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A FIELD TEST OF LEAD-BASED PAINT TESTING TECHNOLOGIES:

TECHNICAL REPORT

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CONTRIBUTING ORGANIZATIONS

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Midwest Research Institute

Midwest Research Institute (MRI) was responsible for initiating the pilot study on schedule, for overall production of the Quality Assurance Project Plan for both the pilot and the full study, for providing input to the design of the study, for planning and supervising the field work, for collecting paint samples, for the laboratory analysis of paint chip samples, and for writing sections of the technical report.

QuanTech

QuanTech (formerly David C. Cox & Associates) was responsible for the design of the study and contributions to the Quality Assurance Project Plan for the pilot and full studies, for participation in field work, for data management and statistical analysis, and for overall production of the technical and summary reports.

U.S. Environmental Protection Agency

The U.S. Environmental Protection Agency (EPA) co-funded the study and was responsible for managing the study, for reviewing study documents, and for arranging for the peer review of the final report. The EPA Project Leader was John Schwemberger. The EPA Work Assignment Managers were John Scalera and John Schwemberger. The EPA Project Officers were Jill Hacker, Samuel Brown, and Janet Remmers. Cindy Stroup was the Branch Chief of the Technical Programs Branch and initiated this study.

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EXECUTIVE SUMMARY

BACKGROUND

This study was undertaken by the U.S. Environmental Protection Agency (EPA) and the U.S. Department of Housing and Urban Development (HUD) to collect information needed for the development of federal guidance on testing paint for lead. Prior to this study, lead testing information was inadequate as little formal evaluation had been done of the various field testing methodologies.

The impetus for this study came from the passage of Title X (Section 1017 of the Residential Lead-Based Paint Hazard Reduction Act of 1992), which mandated that the federal government establish guidelines for lead-based paint hazard evaluation and reduction. This study was designed to produce the type of detailed information EPA and HUD needed in order to respond to that mandate, and focused on two field technologies that are used for testing for lead in paint: portable X-ray fluorescence (XRF) instruments and chemical test kits. A pilot study was conducted during March and April 1993 in Louisville, Kentucky. The full study was conducted from July through October 1993 in Denver, Colorado and Philadelphia, Pennsylvania.

This is the full technical report of the study. The summary report, entitled *A Field Test of Lead-Based Paint Testing Technologies: Summary Report* (EPA 747-R-95-002a), contains an overview of the results from the study. Both reports are available from the National Lead Information Center Clearinghouse (1-800-424-LEAD).

TECHNOLOGIES EVALUATED

This study evaluated XRF instruments and chemical test kits. XRF instruments measure lead in paint by directing high energy X-rays and gamma rays into the paint, causing the lead atoms in the paint to emit X-rays which are detected by the instrument and converted to a measurement of the amount of lead in the paint. Chemical test kits detect the presence of lead in paint by a chemical reaction that occurs when chemicals in the kit are

exposed to lead. This reaction causes a color change to occur if lead is present in the paint.

Laboratory spectroscopic analysis of paint samples was conducted to determine the actual levels of lead in the paint. The laboratory results were used as a benchmark for comparison to the XRF and test kit results.

STUDY OBJECTIVES

The overall study goal was to collect information about field measurement methodologies sufficient to allow EPA and HUD to establish guidance and protocols for lead hazard identification and evaluation. In order to achieve that goal, the study had to be designed and conducted with sufficient rigor and appropriate quality assurance.

To ensure adequacy of the resulting data, six specific study objectives were developed: three primary and three secondary. The results are presented in this report in two ways: overall conclusions and testing recommendations are made in light of the overall study goal, and results are provided in terms of the specific study objectives.

The three primary study objectives were: (1) to characterize the performance (precision and accuracy) of portable XRF instruments under field conditions; (2) to evaluate the effect on XRF performance of interference from the material (the substrate) underlying the paint; and (3) to characterize the relationship between test kit results and the actual lead level in the paint (operating characteristic curves).

The three secondary study objectives were: (4) to understand XRF behavior in the field through the investigation of XRF measurements that were very different than their corresponding lab result; (5) to evaluate field quality assurance and control methods; and (6) to investigate the variability of lead levels in the paint within the study sampling locations.

FIELD TESTING

Three primary concerns of the field testing portion of the study were consistency, real world comparability, and quality control. Due to the differences among the three measurement methods: XRF, test kits, and laboratory analysis, field testing approaches necessarily varied somewhat. In order to ensure consistency, testing was standardized as much as possible. A template was designed for test locations throughout the study housing units, and the different measurement methods were systematically assigned to consistent test locations within the template. This approach ensured results could be compared across different test locations and measurement methods.

At each test location, chemical test kits were tested first. The individuals who did the field testing of the test kits were selected to represent typical homeowners who might purchase test kits for their personal use. That is, they did not have any specific scientific background nor prior training. To further replicate “real world” use, the test kits were rotated among the testers during the study. One of the test kits was an exception to this. It was a kit which is only used by state-certified inspectors. For that kit, a state-certified inspector was brought in and that particular kit was not included in the kit rotation. After each tester completed a test location, the used area of the template was covered to prevent subsequent testers from observing the results obtained by prior testers.

Once test kit testing was finished, paint samples were taken. Paint was removed from a specified location on the template and sent to a laboratory for spectroscopic analysis. A modified NIOSH method 7082 was followed with all appropriate quality control samples including laboratory and field duplicates.

XRF testing was the final step in the field portion of the study. It was conducted by trained and licensed XRF instrument operators employed by independent testing companies. XRF testing was carried out on the portions of the templates designated for this purpose. A number of quality control procedures were employed, including the use of National Institute of Standards and Technology (NIST) Standard Reference Material (SRM) paint films. The NIST SRM paint film is a thin layer of paint with a known level of lead enclosed between two layers of plastic. A portion of the template was scraped bare of paint, revealing the material underneath the paint, the substrate, which was either

brick, concrete, drywall, metal, plaster or wood. The NIST SRM paint film was placed on the bare substrate and a reading was taken in order to determine if the substrate interfered with the XRF reading. In addition, blocks of known substrate materials, called control blocks, were utilized in the field. The NIST SRM paint film was placed on the appropriate block and XRF readings taken in order to determine if control block substrates could be surrogates for the substrates underlying the painted areas tested.

STUDY RESULTS:

Laboratory Analysis Results

There were two key results of the laboratory analyses. First, laboratory analysis results exhibited a wide range of lead levels with a distribution similar to that reported in the 1990 HUD National Survey of Lead-Based Paint in Housing. Second, lead levels appear to vary significantly across the same painted surface.

Two federal thresholds have been established to define lead-based paint on painted architectural components. If paint is found to contain lead equal to or greater than these thresholds, it is characterized as lead-based paint. The federal threshold in milligrams lead per unit area is 1.0 mg/cm^2. The federal threshold in percent lead by weight is 0.5%. Approximately 20% of the samples analyzed in this study were equal to or greater than the federal threshold of 1.0 mg/cm^2, while 29% were equal to or greater than the federal threshold of 0.5% lead. A rough numerical equivalence between results reported as mass of lead per unit area (mg/cm^2) and as percent lead by weight (%) was found in the study data. That is, 1.0 mg/cm^2 lead was found to be roughly equivalent to 1% lead by weight.

The variability of a set of test results is the extent to which the results in the set differ from one another. The standard deviation is a statistical measure of the extent that actual test results tend to spread about an average value. The typical relative standard deviation for laboratory analytical measurements in the study samples was 13%. Variability between field duplicate samples, taken nine inches apart at a subset of test locations, was much larger, between 30% - 60%, indicating significant variability in lead levels across the same painted

surface. The statistical analysis of the data took variability in lead levels into account.

Chemical Test Kit Results

The primary result of the test kit evaluation is that they varied widely in their performance in classifying paint against either the 1.0 mg/cm^2 or 0.5% threshold. No single kit achieved a low rate of both false positive and false negative results and their performance varied across substrates.

A false negative result occurs when the kit fails to detect the presence of lead in paint equal to or greater than the federal threshold, but in fact, the paint is shown by laboratory analysis to contain lead equal to or greater than the threshold. Similarly, a false positive result occurs when the kit detects lead equal to or greater than the federal threshold, but laboratory analysis shows that the paint does not contain lead equal to or greater than the threshold.

No kit in the study achieved low rates of both false positive and false negative results. Two out of six kits were prone to false negative results. Negative test results obtained with these two kits do not necessarily indicate the absence of lead. The other four kits had a tendency to produce false positive results, even at levels of lead well below the federal thresholds.

Further, the performance of the test kits varied with different types of substrates. Most kits usually produced a positive result on at least one substrate, even for very low lead levels. This suggests positive interferences with the chemicals in the kits. On the other hand, some test kits demonstrated negative interferences on some substrates, as indicated by not always giving a positive result for high levels of lead.

XRF Results

The primary result of the XRF testing is that K-shell instruments were often effective in classifying paint samples against the federal threshold of 1.0 mg/cm^2, when using an inconclusive classification range, laboratory confirmation, and

substrate correction, as needed. Generally, L-shell instruments had extremely high false negative rates, making them ineffective in classifying paint against the 1.0 mg/cm^2 threshold.

In this study, measurement bias, or bias, is the tendency of a set of test results to be either greater or less than the laboratory measurements of the lead content of the paint. If test results tend to be greater than the laboratory results, they are said to exhibit positive bias. If the test results tend to be less than the laboratory results, they exhibit negative bias. Results of tests using XRF instruments showed both positive and negative bias. Biases of the K-shell XRF instruments were strongly dependent on the underlying substrate. One K-shell instrument exhibited much less bias than the other XRF instruments. L-shell instruments generally had large negative biases at the 1.0 mg/cm^2 threshold that were usually independent of the substrate.

Substrate correction, using NIST SRM readings on either the scraped substrates or the control blocks, did not reduce bias for L-shell instruments. For K-shell instruments, results were mixed. Control block correction reduced bias for two instruments on some substrates. Correction using NIST SRM readings on the scraped substrate was effective for two instruments on most substrates, and for another instrument on some substrates.

The variability of the results from each XRF instrument was estimated by calculating a standard deviation. The results of most K-shell instruments exhibited high variability at the federal threshold of 1.0 mg/cm^2. The variability in the results from the L-shell instruments was significantly lower than that of K-shell instruments.

Despite their generally high variability and bias, K-shell instruments were often effective in classifying the paint samples in this study against the federal threshold of 1.0 mg/cm^2 when using an inconclusive classification range of 0.4 to 1.6 mg/cm^2 with mandatory laboratory confirmation. Without using an inconclusive range and laboratory confirmation, only two of the K-shell instruments had both false positive and false negative rates below 10%.

Generally, L-shell instruments had extremely high false negative rates. One L-shell instrument had moderate to high

false negative rates, depending on the width of the inconclusive range, but still gave low readings on some samples with high levels of lead.

OVERALL RECOMMENDATIONS FOR TESTING

XRF Instrument Conclusions

The primary XRF conclusion is that testing by K-shell XRF instruments, with laboratory confirmation of inconclusive XRF results, and with substrate correction in cases where this is effective in reducing bias, is a viable way to test for lead-based paint. This approach can produce satisfactory results for classifying the paint on architectural components using the federal threshold of 1.0 mg/cm^2.

Further, the variability found in paint samples located approximately nine inches apart supports the conclusion that the most effective method of XRF testing of a single architectural component, such as a window sill, wall, or door, is to obtain readings at different points on the component, and compute their average. This would replace the current practice which is to average a number of XRF readings taken at a single point.

Chemical Test Kit Conclusions

The conclusion of this study is that test kits should not be used for lead paint testing. Test kits cannot determine the extent of lead-based paint in a home and the need for protecting the occupants, especially when repairs or renovations are carried out. Homeowners and renters cannot be confident that test kits will discriminate accurately between lead-based paint and non-lead based paint. They should not make decisions on repairs, renovations or abatements based on test kit results.

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1 DESCRIPTION OF THE STUDY

1.1 BACKGROUND

Lead-based paint (LBP) in older housing, especially lead-based paint in poor condition, is recognized as a major cause, both direct and indirect, of elevated blood lead levels in children between 1 and 6 years old. Exposure to lead in paint can come from the paint chips themselves, from dust caused by abrasion of paint on friction surfaces, or from chalking of exterior paint. The Lead-Based Paint Poisoning Prevention Act of 1971, as amended by the Housing and Community Development Act of 1987, established 1.0 mg/cm^2 as the federal threshold requiring abatement of lead-based paint in public and Indian housing developments nationwide. To implement this legislation, Congress required the U.S. Department of Housing and Urban Development (HUD) to complete testing for lead-based paint in all public and Indian housing by December, 1994. In response to this requirement, HUD, with substantial input from the Environmental Protection Agency (EPA), published interim guidelines for testing and abatement of LBP in public and Indian housing in April, 1990. At the time the HUD Guidelines were published, the research conducted to evaluate the performance of X-ray fluorescence (XRF) instruments and chemical test kits in detecting LBP at or above the federal threshold was limited. The recommended approach was to perform XRF testing, with laboratory confirmation of inconclusive results. The Guidelines recommended that test kits should not be used as a primary testing method. Federal guidance documents available from the National Lead Information Center Clearinghouse also did not recommend the use of test kits by homeowners or renters.

The Residential Lead-Based Paint Hazard Reduction Act of 1992 ("Title X") mandated the evaluation and reduction of lead-based paint hazards in the nation's existing housing. Title X also established 0.5% lead as an alternative to the 1.0 mg/cm^2 threshold. Section 1017 of Title X required HUD to develop guidelines for federally-supported lead-based paint hazard evaluation and reduction activities. HUD is complying with this requirement by preparing a major revision and expansion of the 1990 Guidelines. To support the testing and inspection portion of the revised Guidelines, EPA and HUD funded this field study of technologies used to detect and measure lead in paint. It is the

first comprehensive evaluation of XRF instruments and test kits under field conditions.

1.2 STUDY OBJECTIVES

The overall study goal was to collect information about field measurement methodologies sufficient to allow EPA and HUD to establish guidance and protocols for lead hazard identification and evaluation. In order to achieve that goal, the study had to be designed and conducted with sufficient rigor and appropriate quality assurance.

To ensure adequacy of the resulting data, six specific study objectives were developed: three primary and three secondary. The results are presented in this report in two ways: overall conclusions and testing recommendations are made in light of the overall study goal, and results are provided in terms of the specific study objectives.

The three primary study objectives were: (1) to characterize the performance (precision and accuracy) of portable XRF instruments under field conditions; (2) to evaluate the effect on XRF performance of interference from the material (the substrate) underlying the paint; and (3) to characterize the relationship between test kit results and the actual lead level in the paint (operating characteristic curves).

The three secondary study objectives were: (4) to understand XRF behavior in the field through the investigation of XRF measurements that were very different than their corresponding lab result; (5) to evaluate field quality assurance and control methods; and (6) to investigate the variability of lead levels in the paint within the study sampling locations.

This study differs from previous studies conducted to measure lead in paint because the study included a larger number of samples and more diverse testing locations, and was designed so that test results obtained at different locations could be compared. Paint from a total of 1,290 locations in 22 housing units in three cities was tested. The tested locations were free from identifiable biases and represent a variety of paint types,

substrates, architectural designs, and lead levels in paint. The study was designed to evaluate field testing technologies used to identify lead-based paint that were commercially available or were working prototypes as of June, 1993. These technologies included six types of XRF instruments and six chemical test kits. Spectroscopic laboratory analysis was used to verify results obtained by the XRF instruments and chemical test kits.

1.3 APPROACH

The study began in March 1993 in Louisville, Kentucky, with a pilot conducted at a vacant public housing development built in 1937. Testing was conducted at 100 locations in 4 units in 2 buildings. The pilot had several objectives. First, it was important to determine the feasibility of collecting large numbers of paint samples in the field while ensuring the quality of the samples, and to develop and test a system for labelling and tracking the samples. Removal of paint with a heat gun and paint scraper proved to be a successful technique. A barcode system that labelled and tracked samples was developed and tested. A working system for selecting and marking test locations was developed. The field practicality of the test kits for large testing programs was evaluated. Procedures for monitoring XRF testing and recording of data were developed. Field testing sequences to minimize the potential for variability in XRF results caused by frequent substrate changes were used. Time estimates for all aspects of sample collection and testing were made. The schedule and logistics for the full study were based on these time estimates. A database structure was developed for storing and retrieving study data.

The full study was conducted in two cities, Denver in July and August 1993 and Philadelphia in September and October 1993. Denver and Philadelphia were specifically chosen because housing was available that met study criteria and because the public housing authorities in those cities were willing to work closely with EPA and its contractors. The study tested units from both multifamily housing, where units tend to be quite similar to each other, and from single-family homes. A total of 10 scattered-site single-family homes were tested in Denver; eight were built between 1943 and 1952, while two were older, dating from 1890 and 1905. In Philadelphia, eight units in two buildings in a single multifamily development built in 1942 were tested. Including

those in the pilot study, a total of 1,290 individual test locations on 6 substrate types in the 22 housing units were tested. There were 100 test locations in Louisville, 750 in Denver and 440 in Philadelphia. The breakdown of testing locations by substrate was: 93 brick, 226 concrete, 124 drywall, 217 metal, 242 plaster, and 388 wood substrates.

1.4 TECHNOLOGIES

Chemical test kits detect the presence of lead in paint by a chemical reaction that occurs when chemicals in the kit are exposed to lead. This reaction causes a color change to occur if lead is present in the paint. The test kits in the study represented the range of kits available at the time the study was conducted. Test kits from five different manufacturers were examined in this study: three rhodizonate based kits, two sodium sulfide based kits, and one proprietary kit. Both of the most common types of chemical test kits, rhodizonate based kits and sodium sulfide based kits, were used in the pilot study. The rhodizonate kits included were LeadCheck (also called LeadCheck II) and the sanding and coring versions of Lead Alert; the sodium sulfide kits were Lead Detective and the Massachusetts state-approved kit. The pilot study also included the Lead Zone kit, which utilizes proprietary chemistry. It was expected that the results of the pilot study would be similar for kits based on similar chemistry, that is, rhodizonate or sodium sulfide, so that fewer kits would need to be included in the full study. However, the test results were not similar for kits utilizing similar chemistry, so the same six kits were included in the full study.

Portable XRF instruments direct high energy X-rays and gamma rays into paint. These high-energy rays strike lead atoms, causing electrons to be ejected from their electron orbits, or shells. In a process called fluorescence, other electrons refill the voids left by the ejected electrons, producing X-rays. These X-rays have specific frequencies based on differences in energy between the electron shells which contained the emitted electrons and the electron shells which received the electrons. The amount of X-ray energy emitted at several specific frequencies, in this case called K-shell or L-shell X-ray energy, is measured by detectors on XRF instruments and used to calculate the amount of lead in paint.

XRF instruments are classified by the type of X-ray energy that they detect, K-shell X-rays, L-shell X-rays, or both. K-shell X-rays are more highly penetrating than L-shell X-rays since L-shell X-rays have lower energy. For this reason, K-shell X-rays are more useful for detecting lead in deeper layers of paint. Two of the XRF instruments in this study detected K-shell X-rays, two XRF instruments detected L-shell X-rays, and two instruments detected both K-shell and L-shell X-rays.

Efforts were made to include a representative example of every XRF instrument available at the time of the study. Six types of XRF instruments were in the study. The MAP-3, the Microlead I, and the XK-3 were included because they were the most commonly used instruments for LBP testing when the study began. The X-MET 880 was included because it performed successfully in the pilot study. After completion of the pilot study, all other known manufacturers of XRF instruments or working prototypes were invited to participate in a day of ruggedness testing to determine whether the instruments were portable and could function reliably throughout a full day of field testing. As a result, two additional instruments, the Lead Analyzer and a prototype of the XL, were included in the full study. Since the conclusion of the field portion of the study, new XRF instruments and modified versions of some tested instruments have become commercially available.

The third type of technology in the study was laboratory analysis which was used to verify results obtained by the two field technologies: chemical test kits and XRF instruments. For this study, the laboratory instrument used was an atomic emission spectrophotometer. The laboratory procedure involved dissolving paint samples in acid, then filtering and diluting them. A portion of the dissolved sample was placed in the spectrophotometer and heated to extremely high temperatures by a device inside the spectrophotometer called a high temperature atomizer. At very high temperatures, most of the sample is broken down into individual atoms. Individual atoms absorb and re-emit energy produced by the atomizer. Atoms of different chemical elements re-emit energy at different energy levels. A detector in the spectrophotometer sorts and measures the energy re-emitted by the atoms of different chemical elements. In this way, the amount of energy re-emitted by lead atoms is measured and then used to calculate the amount of lead in the sample. The particular type of spectrophotometer used in this study was an

inductively coupled plasma atomic emission spectrophotometer (ICP). The analytical laboratory results were continually evaluated by using reference materials to assure the accuracy of the laboratory analysis of field samples.

Chemical test kit results were reported as either negative or positive indicating the absence of lead or presence of lead, respectively. XRF instruments and laboratory analysis results were reported as quantitative measures of lead. XRF instruments report their results as mass of lead per unit area (mg/cm^2). Laboratory analysis results were reported both as mass of lead per unit area (mg/cm^2) and percent lead by weight (%).

1.5 FIELD TESTING

Templates were designed for marking test locations in the study housing units so that results could be compared for different test technologies and locations. The most commonly used template, shown in Figure 1-1, was a rectangle 14 inches long and 4 inches wide. For certain locations such as door frames, a thin version of the template, 2 inches by 14 inches, was needed. On the left of the most commonly used template was a square 4 x 4 inches; in the center, a second 4 x 4 inch square was divided into four 2 x 2 inch subsquares; the remaining 6 x 4 inch rectangle on the right of the template was divided into six vertical strips each 1 x 4 inches. One of the 2 x 2 inch subsquares was randomly selected as the location for paint sampling for laboratory analysis. At 10% of locations in the full study, a duplicate paint sample was taken adjacent to the right end of the template for use in assessing variability in the paint lead levels. Following paint sampling, the remainder of the center 4 x 4 inch square was scraped to remove all remaining paint. It was then used for taking XRF measurements on bare substrates both with and without the standard reference material paint films (SRM 2579) developed by the National Institute of Standards and Technology (NIST). The NIST SRM paint film is a thin layer of paint with a known level of lead enclosed between two layers of plastic. The 4 x 4 inch square on the left of the template was used for XRF measurements on paint. The six 1 x 4 inch strips were randomly assigned as testing locations for the six chemical test kits. Each of the testing locations in the study was selected and marked by the field statisticians using

the template and an indelible ink marker. Each test location was numbered for identification and sample tracking.

The first step in the full study was to test the six chemical test kits. Testers for five of the six test kits were individuals without any special scientific background or prior training. They were selected to represent typical homeowners who might purchase kits for their personal use. The testers were trained by field supervisors to ensure that study protocols were followed. The training did not provide the testers with knowledge about test kit operation beyond the information contained in the manufacturer's instructions. These five kits were rotated among the testers during the study. The sixth kit, tested by a state-certified inspector, was not part of the kit rotation. After each tester had completed the testing at a location, the strip of the test location where the color change could be observed was taped over to prevent subsequent testers from knowing the result of the test.

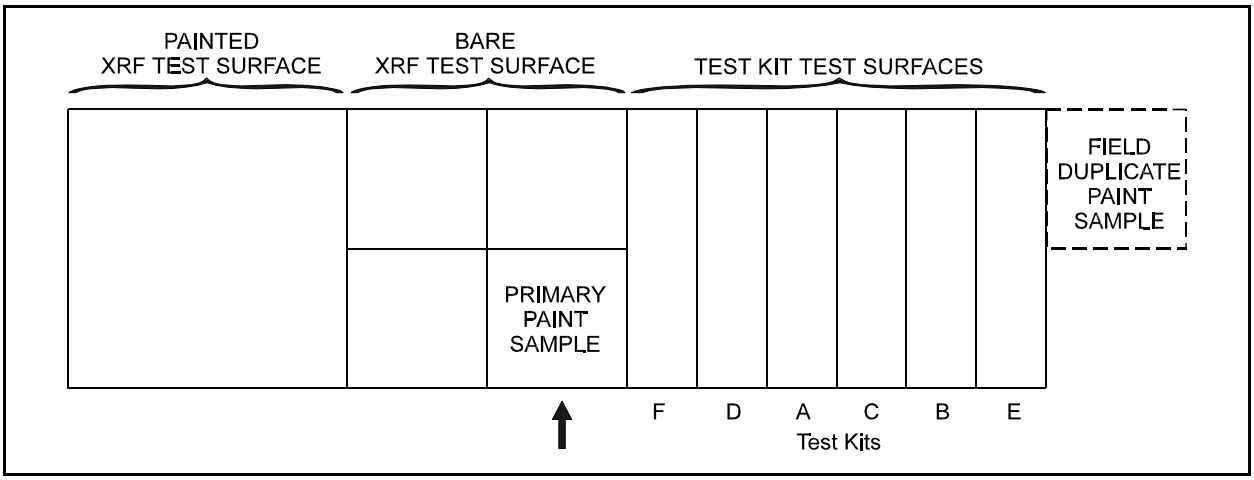

Figure 1-1. Full study template.

After test kit testing was completed, paint chip samples were taken and sent to the laboratory for ICP spectroscopic analysis. Paint samples were homogenized by grinding to a powder, and, if necessary, subsampled prior to analysis. Subsampling was necessary because the total mass of many samples was too large for a single laboratory analysis.

The third and final step in the field study was XRF testing. It was conducted by trained and licensed XRF instrument operators employed by independent testing companies. Within each unit, test locations from each substrate type were tested as a group. For example, all locations on metal substrates were tested, then all locations on wood substrates were tested, etc. This was done to minimize the potential for XRF variability caused by repeated substrate changes. However, the order of substrates tested within a unit was varied. Quality control checks were also performed on six control blocks, each composed of a different substrate, combined with the NIST SRM paint films. To ensure that the testing protocol was followed exactly, and to ensure accurate recording of data, during testing each XRF instrument operator was observed by a full-time monitor who recorded the results and reported to a field supervisor.

1.6 REPORT ORGANIZATION

This report is the full technical report of study procedures. Testing recommendations, study conclusions, and study results are presented in chapter 2. Chapter 3 contains

detailed discussions on study design. Chapter 4 presents paint-chip data. Chapter 5 presents analysis of test kit data, including operating characteristic curves for lead testing kits. Chapter 6 is the analysis of XRF testing data. Chapter 7 presents a discussion about data quality assurance and quality control. Chapters 8 and 9 contain the bibliography and glossary, respectively. The appendices contain testing protocols used in this study and other technical issues.

1.7 PEER REVIEW

The technical report on this study was reviewed independently by members of a peer review panel. Comments which are important for interpreting the study results or which had an important impact on the report are discussed below.

A comment from a number of reviewers related to the representativeness of the study paint samples and the fact that the sample was not selected randomly from the national housing stock. Although the sample was not randomly selected, the sample did include different substrate materials, housing components, paint thicknesses, and lead levels. The housing in the study included both single-family homes and multifamily housing. The distribution of lead levels in the study is similar to the distribution in the HUD National Survey of pre-1980 housing.

A comment from the reviewers related to the training received by the individuals who, as representative homeowners or renters, applied the test kits. There were concerns that it would have been more appropriate to have no training to better simulate what a homeowner or renter would encounter. However, the training did not give the individuals in the study any more information beyond what could have been obtained from a careful reading of the kit instructions. The kits were rotated among the testers to reduce the chance of an individual becoming an expert with a single kit. Nevertheless, it is probably fair to say that the training, the availability of on-site supervisors, and the large number of tests performed by the individual testers provided conditions that exceeded what would be typical for a homeowner or renter who purchased a test kit.

A comment was made concerning the impact of spatial variation and laboratory measurement error on the false positive

and false negative rates calculated from the study data. A simulation study was conducted to address this comment and the results included in the final technical report. The simulation study demonstrated that the false positive and false negative rates were robust, and therefore accurately portrayed performance of the technologies in the study. Another reviewer comment on the statistical analysis of paint samples with lead below detection levels led to an improvement in the approach for estimating model parameters.

A number of reviewers commented on the length of the technical report. In response to those comments, a summary report was developed from the technical report to make the information in the technical report accessible to a wider audience.

EPA has established a public record for the peer review under administrative record 142. The record is available in the TSCA Nonconfidential Information Center, which is open from noon to 4 PM Monday through Friday, except legal holidays. The TSCA Nonconfidential Information Center is located in Room NE-B607, Northeast Mall, 401 M Street SW, Washington, D.C.

2 STUDY CONCLUSIONS, TESTING RECOMMENDATIONS, AND STUDY RESULTS

This section provides conclusions and recommendations for testing as well as results from the study. It is divided into three subsections. Section 2.1 contains conclusions and recommendations for testing for lead-based paint, section 2.2 contains a summary of results organized by study objectives, and section 2.3 contains detailed study results. The conclusions, recommendations, and results are based on the samples and data collected in this study, and are specific to the laboratory analysis method, chemical test kits, and XRF instruments used.

2.1 CONCLUSIONS AND RECOMMENDATIONS FOR TESTING

2.1.1 XRF Instrument Conclusions

The primary XRF conclusion is that testing using K-shell XRF instruments, with laboratory confirmation of inconclusive XRF results, and with substrate correction in cases where this is effective in reducing bias, is a viable way to test for lead-based paint. This approach can be expected to produce satisfactory results for classifying the paint on architectural components as either above or below the federal threshold of 1.0 mg/cm^2.

Currently, a common practice is to average a number of readings taken at a single point on an architectural component. The study demonstrated that the most effective method of XRF testing is to obtain readings at different points on the component and compute their average. This recommendation is supported by the variability found in paint samples located approximately nine inches apart, and evidence that a single XRF reading at one point provided almost as much information as an average of three XRF readings at the same point.

2.1.2 Chemical Test Kit Conclusions

The conclusion of this study is that test kits should not be used for lead paint testing. Test kits cannot determine the extent of lead-based paint in a home and the need for protecting

the occupants, especially when repairs or renovations are carried out. Homeowners and renters cannot be confident that test kits will discriminate accurately between lead-based paint and non-lead based paint. They should not make decisions on repairs, renovations or abatements based on test kit results.

2.2 RESULTS FOR STUDY OBJECTIVES

2.2.1 Precision and Accuracy of XRF Instruments

The first primary objective of this study was to characterize the precision and accuracy of XRF instruments on common substrates under field conditions. The results of the study showed that most K-shell instruments exhibited relatively high variability and a high degree of bias at lead levels close to the federal threshold of 1.0 mg/cm^2. Nevertheless, K-shell XRF instruments reliably classified the paint samples in this study vis-a-vis the federal threshold of 1.0 mg/cm^2, provided a suitable inconclusive range and substrate correction (where appropriate) were used.

Test results using L-shell instruments generally exhibited large negative biases which increased with the lead level in the paint. Bias for L-shell instruments was usually substantial at 1.0 mg/cm^2 lead. L-shell instruments were less variable than K-shell instruments. As a consequence of the large negative biases, L-shell instruments exhibited a high rate of false negative results when classifying paint using the 1.0 mg/cm^2 threshold. When an inconclusive range was added, L-shell instruments, with one exception, still had high rates of false negatives. The one exception exhibited reductions in the rate of false negatives as the inconclusive range was lengthened.

2.2.2 Substrate Interference

The second primary objective of the study was to evaluate the effect on the performance of XRF instruments of interference or bias attributable to the underlying substrate and, hence, to evaluate the utility of different approaches for adjusting XRF readings for this bias. The results of the study showed that biases of most K-shell instruments were strongly substrate dependent. Test results using L-shell instruments generally

exhibited large negative biases at the 1.0 mg/cm^2 threshold that were usually independent of the substrate.

Substrate correction obtained using readings on NIST SRM paint films placed on test location areas scraped bare of paint reduced bias for two of the K-shell instruments, and for a third on metal and wood substrates. The already low bias of the fourth K-shell instrument's results was unchanged. Substrate correction using NIST SRM paint films over control blocks was effective in reducing bias for one K-shell instrument, and somewhat effective for a second on plaster, concrete and metal. No method of substrate correction reduced the bias of L-shell readings.

2.2.3 Large XRF Errors

A secondary objective of the study was to investigate large errors in the XRF measurements, i.e., measurements that were very different than their corresponding lab results. The results of the study showed that the incidence of large XRF errors was very low (0.6%). Moreover, many of the large errors occurred for several instruments at the same test location. This suggests a common cause other than mere erratic behavior on the part on any single XRF instrument.

2.2.4 Field Quality Assurance and Quality Control Methods

Another secondary objective of the study was to evaluate field quality assurance and quality control methods. The study results showed that NIST SRM readings on control blocks were unable to predict XRF instrument performance on painted components in most cases. In particular, the study results showed that erratic behavior in XRF readings taken on control blocks was not necessarily predictive of similarly erratic behavior on actual paint samples. Finally, with the exception of two K-shell instruments used on some substrates, substrate correction using readings on NIST SRM paint films placed on control blocks of substrate materials brought to the site was not effective in reducing biases of readings attributable to substrate interference.

2.2.5 Operating Characteristic Curves for Test Kits

The third primary objective of the study was to estimate the operating characteristic curve for each test kit under field conditions. The results of the study showed that the probability of a positive classification when the sample's lead level was equal to the federal thresholds varied depending on the kit and substrate and that high levels of lead would not always be detected by some test kits. Furthermore, there were numerous cases of positive test results at lead levels well below the federal thresholds. None of the test kits used in this study demonstrated low rates of both false positive and false negative results when compared to laboratory analytical results using the federal thresholds, 1.0 mg/cm^2 and 0.5%.

2.2.6 Variability of Lead Levels in Paint

The third secondary objective of the study was to investigate the variability of lead levels in paint using laboratory measurements of field duplicate samples. The study results showed that the typical relative standard deviation for laboratory analytical measurements in the study samples was 13%. Variability between field duplicate samples was much larger, between 30% - 60% at one standard deviation, indicating significant variability in lead levels between paint samples approximately 9 inches apart. This variability in lead levels within single architectural components, called spatial variability, was the primary cause of variability in the paint samples.

2.3 DETAILED STUDY RESULTS

2.3.1 Lead Levels in the Study Samples

1. **Of the 1,290 paint samples collected and analyzed in the laboratory in this study, approximately 20% contained lead at a level equal to or greater than 1.0 mg/cm^2, one of the federal thresholds for defining LBP on painted surfaces. Approximately 29% of the samples contained lead equal to or greater than 0.5% by weight, the other federal threshold for LBP on painted surfaces.**

Lead levels in the samples were reported by the laboratory as mass per unit area (mg/cm^2 lead) and percent lead by weight (%). Table 2-1 presents a cross-tabulation of lead levels expressed in mg/cm^2 and percent lead by weight. The arithmetic mean lead level in the study samples was 1.17 mg/cm^2 (1.12%). The median lead level of the study samples was 0.20 mg/cm^2 (0.20%). The 25th and 75th percentiles were 0.03 mg/cm^2 (0.05%) and 0.62 mg/cm^2 (0.72%). The minimum and maximum values were 0.0001 mg/cm^2 (0.0004%) and 37.29 mg/cm^2 (34.56%).

Table 2-1. Cross-Tabulation of Paint Sample Lead Levels in mg/cm^2 Lead and Percent Lead by Weight.

Percent Lead by Weight	**mg/cm^2 Lead**			
	< 0.5	0.5 - 1.0	≥ 1.0	**Totals**
< 0.5	874	42	2	918
0.5 - 1.0	36	44	14	94
≥ 1.0	16	25	237	278
Totals	926	111	253	1,290

2. **For the paint samples, lead levels expressed in mg/cm^2 and lead levels expressed in percent lead by weight were roughly equivalent, as shown by the distribution in Table 2-1. A level of 1.0 mg/cm^2 was roughly equivalent to 1.0% by weight and a level of 0.5% by weight was roughly equivalent to 0.5 mg/cm^2.**

The overall average ratio between the two types of measurement units for the 1,290 primary paint samples analyzed in the laboratory was 1.00. In 80% of the samples, the ratio was between 0.25 and 2.34. A regression plot of results expressed in percent lead by weight (%) versus mass of lead per unit area (mg/cm^2) using a logarithmic scale showed good agreement between the two types of measurement units (R^2 = 0.91), with the following relationship:

$$\textit{PERCENT LEAD} = 0.96 \times (\textit{AREA LEAD})^{0.85}, \text{ where}$$

PERCENT LEAD = percent lead by weight (%) and

AREA LEAD = mass of lead per unit area (mg/cm^2).

This relationship suggests that 0.5% lead is roughly equivalent to 0.5 mg/cm^2 lead, while 1.0 mg/cm^2 lead is roughly equivalent to 1.0% lead. This demonstrates that the threshold of 1.0 mg/cm^2 lead is typically less stringent than 0.5% lead.

2.3.2 XRF Instruments

1. **Most K-shell instruments exhibited relatively high variability, even for paint with low levels of lead. The amount of variability was sometimes related to the level of lead in the sample.**

Table 2-2 shows estimated standard deviations for each substrate for results using the K-shell XRF instruments at lead levels of 0.0 mg/cm^2 and 1.0 mg/cm^2, for a single 15-second (nominal) reading taken on the painted surface of each test location. For XRF instrument results that showed significant variation between instruments and/or cities in the study, a range of values for the standard deviation is also presented. In these cases, the single value in the table represents the single instrument, or a group of similar instruments, with the largest number of readings taken. These estimated standard deviations take into

Table 2-2. Estimated Standard Deviation at 0.0 mg/cm^2 and 1.0 mg/cm^2 Lead for One Nominal 15-Second Paint Reading for K-Shell XRF Instruments, by Substrate.

SUBSTRATE	LEAD ANALYZER K-SHELL		MAP-3 K-SHELL		MICROLEAD I		XK-3	
	0.0 mg/cm^2	1.0 mg/cm^2	0.0 mg/cm^2	1.0 mg/cm^2	0.0 mg/cm^2	1.0 mg/cm^2	0.0 mg/cm^2	1.0 mg/cm^2
Brick	0.17	0.23	0.93	0.93	0.59	0.55	0.60	0.60
Concrete	0.11	0.37	0.90	1.00	0.61 (0.48-1.24)	0.72 (0.48-1.31)	0.64 (0.51-0.85)	0.64 (0.51-0.85)

Drywall	0.08	0.35	0.38	0.38	0.34 (0.34-0.53)	0.34 (0.34-0.53)	0.36 (0.21-0.36)	0.56 (0.55-0.56)
Metal	0.18	0.41	0.37	0.55	0.62 (0.37-0.81)	0.68 (0.55-0.81)	0.52 (0.34-0.70)	1.06 (0.49-1.63)
Plaster	0.14	0.24	0.81	0.87	0.55 (0.37-1.01)	0.64 (0.46-1.01)	0.55 (0.40-0.55)	0.63 (0.40-0.81)
Wood	0.08	0.43	0.49	0.67	0.62 (0.50-1.06)	0.92 (0.55-1.06)	0.49 (0.25-0.51)	0.69 (0.44-1.15)

Ranges presented for XRFs demonstrating significant variability between different instruments.

Table 2-3. Estimated Standard Deviation at 0.0 mg/cm^2 and 1.0 mg/cm^2 Lead for One Nominal 15-Second Reading on Control Blocks for K-Shell XRF Instruments, by Substrate.

SUBSTRATE	LEAD ANALYZER K-SHELL		MAP-3 K-SHELL		MICROLEAD I		XK-3	
	0.0 mg/cm^2	1.0 mg/cm^2	0.0 mg/cm^2	1.0 mg/cm^2	0.0 mg/cm^2	1.0 mg/cm^2	0.0 mg/cm^2	1.0 mg/cm^2
Brick	0.11	0.24	0.72	0.61	0.48 (0.28-0.76)	0.40 (0.26-0.61)	0.33	0.41
Concrete	0.11	0.24	0.64	0.67	0.38 (0.31-0.51)	0.50 (0.41-0.68)	0.41	0.50
Drywall	0.07	0.19	0.28	0.34	0.29 (0.21-0.42)	0.29 (0.25-0.44)	0.32	0.45
Metal	0.15	0.22	0.21	0.25	0.27 (0.22-2.39)	0.36 (0.21-2.14)	0.38	0.47
Plaster	0.09	0.20	0.69	0.57	0.49 (0.33-0.66)	0.47 (0.30-0.69)	0.50	0.70
Wood	0.03	0.18	0.24	0.24	0.26 (0.24-2.08)	0.33 (0.23-2.25)	0.39	0.43

Ranges presented for XRFs demonstrating significant variability between different instruments.

account several sources of variability in addition to instrumental variation. These include site-specific factors such as the substrate composition and the age and thickness of the paint. The MAP-3, Microlead I, and XK-3 results exhibited similar high levels of variability. The Lead Analyzer's results were significantly less variable than the other three. Generally, the instruments' results showed higher variability at 1.0 mg/cm^2 lead than at 0.0 mg/cm^2. The difference in variability at the two levels was greatest for the Lead Analyzer's results and least for the MAP-3's results. Variability of control block quality control test results was significantly lower than results for field test locations. Table 2-3 is the companion to Table 2-2 for control block test results. The standard deviation at 0.0 mg/cm^2 was estimated using XRF test results on the bare control blocks. The standard deviation at 1.0 mg/cm^2 was

estimated using XRF test results from control blocks covered with the NIST SRM 2579 paint film that has a lead level of 1.02 mg/cm^2. As in Table 2-2, the Lead Analyzer's results were less variable than the results of the other three instruments. For tests on control blocks, the Lead Analyzer's results were more variable at 1.0 mg/cm^2 than at 0.0 mg/cm^2. However, the other three instruments' results showed similar variability on the control blocks at the two levels, 0.0 and 1.0 mg/cm^2.

2. **Biases of most K-shell instruments were strongly substrate dependent.**

Bias of an XRF instrument is defined as the average difference between XRF readings and the true lead level in the paint. Table 2-4 shows biases of the K-shell XRF instruments on the field samples. The results of the Lead Analyzer exhibited low bias on all substrates. The MAP-3's results showed negative bias on brick, concrete, and plaster; positive bias on metal; and low bias on wood and drywall with the exception of wood at 1.0 mg/cm^2.

Table 2-4. Bias at 0.0 mg/cm^2 and 1.0 mg/cm^2 Lead for One Nominal 15-Second Reading for K-Shell XRF Instruments, by Substrate.

SUBSTRATE	LEAD ANALYZER K-SHELL		MAP-3 K-SHELL		MICROLEAD I		XK-3	
	0.0 mg/cm^2	1.0 mg/cm^2	0.0 mg/cm^2	1.0 mg/cm^2	0.0 mg/cm^2	1.0 mg/cm^2	0.0 mg/cm^2	1.0 mg/cm^2
Brick	0.08	-0.21	-0.60	-0.80	0.10	-0.33	0.86	0.88
Concrete	0.02	-0.01	-0.66	-0.45	0.28 (-0.03-0.89)	0.38 (0.01-1.23)	1.08 (0.66-1.84)	1.75 (0.23-2.57)
Drywall	-0.02	0.18	0.01	-0.12	0.02 (0.00-0.66)	0.22 (0.16-1.79)	-0.33 (-0.33-0.25)	-0.09 (-0.09-0.18)
Metal	0.06	0.02	0.33	0.42	0.35 (-0.42-1.08)	0.45 (-0.17-1.36)	0.45 (0.26-1.48)	0.86 (0.81-1.69)
Plaster	0.03	-0.11	-0.68	-0.55	0.01 (-0.09-0.22)	0.06 (-0.32-0.18)	0.54 (0.38-1.68)	0.57 (0.18-1.63)
Wood	0.01	0.28	-0.05	0.36	0.00 (0.00-0.60)	0.43 (0.18-0.90)	-0.07 (-0.07-0.93)	0.35 (0.31-1.23)

Ranges presented for XRFs demonstrating significant variability between different instruments.

Table 2-5. Bias at 0.0 mg/cm² and 1.0 mg/cm² Lead for One Nominal 15-Second Reading on Control Blocks for K-Shell XRF Instruments, by Substrate.

SUBSTRATE	LEAD ANALYZER K-SHELL		MAP-3 K-SHELL		MICROLEAD I		XK-3	
	0.0 mg/cm²	1.0 mg/cm²	0.0 mg/cm²	1.0 mg/cm²	0.0 mg/cm²	1.0 mg/cm²	0.0 mg/cm²	1.0 mg/cm²
Brick	0.05	0.08	-1.18	-0.05	0.47 (-0.10-0.51)	0.45 (-0.31-0.54)	0.97	1.10
Concrete	-0.01	0.06	-1.20	-0.18	0.57 (0.15-1.43)	0.70 (0.25-1.59)	0.89	1.00
Drywall	-0.01	0.06	-0.10	0.04	0.03 (-0.62-0.14)	0.12 (-0.56-0.18)	0.17	0.48
Metal	-0.01	0.11	0.23	0.18	-0.34 (-0.82-2.25)	-0.35 (-0.84-2.00)	1.10	1.34
Plaster	-0.03	0.05	-1.38	-0.64	0.45 (0.06-1.13)	0.40 (0.09-1.02)	0.83	0.83
Wood	-0.00	0.04	-0.27	-0.14	0.15 (-0.22-1.57)	0.18 (-0.05-1.47)	0.25	0.49

Ranges presented for XRFs demonstrating significant variability between different instruments.

The Microlead I's results were mostly positively biased, but with large differences between individual instruments. The XK-3's results showed large positive biases except on wood and drywall, and also exhibited substantial variation between individual instruments. Table 2-5 shows biases for the K-shell instruments' results, estimated using control block readings. For the Lead Analyzer, control block biases were very small. For the MAP-3, the control block result biases were generally of the same sign, positive or negative, as the field sample result biases, but the magnitudes were very different. For the Microlead I, sporadic agreement existed between control block and field sample result biases. For example, the control block results showed negative bias on metal, while the field

sample result biases. For example, the control block results showed negative bias on metal, while the field sample results showed a positive bias on the same substrate. For the XK-3, the control block result biases usually tracked the field sample result biases.

3. **With the exception of the XL prototype, test results using L-shell instruments exhibited large negative biases at the 1.0 mg/cm² threshold. However, test results using L-shell instruments were less variable than results obtained using K-shell instruments.**

   Table 2-6 shows estimated biases of field sample results using L-shell instruments at 0.0 mg/cm² and 1.0 mg/cm². The instruments' results show little bias at 0.0 mg/cm². However, large negative biases, typically between -0.7 and -0.9 mg/cm², at 1.0 mg/cm² lead, are shown for all L-shell instruments' results except those obtained using the XL.

   Standard deviations were usually 0.2 mg/cm² or less for field sample test results at both 0.0 and 1.0 mg/cm² lead, although the MAP-3's L-shell results showed slightly higher variability than this on metal. Variability of control block results was significantly lower for all L-shell instruments compared to K-shell instruments' results.

Table 2-6. Bias at 0.0 mg/cm² and 1.0 mg/cm² Lead for One Nominal 15-Second Reading for L-Shell XRF Instruments, by Substrate.

SUBSTRATE	LEAD ANALYZER L-SHELL		MAP-3 L-SHELL		XL		X-MET 880	
	0.0 mg/cm²	1.0 mg/cm²	0.0 mg/cm²	1.0 mg/cm²	0.0 mg/cm²	1.0 mg/cm²	0.0 mg/cm²	1.0 mg/cm²
Brick	0.01	-0.77	0.01	-0.88	0.11	-0.40	0.03	-0.74
Concrete	0.01	-0.84	-0.14	-0.94	0.07	-0.15	0.05	-0.89
Drywall	-0.01	-0.70	-0.12	-0.62	0.08	-0.63	0.04	-0.74
Metal	0.01	-0.79	0.04	-0.69	0.07	-0.10	0.11	-0.77

Plaster	0.002	-0.80	-0.12	-0.96	0.08	-0.26	0.05	-0.88
Wood	-0.02	-0.74	-0.08	-0.65	0.06	-0.30	0.04	-0.70

4. **The XL results showed smaller biases at 1.0 mg/cm² than results of the other L-shell instruments, but still showed large negative biases at higher lead levels.**

   Biases of the XL's results at 1.0 mg/cm² lead range from -0.10 to -0.63 mg/cm². There was some variation in bias between different XL machines on metal and wood at 1.0 mg/cm². The instrument's results showed large negative biases at higher lead levels. For example, it read 1.0 mg/cm² or less on 26% of the samples with lead levels of 10.0 mg/cm² or greater. The XL instruments used in this study were prototype models.

5. **Substrate correction obtained using readings for NIST SRM paint films placed on test location areas scraped bare of paint reduced bias for results using the Microlead I and the XK-3, and for the MAP-3 K-shell instrument results on metal and wood substrates. The already low bias of the Lead Analyzer's K-shell results was unchanged.**

   Two methods of substrate correction using NIST SRM paint films placed on the bare substrate were analyzed. In the first method, called "full" correction, readings were taken at each individual test location after the NIST SRM paint film was placed on the bare area of the substrate. These readings provided an offset value used to correct the paint sample readings taken at that location. The second method, called "average" correction, used the average of all readings taken after the NIST SRM paint film was placed on the bare area at test locations of the same substrate in the entire dwelling unit. These average readings provided an offset value used to correct paint sample readings taken on the same substrate in a dwelling unit. Full correction is not a practical method, while average correction approximates the method recommended in the 1990 HUD Guidelines. The two methods were found to give approximately the same results.

6. **With the exception of the XK-3 and the MAP-3 on some substrates, substrate correction using readings for NIST SRM paint films placed on control blocks of substrate materials brought to the site was not effective in reducing biases of K-shell readings attributable to substrates.**

   A third method of correcting for bias attributable to substrates, called "control block" correction, used the average of readings taken on control blocks after the SRM paint film was placed on the control block. These average readings provided an offset value used to correct paint sample readings taken on the same substrate. Control block correction was not a generally effective technique to detect location-dependent substrate characteristics which cause the results to show bias. An exception was the XK-3 instrument. This instrument's results typically exhibited positive bias which <u>was</u> reduced significantly by control block correction. For the MAP-3, control block correction was somewhat effective in reducing bias for plaster, concrete, and metal. For the Microlead I, control block correction actually increased bias for metal and plaster.

7. **No method of substrate correction reduced the bias of L-shell readings.**

   Neither the use of control blocks nor readings taken after placing NIST SRM paint films on scraped substrates was effective in reducing the biases in L-shell readings. This is because L-shell result bias is caused by difficulty in detecting lead in deeper layers of paint, which was not simulated by usage of the NIST SRM paint films.

8. **Despite the generally high variability and bias of their results, K-shell XRF instruments reliably classified the paint samples in this study using the federal threshold of 1.0 mg/cm^2, with laboratory confirmation of XRF readings between 0.4 and 1.6 mg/cm^2 and correction of biases attributable to substrates as needed.**

   Classify a paint sample as positive if the first 15-second (nominal) K-shell XRF reading (substrate corrected as appropriate) taken on paint is 1.6 mg/cm^2 or greater, as negative if the reading is 0.4 mg/cm^2 or less; otherwise the

paint sample is classified as inconclusive. Inconclusive readings are to be resolved by laboratory analysis. Using the ICP spectroscopic analysis of the paint sample to determine whether the lead level was actually greater than or equal to 1.0 mg/cm^2, the overall false positive, false negative and inconclusive rates for the K-shell XRF instruments are shown in Table 2-7. With the exception of the XK-3 false positive rate, all error rates were below 10%. The false positive rate for the XK-3 was dramatically reduced by either method of substrate correction. For each substrate type, most error rates were still below 10%. The exceptions were MAP-3 false negative rates on concrete and plaster, the Microlead I false positive rate on wood, and

Table 2-7. False Positive, False Negative and Inconclusive Percentages for K-Shell XRF Instruments, Based on One Nominal 15-Second Reading With an **INCONCLUSIVE RANGE OF 0.4 - 1.6 mg/cm²** (1.0 mg/cm² Threshold).

INSTRUMENT	FALSE POSITIVE PERCENTAGE	FALSE NEGATIVE PERCENTAGE	INCONCLUSIVE PERCENTAGE
Lead Analyzer K-shell	0.5%	1.4%	18%
MAP-3 K-shell	2.3%	3.7%	23%
Microlead I	7.5%	1.1%	30%
XK-3	22%	1.1%	35%
XK-3 (Average Corrected)	2.3%	4.2%	25%
XK-3 (Control Block Corrected)	3.5%	4.0%	25%

the XK-3 false negative rate on metal. It is important to remember that these classification results apply strictly only to the set of samples and instruments in this study. Classification results for a different set of samples or instruments could be different.

9. **When the laboratory confirmation range was narrowed to 0.7 to 1.3 mg/cm², thereby substantially reducing the inconclusive percentages, the K-shell instruments continued to reliably classify paint samples in this study.**

Table 2-8 shows similar data to Table 2-7 with the narrower inconclusive range. Results of the Microlead I and the XK-3 both needed substrate correction to achieve satisfactory false positive rates. For each substrate type, error rates were generally below 10%. The exceptions were MAP-3 false negative rates on concrete and plaster, the Microlead I false negative rate on concrete, XK-3 false negative rates on metal and plaster, and the XK-3 false positive rate on concrete. Inconclusive percentages are reduced by at least 50% for all XRF instruments compared to the inconclusive percentages when classifying paint samples using the 0.4 - 1.6 mg/cm² inconclusive range.

Table 2-8. False Positive, False Negative and Inconclusive Percentages for K-Shell XRF Instruments, Based on One Nominal 15-Second Reading With an **INCONCLUSIVE RANGE OF 0.7 - 1.3 mg/cm²** (1.0 mg/cm² Threshold).

INSTRUMENT	FALSE POSITIVE PERCENTAGE	FALSE NEGATIVE PERCENTAGE	INCONCLUSIVE PERCENTAGE
Lead Analyzer K-shell	1.2%	2.7%	6.0%
MAP-3 K-shell	4.1%	4.6%	11%
Microlead I	12%	2.1%	15%
Microlead I (Average Corrected)	4.9%	5.3%	12%
XK-3	30%	1.7%	17%
XK-3 (Average Corrected)	5.5%	6.6%	12%
XK-3 (Control Block Corrected)	6.5%	6.8%	12%

10. **Without a laboratory confirmation range, the K-shell instruments' performance differed when classifying paint samples in this study using the federal threshold of 1.0 mg/cm².**

Based on readings obtained using the K-shell instruments, paint samples were classified as positive if the XRF reading was 1.0 mg/cm² or higher and negative otherwise. There was no inconclusive range. False positive and false negative rates for the K-shell instruments' results are shown in Table 2-9. As expected, these rates are higher than when inconclusive ranges were used, but still no greater than 11% overall when substrate correction methods are employed as needed. False positive and false negative rates for readings on particular substrates were substantially higher than the overall rates as exemplified by the following ranges. For all of the K-shell instruments, the lowest false positive or false negative rate on a particular substrate was less than 2.0%. However, on the high end, the Lead Analyzer's false negative rate on concrete was 11%, the MAP-3's false negative rate on concrete was 24%, the Microlead I's false positive rate on wood was 26%, and the XK-3's false positive rate on concrete was 66%.

Table 2-9. False Positive and False Negative Percentages for K-Shell XRF Instruments, Based on One Nominal 15-Second Reading With **NO INCONCLUSIVE RANGE** (1.0 mg/cm^2 Threshold).

INSTRUMENT	FALSE POSITIVE PERCENTAGE	FALSE NEGATIVE PERCENTAGE
Lead Analyzer K-shell	3.1%	5.9%
MAP-3 K-shell	8.0%	8.3%
Microlead I	20%	3.8%
Microlead I (Average Corrected)	9%	9%
XK-3	40%	3.6%
XK-3 (Average Corrected)	11%	10%
XK-3 (Control Block Corrected)	11%	11%

11. **With the exception of the XL, L-shell instruments performed poorly when classifying paint using the 1.0 mg/cm^2 threshold, because of a high rate of false negative results.**

Table 2-10 shows false positive, false negative and inconclusive percentages for tests using L-shell instruments and an inconclusive range of 0.4 to 1.6 mg/cm^2. With the exception of the XL, the false negative rates for the L-shell instruments' results were very high, due to the large negative biases shown in the results using these instruments. False positive rates were very low for all L-shell instruments' results.

Table 2-10. False Positive, False Negative and Inconclusive Percentages for L-Shell XRF Instruments, Based on One Nominal 15-Second Reading with an **INCONCLUSIVE RANGE OF 0.4 - 1.6 mg/cm^2** (1.0 mg/cm^2 Threshold).

INSTRUMENT	FALSE POSITIVE PERCENTAGE	FALSE NEGATIVE PERCENTAGE	INCONCLUSIVE PERCENTAGE
Lead Analyzer L-shell	0.0%	66%	6%
MAP-3 L-shell	0.0%	37%	12%

XL	0.1%	11%	15%
X-MET 880	0.0%	66%	7%

12. **Although the XL prototype had a lower rate of false negative results than the other L-shell instruments, it still exhibited false negative results at very high lead levels.**

As shown in Table 2-10, the XL had a false negative rate of approximately 11% and a false positive rate of 0.1% using an inconclusive range of 0.4 to 1.6 mg/cm^2. However, of the 38 instances where the ICP measurement exceeded 10 mg/cm^2, 2 of the XL readings were below 0.4 mg/cm^2 and one was equal to 0.4 mg/cm^2. In all 3 cases, a paint sample with an ICP result above 10 mg/cm^2 was classified as negative for lead-based paint. With a narrower inconclusive range of 0.7 to 1.3 mg/cm^2, the XL had an overall false negative rate of 24.1% and a 0.2% false positive rate. Classifying the XL results without an inconclusive range yielded a 41.8% false negative rate and a 0.5% false positive rate.

13. **Generally, a single XRF reading at one point of an architectural component provided almost as much accuracy as an average of three XRF readings at the same point.**

When paint samples were classified as positive for XRF results 1.6 mg/cm^2 or greater, negative for XRF results 0.4 mg/cm^2 or less, or inconclusive, otherwise, and the results were compared to the lead level obtained from the ICP spectroscopic analysis of the paint sample, there was very little difference in the false positive and false negative rates for the average of three 15-second readings versus a single 15-second reading. The small improvement in classification accuracy did not justify the additional time and expense of taking three readings at the same point. This remained true when substrate correction and different inconclusive ranges were employed.

A similar conclusion was reached when the precision of the average of three 15-second readings, as measured by its standard deviation, was compared to that of a single reading. If the three readings were statistically independent, one would expect the standard deviation of the average to be 58% of the standard deviation of a single

reading. However, it was found that the standard deviation of the average was much greater than this. For L-shell instruments, the standard deviation of the average was typically at least 95% of the standard deviation of a single reading. For K-shell instruments, the standard deviation of the average was typically between 76% and 93% of the standard deviation of a single reading.

There are two reasons why taking the average of three readings did not produce the expected gains in precision. First, with the exception of the MAP-3 K-shell instrument's readings, successive readings at the same point were positively correlated. Thus, the reduction in variability from averaging repeat readings was less than would be achieved if successive readings had been statistically independent. The second reason why the average produced a smaller reduction in variability than expected is that repeated readings reduced only the component of variability due solely to the performance of the instrument. The study data clearly demonstrated that there were additional sources of variability that were generally at least as large as the component due to the performance of the XRF instrument. Taking repeated readings does not reduce the variability due to these other sources. The additional variability was due to location-specific factors, such as paint and substrate composition.

2.3.3 Chemical Test Kits

1. **None of the test kits used in this study demonstrated low rates of both false positive and false negative results when compared to laboratory analytical results using the federal thresholds, 1.0 mg/cm² and 0.5%.**

   Table 2-11 shows overall false positive and false negative rates for the test kits compared to laboratory analytical results using the 1.0 mg/cm² threshold. Table 2-12 shows the corresponding rates for the 0.5% threshold. Rates for the Lead Alert kits exclude results of tests on painted plaster substrates since the manufacturer does not recommend use of these kits on plaster. For the 1.0 mg/cm² threshold, State Sodium Sulfide and LeadCheck had low false negative

rates, but high false positive rates. Lead Alert: Sanding had a low false positive rate, but a high false negative rate. The other three kits tested, Lead Zone, Lead Detective, and Lead Alert: Coring, had moderate to high rates of both false positive and false negative results. For the 0.5% threshold, State Sodium Sulfide had a low false negative rate and Lead Alert: Sanding had a low false positive rate. False negative rates for LeadCheck and false positive rates for Lead Alert: Coring were slightly above 10%. Lead Zone and Lead Detective had high rates of both false positive and false negative results. As was pointed

Table 2-11. Overall False Positive and False Negative Rates for Test Kits Compared to Laboratory Analytical Results Using the 1.0 mg/cm^2 Threshold.

TEST KIT	FALSE POSITIVE PERCENTAGE	FALSE NEGATIVE PERCENTAGE
LeadCheck	46%	6%
Lead Alert: Coring	15%	24%
Lead Alert: Sanding	9%	53%
Lead Detective	36%	23%
Lead Zone	28%	14%
State Sodium Sulfide	65%	1%

Table 2-12. Overall False Positive and False Negative Rates for Test Kits Compared to Laboratory Analytical Results Using the 0.5% Threshold.

TEST KIT	FALSE POSITIVE PERCENTAGE	FALSE NEGATIVE PERCENTAGE
LeadCheck	42%	11%
Lead Alert: Coring	11%	36%
Lead Alert: Sanding	10%	67%
Lead Detective	32%	27%
Lead Zone	25%	25%
State Sodium Sulfide	62%	6%

out for XRFs, it is important to remember that these classification results apply strictly only to the set of samples and kits in this study. Classification results for a different set of samples or kits could be different.

2. **The substrate underlying the paint sometimes affected false positive and false negative rates for test kits.**

LeadCheck: For both federal thresholds, the false positive rate on drywall was considerably lower than on the other five substrates. False negative rates in mg/cm^2 on concrete and plaster were higher than on the other substrates. For percent by weight, false negative rates were higher on

concrete, drywall, metal, and plaster than on brick and wood. Some of these differences in false negative rates may be caused by sulfates found in plaster dust, gypsum and stucco. The kit includes a confirmation procedure to guard against false negative results caused by sulfates.

Lead Alert: Coring: The manufacturer states that this kit is prone to negative interferences from gypsum and plaster dust. High false negative rates were observed on plaster and drywall for percent lead by weight measurements and on plaster for mg/cm^2 measurements. However, the sample size for drywall was very small. False negative rates on brick were much lower than on the other substrates for both types of measurements. For mg/cm^2 measurements, false positive rates were lowest on plaster and drywall substrates, and highest on brick. For percent lead by weight measurements, false positive rates were lowest on drywall, plaster, and wood substrates, and highest on brick.

Lead Alert: Sanding: This kit had a very similar pattern to Lead Alert: Coring with high false negative rates on plaster and drywall, and the highest false positive rate on brick.

Lead Detective: The manufacturer does not recommend use on metal, but does recommend application on wood, drywall, and plaster. False positive rates were consistent for both types of measurements on all substrates except brick, which had a higher false positive rate. False negative rates were lowest on wood and highest on brick and concrete substrates. (Results were observed showing that drywall had the highest false negative rate for percent lead by weight units, but the sample size was very small.) Thus, this kit did not perform much better on wood, plaster, and drywall than on metal so that the manufacturer's recommendations were not borne out by the study data.

Lead Zone: The manufacturer's instructions only mention testing on wood and metal. False positive rates were the same on all substrates for both types of measurements. False negative rates were lower on brick, wood, and concrete, and higher on the other substrates. The false negative rate on metal was the highest of all substrates using percent lead by weight measurements. The

manufacturer's instructions do not include mention of using this kit on substrates where it performed similarly to its performance on wood, but do mention its use on metal, where its false negative rate was substantially larger than its false negative rate on wood.

State Sodium Sulfide: The instructions contain a caution not to test directly on metal. For metal substrates, a paint chip can be removed and tested separate from the substrate. This kit had very high false positive rates for both types of measurements on all substrates except drywall. False negative rates were low on all substrates for mg/cm^2 measurements. For percent lead by weight measurements, this kit had higher false negative rates on metal, plaster, and drywall than on the other substrates.

3. **The probability of a positive classification when the sample's lead level was equal to the federal thresholds varied depending on the kit and substrate. High levels of lead would not always be detected using test kits alone.**

Table 2-13 shows the probability of a positive result using a test kit on paint with a lead level equal to the 1.0 mg/cm^2 federal threshold, as estimated from the statistical model developed in this study. Table 2-14 is the companion table for the other federal threshold of 0.5% by weight. Considerable variation among results for each kit and each substrate is seen in the tables.

High levels of lead were not always detected with complete certainty using test kits. The statistical model estimated the limiting probability of a positive test kit result at high levels of lead using the laboratory ICP spectroscopic results reported in mg/cm^2 units. In a number of cases, the limiting probability was much lower than the desired value of 100%. This occurred for four of the six kits: Lead Alert: Coring on metal; Lead Alert: Sanding on concrete, metal, and wood; Lead Detective on concrete, metal, and plaster; and Lead Zone on plaster.

Table 2-13. Probability of a Positive Test Kit Result at 1.0 mg/cm^2 Lead.

TEST KIT	Brick	Concrete	Drywall	Metal	Plaster	Wood
LeadCheck	0.95	0.69	0.49	0.93	0.69	0.91
Lead Alert: Coring	0.93	0.27	N/A	0.66	N/A	0.57
Lead Alert: Sanding	N/A	0.50	N/A	0.39	N/A	0.02
Lead Detective	0.81	0.58	0.34	0.74	0.51	0.78
Lead Zone	0.82	0.27	0.64	0.59	0.55	0.80
State Sodium Sulfide	0.99	0.95	0.68	0.94	0.95	0.95

Table 2-14. Probability of a Positive Test Kit Result at 0.5% Lead.

TEST KIT	Brick	Concrete	Drywall	Metal	Plaster	Wood
LeadCheck	0.95	0.68	0.48	0.62	0.68	0.83
Lead Alert: Coring	0.73	0.23	N/A	0.26	N/A	0.28
Lead Alert: Sanding	N/A	0.13	N/A	0.05	N/A	0.03
Lead Detective	0.80	0.55	0.31	0.43	0.46	0.58
Lead Zone	0.81	0.51	0.55	0.19	0.53	0.62
State Sodium Sulfide	0.998	0.93	0.59	0.83	0.91	0.87

4. **The lead level at which there was a 50% chance of the occurrence of a positive test kit result varied depending on the kit and substrate. In many cases, positive results occurred even when paint with very low lead levels was tested.**

   Table 2-15 shows the lead level in mg/cm^2 at which each kit had an estimated 50% probability of a positive result, by substrate. Table 2-16 is the companion table in percent lead by weight measurements. There was significant variation in 50% probability levels for different kits used on the same substrate. There was also significant variation in the 50% probability levels for the same kit used on different substrates. One exception, the State Sodium Sulfide kit, reached a 50% probability of a positive result at low lead levels on all substrates for both types of measurements.

   The statistical model used to analyze the test kit data also provided estimates of the limiting probability of a positive result as the lead level in the paint sample approached zero using the laboratory ICP spectroscopic results reported in mg/cm^2 units. It is desirable that this limiting probability be zero; otherwise, the kit will produce some positive results even for paint samples with very low lead levels. However, every kit exhibited a non-zero limiting probability of a positive result on at least one substrate. This occurred on metal substrates for all six kits. With the sodium sulfide kits, Lead Detective and State Sodium Sulfide, most substrates had a non-zero limiting probability of a positive result. For the other 4 test kits, limiting

probabilities of a positive result equaled or exceeded 20% for LeadCheck on metal and plaster, Lead Alert: Coring on brick, and Lead Zone on concrete. For LeadCheck, Lead

Table 2-15. Lead Level in mg/cm^2 at Which There is a 50% Probability of a Positive Test Kit Result.

TEST KIT	Brick	Concrete	Drywall	Metal	Plaster	Wood
LeadCheck	0.02	0.19	1.14	0.34	0.13	0.03
Lead Alert: Coring	0.33	1.84	N/A	0.65	N/A	0.77
Lead Alert: Sanding	N/A	N/A	N/A	N/A	N/A	1.24
Lead Detective	0.05	0.60	N/A	0.55	0.98	0.20
Lead Zone	0.08	1.38	0.31	0.82	0.71	0.15
State Sodium Sulfide	0.01	0.01	0.08	0.08	0.02	0.04

Table 2-16. Lead Level in Percent Lead by Weight at Which There is a 50% Probability of a Positive Test Kit Result.

TEST KIT	Brick	Concrete	Drywall	Metal	Plaster	Wood
LeadCheck	0.02	0.16	0.56	0.32	0.14	0.07
Lead Alert: Coring	0.13	1.14	N/A	1.09	N/A	0.97
Lead Alert: Sanding	N/A	0.88	N/A	N/A	N/A	1.68
Lead Detective	0.01	0.33	N/A	0.63	0.58	0.36
Lead Zone	0.07	0.49	0.35	1.03	0.44	0.26
State Sodium Sulfide	0.01	0.01	0.13	0.08	0.02	0.09

Detective and State Sodium Sulfide, limiting probabilities for the wood substrate were positive.

2.3.4 Paint Chip Sampling and Analysis

1. **Lead levels in paint showed significant variation within individual architectural components such as doors, walls, and baseboards.**

   Duplicate paint samples were taken approximately 9 inches apart on the same component at 10% of the test locations in the full study in Denver and Philadelphia. Duplicate paint samples taken from the same component were called duplicate pairs. The estimated median ratio of the larger to the smaller ICP spectroscopic result, measured in mg/cm^2, for

duplicate pairs was 1.6 in Denver and 1.3 in Philadelphia. The corresponding median ratios for percent lead by weight units were 1.5 and 1.2. The estimated 95th percentile for the ratio in mg/cm^2 was 3.7 in Denver and 2.1 in Philadelphia. The corresponding 95th percentile ratios for percent lead by weight units were 3.1 and 1.9. There was slightly greater variability in lead levels within architectural components when measured in mg/cm^2 than in percent lead by weight. The extent to which greater variability would be observed between samples taken farther apart than 9 inches is not addressed by the study data.

Variability in duplicate samples could result in different classification of paint depending on which member of the pair was compared to the federal threshold. If the lead level of a paint sample was equal to or greater than the federal threshold, it was classified as positive for lead-based paint. Likewise, if the sample was less than the federal threshold, then it was classified as negative. Of 128 total duplicate pairs in the study, 10 (8%) had different classifications, one sample positive and the other negative for lead, compared to the 1.0 mg/cm^2 threshold, while 8 (6%) had different classifications compared to the 0.5% threshold.

Spatial variation in lead levels within single architectural components complicated the statistical analysis of XRF and test kit performance data in the study. Complex statistical models were needed to account for the impact of spatial variation on estimates of XRF measurement bias and standard deviation. Spatial variation had a smaller impact on the test kit data analysis.

2. **Variation between members of laboratory duplicate subsample pairs was much smaller than variation between members of duplicate samples obtained in the field.**

Laboratory analytical measurement error for ICP spectroscopic analysis of 2 x 2 inch paint chip samples, including homogenization, subsampling and instrumental error, can be quantified using the ratio of the larger to the smaller ICP measurement for a pair of subsamples of the same sample. The estimated median for this error ratio was 1.13 for samples taken from smooth substrates with no

unusual difficulty in paint removal. The estimated 95th percentile for the error ratio was 1.4. These ratios apply to laboratory results reported in both in mg/cm^2 and percent lead by weight units.

Laboratory measurement error was approximately constant across metal, wood, plaster, and drywall substrates, across cities, and across samples within a substrate or within a city. For samples taken on rough substrates such as brick or concrete, total laboratory analytical measurement error was higher: the estimated median ratio was 1.2 and the estimated 95th percentile ratio was 1.8.

Only two laboratory duplicate pairs out of a total of 171 (1%) had different classifications, one of the pair positive and one negative, with respect to the 1.0 mg/cm^2 threshold. For the 0.5% threshold, three subsample pairs out of 171 (2%) had different classifications.

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Chapter 3 Summary: Design Elaboration

GENERAL DESIGN ELEMENTS

- **Testing Sites: Four multifamily units in Louisville, ten single family units in Denver, & eight multifamily units in Philadelphia**
- **Number of Test Locations: 100 in Louisville, 750 in Denver, & 440 in Philadelphia.**
- **Range of Substrate Types: brick, concrete, plaster, drywall, metal, & wood.**
- **Blind Testing: Testers were blind to previous test results.**

SPECIFIC DESIGN ELEMENTS - COLLECTION OF PAINT CHIP SAMPLES

- **Areas Collected: samples were approximately 25 cm^2 each.**
- **All Paint Layers Collected: collection of all paint layers was the 1st priority, avoiding substrate inclusion was a 2nd priority.**
- **Field Duplicates Collected: collected in Denver and Philadelphia at a rate of 10%**
- **Uniform Collection Protocols: included specific step-by-step instructions.**

SPECIFIC DESIGN ELEMENTS - LABORATORY ANALYSIS OF PAINT CHIP SAMPLES

- **Inductive coupled plasma atomic emission spectrometry (ICP) Detection: lead measurements on prepared samples were performed using ICP**
- **Validated Sample Preparation Procedure: a hot plate digestion procedure was investigated for lead recoveries prior to use for processing field paint-chip samples.**
- **Sample Homogenization: all samples homogenized prior to preparation.**
- **Preparation Sample Size: sample size of a nominal 0.5 grams for adequate recovery.**
- **Reporting of Lead Results in both Mass/Area and Mass/Mass: mg/cm^2 and mg/g.**
- **Blind Samples: included in each batch of field paint-chip samples.**
- **Laboratory Duplicates Processed: included in each batch of field paint-chip samples.**
- **Method Blanks Processed: included in each batch of field**

Chapter 3 Summary: Design Elaboration (continued)

SPECIFIC DESIGN ELEMENTS - TEST KIT MEASUREMENTS

- **Wide Range of Test Kits: Six test kits included.**
- **Professional State of Massachusetts Test kit tester: included as one of the six kits, applied by a professional tester.**
- **Simulated Homeowners: simulated homeowners were used to perform measurements for five kits marketed for home owner use.**
- **Training: simulated homeowners received some limited in-field training on test kit handling.**
- **Adherence to Manufacturer Instructions: protocols followed by simulated homeowners were done in accordance to the manufacturers instructions with only minor exceptions.**

SPECIFIC DESIGN ELEMENTS - XRF INSTRUMENT MEASUREMENTS

- **Range of XRF Instruments: Six XRF instruments were included.**
- **Independent XRF operators: XRF instruments were operated by testing contractors.**
- **Specified Testing Order and Testing Protocols: testing was performed using a specific substrate testing order with detailed testing protocols.**
- **Common Reading Times: standard testing performed at all locations used a common nominal reading time for all instruments.**
- **Alternative Reading Times: additional testing was performed at some locations using longer reading times for one instrument.**
- **Field QC Measurements: a number of specific field QC measurements were included.**
- **Bare Substrate Measurements: measurements were taken on both scraped substrates and scraped substrates covered by NIST standard reference material paint films.**

3 DESIGN ELABORATION

This section describes the design elements that were incorporated into the testing tasks to aid in achieving the study objectives:

- to characterize the precision and accuracy of portable XRF instruments under field conditions
- to evaluate the effect on XRF performance of interference from the material (the substrate) underlying the paint
- to characterize the relationship between test kit results and the actual lead level in the paint
- to investigate XRF measurements that were very different than their corresponding laboratory results.
- to evaluate field quality assurance and control methods
- to investigate the variability of lead levels in the paint within the study sampling locations

The study design elements, discussed below, include both general design elements common to all testing tasks and specific testing design elements.

3.1 GENERAL DESIGN ELEMENTS

General design elements incorporated into the study included use of variable sampling sites, a large number of sampling locations at each site, inclusion of specific substrate types, use of a standardized marking template, targeted locations near the 1.0 mg/cm^2 lead level, a controlled sample identification transfer system, and testing in a blind manner. Each of these general design elements is discussed in this section. Specific design elements is discussed later in sections 3.2 through 3.5.

3.1.1 Site Selection

A total of three different cities were included in the study. The pilot study was conducted in four multifamily housing units, two units each inside two buildings at one development in Louisville, Kentucky. The full study was conducted using ten single family homes in Denver, Colorado and eight multifamily housing units inside two buildings at one development in Philadelphia, Pennsylvania. A summary of the units by age included in the study is presented in Table 3-1. For the pilot study, each set of two units within a building were assigned a

single dwelling number for testing as shown in Table 3-1. For the full study, each unit was assigned a separate dwelling number for testing.

Table 3-1. A Summary of Units Selected for the Study.

CITY	DWELLING No.	YEAR BUILT
Louisville	1	1937
	2	1937
Denver	1	1943
	2	1948
	3	1952
	4	1905
	5	1949
	6	1948
	7	1952
	8	1890
	9	1949
	10	1947
Philadelphia	1	1942
	2	1942
	3	1942
	4	1942
	5	1942
	6	1942
	7	1942
	8	1942

Primary considerations for selection of housing units were to use units with a wide distribution of surface types having variable painting histories and to select units that were likely to represent those that are currently being routinely tested for LBP. Therefore, both multifamily housing and scattered site housing units were included in the study to generate results that represent both these types of housing.

Selection of the study sites was performed through cooperation of the Housing Authority in each city. The following criteria were used to select the units for the study:

1. Units had to be available and vacant March through October 1993.
2. All units had to be constructed prior to 1970.
3. Overall, all six substrate targeted types had to be represented. Substrate types are discussed in section 3.1.3.
4. Laboratory or XRF testing data had to be available. These

data were needed to help select painted components in the 0.5 to 1.5 mg/cm^2 range for testing whenever possible. Selection of painted components in this range would provide lead testing results close to the current 1.0 mg/cm^2 HUD action level.

5. Overall, the units had to be free from obvious sources of bias and represent a variety of paint types, architectural designs, and lead levels in paint.
6. Housing Authorities had to be willing to accept the damage associated with testing, including the removal of selected pieces of substrate when possible for archive purposes, provided that repairs were made prior to leaving the site.
7. The neighborhoods in which the units were located had to be sufficiently safe to permit uninterrupted testing.
8. Availability of good lighting and electricity was desirable but not mandatory.

The final set of units included in the study were selected following site inspections by study team members. Large variability in paint colors and collected paint chip sample masses were observed across the three cities. Total paint chip sample mass ranges for approximately 25 cm^2 areas collected in the full study were wide: 0.0454 grams to 25.1782 grams for Denver and 0.1362 grams to 11.1902 grams in Philadelphia. This suggests that selected sampling locations had wide variability in painting histories, as desired for the full study. The mean value of the total paint chip sample mass for the samples collected in Philadelphia (3.1586 grams) was higher than those collected in Denver (2.4306 grams). This suggests that thicker paints were present in the multifamily housing units sampled in Philadelphia than in the single family housing units sampled in Denver. However, the sample mass for samples collected in Louisville were less variable, ranging from 0.3116 grams to 4.3604 grams with a mean of 1.7417 grams.

3.1.2 Location Selection

A sampling location was a test area on a painted building component where lead measurements were taken using each of the testing methodologies included in the study. Examples of building components included walls, baseboards, doors, and window frames.

For the pilot study in Louisville, a total of 25 sampling

locations per housing unit were targeted for testing for a total of 100 locations.

For the full study, a total of 75 sampling locations per house in Denver and 55 sampling locations per housing unit in Philadelphia were targeted for testing for a total of 1,190 locations.

Selection of sampling locations was performed while simultaneously adhering to the following five constraints:

1. Meet the targeted number of total sampling locations as described above.
2. Meet targeted number of sampling locations on different substrate types as described in section 3.1.3.
3. Select as many sampling locations as possible with lead levels close to 1.0 mg/cm^2 as was described in section 3.1.1.
4. Select surfaces that were capable of being testing using a portable XRF. The surface could not be ornately curved and must be able to accept the face-plate geometry of each XRF evaluated in the study.
5. Selected surfaces could not be ceilings. This constraint was used to avoid potential safety hazards resulting from attempting to use test kit chemicals in over-head positions.

The actual number of sampling locations for each substrate type are discussed further in the following section. Lead levels from laboratory analysis are discussed in chapter 4.

3.1.3 Substrate Selection

A key general design element for the study was to include painted substrates that were commonly found in residential housing. A total of six commonly encountered substrates were targeted for inclusion: brick, concrete, metal, drywall, plaster, and wood. This design element was included to provide information on the possible influence of substrates on XRF and test kit results.

During the pilot study in Louisville, attempts were made to distribute the total number of sampling locations equally among the targeted substrates. However, success was limited by lack of painted brick and drywall present at this site.

Prior to the full study in Denver and Philadelphia, attempts were made to identify 200 sampling locations for each targeted substrate. However, site inspections made by the study team indicated that painted brick was relatively uncommon and that only about half the targeted number of sampling locations for this substrate were likely to be obtained. Because of the presence of a wide variety of painted wood building components in selected units, additional painted wood sampling locations were selected in place of painted brick sampling locations that could not be found. The estimated number of sample locations targeted for the full study, based on the pre-sampling site inspections, and for the pilot is presented in Table 3-2 along with the actual number of sample locations achieved.

3.1.4 Logistical Considerations

The study required the performance of three major testing tasks: (1) XRF measurements, (2) test kit applications, and (3) paint chip collection for laboratory testing. Of the three major testing tasks, only the XRF measurements are non-destructive. The other two testing tasks are destructive. The best approach to performing these tasks in the field, with respect to testing of the exact same areas, would be to perform the tasks in the following order:

1. Perform XRF measurements on the painted surface.
2. Perform test kit applications on the painted surface.
3. Collect a paint chip from painted surface for later laboratory analysis.
4. Remove remaining paint from surface.
5. Perform XRF measurements on scraped surface. (This is a design element for XRF measurements described in section 3.5.2.8.)

However, testing on the exact same area in the above order could not be performed because of the following four major reasons:

1. Destructive testing by the test kit applications would interfere with collection of the paint chip for laboratory analysis.
2. Destructive testing by the test kit applications would interfere with XRF measurements on the scraped surface.
3. Large amounts of time would elapse between the XRF

measurements on the paint surface and XRF measurements on the bare substrate. This would be undesirable because of the potential for instrumental drift. If the elapsed time between the XRF measurements on paint and bare substrates were minimized by requiring the testing personnel to perform their tasks sequentially, one location at a time, a large amount of idle time would be introduced causing large increases in required field testing time and a large impact on project costs.

Table 3-2. Number of Sampling Locations by Substrate.

Substrate	Denver[a]	Philadelphia[a]	Louisville[a]	Total[a]
Brick	80 / 81	8 / 12	0 / 0	88 / 93
Concrete	170 / 98	48 / 120	20 / 8	238 / 226
Drywall	170 / 105	8 / 8	20 / 11	198 / 124
Metal	60 / 62	128 / 127	20 / 28	208 / 217
Plaster	100 / 101	120 / 121	20 / 20	240 / 242
Wood	170 / 303	128 / 52	20 / 33	318 / 388
Total	750 / 750	440 / 440	100 / 100	1290 / 1290

[a] target number / actual number

4. Holding multiple test personnel at or near the location to perform all the testing tasks would increase the risk of passage of previous results to later testers. In addition, an increased risk of inadvertent exposure of testers to XRF radiation and problems caused by excessive traffic flow would also exist.

To maintain adequate supervision, project performance control, and expenditure control, the design of the study needed to be such that a group of test personnel performing the same types of tests could be cycled through a given housing unit in a given time period. Therefore, the design of the study included testing at each sampling location in different areas that were identified through the use of the standardized location marking template. This template specified area blocks for performing different tests as discussed later in section 3.1.5. Using this template, the order of testing for each unit included in the pilot study was as follows:

1. Collect all paint samples for laboratory analysis and remove remaining paint from the surface as specified by the template for later XRF bare substrate measurements;
2. Perform all test kit applications;
3. Perform XRF measurements on the painted surface; and
4. Perform XRF measurements on the scraped surface.

The order of testing for each unit was changed from the pilot to the full study. The change made was to perform the test kit applications before the paint chip collection. This was done to force the testers to make a decision as to when they had reached the last layer of paint without the visual aid provided by the bare substrate area generated during paint chip collection. The order of testing for each unit included in the full study was as follows:

1. Perform all test kit applications;
2. Collect all paint samples for laboratory analysis and remove remaining paint from surface for later XRF bare substrate measurements;
3. Perform XRF measurements on the painted surface; and
4. Perform XRF measurements on the scraped surface.

3.1.5 Standardized Location Marking Template

A standardized format, referred to as a template, was used to mark each sampling location for XRF readings, test kit applications, and paint chip collection. The template, shown in Figure 3-1, includes separate blocks to identify areas where the two different types of measurements were to be taken and the paint chip samples were to be removed for laboratory analysis. Use of this template enhanced the comparability of lead results by providing a mechanism to take lead measurements in a uniform manner at different locations.

As shown in Figure 3-1, the template was changed from the pilot to the full study. The changes made in the template were a result of increased awareness of spatial variations in lead concentrations from data provided by EPA/ORD. Spatial variations in lead concentrations refer to surface-to-surface or two-dimensional variability in lead concentration. Because of the potential spatial variations, the following three changes were made:

1. Paint chip collection areas were moved to roughly the center of the template with XRF readings and test kit applications to either side;
2. Positions of test kit applications were randomized as discussed later in section 3.4.2.1; and
3. Collection of field duplicate paint chip samples was moved just outside the upper right hand corner of the horizontally placed template.

Two general orientations of the template were used. One is the horizonal orientation as shown in Figure 3-1. The other was a vertical orientation (90° clockwise turn of the template, with test kit areas at the bottom), which was used when the targeted location would not accommodate the horizonal orientation. Under a few circumstances, driven by field conditions, some targeted locations required alterations to the standardized template.

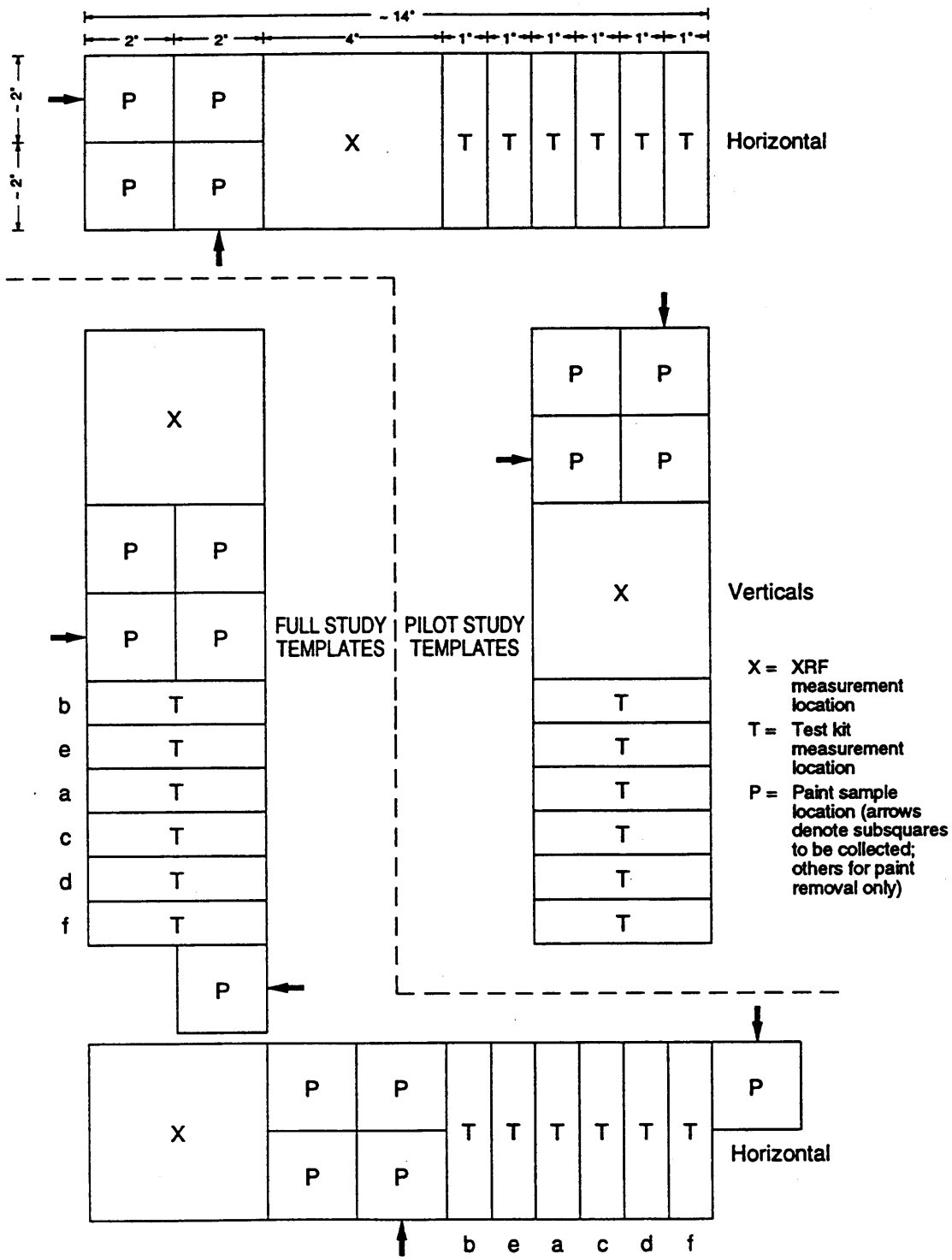

Figure 3-1. Sampling location templates used in the study.

Examples of these are shown in Figure 3-2. All locations, including altered versions of the standardized template, were marked under the direct supervision of a statistician knowledgeable on the design aspects of the study.

3.1.6 Sample ID Transfer

Control over the potential of misidentified sampling locations was achieved through the use of pre-printed sticky-back barcode labels. Large numbers of identically numbered labels matching the sample location number were affixed to each location for use on all data forms throughout the study. This general design element reduced the potential for generation of a transcription error for data-recording activities during all phases of testing. Control over the potential transcription errors in the laboratory was achieved by submitting a set of barcode labels with each paint chip sample collected in the field for later use in the laboratory.

3.1.7 Blind Testing

Performance of testing in a manner that would reduce the potential for inadvertent passage of testing results between different test personnel was of primary importance in the study. Several general procedures were incorporated into the study to achieve testing in a blind manner. These included actively directing all testing personnel to perform measurements independently, prohibition of any discussion of results with other test personnel, active supervision of sites at all times during testing activities, and collection of all data forms at the end of each day of testing. In addition, two specific field testing procedures for test kit applications and one additional field testing procedure for XRF measurements were incorporated. For test kit applications in the full study, a maximum number of three testers were allowed in a given housing unit at one time.

In the pilot study, test kit applications were spread-out by staggering the starting points. All testing marks from test kit applications were hidden from the next tester, using tape as described later in section 3.4.2.2. For XRF measurements, only one XRF operator with monitor was allowed in a given housing unit at one time, as described later in section 3.5.2.11.

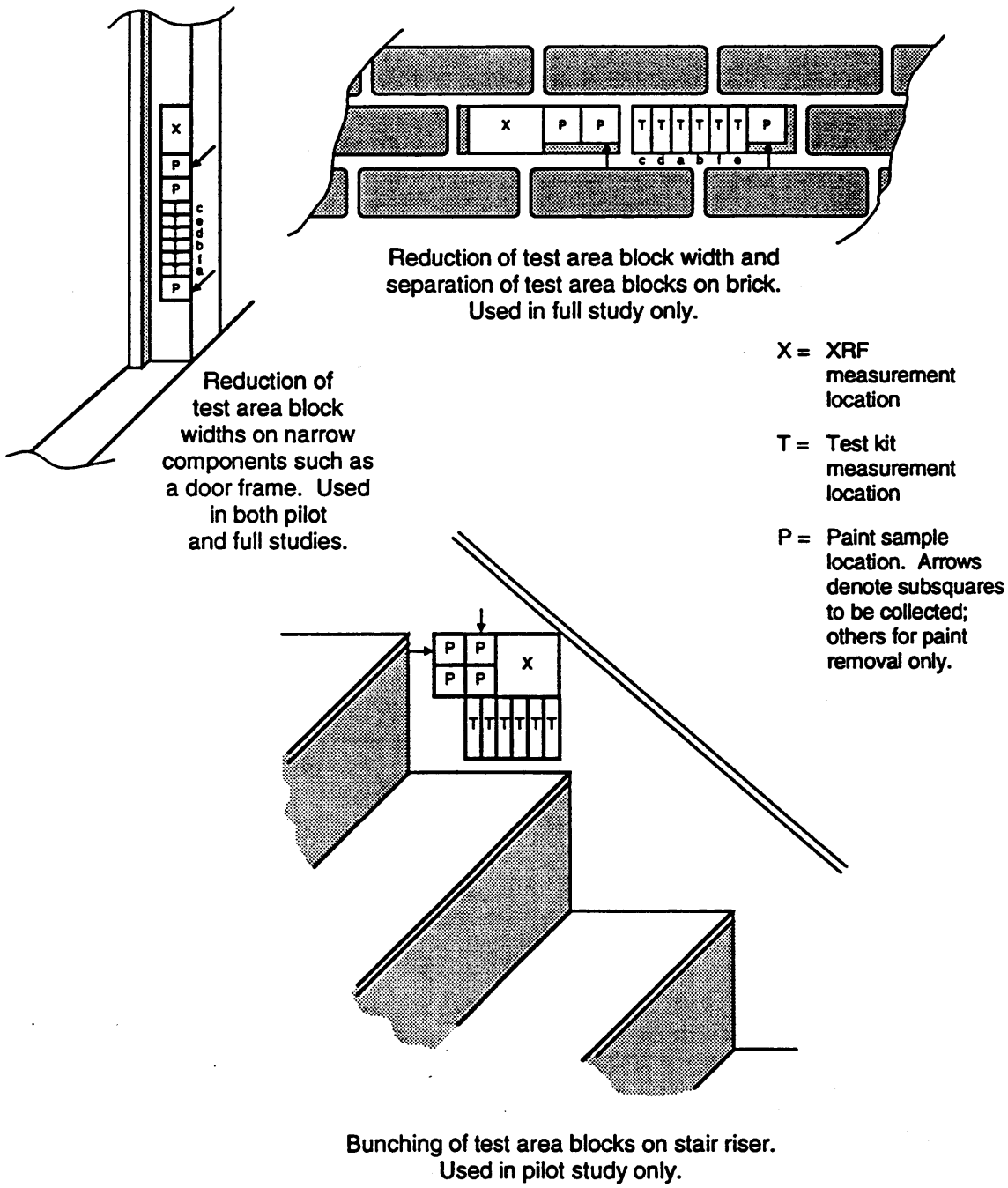

Figure 3-2. Examples of alternative sample location templates used in the study.

3.2 COLLECTION OF PAINT CHIP SAMPLES

The collection of paint chip samples is discussed in this section. Further details on sampling procedures can be found in the appendices of this report. Laboratory handling of collected paint samples is discussed later in section 3.3.

3.2.1 Paint Collection Method Selection

In general, there are three methods for removing the paint: the heat gun method, the cold scraping method, and the coring method. The heat gun method uses a heat gun to soften the paint to aid in removal from the substrate using scraping and cutting tools. The cold scraping method is the same as the heat gun method but without the use of the heat gun. The coring method is a cold cutting removal method that incorporates use of a sharpened cylindrical cutting tool. The heat gun method for removing paint was selected as the primary method of choice for use in the study because of the following advantages:

a) The heat gun method can be used on a wider variety of surfaces than the coring method. For example, the coring method is not very effective on very hard substrates such as brick or concrete because of coring tool damage that occurs when cutting against these hard surfaces.
b) The heat gun method can be easier to perform on some surfaces than the cold scraping method. Cold paint removal generally requires more force.
c) Both lead-area and lead-concentration determinations can be performed on the collected sample with the heat gun method. For the coring method, only lead-area determinations are possible because this method requires the use of tape to hold the painted surface intact during the coring process. The tape adds mass to the sample and cannot be easily removed without damaging the paint film, thus eliminating the ability to determine lead-concentration results.

The primary disadvantage of the heat gun method is that its use can result in generation of organic based fumes during heating of the paint. Therefore, the heat gun method requires the use of a respirator to protect workers from the generated fumes. For this study, separation of different field activities required for logistical reasons, previously discussed in section

3.1.4, limited the need for fume protection only to those directly involved with paint collection; that is paint samplers and field supervisory staff.

The cold scraping method of paint removal was selected as the secondary method. A secondary method was needed because the heat gun method is not very effective on building components with high heat capacities. For example, concrete has a high heat capacity. A heat gun cannot generally apply sufficient heat to soften the paint and keep it soft during the scraping process on this substrate. For substrates that have high heat capacities, cold scraping using chisels are usually more effective than a heat gun.

3.2.2 Paint Collection Design Elements

A discussion of design elements for paint collection is presented in this section.

3.2.2.1 Collection of Large Surface Areas

Common methods used to apply paint to surfaces can result in variability of paint thickness on painted building components. These variations in paint thickness can result in spatial variations in lead levels. Collection of a large surface area was incorporated as a study design element to aid in reducing variation caused by these potential spatial variations. A metal template with inside dimensions of approximately 2 by 2 inches square was used to score the perimeter of the paint chip collection area as presented later in section 3.2.3. Because of the desire to report lead analysis results in mg/cm^2, actual collection areas were measured in centimeters. Paint chip samples collected in the study were approximately 25 cm^2 in size (5 cm ≈ 2").

The use of large area samples also assured that collected-sample mass would be sufficiently heavy to conduct duplicate sub-sample analyses in the laboratory. Using a nominal 0.5-gram sub-sample mass, a minimum of 1 gram of paint chip sample was required to conduct laboratory duplicate sample analyses. The mean value of the total paint chip sample mass across all three cities, to one decimal place, was 2.6 grams for the approximately 25 cm^2 paint chip collection area. This is discussed further in sections 3.3.2.1 and 3.3.2.3.2.

3.2.2.2 Substrate Inclusion

The following three issues were considered during the design of paint collection protocols with respect to substrate inclusion:

(1) Collection of paint samples without including some substrate is difficult. This is particularly true for soft substrates such as wood or drywall and porous substrates such as some types of brick and concrete. Soft and porous substrates typically adsorb substantial amounts of paint when painted, and this first layer of paint is the most likely to have the highest lead levels, being the oldest layer on a painted surface.
(2) XRF instruments that employ K-shell detection are generally considered to be able to detect lead to depths that go well beyond the paint layers into the underlying substrate, and sometimes beyond.
(3) Test kit protocols are generally written to test only the paint layers and not any paint that may be absorbed into the substrate.

There are four potential testing conditions with respect to collection of paint samples regarding potential substrate inclusion. These four potential testing conditions, lead free and lead present in substrate combined with collection of paint samples with and without substrate inclusion, are summarized in Table 3-3.

Ideally, paint collection should be performed in a manner that collects all paint from the surface and leaves all substrate material intact on the building component; these are testing conditions A and B in Table 3-3. Under test condition A, both XRF and test kit results are directly comparable to the laboratory results generated from the collected paint samples. However, if the substrate contains lead that can be measured by the XRF (issue (2) above) then the XRF results may be biased high with respect to the laboratory results. Bare substrate XRF measurements incorporated into the study as a basic design element, discussed in section 3.5.2.8, can theoretically be used to correct for this high bias by subtracting the XRF bare substrate lead results, that is, background results, from the XRF results on the painted surface. Unfortunately, this technique is limited because it is not possible to separate background results

caused by lead in the substrate and other XRF measurement substrate effects, discussed under section 3.5.2.

As presented in issue (1) above, substrate inclusion into some of the collected paint samples is inevitable. Testing conditions C and D in Table 3-3 summarize the testing outcomes for these conditions. If the substrate is lead free, test condition C, then both XRF and test kit results are directly comparable to the laboratory results generated from the collected

Table 3-3. Potential Testing Outcomes for Different Testing Conditions.

TESTING CONDITION CODE	TESTING CONDITION		LABORATORY mg/cm^2 RESULTS DIRECTLY COMPARABLE TO TEST METHOD?		
	LEAD IN SUBSTRATE?	SUBSTRATE IN COLLECTED PAINT SAMPLE?	XRF	XRF MINUS SUBSTRATE MEASUREMENTS[a]	TEST KITS
A	no	no	yes	yes	yes
B	yes	no	no[b]	yes	yes
C	no	yes	yes[c]	yes[c]	yes[c]
D	yes	yes	yes[d]	yes	no[e]

[a] Correction of background lead remaining in substrate can be compensated by use of (1) the NIST covered bare substrate measurements, corrected for the known NIST lead level or (2) the bare only substrate measurements. (1) was taken at all sampling locations in the full study. (2) was taken at approximately 25% of the sampling locations in the full study.

[b] XRF results may be biased higher than the laboratory results if XRF instrument capable of "reading" the lead in the substrate.

[c] XRF results and test kits will be comparable to laboratory results on a mg/cm^2 basis. However, the laboratory mg/g results will be biased low because of increased collected sample mass.

[d] XRF results may or may not be comparable to laboratory results on a mg/cm^2 basis. The comparability level will be dependent on the amount of XRF measured substrate that is included in the collected paint sample.

[e] Test kit results may not be comparable to laboratory results on a mg/cm^2 basis. The comparability level will be dependent on the amount of lead in the substrate material that is included in the collected paint sample.

paint samples in mg/cm^2. However, if the substrate contains lead, then the XRF results may or may not be directly comparable to laboratory results on a mg/cm^2 basis. The degree of comparability will be dependent on the amount of XRF measured substrate that is included in the collected paint sample. In addition, if the substrate contains lead, then the test kit results may be biased low compared to laboratory results. The amount of bias will be dependent on the amount of lead contaminated substrate that is included in the collected paint sample.

Paint collection protocols used in the study are presented in detail in the appendices of this report and are summarized in

section 3.2.3. These protocols were designed to maximize the comparability of laboratory results to XRF and test kit results.

A priority was given to collection of as much paint as possible from targeted sample locations. The second priority was to minimize the inclusion of substrate. In other words, the paint chip collection rule adopted for this study was to collect all visible paint even if that meant collecting some substrate. However, if possible, substrate inclusion during paint collection was avoided. Collection of all of the paint is a logical necessity required to make any reasonable comparisons between testing technologies. It was believed that use of the second priority, minimizing substrate collection, would reduce the potential comparability problem for test kits identified in testing condition D. The potential comparability problem for XRF results identified in testing condition B have to be controlled through background subtraction, as discussed above, since no reliable field method was available for assessing the magnitude and depth of lead present in the underlying substrate.

3.2.2.3 Collection of Field Duplicate Paint Chip Samples

As previously discussed in section 3.2.2.1., spatial variations in lead levels were expected to be encountered during performance of the study. Collection of duplicate samples at the sampling locations, called field duplicate samples or side-by-side samples, was included as a design element to provide information on the magnitude of potential spatial variations in lead across the sampling location area.

For the pilot study, one field duplicate sample was collected in addition to the regular paint sample at each sampling location. As previously discussed in section 3.1.5 and shown in Figure 3-1, the regular and the field duplicate samples were selected at random from within the nominal 4 in. by 4 in. area targeted for paint collection. A hand-held calculator was used to determine a random number for assignment of the sampling positions within this targeted sampling area.

For the full study, field duplicate samples were collected at approximately 10% of the sampling locations. Similar to the pilot study, the regular samples were selected at random from within the nominal 4 inch by 4 inch area targeted for paint collection. A hand-held calculator was used to determine a random number for assignment of the sampling position within this

targeted sampling area. However, the location of the field duplicate sample was different from that in the pilot. Collection of field duplicate samples was moved to the end of the sampling location as shown in Figure 3-1. This change was made to provide information on potential spatial variations of lead across a wider area than was obtained for the pilot study. It was believed that variability data across this wider width would better characterize the actual lead variability experienced by all the different tests being conducted in different sub-areas within the template.

3.2.2.4 Collection of Field Blanks

Field blanks are generally included as a design element in any sampling study to provide information on the extent of lead contamination resulting from a combination of laboratory processing and field handling activities. In general, field blanks are identical to field paint-chip samples, except that no sample is actually collected. When sampling involves collection of material on a collection medium such as a filter, field blank results can be very useful for assessing contamination experienced by the field paint-chip samples. However, when sampling does not involve the use of a collection medium, such as is the case for paint collection, field blanks have limited value because only a representative collection container can be marked and shipped to the laboratory to serve as a field blank.

Potential contamination resulting from sampling handling such as use of scraping tools and collection trays cannot be assessed in a manner that would be representative of the collected paint samples. Therefore, field blanks are limited to monitoring the incidence of sample container contamination resulting from the handling of the samples in the field, shipping samples to the analytical laboratory, and storing samples prior to analysis. Despite this limitation, field blanks were included in the study. The targeted number of field blanks to collect was at least one for each housing unit sampled. The total number of field blanks actually collected during the study were as follows: 10 in Louisville, 13 in Denver, and 8 in Philadelphia.

Because of the relatively high levels of lead of interest in the study, contamination was not believed to be a significant concern. Therefore, the limited value of the field blanks was not believed to be a problem. The largest potential source of

contamination for such a study as this one is from cross contamination of previously collected samples to the sample being currently collected. Extra attention to housekeeping and personal hygiene was mandated during collection of paint samples to minimize any cross-contamination of the samples. The following steps were implemented to avoid contamination in the field:

- The sampling collection materials were stored in sealed containers until used.
- A new sample collection tray was constructed at each sampling location using a new sheet of white paper. (See section 3.2.3 below.)
- Hands were kept clean by washing them with pre-moistened baby wipes.
- Reusable paint sampling equipment (templates, scrapers, etc.) were thoroughly cleaned between uses with pre-moistened baby wipes.

3.2.3 Paint Collection Protocol Summary

Collection of each paint chip sample required four major steps: (1) Marking the collection area, (2) Setting up a paint collection tray, (3) Removing the paint, and (4) Transferring the collected sample to the paint collection container. A detailed protocol describing paint collection is presented in the appendices of this report. A summary of the collection steps is presented below.

(1) Marking the collection area. Sample location areas selected for paint collection were marked as discussed in section 3.1.5. Pre-numbered barcode labels for identifying sample containers and data entries on paint collection forms were placed at each sampling location as described in sections 3.1.6. The outline of the paint collection area was further defined, after setting up a collection tray and prior to paint removal, by cutting down through all paint layers around the marked area with the aid of a metal template and a utility knife. Use of the square template with internal dimensions of approximately 25 cm^2 helped improve uniformity of collection areas at different sampling locations.

(2) Setting up a paint collection tray. A collection tray was

placed beneath each sample collection area to catch any paint during the removal process. The collection tray was prepared using a sheet of clean white paper, one edge taped immediately below the sample collection area. The tray was formed by pulling the two adjacent corners together (opposite from the taped edge), overlapping slightly to form a funnel, and taping it together using a piece of masking tape. The collection tray was completed by folding up a portion of the bottom and taping it in place to permanently close off the funnel bottom. This was done in a manner that no sticky tape surfaces were exposed on the inside of the closed bottom funnel. The result was a clean, wide, disposable collection tray capable of catching all paint removed from the collection area.

(3) Removing the paint. A variety of scraping and cutting tools were used to complete paint removal from the targeted collection area after softening the paint with the heat gun. Removed paint was deposited into the collection tray placed immediately below the collection area. Following transfer of the paint to the paint collection container, described in step 4 below, and collection of any required field duplicate samples, previously described in section 3.2.2.3, additional paint was removed from the sampling location to provide a sufficiently large area for later XRF measurements on the bare substrate.

(4) Transferring the collected sample to the paint collection container. Following completion of paint removal from the targeted collection area, paint in the collection tray was transferred into a labeled plastic centrifuge tube for shipment back to the laboratory. The transfer of paint to the centrifuge tube was performed by carefully tapping the collected paint into the paint collection container. A fold creased into the side of the closed bottom paper funnel was used to aid in the transfer of paint into the labeled plastic centrifuge tube. The dimensions of the collection area were measured and recorded on a field data form following completion of paint transfer to the collection container. The field crew used rulers with metric only units to reduce any potential confusion as to sampling area units for dimensions recorded on field data forms.

3.2.4 Summary of Field Observations

A summary of observations on collection of paint samples is presented in this section.

3.2.4.1 Paint Collection Time Requirements

Collection of a large number of paint samples is a labor-intensive activity that requires a fair amount of physical strength to scrape samples away from the substrate. Collection time requirements included time to perform the following large list of activities:

- locate the samples;
- mark the sampling areas by cutting an outline of the sampling area down through all the paint layers;
- make a sample collection tray for each sample;
- remove the paint samples;
- transfer the paint samples to collection containers;
- measure collection areas and fill out the data forms;
- store the samples for later shipment to the laboratory;
- collect the field blanks;
- clean equipment and tools between samples;
- handle respirators;
- remove paint from areas targeted for XRF bare substrate measurements; and
- take periodic breaks.

The largest amount of time was spent on removing the paint from the sampling location.

A review of collection activities in Denver suggests that approximately 30 paint samples can be collected by a single sample collection person in an eight-hour day.

3.2.4.2 Collection Difficulties Encountered

Some difficulties were encountered during the paint collection portion of the study. These difficulties are summarized in this section.

The most common paint collection difficulties encountered during the study were related to substrate inclusion. As discussed in section 3.2.2.2, the paint chip collection rule used in this study was to collect all visible paint even if it meant collecting some of the substrate. Therefore, it was anticipated

that some substrate inclusion would be experienced. Collection of paint from plaster, drywall, brick, concrete and soft wood generally resulted in some substrate inclusion. Removal of paint from drywall samples generally resulted in a separation from the substrate at the paper layer that forms the top of this building material. Paper was commonly observed in these samples. Much of the brick and concrete encountered in Denver was very porous and had to be sampled using the cold scraping method with the aid of chisels. The use of chisels was effective for maximizing paint collection but was not particularly effective for minimizing substrate inclusion. Removal of paint from soft wood was also found to be difficult without including some substrate.

Supervisors performed several activities in addition to directing field personal. Field supervisors routinely reviewed sampling locations following completion of a specific testing activity. In Denver, a review of sampling locations following completion of paint removal activities and prior to test kit and XRF testing revealed that two locations appeared to have traces of visible paint embedded in the surface of areas where paint was originally removed for collection of laboratory samples. Additional paint collection was performed from these two areas. These extra samples were labeled with the sampling location ID numbers, 80540 and 80667, preceded by an "E" to indicate that they were extra scrapings. These extra samples were processed through the laboratory as separate samples. Both lead-concentration and lead-area results were determined for these extra samples. The extra sample lead result summed with the originally collected sample lead result gives the total lead for these two sampling locations.

A review of field forms from Philadelphia while paint collection teams were still in the field resulted in collection of additional samples at seven sampling locations. These seven samples were collected to verify originally collected samples and associated collection area measurement data. These samples, collected side-by-side to the originals, were labeled with the sample location ID numbers, preceded by an "R" to indicate that they were "repeat" samples. A statistician present in the field was consulted to provide direction on where to collect these repeat samples with respect to obtaining samples within established study objectives.

Variability in lead analysis results between the originally

collected and repeat samples, "R" samples, were less than the generally observed variability between field duplicate samples collected over the width of the sampling location template for the full study. This indicates that no significant differences exist between the original samples and the repeat samples. For statistical analysis, only the repeat sample lead results were used with one exception; sample number 81806. For this sample, the original lead result was used for statistical analysis because it was the only one of the original seven samples that had a laboratory duplicate analysis, whereas the corresponding repeat sample did not have a laboratory duplicate analysis. Laboratory duplicates are discussed in section 3.3.2.3.2. The use of the lead results from the original sample of 0.039 mg/g and 0.010 mg/cm^2 are not believed to cause any statistical analysis problems, as the lead results for the repeat sample were 0.032 mg/g and 0.007 mg/cm^2.

3.3 LABORATORY ANALYSIS OF PAINT CHIP SAMPLES

3.3.1 Selection of Laboratory Methods

3.3.1.1 Selection of Inductively Coupled Plasma-Atomic Emission Spectrometry (ICP)

The most common methods used to analyze lead in paint chip samples in the laboratory are atomic absorption spectrometry (AAS) or inductively coupled plasma-atomic emission spectrometry (ICP) preceded by a sample preparation process that includes sample homogenization, subsampling, and an extraction (digestion) procedure that uses acids to dissolve the lead in the samples. In this study, ICP was selected for measurements of lead in digested samples because this instrumental technique has multi-element capability; that is, it provides concentration data on other elements contained in the sample. As a result, this instrumental technique can offer concentration data for analytes that may be interferants using portable XRF technologies or chemical test solutions in lead test kits. Measurement processes for lead in paint using ICP techniques have been well characterized and are detailed in published analytical methods such as ASTM E1613-94. In addition, use of ICP for lead-containing paint samples is one of the techniques recommended for confirmation testing in the HUD Guidelines [2].

3.3.1.2 Selection of Sample Preparation Method

At the initiation of this study, a draft EPA report [3], indicated that a NIOSH method 7082 would be an acceptable sample preparation method for the study since it was shown to produce high lead recoveries from paint samples. NIOSH method 7082 is designed to prepare and analyze air filter samples for analysis of a wide variety of inorganic components that also included lead. Because it is specifically written for air filter samples, modifications to NIOSH 7082 are required to make it applicable to processing paint chip samples. Based on the EPA report, this method with appropriate modifications, was selected to digest paint samples for this study.

Prior to initiation of laboratory analysis on collected field paint-chip samples, a set of experiments were conducted for the following three reasons: (1) to familiarize the laboratory with the modified NIOSH method 7082; (2) to assure that the modifications to the method were appropriate, and (3) to determine the appropriate sample mass that could be processed using the modified NIOSH method 7082. Processing large sample masses was desirable because analysis of small aliquots could result in increased subsampling variance. A discussion of the experiments is presented in appendix AAA. These experiments suggested that subsample aliquot mass was an important factor in obtaining lead recovery. Therefore, sample aliquot masses were limited to a maximum of a nominal 0.5 gram to assure that satisfactory lead recoveries could be achieved.

3.3.2 <u>Laboratory Analysis Design Elements</u>

A discussion of design elements included for laboratory processing of collected field paint-chip samples is presented in this section.

3.3.2.1 Mass/Area and Mass/Mass Reporting of Data

Two different lead reporting units were selected for inclusion in the study: mass/area results, mg/cm^2, and mass/mass results, mg/g or percent by weight. Both units were included because action levels have been expressed in both kinds of units. In addition, mass/area units were needed from the laboratory to provide a means of comparing results with portable XRF data taken in the field.

Two options were available for generation of both units from the collected paint samples. One option was to digest and analyze the entire sample and divide the quantity of lead found in the sample digest by the sample area. The second option for analytical measurements was to digest and analyze a subsample and calculate the lead concentration in the collected sample from data determined on the subsample. As discussed in section 3.3.1.2, a 0.5 gram nominal sample mass limitation existed for the laboratory sample preparation process. The first option was not possible due to the design element of large collection areas discussed in section 3.2.2. Collected sample masses were expected to commonly exceed the mass limitation, 0.5 gram for effective sample extraction, imposed by laboratory processing. Actual collected sample masses from Denver ranged from 0.0454 to 25.1792 grams with an arithmetic mean of 2.4306 grams. Collected sample masses from Philadelphia had a smaller range, 0.1362 to 11.1902 grams, but a higher arithmetic mean of 3.1586 grams. Collected sample masses from Louisville ranged from 0.3116 to 4.3604 grams with an arithmetic mean of 1.7417 grams. Therefore, the second option was used in this study.

The second option for analytical measurements required homogenizing the sample, removing a sub-sample and digesting the sub-sample. To use this option, the entire sample mass, the sample area, and the sub-sample mass must be measured.

3.3.2.2 Homogenization and Subsampling

To use the selected option for analytical measurements of field paint-chip samples, a homogenization procedure was required to permit representative sub-sampling. Homogenization refers to the process of grinding and mixing the sample to achieve a uniform distribution of lead within the resulting powdered sample. The concentration data, mg/g, of the subsample is representative of the lead concentration in the collected sample only if the sample is uniformly homogenized.

The procedure used in the study to homogenize the samples was based on the fact that substances become brittle when cooled to low temperatures. A summary of the procedure is as follows:

- Place a sample inside a plastic centrifuge tube
- Immersed the container with sample into dry ice
- Allow sample to cool

- Pulverized the sample against the bottom of the sample container using a plastic rod.
- Roll the cylindrical sample containers holding the crushed samples to mix the contents.

This homogenization procedure, detailed in the appendices E and EE, was performed after determination of total sample mass to avoid any potential mass errors caused by convection losses during homogenization.

The ratio of the total sample mass to the subsample mass can be used to calculate the total amount of lead in the collected sample from the measured amount of lead in the subsample. Because the area of the sample is known, the concentration of lead in this area can be determined by dividing the lead amount in the collected sample by the measured area of the collected sample. This mass/area calculation for the collected field paint-chip sample is performed using the following mathematical expression:

$$\text{mg of lead/cm}^2 = [(A/B)(C)]/D$$

where: A = total sample mass, grams
B = subsample mass, grams
C = mg of Pb in subsample, laboratory measured
D = area of total sample, cm^2, field measured

The protocol used to measure the total sample mass and subsample mass is detailed in the appendices E, F and EE of this report.

3.3.2.3 Sample Preparation QC Samples

Sample preparation QC samples are samples submitted to the laboratory in addition to field paint-chip samples for the purposes of providing information on laboratory analysis performance, that is, the accuracy and precision of lead measurement. These QC samples were incorporated into each group of samples digested during laboratory processing. Laboratory processing of samples is performed in a batch mode of operation and a group of samples digested together is typically referred to as a sample preparation batch. A typical sample preparation batch included the following 49 samples:

- 40 field paint-chip samples
- 1 blind sample, National Institute of Standards and Technology (NIST) Standard Reference Material (SRM) No. 1579a
- 2 blind samples, American Industrial Hygiene Association (AIHA) Environmental Lead Proficiency Analytical Testing (ELPAT) performance evaluation materials
- 2 method blanks
- 4 laboratory duplicate samples

Blind samples were generated by a person independent from the laboratory while method blanks and laboratory duplicate samples were generated by the laboratory. These three types of QC samples are summarized in Table 3-4 along with targeted data quality objectives for the study. A detailed discussion of these three types of QC samples is provided in the following three sections.

3.3.2.3.1 Blind Samples

Blind samples were included in each batch of field paint-chip samples processed in the laboratory. A blind sample is a sample submitted for analysis whose composition is known to the submitter but unknown to the laboratory. Lead recovery data from the blind samples were used as an assessment of accuracy on field paint-chip samples as determined by sample preparation and

Table 3-4. Summary of Sample Preparation QC Samples.

Sample Type	Frequency	Data Quality Objective
Method Blank	1 per 20 field paint-chip samples; minimum of 1 per batch	Measured value less than 10 times the Instrumental Detection Limit (IDL), see Chapter 4 for a detailed discussion of IDL.
Blind Sample: NIST SRM No. 1579	1 per batch	Accuracy of ±25% from certified concentration
Blind Sample: ELPAT	2 per batch	Accuracy of ±25% from consensus concentration Range of duplicate % recoveries of <20%
Laboratory Duplicate Sample	4 per batch	None

analysis activities. The following two types of blind samples were included in each sample preparation batch: NIST SRM 1579a and ELPAT samples.

At the time the laboratory work was performed for this study, NIST SRM 1579a was the only certified lead-based paint sample material available from NIST. Because of the high lead concentration in this SRM, 11.995%, it was desirable to find an additional real-world material that contained lower known lead concentrations, closer to the lead concentrations anticipated in collected field paint-chip samples. Since paint performance evaluation samples, from rounds 02 and 03 prepared for the ELPAT program, were available from AIHA, they were selected to complement the NIST SRM for use as blind samples. Known lead concentrations for the ELPAT samples were consensus determined during the ELPAT program and were provided by AIHA.

Blind samples were generated by placing up to approximately 1 gram of the targeted sample material into clean-labeled, field-sample containers followed by inserting the blind samples into the stream of samples submitted to the laboratory for analysis. For the NIST SRM No. 1579a, blind samples were typically 1 gram aliquots. However, for the ELPAT samples, only limited quantities were available from AIHA. For these blind samples,

the entire contents of the sample container received from AIHA, approximately 0.5 gram, was transferred into a clean field paint-chip sample container.

Labeling used for identification of blind samples was equivalent to that used for the field paint-chip samples. Blind samples were included within groups of field paint-chip samples after field paint-chip samples were homogenized in an effort to submit blinds that were indistinguishable from field paint-chip samples. However, blind samples could not be considered as double blinds because the physical appearance of the field paint-chip samples was typically different than the very finely powdered blind samples.

One NIST and two identical ELPAT samples were placed into each batch of samples digested in the laboratory. The duplicate ELPAT samples were used to provide within-batch repeatability from sub-sampling, extraction and instrumental measurement. Accuracy and precision generated from the blind samples were monitored during the study for performance trends and feedback to the laboratory on analysis performance.

3.3.2.3.2 Laboratory Duplicate Samples

As discussed in section 3.3.2.2, laboratory analysis processing of samples included sample homogenization and sub-sampling procedures. The accuracy of lead data obtained from the laboratory was dependent upon the homogeneity of the paint samples before subsampling.

Laboratory sample preparation and analysis of duplicate subsamples were included as a design element to provide an estimate of the variability caused by the homogenization process. Laboratory duplicate subsamples were processed at a rate of 10%; that is one duplicate sample was processed for every 10 field paint-chip samples. Because duplicate processing of samples consumed a minimum of 1 gram and because of the desire to avoid complete exhaustion of any collected sample, samples with total collected sample mass greater than 1.5 grams were targeted to be used for laboratory duplicates.

3.3.2.3.3 Method Blanks

Method blanks were included in the batches of field paint-chip samples processed in the laboratory. A method blank is a mixture of reagents used for the digestion of field paint-chip samples. This mixture contains no sample matrix and is carried through all steps of the digestion and instrumental measurement process. Method blanks provide information on the potential systematic lead contamination of field resulting from laboratory processing. Method blanks were generated at a frequency of two per batch.

Laboratory technicians inadvertently missed inserting method blanks in two (2) of the 39 sample preparation batches processed for the pilot and full studies. However, at least two field paint-chip samples within each of these two sample preparation batches included non-detectable lead measurements ranging from 0.0058 mg of lead per sample to 0.0062 mg of lead per sample. Therefore, sufficient data was available to assure that no systematic lead contamination of field paint-chip samples occurred in any of the 39 batches as a result of laboratory processing.

3.3.2.4 Instrumental Measurement QC Samples

Instrumental measurement QC samples were analyzed along with field paint-chip samples during instrumental measurement activities to assure adequate instrument performance. These QC samples included daily calibration standards, multiple calibration verification standards, multiple calibration blank samples, and interference check standards. Table 3-5 contains a detailed description, specifications, and frequency of use of the instrumental measurement QC standards. Additional detail on instrumental QC samples is presented in appendices G and FF. All specifications presented in Table 3-5 were met for all reported lead analysis laboratory data.

3.3.2.5 Sample ID Transfer

As presented in section 3.1.6, control over the potential for misidentified sampling locations was achieved through the use of pre-printed sticky-back barcode labels. Samples collected in the field were identified with the barcode label sticker on the sample container and on the entry line of the data form

associated with the sample. Additional barcode labels were placed into a plastic bag along with the sample container for in-laboratory processing of the sample. These extra barcode labels were used to identify laboratory forms and containers used in the digestion and instrumental measurement steps for each sample.

For the pilot study, field duplicate samples collected in the field were identified on the collection containers by inscribing a "D" or "DUP" at the end of the sample location ID number on the container. For laboratory processing of the pilot samples, a "LDP" suffix was used to designate and track

Table 3-5. Instrumental QC Standards and Specification for ICP.

Name	**Use**	**Specification**
ICB - Initial Calibration Blank	Used for initial calibration and zeroing instrument response.	Calibration Standard which contains no lead. Must be measured during calibration and after calibration. Measured value to be less than 5 times the IDL (see Chapter 4).
Calibration Standards	Used to Calibrate instrument. The high standard re-run is used to check for response linearity.	Acid content must be approximately the same as that in the sample digests. Must be measured prior to measuring any sample digests. Correlation Coefficient of $\geq$0.995, as measured using linear regression on instrument response(y) versus concentration(x). The highest level Calibration standard must be measured after calibration. The measured value to fall within $\pm$10% of known value.
ICV - Initial Calibration Verification	Used to verify calibration standard levels.	Concentration of lead to be near the middle of calibration curve. It is made from a stock solution having a different manufacturer or manufacturer lot identification than the calibration standards. Must be measured after calibration and before measuring any sample digests. Measured value to fall within $\pm$10% of known value.
ICS - Interference Check Standard	Used to verify accurate lead response in the presence of possible spectral interferences from other analytes present in samples.	Concentration of lead to be less than 25% of the highest calibration standard, concentrations of interferant are 200 µg/mL of Al, Ca, Fe, and Mg. Must be analyzed at least twice, once before and once after all sample digestates. Measured lead value to fall within ±20% of known value.
CCV - Continuing Calibration Verification	Used to verify freedom from excessive instrumental drift.	Concentration to be near the middle of the calibration curve. Must be analyzed before and after all sample digestates and at a frequency not less than once every ten samples. Measured value to fall within ±10% of known value.
CCB - Continuing Calibration Blank	Used to verify blank response and freedom from carryover.	Calibration Standard which contains no lead. Must be analyzed after each CCV and each ICS. Measured value to be less than 5 times the instrumental detection limit.

laboratory duplicate samples so that there would not be confusion in the laboratory because of the historical use of "D" suffixes to designate laboratory duplicate samples discussed in section 3.3.2.3.2. For example, a laboratory duplicate for a sample that was also a field duplicate sample would be given the designation of "D-LDP" or "DUP-LDP".

For the full study, an improvement was made to the sample identification design for the field duplicate samples. A separate set of barcode labels was used to identify the field duplicate samples using an "S" designation to avoid any potential laboratory confusion. For each sampling location, a set of barcode labels was produced with the location ID number preceded by an "S" prefix. These barcode labels were placed at sampling locations that were targeted for collection of field duplicate paint samples, used for field forms, and were submitted to the laboratory along with the field duplicate samples for use in the laboratory. Laboratory duplicate sample preparations were identified using a "D" prefix to the sample ID number in accordance with historical precedent.

3.3.3 Summary of Laboratory Processing

Paint chip samples were grouped together and processed through the laboratory in batches. Batching refers to the process of grouping the samples together for submission to performance of specific laboratory tasks. A total of three sample batchings were performed within the laboratory. The tasks performed in each designated batch include the following:

- weighing (1st batching)
- homogenization (1st batching)
- re-weighing (1st batching)
- sub-sampling (1st batching)
- sample digestion (2nd batching)
- instrumental measurement (3rd batching)

Details on these tasks are presented in the appendices E, F, G, EE, and FF of this report.

The total laboratory processing scheme, diagrammed in Figure 3-3, presents the three sample batching processes. Each of these batching processes is discussed below along with a summary of the

laboratory activities preformed on the samples within each batch.

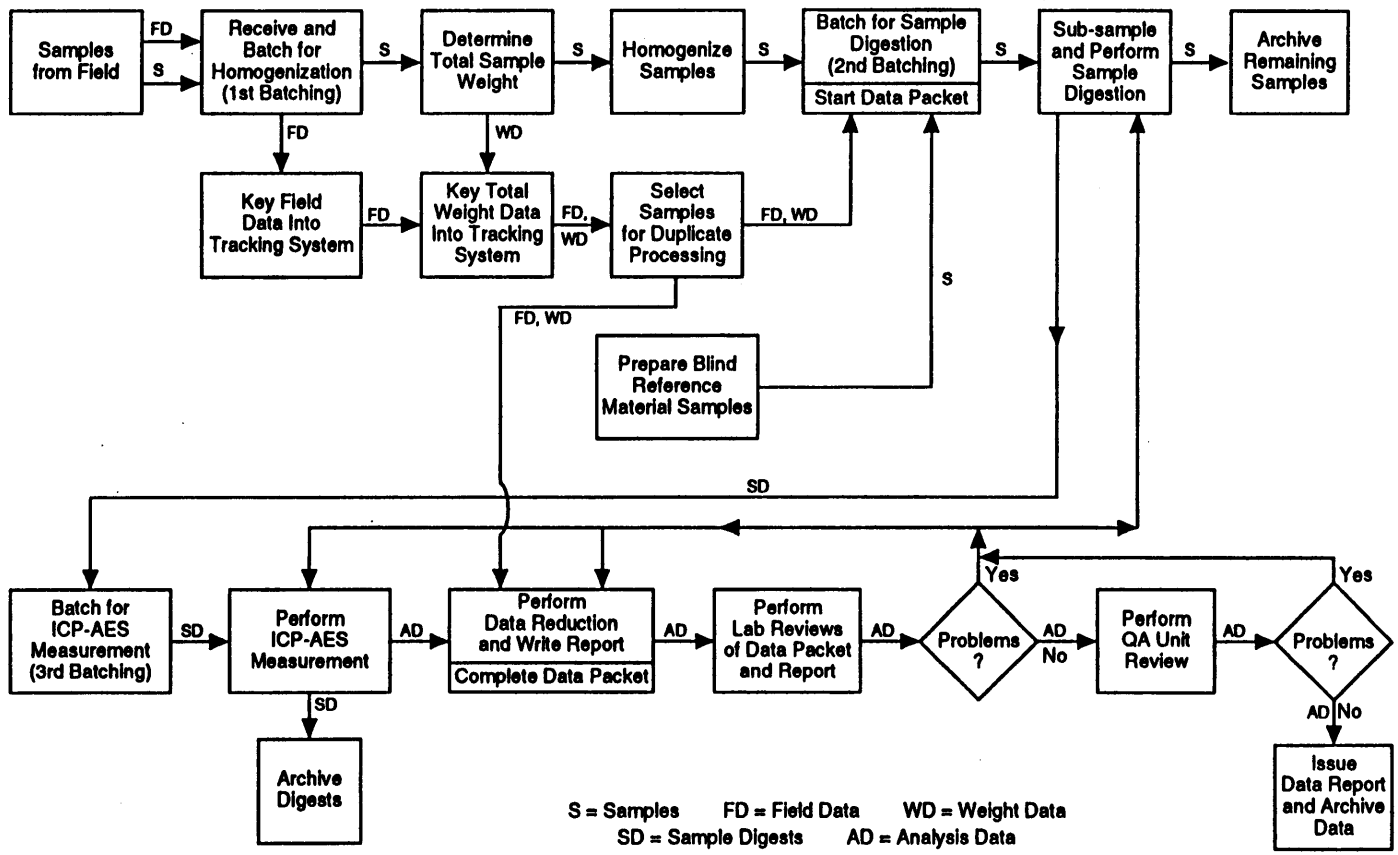

Figure 3-3. Flow Diagram of Laboratory Processing

3.3.3.1 First Batching of Paint Chip Samples

The first batching of samples was the grouping of samples to determine sample mass and homogenize the samples. The number of samples in each batch varied from batch to batch. Usually a batch contained less than 100 samples.

Total sample mass was determined by subtracting the original field collection container mass, or empty sample container mass, from the total paint sample plus container mass. Protocols for performing total sample mass determinations are detailed in the appendices E and EE of this report.

Homogenization of all samples within the batch, summarized in section 3.2.2.2, was performed after determination of total sample mass.

3.3.3.2 Second Batching of Paint Chip Samples

The second batching of samples was the grouping of samples for acid digestion. A typical batch, described in section 3.3.2.3, included 40 field paint-chip samples and nine sample preparation QC samples for a total of 49 samples.

This batching was performed, by a designated sample custodian who was independent of the laboratory, in a manner that distributed samples from each housing unit among a minimum of three different sample preparation batches. This distribution was achieved by randomly selecting a sample collected from each housing unit in a sequential manner until the number of field paint-chip samples needed to make up a sample preparation batch, 40, was reached. Distributing samples in this manner was done to prevent the loss of an entire housing unit under the potential conditions of a complete failure in processing an individual sample preparation batch. In addition, batching was performed so that field duplicate samples would not be processed in the same batch. This assured that the variability between field duplicate samples would include between-batch variability.

After assembling a batch, the total sample mass data for samples within the batch was reviewed to identify a subset of samples that could be used for laboratory duplicate samples. All samples with total sample mass greater 1.5 grams were identified as candidates for laboratory duplicate samples as described in

section 3.3.2.3.2. Four (4) of these samples were randomly assigned for duplicate extraction and analysis. Prior to submission of the batch to the laboratory, the sample custodian inserted the blind samples into each batch as discussed in section 3.3.2.3.1.

3.3.3.3 Third Batching of Paint Chip Samples

An instrumental measurement batch is a group of digested paint samples that are analyzed together in a sequential manner following calibration of the ICP instrument. An instrumental measurement batch size can vary from one to approximately 200 digested paint samples. Because it is generally more efficient to process large batches than small batches, a third batching of paint chip samples was performed prior to instrumental measurement to group together samples from multiple sample preparation batches. This batching was performed by laboratory personnel on an as-needed basis as samples became available for instrumental lead measurement. Because only a small volume of the digest, 10 mL, was required for instrumental lead measurement, replicate analyses could be performed if needed. For example, replicate analysis would be required if an instrumental measurement QC sample failed to meet the specifications as described in section 3.3.2.4.

3.3.4 Summary of Laboratory Data Handling and Reporting

Similar to original XRF and test kit field data, original paint chip sample data generated in the field was hand carried back to the laboratory by field personnel upon completion of the field activities. Copies of original data forms were made and transmitted to the statistical analysis team on a routine basis during performance of field activities to provide for backup in case of inadvertent loss of original data forms. In addition, copies were also shipped with field paint-chip samples to the laboratory. The designated sample custodian received the field paint-chip samples and recorded their receipt and field collection data into a computerized system for tracking samples.

During the laboratory processing of the samples, a data packet was used to store and transmit sample information. The data packet is a folder that contains all laboratory-generated data and copies of field data forms received with samples. Following the instrumental analysis, information placed into the

data packet was used to produce a final data report. The final data report, including both hard copy results and electronic results, was sent to the statistical analysis team for data analysis.

Electronic data transfer, via floppy disk, was used to transmit analytical data between the laboratory and the statistical analysis team. Laboratory data, identical to the data presented in the hard-copy final data reports, were transmitted using data tables in a fixed ASCII format for retrieval and data analysis by the statistical analysis team.

Errors in analytical data from the laboratory were minimized by using four methods. The first was to use separate raw data entry and calculation spreadsheets. Only the calculation spreadsheet was used to generate data tables that were transmitted to the statistical analysis team. Raw data was only allowed on the raw data entry spreadsheets. After completing raw data entry, calculations and table generation was performed by electronically moving raw data into the calculation spreadsheets. Use of the raw data entry spreadsheets provided protection against inadvertent alteration of the mathematical formulas used to calculate data. Spreadsheets were created and verified using a set of test raw data to determine that formulas contained in the calculation spreadsheet were free from errors. Once created and verified, all formulas in calculation spreadsheet were electronically protected from alteration.

The second method of minimizing errors was the use of direct electronic transfer of instrumental measurement data from the ICP into the raw data entry spreadsheets used for data reduction.

The third method of minimizing errors, a double key entry system, was to perform 100% entry of the hand entered raw data and electronic transfer of instrumental measurement data into the spreadsheets by a second person. Entry errors were identified by subtracting the entire duplicate electronic raw data entries from the original electronic raw data entries. Non-zero values indicated entry errors which were corrected before finalizing data reports.

The final method of minimizing errors was the review and sign off of the data packet, and the review of the final data report by appropriate laboratory, quality assurance, and

supervisory personnel who were involved with the laboratory handling of the samples and sample data. Quality assurance personnel, independent of the laboratory, performed a number of data verifications on random samples within each data packet prior to transfer of any data to the statistical analysis team. A summary of quality assurance review activity and findings is presented in chapter 7 of this report.

3.4 TEST KIT MEASUREMENT DESIGN

A discussion of the design elements related to investigation of test kit performance is presented in this section.

3.4.1 Test Kit Selection

Two basic test kit technologies for detection of lead in paint were available on the market as of the Spring of 1993. The first is based on the formation of black lead sulfide by the reaction of lead in paint with sodium sulfide. The second is based on the formation of a pink complex by reaction of lead in paint with sodium rhodizonate. There are several commercially available kits based on each technology. The kits differ in their testing protocols, delivery systems, and sometimes in the exact formulation of the reagent. This study was designed to examine the performance of a representative set of test kits under real field conditions.

A total of eight test kits were investigated for potential inclusion in the study as listed below:

1. "Acc-U-Test." Manufactured by South Shore Lead Paint Testing Company. This is a sodium sulfide based kit.
2,3. "LeadCheck." Manufactured by Hybrivet Systems. This is a rhodizonate based kit (also called LeadCheck II). This company also manufactures a sodium sulfide kit referred to as LeadCheck I.
4,5. "Lead Alert" and "Lead Alert All-in-One." Manufactured by Frandon/PACE Enterprises. Both are rhodizonate based kits.
6. "Lead Detective." Manufactured by Innovative Synthesis Corporation. This is a sodium sulfide based kit.
7. "Lead Zone." Distributed by Enzone Corporation. This is based on a proprietary formula.
8. "State sodium sulfide kit." The state of Massachusetts has its own sodium sulfide formulation and test protocol, which

must be carried out by certified and licensed inspectors. No professional test kit testing programs other than the one offered by the state of Massachusetts were available during the design of this study.

A total of six test kits, representing the range of test kits available to the public at the time the study was designed, were included in the study. As of the spring of 1993, the three test kits believed to be the leading brands were LeadCheck, Lead Alert, and Lead Detective. Therefore, these were included because it was believed that these kits were being purchased by consumers. In addition, the Lead Alert All-in-One, the Lead Zone and State sodium sulfide kits provided features that were not present in other the three leading brands. These were also included in the study.

The kits included in the study are discussed in this section. All discussions presented in this section are based on the test kits that were purchased directly from the manufacturers in the spring and summer of 1993. Changes made to the kits by manufacturers since this time period are, obviously, not part of this work and, therefore, are not discussed.

3.4.2 General Test Kit Measurement Design Elements

3.4.2.1 Test kit Measurement Areas

Each test kit measurement was performed within one of the six rectangular shaped test area blocks marked at each sampling location as previously described in section 3.1 and shown in Figures 3-1 and 3-2. For the full study, each of the test area blocks was randomly marked with a different letter "A" through "F". A hand held calculator was programmed to perform random assignment of the letters to the test area blocks. Test kit measurements were, on the average, performed at approximately an equal distance from the collected paint chip sample by using the lettered testing area blocks and by permanently assigning one of the letters to each test kit. Test kit measurements were performed on the correspondingly lettered test area blocks.

Lettering of the testing area blocks for test kit measurements was a study design improvement that was not part of the pilot study. For the pilot study, testers were spread-out among the sampling locations to reduce the potential for

communication of test results between testers. Testers were directed to use the first unused testing area block available starting from the left hand side for the horizontally drawn sampling location template or the bottom side for a vertically drawn sampling location template as shown in Figures 3-1 and 3-2. Similar to the full study, the test kit measurements were, at an average, performed at an approximately equal distance from the collected paint chip samples. However, in this case, this result was a by-product of the testing directives as opposed to a purposely planned design element.

3.4.2.2 In-Field Access Control of Test Kit Measurement Data

Potential bias caused by inadvertent passage of testing results between testers was an active concern during planning stages of the study. Four procedures were used to minimize this potential information passage for the full study. First, all test kit operators were explicitly told during training that no discussions on results were to be voiced during the field work. Second, in the full study, test kit measurements were performed in two units simultaneously (three testers per unit) to reduce the number of people within a building at any given time. Third, all written test kit protocols included covering the tested location with duct tape immediately after the test was completed (or, for one kit, a disposable beaker was used in place of the duct tape). This assured that any following tester would not be able to easily see or interpret a previous tester's result. Finally, all test kit data forms were collected on site by the acting field supervisor at the end of each day of testing. No field data were allowed to remain in the hands of any given tester overnight. These four procedures were also used during the pilot study with the exception of the second procedure. As discussed in section 3.4.2.1, inadvertent passage of testing results in the pilot study was controlled by spreading testers out among the sampling locations as opposed to limiting the number of testers in each unit.

3.4.2.3 Test Kit Testing Personnel

All test kits selected for use in the study, with the exception of the State sodium sulfide kit, were available for purchase by the general public. Instructions supplied with the kits were written for non-technical users. Therefore, all the

kit measurements, with exception of those performed using the State sodium sulfide kit, were performed using simulated homeowners to perform the tests in a manner compliant with the intended market.

Selection criteria for the simulated homeowners included individuals who could follow instructions, who were capable of clearly recording data on data forms, and who did not have any extensive experience using field test kits for lead measurements. In addition, selection was limited to those without bench-science backgrounds, since such testers would have an unusual degree of expertise in chemical testing as compared to a typical homeowner. All individuals targeted for selection were checked for potential color blindness and removed from consideration if found to have this genetic disorder. Such a disorder would have seriously handicapped their ability to make positive lead identification with the rhodizonate-based kits.

Full study testers were recruited locally by placing help wanted advertisements in local newspapers combined with thorough background screening and one-on-one interviews. Pilot study testers were recruited from the staff pool of one of the prime contractors for the study. A summary of simulated homeowner backgrounds is presented in Tables 3-6, 3-7 and 3-8. Only one of the simulated homeowners used, one in Philadelphia, had any previous experience using test kits. This person had used one kit provided by a public housing authority back in 1987. This limited, not recent experience, was not believed to be in conflict with the desire to simulate a homeowner.

The simulated homeowners received some limited in-field training to perform test kit measurements for all the kits in the study, with the exception of the State sodium sulfide kit, using the established protocols. The limited training consisted of a review of the project, study objectives, test kit protocols, and reporting practices to be followed. Training was purposely performed as close to the start of field measurements as possible. For the pilot study, training was conducted off-site immediately before traveling to Louisville. For the full study, training was conducted on-site in each city one day prior to initiating testing.

3.4.2.4 Test Kit Assignments and Performance Order

For the full study, test kit assignments for the simulated homeowners were rotated so that each simulated homeowner performed tests using each of the test kits. In the pilot study, test kit assignments were held constant with the exception that one kit, Lead Alert: Coring (see section 3.4.3.1.2), which was assigned to an additional person as an aid to complete testing on schedule. For the full study, when permitted under field conditions, test kit assignments were changed each time a new housing unit was started. These assignment changes were incorporated as a design element to aid in reducing the potential for a tester becoming too familiar with a given test kit as a result of repetitive testing at an excessively large number of locations. It was believed that, under standard testing conditions, a real homeowner would not have the opportunity to gain experience from large numbers of repetitive tests. More frequent rotation of test kits between personnel than the change

Table 3-6. Background Summary of Simulated Homeowners Used for Operation of Test Kits in Louisville.

Person[a]	Background
RD	Male, BS in Mathematics
TH	Male, BA in Business Administration
JJ	Male, BS in Mechanical Engineering
KL	Female, Administrative Assistant
LY	Female, Administrative and Technical Assistant

[a] Initials of person

Table 3-7. Background Summary of Simulated Homeowners Used for Operation of Test Kits in Denver.

Person[a]	Background
DD	Male, retired from military, experienced in Health Care
HF	Male, BS in Geology, pursuing masters degree in Geology
AG	Male, BS in Environmental Design, pursuing degree in Civil Engineering.
BH	Male, pursuing bachelors degree
BN	Male, retired from military, pursuing bachelors degree in Science and Engineering
KS	Male, pursuing bachelors degree in Earth Sciences

[a] Initials of person

Table 3-8. Background Summary of Simulated Homeowners Used for Operation of Test Kits in Philadelphia.

Person[a]	Background
DC	Male, two years of college in business management
JM	Male, 2 years of college
PM	Female, BS in Civil Engineering
GM	Female, two yr degree in Computer Science
MS	Female, degree in Art
DY	Male, degree in Mathematics

[a] Initials of person

each time a new housing unit was started was not considered practical under field conditions and resource restrictions experienced during the study.

For the full study, the test kit measurements were performed prior to any collection of paint chip samples. As previously discussed in section 3.1.4, this order of testing was done to force the testers to make a decision as to when they had reached the last layer of paint without the visual aid provided by the bare substrate area generated during paint chip collection. This was different than what was performed during the pilot study where collection of paint samples was performed at about the same time as the test kit measurements.

For the full study, a total of six simulated homeowners were used in both Denver and Philadelphia. In Denver, one of the simulated homeowners was used more for general field support activities rather than as a primary test kit tester. This person performed only a small number of measurements using one of the test kits, less than 7% of the measurements for that kit, as opposed to all the other testers, who each performed a larger number of test kit measurements using all of the test kits targeted for use by simulated homeowners. A summary of the number of test kit measurements made by each tester is presented in Table 3-9. For the pilot study, a given test kit was assigned to a specific tester for all test locations as opposed to using a rotation mechanism incorporated into the design for the full study. Towards the end of the test kit measurement period, one of the testers that had completed his assigned testing was put to work as a second tester, making measurements with one of the other test kits. This was required to complete this phase of testing because the performance of the other kit was proceeding at a much slower rate than the others. Twenty percent of the sampling locations for this other kit were performed by the second tester.

3.4.3 Test Kit Descriptions and Protocol Summaries

The final test kit protocols, used for both the pilot and full studies, were based on the original manufacturer's instructions supplied with the test kits purchased directly from the manufacturers with a few exceptions that are discussed in sections 3.4.3.2, 3.4.3.3, and 3.4.3.4. To avoid potential in-field changes of established protocols, manufacturer

representatives were not allowed to come to the testing site during performance of the study. Manufacturers original test kit instructions were re-written to clarify testing steps and to

Table 3-9. Summary of Number of Test Kit Measurements Made by the Simulated Homeowners.

Person	City	Number of Measurements				
		LeadCheck	Lead Alert: Coring	Lead Alert: Sanding	Lead Detective	Lead Zone
RD	Louisville	0	20	100	0	0
TH	Louisville	0	0	0	0	99
JJ	Louisville	0	0	0	100	0
KL	Louisville	0	80	0	0	0
LY	Louisville	100	0	0	0	0
DD	Denver	150	210	60	74	150
HF	Denver	0	51	0	0	0
AG	Denver	150	188	61	150	75
BH	Denver	150	150	46	150	150
BN	Denver	149	75	36	224	148
KS	Denver	151	75	60	150	225
DC	Philadelphia	108	57	40	109	55
JM	Philadelphia	57	86	37	53	18
PM	Philadelphia	55	79	62	110	57
GM	Philadelphia	55	54	78	55	75
MS	Philadelphia	55	55	58	55	143
DY	Philadelphia	110	108	40	57	92
Totals		1290	1288	678	1287	1287

include items specific to the study, such as the consistent pre-preparation of test surfaces and recording of test results using pre-printed data reporting forms.

The data forms used for the full study included recording blocks for the sampling location number, starting time for the test, the time that the observation of the testing result was made, a positive or negative assessment of the result, and a comment area. Within the comment area were a set of five shaded blocks for use by the sodium sulfide based kit testers to use to record the observed darkness of the test result.

Some test kits used in the study had manufacturers instructions for partial paint testing (such as surface-only lead detection). Partial paint testing procedures were not included in the study because of their lack of comparability to ICP results.

All manufacturers were provided with copies of written study protocols prior to full study field testing activities (Denver and Philadelphia) and were requested to comment on them for potential corrections or clarifications. The test kit protocols used in the study are summarized in section 3.4.3.1 below and are presented in the appendices of this report.

3.4.3.1 Descriptions of Test Kits Included in the Full Study

A description of the test kits along with a summary of the protocols used in the full study is presented below.

3.4.3.1.1 Lead Alert (Kit No. 1040)

The Lead Alert kit No. 1040 used for the study, referred to as Lead Alert: Sanding, is a rhodizonate based testing kit. The kit contained sand paper, a leaching solution, an indicating solution with a chemical tablet, cotton swabs, and a verification test card. The indicating solution was prepared by placing a supplied chemical tablet into a specified dropper bottle followed by a vigorous shaking procedure. Additional sand paper was obtained from the manufacturer for use in this study. Additional cotton swabs were purchased locally for use as needed to perform testing.

The test location (painted surface) was prepared by cleaning the surface with a baby wipe followed by taping a paper funnel immediately below the sampling location to catch any removed paint. Sand paper was used to remove the paint down to the substrate surface.

Testing was performed by applying two drops of leaching solution to a cotton swab followed by picking up a small amount of the fine paint particles onto the moistened swab from paint in the paper funnel. Another drop of leaching solution was added over the paint particles on the applicator tip and allowed to leach the lead from the paint for 30 seconds. Two drops of

indicating solution were added and the cotton swab was then examined for a color change. A pink to rose/red color was considered positive for lead. A orange or yellow color was not considered positive for lead. The verification test card, which contains lead, was used to verify the integrity of the chemical reagents upon opening each new kit for a given day of testing. A positive test result on the verification test card indicated that the chemical reagents were working properly and that the kit could be used for testing sampling locations. Only new kits were used for each day of testing. Test kits from previous work days were kept separate from new kits and were disposed of as chemical waste at the end of the study. A total of 20 double tipped cotton swabs were supplied with the kit for a maximum of 40 tests per kit.

Experience gained from the pilot study use of the sanding portion of this test kit, discussed in section 3.4.3.3, resulted in reducing the planned number of sampling locations for this test kit to 25% of the total. This smaller targeted number of sampling locations was planned because of the large differences in execution time between this test kit using the sanding method and other test kit methods. Testers were instructed to perform measurements at additional locations if time was available after completing the planned number of sampling locations. As shown in Table 3-9, the total number of sampling locations that were actually completed in the full study was close to 49% of the total (578 out of 1190).

3.4.3.1.2 Lead Alert All-in-One (Kit No. 1010)

The Lead Alert All-in-One kit No. 1010 used for the study, referred to as Lead Alert: Coring, is a rhodizonate-based testing kit clearly marked in the supplied instruction set as for use by "do-it-yourselfers". The kit contained supplies and materials to perform lead testing using three different methods: a total lead method using a coring technique, a surface lead method using a sanding technique and a total lead method using a sanding technique. The total lead method using the coring technique was selected for use in the study. The other total lead method using the sanding technique contained in the kit was the same as that described for the Lead Alert kit which was included in the study and is described in section 3.4.3.1.1. The total lead method using the coring technique contained a leaching solution, an indicating solution with a chemical tablet, plastic

rods, small plastic vials, cotton swabs, a pad of sticky edged paper squares, and a verification test card. A coring tool and brush were supplied by the manufacturer separate from the kit. The indicating solution was prepared by placing the chemical tablet into a specified dropper bottle followed by a vigorous shaking procedure.

The test location (painted surface) was prepared by cleaning the surface with a baby wipe followed by sticking a folded square of paper immediately below the sampling location to catch any removed paint. A clean coring tool was used to remove the paint down to the substrate surface. Coring tools were cleaned between each use by wiping with a tissue and repeated brushing out using the coring tool brush.

Testing was performed by placing the removed paint sample into a plastic vial, crushing the sample with a plastic rod, adding leaching solution, and then followed by adding indicating solution to a cotton swab and touching the swab to the paint-leaching solution mixture in the plastic vial. The cotton swab was examined for a color change. A pink to rose/red color was considered positive for lead. The verification test card, which contained lead, was used to verify the integrity of the chemical reagents upon opening each new kit for a given day of testing. A positive test result on the verification test card indicated that the chemical reagents were working properly and that the kit could be used for testing sampling locations. Only new kits were used for each day of testing. Test kits from previous work days were kept separate from new kits and were disposed of as chemical waste at the end of the study. A total of 20 plastic vials and rods were in each kit for a maximum 20 tests per kit.

3.4.3.1.3 LeadCheck

The LeadCheck kit used for the study is a rhodizonate based testing kit. The kit box, purchased in bulk, contained 12 swabs, one for each test, and several verification test cards. Each swab was constructed from a cylindrical paper tube tipped with a fibrous applicator. Inside the tube were two chemical containing ampules (assumed to be a leaching solution and an indicating solution).

The test location (painted surface) was prepared by cleaning the surface with a baby wipe followed by exposing all layers of

paint down to the substrate surface (not cutting into the substrate) using a utility knife to cut through the paint.

Testing was initiated by crushing both chemical containing ampules inside the swab followed by squeezing the chemicals up into the fibrous applicator. The testing was completed by rubbing the chemical containing applicator tip on the exposed paint layers for 30 seconds and examining the fibrous swab tip for a color change. If a pink to red color was observed on the swab tip, then the test was considered positive for lead. If no color change was observed, the verification test card, which contains lead, was used to verify the integrity of the chemical reagents in the swab that was used to generate the indicated negative test result. If the chemicals were shown to be working through the use of the verification test card, the negative test result was confirmed by observing the notches for potential color changes at 30 minute and 60 minute intervals. If the chemicals were shown to be not working through the use of the verification test card, then a new swab was used for the test. If no color change was observed after the 30 minute and 60 minute intervals, then the test was negative for lead.

Because of the requirement to examine negative results over time, all test locations for this test kit were covered with a small disposable beaker using tape to permit undamaged examination at a later time. The beakers were temporarily removed to perform the later time period examinations. The beakers were placed over the measurement area regardless of the outcome of the test to avoid passage of testing information to other testers.

3.4.3.1.4 Lead Detective

The Lead Detective kit used for the study is a sodium sulfide based testing kit. The kit contained two pairs of plastic gloves, a magnifying glass, four single edged razor blades, a pair of plastic tweezers, a bottle containing water, a dropper bottle containing sodium sulfide crystals, and a plastic bag containing a lead paint chip. The sodium sulfide solution was prepared by pouring the water into the dropper bottle containing the crystals followed by a vigorous shaking procedure.

The test location (painted surface) was prepared by cleaning the surface with a baby wipe followed by exposing all layers of paint down to the substrate surface (not cutting into the substrate) using a utility knife to cut through the paint. The removed paint chip was retained for potential further testing.

Testing was performed by applying the sodium sulfide solution to the exposed paint layers followed by examination for a color change. A black or gray color was considered positive for lead. Negative or doubtful observations were further investigated by testing the paint chip removed from the sampling location during cutting through the paint. The lead paint chip supplied with the kit was used to verify the integrity of the chemical reagents upon opening each new kit for a given day of testing. A positive test result on the lead paint chip indicated that the chemical reagents were working properly and that the kit could be used for testing sampling locations. Only new kits were used for each day of testing. Test kits from previous work days were kept separate from new kits and were disposed of as chemical waste at the end of the study.

3.4.3.1.5 Lead Zone

The Lead Zone kit used for the study is based on a proprietary formula. The kit contained a chemical impregnated pad, a plastic dropper and a verification test card.

The test location (painted surface) was prepared by cleaning the surface with a baby wipe followed by exposing all layers of paint down to the substrate surface (not cutting into the substrate) using a utility knife to cut through the paint.

Testing was performed by wetting a section of the chemically impregnated pad with a few drops of water followed by pressing the pad against the exposed paint layers for a two minute period and examining the pad for a color change. If a pink to purple color was observed on the pad, then the test was considered positive for lead. For the full study, ASTM type I water was supplied to the field testers from the laboratory. For the pilot, locally obtained bottled drinking water was used. The integrity of the chemical reagents in the pads was verified using the verification test card, which contains lead. The verification test card was used after opening each kit and after each negative result by pressing the wetted section of

impregnated pad against the verification card. A positive test result on the verification test card indicated that the chemical reagents were working properly. A total of six sections of pads were in each kit for a maximum five tests per kit, as one pad is used-up for the initial verification check.

3.4.3.1.6 State Sodium Sulfide Kit

The State sodium sulfide kit is a lead measurement procedure that is performed by a professional lead inspector, trained and licensed by the state of Massachusetts. This test kit uses an approximately 6% to 8% aqueous solution of sodium sulfide for making lead measurements. This is the same concentration of sodium sulfide solution supplied with the Lead Detective test kit described in section 3.4.3.1.4. The primary difference between these two kits is that the State sodium sulfide kit is performed by a professional lead inspector and the sodium sulfide solution must be obtained from the State of Massachusetts. All other equipment needed to perform measurements using this kit was provided by the professional lead inspector.

The test location (painted surface) was prepared in a manner similar to that described in section 3.4.3.1.4; the surface was cleaned with a baby wipe followed by exposing all layers of paint down to the substrate surface (not cutting into the substrate) using a utility knife to cut through the paint.

Testing was performed by applying the sodium sulfide solution to the exposed paint layers using a cotton tipped swab followed by examination for a color change. A black or gray color was considered positive for lead. The protocol for the State sodium sulfide kit called for removing paint chips from metal substrates and testing the chip separate from the metal surface. Under some conditions, as judged by the professional lead inspector, comparison tests on adjacently placed cuts were used to aid in making a determination of positive or negative results. In these cases, comparisons would be performed by applying water to one of the cuts and sodium sulfide solution to the other.

3.4.3.2 Manufacturer Instruction Changes for the Full Study

As described in section 3.4.3, the final test kit instruction sets were written following the manufacturer's instructions with a few exceptions. One exception to these instructions was incorporated into the instruction set used in the full study for the Lead Detective kit. The manufacturers instruction set for this kit required that the sodium sulfide solution be applied to the paint surface without touching the tip of the reagent bottle to the paint. However, in practice, this is very difficult and sometimes not possible for vertical surfaces. Therefore, a step was added to the protocol to include the use of a cotton tipped swab to apply the reagent to the painted surface. This was the same technique used by the professional lead inspector for applying the reagent to the painted surface described in section 3.4.3.1.6.

Second, lead acetate impregnated test strips were included into the general test kit supplies for the study to perform verification tests of the kits on an as needed basis. In general, these test strips were used to provide backup replacements for verification test cards for LeadCheck, Lead Alert, Lead Alert All-in-One, and Lead Zone, and backup replacements for the lead paint chip for Lead Detective. Because of the large number of tests performed during the study, the supplied verification test cards and lead paint chips were not always sufficient to meet the testing needs. The lead acetate impregnated test strips were only sporadically used and only when test kit supplied materials were insufficient to meet needs. The lead acetate impregnated test strips contained approximately 0.5% lead by weight. They were sized such that the lead per unit area contents were approximately 0.4 mg of lead per cm^2 of surface area (one side).

The instructions for the Lead Alert kit were supplied directly from the manufacturer separate from the kits. In this case, the manufacturer requested that a newer version of the instructions be used for the study since the instruction set was under revision during the planning stages of the full study.

3.4.3.3 Descriptions of Test Kits Included in the Pilot Study

Test kits included in the pilot study were the same as those included in the full study as described in section 3.4.3.1. The protocols used to perform lead measurements with these test kits were the same as those used in the full study with one exception. The exception is for the Lead Alert: Sanding kit, and is discussed below.

The protocol used for the Lead Alert: Sanding kit in the pilot study was the same as that used in the full study except that exposed paint layers were tested as opposed to testing the paint sanding dust as described in section 3.4.3.1.1. The paint layers were exposed for testing using the cutting method common to a number of the other kits described in section 3.4.3.1. The sanding method was attempted in the pilot, but abandoned for the cutting approach after the first location because the sanding technique for total lead measurement was considered too time consuming to be practical for a large number of locations. Feedback from the manufacturer on use of this change after completion of the pilot resulted in modifying the testing plans to incorporate the sanding method. This change required a reduction in the targeted number of sampling locations for this kit as previously discussed in section 3.4.3.1.1

3.4.3.4 Manufacturer Instruction Changes for the Pilot Study

A change to the manufacturers instruction set for the Lead Detective kit was incorporated into the pilot study in a manner similar to that described for the full study. As presented in section 3.4.3.2, the change was related to the application of the sodium sulfide reagent to the testing surface. However, for the pilot, the change was to include a step to use a disposable plastic stirring rod or toothpick to direct the reagent from the reagent bottle to the painted surface, as opposed to the use of a cotton tipped swab used for the full study.

3.4.4 <u>Summary of Field Observations</u>

Recording of unusual events was encouraged throughout the course of the study. Supervisors, on site at all times during field activities, recorded field observations in bound pre-numbered notebooks. Testers used data forms (specific data blocks for time entries and comment columns present on all data recording forms) to record field observations. These

observations were reviewed to aid in data interpretation and to provide supplemental study data. A summary of pertinent observations is presented in this section.

3.4.4.1 Testing Time Requirements

The testing time requirements included time to perform the following large list of activities:

- perform the test;
- fill out the data forms;
- move to the next testing location; and
- periodic breaks.

Typical learning curve characteristics were observed for the time required to perform the test kit measurements.

A review of combined testing times from all three cities suggests that after achieving some familiarity with the kits, approximately 80 measurements could be performed in eight hours using any of the test kits, including the State sodium sulfide kit, with the exception of the sanding method used in the Lead Alert: Sanding kit. Only about 40 measurements could be performed in eight hours using this kit.

3.4.4.2 Testing Difficulties Encountered with Test Kits

3.4.4.2.1 Louisville

As discussed in section 3.4.3.3, the sanding method used to exposed the paint layers for the Lead Alert: Sanding kit was changed to a cutting method after the first location.

Some procedural difficulties were encountered applying the reagents to the testing locations on vertical surfaces as written by the manufacturer using Lead Detective. These procedural difficulties were handled by including an additional step in the testing protocol as discussed in detail in section 3.4.3.4.

3.4.4.2.2 Denver

Some procedural difficulties were encountered using the Lead Alert: Coring kit. The procedural difficulties encountered were

with the indicator solutions. The indicator solution for the kit, prepared by dissolving a chemical tablet in a supplied liquid, was observed by a field supervisor to be unusually dark in color. Normally the indicator solution is a bright orange. The field supervisor observed a dark rose colored indicator solution and stopped the measurement work to investigate the observation. A verification card, which contains lead, was used to check on the measurement response using the dark rose colored indicator solution and found to be working, giving a positive response on the test. However, it was believed that response time appeared to be slightly slower than normal.

As an interim correction, directives were given by the field supervisor to dispose of any kit that was found to have a dark rose colored indicator solution. Under these conditions, a new kit was to be opened and utilized for testing. Because the indicator solutions for both Lead Alert: Coring and Lead Alert: Sanding kits were visually identical, these interim instructions were given to testers performing both of these test kits.

A duplicate measurement, using a new test kit, was performed within the targeted testing area block at the sampling location under evaluation at the time the indicator solution problem was first observed by the field supervisor. The new measurement result was the same as the original measurement, which was negative for lead.

The manufacturer was contacted about the dark colored indicator problem and agreed with the interim corrective action that was used to temporarily resolve the problem. In addition, the manufacturer recommended avoiding excessive shaking of the reagent. This additional recommendation was based on a belief that the problem was caused by a excessive chemical tablet softness for a specific batch of tablets received from the manufacturer's supplier. This excessive softness problem likely resulted in chemical dissolution rates in excess of normal rates, making the indicator solution darken more than normal.

The manufacturer's recommendations were followed until replacement tablets were received from the manufacturer to permanently correct the problem.

The same indicator solution problems encountered for the Lead Alert: Coring kit were also sporadically observed for the

Lead Alert: Sanding kit. These problems were handled using the same interim corrective action discussed above. This resulted in a greater than anticipated consumption rate for the Lead Alert: Sanding kits. As the last few units were being tested, it was recognized that the number of kits available in the field would not be sufficient to complete testing. As an interim solution, while waiting for delivery of additional Lead Alert: Sanding kits, the indicator solutions from the Lead Alert: Coring kits were substituted for use. This substitution was believed to be reasonable since both kits included the sanding technique for total lead measurements. The manufacturer was contacted to verify this substitution. Information received from the manufacturer indicated that there were differences in the formulation of the final indicator solutions between the two kits and that this substitution should not be made. In-progress testing using the substituted indicator solution, which was limited to a single tester in one housing unit, was terminated until new Lead Alert: Sanding kits were received in the field. After receipt of the new Lead Alert: Sanding kits, all sampling locations tested using the substituted indicator solution were re-tested on a clean surface within the original rectangular shaped test area blocks. The original data generated at these sampling locations using the substituted indicator solution were voided in the field and not used for later statistical analysis.

3.4.4.2.3 Philadelphia

Some procedural difficulties were encountered using the LeadCheck kit. The procedural difficulties revolved around misinterpretation as to the procedure for performing verification checks of negative responses at the 30-minute intervals. Two different testers were observed to attempt using an additional swab at the 30-minute interval for verifying the negative result as opposed to visual examination of the coloration of the chemicals remaining on the exposed paint layers from the initial swabbing of the surface. This problem was corrected immediately by the acting field supervisor and was not observed to be a problem for any further testing in Philadelphia.

Some additional difficulties were encountered using Lead Alert: Sanding kit. The difficulties were similar to those experienced in Denver and revolved around problems with substitution of the indicator solution from the Lead Alert: Coring kit. In this situation, a tester had consumed all the

reagents from the Lead Alert: Sanding kit and replaced it with those from the Lead Alert: Coring kit because it looked the same. The problem was identified by the acting field supervisor after six locations had been affected. The situation was corrected and re-testing was performed at the six affected locations. The re-testing was performed on a clean surface within the original rectangular shaped test area blocks targeted for the test kit. The original data generated at these sampling locations using the substituted indicator solution were voided in the field and not used for later statistical analysis.

3.5 XRF TESTING

A discussion of the design elements related to investigation of XRF instrument performance is presented in this section.

3.5.1 XRF Instrument Selection

All field portable XRF instruments that were commercially available for lead-based paint (LBP) testing in the Spring of 1993 were considered candidates for inclusion in the study. Field portable XRF instruments for lead-based paint (LBP) are generally classified into two categories based on the type of X-ray emission lines that are used for the determination of lead. These include K-shell and L-shell emission lines that correspond to the X-ray fluorescence transitions from electron orbitals of the lead atom. K-shell X-ray emission lines are more energetic than the L-shell lines and therefore, are generally expected to have better penetrating power through multiple paint layers than the L-shell emissions. However, in general, background emission spectra near the K-shell lines are more complex than those near the L-shell lines. This makes precise measurement of the K-shell emissions more difficult than the L-shell emissions. Commercially available field portable XRF instruments include those that use the K-shell emissions, those that use L-shell emissions, and those that are capable of using both types of emissions for measurement of lead.

Three XRF instruments were in wide use for LBP testing in housing at the start of this study. These are the MAP-3 spectrum analyzer manufactured by Scitec Corporation, the Microlead I revision 4 manufactured by Warrington, Inc., and the XK-3 manufactured by Princeton Gamma-Tech, Inc.. The MAP-3 uses both K-shell and L-shell X-rays while the other two use only K-shell

X-rays for lead measurements. Because of their wide use in LBP testing these instruments were used in the pilot study and the field study; one of each in the pilot study and two of each in the field study.

Another instrument, the X-MET 880, manufactured by Outokumpu Electronics, was reported to be sporadically used for LBP testing. Unlike the previous three instruments, the X-MET 880 operates with L-shell X-rays only. For both studies, one X-MET 880 was included. A summary of the XRF instruments included in the pilot study is presented in Table 3-10.

In order to achieve the goal of updating the federal guidelines for testing lead in paint, it was desirable to include any prototype XRF instrument that was likely to be available for LBP testing in the near future. Following completion of the pilot study, XRF manufacturers that were reported to have such prototypes were contacted and asked to participate. Because of the large amount of resources involved with the addition of each XRF instrument to the study, a ruggedness test was used to select XRF prototype instruments. This ruggedness test was designed to provide evidence that a given XRF prototype would be able to operate under the field conditions anticipated during performance of the study. No assessment of testing performance in terms of accuracy was included in this ruggedness test. The ruggedness test consisted of a series of measurements, similar to those planned for the full study. Testing locations were placed in a variety of areas to simulate the environment likely to be encountered during performance of the full study. Locations included indoor and outdoor testing areas: cold and hot temperatures, narrow and wide testing areas, and low and high testing areas. Any XRF instrument that was able to continue to operate and generate lead results during an entire day of testing was included in the full study. Two additional instruments qualified and therefore, were included in the full study. These were the Lead Analyzer, a K- and L-shell instrument manufactured by TN Technologies, Inc., and the XL, an L-shell instrument manufactured by Niton Corporation. A summary of the XRF instruments included in the two cities tested during the full study is presented in Tables 3-11 and 3-12. It is worth noting that the XL has undergone significant modifications by the manufacturer since its inclusion in the study.

Table 3-10. Summary of XRF Instruments Used in the Pilot: Louisville.

XRF Model	Source Type ([a])	Detector Type	XRF Code No.[b]	Date of Source (month/year)	Testing Dates in 1993 (month/day)
MAP-3	Co^{57} (40)	Ambient Silicon	13	1/93	03/31-04/01
Microlead I revision 4	Co^{57} (10)	Cesium Iodide	24	3/93	03/31-04/01
XK-3	Co^{57} (10)	Proportional Counter (Xenon gas)	31	3/93	03/29-30
X-MET 880	Cd^{109} (5)[c]	Proportional Counter (Argon gas)	51	8/92	03/29-30

[a] Parenthetical numbers are approximate source strengths for new sources in millicurie.
[b] Code Nos., created for this report, are used to discuss lead testing results for specific instruments.
[c] A number of radiation sources are sold for this instrument. Only this source was present in Louisville.

3.5.2 XRF Measurement Design Elements

Anecdotal evidence from previous laboratory and field studies suggested that accuracy of lead measurements using XRF instruments can vary due to a wide variety of factors including: short-term and long-term drift, differences in substrates, potential memory effects when switching between substrates, and differences in surface exposure time. Each of these factors was considered and the resulting design included a large number of specific design elements that were used to direct XRF comparison testing to achieve the study objectives. Specific XRF testing design elements included in this study are listed below and are discussed in detail in the following sections:

- Use of Independent Contractors and Monitors
- Adherence to Manufacturer Protocols
- Specified Testing Order
- XRF Variability QC Checks
- XRF Measurement Definitions
- XRF Measurements at Standard Locations
- XRF Measurements at "Special" Locations
- Bare Substrate Measurements (with and without NIST films)

- Field QC Samples for XRF Measurements
- Recording of K- and L-shell XRF Data
- Safety Considerations for XRF Testing

Table 3-11. Summary of XRF Instruments Used in the Full Study: Denver.

XRF Model	Source Type ([a])	Detector Type	XRF Code No.[b]	Date of Source (month/year)	Testing Dates in 1993 (month/day)
Lead Analyzer	Cd^{109} (30)	Cooled Mercuric Iodide	1	4/93	08/04-14
MAP-3	Co^{57} (40)	Ambient Silicon	10 11	7/93 7/93	08/04-21 08/04-21
Microlead I revision 4	Co^{57} (10)	Cesium Iodide	20 21 22	8/93 5/93 8/93	08/07-18 08/9-10,16,18-19 8/11-14,17
XK-3	Co^{57} (10)	Proportional Counter (Xenon gas)	30 31	3/93 3/93	08/05-16 08/06-17
XL	Cd^{109} (10)	Silicon	40 41	7/93 7/93	08/10-14 08/16-20
X-MET 880	Cm^{244} (100)[c]	Proportional Counter (Argon gas)	50	9/91	08/04-14

[a] Parenthetical numbers are approximate source strengths for new sources in millicurie.
[b] Code Nos., created for this report, are used to discuss lead testing results for specific instruments.
[c] Two sources were present in this instrument. Only this source was used for lead measurements in Denver.

3.5.2.1 Use of Independent Contractors and Monitors

XRF testing was performed by lead testing companies, independent from the manufacturers that owned XRF instruments or leased/rented XRF instruments from the manufacturers. The testing companies used for this study were selected from lists of testing contractors supplied by the XRF manufacturers with one exception. For the prototype XRF instrument, cooperative efforts were exerted to use an independent testing company that the manufacturer would be willing to train and supply with prototype instruments for use in the study. During the field testing activities, the testing company personnel were requested not to contact the manufacturers unless approved by the field supervisor.

As an aid to reduce testing errors and deviations from the testing protocol, a data recording monitor, independent of the XRF testing contractor, was assigned to each XRF tester. This

recording monitor was trained on-site in the field to perform the work according to study protocols. The use of the independent monitors helped assure that all data taken by the XRF tester

Table 3-12. Summary of XRF Instruments Used in the Full Study: Philadelphia

XRF Model	Source Type ([a])	Detector Type	XRF Code No.[b]	Date of Source (month/year)	Testing Dates in 1993 (month/day)
Lead Analyzer	Cd^{109} (30)	Cooled Mercuric Iodide	1 2	4/93 4/93	10/11-21 10/21-25
MAP-3	Co^{57} (40)	Ambient Silicon	10 12	7/93 7/93	10/06-25 10/06-25
Microlead I revision 4	Co^{57} (10)	Cesium Iodide	20 23 21	8/93 9/93 10/93	10/11-25 10/11-14,18-25 10/15
XK-3	Co^{57} (10)	Proportional Counter (Xenon gas)	30 32	3/93 4/93[d]	10/11-25 10/11-25
XL	Cd^{109} (10)	Silicon	42	7/93	10/11-25
X-MET 880	Cm^{244} (100)[c]	Proportional Counter (Argon gas)	50	9/91	10/11-25

[a] Parenthetical numbers are approximate source strengths for new sources in millicurie.
[b] Code Nos., created for this report, are used to discuss lead testing results for specific instruments.
[c] Two sources were present in this instrument. Only this source was used for lead measurements in Philadelphia.
[d] Interpreted from 6 month old recorded source age.

would be recorded and helped increase the speed of the field testing task.

3.5.2.2 Adherence to Manufacturer Protocols

Although the study design included highly structured testing procedures as described in sections 3.5.2.3 through 3.5.3.11, XRF testers were required to operate XRF instruments in accordance with generally recommended manufacturer protocols (with one exception discussed below). This requirement was included to generate data in a manner that was consistent with general LBP testing in housing. Examples of generally recommended manufacturer protocols include the use of instrument warm-up periods and the use of beginning-of-day test block checks or adjustments.

The one exception to using generally recommended manufacturer protocols was for the Microlead I. The manufacturer recommends discarding one reading when changing substrates. The discarding of one reading when changing substrates was not followed for the three reasons listed below:

- During the pilot study no unusual readings were observed when substrates were changed during performance of the XRF variability QC checks described later in section 3.5.2.4.
- It was desirable to discourage the discarding of any readings by all the operators in order to provide data that would permit investigating the occurrence and causes of large XRF measurement errors that fall well outside the normal range; and
- Similar substrates were grouped and tested together to control substrate transition points as discussed in section 3.5.2.3. In addition, continuing control block readings were taken at these transition points to provide information on potential substrate change affects as discussed in section 3.5.2.9.2.

Even though the Microlead I testers were not permitted to discard any readings, all XRF instrument testers, included those operating the Microlead I, were allowed to take additional readings if desired or needed to meet manufacturer's directives as long as all the readings were recorded by the monitors. Virtually all of the XRF testers made and recorded additional readings at various times throughout the testing. However, any additional readings recorded by the monitors were not used for statistical analysis unless it was clear that the XRF tester made an error making the original readings.

3.5.2.3 Specified Testing Order

XRF testing was performed in a specific testing order with respect to substrate type. All like substrates in a unit were tested together before moving to the next substrate. This design element was performed to systematically control changes in substrate type. Continuing control block readings, discussed in section 3.5.2.9.2, were included to monitor potential effects from changing substrates during transition from one substrate to another.

For the pilot study, XRF testing was performed in a total of four units grouped under two dwelling numbers consisting of two housing units each. Individual housing units did not have sufficient numbers of potential sampling locations to provide enough locations near the 1.0 mg/cm^2 lead level based on data supplied by the housing authority. Sampling locations with like substrates were sequentially numbered together within each dwelling number. The order of testing was wood, drywall, plaster, concrete, and metal. Brick was not included because this painted substrate was not present at the Louisville site. XRF testing was performed starting from the lowest sample location number and progressing in an increasing fashion.

For the full study, sampling locations with like substrates were sequentially numbered together within each of the housing units. Targeted substrates were grouped into two categories based on relative density with respect to XRF testing: high density materials that included metal, brick, and concrete, and low density materials that included wood, drywall and plaster. The testing order was changed from the pilot to the full study to alternate between these high density and low density materials. The order of testing for the full study was metal, wood, brick, drywall, concrete, and plaster. This change from the pilot was made to investigate potential memory effects on XRF lead responses when switching between substrates of different densities. It was believed that alternating between the high and low density materials would provide the best opportunity for observing any potential memory affects.

XRF testing in the full study was performed in a manner similar to the pilot study, that is, progressing through the sample location numbers in an increasing fashion. However, the starting substrate for each house or unit was systematically varied among the substrate types. This design element was added to the full study to avoid any potential bias that could occur from testing the like substrates in each housing unit at the same time period of the testing day. Once the starting substrate was selected, testing of other substrates was determined by the testing order list. After the last substrate on the list was completed, testing was resumed on the first substrate on the list, that is, metal.

In Denver, sample location numbering for a given housing unit was performed according to the targeted testing order: metal, wood, brick, drywall, concrete, and plaster. Because of the desire to initiate testing on different substrate types in different housing units, testing was performed in compliance to the targeted testing order by starting with the lowest sampling location number corresponding to the starting substrate type. Testing was performed from that starting point through the sample location numbers in an increasing fashion. When the highest sampling location number had been tested, testing was resumed starting from the lowest sample location number on the next substrate. In Philadelphia, the procedure was simplified by initiating the numbering at the targeted starting substrate type for that unit as opposed to numbering the all units in an identical manner starting with the same substrate type.

3.5.2.4 XRF Variability QC Checks

In the pilot study, additional XRF measurement replicates were taken at each sampling location that was of a different substrate type from the previous sampling location. These additional XRF measurements, called "variability QC checks", were performed to provide information on potential memory effects when changing substrates. This design element was not included in the full study after data obtained in the pilot suggested that the potential memory effects were not observable, if present, using this type of QC check.

3.5.2.5 XRF Measurement Definitions

An XRF measurement for this study was defined as a set of replicate XRF readings over a fixed duration of time called a reading time. Reading time and numbers of replicates were variables for consideration in the design of this study. A single reading time was defined as single open shutter event that included exposure of the painted surface to energy in the form of gamma rays or high energy X-rays, emission of X-rays from fluorescence transitions within lead atoms residing in the painted surface, counting of the X-rays received at the detector, electronic processing of the detector signals, and displaying a lead-area value result in mg/cm^2.

Reading times discussed in this report are referred to as nominal reading times. The word nominal is used to designate the

reading time that would result if the XRF instrument were using a new radiation source. For the full study, the nominal reading time at a standard sampling location, discussed later in this section, was defined as a 15-second reading. The actual reading time for a given XRF instrument was generally slightly longer than this nominal 15-second time because radiation source materials used in XRF instruments decay. As the radiation source decays, the reading time must be lengthened to produce a constant radiation exposure of the painted testing surface. Because of radiation source decay and because of the desire to perform testing using a relatively constant radiation energy exposure of the testing surfaces among different instruments, efforts were made to include XRF instruments into the study that had relatively new radiation sources. Adjustment to the reading time beyond the nominal time was done based on the decay rate curve for the radiation source material. Some instruments, including the MAP-3, Microlead I, and the XK-3, performed this adjustment automatically. Other instruments, such as the Lead Analyzer and the X-MET 880, required manual adjustment of the reading time for this study. The XL was factory set to a 15-second reading time for the entire study with no adjustment based on source date. For the Lead Analyzer, manual adjustments were made with the aid of an adjustment table based on the radiation source age for Cd^{109} as shown in Table 3-13. Although no Co^{57} manual adjustments were required, adjustment figures are also shown for this source material as an aid to understanding the relationship between the actual reading times and the nominal reading times used in this study for Co^{57} sources. The Cm^{244} source used in the X-MET 880 for the full study has a half-life of approximately 18 years. This long half life, more than ten times that of Cd^{109}, results in a much slower decay rate than the other radiation sources used in this study. This reduces the magnitude and frequency of reading time adjustments needed to maintain a constant energy exposure of the testing surfaces. Based on the Cm^{244} decay rate and a source date of September 1991, a one second increase to the nominal 15-second reading time was used for this instrument for a total of 16 seconds.

Reading time for XRF instruments are sometimes discussed using three separate measurement time period terms: clock time, live time, and dead time. These measurement terms need some discussion to explain setting of reading times among the different XRF instruments included in this study. With the exception of clock time, the definitions for these measurement

terms vary among different XRF manufacturers. This definition variability is related to differences in electronic signal processing among different XRF instruments. However, despite these differences, the following approximate definitions represent the ideas needed in this report to discuss the reading times set for each XRF instrument.

Clock time Clock time, sometimes referred to as real time, is the total time that elapses while making a single lead measurement. Clock time is equal to live time plus dead time.

Live time Live time, also referred to as counting time, is the time the detector is actively accumulating X-rays for producing a lead result.

Table 3-13. Adjustment of Nominal 15 Second XRF Reading Times for Age of Source.

Source Age (Days)	Reading Times (Seconds)[a] For Cd^{109}	Reading Times (Seconds)[b] For Co^{57}	Source Age (Days)	Reading Times (Seconds)[a] For CD^{109}	Reading Times (Seconds)[b] For Co^{57}
0	15.0	15.0	210	20.6	25.6
10	15.2	15.4	220	20.9	26.3
20	15.5	15.8	230	21.2	27.0
30	15.7	16.2	240	21.5	27.7
40	15.9	16.6	250	21.8	28.4
50	16.2	17.0	260	22.2	29.1
60	16.4	17.5	270	22.5	29.9
70	16.7	17.9	280	22.8	30.6
80	16.9	18.4	290	23.2	31.4
90	17.2	18.9	300	23.5	32.2
100	17.4	19.4	310	23.9	33.1
110	17.7	19.9	320	24.2	33.9
120	18.0	20.4	330	24.6	34.8
130	18.2	20.9	340	25.0	35.7
140	18.5	21.4	350	25.4	36.6
150	18.8	22.0	360	25.7	37.6
160	19.1	22.6	370	26.1	38.5
170	19.4	23.2	380	26.5	39.5
180	19.7	23.7	390	26.9	40.6
190	19.9	24.4	400	27.3	41.6
200	20.2	25.0	410	27.7	42.7

[a] The half life of Cd^{109} is 462.0 days [4].
[b] The half life of Co^{57} is 271.8 days [4].

Dead time Dead time is the time the detector is **not** actively accumulating X-rays for producing a lead result.

Some XRF instruments, such as the Microlead I and XK-3, have no appreciable dead time. Therefore, for these instruments,

clock time is equal to live time, and the reading time used in this study is equivalent to a live time measurement. For instruments that are reported to have some dead time, such as the Lead Analyzer, the MAP-3, the XL, and the X-MET 880, the amount of dead time is sometimes variable depending on the substrate and lead level being tested. For the MAP-3, the XL, and the X-MET 880, reading times were set according to live time. For the Lead Analyzer, reading times were set according to clock time. Therefore, the live time for Lead Analyzer, as used in this study, was slightly less than the live times used for MAP-3, the XL, and the X-MET 880.

The MAP-3 had three reading modes of operation: the "screen" mode that used a nominal 15-second reading, a "test" mode that used a nominal 60-second reading, and "confirm" mode that used a nominal 240-second reading. Other instruments generally relied on the use of one reading mode. Investigation of the three reading modes for this instrument was included by using three different types of sampling locations: "standard" sampling locations, "special" sampling locations, and "special-special" sampling locations. The "special-special" locations were used only in the full study and not in the pilot. The difference between these sampling locations was that the "special" and "special-special" sampling locations received some extra XRF testing in addition to the standard testing using different measurement definitions, as presented in Table 3-14 and Table 3-15. In the pilot, all instruments under investigation were included in this extra testing. For the full study, extra testing was performed only by the MAP-3 instruments on separate testing days referred to as "special" testing days. Further details on testing at standard and "special" sampling locations are discussed later in sections 3.5.2.6 and 3.5.2.7.

Use of these different types of sampling locations permitted an investigation into the alternative modes of operation in a manner that conserved study resources. Conservation of study resources was accomplished by limiting the numbers of "special" and "special-special" locations in the study. "Special" sampling locations were designated at a rate of approximately 25% of the total for the pilot and full studies. For the full study, approximately 25% of the "special" sampling locations were designated as "special-special" sampling locations.

3.5.2.6 XRF Measurements at Standard Locations

Two goals were desired with respect to operation of the XRF instruments for this study. One was to use the XRF instruments in accordance with manufacturer recommendations as discussed in section 3.5.2.2. The other goal was to make reading times approximately the same. This goal was aimed at reducing the

Table 3-14. Pilot Study: XRF Measurement Definitions.

XRF MODEL	No. of Replicates - Nominal Reading time (sec.)			
	Standard Locations[a]	**"Special" Locations[a]**	**Beginning and Ending Control Block Readings[a]**	**Continuing Control Blocks Readings[a]**
MAP-3	1 - 60	3 - 15	3 - 60	1 - 60
Microlead I revision 4	3 - 15[b]	4 - 15[b]	9 - 15[c]	3 - 15[b]
XK-3	3 - 15	4 - 15	9 - 15	3 - 15
X-MET 880	3 - 15	1 - 60	9 - 15	3 - 15

[a] If the substrate was concrete, an extra measurement was performed.
[b] Data was collected using a single trigger pull event; the trigger was held down through consecutive clicks, three or four, with each replicate reading recorded during the measurement cycle.
[c] Data collected as three groups of three replicate readings. Each group of three replicate readings was collected using a single trigger pull event; the trigger was held down through three consecutive clicks with each replicate reading recorded during the measurement cycle.

Table 3-15. Full Study: XRF Measurement Definitions.

XRF MODEL	No. of Replicates - Nominal Reading time (sec.)			
	Standard Locations	**"Special" Locations**	**Beginning and Ending Control Block Readings**	**Continuing Control Block Readings**
Lead Analyzer	3 - 15	na	3 - 15	3 - 15
MAP-3	3 - 15	1 - 60[a] 1 - 240[b]	3 - 15	3 - 15
Microlead I revision 4	3 - 15[c]	na	3 - 15[c]	3 - 15[c]
XK-3	3 - 15	na	3 - 15	3 - 15
XL	3 - 15	na	3 - 15	3 - 15
X-MET 880	3 - 15	na	3 - 15	3 - 15

na	not applicable, no measurement definition
[a]	Measurement definition at "Special" locations
[b]	Measurement definition at "Special-special" locations
[c]	Collection of the three replicate readings was performed using a single trigger pull event; the trigger was held down through three consecutive clicks with each replicate reading recorded during the measurement cycle.

potential for unfair advantage to XRF instruments operating under extended reading times since measurement precision might be improved by acquiring X-ray counts for longer time periods. Differences among the XRF instruments, as presented through the previous discussion of clock time, live time and dead time, limited the ability to make measurements using exactly the same exposure times. However, it is believed that any potential differences in counting statistics among the instruments were minimized during the measurements at standard sampling locations.

For the pilot, the design elements for XRF testing, including the XRF measurement definitions, were influenced by information from the unpublished November 30, 1992 version of the paint testing chapter of the HUD Guidelines [5]. Standard sampling locations were tested using reading times that were considered, with respect to these draft guidelines, to be the most common for LBP testing by each specific instrument. The Microlead I and the XK-3 were set to take readings the way it is commonly done for these instruments; three nominal 15-second readings. Collection of the three replicate readings using the Microlead I was performed using a single trigger pull event, that is, the trigger was held down through three consecutive clicks with each replicate reading recorded during the measurement cycle. This mode of taking multiple replicate measurements at a specific sampling location, which is unique to the Microlead I, was used throughout the pilot and full studies. The X-MET 880, not commonly used for LBP testing, was arbitrary set to take readings using the same measurement definition used for the Microlead I and the XK-3 as shown in Table 3-14. The MAP-3 was operated in the "test mode" using a single nominal 60-second reading time.

For the full study, at all standard sampling locations, a reading was defined as a nominal 15-second exposure time for all XRF instruments including the MAP-3 as shown in Table 3-15. This

was done to achieve the second goal with respect to operation of the XRF instruments; that is, to perform reading times of approximately the same length as previously discussed.

3.5.2.7 XRF Measurements at "Special" Locations - Use of Alternative Measurement Times

As discussed in section 3.5.2.5, "special" sampling locations, which included both "special" and "special-special" sampling locations for the full study, were used to perform additional testing using alternative measurement definitions.

For the full study, performance of additional testing at the "special" sampling locations was limited to the MAP-3 as shown in Table 3-15. The MAP-3 was investigated using two alternative measurement times: the test mode using a nominal 60-second reading and the confirm mode using a nominal 240-second reading. All "special" sampling locations were tested using the nominal 60-second reading. The "special-special" sampling locations were further tested using the nominal 240-second reading. A summary of measurement definitions used for XRF testing across the different types of sampling locations in the pilot and full studies are shown in Tables 3-14 and 3-15. Discussions of measurements on control blocks are presented in section 3.5.2.9.

For the pilot study, different measurement definitions for "special" sampling locations were used for all four instruments to provide comparison testing information among instruments at approximately equivalent nominal reading times as shown in Table 3-14. For example, measurement for the X-MET 880 at the "special" sampling locations was performed using one nominal 60-second reading, which is equivalent to MAP-3 data generated at the standard sampling locations. Since the Microlead I and the XK-3 had fixed reading times, use of an additional replicate provided a combined set of readings, four nominal 15-second readings, that was equivalent in duration to the measurement time for the MAP-3 at the standard sampling locations. Use of nominal 15-second readings for the MAP-3 at "special" locations provided data equivalent in duration to the measurement times for the other three XRF instruments at the standard sampling locations.

3.5.2.8 Bare Substrate Measurements (with and without NIST films)

Each sampling location included painted testing areas and bare XRF testing areas (areas scraped free of surface paint following removal of paint chip samples) for making XRF measurements as described in section 3.1.5 and diagrammed in Figure 3-1. Measurements at the bare substrate testing areas were included in the study to provide information on the potential of substrate correction. Substrate correction, a type of background correction method, refers to a method of improving the accuracy of lead testing results by subtracting lead testing results obtained from the bare substrates from lead results on painted surfaces.

Statistical analysis of historical XRF data from previous studies was hampered by the fact that some of the XRF instruments truncate lead measurement values at zero, that is, no negative values were allowed to be displayed. However, negative values can be generated by most XRF instruments, and negative values can be useful for statistical analysis. Therefore, to avoid censoring of negative readings, a NIST standard film, containing lead, was routinely placed over the bare substrate to elevate the measured lead result by a known value. The NIST standard films, NIST SRM 2579, are a set of five films containing known levels of lead ranging from less than 0.0001 mg/cm^2 to 3.53 mg/cm^2. These films, approximately 12 to 13 mil thick, have been reported to consist of approximately 2 mil of paint on top of 8 mil of mylar backing plus a plastic coating [6]. (A mil is a common unit of measure in the coatings industry and is equal to 1/1000 of an inch.) This design element of using the films to elevate the measured lead result was used both for bare substrate areas at each sampling location and for control block measurements described in section 3.5.2.9.

For the pilot, bare substrate areas at each location were measured while covered with the 1.02 mg/cm^2 NIST standard film. If the substrate was concrete, an additional measurement was made using the 3.53 mg/cm^2 NIST standard film. Concrete substrates were targeted for the additional measurement using a higher lead standard film because an XRF expert suggested that on dense substrates, the higher lead levels constitute a greater challenge than lower levels.

For the full study, bare substrate areas at each location were measured while covered with the 1.02 mg/cm^2 NIST standard film. Use of the higher standard for concrete was dropped based

on lack of evidence from pilot data that it was needed. However, measurement of the bare substrates without any films was added to the design to provide supplemental XRF measurement data as suggested by some reviewers during the planning stages for the full study. Bare substrate measurements were performed by all instruments at the locations identified as "specials," which was approximately 25% of the locations. Bare substrate measurements made by the MAP-3 instruments were performed using the test mode, a nominal reading time of 60-seconds, only during days when the "special" sampling locations were being tested using alternative measurement definitions as described in section 3.5.2.7.

3.5.2.9 Field QC Samples for XRF Measurements

As previously discussed in section 3.5.2, design elements were needed to investigate the anecdotal evidence that XRF instruments were subject to both systematic and intermittent sources of error. Field QC samples were developed to aid in this investigation and to provide information on the potential use of standard field QC procedures for performing substrate correction. The field QC samples developed for this study consisted of control blocks constructed from various building materials representing the commonly encountered substrates. These QC samples were measured while covered with NIST standard films to provide lead measurement results on the known lead level standard films. In the full study, these QC samples were also measured bare, not covered with the NIST standard films.

For the pilot, a total of six different types of control blocks were constructed. These control blocks were each approximately 4 inches by 4 inches by y inches where y equalled approximately ¾ inches for wood (pine), 2 inches for concrete (with aggregate), ½ inches drywall, 1 inch for plaster, and y was 20- to 25-gauge for metal. A brick was also included as a control block. In addition to these blocks, a 12 inch thick styrofoam support block was included to support the control blocks during XRF measurement activities. The styrofoam block was used to eliminate the potential for underlying materials to affect the XRF measurement values taken using the control blocks. A total of two complete sets of blocks were constructed.

For the full study, ten complete sets of the same types of control blocks included in the pilot were constructed.

For each XRF testing day, all control block measurements made by a specific XRF operator were always performed on the same set of control blocks at a fixed location within the housing unit. These restrictions were made to assure that differences observed in control block data would be free of potential effects caused by physical differences in individual control blocks and placements within the units. One set of control blocks was used for each of two units tested together in the pilot study. For the full study, a separate set of control blocks was placed in each housing unit.

3.5.2.9.1 Beginning and Ending Control Block Testing

As previously discussed in section 3.5.2, information was needed to investigate a tendency for some instruments to "drift" over the course of a day's measurements. Information on day-long drift was obtained by requiring all XRF operators to take measurements on all types of the control blocks at the beginning and at the end of each testing day.

For the pilot, beginning and ending control block testing were performed using the same reading times as those used for the standard sampling location measurements, but at a three fold increase in replications as shown in Table 3-14. This large number of replications was included to assure collection of sufficient data to assess short term stability during the collection of the control block data. All control blocks were measured while covered with the 1.02 mg/cm^2 NIST standard film. If the substrate was concrete, an additional measurement was made using the 3.53 mg/cm^2 NIST standard film. Use of this additional higher lead level film is discussed in section 3.5.2.8.

For the full study, beginning and ending testing were performed using the same reading times as those used for the standard sampling location measurements as shown in Table 3-15. The increase in replication of reading times used in the pilot was dropped due to lack of need based on the pilot data. Instead, three different measurements were made on all the control blocks: one while covered with a 3.53 mg/cm^2 NIST standard film, one while covered with a 1.02 mg/cm^2 NIST standard film, and one on the bare substrate. This change was made to gather potential drift data over a range of lead levels as opposed to one lead level used in the pilot with the exception of the concrete substrate.

3.5.2.9.2 Substrate Change Control Block Testing

As discussed in section 3.5.2, information was needed to investigate a tendency for some instruments to show unusually high variability of measurements taken immediately after a change in substrate. Information on potential short term "drift" during the day was also needed. Information on these two issues was obtained by requiring all XRF operators to make measurements on specific control blocks every time a substrate change was made during testing of the sampling locations. These measurements are referred to as "continuing control block readings".

For the pilot, continuing control block readings were performed using the same reading times as those used for the standard sampling location measurements as shown in Table 3-14. If the new sampling location was of a different substrate type from the last sampling location, a control block measurement was required on the new substrate type. The control block matching the new sampling location was measured while covered with the 1.02 mg/cm^2 NIST standard film. If the substrate was concrete, an additional measurement was made on these areas using the 3.53 mg/cm^2 NIST standard film. Use of this additional higher lead level film is discussed in section 3.5.2.8. During the performance of the pilot, an addition was made to include continuing control block readings on substrates matching the last sampling location. Therefore, most of the continuing control block readings taken during the pilot included readings on substrates matching the last and new sampling locations. This feature was added as an up-front design element for the full study.

For the full study, continuing control block readings were performed using the same reading times as those used for the standard sampling location measurements as shown in Table 3-15. If the new sampling location was a change in substrate type from the last sampling location, continuing control block readings were required on both substrates types, last and new. Three different measurements were made on each of the two matching control blocks: one while covered with a 3.53 mg/cm^2 NIST standard film, one while covered with a 1.02 mg/cm^2 NIST standard film, and one on the bare substrate. This change was made to match beginning and ending control block testing previously discussed in section 3.5.2.9.1.

Because of concerns related to increases in testing time for the MAP-3 instruments during the "special" measurement days discussed in section 3.5.2.7, continuing control block readings for the MAP-3 instruments were not performed during "special" measurement days.

3.5.2.10 Recording of K- and L-shell Data

Two of the XRF instruments included in the study had the capability to simultaneously make both K- and L-shell measurements. These were the MAP-3 and the Lead Analyzer. For these instruments, both forms of data were collected during the study.

3.5.2.11 Safety Considerations

XRF instruments use radioactive isotopes that continuously emit energy in the form of high energy X-rays, or gamma rays or other decay particles. Because these energy emissions can cause damage to body tissue, radiation safety was a primary consideration during XRF testing.

Several design elements were included to address safety concerns and manage the potential hazard during the study. These included use of (1) properly trained and licensed operators, (2) additional hazard instructional training for both operators and monitors, (3) limiting the number of testers in a given unit, (4) elimination of some potential testing areas from use during selection of sampling locations, (5) use of specific unit entry protocols, (6) use of warning signs and door guards, (7) monitoring by the field supervisors, and (8) use of radiation badges. Each of these is discussed further below:

(1) Use of Licensed Operators. Only licensed operators were used for handling the XRF instruments. These operators were training by the XRF manufacturers for use of the instruments under their control with one exception. The exception was for one of the Microlead I operators who was trained by a manufacturer to operate the MAP-3 but not specially trained by a manufacturer to operate the Microlead I. However, this operator did receive training for the Microlead I by a senior testing company staff member who had received training directly from the manufacturer to operate the Microlead I.

(2) Additional Hazard Instructional Training. Prior to XRF testing, all operators and monitors were assembled together and given detailed instructions as to the requirements for performance of the study. During this training, safety concerns and the potential hazards were discussed with the goal of increasing awareness to eliminate any inadvertent radiation exposure.

(3) Limiting the Number of Testers in a Given Unit. Only one XRF operator with monitor was allowed in a given unit at a time. This eliminated the potential for a second operator to inadvertently expose the first during performance of testing activities. In addition, any potential electronic interferences from one XRF instrument on another were also eliminated. Field supervisors were allowed to enter units to observe testing and perform other supervisory activities.

(4) Elimination of Potential Testing Areas from Use. During the process of identifying sampling locations, discussed in section 3.1.4, some areas were excluded from consideration because of the potential inadvertent radiation exposure hazard. Examples include the back of an main entry door that would have to be exposed to radiation in a manner that would be not permit the operator or monitor to observe traffic coming up to the door, walls that were facing stairways common to multiple housing units, and walls that were facing occupied areas.

(5) Use of Specific Entry Protocols. Entry into a building undergoing XRF testing was performed using an established verbal call and wait for reply procedure. In addition to instructing all field personnel in the procedure, the procedure was posted on the doors of housing units that were not assigned guards as described below.

(6) Use of Warning Signs and Door Guards. Brightly colored warning signs were posted at the entry to each housing unit in Louisville and Denver. In Philadelphia, the testing site was such that the signs would only result in attraction of people and therefore, would have failed to be an effective deterrent to entry. Therefore, guard personnel were used to stop outside people from entering the buildings undergoing XRF testing.

(7) Monitoring by the Field Supervisors. All XRF testing was actively monitored by field supervisors. Part of the field supervisors duty was to assure that testing was conducted in a safe manner.

(8) Use of Radiation Badges. All field personnel involved with XRF testing were assigned radiation badges for monitoring levels of any inadvertent exposure. Badges were worn by XRF operators, monitors, field supervisors, and any outside observers visiting the XRF testing site.

3.5.3 Summary of Field Observations

Recording of unusual events was encouraged throughout the course of the study. Supervisors, on site at all times during field activities, recorded field observations in bound pre-numbered notebooks. Testers used data forms to record field observations. All data forms used in the study contained data blocks for recording time entries and comments or observations. This information was reviewed to aid in data interpretation and to provide supplemental study data. A summary of observations deemed by the field supervisory team to be of importance for supplemental XRF testing information is presented in this section.

3.5.3.1 Testing Time Requirements

The primary goal of the pilot study was to determine operating logistics for use in planning resources for the full study. Time requirement estimates used for planning the pilot study were determined by making assumptions that were based on reported XRF operations. These estimates were increased slightly to assure that the pilot study testing could be completed within planned times. Actual performance times in the field for both the pilot study and full study were recorded in time measurement entry blocks on the data forms.

The testing time requirements included the time to perform the following list of activities:

- warm up the instrument;
- perform field QC sample measurements;
- perform sampling location measurements;
- fill out the data forms;

- move to the next testing location; and
- periodic breaks.

Typical learning curve characteristics were observed for the time required to perform the XRF testing. Inclusion of the highly specified measurement procedures designed into the study is suspected to be a contributing factor to the increased testing times required during the initial days of XRF testing. The XRF operators had been trained to run their instruments but not in a manner called for by the detailed measurement protocols used in the study, particularly with respect to performance of the field QC sample measurements.

A review of testing times from Denver suggests that after achieving some familiarity with the testing protocols, approximately 60 to 100 of the standard sampling locations, including field QC samples, could be tested in eight hours with any of the XRF instruments in the study.

3.5.3.2 Testing Difficulties Encountered in the Performance of XRF Measurements

Some difficulties were encountered during performance of the XRF testing portion of the study. These difficulties are summarized in the following sections.

3.5.3.2.1 Factory Modifications between Denver and Philadelphia

Testing in a manner completely independent from manufacturer influence was not possible during the study. For example, prototype instruments had to be obtained through direct cooperation with the manufacturer. Some modifications of instruments were performed between Denver and Philadelphia and are discussed in this section.

The XL used for Philadelphia was different from the XL instruments used in Denver, as indicated in Tables 3-11 and 3-12. The manufacturer verbally informed the study team that modification of the XL was performed between use in the two cities. However, the exact nature of the modification is not known.

The Lead Analyzer used for much of the Philadelphia Testing

was modified after the Denver testing and before the Philadelphia testing. This information was provided to the study team by the manufacturer in writing. The nature of the modification was limited to a re-calibration of the instrument using a different set of standards from those used prior to shipment of the instrument to Denver.

The same two MAP-3 instruments were used in both Denver and Philadelphia, as indicated in Tables 3-11 and 3-12, even though different independent subcontractors were used for one of these two instruments. It was observed that these instruments were capable of displaying negative lead readings in both cities. However, in Denver, the instruments could not store negative readings, whereas in Philadelphia, the instruments could store negative readings.

3.5.3.2.2 Instrument Operational Problems and Failures

Some XRF instrument operational problems occurred during testing for the pilot and full studies. All of the operational problems can be summarized in three categories: battery problems, instrument failures, and data storage problems. Each of these is discussed below.

Battery related problems, observed in the pilot and full study, were the most common of the XRF instrument operational problems experienced in the study. In general, low battery warnings on instruments were handled by re-charging the instruments or by making interim connections to local line power for completion of testing in a given day. In a few cases, the battery problems involved failures that required use of backup instruments. In Denver, the XL and one of the Microlead I instruments required use of backup instruments as indicated in Table 3-11. In Philadelphia, one of the Microlead I instruments required use of backup instruments as indicated in Table 3-12. In addition, the Lead Analyzer also experienced an operational failure in Philadelphia and had to be replaced by a backup instrument as indicated in Table 3-12. However, whether this failure was due to a battery related problem is not known.

Three of the XRF instruments included in the study had the capability to electronically store lead readings for later retrieval and examination. These included the X-MET 880, the MAP-3 and the Lead Analyzer. For these instruments, captured

electronic data were collected for later comparison to real-time hard-copy data forms. Problems with electronic collection of data occurred during the full study. In Denver, data storage failures were observed for the X-MET 880 and one of the MAP-3 instruments. In Philadelphia, data storage failures were again observed for the X-MET 880.

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Chapter 4 Summary: Paint-Chip Sample Data

- **Of the 1,290 paint samples collected and analyzed in the laboratory in this study, approximately 20% contained lead at a level equal to or greater than 1.0 mg/cm^2, one of the federal thresholds for defining LBP on painted surfaces. Approximately 29% of the samples contained lead equal to or greater than 0.5% by weight, the other federal threshold for LBP on painted surfaces.**

- **For the paint samples, lead levels expressed in mg/cm^2 and lead levels expressed in percent lead by weight were roughly equivalent. A level of 1.0 mg/cm^2 was roughly equivalent to 1.0% by weight and a level of 0.5% by weight was roughly equivalent to 0.5 mg/cm^2.**

- **Lead levels in paint showed significant variation within individual architectural components such as doors, walls, and baseboards.**

- **Variation between members of laboratory duplicate subsample pairs was much smaller than variation between members of duplicate samples obtained in the field.**

4 PAINT-CHIP SAMPLE DATA

4.1 DESCRIPTIVE STATISTICS ON LABORATORY ANALYSES

Descriptive statistics of the paint-chip samples address the study objective to investigate the variability of lead levels in the paint within the study sampling locations. Paint-chip samples were collected at 1,290 sampling locations in the three cities. There were 100 locations in two multifamily buildings in Louisville, 750 in ten single-family houses in Denver, and 440 in eight multifamily units in Philadelphia. As described in section 4.3.2 below, two paint samples (field duplicates) were collected at a subset of locations in order to estimate the variability in lead levels between samples collected a short distance apart on the same component. As described in section 4.3.1, some samples were analyzed in duplicate to estimate the variability of the analytical process. Thus, some locations have more than one sample and some samples have more than one reported measurement. A single measurement at each sampling location was designated as the primary measurement. Descriptive statistics on these 1,290 primary measurements are provided below.

Each sample was analyzed using a modified NIOSH 7082 method applied to a 0.5 gram subsample of the original sample (if it weighed more than 0.5 gram), taken after homogenization of the sample. Results were reported both in area units (mg/cm^2 lead) and in percent by weight units (% of lead in the sample by mass).

Samples were collected from six substrate types: brick, concrete, drywall, metal, plaster, and wood. The number of sampling locations for each substrate in each city and overall is shown in Table 4-1. The target allocations for the Denver and Philadelphia parts of the study are shown in Table 4-2; there were no targets for Louisville since this was a pilot study.

These counts were dictated by the realities of the testing sites. Philadelphia and Louisville were older public housing projects with very little painted brick and drywall. The Denver site consisted of 10 single-family homes, in which metal substrates were in short supply. By contrast, there were many metal components in Louisville and Philadelphia. Given the constraints of field sampling, the actual number of each substrate achieved was close to the target allocations for Denver and Philadelphia. The overall target was achieved exactly. For each substrates, Table 4-3 presents summary statistics for the

mass in grams of the primary paint-chip samples taken from each sampling location in each city and aggregated across cities.

Table 4-1. Number of Sampling Locations by Substrate and Overall.

SUBSTRATE	ALL CITIES	DENVER	PHILADELPHIA	LOUISVILLE
Brick	93	81	12	0
Concrete	226	98	120	8
Drywall	124	105	8	11
Metal	217	62	127	28
Plaster	242	101	121	20
Wood	388	303	52	33
Total	1,290	750	440	100

Table 4-2. Target Sample Allocations for Denver and Philadelphia by Substrate.

SUBSTRATE	DENVER	PHILADELPHIA	TOTAL
Brick	80	8	88
Concrete	170	48	218
Drywall	170	8	178
Metal	60	128	188
Plaster	100	120	220
Wood	170	128	298
Total	750	440	1,190

Table 4-4 presents summary statistics for the primary paint-chip samples in mg/cm^2 lead, first by housing unit in each city, then aggregated by city, and, finally, aggregated across cities for the overall study. Table 4-5 gives the same statistics by substrate for each city and overall. The same statistics for interior, exterior and common areas are provided in Table 4-6. Tables 4-7, 4-8, and 4-9 are the companion tables in percent by weight units.

In Denver, two units of the ten tested (unit numbers 3 and 8) stand out as having unusually high lead levels. Unit number 8, built in 1890, was the oldest unit studied and had high levels of lead throughout. Unit number 3 had several very high levels on wood substrates which created a skewed distribution. Apart

from these two units, lead levels in Denver were fairly consistent from house to house, although unit number 4 had some high levels in percent by weight. Levels in Philadelphia and

Table 4-3. Summary Statistics of Primary Sample Mass (grams) Categorized by City and Substrate.

CITY	SUBSTRATE	SAMPLE SIZE	MINIMUM	25TH PERCENTILE	MEDIAN	GEOMETRIC MEAN	ARITHMETIC MEAN	75TH PERCENTILE	MAXIMUM	STANDARD DEVIATION
DENVER	Brick	81	0.21	1.34	4.26	2.86	4.61	6.79	22.85	3.83
	Concrete	98	0.35	1.79	2.92	2.87	3.59	4.91	11.01	2.31
	Drywall	105	0.34	1.19	1.91	1.85	2.29	6.24	3.15	1.46
	Metal	62	0.06	1.30	0.54	0.54	0.75	0.97	2.23	0.61
	Plaster	101	0.40	1.38	2.16	2.29	3.12	3.39	25.18	3.29
	Wood	303	0.05	0.92	1.43	1.36	1.64	2.17	5.82	0.98
PHILADELPHIA	Brick	12	1.60	3.63	5.09	4.78	5.30	6.44	11.19	2.48
	Concrete	120	0.60	3.09	3.73	3.24	3.60	4.34	7.16	1.34
	Drywall	8	0.54	0.79	1.54	1.36	1.64	2.36	3.24	1.02
	Metal	127	0.21	1.43	2.38	2.00	2.36	3.19	7.31	1.20
	Plaster	121	1.21	2.89	3.63	3.62	3.84	4.59	8.58	1.31
	Wood	52	0.14	1.56	2.17	1.91	2.28	3.03	4.66	1.07
LOUISVILLE	Concrete	8	0.50	1.45	2.15	1.86	2.17	2.70	4.28	1.13
	Drywall	11	0.57	0.73	0.83	0.88	0.91	1.17	1.34	0.24
	Metal	28	0.67	1.48	1.80	1.75	1.89	2.24	4.36	0.75
	Plaster	20	0.88	1.78	2.36	2.14	2.24	2.56	3.19	0.62
	Wood	33	0.31	0.98	1.45	1.32	1.49	1.82	3.91	0.73
ALL CITIES	Brick	93	0.21	1.60	4.26	3.05	4.70	6.79	22.85	3.68
	Concrete	226	0.35	2.25	3.48	3.02	3.54	4.42	11.01	1.83
	Drywall	124	0.34	1.06	1.74	1.70	2.12	2.79	6.24	1.43
	Metal	217	0.06	0.79	1.70	1.35	1.84	2.62	7.31	1.23
	Plaster	242	0.40	2.04	3.04	2.86	3.40	4.16	25.18	2.37

	Wood	388	0.05	0.96	1.51	1.42	1.71	2.34	5.82	1.00
	All	1,290	0.05	1.21	2.17	1.97	2.63	3.49	25.18	2.07

Table 4-4. Summary Statistics of ICP Analyses of Primary Samples (mg/cm^2 lead) Categorized by Unit and City.

CITY	UNIT ID	YEAR BUILT	SAMPLE SIZE	MINIMUM	25TH PERCENTILE	MEDIAN	GEOMETRIC MEAN	ARITHMETIC MEAN	75TH PERCENTILE	MAXIMUM	STANDARD DEVIATION
DENVER	1	1943	75	0.0001	0.0006	0.003	0.01	0.19	0.02	3.07	0.53
	2	1948	75	0.0003	0.03	0.09	0.10	0.47	0.41	2.89	0.74
	3	1952	75	0.0001	0.04	0.09	0.08	2.19	0.24	30.11	6.44
	4	1905	75	0.0003	0.002	0.02	0.03	0.40	0.22	5.75	0.99
	5	1949	75	0.0002	0.01	0.08	0.05	0.45	0.23	3.61	0.86
	6	1948	75	0.0002	0.01	0.18	0.08	0.50	0.71	3.16	0.69
	7	1952	75	0.0007	0.03	0.08	0.12	0.76	0.63	5.06	1.34
	8	1890	75	0.0004	0.62	6.67	2.34	8.32	13.07	37.29	8.58
	9	1949	75	0.0003	0.02	0.20	0.08	0.31	0.47	2.27	0.39
	10	1947	75	0.0005	0.003	0.04	0.03	0.27	0.15	4.55	0.72
	ALL	N/A	750	0.0001	0.01	0.08	0.07	1.39	0.50	37.29	4.19
PHILADELPHIA	1	1942	55	0.0004	0.05	0.60	0.23	0.93	1.31	3.84	1.11
	2	1942	55	0.0002	0.05	0.37	0.12	0.44	0.57	2.57	0.48
	3	1942	55	0.0003	0.21	0.38	0.32	0.68	0.79	3.97	0.76
	4	1942	55	0.0021	0.24	0.35	0.26	0.41	0.43	2.67	0.42
	5	1942	55	0.0005	0.25	0.39	0.40	1.05	1.68	7.23	1.42
	6	1942	55	0.0017	0.26	0.46	0.25	0.53	0.60	6.50	0.86
	7	1942	55	0.0078	0.09	0.17	0.14	0.27	0.26	4.48	0.61
	8	1942	55	0.0028	0.13	0.20	0.27	0.73	0.90	6.07	1.16

	ALL	1942	440	0.0002	0.16	0.32	0.23	0.63	0.61	7.23	0.94
LOUISVILLE	1	1937	50	0.0001	0.12	0.29	0.20	1.87	2.56	14.05	3.02
	2	1937	50	0.0004	0.14	0.45	0.28	2.00	2.84	11.64	3.22
	ALL	1937	100	0.0001	0.13	0.39	0.24	1.93	2.56	14.05	3.11
ALL CITIES	ALL	N/A	1290	0.0001	0.03	0.20	0.12	1.17	0.62	37.29	3.38

Table 4-5. Summary Statistics of ICP Analyses of Primary Samples (mg/cm^2 lead) Categorized by Substrate.

CITY	SUBSTRATE	SAMPLE SIZE	MINIMUM	25TH PERCENTILE	MEDIAN	GEOMETRIC MEAN	ARITHMETIC MEAN	75TH PERCENTILE	MAXIMUM	STANDARD DEVIATION
DENVER	Brick	81	0.0001	0.003	0.28	0.10	3.86	1.67	34.09	7.52
	Concrete	98	0.0005	0.01	0.03	0.05	1.14	0.28	15.98	2.88
	Drywall	105	0.0001	0.01	0.04	0.02	0.11	0.11	0.91	0.18
	Metal	62	0.0002	0.002	0.05	0.03	0.45	0.73	2.44	0.72
	Plaster	101	0.0003	0.01	0.11	0.08	1.59	0.26	37.29	5.21
	Wood	303	0.0001	0.03	0.12	0.12	1.37	1.22	30.11	3.79
PHILADELPHIA	Brick	12	0.0011	0.002	0.03	0.02	0.12	0.16	0.71	0.21
	Concrete	120	0.0013	0.07	0.26	0.17	0.40	0.47	3.60	0.55
	Drywall	8	0.0003	0.0004	0.002	0.001	0.002	0.002	0.01	0.00
	Metal	127	0.0020	0.20	0.39	0.40	0.99	1.27	6.50	1.31
	Plaster	121	0.0005	0.20	0.30	0.27	0.43	0.44	2.64	0.48
	Wood	52	0.0002	0.21	0.79	0.38	0.98	1.21	7.23	1.17
LOUISVILLE	Concrete	8	0.0400	0.14	0.25	0.32	0.92	1.48	3.54	1.34
	Drywall	11	0.0001	0.00	0.00	0.0005	0.0004	0.001	0.001	0.0005
	Metal	28	0.0040	0.17	0.43	0.34	0.68	0.72	4.03	0.91

	Plaster	20	0.1000	0.23	0.37	0.68	1.68	3.57	5.53	2.01
	Wood	33	0.0001	0.13	2.43	0.72	4.03	7.01	14.05	4.34
ALL CITIES	Brick	93	0.0001	0.003	0.19	0.08	3.38	0.65	34.09	7.13
	Concrete	226	0.0005	0.02	0.19	0.10	0.74	0.43	15.98	1.98
	Drywall	124	0.0001	0.002	0.02	0.01	0.09	0.08	0.90	0.17
	Metal	217	0.0002	0.10	0.34	0.19	0.79	0.90	6.50	1.14
	Plaster	242	0.0003	0.10	0.23	0.18	1.02	0.42	37.29	3.47
	Wood	388	0.0001	0.04	0.17	0.16	1.54	1.36	30.11	3.68

Table 4-6. Summary Statistics of ICP Analyses of Primary Samples (mg/cm^2 lead) Categorized by City and Interior, Exterior, and Common Areas.

CITY	LOCATION	SAMPLE SIZE	MINIMUM	25TH PERCENTILE	MEDIAN	GEOMETRIC MEAN	ARITHMETIC MEAN	75TH PERCENTILE	MAXIMUM	STANDARD DEVIATION
DENVER	Interior	447	0.0001	0.008	0.07	0.05	0.83	0.21	37.29	3.64
	Exterior	303	0.0001	0.007	0.25	0.13	2.20	1.86	30.11	4.78
PHILADELPHIA	Interior	385	0.0002	0.20	0.36	0.30	0.64	0.62	7.23	0.84
	Common	55	0.0011	0.007	0.03	0.05	0.60	0.18	6.50	1.47
LOUISVILLE	Interior	91	0.0001	0.12	0.32	0.34	1.34	2.11	11.64	2.16
	Exterior	9	0.0360	3.54	10.16	4.30	7.94	10.91	14.05	4.73
ALL CITIES	N/A	1290	0.0001	0.03	0.20	0.12	1.17	0.62	37.29	3.38

Table 4-7. Summary Statistics of ICP Analyses of Primary Samples (percent by weight lead) Categorized by Unit and City.

CITY	UNIT ID	YEAR BUILT	SAMPLE SIZE	MINIMUM	25TH PERCENTILE	MEDIAN	GEOMETRIC MEAN	ARITHMETIC MEAN	75TH PERCENTILE	MAXIMUM	STANDARD DEVIATION
DENVER	1	1943	75	0.0005	0.002	0.01	0.02	0.30	0.08	5.26	0.90
	2	1948	75	0.0020	0.07	0.13	0.20	1.14	0.45	8.87	2.10
	3	1952	75	0.0006	0.09	0.20	0.21	3.22	0.48	34.56	7.80
	4	1905	75	0.0008	0.01	0.04	0.05	0.83	0.29	11.37	2.18
	5	1949	75	0.0010	0.03	0.12	0.09	0.49	0.24	3.25	0.89
	6	1948	75	0.0010	0.02	0.35	0.14	0.66	1.28	3.02	0.80
	7	1952	75	0.0020	0.05	0.15	0.19	0.90	0.99	7.72	1.56
	8	1890	75	0.0030	0.62	3.09	1.46	3.46	5.28	14.83	3.14
	9	1949	75	0.0007	0.03	0.24	0.12	0.43	0.46	4.83	0.71
	10	1947	75	0.0007	0.002	0.02	0.02	0.16	0.09	2.21	0.40
	ALL	N/A	750	0.0005	0.02	0.13	0.11	1.16	0.72	34.56	3.11
PHILADELPHIA	1	1942	55	0.0009	0.08	0.38	0.25	1.10	1.27	8.05	1.74
	2	1942	55	0.0010	0.07	0.31	0.17	0.75	0.65	6.80	1.37
	3	1942	55	0.0010	0.15	0.28	0.27	0.65	0.78	6.42	1.08
	4	1942	55	0.0020	0.18	0.27	0.25	0.52	0.37	9.40	1.27
	5	1942	55	0.0010	0.17	0.26	0.33	0.91	0.83	4.52	1.31
	6	1942	55	0.0010	0.15	0.31	0.20	0.50	0.43	9.75	1.29
	7	1942	55	0.0040	0.08	0.13	0.13	0.34	0.20	9.44	1.26
	8	1942	55	0.0006	0.12	0.21	0.26	0.75	0.81	12.16	1.70
	ALL	1942	440	0.0006	0.13	0.26	0.22	0.69	0.56	12.16	1.40

LOUISVILLE	1	1937	50	0.0004	0.16	0.39	0.34	2.69	4.73	13.62	4.03
	2	1937	50	0.0008	0.21	0.46	0.46	2.64	3.68	14.16	4.09
	ALL	1937	100	0.0004	0.17	0.44	0.40	2.67	4.14	14.16	4.04
ALL CITIES	ALL	N/A	1290	0.0004	0.05	0.20	0.15	1.12	0.72	34.56	2.79

Table 4-8. Summary Statistics of ICP Analyses of Primary Samples (percent by weight lead) Categorized by Substrate.

CITY	SUB	SAMPLE SIZE	MINIMUM	25TH PERCENTILE	MEDIAN	GEOMETRIC MEAN	ARITHMETIC MEAN	75TH PERCENTILE	MAXIMUM	STANDARD DEVIATION
DENVER	Brick	81	0.0008	0.01	0.15	0.10	1.28	0.98	9.33	2.26
	Concrete	98	0.0007	0.004	0.04	0.05	0.76	0.29	9.69	1.78
	Drywall	105	0.0005	0.01	0.04	0.03	0.14	0.11	1.44	0.28
	Metal	62	0.0020	0.02	0.20	0.17	1.61	1.71	16.68	3.14
	Plaster	101	0.0007	0.03	0.14	0.09	0.59	0.32	8.49	1.35
	Wood	303	0.0008	0.07	0.20	0.22	1.70	1.61	34.56	4.26
PHILADELPHIA	Brick	12	0.0006	0.001	0.02	0.01	0.06	0.07	0.35	0.10
	Concrete	120	0.0010	0.08	0.20	0.14	0.27	0.30	2.32	0.36
	Drywall	8	0.0010	0.001	0.002	0.002	0.002	0.002	0.01	0.002
	Metal	127	0.0040	0.21	0.46	0.51	1.37	1.17	12.16	2.27
	Plaster	121	0.0010	0.13	0.22	0.20	0.30	0.30	1.89	0.31
	Wood	52	0.0009	0.33	0.91	0.52	1.14	1.41	5.65	1.18
LOUISVILLE	Concrete	8	0.1200	0.17	0.23	0.44	1.21	1.77	5.21	1.89
	Drywall	11	0.0004	0.0008	0.001	0.001	0.001	0.002	0.003	0.0008
	Metal	28	0.0100	0.25	0.46	0.50	1.07	0.95	5.63	1.60
	Plaster	20	0.1300	0.34	0.47	0.82	1.65	3.33	5.11	1.83

	Wood	33	0.0005	0.31	4.73	1.39	5.89	11.10	14.16	5.38
ALL CITIES	Brick	93	0.0006	0.004	0.09	0.07	1.12	0.42	9.33	2.15
	Concrete	226	0.0007	0.03	0.15	0.09	0.52	0.30	9.69	1.27
	Drywall	124	0.0004	0.002	0.03	0.02	0.12	0.09	1.44	0.26
	Metal	217	0.0020	0.16	0.40	0.37	1.40	1.27	16.68	2.48
	Plaster	242	0.0007	0.10	0.21	0.16	0.53	0.35	8.49	1.10
	Wood	388	0.0005	0.08	0.31	0.29	1.98	1.94	34.56	4.27

Table 4-9. Summary Statistics of ICP Analyses of Primary Samples (percent by weight lead) Categorized by City and Interior, Exterior, and Common Areas.

CITY	LOCATION	SAMPLE SIZE	MINIMUM	25TH PERCENTILE	MEDIAN	GEOMETRIC MEAN	ARITHMETIC MEAN	75TH PERCENTILE	MAXIMUM	STANDARD DEVIATION
DENVER	Interior	447	0.0005	0.02	0.10	0.07	0.54	0.26	15.72	1.61
	Exterior	303	0.0007	0.02	0.28	0.21	2.07	2.58	34.56	4.34
PHILADELPHIA	Interior	385	0.0009	0.15	0.27	0.27	0.58	0.56	7.18	0.88
	Common	55	0.0006	0.004	0.06	0.07	1.45	0.28	12.16	3.12
LOUISVILLE	Interior	91	0.0004	0.15	0.39	0.30	1.97	2.98	13.40	3.14
	Exterior	9	0.1835	5.21	12.61	6.28	9.73	13.28	14.16	5.37
ALL CITIES	N/A	1290	0.0004	0.05	0.20	0.15	1.12	0.72	34.56	2.79

Louisville show less variation from unit to unit than in Denver.

The arithmetic mean lead level in both types of units was highest in Louisville, with Denver next and Philadelphia last. However, Denver had the lowest median level. This is explained by the impact of the two units in Denver with the very high levels. If these units are excluded, the arithmetic mean level in Denver drops from 1.39 mg/cm^2 lead to 0.42 mg/cm^2 lead and from 1.16% lead to 0.62% lead. Thus, without units number 3 and number 8, Denver also has the lowest arithmetic mean levels, and its levels are quite comparable to those in Philadelphia. The higher mean levels in Louisville are partially explained by very high levels of lead on exterior samples in Louisville (see Tables 4-6 and 4-9). However, interior lead levels in Louisville are also higher than interior levels in the other two cities.

Arithmetic mean lead levels are fairly consistent across cities for metal and drywall, and are somewhat consistent for concrete. For brick, plaster and wood, more significant variations between cities are apparent. For wood, some of the differences are explained by the lack of exterior samples in Philadelphia. For brick, the sampling locations in Philadelphia had been first painted only recently, explaining the low lead levels as compared to Denver. For plaster, the source of the variation is unexplained.

Across the whole study, median lead levels are comparable for all substrates except drywall, which has much lower levels. Arithmetic mean levels are highest for brick in area units and for wood in percent by weight units. It is likely that the percent by weight lead in the brick samples was diluted by inclusion of substrate caused by difficulties encountered while removing samples.

Figures 4-1 through 4-10 graphically depict the frequency distribution of lead levels in mg/cm^2 for all data combined, by city, and by substrate. The horizontal axis shows the midpoint of the corresponding interval. For example, the second interval, with midpoint 0.3 mg/cm^2 lead, contains all samples with lead level greater than or equal to 0.2 mg/cm^2 and less than 0.4 mg/cm^2. The last bar in each figure covers all samples with lead levels 3.8 mg/cm^2 or greater. Figures 4-11 through 4-20 are the companion graphs for percent by weight units.

The figures confirm the highly skewed distribution of the sample lead levels suggested by the summary statistics in Tables 4-3 through 4-9. Data collected in Denver were the most skewed.

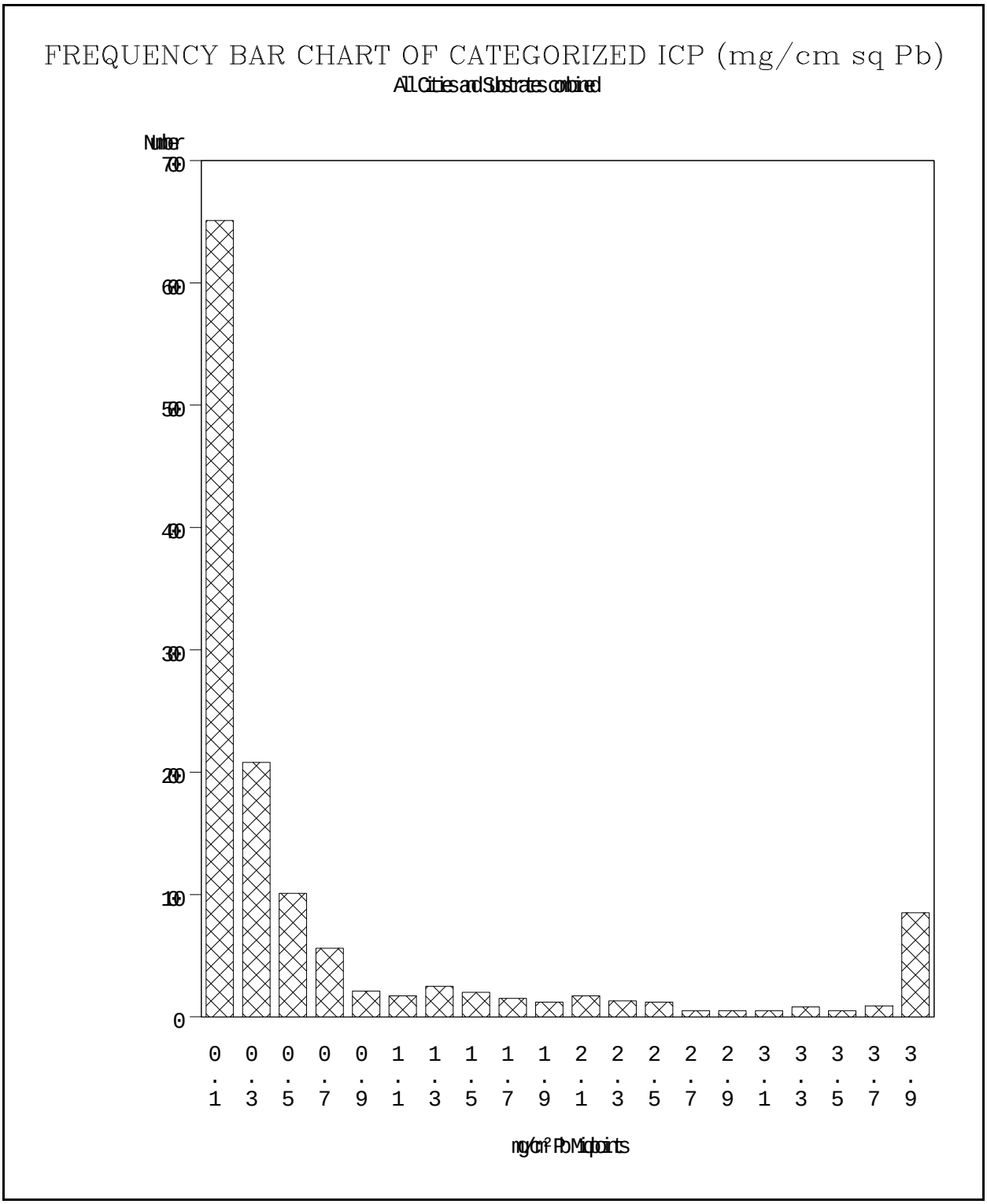

Figure 4-1. Frequency bar chart of primary ICP measurements (mg/cm^2 lead) for all substrates combined in all cities. A total of 253 measurements (19.6%) were greater than or equal to 1.0 mg/cm^2.

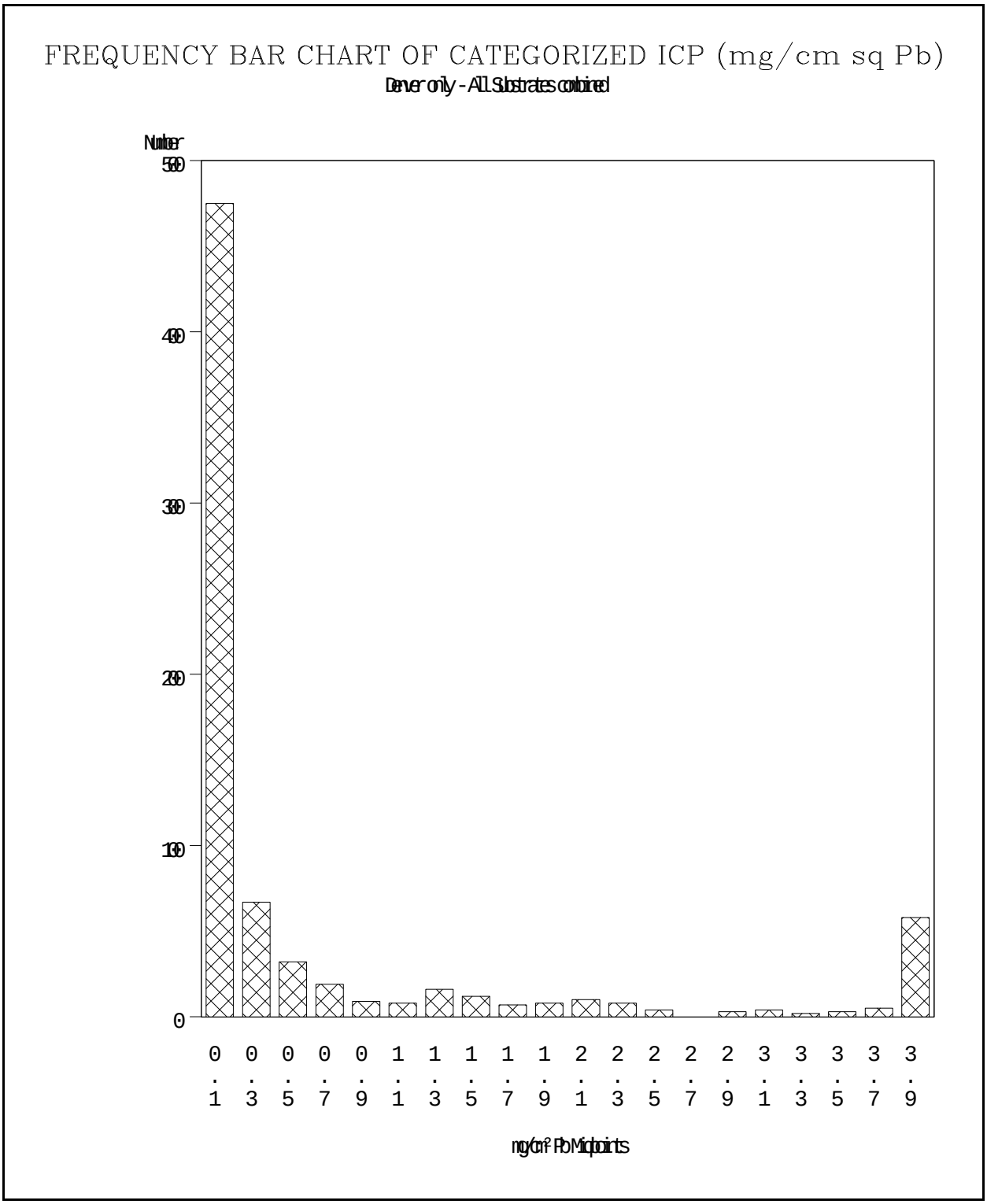

Figure 4-2. Frequency bar chart of primary ICP measurements (mg/cm^2 lead) for all substrates combined in Denver. A total of 148 measurements (19.7%) were greater than or equal to 1.0 mg/cm^2.

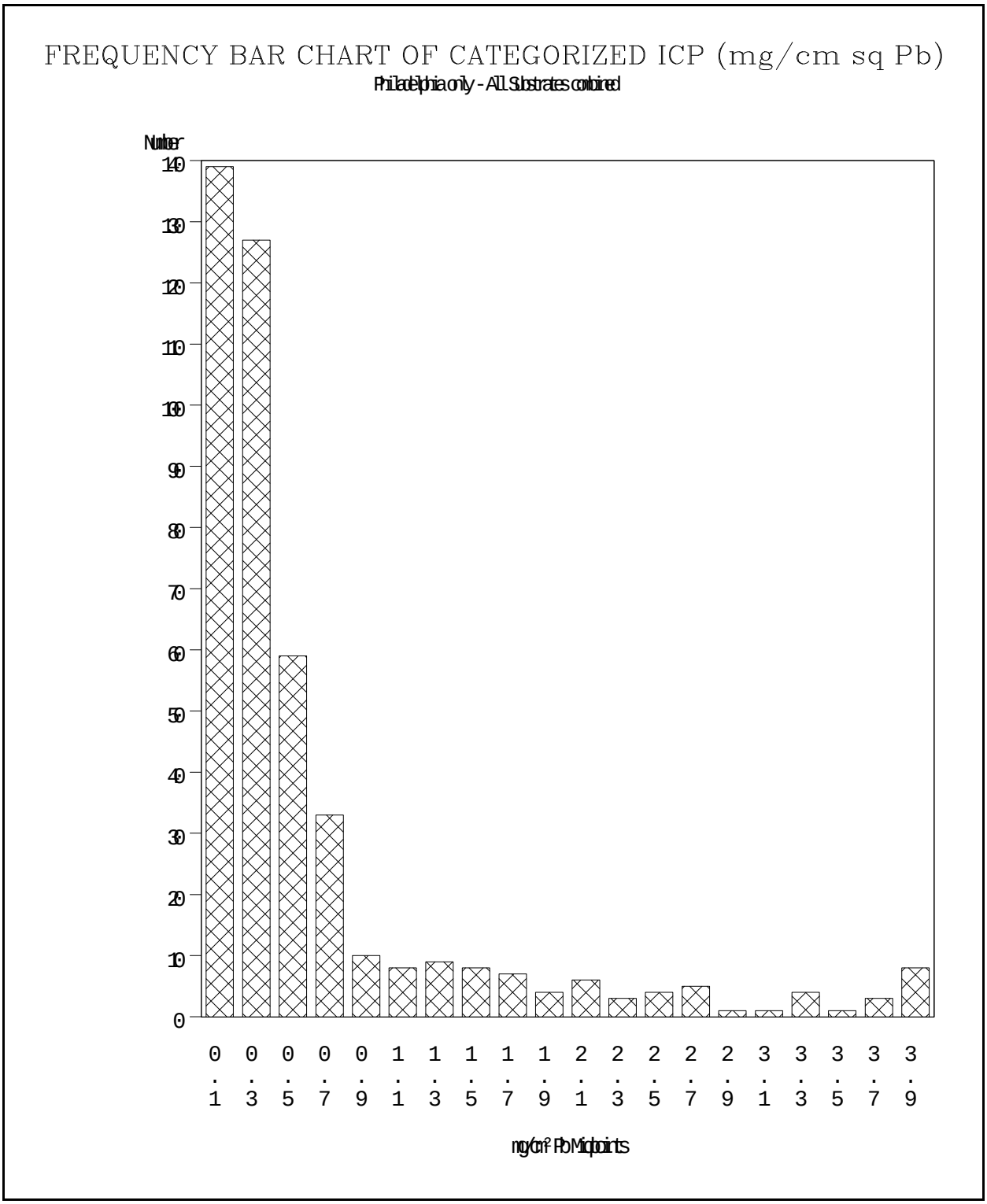

Figure 4-3. Frequency bar chart of primary ICP measurements (mg/cm^2 lead) for all substrates combined in Philadelphia. A total of 72 measurements (16.4%) were greater than or equal to 1.0 mg/cm^2.

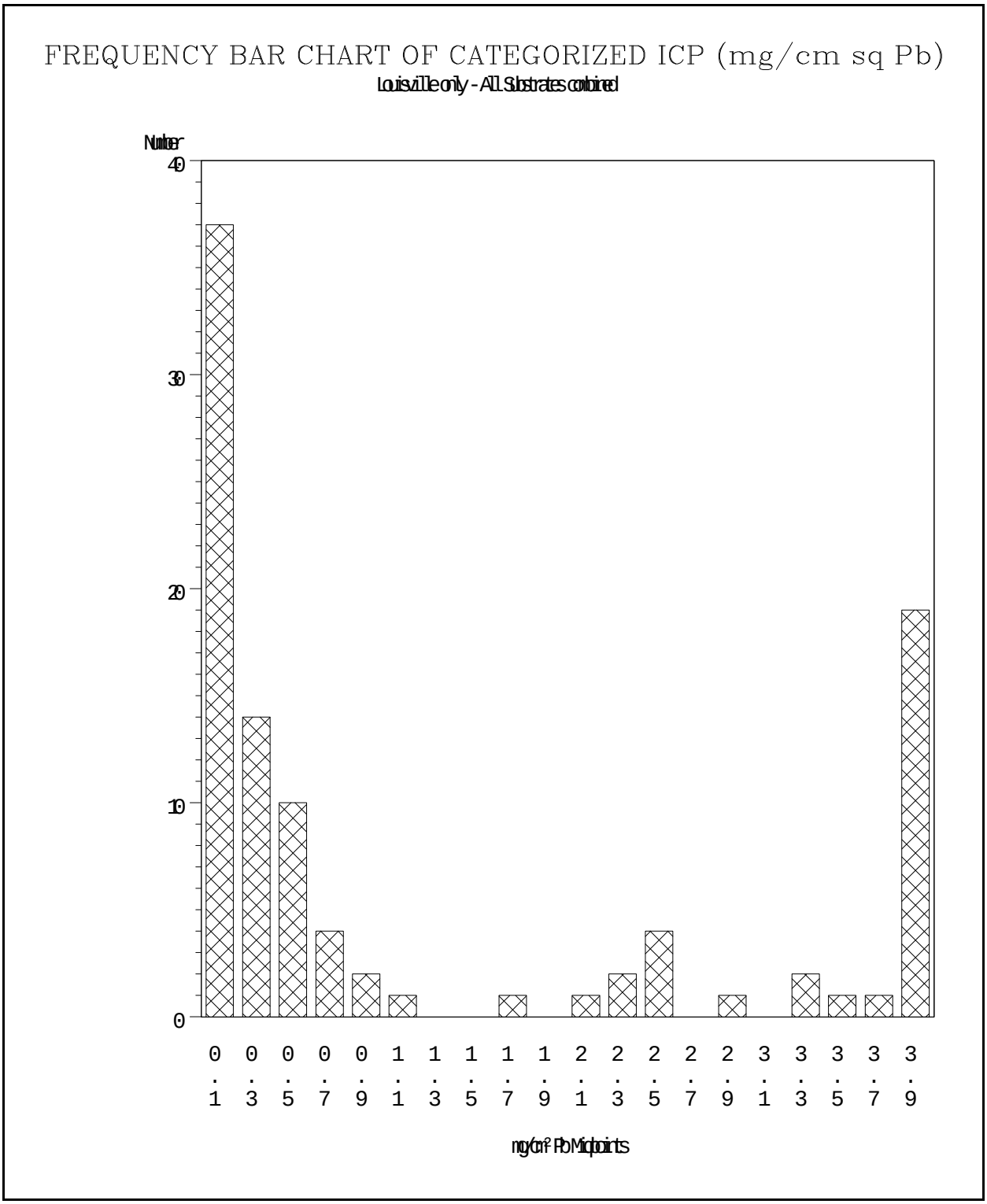

Figure 4-4. Frequency bar chart of primary ICP measurements (mg/cm^2 lead) for all substrates combined in Louisville. A total of 33 measurements (33.0%) were greater than or equal to 1.0 mg/cm^2.

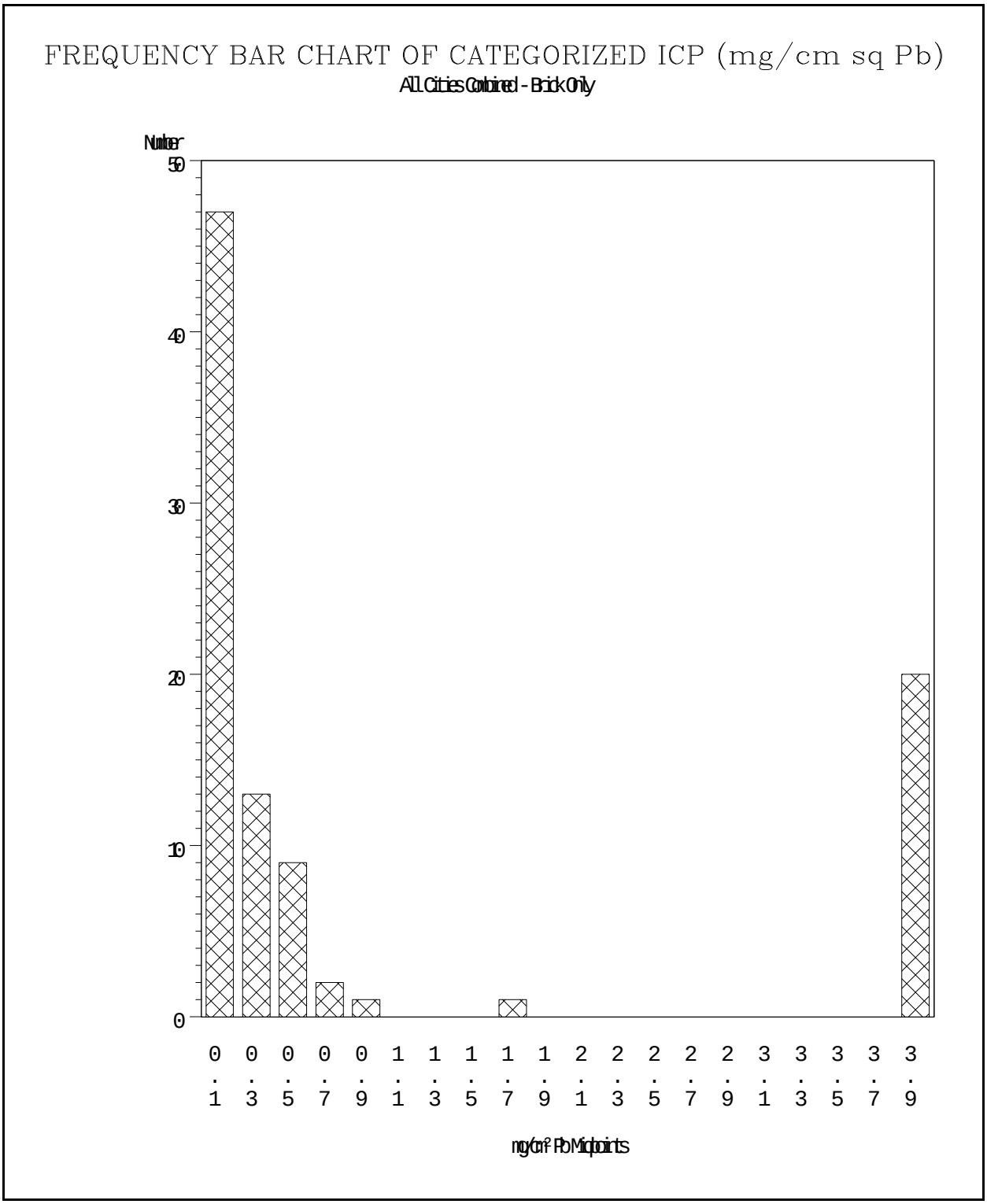

Figure 4-5. Frequency bar chart of primary ICP measurements (mg/cm^2 lead) for brick in all cities. A total of 21 measurements (22.6%) were greater than or equal to 1.0 mg/cm^2.

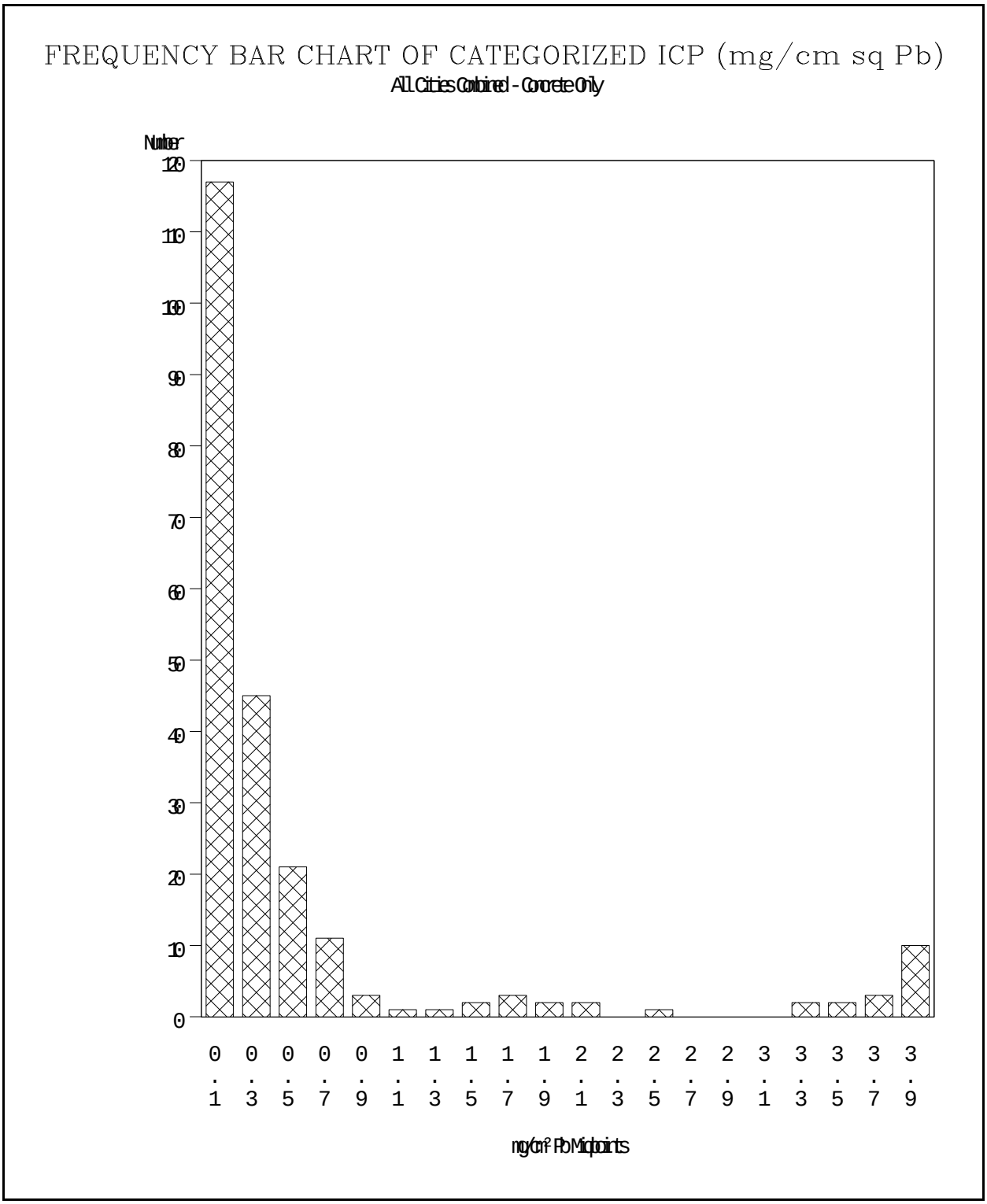

Figure 4-6. Frequency bar chart of primary ICP measurements (mg/cm^2 lead) for concrete from all cities. A total of 29 measurements (12.8%) were greater than or equal to 1.0 mg/cm^2.

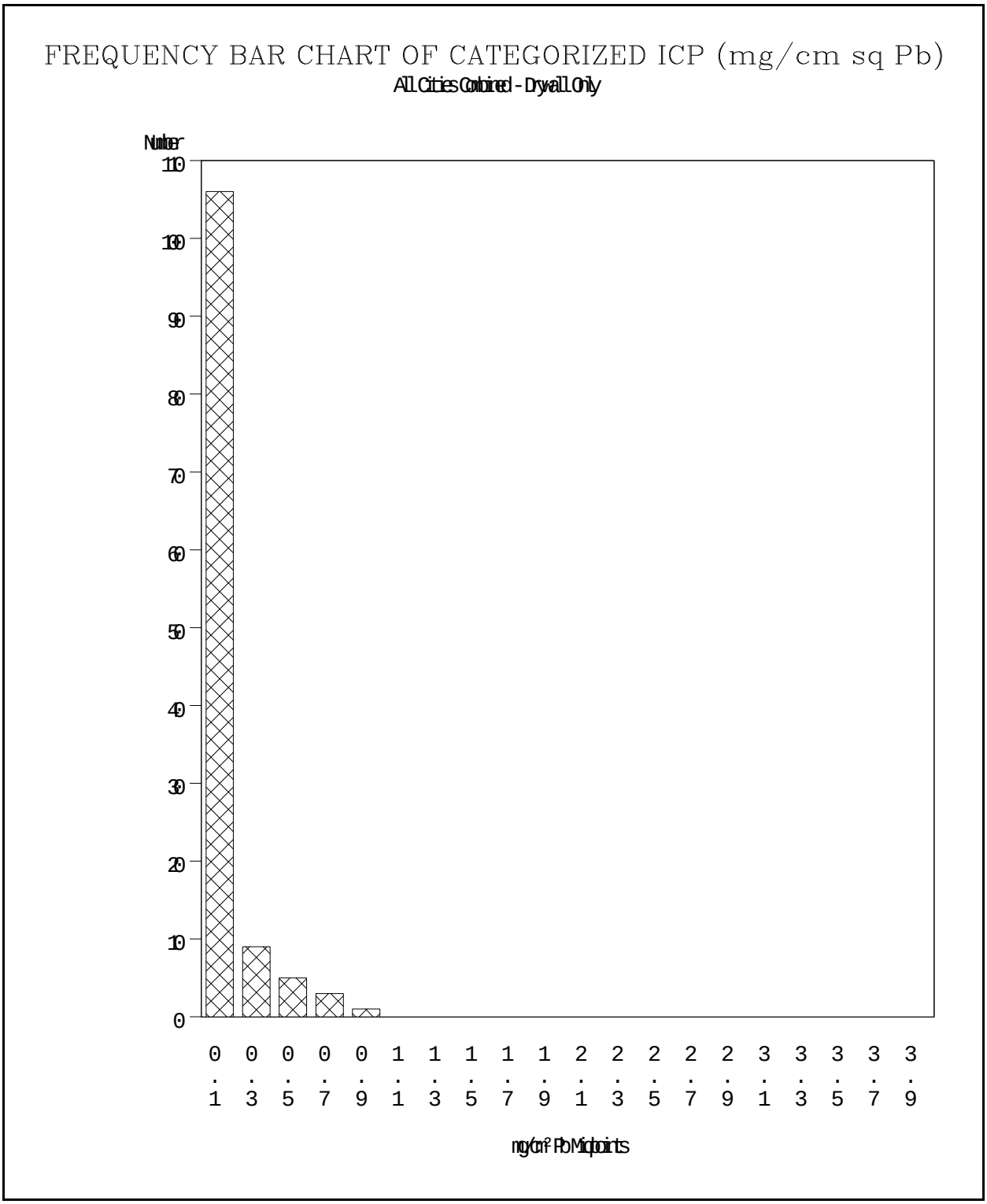

Figure 4-7. Frequency bar chart of primary ICP measurements (mg/cm^2 lead) for drywall in all cities. Out of 124 measurements, none were greater than or equal to 1.0 mg/cm^2.

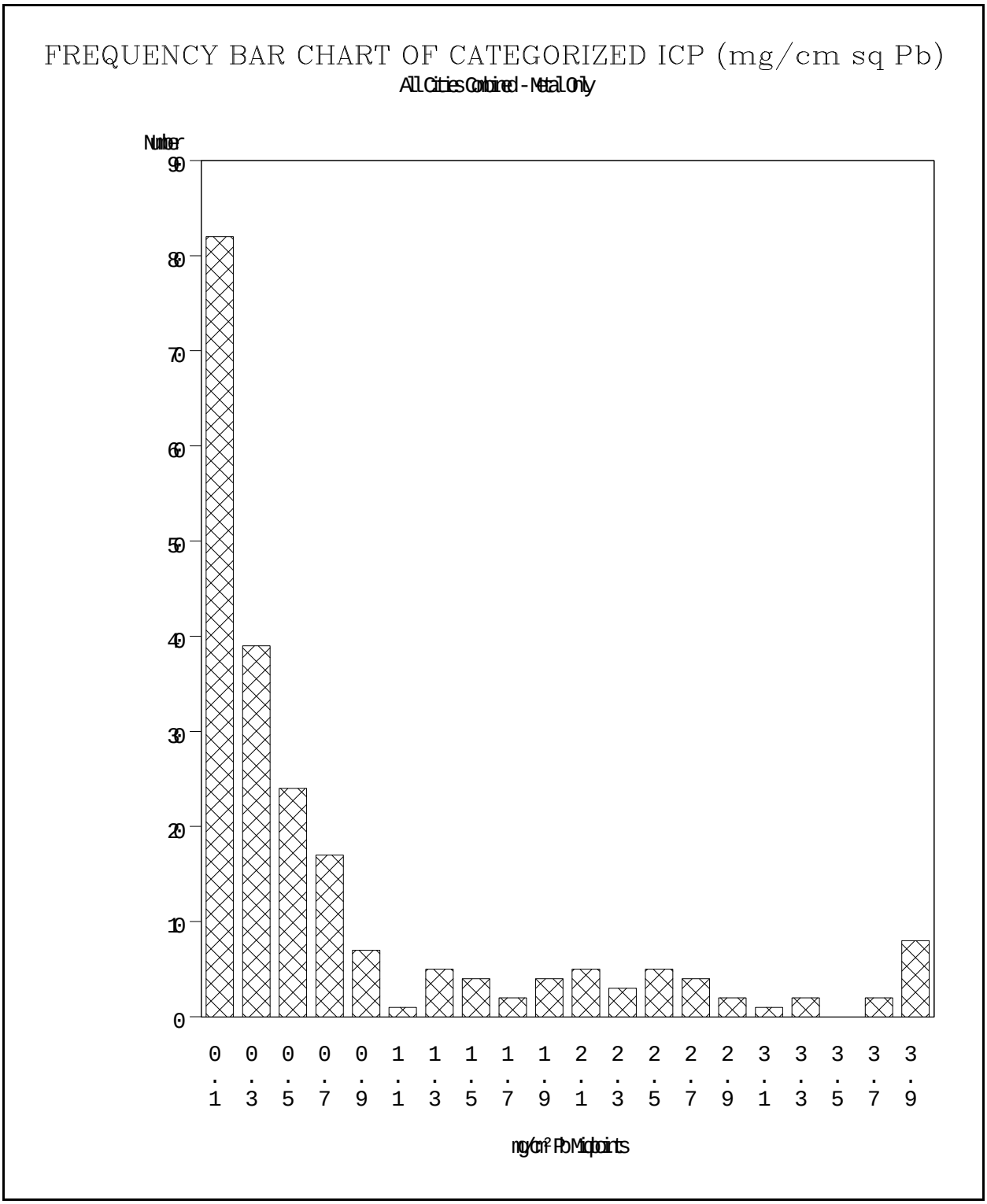

Figure 4-8. Frequency bar chart of primary ICP measurements (mg/cm^2 lead) for metal in all cities. A total of 48 measurements (22.1%) were greater than or equal to 1.0 mg/cm^2.

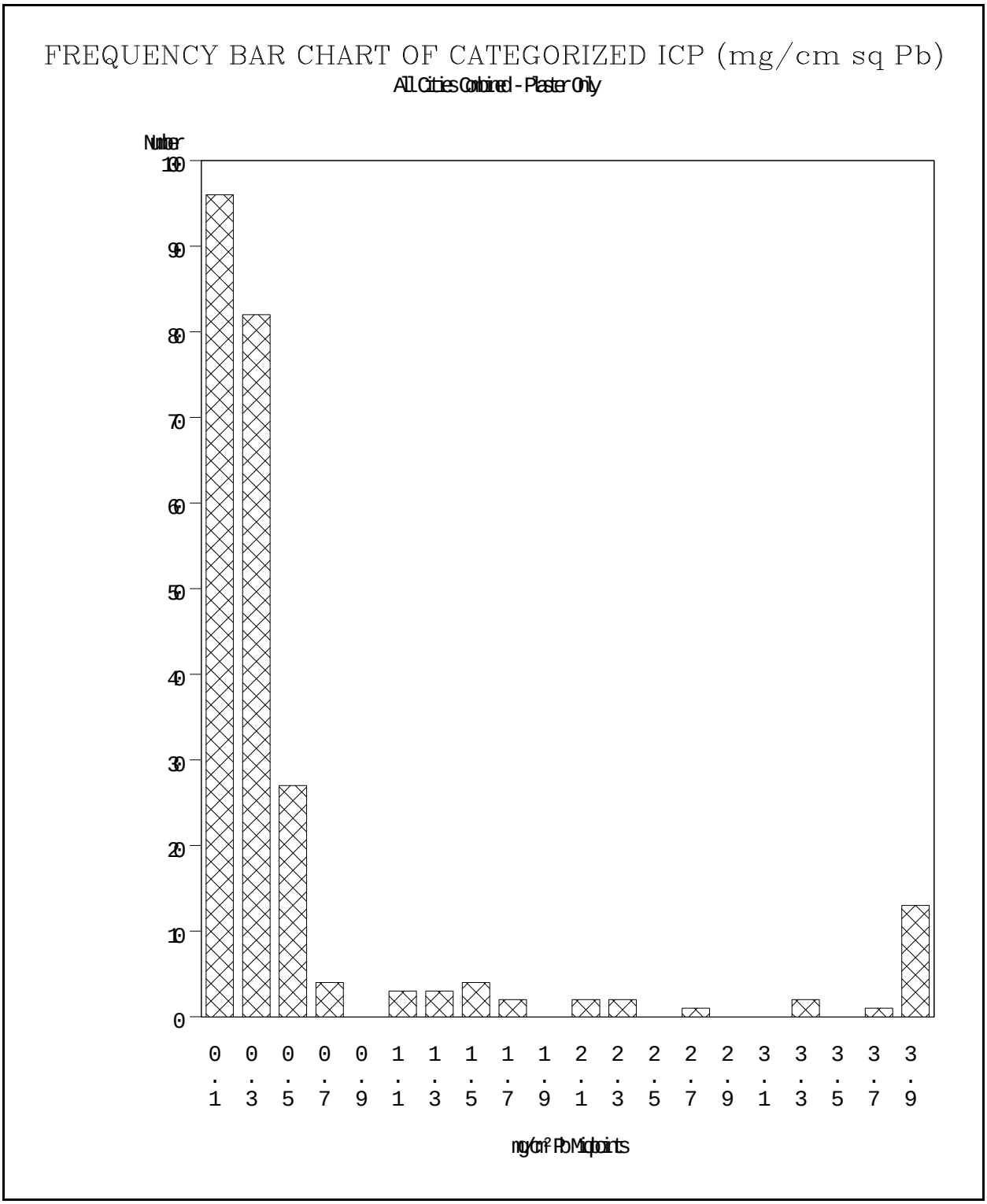

Figure 4-9. Frequency bar chart of primary ICP measurements (mg/cm^2 lead) for plaster in all cities. A total of 33 measurements (13.6%) were greater than or equal to 1.0 mg/cm^2.

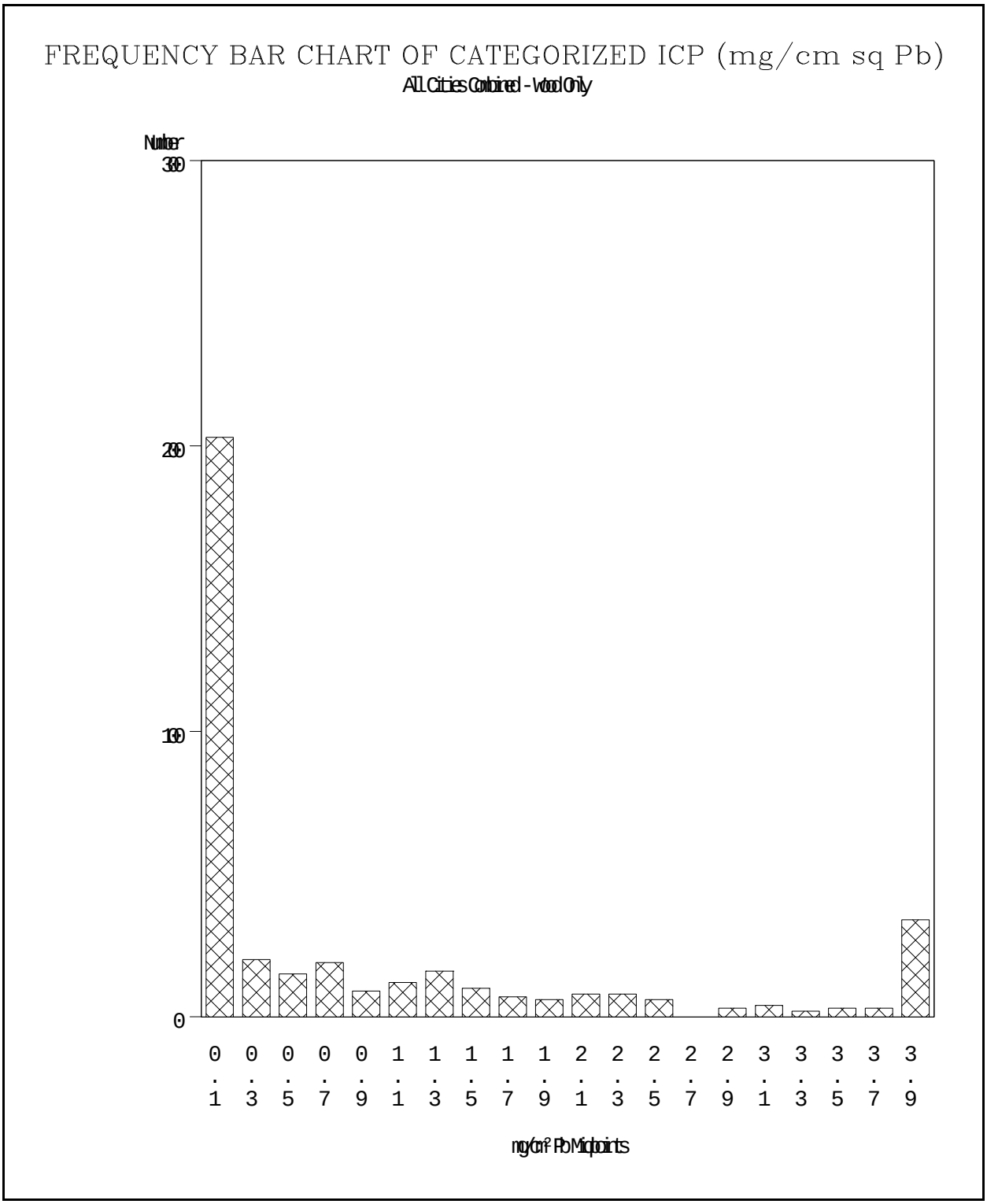

Figure 4-10. Frequency bar chart of primary ICP measurements (mg/cm^2 lead) for wood in all cities. A total of 122 measurements (31.4%) were greater than or equal to 1.0 mg/cm^2.

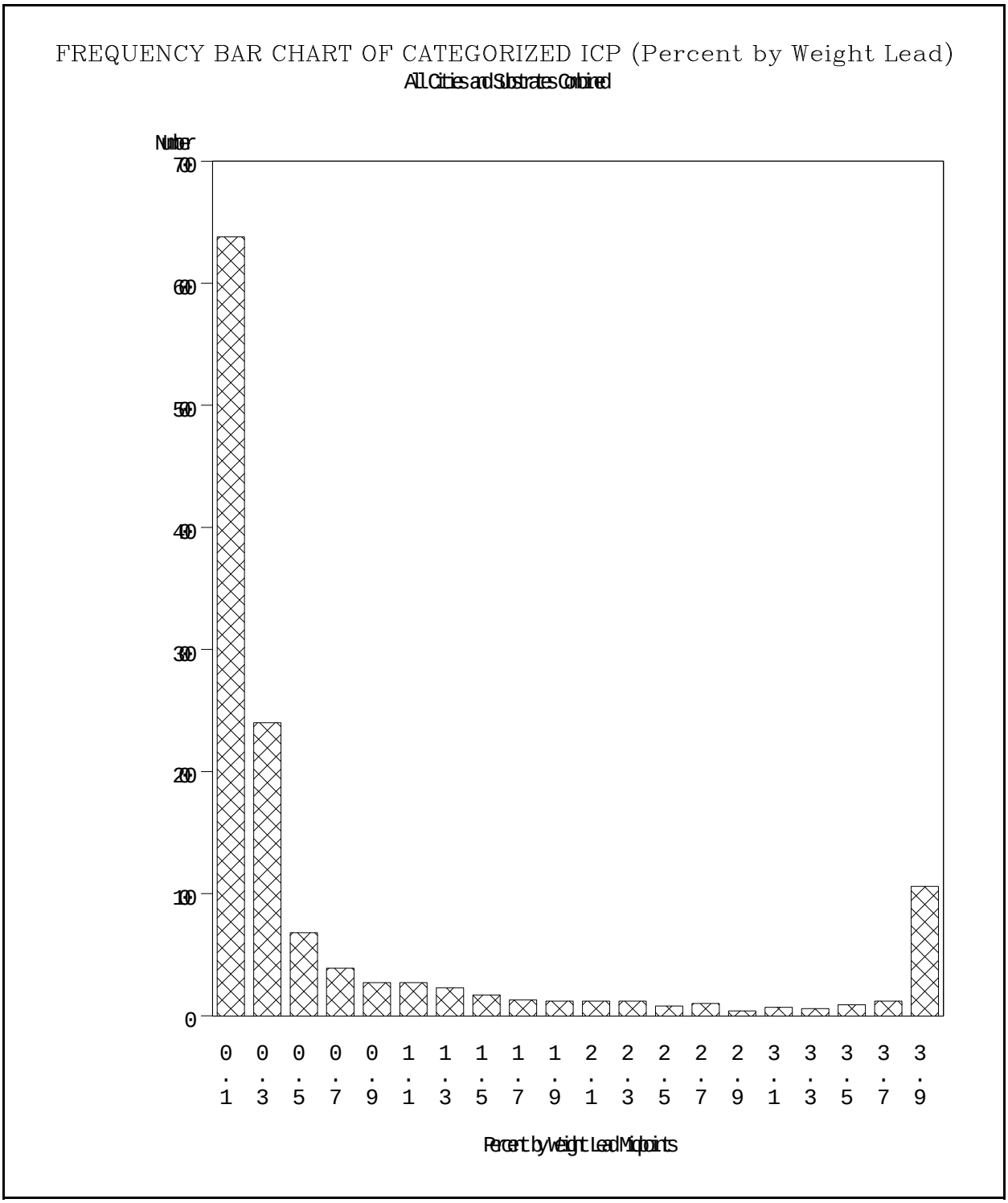

Figure 4-11. Frequency bar chart of primary ICP measurements (percent by weight lead) for all substrates and cities combined. A total of 372 (28.8%) measurements were greater than or equal to 0.5% lead.

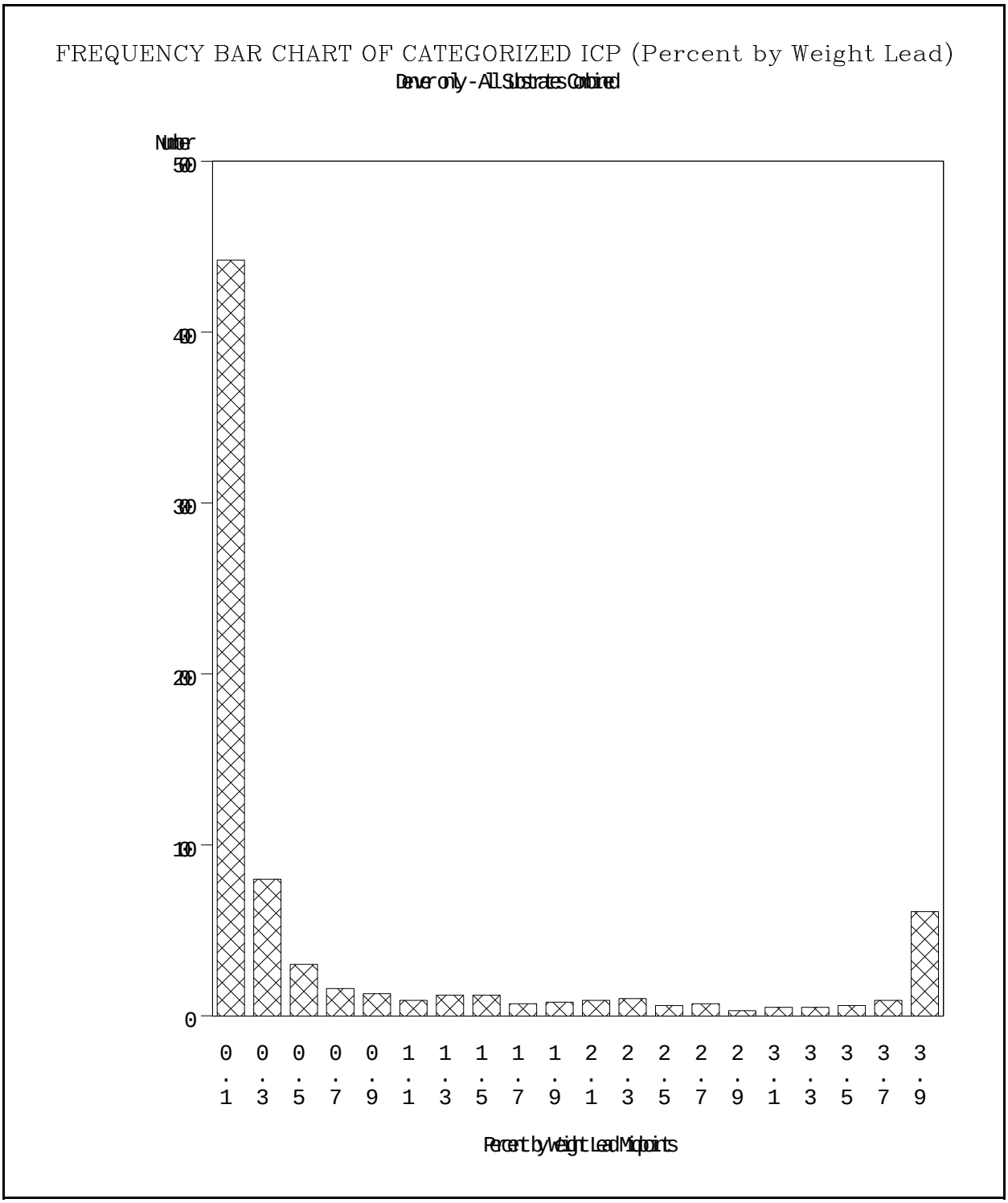

Figure 4-12. Frequency bar chart of primary ICP measurements (percent by weight lead) for all substrates combined in Denver. A total of 209 (27.9%) measurements were greater than or equal to 0.5% lead.

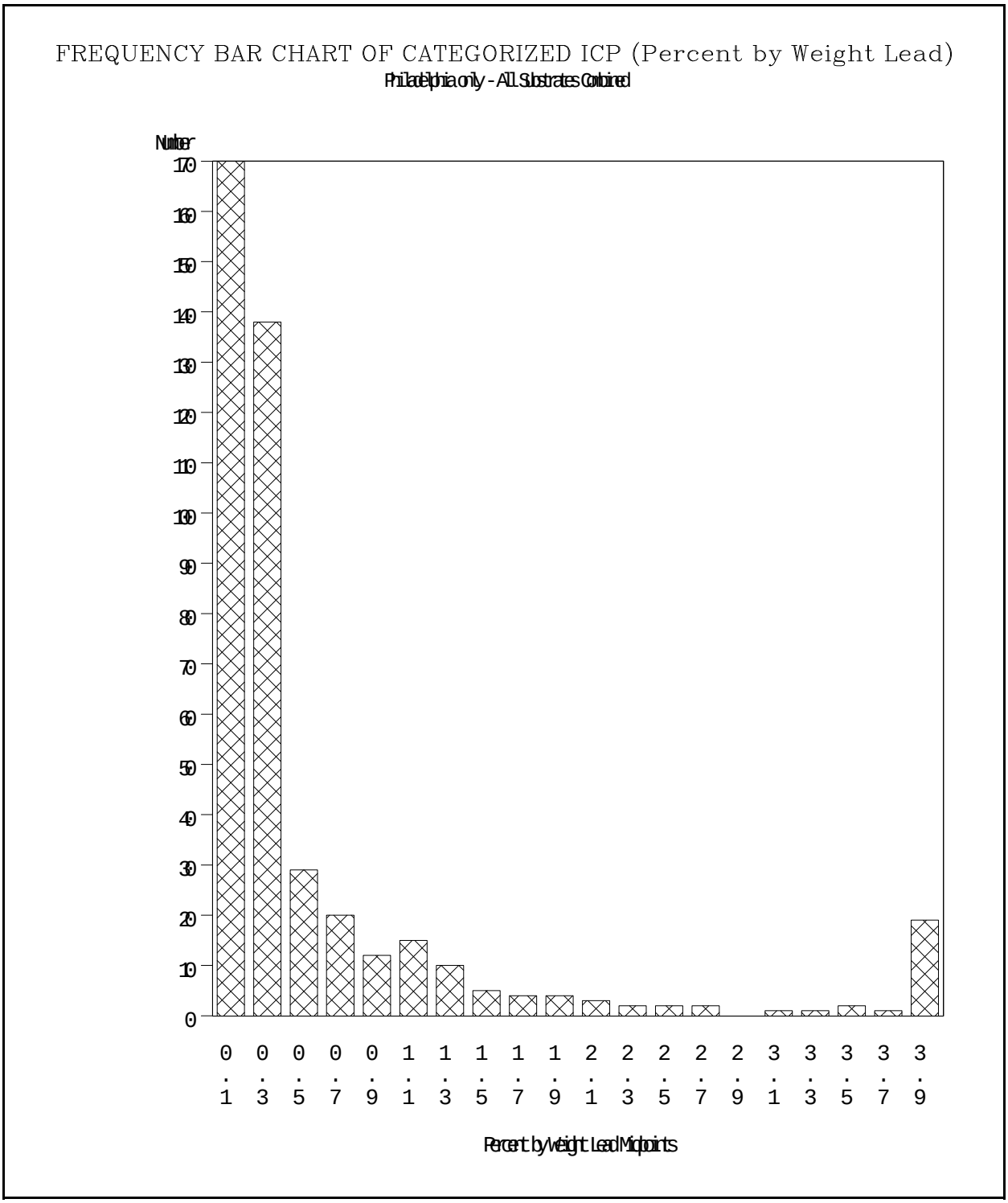

Figure 4-13. Frequency bar chart of primary ICP measurements (percent by weight lead) for all substrates combined in Philadelphia. A total of 116 (26.4%) measurements were greater than or equal to 0.5% lead.

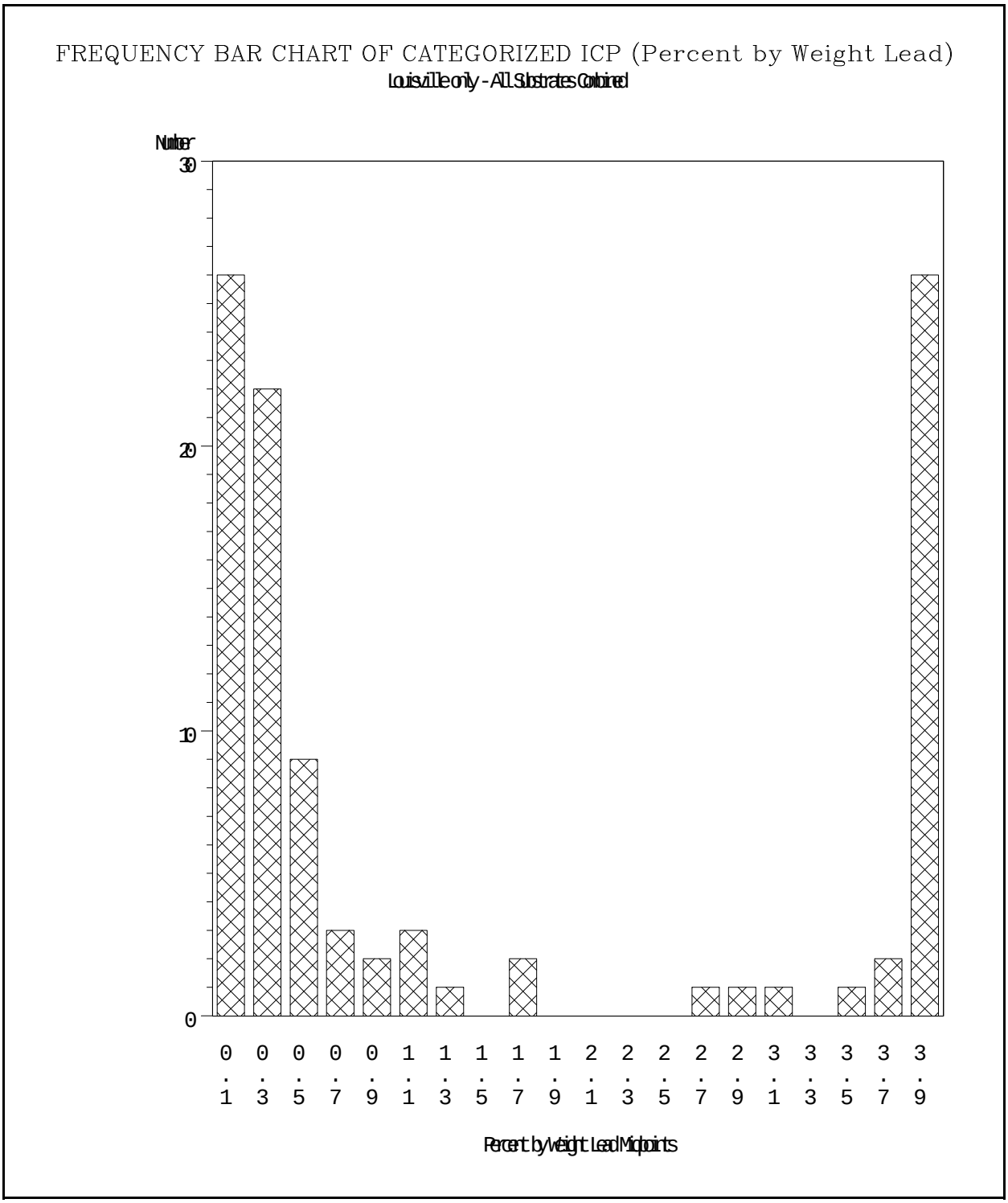

Figure 4-14. Frequency bar chart of primary ICP measurements (percent by weight lead) for all substrates combined in Louisville. A total of 47 (47.0%) measurements were greater than or equal to 0.5% lead.

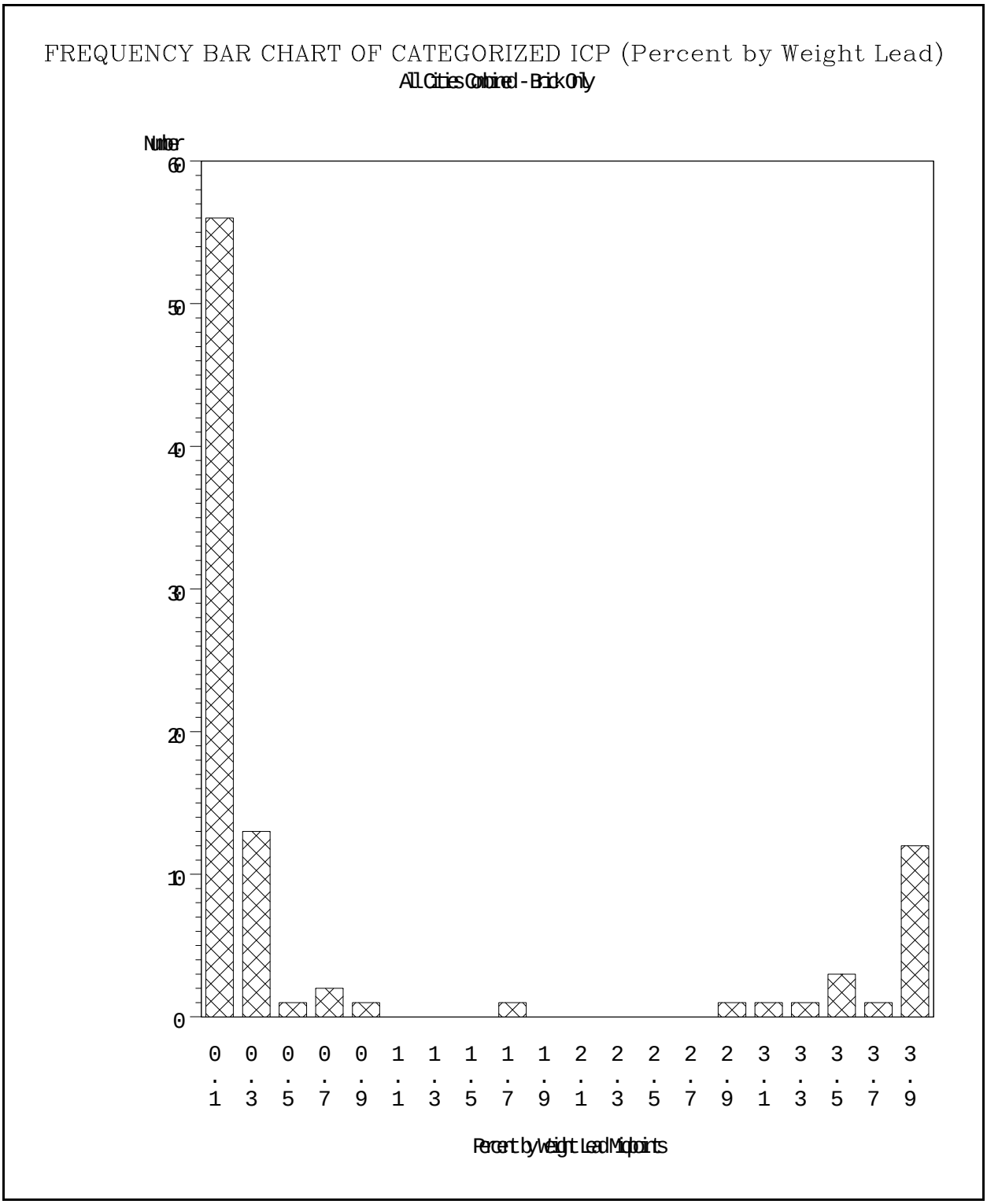

Figure 4-15. Frequency bar chart of primary ICP measurements (percent by weight lead) for brick in all cities. A total of 23 measurements (24.7%) were greater than or equal to 0.5% lead.

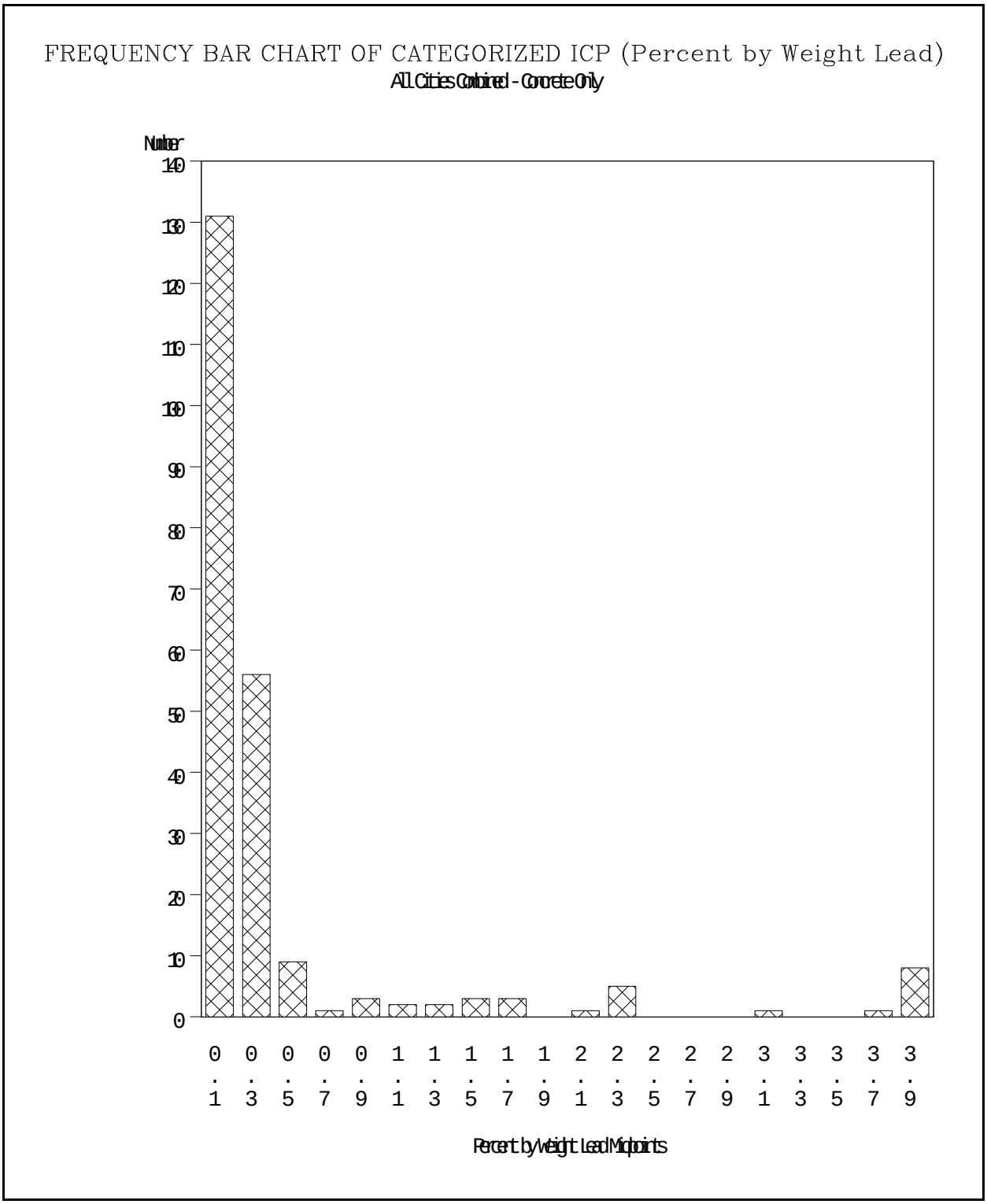

Figure 4-16. Frequency bar chart of primary ICP measurements (percent by weight lead) for concrete in all cities. A total of 33 measurements (14.6%) were greater than or equal 0.5% lead.

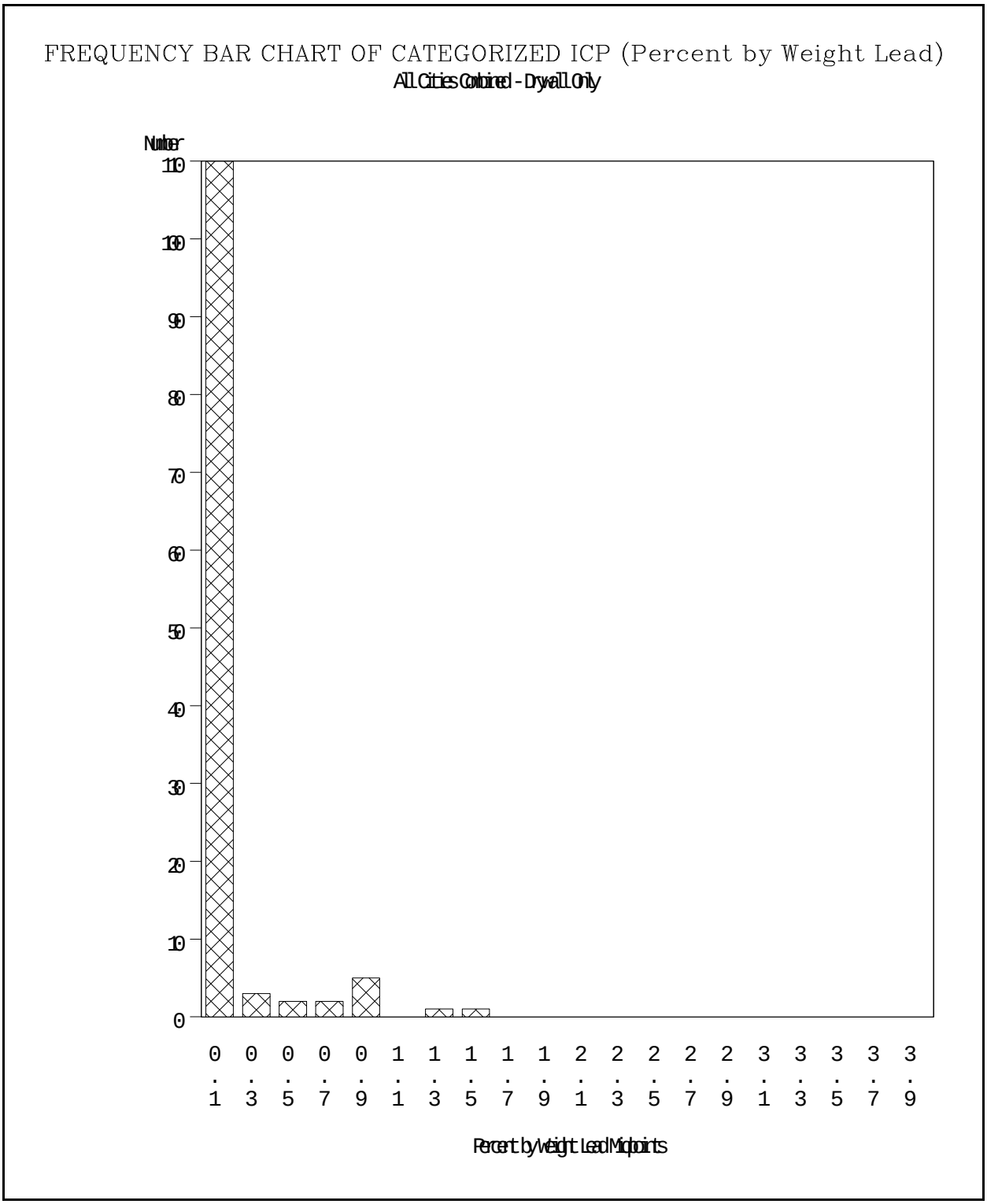

Figure 4-17. Frequency bar chart of primary ICP measurements (percent by weight lead) for drywall in all cities. A total of 10 measurements (8.1%) were greater than or equal to 0.5% lead.

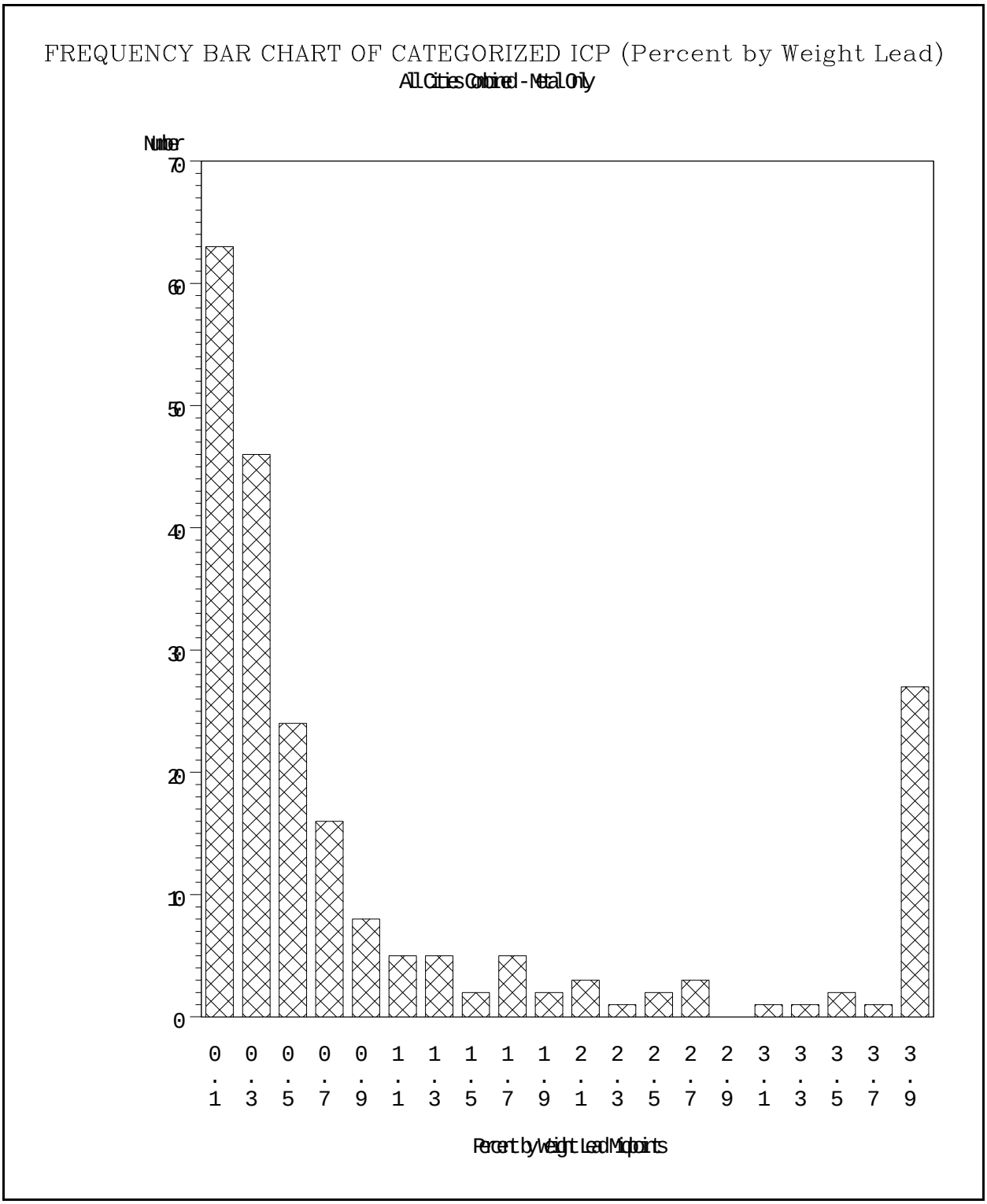

Figure 4-18. Frequency bar chart of primary ICP measurements (percent by weight lead) for metal in all cities. A total of 96 measurements (44.2%) were greater than or equal to 0.5% lead.

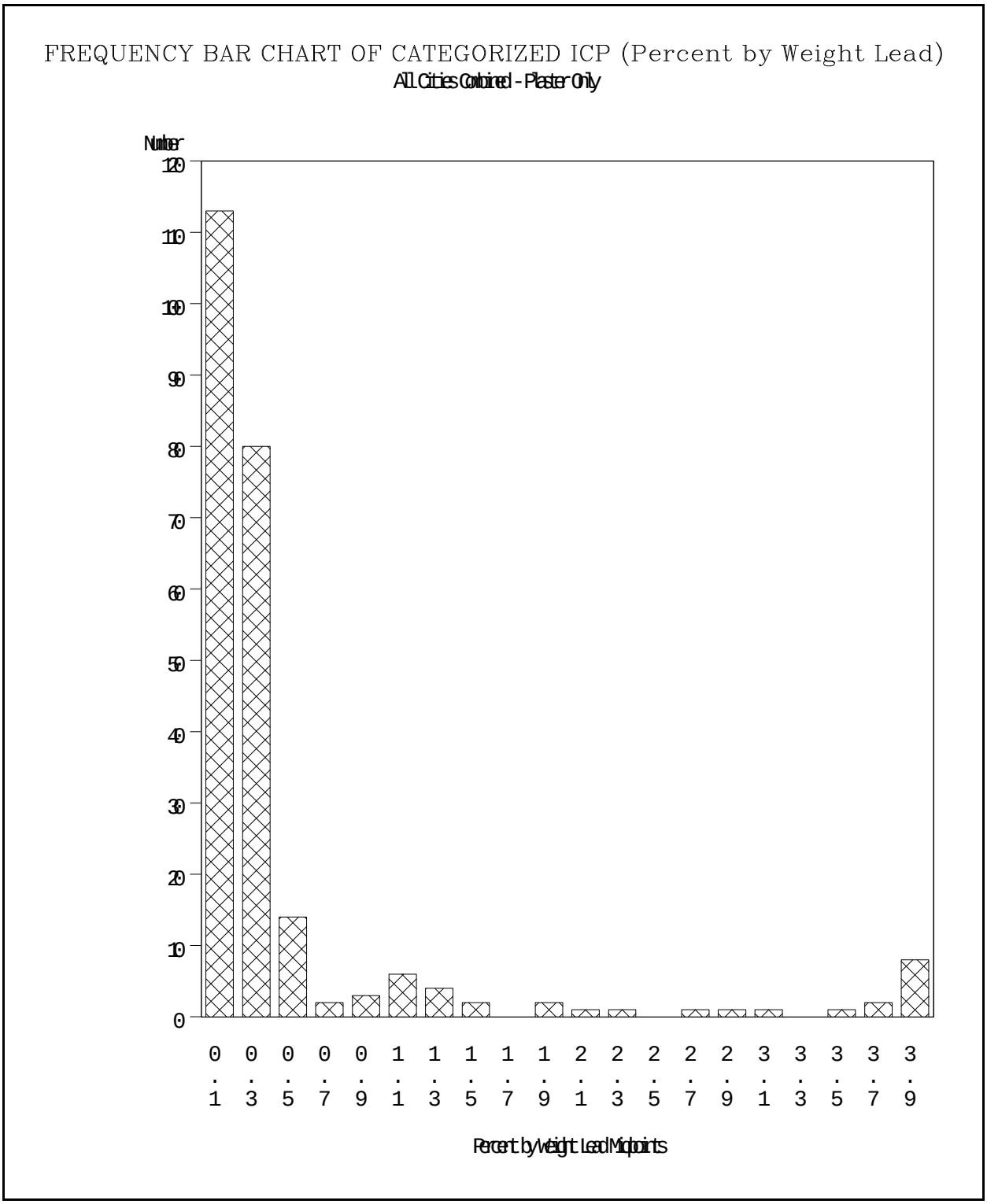

Figure 4-19. Frequency bar chart of primary ICP measurements (percent by weight lead) for plaster in all cities. A total of 39 measurements (16.1%) were greater than or equal to 0.5% lead.

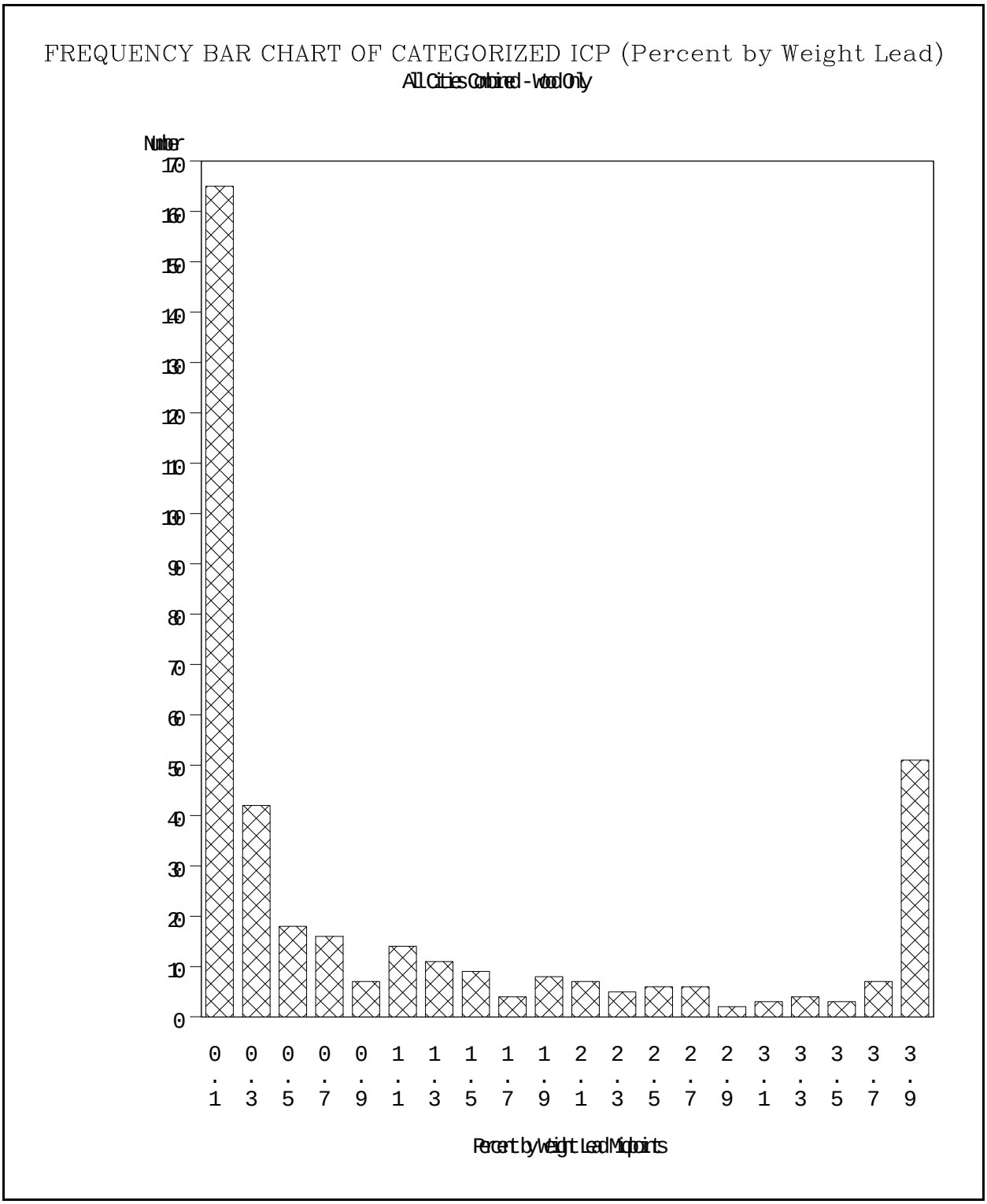

Figure 4-20. Frequency bar chart of primary ICP measurements (percent by weight lead) for wood in all cities. A total of 171 measurements (44.1%) were greater than or equal to 0.5% lead.

Data from Philadelphia and Louisville were comparable, except that Louisville data have a higher concentration of high levels of lead. The same is true for distributions by substrate, except that the distribution for brick appears bimodal.

Although the housing units tested in this study were not selected as a random sample of housing nationwide, they are free from identifiable biases. The tested locations represent a wide variety of paint types, substrates, and architectural designs. As a result, the distribution of lead levels in the paint samples is similar to that reported in the 1990 HUD National Survey of Lead-Based Paint in Housing. This suggests that the performance of XRFs and test kits on the study samples is representative of typical performance in real-world testing.

4.2 RELATIONSHIP BETWEEN AREA AND PERCENT BY WEIGHT UNITS

The relationship between paint lead levels expressed in area units (mg/cm^2) and percent by weight units is of interest as part of the overall study goal of collecting information needed to establish guidance and protocols for lead hazard identification and evaluation, since the federal standard for lead in paint has been expressed in both units (as 1.0 mg/cm^2 and as 0.5%). The present study provides a large database of 1,290 primary samples measured both ways. Within the study, there are no biases in the selection of samples to be measured in each unit - all samples are reported in both units. The objective of this section is to describe the relationship between the two types of units in this study, and to suggest some approximate conversion relationships between them.

The relationship between the two types of units is described by the following equation:

$$(mg/cm^2)/\% = (mg/cm^2)/(0.1 * mg/g) = 10 * (g/cm^2)$$

Thus, the relationship depends only on the area density of the paint sample measured in grams per square centimeter. In this study, all samples have the same area (to a very close approximation). Thus, the relationship between mg/cm^2 lead and percent by weight lead in this study depends only on the sample mass. The degree to which the ratio between the two types of units varies is therefore, for this study data, purely a function of the variation in the masses of the samples collected.

Table 4-10 shows the arithmetic mean ratio (mg/cm^2 lead)/(percent by weight lead) for the primary samples, by city

Table 4-10. Arithmetic Mean Ratio (mg/cm^2 lead)/(percent by weight lead) by City and Substrate.

SUBSTRATE	ALL CITIES	DENVER	PHILADELPHIA	LOUISVILLE
Brick	1.74	1.68	2.13	N/A
Concrete	1.35	1.36	1.38	0.85
Drywall	0.83	0.88	0.66	0.38
Metal	0.72	0.29	0.92	0.74
Plaster	1.30	1.19	1.46	0.87
Wood	0.66	0.63	0.87	0.59
Overall	1.00	0.92	1.22	0.68

and substrate and overall by substrate. The first point to note is that the overall average ratio is 1.00. Thus, as a simple rule of thumb, mg/cm^2 lead may be equated numerically to percent by weight lead based on these samples. However, the ratio varies widely over the samples. The 10th percentile is 0.25, while the 90th percentile is 2.34. Thus, in 80% of the samples, the conversion factor is between 0.25 and 2.34.

As Table 4-10 shows, even the arithmetic mean ratio varies substantially between cities and between substrates. Some of these differences are related to the difficulty of removing a paint-chip sample from the substrate, and the corresponding potential for including pieces of the substrate in the sample. Unless the substrate in the sample contains as much or more lead as the paint itself (an unlikely occurrence), the percent by weight values reported by the laboratory will be lower than if no substrate is included in the sample. However, substrate inclusion will generally have a much smaller effect on the reported mg/cm^2 values. Thus, samples which include substrate will have higher ratios (mg/cm^2 lead)/(percent lead) than those that do not. Brick and concrete samples were especially prone to substrate inclusion. It was often impossible to remove all the paint from these substrates without including parts of the substrate. Substrate inclusion was fairly common with soft plaster samples, particularly if the plaster was in poor condition. Substrate was sometimes included with wood and drywall samples, but this was less important since wood and drywall paper are much lighter than the other substrates.

Finally, clean samples were the rule with metal. Indeed, it was often not possible to remove factory-applied primer from metal components such as door frames.

The above observations are borne out by Table 4-10. Brick, concrete and plaster samples have the highest ratios of mg/cm^2 lead to percent by weight lead. Metal, wood and drywall have ratios less than 1.0 and are generally comparable. Two exceptions are metal in Denver and drywall in Louisville. The metal samples in Denver were in many cases taken from weathered exterior surfaces which were noted in the field to have thin paint, resulting in lighter samples. The sample masses taken from metal substrates in Denver ranged from 0.06 to 2.23 gram with a 0.75 gram mean compared to 0.21 to 7.31 gram with a 2.36 gram mean for Philadelphia and 0.67 to 4.36 gram with a 1.89 gram mean for Louisville. The drywall ratio for Louisville is low without apparent explanation.

Regression analyses [7] were conducted on the primary sample data to explore the utility of simple conversion formulas between the two types of units. Because of the highly skewed nature of the lead levels, a logarithmic transformation was applied to the data. The regression model is

$$log(PERCENT\ LEAD) = A + B * log(MG/CM^2\ LEAD)$$

where *PERCENT LEAD* is percent by weight lead and MG/CM^2 is mg/cm^2 lead.

Table 4-11 shows the regression coefficients and correlations for the model, both separately by substrate and overall. Although the model fits less well on metal than on the other substrates, a single overall model of the relationship may be used across all substrates. The relationship, converted back into the measurement scale, is

$$PERCENT\ LEAD = 0.96 * (MG/CM^2 LEAD)^{0.85}$$

where *PERCENT LEAD* is percent by weight lead and MG/CM^2 is mg/cm^2 lead.

For example, this formula predicts that a sample with 1.0 mg/cm^2 lead will have 0.96% lead by weight. A sample with 3.0 mg/cm^2 lead is predicted to have 2.44% lead by weight.

Conversely, a sample with 0.5% lead by weight is predicted to contain 0.46 mg/cm^2 lead.

Although the regression analysis indicates a good fit of these simple models to the study data, caution should still be

Table 4-11. Regression Coefficients and Correlations Measured in log(mg/cm^2) Units by Substrate and Overall.

SUBSTRATE	INTERCEPT	SLOPE	CORRELATION
Brick	-0.73	0.76	0.97
Concrete	-0.32	0.92	0.96
Drywall	-0.34	0.83	0.97
Metal	0.24	0.75	0.93
Plaster	-0.37	0.84	0.96
Wood	0.34	0.86	0.97
Overall	-0.04	0.85	0.95

exercised in using the resulting models for predictive purposes. First, there is considerable variation in the mass of paint samples, so that the relationship between the two types of units varies from sample to sample. Second, the relationship between the units estimated from the data in this study was influenced by the extent to which substrate was included in some samples.

4.3 VARIATION BETWEEN LABORATORY AND FIELD DUPLICATES

Analyses on the variation between laboratory and field duplicates presented below address the study objective to investigate the variability of lead levels in the paint within the sampling locations. These analyses also address the overall study goal to collect information about field measurement methodologies for use in establishing guidance and protocols for lead hazard identification and evaluation.

4.3.1 Variation Between Laboratory Duplicates

Total laboratory measurement variability is due to the combined effect of variability in subsampling prior to analysis, in the laboratory sample preparation and in the instrumental measurement. Total laboratory variability can be estimated by comparing measurements on a primary sample to those on the corresponding laboratory duplicate samples. Primary and duplicate samples were subsamples taken from the same homogenized physical sample and carried through the same extraction and analysis procedures. The assignment of primary versus duplicate within a pair was performed arbitrarily by the laboratory during

subsampling of the physical sample. In general, the first subsample taken was assigned as primary. Thus, the pair will often be referred to as a "(laboratory) duplicate pair". In this study, a total of 171 duplicate pairs were analyzed by the laboratory. Ten of these duplicate pairs (5.8%) were not used in estimating laboratory variability because at least one non-detectable[1] lead result was present in the pair. After eliminating these ten pairs, 161 duplicate pairs (78 from Denver, 44 from Philadelphia, and 39 from Louisville) were used for estimating laboratory variability. The laboratory duplicate data using mg/cm^2 results for these 161 pairs, sorted and presented in decreasing order according to the ratio of the larger lead result to the smaller lead result, is shown in Table 4-12. Table 4-13 is the companion table in percent by weight units. Note that the pairs eliminated from this analysis are not listed in the tables.

As shown in section 4.1 above, the distribution of lead levels in the study samples is highly skewed. Therefore, a logarithmic transformation was first applied to the data. Only area units (mg/cm^2 lead) were used in the estimation of variation between laboratory duplicates, since the variability in duplicate subsamples measured in percent by weight lead is the same. This can be seen as follows. Let AREA1 be the lead level in the primary subsample of a pair in mg/cm^2 and AREA2 the lead level in the duplicate subsample; let PERCENT1 and PERCENT2 be the corresponding percent by weight measurements. As shown in section 4.2,

AREA1/PERCENT1 = 10 x SAMPLE MASS/SAMPLE AREA = AREA2/PERCENT2

since both subsamples are taken from the <u>same</u> physical sample, that is, both subsamples have the same sample mass and sample area. Rearranging the above equation gives

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[1] A discussion on the determination of non-detectable status, as well as values used as non-detectable is presented in Section 4.4.1. As reported in Section 4.4.1, 4.2% (54 out of 1,290 samples) of the primary samples were reported as non-detectable. A pair of results is identified as non-detectable if the pair had at least one non-detectable result. Therefore, pairs of results will have a higher percentage of non-detectable results since there are two opportunities for the pair to be identified as non-detectable.

$$AREA2/AREA1 = PERCENT2/PERCENT1.$$

Thus, the ratio of lead levels in area units for the subsample pair is the same as the ratio of percent by weight units. Taking logarithms,

Table 4-12. Lead Levels in Laboratory Duplicate Samples in **Area Units (mg/cm²)**, Sorted by RATIO1.

SAMPLE ID	AREA1	AREA2	SUBSTRATE	CITY	LOG(AREA1)	LOG(AREA2)	RATIO1	RATIO2
905593	0.1359	0.1358	Plaster	Louisville	-1.99595	-1.99678	1.00084	0.99916
81441	0.2682	0.2674	Metal	Philadelphia	-1.31606	-1.31901	1.00295	0.99705
905598	3.5437	3.5329	Concrete	Louisville	1.26516	1.26211	1.00305	0.99696
905524	0.4315	0.4330	Plaster	Louisville	-0.84051	-0.83701	1.00351	1.00351
905501	11.6371	11.5957	Wood	Louisville	2.45420	2.45063	1.00357	0.99644
905527	0.3942	0.3927	Plaster	Louisville	-0.93091	-0.93461	1.00371	0.99630
905604	0.2233	0.2241	Metal	Louisville	-1.49942	-1.49551	1.00391	1.00391
905528	5.8033	5.7732	Plaster	Louisville	1.75844	1.75323	1.00522	0.99481
80314	2.0720	2.0612	Wood	Denver	0.72851	0.72330	1.00522	0.99481
905608	0.1686	0.1674	Metal	Louisville	-1.78021	-1.78712	1.00694	0.99311
905597	0.2368	0.2351	Concrete	Louisville	-1.44057	-1.44760	1.00706	0.99299
80510	2.2623	2.2788	Metal	Denver	0.81637	0.82366	1.00732	1.00732
80320	0.1893	0.1908	Wood	Denver	-1.66463	-1.65674	1.00793	1.00793
905607	0.7848	0.7913	Metal	Louisville	-0.24231	-0.23412	1.00823	1.00823
905506	11.2622	11.3707	Wood	Louisville	2.42145	2.43104	1.00963	1.00963
80071	1.3858	1.3713	Plaster	Denver	0.32626	0.31572	1.01060	0.98951
80947	0.0016	0.0016	Concrete	Denver	-6.44402	-6.45668	1.01274	0.98742
81208	0.7716	0.7829	Metal	Philadelphia	-0.25924	-0.24475	1.01459	1.01459
905524	0.4823	0.4752	Plaster	Louisville	-0.72913	-0.74407	1.01505	0.98518
80329	0.0725	0.0713	Wood	Denver	-2.62444	-2.64016	1.01584	0.98441
81958	0.2046	0.2079	Brick	Philadelphia	-1.58675	-1.57065	1.01623	1.01623
905600	0.5586	0.5475	Metal	Louisville	-0.58236	-0.60243	1.02028	0.98013
81756	0.5149	0.5037	Plaster	Philadelphia	-0.66382	-0.68583	1.02226	0.97823
80363	3.7741	3.6919	Concrete	Denver	1.32818	1.30613	1.02229	0.97820
80411	1.5483	1.5829	Wood	Denver	0.43718	0.45923	1.02230	1.02230
80958	0.1783	0.1742	Concrete	Denver	-1.72429	-1.74767	1.02365	0.97689
905540	0.4469	0.4579	Metal	Louisville	-0.80552	-0.78119	1.02463	1.02463
81846	0.1731	0.1775	Metal	Philadelphia	-1.75406	-1.72901	1.02537	1.02537
905548	4.0336	4.1458	Metal	Louisville	1.39467	1.42209	1.02781	1.02781
80724	20.5851	20.0229	Wood	Denver	3.02457	2.99688	1.02807	0.97269
80908	1.6670	1.7164	Brick	Denver	0.51100	0.54023	1.02967	1.02967
80208	21.4584	22.1231	Wood	Denver	3.06612	3.09662	1.03098	1.03098
905591	0.2174	0.2242	Plaster	Louisville	-1.52613	-1.49522	1.03139	1.03139
905535	0.9096	0.8814	Metal	Louisville	-0.09478	-0.12624	1.03195	0.96904
80572	0.7489	0.7250	Drywall	Denver	-0.28916	-0.32164	1.03301	0.96805
80741	27.2060	28.1517	Brick	Denver	3.30344	3.33761	1.03476	1.03476

AREA1 = Lead level in primary sample (mg/cm^2)	AREA2 = Lead level in lab duplicate sample (mg/cm^2)
RATIO1 = max(AREA1, AREA2) ÷ min(AREA1, AREA2)	RATIO2 = AREA1 ÷ AREA2

Table 4-12 (cont). Lead Levels in Laboratory Duplicate Samples in **Area Units (mg/cm²)**, Sorted by RATIO1.

SAMPLE ID	AREA1	AREA2	SUBSTRATE	CITY	LOG(AREA1)	LOG(AREA2)	RATIO1	RATIO2
905595	0.4329	0.4482	Concrete	Louisville	-0.83724	-0.80250	1.03534	1.03534
80547	1.8858	1.9535	Wood	Denver	0.63433	0.66962	1.03592	1.03592
905523	4.5359	4.7019	Plaster	Louisville	1.51202	1.54797	1.03661	1.03661
81944	0.0902	0.0936	Metal	Philadelphia	-2.40561	-2.36830	1.03802	1.03802
80022	0.2119	0.2202	Wood	Denver	-1.55178	-1.51326	1.03927	1.03927
81855	0.4976	0.4785	Metal	Philadelphia	-0.69800	-0.73712	1.03990	0.96163
81541	0.4928	0.5126	Metal	Philadelphia	-0.70761	-0.66822	1.04018	1.04018
81719	0.5900	0.6146	Metal	Philadelphia	-0.52762	-0.48686	1.04159	1.04159
81357	0.2474	0.2374	Metal	Philadelphia	-1.39683	-1.43801	1.04204	0.95966
80013	0.6314	0.6592	Wood	Denver	-0.45986	-0.41670	1.04411	1.04411
905512	10.9131	11.4086	Wood	Louisville	2.38996	2.43437	1.04541	1.04541
905531	0.2780	0.2908	Concrete	Louisville	-1.28014	-1.23499	1.04618	1.04618
905544	0.7229	0.7570	Metal	Louisville	-0.32444	-0.27836	1.04716	1.04716
81651	0.3393	0.3554	Concrete	Philadelphia	-1.08099	-1.03457	1.04752	1.04752
80628	5.0579	5.3035	Wood	Denver	1.62095	1.66836	1.04855	1.04855
80032	0.0751	0.0788	Wood	Denver	-2.58893	-2.54072	1.04940	1.04940
80272	0.1104	0.1050	Plaster	Denver	-2.20374	-2.25370	1.05123	0.95126
905501	8.5903	9.0399	Wood	Louisville	2.15063	2.20165	1.05234	1.05234
80169	0.1109	0.1167	Plaster	Denver	-2.19913	-2.14798	1.05248	1.05248
80979	0.0061	0.0064	Concrete	Denver	-5.10111	-5.04990	1.05255	1.05255
80479	0.2172	0.2294	Drywall	Denver	-1.52694	-1.47233	1.05612	1.05612
80909	0.2890	0.3058	Brick	Denver	-1.24133	-1.18489	1.05806	1.05806
80742	17.4958	18.5579	Brick	Denver	2.86196	2.92089	1.06071	1.06071
80845	0.3428	0.3224	Brick	Denver	-1.07046	-1.13187	1.06333	0.94044
80116	0.0623	0.0585	Wood	Denver	-2.77628	-2.83839	1.06408	0.93978
80117	0.1274	0.1196	Wood	Denver	-2.06011	-2.12352	1.06546	0.93856
81910	1.7460	1.6372	Concrete	Philadelphia	0.55730	0.49299	1.06642	0.93771
80209	30.1056	28.1478	Wood	Denver	3.40471	3.33747	1.06955	0.93497
80417	1.8030	1.6821	Wood	Denver	0.58947	0.52001	1.07193	0.93290
80270	0.1558	0.1452	Plaster	Denver	-1.85886	-1.92944	1.07313	0.93186
81510	0.4210	0.4518	Concrete	Philadelphia	-0.86519	-0.79458	1.07316	1.07316
80170	0.2620	0.2811	Plaster	Denver	-1.33956	-1.26890	1.07322	1.07322
81615	0.4212	0.4524	Plaster	Philadelphia	-0.86477	-0.79317	1.07423	1.07423
80468	0.2834	0.3056	Drywall	Denver	-1.26072	-1.18545	1.07818	1.07818
81225	1.2017	1.2965	Wood	Philadelphia	0.18370	0.25966	1.07892	1.07892
81905	0.0516	0.0477	Concrete	Philadelphia	-2.96501	-3.04199	1.08002	0.92591

AREA1 = Lead level in primary sample (mg/cm^2)	AREA2 = Lead level in lab duplicate sample (mg/cm^2)
RATIO1 = max(AREA1, AREA2) ÷ min(AREA1, AREA2)	RATIO2 = AREA1 ÷ AREA2

Table 4-12 (cont). Lead Levels in Laboratory Duplicate Samples in **Area Units (mg/cm²)**, Sorted by RATIO1.

SAMPLE ID	AREA1	AREA2	SUBSTRATE	CITY	LOG(AREA1)	LOG(AREA2)	RATIO1	RATIO2
80918	0.1942	0.1797	Brick	Denver	-1.63907	-1.71652	1.08053	0.92547
81920	2.6400	2.8559	Plaster	Philadelphia	0.97079	1.04939	1.08178	1.08178
905573	10.1643	11.0217	Wood	Louisville	2.31889	2.39987	1.08436	1.08436
905527	0.3096	0.3360	Plaster	Louisville	-1.17258	-1.09063	1.08540	1.08540
80274	0.1454	0.1578	Plaster	Denver	-1.92834	-1.84636	1.08543	1.08543
81624	2.2528	2.4476	Metal	Philadelphia	0.81219	0.89511	1.08646	1.08646
905590	4.8503	5.2775	Plaster	Louisville	1.57904	1.66346	1.08809	1.08809
81524	0.1758	0.1615	Plaster	Philadelphia	-1.73858	-1.82331	1.08843	0.91876
80158	0.2952	0.2711	Plaster	Denver	-1.22003	-1.30512	1.08881	0.91843
81708	0.2043	0.2225	Metal	Philadelphia	-1.58817	-1.50260	1.08933	1.08933
80765	15.9796	17.4298	Concrete	Denver	2.77132	2.85818	1.09075	1.09075
905521	4.1370	3.7861	Plaster	Louisville	1.41998	1.33134	1.09268	0.91518
80174	0.2166	0.2371	Plaster	Denver	-1.52966	-1.43944	1.09441	1.09441
81508	0.3487	0.3819	Concrete	Philadelphia	-1.05360	-0.96249	1.09539	1.09539
80565	0.9049	0.8230	Drywall	Denver	-0.09993	-0.19474	1.09945	0.90955
80212	20.5478	22.6199	Wood	Denver	3.02276	3.11883	1.10084	1.10084
80210	24.7695	22.4571	Wood	Denver	3.20961	3.11161	1.10297	0.90664
81931	0.1150	0.1271	Plaster	Philadelphia	-2.16300	-2.06310	1.10506	1.10506
81745	0.7352	0.6634	Plaster	Philadelphia	-0.30760	-0.41041	1.10828	0.90230
80416	3.6117	4.0057	Wood	Denver	1.28419	1.38773	1.10909	1.10909
81409	0.5719	0.6349	Concrete	Philadelphia	-0.55881	-0.45437	1.11009	1.11009
80461	0.1081	0.0967	Drywall	Denver	-2.22470	-2.33562	1.11731	0.89500
80114	0.0887	0.0992	Wood	Denver	-2.42204	-2.31062	1.11787	1.11787
81532	0.3455	0.3864	Plaster	Philadelphia	-1.06268	-0.95101	1.11814	1.11814
81835	0.2593	0.2312	Plaster	Philadelphia	-1.34981	-1.46434	1.12135	0.89178
80929	0.0160	0.0142	Drywall	Denver	-4.13767	-4.25240	1.12157	0.89160
80560	0.0487	0.0434	Drywall	Denver	-3.02126	-3.13637	1.12201	0.89126
80978	0.0094	0.0084	Concrete	Denver	-4.66280	-4.78310	1.12784	0.88665
80171	0.4075	0.3599	Plaster	Denver	-0.89776	-1.02182	1.13208	0.88333
81347	0.4648	0.4102	Plaster	Philadelphia	-0.76613	-0.89111	1.13313	0.88251
80225	14.6201	12.9019	Wood	Denver	2.68239	2.55737	1.13317	0.88248
905511	10.2236	11.6002	Wood	Louisville	2.32469	2.45102	1.13466	1.13466
905514	0.2103	0.1844	Wood	Louisville	-1.55943	-1.69084	1.14044	0.87686
80463	0.0595	0.0517	Drywall	Denver	-2.82144	-2.96288	1.15193	0.86811
81806	0.0096	0.0083	Brick	Philadelphia	-4.64599	-4.79633	1.16223	0.86042
80533	2.1287	1.8261	Wood	Denver	0.75551	0.60217	1.16572	0.85784

AREA1 = Lead level in primary sample mg/cm^2	AREA2 = Lead level in lab duplicate sample mg/cm^2
RATIO1 = max(AREA1, AREA2) ÷ min(AREA1, AREA2)	RATIO2 = AREA1 ÷ AREA2

Table 4-12 (cont). Lead Levels in Laboratory Duplicate Samples in **Area Units (mg/cm²)**, Sorted by RATIO1.

SAMPLE ID	AREA1	AREA2	SUBSTRATE	CITY	LOG(AREA1)	LOG(AREA2)	RATIO1	RATIO2
80444	0.1031	0.0881	Wood	Denver	-2.27167	-2.42962	1.17111	0.85389
80922	0.0429	0.0503	Brick	Denver	-3.14982	-2.98935	1.17406	1.17406
80464	0.1239	0.1455	Drywall	Denver	-2.08804	-1.92737	1.17429	1.17429
80721	13.0686	11.1193	Wood	Denver	2.57021	2.40869	1.17530	0.85085
80844	0.4949	0.4168	Brick	Denver	-0.70332	-0.87527	1.18762	0.84202
905587	3.9644	4.7166	Plaster	Louisville	1.37737	1.55108	1.18972	1.18972
81812	0.2410	0.2878	Concrete	Philadelphia	-1.42308	-1.24539	1.19446	1.19446
81824	0.2982	0.2492	Plaster	Philadelphia	-1.21006	-1.38942	1.19645	0.83580
80928	0.0058	0.0070	Drywall	Denver	-5.14131	-4.96042	1.19829	1.19829
80766	12.3937	10.2811	Plaster	Denver	2.51719	2.33031	1.20549	0.82954
80373	0.0056	0.0046	Plaster	Denver	-5.18499	-5.37736	1.21212	0.82500
80751	7.3083	6.0259	Concrete	Denver	1.98901	1.79606	1.21283	0.82452
905588	0.3448	0.2842	Plaster	Louisville	-1.06486	-1.25808	1.21314	0.82430
81724	0.7928	0.6514	Wood	Philadelphia	-0.23217	-0.42866	1.21712	0.82161
80271	0.3378	0.2760	Plaster	Denver	-1.08530	-1.28725	1.22378	0.81714
80051	0.0030	0.0025	Brick	Denver	-5.80250	-6.00759	1.22764	0.81457
81452	0.4689	0.5765	Wood	Philadelphia	-0.75728	-0.55073	1.22943	1.22943
80953	0.5879	0.4768	Concrete	Denver	-0.53127	-0.74066	1.23293	0.81108
81638	2.7076	2.1944	Metal	Philadelphia	0.99606	0.78592	1.23385	0.81047
905507	0.5713	0.4630	Wood	Louisville	-0.55981	-0.77011	1.23404	0.81034
80159	0.2330	0.2908	Plaster	Denver	-1.45680	-1.23526	1.24800	1.24800
80126	0.1246	0.0999	Wood	Denver	-2.08233	-2.30399	1.24815	0.80119
81607	0.5449	0.4350	Plaster	Philadelphia	-0.60719	-0.83243	1.25263	0.79832
905564	15.2450	12.0912	Wood	Louisville	2.72425	2.49248	1.26083	0.79313
81646	0.3130	0.3968	Concrete	Philadelphia	-1.16149	-0.92432	1.26765	1.26765
81213	3.6850	4.6756	Metal	Philadelphia	1.30428	1.54236	1.26882	1.26882
905545	0.4024	0.5155	Metal	Louisville	-0.91027	-0.66255	1.28110	1.28110
81557	0.3514	0.2727	Wood	Philadelphia	-1.04583	-1.29931	1.28850	0.77610
81425	1.5011	1.1636	Plaster	Philadelphia	0.40622	0.15148	1.29014	0.77511
81412	0.7936	1.0251	Concrete	Philadelphia	-0.23123	0.02481	1.29180	1.29180
80435	2.2876	1.7599	Wood	Denver	0.82750	0.56525	1.29985	0.76932
905605	0.2324	0.3033	Metal	Louisville	-1.45920	-1.19305	1.30493	1.30493
81335	0.4394	0.5858	Concrete	Philadelphia	-0.82244	-0.53469	1.33342	1.33342
81307	1.2500	1.6714	Wood	Philadelphia	0.22316	0.51368	1.33712	1.33712
81434	0.3569	0.2569	Plaster	Philadelphia	-1.03027	-1.35919	1.38946	0.71971
81318	0.0040	0.0028	Brick	Philadelphia	-5.52899	-5.86748	1.40283	0.71285

AREA1 = Lead level in primary sample (mg/cm^2)	AREA2 = Lead level in lab duplicate sample (mg/cm^2)
RATIO1 = max(AREA1, AREA2) ÷ min(AREA1, AREA2)	RATIO2 = AREA1 ÷ AREA2

Table 4-12 (cont). Lead Levels in Laboratory Duplicate Samples in **Area Units (mg/cm²)**, Sorted by RATIO1.

SAMPLE ID	AREA1	AREA2	SUBSTRATE	CITY	LOG(AREA1)	LOG(AREA2)	RATIO1	RATIO2
80957	0.1385	0.0965	Concrete	Denver	-1.97703	-2.33821	1.43503	0.69685
80948	0.0034	0.0024	Concrete	Denver	-5.68693	-6.05334	1.44255	0.69322
80346	0.0020	0.0014	Drywall	Denver	-6.19972	-6.57128	1.45000	0.68966
80519	3.1580	2.1483	Wood	Denver	1.14994	0.76466	1.47002	0.68026
80356	0.0154	0.0101	Concrete	Denver	-4.17404	-4.59621	1.52527	0.65562
905541	0.4684	0.7342	Metal	Louisville	-0.75852	-0.30896	1.56761	1.56761
80664	0.0808	0.0508	Drywall	Denver	-2.51590	-2.98065	1.59161	0.62830
80921	0.2943	0.1847	Brick	Denver	-1.22305	-1.68918	1.59382	0.62742
905592	3.02108	4.86975	Plaster	Louisville	1.10562	1.58304	1.61192	1.61192
905521	3.78884	2.30963	Plaster	Louisville	1.33206	0.83709	1.64045	0.60959
80964	0.00229	0.00379	Concrete	Denver	-6.07920	-5.57539	1.65502	1.65502
81255	0.05526	0.09514	Plaster	Philadelphia	-2.89571	-2.35241	1.72168	1.72168
80239	0.12735	0.21952	Wood	Denver	-2.06082	-1.51631	1.72375	1.72375
80917	0.03684	0.06983	Brick	Denver	-3.30117	-2.66169	1.89549	1.89549
80711	6.67299	3.49165	Wood	Denver	1.89807	1.25037	1.91113	0.52325
80050	0.00249	0.00119	Brick	Denver	-5.99547	-6.73380	2.09244	0.47791
905606	0.57670	1.85511	Metal	Louisville	-0.55044	0.61794	3.21678	3.21678

AREA1 = Lead level in primary sample (mg/cm²) AREA2 = Lead level in lab duplicate sample (mg/cm²)
RATIO1 = max(AREA1, AREA2) ÷ min(AREA1, AREA2) RATIO2 = AREA1 ÷ AREA2

Table 4-13. Lead Levels in Laboratory Duplicate Samples in **Percent by Weight Units**, Sorted by RATIO1.

SAMPLE ID	PERCENT1	PERCENT2	SUBSTRATE	CITY	LOG(PERCENT1)	LOG(PERCENT2)	RATIO1	RATIO2
80947	0.0008	0.0008	Concrete	Denver	-7.13090	-7.13090	1.00000	1.00000
905593	0.1324	0.1323	Plaster	Louisville	-2.02179	-2.02263	1.00084	0.99916
81441	0.1420	0.1416	Metal	Philadelphia	-1.95193	-1.95475	1.00282	0.99718
905598	5.2076	5.1917	Concrete	Louisville	1.65012	1.64707	1.00305	0.99696
905524	0.4258	0.4273	Plaster	Louisville	-0.85378	-0.85028	1.00351	1.00351
905501	11.1032	11.0637	Wood	Louisville	2.40723	2.40367	1.00357	0.99644
905527	0.4176	0.4161	Plaster	Louisville	-0.87319	-0.87690	1.00371	0.99630
905604	0.1945	0.1952	Metal	Louisville	-1.63741	-1.63350	1.00391	1.00391
80314	2.2774	2.2656	Wood	Denver	0.82303	0.81784	1.00521	0.99482
905528	4.8594	4.8342	Plaster	Louisville	1.58091	1.57571	1.00522	0.99481
905608	0.2215	0.2200	Metal	Louisville	-1.50728	-1.51419	1.00694	0.99311
905597	0.1870	0.1857	Concrete	Louisville	-1.67659	-1.68362	1.00706	0.99299
80510	3.0185	3.0406	Metal	Denver	1.10476	1.11205	1.00732	1.00732
80320	0.1405	0.1416	Wood	Denver	-1.96255	-1.95475	1.00783	1.00783
905607	0.8378	0.8446	Metal	Louisville	-0.17703	-0.16883	1.00823	1.00823
905506	14.1613	14.2977	Wood	Louisville	2.65051	2.66010	1.00963	1.00963
80071	1.0038	0.9932	Plaster	Denver	0.00379	-0.00682	1.01067	0.98944
81208	0.7188	0.7293	Metal	Philadelphia	-0.33017	-0.31567	1.01461	1.01461
905524	0.5180	0.5103	Plaster	Louisville	-0.65785	-0.67279	1.01505	0.98518
81958	0.0757	0.0769	Brick	Philadelphia	-2.58098	-2.56525	1.01585	1.01585
80329	0.0762	0.0750	Wood	Denver	-2.57439	-2.59027	1.01600	0.98425
905600	0.7269	0.7125	Metal	Louisville	-0.31891	-0.33899	1.02028	0.98013
81756	0.2625	0.2568	Plaster	Philadelphia	-1.33750	-1.35946	1.02220	0.97829
80411	1.4407	1.4728	Wood	Denver	0.36513	0.38717	1.02228	1.02228
80363	2.2235	2.1750	Concrete	Denver	0.79908	0.77703	1.02230	0.97819
80958	0.0870	0.0850	Concrete	Denver	-2.44185	-2.46510	1.02353	0.97701
905540	0.5088	0.5213	Metal	Louisville	-0.67573	-0.65140	1.02463	1.02463
81846	0.1202	0.1232	Metal	Philadelphia	-2.11860	-2.09395	1.02496	1.02496
905548	5.5140	5.6673	Metal	Louisville	1.70729	1.73471	1.02781	1.02781
80724	10.2034	9.9248	Wood	Denver	2.32272	2.29504	1.02807	0.97270
80908	0.9804	1.0095	Brick	Denver	-0.01979	0.00946	1.02968	1.02968
80208	22.5540	23.2526	Wood	Denver	3.11591	3.14642	1.03097	1.03097
905591	0.2381	0.2455	Plaster	Louisville	-1.43522	-1.40431	1.03139	1.03139
905535	1.1665	1.1304	Metal	Louisville	0.15402	0.12257	1.03195	0.96904
80572	0.5551	0.5374	Drywall	Denver	-0.58861	-0.62101	1.03294	0.96811

PERCENT1 = Lead level in primary sample (%)	PERCENT2 = Lead level in lab duplicate sample (%)
RATIO1 = max(PERCENT1, PERCENT2) ÷ min(PERCENT1, PERCENT2)	RATIO2 = PERCENT1 ÷ PERCENT2

Table 4-13 (cont). Lead Levels in Laboratory Duplicate Samples in **Percent by Weight Units**, Sorted by RATIO1.

SAMPLE ID	PERCENT1	PERCENT2	SUBSTRATE	CITY	LOG(PERCENT1)	LOG(PERCENT2)	RATIO1	RATIO2
80741	9.3293	9.6536	Brick	Denver	2.23316	2.26733	1.03476	1.03476
905595	0.5141	0.5323	Concrete	Louisville	-0.66535	-0.63062	1.03534	1.03534
80547	1.4193	1.4703	Wood	Denver	0.35016	0.38547	1.03593	1.03593
905523	3.6688	3.8031	Plaster	Louisville	1.29985	1.33581	1.03661	1.03661
81944	0.0726	0.0753	Metal	Philadelphia	-2.62279	-2.58628	1.03719	1.03719
80022	0.2370	0.2463	Wood	Denver	-1.43970	-1.40120	1.03924	1.03924
81855	0.3543	0.3407	Metal	Philadelphia	-1.03761	-1.07675	1.03992	0.96161
81541	0.3451	0.3590	Metal	Philadelphia	-1.06392	-1.02443	1.04028	1.04028
81719	0.2971	0.3095	Metal	Philadelphia	-1.21369	-1.17280	1.04174	1.04174
81357	0.2445	0.2347	Metal	Philadelphia	-1.40854	-1.44945	1.04176	0.95992
80013	0.5077	0.5301	Wood	Denver	-0.67786	-0.63469	1.04412	1.04412
905512	13.0290	13.6206	Wood	Louisville	2.56718	2.61158	1.04541	1.04541
905531	0.2603	0.2723	Concrete	Louisville	-1.34600	-1.30085	1.04618	1.04618
905544	0.8059	0.8439	Metal	Louisville	-0.21577	-0.16968	1.04716	1.04716
81651	0.2053	0.2150	Concrete	Philadelphia	-1.58328	-1.53712	1.04725	1.04725
80628	2.2156	2.3232	Wood	Denver	0.79552	0.84295	1.04856	1.04856
80032	0.0710	0.0745	Wood	Denver	-2.64508	-2.59696	1.04930	1.04930
80272	0.0934	0.0889	Plaster	Denver	-2.37086	-2.42024	1.05062	0.95182
905501	8.6911	9.1460	Wood	Louisville	2.16230	2.21332	1.05234	1.05234
80169	0.1345	0.1416	Plaster	Denver	-2.00619	-1.95475	1.05279	1.05279
80479	0.1056	0.1115	Drywall	Denver	-2.24810	-2.19373	1.05587	1.05587
80909	0.1480	0.1566	Brick	Denver	-1.91054	-1.85406	1.05811	1.05811
80742	4.6314	4.9126	Brick	Denver	1.53286	1.59180	1.06072	1.06072
80845	0.1972	0.1854	Brick	Denver	-1.62354	-1.68524	1.06365	0.94016
80116	0.0779	0.0732	Wood	Denver	-2.55233	-2.61456	1.06421	0.93967
80117	0.1299	0.1219	Wood	Denver	-2.04099	-2.10455	1.06563	0.93841
81910	1.0528	0.9872	Concrete	Philadelphia	0.05145	-0.01288	1.06645	0.93769
80209	34.1918	31.9683	Wood	Denver	3.53199	3.46474	1.06955	0.93497
80417	1.3102	1.2223	Wood	Denver	0.27018	0.20073	1.07191	0.93291
80170	0.1698	0.1822	Plaster	Denver	-1.77313	-1.70265	1.07303	1.07303
81510	0.2660	0.2855	Concrete	Philadelphia	-1.32426	-1.25351	1.07331	1.07331
80270	0.1303	0.1214	Plaster	Denver	-2.03792	-2.10866	1.07331	0.93170
80979	0.0027	0.0029	Concrete	Denver	-5.91450	-5.84304	1.07407	1.07407
81615	0.2516	0.2703	Plaster	Philadelphia	-1.37991	-1.30822	1.07432	1.07432
80468	0.1673	0.1803	Drywall	Denver	-1.78797	-1.71313	1.07770	1.07770

PERCENT1 = Lead level in primary sample (%)	PERCENT2 = Lead level in lab duplicate sample (%)
RATIO1 = max(PERCENT1, PERCENT2) ÷ min(PERCENT1, PERCENT2)	RATIO2 = PERCENT1 ÷ PERCENT2

Table 4-13 (cont). Lead Levels in Laboratory Duplicate Samples in **Percent by Weight Units**, Sorted by RATIO1.

SAMPLE ID	PERCENT1	PERCENT2	SUBSTRATE	CITY	LOG(PERCENT1)	LOG(PERCENT2)	RATIO1	RATIO2
81225	0.6774	0.7309	Wood	Philadelphia	-0.38949	-0.31348	1.07898	1.07898
80918	0.0854	0.0791	Brick	Denver	-2.46041	-2.53704	1.07965	0.92623
81905	0.0431	0.0399	Concrete	Philadelphia	-3.14423	-3.22138	1.08020	0.92575
81920	1.4449	1.5631	Plaster	Philadelphia	0.36804	0.44667	1.08180	1.08180
905573	12.5035	13.5582	Wood	Louisville	2.52601	2.60699	1.08436	1.08436
80274	0.1066	0.1157	Plaster	Denver	-2.23867	-2.15675	1.08537	1.08537
905527	0.3360	0.3647	Plaster	Louisville	-1.09069	-1.00874	1.08540	1.08540
81624	0.8334	0.9054	Metal	Philadelphia	-0.18224	-0.09938	1.08639	1.08639
905590	4.6386	5.0472	Plaster	Louisville	1.53440	1.61882	1.08809	1.08809
81524	0.1458	0.1339	Plaster	Philadelphia	-1.92552	-2.01066	1.08887	0.91838
80158	0.2702	0.2481	Plaster	Denver	-1.30859	-1.39392	1.08908	0.91821
81708	0.1344	0.1464	Metal	Philadelphia	-2.00693	-1.92141	1.08929	1.08929
80765	6.2109	6.7745	Concrete	Denver	1.82631	1.91317	1.09074	1.09074
905521	4.4742	4.0947	Plaster	Louisville	1.49833	1.40969	1.09268	0.91518
80174	0.2373	0.2598	Plaster	Denver	-1.43843	-1.34784	1.09482	1.09482
81508	0.2296	0.2515	Concrete	Philadelphia	-1.47142	-1.38031	1.09538	1.09538
80565	0.4129	0.3756	Drywall	Denver	-0.88455	-0.97923	1.09931	0.90966
80212	21.9811	24.1977	Wood	Denver	3.09018	3.18626	1.10084	1.10084
80210	22.8237	20.6929	Wood	Denver	3.12780	3.02979	1.10297	0.90664
81931	0.0735	0.0812	Plaster	Philadelphia	-2.61047	-2.51084	1.10476	1.10476
81745	0.3620	0.3266	Plaster	Philadelphia	-1.01611	-1.11902	1.10839	0.90221
80416	2.3522	2.6088	Wood	Denver	0.85535	0.95889	1.10909	1.10909
81409	0.2996	0.3326	Concrete	Philadelphia	-1.20531	-1.10081	1.11015	1.11015
80114	0.0843	0.0942	Wood	Denver	-2.47337	-2.36234	1.11744	1.11744
80461	0.0780	0.0698	Drywall	Denver	-2.55105	-2.66212	1.11748	0.89487
81532	0.1801	0.2014	Plaster	Philadelphia	-1.71424	-1.60246	1.11827	1.11827
81835	0.1536	0.1370	Plaster	Philadelphia	-1.87340	-1.98777	1.12117	0.89193
80929	0.0083	0.0074	Drywall	Denver	-4.79150	-4.90628	1.12162	0.89157
80560	0.0385	0.0343	Drywall	Denver	-3.25710	-3.37261	1.12245	0.89091
80978	0.0053	0.0047	Concrete	Denver	-5.24005	-5.36019	1.12766	0.88679
80171	0.2412	0.2130	Plaster	Denver	-1.42213	-1.54646	1.13239	0.88308
81347	0.3557	0.3139	Plaster	Philadelphia	-1.03367	-1.15868	1.13316	0.88249
80225	19.1533	16.9024	Wood	Denver	2.95248	2.82746	1.13317	0.88248
905511	12.6130	14.3114	Wood	Louisville	2.53472	2.66105	1.13466	1.13466
905514	0.2992	0.2623	Wood	Louisville	-1.20666	-1.33808	1.14044	0.87686

PERCENT1 = Lead level in primary sample (%)	PERCENT2 = Lead level in lab duplicate sample (%)
RATIO1 = max(PERCENT1, PERCENT2) ÷ min(PERCENT1, PERCENT2)	RATIO2 = PERCENT1 ÷ PERCENT2

Table 4-13 (cont). Lead Levels in Laboratory Duplicate Samples in **Percent by Weight Units**, Sorted by RATIO1.

SAMPLE ID	PERCENT1	PERCENT2	SUBSTRATE	CITY	LOG(PERCENT1)	LOG(PERCENT2)	RATIO1	RATIO2
81806	0.0039	0.0034	Brick	Philadelphia	-5.54678	-5.68398	1.14706	0.87179
80463	0.0301	0.0261	Drywall	Denver	-3.50323	-3.64582	1.15326	0.86711
80533	2.0857	1.7892	Wood	Denver	0.73510	0.58177	1.16572	0.85784
80444	0.0762	0.0651	Wood	Denver	-2.57439	-2.73183	1.17051	0.85433
80922	0.0190	0.0223	Brick	Denver	-3.96332	-3.80317	1.17368	1.17368
80464	0.1416	0.1663	Drywall	Denver	-1.95475	-1.79396	1.17444	1.17444
80721	6.6588	5.6656	Wood	Denver	1.89594	1.73441	1.17530	0.85084
80844	0.2566	0.2160	Brick	Denver	-1.36024	-1.53248	1.18796	0.84178
905587	3.6765	4.3740	Plaster	Louisville	1.30196	1.47568	1.18972	1.18972
81812	0.1246	0.1489	Concrete	Philadelphia	-2.08265	-1.90448	1.19502	1.19502
81824	0.1272	0.1063	Plaster	Philadelphia	-2.06199	-2.24149	1.19661	0.83569
80051	0.0030	0.0025	Brick	Denver	-5.80914	-5.99146	1.20000	0.83333
80766	3.5790	2.9689	Plaster	Denver	1.27508	1.08819	1.20550	0.82953
80373	0.0046	0.0038	Plaster	Denver	-5.38170	-5.57275	1.21053	0.82609
80751	2.3544	1.9412	Concrete	Denver	0.85629	0.66331	1.21286	0.82450
905588	0.3975	0.3277	Plaster	Louisville	-0.92249	-1.11570	1.21314	0.82430
80928	0.0037	0.0045	Drywall	Denver	-5.59942	-5.40368	1.21622	1.21622
81724	0.4636	0.3809	Wood	Philadelphia	-0.76873	-0.96522	1.21712	0.82161
80271	0.2852	0.2331	Plaster	Denver	-1.25456	-1.45629	1.22351	0.81732
81452	0.2853	0.3507	Wood	Philadelphia	-1.25421	-1.04782	1.22923	1.22923
80953	0.3572	0.2897	Concrete	Denver	-1.02946	-1.23891	1.23300	0.81103
81638	1.7495	1.4179	Metal	Philadelphia	0.55933	0.34918	1.23387	0.81046
905507	0.4656	0.3773	Wood	Louisville	-0.76453	-0.97483	1.23404	0.81034
80159	0.1490	0.1859	Plaster	Denver	-1.90381	-1.68255	1.24765	1.24765
80126	0.1337	0.1071	Wood	Denver	-2.01216	-2.23399	1.24837	0.80105
81607	0.2093	0.1670	Plaster	Philadelphia	-1.56399	-1.78976	1.25329	0.79790
905564	15.2842	12.1223	Wood	Louisville	2.72682	2.49505	1.26083	0.79313
81646	0.1314	0.1666	Concrete	Philadelphia	-2.02951	-1.79216	1.26788	1.26788
81213	2.4975	3.1689	Metal	Philadelphia	0.91529	1.15338	1.26883	1.26883
905545	0.3335	0.4272	Metal	Louisville	-1.09821	-0.85050	1.28110	1.28110
81557	0.2482	0.1926	Wood	Philadelphia	-1.39352	-1.64714	1.28868	0.77599
81425	0.6856	0.5314	Plaster	Philadelphia	-0.37746	-0.63224	1.29018	0.77509
81412	0.3643	0.4705	Concrete	Philadelphia	-1.00978	-0.75396	1.29152	1.29152
80435	2.0611	1.5856	Wood	Denver	0.72324	0.46096	1.29989	0.76930
905605	0.1426	0.1861	Metal	Louisville	-1.94747	-1.68132	1.30493	1.30493

PERCENT1 = Lead level in primary sample (%)	PERCENT2 = Lead level in lab duplicate sample (%)
RATIO1 = max(PERCENT1, PERCENT2) ÷ min(PERCENT1, PERCENT2)	RATIO2 = PERCENT1 ÷ PERCENT2

Table 4-13 (cont). Lead Levels in Laboratory Duplicate Samples in **Percent by Weight Units**, Sorted by RATIO1.

SAMPLE ID	PERCENT1	PERCENT2	SUBSTRATE	CITY	LOG(PERCENT1)	LOG(PERCENT2)	RATIO1	RATIO2
81335	0.2577	0.3436	Concrete	Philadelphia	-1.35596	-1.06828	1.33333	1.33333
81307	1.0086	1.3486	Wood	Philadelphia	0.00856	0.29907	1.33710	1.33710
80948	0.0018	0.0013	Concrete	Denver	-6.31997	-6.64539	1.38462	0.72222
81434	0.1253	0.0902	Plaster	Philadelphia	-2.07704	-2.40573	1.38914	0.71987
80346	0.0021	0.0015	Drywall	Denver	-6.16582	-6.50229	1.40000	0.71429
81318	0.0017	0.0012	Brick	Philadelphia	-6.37713	-6.72543	1.41667	0.70588
80957	0.0391	0.0272	Concrete	Denver	-3.24163	-3.60454	1.43750	0.69565
80519	2.0474	1.3928	Wood	Denver	0.71657	0.33132	1.46999	0.68028
80356	0.0081	0.0053	Concrete	Denver	-4.81589	-5.24005	1.52830	0.65432
905541	0.5246	0.8224	Metal	Louisville	-0.64511	-0.19555	1.56761	1.56761
80664	0.0490	0.0308	Drywall	Denver	-3.01593	-3.48024	1.59091	0.62857
80921	0.1558	0.0978	Brick	Denver	-1.85918	-2.32483	1.59305	0.62773
905592	2.41634	3.89495	Plaster	Louisville	0.88225	1.35968	1.61192	1.61192
905521	3.74638	2.28375	Plaster	Louisville	1.32079	0.82582	1.64045	0.60959
80964	0.00090	0.00150	Concrete	Denver	-7.01312	-6.50229	1.66667	1.66667
81255	0.01770	0.03050	Plaster	Philadelphia	-4.03419	-3.49003	1.72316	1.72316
80239	0.19550	0.33710	Wood	Denver	-1.63219	-1.08738	1.72430	1.72430
80917	0.01610	0.03050	Brick	Denver	-4.12894	-3.49003	1.89441	1.89441
80711	3.53050	1.84730	Wood	Denver	1.26144	0.61373	1.91117	0.52324
80050	0.00310	0.00150	Brick	Denver	-5.77635	-6.50229	2.06667	0.48387
905606	0.64994	2.09072	Metal	Louisville	-0.43087	0.73751	3.21678	3.21678

PERCENT1 = Lead level in primary sample (%)
PERCENT2 = Lead level in lab duplicate sample (%)
RATIO1 = max(PERCENT1, PERCENT2) ÷ min(PERCENT1, PERCENT2)
RATIO2 = PERCENT1 ÷ PERCENT2

$$LOG(AREA2) - LOG(AREA1) = LOG(PERCENT2) - LOG(PERCENT1),$$

which shows that the variation between the pairs is identical in the two sets of units when a logarithmic transformation is applied.

The first step in the analysis was to fit a regression with LOG(AREA1) as the independent variable and LOG(AREA2) as the dependent variable. The regression was performed separately by city. Table 4-14 shows the correlations and regression coefficients obtained.

The regressions are very close to a perfect fit of LOG(AREA2) = LOG(AREA1) and the correlations are high. Figures 4-21 through 4-23 show the residual plots from the regressions. The residual plots indicate little dependence of variability on the level of lead in the sample. There are two possible outliers in the residual plots, one in Philadelphia and one in Louisville. The Philadelphia point has AREA1 = 0.05526 mg/cm^2, AREA2 = 0.09514 mg/cm^2 and ratio 1.72; the substrate for the sample was plaster. The Louisville point has AREA1 = 0.5767 mg/cm^2, AREA2 = 1.85511 mg/cm^2 and ratio 3.22; the substrate was metal. These data points will be examined later in this section after development of a statistical model and outlier criterion.

The residual plots suggest the following simple model for measurement variability between laboratory subsamples:

$$LOG(AREAi) = LOG(LEAD) + e_i$$

where lead is the true lead level in the sample and the errors e_i are independent with normal distribution $N(0,F^2)$. The variance F^2 is independent of lead as indicated by the residual plots. It follows from the model that

$$LOG(AREA2) - LOG(AREA1) = N(0,2F^2).$$

Hence, $s^2 = [LOG(AREA2) - LOG(AREA1)]^2/2$ is an unbiased estimator of F^2 and s^2/F^2 has a chi-square distribution with 1 degree of freedom. An overall estimator of F^2 can therefore be obtained by taking the mean value of s^2 over all sample pairs in the city. However, before calculating this estimate it is appropriate to undertake an outlier analysis because the residual plots indicate two possible outliers.

A simple outlier criterion can be developed as follows. For a fixed city, let N be the number of subsample pairs, s_i^2 the

Table 4-14. Correlations and Regression Coefficients for Regression of LOG(AREA2) Against LOG(AREA1) by City.

CITY	N	CORRELATION	SLOPE	INTERCEPT
Denver	78	0.997	1.003	0.056
Philadelphia	44	0.991	1.020	0.032
Louisville	39	0.987	0.984	0.056

estimated variances for the pairs, i = 1,...,N, and F^2 the true variance. A value s_i^2 will be considered an outlier if s_i^2/F^2 exceeds the 95th percentile for the maximum of a sample of size N from the chi-square distribution with 1 degree of freedom, denoted M(1,N,95). Replacing the unknown value F^2 by its estimate

$$s_{av}^2 = (s_1^2 + \ldots + s_N^2)/N,$$

the outlier cutoff becomes M(1,N,95) * s_{av}^2. Table 4-15 shows the results of the outlier analysis and the revised values of s_{av}^2 and s_{av} (its square root) computed after excluding the outliers, if any.

Thus, of the two potential outliers suggested by the regression residual plots, only one, in Louisville, fails the outlier criterion. This pair has a primary measurement equal to 0.5767 mg/cm^2 lead and a duplicate equal to 1.85511 mg/cm^2 lead.

Laboratory measurement variability is statistically significantly higher for the Denver samples than for those from Philadelphia or Louisville, based on an F-test at the 5% significance level. This may be related to the greater variety of substrates, both in terms of the underlying material and its condition, encountered in Denver. For example, there is more substrate inclusion in some Denver samples than was observed in collected samples for the other two cities, and this could lead to difficulty in homogenization resulting in greater variability between subsamples. Table 4-16 shows estimated standard deviations for laboratory duplicates in the three cities by substrate, with associated sample sizes. It is clear that the greater variability in Denver is attributable to the brick and concrete substrates; material from these substrates was often included in the paint-chip samples due to the rough surfaces

encountered in Denver. Data from brick and concrete samples in Philadelphia and Louisville were less variable. A major reason for this was the smooth poured concrete that predominated in

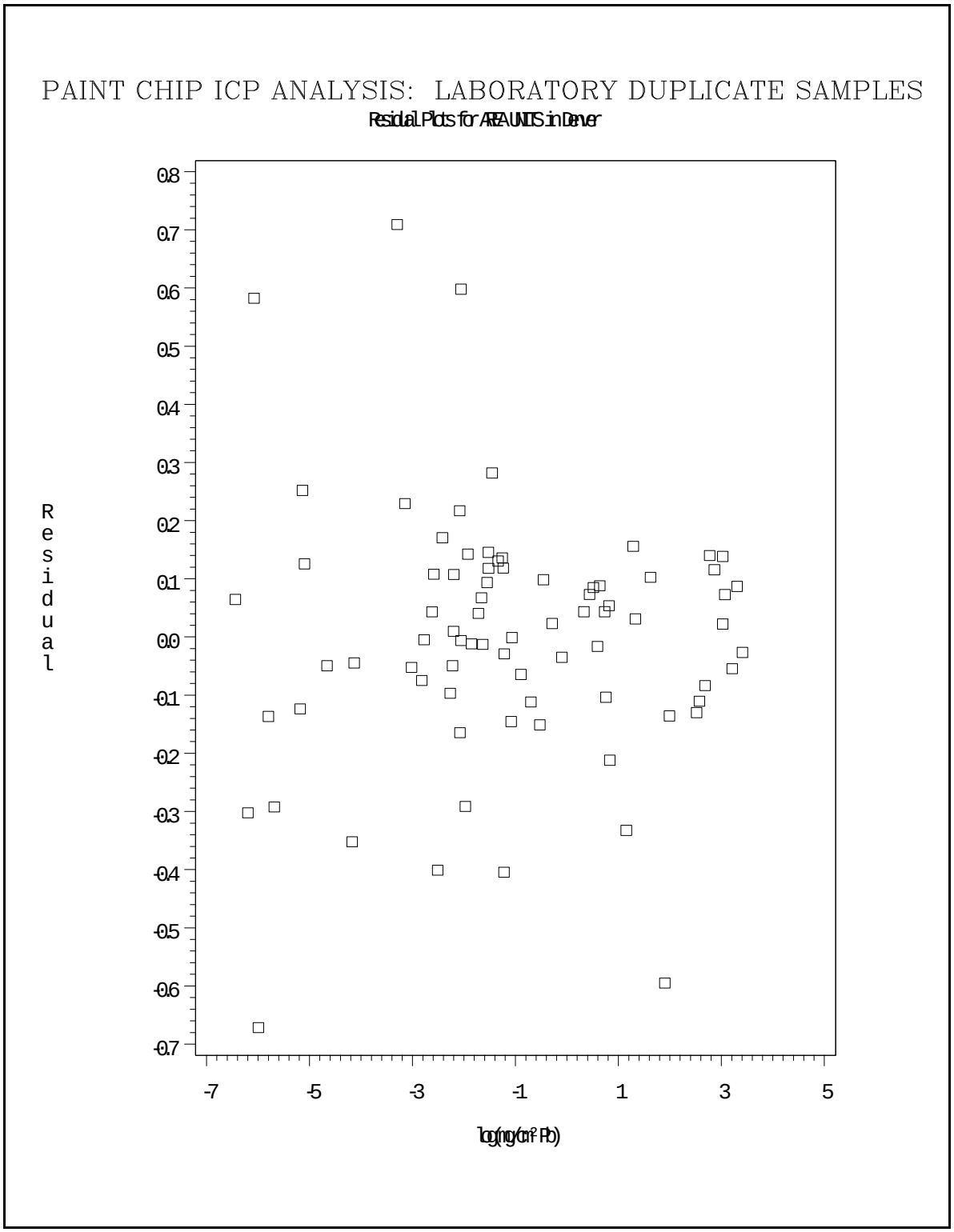

Figure 4-21 Plot of residuals from regression of log(lab duplicate) versus log(primary sample) in Denver (mg/cm^2 Pb).

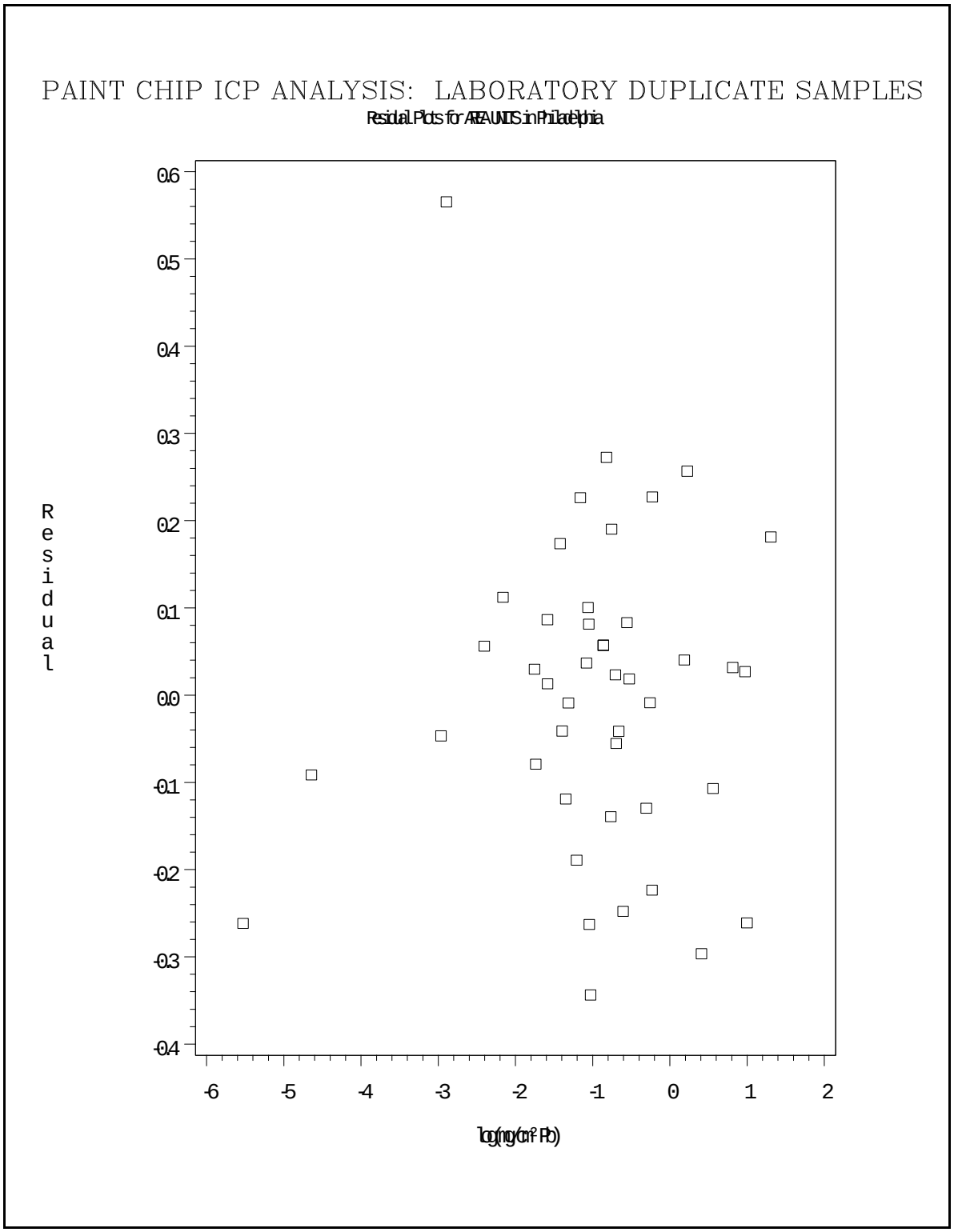

Figure 4-22. Plot of residuals from regression of log(lab duplicate) versus log(primary sample) in Philadelphia (mg/cm² Pb).

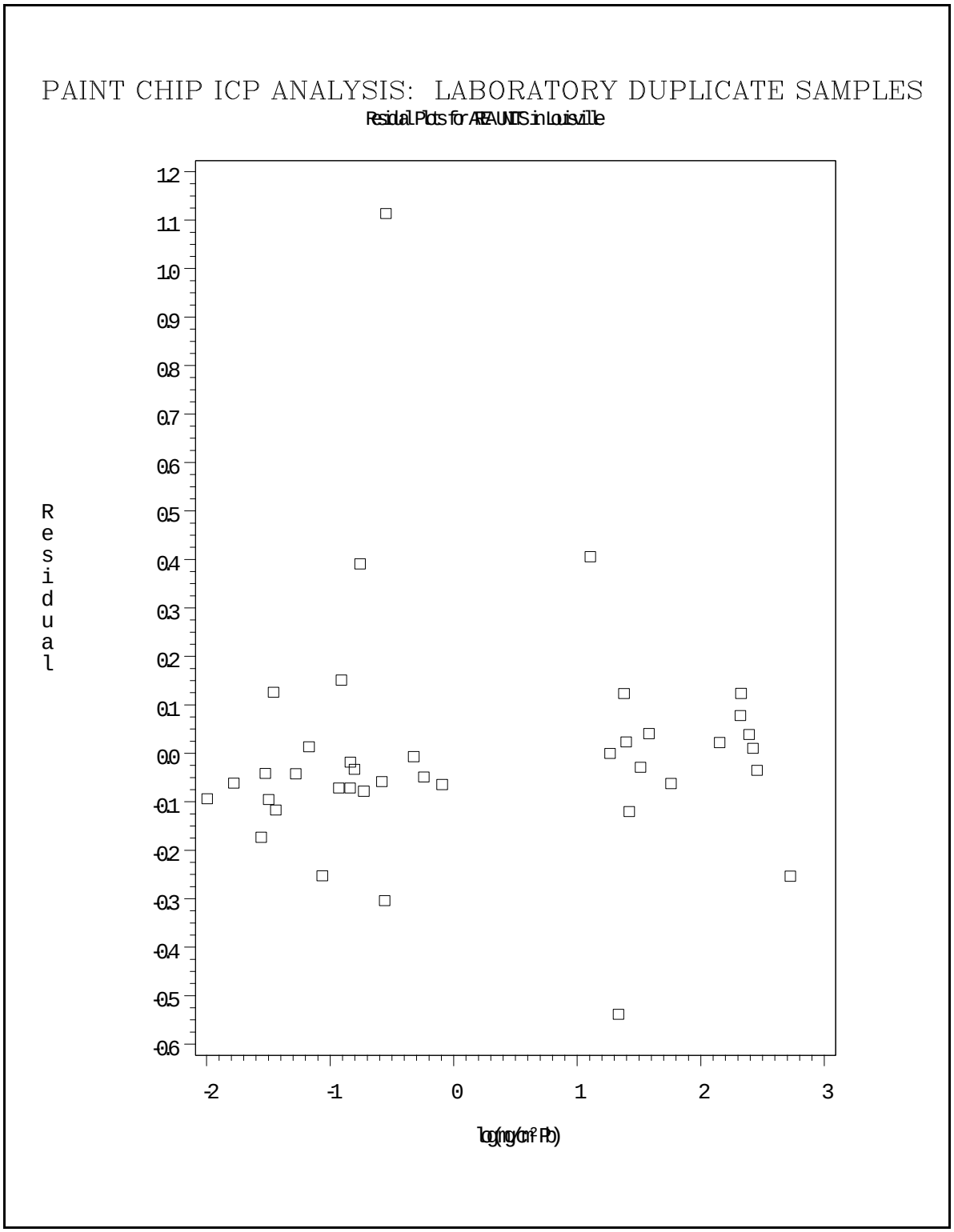

Figure 4-23. Plot of residuals from regression of log(lab duplicate) versus log(primary sample) in Louisville (mg/cm² Pb).

Table 4-15. Outlier Analysis for Laboratory Duplicate Data for mg/cm^2 Lead on the Log Scale.

	DENVER	PHILADELPHIA	LOUISVILLE
N	78	44	39
s_{av}^2	0.0252	0.0159	0.0310
M(1,N,95)	11.62	10.56	10.34
Outlier Cutoff	0.2934	0.1682	0.3201
Outlier Values	None	None	0.6826
Revised s_{av}^2	0.0252	0.0159	0.013811
Revised s_{av}	0.1589	0.1262	0.1175

Table 4-16. Estimated Standard Deviations for mg/cm^2 Lead on the Log Scale for Laboratory Measurement Variability by Substrate and City with Associated Sample Sizes (Outliers Excluded).

SUBSTRATE	DENVER			PHILADELPHIA			LOUISVILLE		
	N[a]	s_{av}	95% Interval[b]	N[a]	s_{av}	95% Interval[b]	N[a]	s_{av}	95% Interval[b]
Brick	12	0.2316	0.526, 1.900	3	0.1513	0.658, 1.521	0	N/A	N/A
Concrete	12	0.1867	0.596, 1.678	10	0.1167	0.724, 1.382	4	0.0203	0.945, 1.058
Drywall	12	0.1429	0.673, 1.486	0	N/A	N/A	0	N/A	N/A
Metal	1	0.0052	0.986, 1.015	12	0.0718	0.820, 1.220	11	0.1242	0.709, 1.411
Plaster	13	0.0908	0.778, 1.286	14	0.1582	0.660, 1.516	14	0.1420	0.675, 1.482
Wood	28	0.1429	0.673, 1.486	5	0.1535	0.660, 1.530	9	0.0890	0.781, 1.280
All	78	0.1596	0.643, 1.556	44	0.1262	0.705, 1.419	38	0.1175	0.722, 1.385

[a] N represents the number of paired results.
[b] A 95% probability interval for the ratio of laboratory duplicates under the assumption the standard deviation is the true value.

these cities by contrast to the rough concrete block that was common in Denver.

In Denver, the subsampling variability in data from rough substrates such as brick (0.2316 mg/cm^2 lead, on the log scale) or concrete (0.1867 mg/cm^2 lead on the log scale) may be up to twice as large as for the smoother surfaces (for example, 0.0908 mg/cm^2 lead on the log scale for plaster data). If the Denver brick and concrete pairs are excluded from the analysis, the average standard deviation in Denver is reduced to 0.1296 mg/cm^2 lead which is a result similar to what was found for Philadelphia and Louisville data. Aggregating across all three cities, the estimate of standard deviation on the log scale for laboratory duplicate samples is 0.125 mg/cm^2 lead for samples taken from smooth substrates with no unusual difficulty in paint removal.

A standard deviation of 0.125 on the log scale translates to a standard deviation factor of exp(0.125) = 1.13 on the original measurement scale in mg/cm^2 lead. Thus, using the normal distribution, 68% of laboratory measurements will be within a factor of 1.13 (one standard deviation) of the true lead level in the sample, that is, will be between 88% and 113% of the true value. Likewise, 95% of the measurements will be within a factor of 1.28 (two standard deviations) of the true value, that is, between 78% and 128% of the true value.

4.3.2 Variation Between Field Duplicates

Field duplicate paint samples were taken in all three cities in order to estimate the variability to be expected between samples taken a short distance apart. The variability in such samples is due to the combined effect of laboratory measurement variability and variation in the true lead level between the two samples of the pair. Thus, field

duplicate variability is expected to be greater than laboratory duplicate variability to the extent that the true lead level differs between the primary and field duplicate samples.

In Louisville, a field duplicate was taken at all 100 sampling locations. The primary sample and the field duplicate were true side-by-side samples, with a distance between centers averaging just over 2 inches (the paint samples were 2" x 2" squares). In Louisville, the assignment of primary versus duplicate within a field duplicate pair was arbitrarily performed in the field during collection. The first sample collected was assigned as primary. A total of 17 pairs out of 100 were eliminated for the variability analysis: eleven pairs where a collection or analytical problem was encountered for at least one of the samples and the six pairs where at least one of the samples was non-detectable. After eliminating the seventeen pairs, a total of 83 pairs remained.

In Denver and Philadelphia, the design called for a field duplicate at 10% of the sampling locations. Depending on the orientation of the marking template, the field duplicate sample was taken at the right end or bottom of the marking template. Thus, these field duplicates were not true side-by-side samples. The distance between centers of the primary and field duplicate samples averaged just over 9 inches. Ten pairs were eliminated from the variability analysis where at least one of the samples was non-detectable, six from Denver and four from Philadelphia. After eliminating the ten pairs, a total of 74 field duplicates remained in Denver, with 44 in Philadelphia. The field duplicate sample data using mg/cm^2 results, sorted and presented in decreasing order according to the ratio of the larger lead result to the smaller lead result, is presented in Table 4-17. Table 4-18 is the companion table in percent by weight units. Note that the pairs eliminated from the variability analysis are not listed in these tables.

The same approach to analysis of field duplicate variability was followed as for the laboratory duplicates. However, the variability between pairs is no longer the same for both sets of units, because the two analyses were conducted on different physical samples. Thus, all the analyses must be carried out in both units. Letting AREA1 and AREA2 be the lead levels in mg/cm^2 in the primary and field duplicate samples, respectively, and PERCENT1 and PERCENT2 the corresponding measurements in percent

by weight, regressions of LOG(AREA2) versus LOG(AREA1) and LOG(PERCENT2) versus LOG(PERCENT1) were carried out by city. Tables 4-19 and 4-20 show the results.

Comparing Table 4-19 to 4-14 shows that for Denver and Philadelphia, the correlations for the field duplicate pairs are lower than for the laboratory duplicates. This indicates greater variation between field duplicates than between laboratory duplicates in these cities. For Louisville, field duplicate and laboratory duplicate correlations are comparable. Figures 4-24 through 4-26 show the residual plots from the LOG(AREA) regressions; Figures 4-27 through 4-29 show the corresponding plots for the LOG(PERCENT) regressions. Both Denver plots appear to have constant variance; there is a single outlier in each. This point has primary lead levels of 0.25281 mg/cm^2 and 0.484%, and field duplicate levels of 0.00427 mg/cm^2 and 0.008%. Thus,

Table 4-17. Lead Levels in Field Duplicate Samples in **Area Units (mg/cm²)**, Sorted by RATIO1.

SAMPLE ID	AREA1	AREA2	SUBSTRATE	CITY	LOG(AREA1)	LOG(AREA2)	RATIO1	RATIO2
905549	0.1280	0.1276	Metal	Louisville	-2.05601	-2.05863	1.00262	0.99738
905512	10.9131	10.8747	Wood	Louisville	2.38996	2.38643	1.00353	0.99648
81448	0.4329	0.4350	Metal	Philadelphia	-0.83736	-0.83241	1.00497	1.00497
905608	0.1686	0.1673	Metal	Louisville	-1.78021	-1.78785	1.00767	0.99239
905568	2.5539	2.5767	Wood	Louisville	0.93763	0.94650	1.00891	1.00891
80924	0.0214	0.0216	Drywall	Denver	-3.84483	-3.83321	1.01169	1.01169
905565	8.6914	8.5857	Wood	Louisville	2.16234	2.15010	1.01231	0.98784
80808	0.0693	0.0683	Metal	Denver	-2.66974	-2.68414	1.01450	0.98571
905567	0.1209	0.1227	Wood	Louisville	-2.11318	-2.09779	1.01551	1.01551
905526	0.1840	0.1812	Plaster	Louisville	-1.69262	-1.70840	1.01591	0.98434
80646	0.0718	0.0730	Wood	Denver	-2.63429	-2.61771	1.01672	1.01672
80617	2.1921	2.1539	Wood	Denver	0.78485	0.76730	1.01770	0.98261
81930	0.0737	0.0751	Plaster	Philadelphia	-2.60748	-2.58933	1.01831	1.01831
905588	0.3381	0.3448	Plaster	Louisville	-1.08427	-1.06486	1.01960	1.01960
89257	0.2757	0.2823	Plaster	Denver	-1.28833	-1.26493	1.02368	1.02368
905597	0.2301	0.2368	Concrete	Louisville	-1.46928	-1.44057	1.02913	1.02913
905573	10.1643	9.8715	Wood	Louisville	2.31889	2.28965	1.02966	0.97119
905547	1.0502	1.0852	Metal	Louisville	0.04903	0.08179	1.03330	1.03330
80558	0.0203	0.0197	Drywall	Denver	-3.89566	-3.92917	1.03408	0.96704
905600	0.5586	0.5785	Metal	Louisville	-0.58236	-0.54728	1.03571	1.03571
905502	5.4967	5.7013	Wood	Louisville	1.70414	1.74070	1.03723	1.03723
905541	0.4515	0.4684	Metal	Louisville	-0.79527	-0.75852	1.03744	1.03744
905585	0.3200	0.3082	Plaster	Louisville	-1.13937	-1.17692	1.03827	0.96314
80433	2.1001	2.1869	Wood	Denver	0.74197	0.78250	1.04136	1.04136
905591	0.2084	0.2174	Plaster	Louisville	-1.56853	-1.52613	1.04331	1.04331
905537	2.8439	2.9747	Metal	Louisville	1.04517	1.09015	1.04601	1.04601
80549	0.0045	0.0043	Wood	Denver	-5.39924	-5.44450	1.04630	0.95575
905589	0.1335	0.1398	Plaster	Louisville	-2.01345	-1.96787	1.04663	1.04663
81654	0.2898	0.3042	Concrete	Philadelphia	-1.23843	-1.19004	1.04958	1.04958
81439	0.3243	0.3405	Metal	Philadelphia	-1.12599	-1.07731	1.04989	1.04989
80108	0.9288	0.8833	Metal	Denver	-0.07381	-0.12410	1.05158	0.95095
80141	1.7464	1.6601	Wood	Denver	0.55758	0.50687	1.05202	0.95055
81858	0.1639	0.1736	Wood	Philadelphia	-1.80832	-1.75112	1.05887	1.05887
80358	0.2170	0.2299	Concrete	Denver	-1.52772	-1.47007	1.05935	1.05935
80654	0.0933	0.0988	Drywall	Denver	-2.37236	-2.31456	1.05951	1.05951

AREA1 = Lead level in primary sample (mg/cm^2)	AREA2 = Lead level in field duplicate sample (mg/cm^2)
RATIO1 = max(AREA1, AREA2) ÷ min(AREA1, AREA2)	RATIO2 = AREA1 ÷ AREA2

Table 4-17 (cont). Lead Levels in Field Duplicate Samples in **Area Units (mg/cm²)**, Sorted by RATIO1.

SAMPLE ID	AREA1	AREA2	SUBSTRATE	CITY	LOG(AREA1)	LOG(AREA2)	RATIO1	RATIO2
905542	0.5319	0.5882	Metal	Louisville	-0.63125	-0.53075	1.10573	1.10573
905592	3.3425	3.0211	Plaster	Louisville	1.20671	1.10562	1.10638	0.90384
81355	0.0036	0.0032	Metal	Philadelphia	-5.62960	-5.73218	1.10802	0.90251
905576	2.2281	2.0067	Wood	Louisville	0.80114	0.69649	1.11033	0.90064
80230	0.0006	0.0005	Wood	Denver	-7.41858	-7.52394	1.11111	0.90000
81239	0.0821	0.0738	Concrete	Philadelphia	-2.50018	-2.60667	1.11236	0.89899
905538	0.18482	0.16607	Metal	Louisville	-1.68837	-1.79535	1.11291	0.89855
905524	0.48233	0.43149	Plaster	Louisville	-0.72913	-0.84051	1.11782	0.89460
905598	3.54366	3.16010	Concrete	Louisville	1.26516	1.15060	1.12138	0.89176
905507	0.64107	0.57132	Wood	Louisville	-0.44461	-0.55981	1.12210	0.89119
80638	0.11112	0.12485	Wood	Denver	-2.19714	-2.08064	1.12356	1.12356
80465	0.06414	0.07214	Drywall	Denver	-2.74669	-2.62915	1.12473	1.12473
905601	2.24057	2.55055	Metal	Louisville	0.80673	0.93631	1.13835	1.13835
905602	0.14263	0.16258	Metal	Louisville	-1.94752	-1.81657	1.13992	1.13992
905522	0.31963	0.27994	Plaster	Louisville	-1.14060	-1.27318	1.14177	0.87583
81523	0.23460	0.26799	Plaster	Philadelphia	-1.44987	-1.31681	1.14233	1.14233
80020	0.10639	0.09312	Wood	Denver	-2.24064	-2.37387	1.14250	0.87527
80106	2.44430	2.79365	Metal	Denver	0.89376	1.02735	1.14292	1.14292
905586	0.25590	0.29327	Plaster	Louisville	-1.36297	-1.22666	1.14603	1.14603
80170	0.26196	0.30027	Plaster	Denver	-1.33956	-1.20307	1.14624	1.14624
905603	0.16660	0.14508	Metal	Louisville	-1.79213	-1.93046	1.14835	0.87081
905546	0.13569	0.15666	Metal	Louisville	-1.99736	-1.85368	1.15452	1.15452
80514	0.58779	0.50853	Wood	Denver	-0.53139	-0.67623	1.15586	0.86516
81850	0.16726	0.14428	Metal	Philadelphia	-1.78821	-1.93600	1.15927	0.86261
905584	4.81985	5.59499	Plaster	Louisville	1.57274	1.72187	1.16082	1.16082
80277	0.08184	0.07044	Plaster	Denver	-2.50299	-2.65299	1.16184	0.86070
81607	0.54488	0.46672	Plaster	Philadelphia	-0.60719	-0.76203	1.16747	0.85656
905604	0.19115	0.22326	Metal	Louisville	-1.65469	-1.49942	1.16798	1.16798
80756	5.13793	6.00396	Concrete	Denver	1.63665	1.79242	1.16856	1.16856
81337	0.29806	0.34923	Plaster	Philadelphia	-1.21046	-1.05202	1.17168	1.17168
81625	0.32528	0.38191	Metal	Philadelphia	-1.12307	-0.96257	1.17410	1.17410
905595	0.43291	0.36861	Concrete	Louisville	-0.83724	-0.99802	1.17444	0.85147
81307	1.25002	1.47425	Wood	Philadelphia	0.22316	0.38815	1.17938	1.17938
80059	0.00276	0.00326	Concrete	Denver	-5.89252	-5.72603	1.18116	1.18116
80852	0.00319	0.00270	Brick	Denver	-5.74773	-5.91450	1.18148	0.84639

AREA1 = Lead level in primary sample (mg/cm^2)	AREA2 = Lead level in field duplicate sample (mg/cm^2)
RATIO1 = max(AREA1, AREA2) ÷ min(AREA1, AREA2)	RATIO2 = AREA1 ÷ AREA2

Table 4-17 (cont). Lead Levels in Field Duplicate Samples in **Area Units (mg/cm²)**, Sorted by RATIO1.

SAMPLE ID	AREA1	AREA2	SUBSTRATE	CITY	LOG(AREA1)	LOG(AREA2)	RATIO1	RATIO2
80371	0.00369	0.00436	Plaster	Denver	-5.60213	-5.43528	1.18157	1.18157
80469	0.06450	0.07626	Drywall	Denver	-2.74109	-2.57361	1.18233	1.18233
80623	0.62523	0.52811	Wood	Denver	-0.46964	-0.63845	1.18390	0.84467
80027	0.01192	0.01005	Wood	Denver	-4.42954	-4.60018	1.18607	0.84312
80439	0.06747	0.08012	Wood	Denver	-2.69607	-2.52423	1.18749	1.18749
81342	0.23624	0.28190	Plaster	Philadelphia	-1.44291	-1.26620	1.19328	1.19328
80314	2.07199	1.73582	Wood	Denver	0.72851	0.55148	1.19367	0.83776
905536	0.15195	0.12649	Metal	Louisville	-1.88420	-2.06762	1.20132	0.83242
80305	0.00158	0.00190	Metal	Denver	-6.45033	-6.26590	1.20253	1.20253
81546	0.4273	0.3540	Metal	Philadelphia	-0.85018	-1.03832	1.20700	0.82850
905563	7.0143	5.7846	Wood	Louisville	1.94795	1.75521	1.21257	0.82469
905539	0.9650	1.1738	Metal	Louisville	-0.03559	0.16022	1.21630	1.21630
905572	7.4964	6.1568	Wood	Louisville	2.01442	1.81756	1.21758	0.82130
905523	5.5332	4.5359	Plaster	Louisville	1.71077	1.51202	1.21988	0.81976
905575	2.1093	2.5786	Wood	Louisville	0.74634	0.94725	1.22251	1.22251
905530	0.0570	0.0699	Concrete	Louisville	-2.86494	-2.66000	1.22746	1.22746
905574	0.0707	0.0871	Wood	Louisville	-2.64974	-2.44084	1.23231	1.23231
905606	0.7117	0.5767	Metal	Louisville	-0.34016	-0.55044	1.23403	0.81036
905540	0.4469	0.5519	Metal	Louisville	-0.80552	-0.59441	1.23505	1.23505
81753	0.5627	0.4548	Plaster	Philadelphia	-0.57497	-0.78792	1.23732	0.80820
80664	0.0808	0.1001	Drywall	Denver	-2.51590	-2.30179	1.23877	1.23877
905587	4.9268	3.9644	Plaster	Louisville	1.59470	1.37737	1.24276	0.80466
80679	0.0730	0.0582	Concrete	Denver	-2.61757	-2.84421	1.25438	0.79720
905562	2.5559	2.0331	Wood	Louisville	0.93842	0.70954	1.25719	0.79542
905544	0.7229	0.5747	Metal	Louisville	-0.32444	-0.55388	1.25790	0.79498
905571	4.1944	3.3166	Wood	Louisville	1.43374	1.19893	1.26467	0.79072
905527	0.3942	0.3096	Plaster	Louisville	-0.93091	-1.17258	1.27338	0.78531
80153	0.0543	0.0698	Wood	Denver	-2.91249	-2.66269	1.28377	1.28377
905505	4.0864	5.3111	Wood	Louisville	1.40766	1.66980	1.29971	1.29971
905548	4.0336	3.0794	Metal	Louisville	1.39467	1.12474	1.30987	0.76344
905566	1.7807	2.3335	Wood	Louisville	0.57701	0.84736	1.31043	1.31043
81921	1.4944	1.1313	Plaster	Philadelphia	0.40175	0.12340	1.32095	0.75703
80531	0.1810	0.2401	Wood	Denver	-1.70898	-1.42670	1.32615	1.32615
905609	0.2443	0.1829	Metal	Louisville	-1.40925	-1.69897	1.33605	0.74848
905501	11.6371	8.5903	Wood	Louisville	2.45420	2.15063	1.35468	0.73818

AREA1 = Lead level in primary sample (mg/cm^2)	AREA2 = Lead level in field duplicate sample (mg/cm^2)
RATIO1 = max(AREA1, AREA2) ÷ min(AREA1, AREA2)	RATIO2 = AREA1 ÷ AREA2

Table 4-17 (cont). Lead Levels in Field Duplicate Samples in **Area Units (mg/cm²)**, Sorted by RATIO1.

SAMPLE ID	AREA1	AREA2	SUBSTRATE	CITY	LOG(AREA1)	LOG(AREA2)	RATIO1	RATIO2
80733	15.0949	20.5565	Brick	Denver	2.71436	3.02318	1.36181	1.36181
81413	1.6797	2.2994	Concrete	Philadelphia	0.51863	0.83264	1.36891	1.36891
81556	1.0628	0.7739	Wood	Philadelphia	0.06086	-0.25635	1.37329	0.72818
80409	0.0084	0.0060	Metal	Denver	-4.78191	-5.10770	1.38512	0.72196
905528	4.1785	5.8033	Plaster	Louisville	1.42995	1.75844	1.38887	1.38887
80507	0.7255	1.0088	Metal	Denver	-0.32087	0.00876	1.39045	1.39045
81458	0.7466	1.0403	Wood	Philadelphia	-0.29219	0.03952	1.39334	1.39334
905520	0.0013	0.0009	Drywall	Louisville	-6.66746	-7.01202	1.41138	0.70853
80545	0.0014	0.0010	Wood	Denver	-6.59294	-6.93821	1.41237	0.70803
81207	3.7589	2.6586	Metal	Philadelphia	1.32413	0.97779	1.41389	0.70727
905596	2.5343	1.7879	Concrete	Louisville	0.92993	0.58102	1.41752	0.70546
81711	0.2668	0.3790	Metal	Philadelphia	-1.32141	-0.97009	1.42094	1.42094
81957	0.8408	0.5846	Wood	Philadelphia	-0.17339	-0.53674	1.43814	0.69534
905515	6.8721	4.7577	Wood	Louisville	1.92747	1.55976	1.44442	0.69232
905590	3.3508	4.8503	Plaster	Louisville	1.20919	1.57904	1.44752	1.44752
80157	0.2329	0.3393	Plaster	Denver	-1.45693	-1.08096	1.45641	1.45641
80827	0.0017	0.0012	Wood	Denver	-6.37126	-6.75075	1.46154	0.68421
81712	0.5893	0.8786	Metal	Philadelphia	-0.52877	-0.12945	1.49081	1.49081
81513	0.3082	0.4623	Concrete	Philadelphia	-1.17688	-0.77143	1.49997	1.49997
81721	1.0578	0.7040	Wood	Philadelphia	0.05621	-0.35103	1.50267	0.66548
81629	2.4184	1.5863	Metal	Philadelphia	0.88310	0.46138	1.52459	0.65592
905599	0.2255	0.3454	Metal	Louisville	-1.48928	-1.06305	1.53147	1.53147
905517	0.0004	0.0006	Drywall	Louisville	-7.85787	-7.42725	1.53821	1.53821
81917	0.1809	0.2784	Concrete	Philadelphia	-1.70992	-1.27855	1.53936	1.53936
80818	0.9102	1.4872	Wood	Denver	-0.09408	0.39686	1.63385	1.63385
81849	0.1813	0.1093	Metal	Philadelphia	-1.70782	-2.21338	1.65792	0.60317
80353	0.0306	0.0180	Drywall	Denver	-3.48643	-4.01961	1.70434	0.58674
80578	0.0068	0.0040	Concrete	Denver	-4.98936	-5.53404	1.72405	0.58003
81939	0.9014	0.5224	Metal	Philadelphia	-0.10382	-0.64932	1.72548	0.57955
905518	0.0005	0.0003	Drywall	Louisville	-7.64087	-8.19313	1.73717	0.57565
905569	0.0001	0.0002	Wood	Louisville	-9.00527	-8.40896	1.81541	1.81541
81812	0.2410	0.4409	Concrete	Philadelphia	-1.42308	-0.81903	1.82952	1.82952
905605	0.4326	0.2324	Metal	Louisville	-0.83788	-1.45920	1.86138	0.53723
80140	1.5719	2.9895	Wood	Denver	0.45227	1.09510	1.90184	1.90184
80762	3.9185	2.0349	Concrete	Denver	1.36572	0.71043	1.92569	0.51929

AREA1 = Lead level in primary sample (mg/cm^2)	AREA2 = Lead level in field duplicate sample (mg/cm^2)
RATIO1 = max(AREA1, AREA2) ÷ min(AREA1, AREA2)	RATIO2 = AREA1 ÷ AREA2

Table 4-17 (cont). Lead Levels in Field Duplicate Samples in **Area Units (mg/cm²)**, Sorted by RATIO1.

SAMPLE ID	AREA1	AREA2	SUBSTRATE	CITY	LOG(AREA1)	LOG(AREA2)	RATIO1	RATIO2
80721	13.0686	25.2705	Wood	Denver	2.57021	3.22964	1.93369	1.93369
80831	0.8303	1.6325	Wood	Denver	-0.18599	0.49013	1.96624	1.96624
81211	2.5598	5.0550	Metal	Philadelphia	0.93991	1.62038	1.97479	1.97479
81315	0.1903	0.3892	Wood	Philadelphia	-1.65931	-0.94356	2.04572	2.04572
905607	0.3825	0.7848	Metal	Louisville	-0.96113	-0.24231	2.05201	2.05201
80035	0.0002	0.0005	Brick	Denver	-8.46840	-7.70626	2.14286	2.14286
81221	1.5256	0.7094	Wood	Philadelphia	0.42236	-0.34332	2.15046	0.46502
80971	0.2784	0.6388	Concrete	Denver	-1.27855	-0.44812	2.29432	2.29432
80848	0.3621	0.8402	Brick	Denver	-1.01583	-0.17414	2.32030	2.32030
81244	0.8357	1.9393	Concrete	Philadelphia	-0.17953	0.66233	2.32068	2.32068
80745	7.7036	19.0541	Brick	Denver	2.04168	2.94728	2.47341	2.47341
905577	0.0009	0.0003	Drywall	Louisville	-7.05633	-8.02761	2.64133	0.37860
80720	1.0439	0.3846	Wood	Denver	0.04294	-0.95553	2.71413	0.36844
80972	4.5535	1.5380	Concrete	Denver	1.51589	0.43046	2.96071	0.33776
81640	0.0032	0.0010	Wood	Philadelphia	-5.76035	-6.85897	3.00000	0.33333
80965	0.00217	0.0070	Concrete	Denver	-6.13303	-4.96758	3.2074	3.20737
80870	0.00623	0.0220	Plaster	Denver	-5.07838	-3.81580	3.5345	3.53451
80449	0.08402	0.2990	Wood	Denver	-2.47670	-1.20738	3.5584	3.55844
80937	0.00077	0.0032	Concrete	Denver	-7.16912	-5.73218	4.2078	4.20779
80875	0.00154	0.0071	Plaster	Denver	-6.47597	-4.94344	4.6299	4.62987
80908	1.66695	0.3115	Brick	Denver	0.51100	-1.16636	5.3514	0.18687
80206	2.07227	11.8282	Metal	Denver	0.72864	2.47049	5.7079	5.70785
80129	0.09266	0.5466	Wood	Denver	-2.37882	-0.60400	5.8992	5.89920
81544	0.06291	0.3841	Metal	Philadelphia	-2.76605	-0.95677	6.1060	6.10602
80229	0.01145	0.0011	Wood	Denver	-4.46977	-6.78554	10.1327	0.09869
80266	0.25281	0.0043	Plaster	Denver	-1.37512	-5.45614	59.2061	0.01689

AREA1 = Lead level in primary sample (mg/cm²) AREA2 = Lead level in field duplicate sample (mg/cm²)
RATIO1 = max(AREA1, AREA2) ÷ min(AREA1, AREA2) RATIO2 = AREA1 ÷ AREA2

Table 4-18. Lead Levels in Field Duplicate Samples in **Percent by Weight Units**, Sorted by RATIO1.

SAMPLE ID	PERCENT1	PERCENT2	SUBSTRATE	CITY	LOG(PERCENT1)	LOG(PERCENT2)	RATIO1	RATIO2
905565	13.6155	13.5834	Wood	Louisville	2.61121	2.60885	1.00237	0.99764
905543	0.0116	0.0115	Metal	Louisville	-4.46071	-4.46437	1.00367	0.99634
80623	0.6611	0.6643	Wood	Denver	-0.41385	-0.40902	1.00484	1.00484
81427	0.3493	0.3511	Plaster	Philadelphia	-1.05182	-1.04668	1.00515	1.00515
905538	0.2718	0.2704	Metal	Louisville	-1.30259	-1.30779	1.00521	0.99482
905511	12.6130	12.7204	Wood	Louisville	2.53472	2.54320	1.00852	1.00852
80106	8.8703	8.9566	Metal	Denver	2.18271	2.19239	1.00973	1.00973
80439	0.1154	0.1166	Wood	Denver	-2.15935	-2.14901	1.01040	1.01040
905588	0.3928	0.3975	Plaster	Louisville	-0.93440	-0.92249	1.01199	1.01199
81850	0.1648	0.1628	Metal	Philadelphia	-1.80302	-1.81523	1.01229	0.98786
905529	0.4417	0.4477	Plaster	Louisville	-0.81710	-0.80372	1.01347	1.01347
81448	0.3941	0.3877	Metal	Philadelphia	-0.93115	-0.94752	1.01651	0.98376
905608	0.2215	0.2179	Metal	Louisville	-1.50728	-1.52394	1.01680	0.98348
80529	0.0290	0.0295	Wood	Denver	-3.54046	-3.52337	1.01724	1.01724
81625	0.8321	0.8163	Metal	Philadelphia	-0.18380	-0.20297	1.01936	0.98101
81752	0.3112	0.3174	Plaster	Philadelphia	-1.16732	-1.14759	1.01992	1.01992
905593	0.1298	0.1324	Plaster	Louisville	-2.04205	-2.02179	1.02046	1.02046
905567	0.3452	0.3523	Wood	Louisville	-1.06359	-1.04318	1.02062	1.02062
80071	1.0038	0.9829	Plaster	Denver	0.00379	-0.01725	1.02126	0.97918
81731	0.2909	0.2973	Concrete	Philadelphia	-1.23478	-1.21301	1.02200	1.02200
905601	5.6275	5.7616	Metal	Louisville	1.72766	1.75122	1.02383	1.02383
905502	8.5182	8.7411	Wood	Louisville	2.14221	2.16804	1.02617	1.02617
81945	0.2117	0.2056	Metal	Philadelphia	-1.55259	-1.58182	1.02967	0.97119
905550	1.0530	1.0856	Metal	Louisville	0.05165	0.08217	1.03099	1.03099
80756	6.2534	6.4476	Concrete	Denver	1.83313	1.86371	1.03106	1.03106
80020	0.2108	0.2179	Wood	Denver	-1.55685	-1.52372	1.03368	1.03368
80465	0.1186	0.1227	Drywall	Denver	-2.13200	-2.09801	1.03457	1.03457
905571	5.7561	5.5528	Wood	Louisville	1.75026	1.71430	1.03662	0.96467
905512	13.0290	12.5670	Wood	Louisville	2.56718	2.53108	1.03676	0.96454
905573	12.5035	12.9654	Wood	Louisville	2.52601	2.56229	1.03694	1.03694
81824	0.1272	0.1225	Plaster	Philadelphia	-2.06199	-2.09964	1.03837	0.96305
905585	0.5035	0.5235	Plaster	Louisville	-0.68613	-0.64718	1.03972	1.03972
81256	0.2676	0.2791	Plaster	Philadelphia	-1.31826	-1.27619	1.04297	1.04297
905547	1.7696	1.6941	Metal	Louisville	0.57076	0.52713	1.04460	0.95731
81546	0.3372	0.3216	Metal	Philadelphia	-1.08708	-1.13445	1.04851	0.95374

PERCENT1 = Lead level in primary sample (%)	PERCENT2 = Lead level in field duplicate sample (%)
RATIO1 = max(PERCENT1, PERCENT2) ÷ min(PERCENT1, PERCENT2)	RATIO2 = PERCENT1 ÷ PERCENT2

Table 4-18 (cont). Lead Levels in Field Duplicate Samples in **Percent by Weight Units**, Sorted by RATIO1.

SAMPLE ID	PERCENT1	PERCENT2	SUBSTRATE	CITY	LOG(PERCENT1)	LOG(PERCENT2)	RATIO1	RATIO2
80219	0.1950	0.2058	Wood	Denver	-1.63476	-1.58085	1.05538	1.05538
81607	0.2093	0.1981	Plaster	Philadelphia	-1.56399	-1.61898	1.05654	0.94649
905537	5.1796	4.8954	Metal	Louisville	1.64473	1.58830	1.05805	0.94513
905576	4.7294	4.4570	Wood	Louisville	1.55380	1.49447	1.06113	0.94239
905506	14.1613	15.0405	Wood	Louisville	2.65051	2.71075	1.06208	1.06208
905603	0.6811	0.6399	Metal	Louisville	-0.38397	-0.44642	1.06444	0.93946
80617	2.5470	2.7162	Wood	Denver	0.93492	0.99923	1.06643	1.06643
81654	0.2101	0.1965	Concrete	Philadelphia	-1.56017	-1.62709	1.06921	0.93527
905507	0.4354	0.4656	Wood	Louisville	-0.83158	-0.76453	1.06935	1.06935
80915	0.0224	0.0209	Brick	Denver	-3.79869	-3.86801	1.07177	0.93304
80654	0.1381	0.1481	Drywall	Denver	-1.97978	-1.90987	1.07241	1.07241
905531	0.2414	0.2603	Concrete	Louisville	-1.42115	-1.34600	1.07805	1.07805
81307	1.0086	1.0887	Wood	Philadelphia	0.00856	0.08498	1.07942	1.07942
905544	0.8059	0.7453	Metal	Louisville	-0.21577	-0.29400	1.08137	0.92475
905526	0.3192	0.2935	Plaster	Louisville	-1.14182	-1.22592	1.08774	0.91934
80469	0.0545	0.0501	Drywall	Denver	-2.90955	-2.99373	1.08782	0.91927
905568	8.4599	7.7643	Wood	Louisville	2.13534	2.04953	1.08960	0.91777
905549	0.2105	0.1927	Metal	Louisville	-1.55848	-1.64638	1.09188	0.91585
905546	0.2387	0.2606	Metal	Louisville	-1.43255	-1.34465	1.09188	1.09188
81523	0.1817	0.1996	Plaster	Philadelphia	-1.70540	-1.61144	1.09851	1.09851
81711	0.4104	0.4511	Metal	Philadelphia	-0.89062	-0.79607	1.09917	1.09917
905541	0.4752	0.5246	Metal	Louisville	-0.74394	-0.64511	1.10388	1.10388
905592	2.6695	2.4163	Plaster	Louisville	0.98191	0.88225	1.10479	0.90515
80433	2.5597	2.3161	Wood	Denver	0.93989	0.83988	1.10518	0.90483
80375	0.0041	0.0037	Plaster	Denver	-5.49677	-5.59942	1.10811	0.90244
81930	0.0672	0.0745	Plaster	Philadelphia	-2.70008	-2.59696	1.10863	1.10863
81917	0.2125	0.2364	Concrete	Philadelphia	-1.54881	-1.44223	1.11247	1.11247
80558	0.0452	0.0503	Drywall	Denver	-3.09666	-2.98975	1.11283	1.11283
905602	0.2746	0.3058	Metal	Louisville	-1.29246	-1.18472	1.11375	1.11375
905522	0.3453	0.3099	Plaster	Louisville	-1.06325	-1.17141	1.11423	0.89748
80153	0.1310	0.1460	Wood	Denver	-2.03256	-1.92415	1.11450	1.11450
905514	0.2992	0.2683	Wood	Louisville	-1.20666	-1.31580	1.11532	0.89661
80371	0.0059	0.0066	Plaster	Denver	-5.13280	-5.02069	1.11864	1.11864
905570	0.1504	0.1343	Wood	Louisville	-1.89430	-2.00769	1.12006	0.89281
80141	3.0756	2.7450	Wood	Denver	1.12350	1.00978	1.12044	0.89251

PERCENT1 = Lead level in primary sample (%)	PERCENT2 = Lead level in field duplicate sample (%)
RATIO1 = max(PERCENT1, PERCENT2) ÷ min(PERCENT1, PERCENT2)	RATIO2= PERCENT1 ÷ PERCENT2

Table 4-18 (cont). Lead Levels in Field Duplicate Samples in **Percent by Weight Units**, Sorted by RATIO1.

SAMPLE ID	PERCENT1	PERCENT2	SUBSTRATE	CITY	LOG(PERCENT1)	LOG(PERCENT2)	RATIO1	RATIO2
81721	1.2142	1.0823	Wood	Philadelphia	0.19409	0.07909	1.12187	0.89137
81342	0.3030	0.3401	Plaster	Philadelphia	-1.19402	-1.07852	1.12244	1.12244
80706	6.4911	5.7784	Metal	Denver	1.87043	1.75413	1.12334	0.89020
81439	1.1662	1.3105	Metal	Philadelphia	0.15375	0.27041	1.12374	1.12374
80549	0.0063	0.0056	Wood	Denver	-5.06721	-5.18499	1.12500	0.88889
81833	0.2238	0.1985	Plaster	Philadelphia	-1.49700	-1.61697	1.12746	0.88695
905508	9.0301	8.0050	Wood	Louisville	2.20057	2.08006	1.12807	0.88647
905598	5.2076	4.6163	Concrete	Louisville	1.65012	1.52960	1.12808	0.88646
905591	0.2107	0.2381	Plaster	Louisville	-1.55723	-1.43522	1.12976	1.12976
80646	0.1219	0.1077	Wood	Denver	-2.10455	-2.22841	1.13185	0.88351
905580	0.0022	0.0024	Drywall	Louisville	-6.14109	-6.01376	1.13579	1.13579
905536	0.2137	0.1879	Metal	Louisville	-1.54295	-1.67176	1.13747	0.87914
80230	0.0049	0.0043	Wood	Denver	-5.31852	-5.44914	1.13953	0.87755
905545	0.3800	0.3335	Metal	Louisville	-0.96753	-1.09821	1.13961	0.87749
80808	0.8759	0.9994	Metal	Denver	-0.13250	-0.00060	1.14100	1.14100
905594	0.1628	0.1423	Plaster	Louisville	-1.81552	-1.94982	1.14373	0.87433
905542	0.7328	0.8392	Metal	Louisville	-0.31082	-0.17533	1.14510	1.14510
80612	0.0041	0.0047	Metal	Denver	-5.49677	-5.36019	1.14634	1.14634
905509	0.3534	0.3077	Wood	Louisville	-1.04011	-1.17847	1.14839	0.87078
905564	13.2784	15.2842	Wood	Louisville	2.58614	2.72682	1.15105	1.15105
80638	0.1250	0.1439	Wood	Denver	-2.07944	-1.93864	1.15120	1.15120
905572	11.2778	9.7818	Wood	Louisville	2.42283	2.28053	1.15293	0.86736
905520	0.0031	0.0027	Drywall	Louisville	-5.78383	-5.92755	1.15457	0.86612
905505	10.8700	12.5618	Wood	Louisville	2.38601	2.53066	1.15564	1.15564
80027	0.0330	0.0285	Wood	Denver	-3.41125	-3.55785	1.15789	0.86364
905562	5.7308	4.9480	Wood	Louisville	1.74585	1.59899	1.15820	0.86341
905600	0.7269	0.6216	Metal	Louisville	-0.31891	-0.47539	1.16939	0.85515
80323	0.0248	0.0211	Wood	Denver	-3.69691	-3.85848	1.17536	0.85081
80778	3.0923	3.6386	Plaster	Denver	1.12892	1.29160	1.17666	1.17666
81921	0.8071	0.6859	Plaster	Philadelphia	-0.21431	-0.37702	1.17670	0.84983
905539	1.3183	1.5521	Metal	Louisville	0.27633	0.43962	1.17738	1.17738
905604	0.1651	0.1945	Metal	Louisville	-1.80149	-1.63741	1.17831	1.17831
80305	0.0289	0.0245	Metal	Denver	-3.54391	-3.70908	1.17959	0.84775
80157	0.1798	0.2121	Plaster	Denver	-1.71591	-1.55070	1.17964	1.17964
80170	0.1698	0.2004	Plaster	Denver	-1.77313	-1.60744	1.18021	1.18021

PERCENT1 = Lead level in primary sample (%)	PERCENT2 = Lead level in field duplicate sample (%)
RATIO1 = max(PERCENT1, PERCENT2) ÷ min(PERCENT1, PERCENT2)	RATIO2= PERCENT1 ÷ PERCENT2

Table 4-18 (cont). Lead Levels in Field Duplicate Samples in **Percent by Weight Units**, Sorted by RATIO1.

SAMPLE ID	PERCENT1	PERCENT2	SUBSTRATE	CITY	LOG(PERCENT1)	LOG(PERCENT2)	RATIO1	RATIO2
80108	4.9727	4.2119	Metal	Denver	1.60396	1.43791	1.18063	0.84700
905584	5.1051	6.0334	Plaster	Louisville	1.63024	1.79731	1.18184	1.18184
80444	0.0762	0.0641	Wood	Denver	-2.57439	-2.74731	1.18877	0.84121
80421	0.0700	0.0833	Wood	Denver	-2.65926	-2.48531	1.19000	1.19000
905597	0.2229	0.1870	Concrete	Louisville	-1.50088	-1.67659	1.19208	0.83887
905566	4.0341	4.8166	Wood	Louisville	1.39478	1.57207	1.19397	1.19397
905521	3.7464	4.4742	Plaster	Louisville	1.32079	1.49833	1.19427	1.19427
81337	0.2736	0.3277	Plaster	Philadelphia	-1.29609	-1.11566	1.19773	1.19773
81315	3.7021	3.0856	Wood	Philadelphia	1.30890	1.12675	1.19980	0.83347
905524	0.5180	0.4258	Plaster	Louisville	-0.65785	-0.85378	1.21645	0.82207
80358	0.2372	0.2888	Concrete	Denver	-1.43885	-1.24202	1.21754	1.21754
905595	0.5141	0.4218	Concrete	Louisville	-0.66535	-0.86322	1.21879	0.82048
81413	0.9388	1.1551	Concrete	Philadelphia	-0.06315	0.14419	1.23040	1.23040
905540	0.5088	0.6277	Metal	Louisville	-0.67573	-0.46564	1.23379	1.23379
81458	0.9341	1.1591	Wood	Philadelphia	-0.06817	0.14764	1.24087	1.24087
905527	0.4176	0.3360	Plaster	Louisville	-0.87319	-1.09069	1.24296	0.80453
81513	0.2526	0.3140	Concrete	Philadelphia	-1.37595	-1.15836	1.24307	1.24307
81858	0.2304	0.1843	Wood	Philadelphia	-1.46794	-1.69119	1.25014	0.79991
905523	4.5865	3.6688	Plaster	Louisville	1.52313	1.29985	1.25016	0.79990
905563	13.4021	10.6820	Wood	Louisville	2.59541	2.36856	1.25464	0.79704
905575	4.2509	5.3574	Wood	Louisville	1.44713	1.67848	1.26031	1.26031
80848	0.3255	0.2571	Brick	Denver	-1.12239	-1.35829	1.26604	0.78986
81207	8.0533	6.3376	Metal	Philadelphia	2.08608	1.84650	1.27072	0.78696
80277	0.2027	0.1589	Plaster	Denver	-1.59603	-1.83948	1.27565	0.78392
905501	11.1032	8.6911	Wood	Louisville	2.40723	2.16230	1.27754	0.78276
80514	0.7684	0.5999	Wood	Denver	-0.26344	-0.51099	1.28088	0.78071
905516	3.5931	4.6274	Wood	Louisville	1.27902	1.53200	1.28786	1.28786
905574	0.1222	0.1574	Wood	Louisville	-2.10218	-1.84895	1.28817	1.28817
905609	0.2720	0.2105	Metal	Louisville	-1.30197	-1.55826	1.29213	0.77392
905548	5.5140	4.2574	Metal	Louisville	1.70729	1.44866	1.29515	0.77211
81712	0.4934	0.6421	Metal	Philadelphia	-0.70644	-0.44301	1.30138	1.30138
905530	0.1219	0.0932	Concrete	Louisville	-2.10482	-2.37273	1.30724	0.76497
81239	0.0740	0.0564	Concrete	Philadelphia	-2.60369	-2.87529	1.31206	0.76216
905606	0.8532	0.6499	Metal	Louisville	-0.15873	-0.43087	1.31278	0.76174
905528	3.6819	4.8594	Plaster	Louisville	1.30342	1.58091	1.31981	1.31981

PERCENT1 = Lead level in primary sample (%)	PERCENT2 = Lead level in field duplicate sample (%)
RATIO1 = max(PERCENT1, PERCENT2) ÷ min(PERCENT1, PERCENT2)	RATIO2= PERCENT1 ÷ PERCENT2

Table 4-18 (cont). Lead Levels in Field Duplicate Samples in **Percent by Weight Units**, Sorted by RATIO1.

SAMPLE ID	PERCENT1	PERCENT2	SUBSTRATE	CITY	LOG(PERCENT1)	LOG(PERCENT2)	RATIO1	RATIO2
80664	0.0490	0.0647	Drywall	Denver	-3.01593	-2.73799	1.32041	1.32041
80507	1.4205	1.8837	Metal	Denver	0.35101	0.63324	1.32608	1.32608
80827	0.0016	0.0012	Wood	Denver	-6.43775	-6.72543	1.33333	0.75000
905587	4.9388	3.6765	Plaster	Louisville	1.59713	1.30196	1.34335	0.74441
81753	0.3918	0.2883	Plaster	Philadelphia	-0.93700	-1.24375	1.35900	0.73583
80679	0.1469	0.1080	Concrete	Denver	-1.91800	-2.22562	1.36019	0.73519
80733	6.8598	4.9200	Brick	Denver	1.92568	1.59331	1.39427	0.71722
905589	0.3881	0.2776	Plaster	Louisville	-0.94645	-1.28150	1.39801	0.71530
905515	10.4921	7.4843	Wood	Louisville	2.35062	2.01281	1.40187	0.71333
80852	0.0077	0.0108	Brick	Denver	-4.86653	-4.52821	1.40260	1.40260
80206	16.6756	23.4386	Metal	Denver	2.81395	3.15438	1.40556	1.40556
80314	2.2774	1.6075	Wood	Denver	0.82303	0.47468	1.41673	0.70585
80545	0.00290	0.0020	Wood	Denver	-5.84304	-6.21461	1.45000	0.68966
80924	0.01480	0.0102	Drywall	Denver	-4.21313	-4.58537	1.45098	0.68919
81355	0.04240	0.0291	Metal	Philadelphia	-3.16061	-3.53702	1.45704	0.68632
81849	0.22530	0.1520	Metal	Philadelphia	-1.49032	-1.88387	1.48224	0.67466
80140	3.39040	5.0758	Wood	Denver	1.22095	1.62448	1.49711	1.49711
905596	3.01411	2.0051	Concrete	Louisville	1.10330	0.69567	1.50326	0.66522
81957	1.19490	0.7883	Wood	Philadelphia	0.17806	-0.23788	1.51579	0.65972
905517	0.00082	0.0013	Drywall	Louisville	-7.10021	-6.67163	1.53507	1.53507
80531	0.29330	0.4522	Wood	Denver	-1.22656	-0.79363	1.54177	1.54177
905586	0.34356	0.5320	Plaster	Louisville	-1.06839	-0.63114	1.54844	1.54844
905590	2.98120	4.6386	Plaster	Louisville	1.09233	1.53440	1.55593	1.55593
81939	0.68950	0.4355	Metal	Philadelphia	-0.37179	-0.83126	1.58324	0.63162
905599	0.44643	0.7099	Metal	Louisville	-0.80647	-0.34261	1.59019	1.59019
81556	1.05850	0.6627	Wood	Philadelphia	0.05685	-0.41143	1.59725	0.62607
81812	0.12460	0.2031	Concrete	Philadelphia	-2.08265	-1.59406	1.63002	1.63002
905518	0.00136	0.0008	Drywall	Louisville	-6.60083	-7.09605	1.64085	0.60944
80762	1.65650	1.0031	Concrete	Denver	0.50471	0.00310	1.65138	0.60555
80059	0.00550	0.0033	Concrete	Denver	-5.20301	-5.71383	1.66667	0.60000
81221	1.31110	0.7826	Wood	Philadelphia	0.27087	-0.24513	1.67531	0.59690
80818	0.89780	1.5122	Wood	Denver	-0.10781	0.41357	1.68434	1.68434
80353	0.05470	0.0324	Drywall	Denver	-2.90589	-3.42960	1.68827	0.59232
81629	4.07130	2.4100	Metal	Philadelphia	1.40396	0.87963	1.68934	0.59195
80745	3.24900	5.4961	Brick	Denver	1.17835	1.70404	1.69163	1.69163

PERCENT1 = Lead level in primary sample (%)	PERCENT2 = Lead level in field duplicate sample (%)
RATIO1 = max(PERCENT1, PERCENT2) ÷ min(PERCENT1, PERCENT2)	RATIO2= PERCENT1 ÷ PERCENT2

Table 4-18 (cont). Lead Levels in Field Duplicate Samples in **Percent by Weight Units**, Sorted by RATIO1.

SAMPLE ID	PERCENT1	PERCENT2	SUBSTRATE	CITY	LOG(PERCENT1)	LOG(PERCENT2)	RATIO1	RATIO2
80257	0.54620	0.9287	Plaster	Denver	-0.60477	-0.07397	1.70029	1.70029
905605	0.25300	0.1426	Metal	Louisville	-1.37435	-1.94747	1.77379	0.56377
81544	0.21240	0.3801	Metal	Philadelphia	-1.54928	-0.96732	1.78955	1.78955
905569	0.00051	0.0009	Wood	Louisville	-7.57143	-6.98134	1.80415	1.80415
81211	7.18480	13.4222	Metal	Philadelphia	1.97197	2.59691	1.86814	1.86814
80721	6.65880	12.4515	Wood	Denver	1.89594	2.52184	1.86993	1.86993
80409	0.05300	0.0282	Metal	Denver	-2.93746	-3.56843	1.87943	0.53208
80875	0.00360	0.0069	Plaster	Denver	-5.62682	-4.97623	1.91667	1.91667
80831	0.79270	1.5227	Wood	Denver	-0.23231	0.42049	1.92090	1.92090
80971	0.17370	0.3427	Concrete	Denver	-1.75043	-1.07090	1.97294	1.97294
80937	0.00170	0.0035	Concrete	Denver	-6.37713	-5.65499	2.05882	2.05882
905607	0.38180	0.8378	Metal	Louisville	-0.96286	-0.17703	2.19423	2.19423
81640	0.01550	0.0070	Wood	Philadelphia	-4.16692	-4.96185	2.21429	0.45161
80578	0.00900	0.0039	Concrete	Denver	-4.71053	-5.54678	2.30769	0.43333
81244	0.43030	1.0690	Concrete	Philadelphia	-0.84327	0.06672	2.48431	2.48431
80972	1.63450	0.60490	Concrete	Denver	0.49134	-0.50269	2.7021	0.37008
905577	0.00263	0.00097	Drywall	Louisville	-5.93908	-6.93590	2.7097	0.36905
80870	0.00620	0.01790	Plaster	Denver	-5.08321	-4.02295	2.8871	2.88710
80720	1.40890	0.48650	Wood	Denver	0.34281	-0.72052	2.8960	0.34530
80965	0.00070	0.00210	Concrete	Denver	-7.26443	-6.16582	3.0000	3.00000
80449	0.11570	0.37420	Wood	Denver	-2.15675	-0.98296	3.2342	3.23423
80035	0.00120	0.00400	Brick	Denver	-6.72543	-5.52146	3.3333	3.33333
80908	0.98040	0.16690	Brick	Denver	-0.01979	-1.79036	5.8742	0.17024
80129	0.12670	0.76760	Wood	Denver	-2.06593	-0.26449	6.0584	6.05841
80229	0.03710	0.00570	Wood	Denver	-3.29414	-5.16729	6.5088	0.15364
80266	0.48400	0.00800	Plaster	Denver	-0.72567	-4.82831	60.5000	0.01653

PERCENT1 = Lead level in primary sample (%)
PERCENT2 = Lead level in field duplicate sample (%)
RATIO1 = max(PERCENT1, PERCENT2) ÷ min(PERCENT1, PERCENT2)
RATIO2= PERCENT1 ÷ PERCENT2

Table 4-19. Correlations and Regression Coefficients for Regression of LOG(AREA2) Against LOG(AREA1) by City.

CITY	N[a]	CORRELATION	SLOPE	INTERCEPT
Denver	74	0.957	0.977	0.011
Philadelphia	44	0.947	1.002	0.040
Louisville	39	0.995	1.002	-0.019

[a] N represents the number of paired results.

Table 4-20. Correlations and Regression Coefficients for Regression of LOG(PERCENT2) Against LOG(PERCENT1) by City.

CITY	N[a]	CORRELATION	SLOPE	INTERCEPT
Denver	74	0.956	0.963	-0.098
Philadelphia	44	0.969	1.017	-0.016
Louisville	39	0.994	1.000	-0.022

[a] N represents the number of paired results.

there is a factor of approximately 60 between the primary and field duplicate lead levels in both types of units. The Philadelphia plots also have constant variance; however, there is an outlier in the area plot that does not appear in the percent plot. This point has primary lead levels of 0.06291 mg/cm^2 and 0.2124% and field duplicate levels of 0.38413 mg/cm^2 and 0.3801%. Thus the ratio of field duplicate to primary lead level is 6.2 in area units (mg/cm^2) but only 1.8 in percent by weight units. The residual plots of the Louisville data show slightly higher variability at the lower lead levels; each has a single outlier. The outlier pair has primary lead levels of 0.00086 mg/cm^2 and 0.00263% and field duplicate lead levels of 0.00033 mg/cm^2 and 0.00097%.

The same statistical model and outlier analysis as used for the laboratory duplicates can be applied to the field duplicates. For example, for the area unit data, the model is

$$LOG(AREAi) = LOG(LEAD) + e_i \quad (i = 1,2)$$

where LEAD now denotes the average true lead level in the area of the component from which the field duplicate samples were taken,

and e_i are random errors that are independent and normally distributed with mean 0 and constant variance F^2. Tables 4-21 and 4-22 show the outlier analysis for the field duplicate data

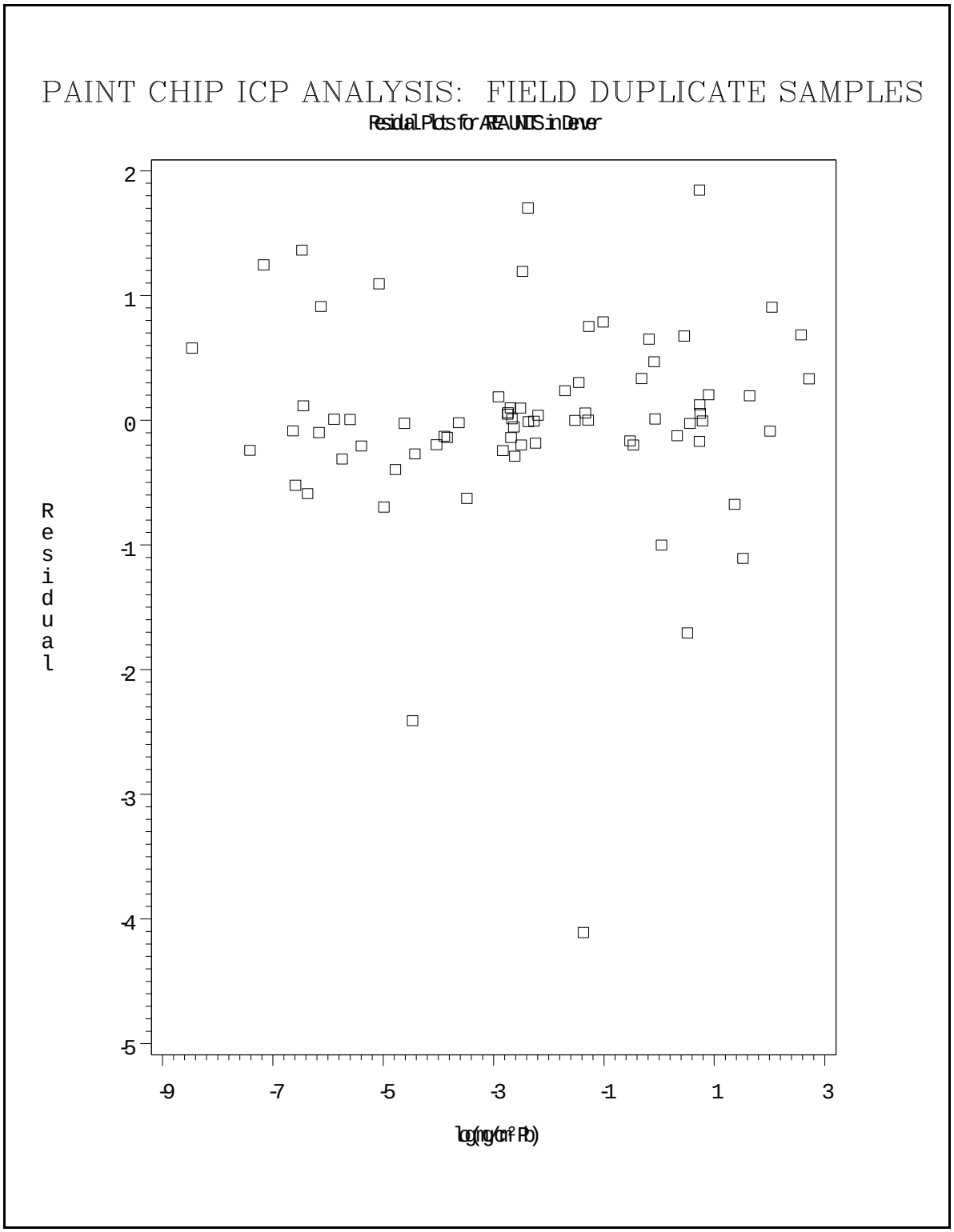

Figure 4-24. Plot of residuals from regression of log(field duplicate) versus log(primary sample) in Denver (mg/cm²).

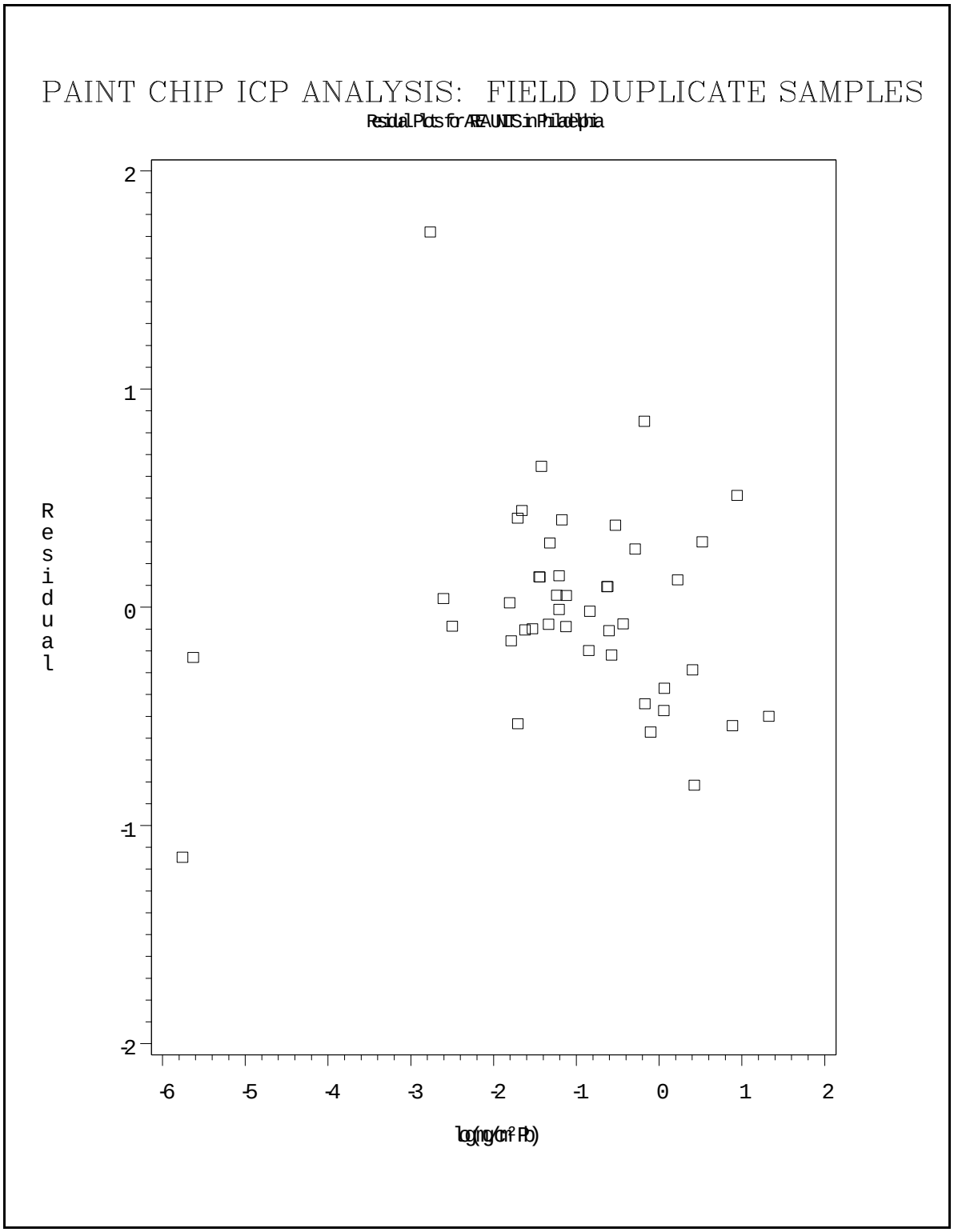

Figure 4-25. Plot of residuals from regression of log(field duplicate) versus log(primary sample) in Philadelphia (mg/cm²).

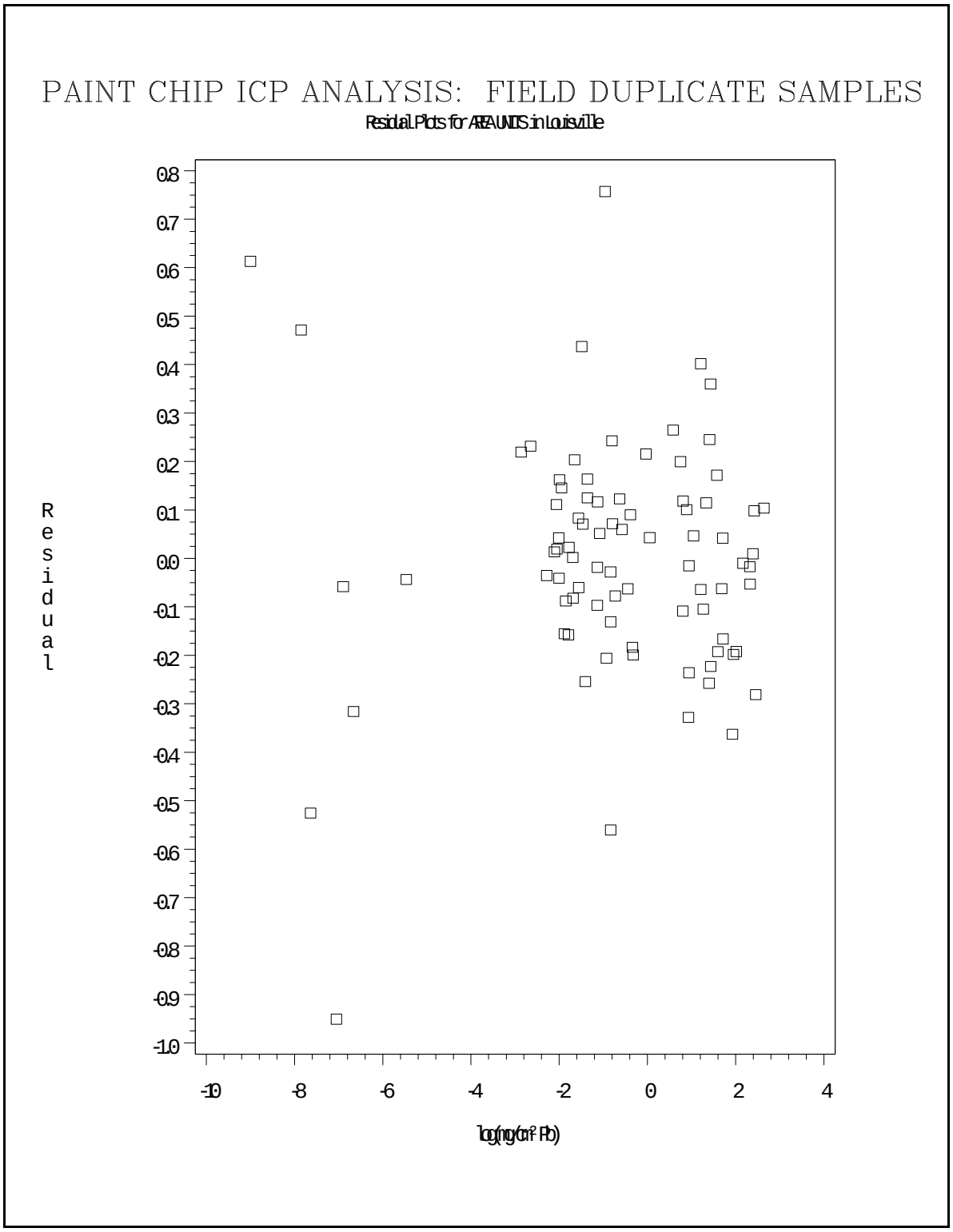

Figure 4-26. Plot of residuals from regression of log(field duplicate) versus log(primary sample) in Louisville (mg/cm²).

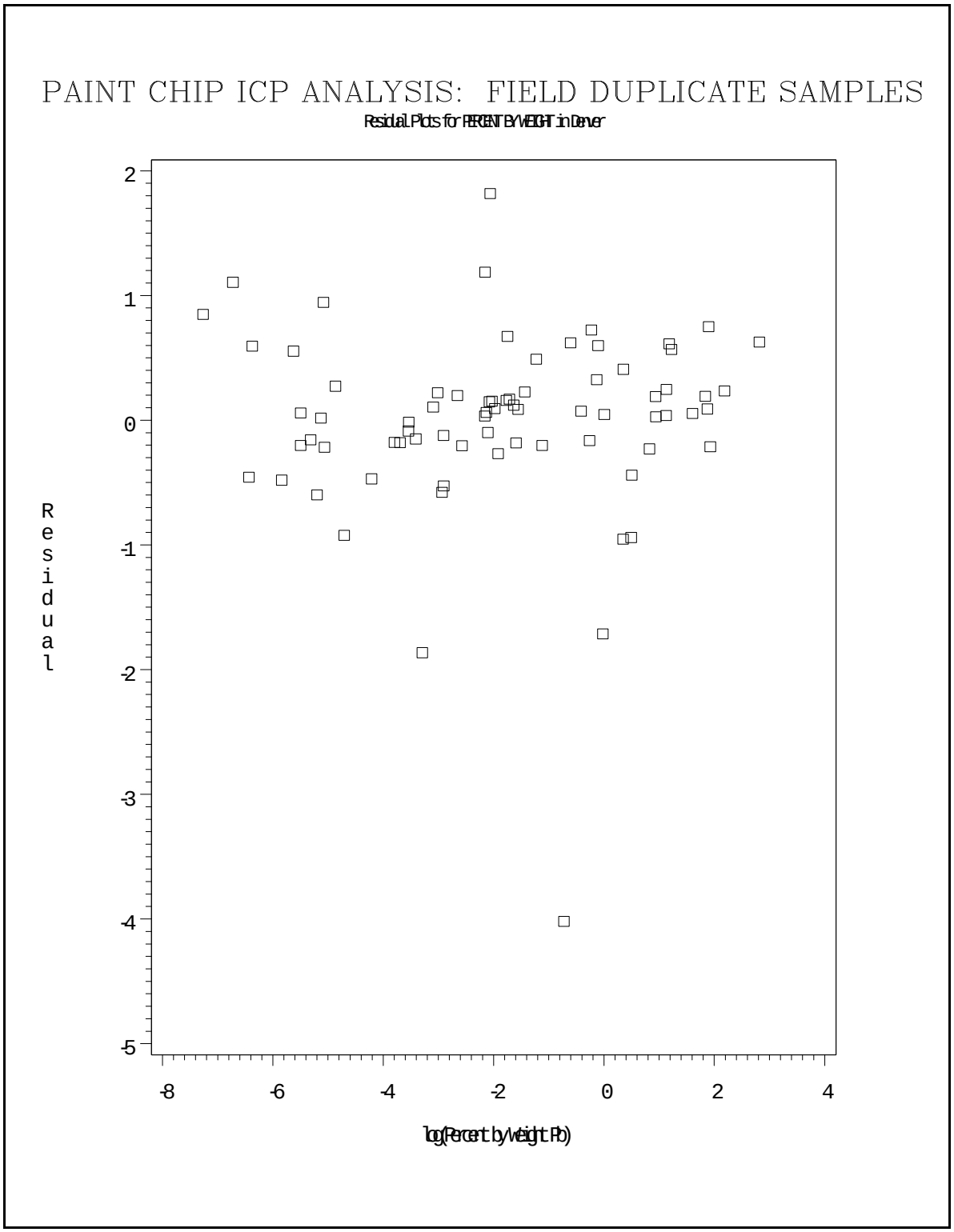

Figure 4-27. Plot of residuals from regression of log(field duplicate) versus log(primary sample) in Denver (percent by weight).

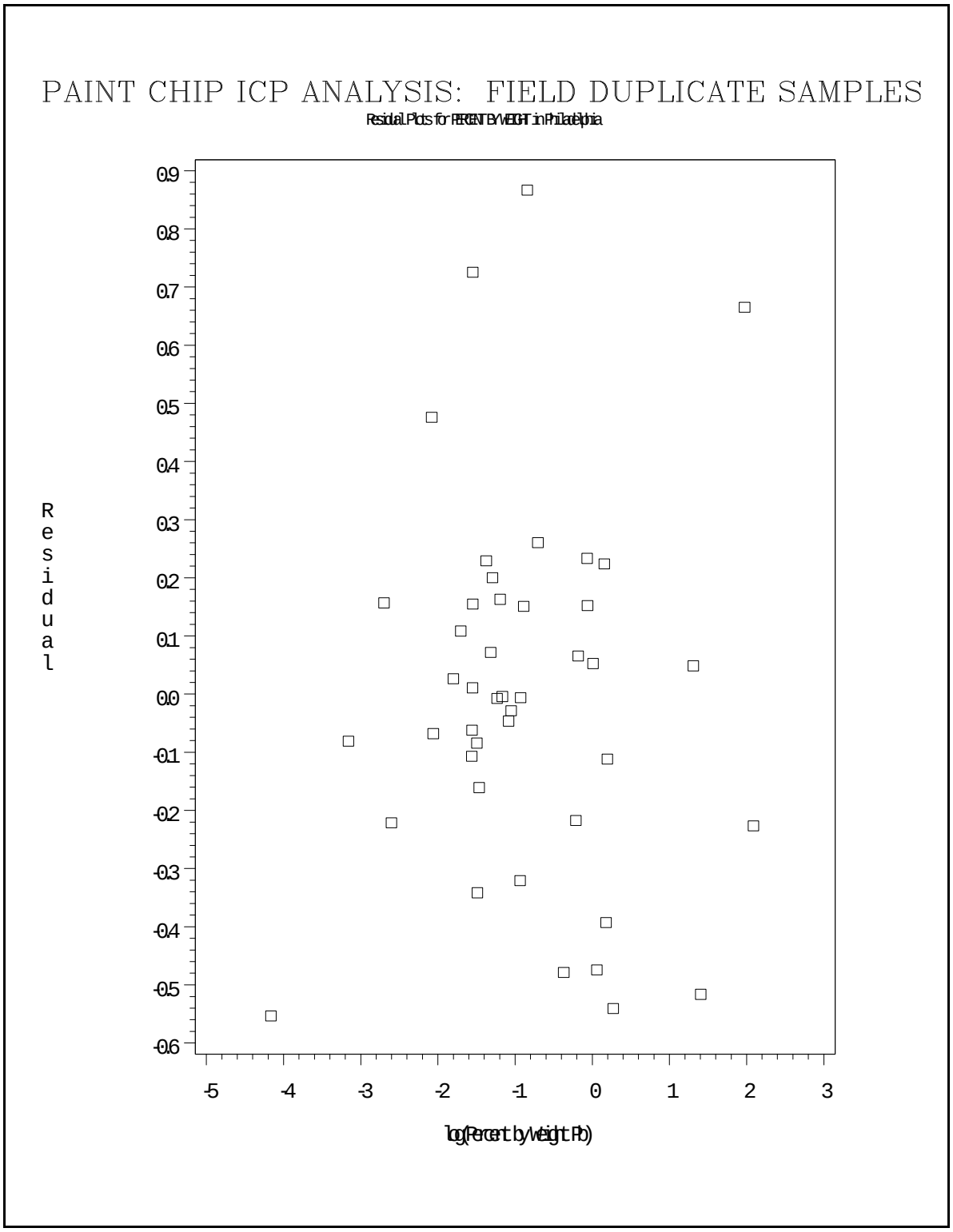

Figure 4-28. Plot of residuals from regression of log(field duplicate) versus log(primary sample) in Philadelphia (percent by weight).

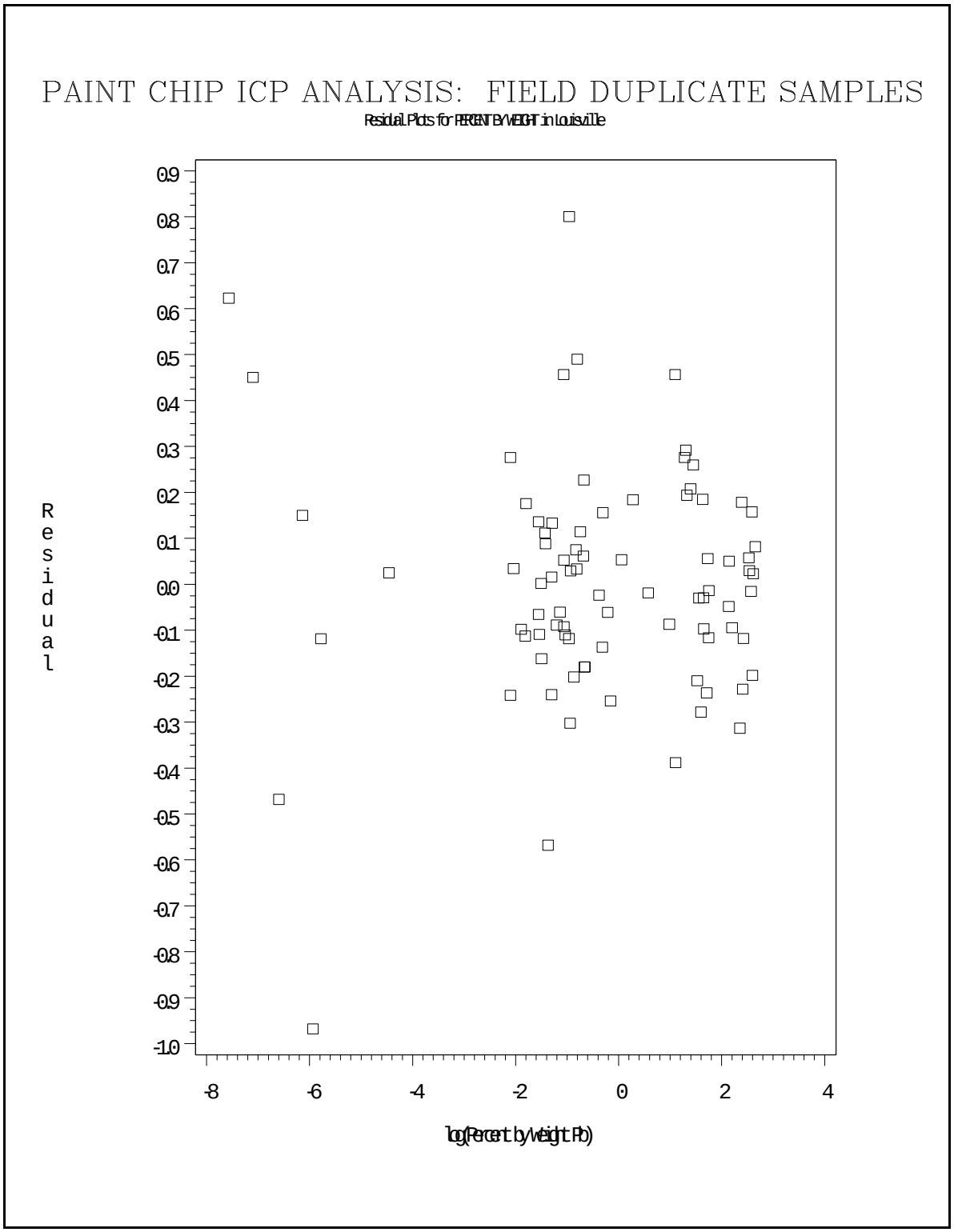

Figure 4-29. Plot of residuals from regression of log(field duplicate) versus log(primary sample) in Louisville (percent by weight).

Table 4-21. Outlier Analysis for Field Duplicate Data (mg/cm^2 lead).

	DENVER	PHILADELPHIA	LOUISVILLE
N	74	44	83
s_{av}^2	0.3310	0.1092	0.0295
M(1,N,95)	11.53	10.56	11.74
Outlier Cutoff	3.8170	1.1533	0.3462
Outlier Values	8.3274	1.6367	0.4587
Revised s_{av}^2	0.2215	0.0737	0.0243
Revised s_{av}	0.4706	0.2715	0.1558

Table 4-22. Outlier Analysis for Field Duplicate Data (Percent by Weight Lead).

	DENVER	PHILADELPHIA	LOUISVILLE
N	74	44	83
s_{av}^2	0.2772	0.0518	0.0318
M(1,N,95)	11.53	10.56	11.74
Outlier Cutoff	3.1966	0.5470	0.3733
Outlier Values	8.4158	None	0.4974
Revised s_{av}^2	0.1658	0.0518	0.0261
Revised s_{av}	0.4071	0.2276	0.1616

in mg/cm^2 lead and percent by weight, respectively. The pairs identified as outliers in the residual plots are confirmed by the outlier criterion.

Tables 4-21 and 4-22 show that, in both sets of units, variability in Denver is highest, with Philadelphia next and Louisville last.

All differences are statistically significant based on an F-test. In Denver and Philadelphia, field duplicate variability is statistically significantly higher than laboratory duplicate variability. In Louisville, the F-test comparing field duplicate and laboratory duplicate variability has a p-value of 0.02 which is of marginal statistical significance given the number of simultaneous comparisons being made. The most probable reason

why field duplicates are much more highly variable than laboratory duplicates in Denver and Philadelphia is because of the 9 inch average distance between primary and field duplicate samples. Most of the field duplicate variability is caused by differences in the true lead level in the pair of samples. In Louisville, where the field duplicates were true side-by-side samples taken an average of only 2 inches apart, there is much less variation in the true lead level so that the variability of field duplicates is only slightly higher than that of laboratory duplicates. However, some of the increase in variability may be due to the additional variability from often analyzing the primary sample and field duplicate sample in two different laboratory batches. By contrast, laboratory duplicate samples were almost always analyzed in the same batch. The difference in field duplicate variability between Denver and Philadelphia is harder to explain. However, the likely reason is the much greater variety of paint and substrates encountered in the single-family housing in Denver as compared to the multifamily development tested in Philadelphia. It is also possible that more of the painting in the Philadelphia units was done by professionals and was therefore more uniform. Finally, comparison of Tables 4-21 and 4-22 shows that the variability in area units in Philadelphia and Denver is slightly higher than variability in percent by weight units.

Tables 4-23 and 4-24 give estimates of field duplicate standard deviation by city and substrate in area and percent by weight units, respectively.

In Denver, variability in area units was consistent across substrates with the exception of drywall where variability is much lower. Although brick and concrete showed higher variability than metal, plaster, and wood, the differences were not statistically significant because of the small sample sizes. Similar variability relationships across substrates occurred for percent by weight units in Denver with the exception of variability on metal. This variability was much lower than it was in area units, primarily because of a single pair of results. For this pair, the primary sample and field duplicate results, in area units, were reported as 2.07227 and 11.82821 mg/cm^2 lead. In percent by weight units, results for this same pair were reported as 16.6756% and 23.4386% lead. Thus, the relative variation was much greater in area units than in percent by weight units.

In Philadelphia, variability was consistent across substrates in both units, except that variability on plaster was much lower than on the other substrates. The very hard and smooth nature of the plaster in Philadelphia may have contributed

Table 4-23. Estimated Standard Deviations on the Log Scale for Field Duplicate Samples in Area Units, with Associated Sample Sizes, by City and Substrate (Outliers Excluded).

SUBSTRATE	DENVER			PHILADELPHIA			LOUISVILLE		
	N[a]	s_{av}	95% Interval[b]	N[a]	s_{av}	95% Interval[b]	N[a]	s_{av}	95% Interval[b]
Brick	7	0.6009	0.189, 5.289	0	N/A	N/A	0	N/A	N/A
Concrete	10	0.5533	0.216, 4.635	8	0.3105	0.423, 2.365	6	0.1324	0.693, 1.443
Drywall	7	0.1640	0.635, 1.576	0	N/A	N/A	4	0.2714	0.471, 2.122
Metal	9	0.4290	0.305, 3.284	14	0.2464	0.505, 1.980	26	0.1751	0.616, 1.625
Plaster	10	0.4569	0.282, 3.548	12	0.1021	0.754, 1.327	19	0.1148	0.728, 1.375
Wood	30	0.4707	0.271, 3.687	9	0.3975	0.332, 3.010	27	0.1417	0.675, 1.481
All	73	0.4706	0.271, 3.686	43	0.2715	0.471, 2.122	82	0.1558	0.650, 1.540

[a] N represents the number of paired results.
[b] A 95% probability interval for the ratio of field duplicates under the assumption the standard deviation is the true value.

Table 4-24. Estimated Standard Deviations on the Log Scale for Field Duplicate Samples in Percent by Weight Units (Percent by Weight Lead), with Associated Sample Sizes, by City and Substrate (Outliers Excluded).

SUBSTRATE	DENVER			PHILADELPHIA			LOUISVILLE		
	N[a]	s_{av}	95% Interval[b]	N[a]	s_{av}	95% Interval[b]	N[a]	s_{av}	95% Interval[b]
Brick	7	0.6063	0.186, 5.369	0	N/A	N/A	0	N/A	N/A
Concrete	10	0.4756	0.268, 3.737	8	0.2792	0.461, 2.168	6	0.1654	0.632, 1.582
Drywall	7	0.1917	0.588, 1.701	0	N/A	N/A	4	0.2431	0.510, 1.962
Metal	9	0.1969	0.579, 1.726	15	0.2356	0.521, 1.921	26	0.1752	0.615, 1.625
Plaster	10	0.3157	0.417, 2.399	12	0.0929	0.773, 1.294	19	0.1558	0.649, 1.540
Wood	30	0.4344	0.300, 3.334	9	0.2828	0.457, 2.190	27	0.1337	0.690, 1.449
All	73	0.4071	0.324, 3.091	44	0.2276	0.532, 1.879	82	0.1616	0.639, 1.565

[a] N represents the number of paired results.
[b] A 95% probability interval for the ratio of field duplicates under the assumption the standard deviation is the true value.

to more uniform paint-chip samples relative to the other substrates. In Louisville, variability was consistent across substrates in both units of measure, except for drywall. This is because all the study lead levels on drywall were extremely low and does not imply that the variability of drywall samples would be higher than the other substrates as a general rule.

In Denver and Philadelphia, there was a generally consistent pattern across substrates of higher variability in area than percent units. However, in Louisville, no such pattern appeared and variability in data expressed as area units and percent by weight units was comparable.

Variability in field duplicate samples could result in inconsistent classification of paint against either the 1.0 mg/cm^2 or 0.5% standards. An inconsistent classification occurs when comparing both results of a pair against a standard and one result is equal to or exceeds the standard while the other result is less than the standard. The following analysis uses results from field duplicate pair samples that were collected including all pairs where either or both of the results from the duplicate samples were reported as non-detectable. Unlike previous analyses which excluded non-detectable results because the values were not exactly known, non-detectable results are included in this analysis since a non-detectable result provides a reliable negative classification relative to the federal standards. The eleven pairs where a collection or analytical problem was encountered for at least one of the samples in Louisville, were eliminated from this analysis.

Of the 128 total field duplicate pairs in Denver and Philadelphia combined, ten (8%) had inconsistent classifications against the 1.0 mg/cm^2 standard and eight (6%) had inconsistent classifications with respect to the 0.5% standard. Of 89 total field duplicate pairs in Louisville, which were taken closer together (2-inch average) than in Denver and Philadelphia (9-inch average), 1 (1%) had an inconsistent classification against the 1.0 mg/cm^2 standard, and 4 (4%) had inconsistent classifications against the 0.5% standard.

Laboratory duplicates, which were not affected by spatial variability, exhibited greater concordance in classification than the field duplicates. Of 171 total laboratory duplicate pairs in all three cities, only 2 (1%) had inconsistent classifications

against the 1.0 mg/cm^2 standard, while 3 (2%) had inconsistent classifications against the 0.5% standard.

The variability estimates shown in Tables 4-21 and 4-22 can be used to calculate the probability of exceeding various size ratios[2] in field duplicate pairs. Table 4-25 shows the probability that the size ratio for a field duplicate pair will exceed various values using the 0.4706 field duplicate standard deviation for the log scale area units in Denver. Table 4-26 shows the same information in percent by weight units using the 0.4071 field duplicate standard deviation for the log scale percent by weight units in Denver. Table 4-27 through 4-30 are the companion tables for Philadelphia and Louisville. The 0.2715 field duplicate standard deviation for the log scale area units in Philadelphia is used to compute the probabilities shown in Table 4-27 and the 0.2276 field duplicate standard deviation for the log scale percent by weight units in Philadelphia is used to compute the probabilities shown in Table 4-28. Similarly, the 0.1558 field duplicate standard deviation for the log scale area units in Louisville is used to compute the probabilities shown in Table 4-29 and the 0.1616 field duplicate standard deviation for the log scale percent by weight units in Louisville is used to compute the probabilities shown in Table 4-30. As can be seen from the tables, large relative differences between field duplicate pairs are possible. For example, 10% of pairs measured in area units from Denver are expected to have ratios of 3 or larger. Table 4-17 shows that 9 of 77 ratios (the outlier is excluded) in Denver actually exceeded 3, in close agreement with the model prediction of 77 * 0.1 = 7.7

4.4 LABORATORY ANALYSIS QUALITY CONTROL (QC) RESULTS

Analyses on the results of laboratory quality control samples are presented below. These analyses support other analyses presented in previous sections which address the study objective to investigate the variability of lead levels in the paint within the sampling locations.

Field paint-chip samples were prepared and analyzed in small

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[2]A size ratio is defined as the ratio of the two results from a pair, either a field or laboratory duplicate pair, with the smaller of the two in the denominator.

groups called batches as discussed under the design elaboration section, section 3.3.3. Each batch was assigned a unique three-character ID. Laboratory QC samples were included in each batch to estimate the analytical performance for field paint-chip samples in the batch. Two general classifications of QC samples were used: (1) instrumental analysis QC samples (placed into

Table 4-25. Ratios for Larger to Smaller of Field Duplicate Pairs, with Associated Probabilities of Exceeding the Ratio, for Area Units in Denver Using the 0.4706 Field Duplicate Standard Deviation for the Log Scale Area Units.

RATIO	PROBABILITY OF EXCEEDING
1.6	50%
2.2	25%
3.0	10%
3.7	5%
5.6	1%

Table 4-26. Ratios for Larger to Smaller of Field Duplicate Pairs, with Associated Probabilities of Exceeding the Ratio, for Percent by weight Units in Denver Using the 0.4071 Field Duplicate Standard Deviation for the Log Scale Percent by Weight Units.

RATIO	PROBABILITY OF EXCEEDING
1.5	50%
1.9	25%
2.6	10%
3.1	5%
4.4	1%

Table 4-27. Ratios for Larger to Smaller of Field Duplicate Pairs, with Associated Probabilities of Exceeding the Ratio, for Area Units in Philadelphia Using the 0.2715 Field Duplicate Standard Deviation for the Log Scale Area Units.

RATIO	PROBABILITY OF EXCEEDING
1.3	50%
1.4	25%
1.9	10%
2.1	5%
2.7	1%

Table 4-28. Ratios for Larger to Smaller of Field Duplicate Pairs, with Associated Probabilities of Exceeding the Ratio, for Percent by weight Units in Philadelphia Using the 0.2276 Field Duplicate Standard Deviation for the Log Scale Percent by Weight Units.

RATIO	PROBABILITY OF EXCEEDING
1.2	50%
1.4	25%
1.7	10%
1.9	5%
2.3	1%

Table 4-29. Ratios for Larger to Smaller of Field Duplicate Pairs, with Associated Probabilities of Exceeding the Ratio, for Area Units in Louisville Using the 0.1558 Field Duplicate Standard Deviation for the Log Scale Area Units.

RATIO	PROBABILITY OF EXCEEDING
1.2	50%
1.3	25%
1.4	10%
1.5	5%
1.8	1%

Table 4-30. Ratios for Larger to Smaller of Field Duplicate Pairs, with Associated Probabilities of Exceeding the Ratio, for Percent by weight Units in Louisville Using the 0.1616 Field Duplicate Standard Deviation for the Log Scale Percent by Weight Units.

RATIO	PROBABILITY OF EXCEEDING
1.2	50%
1.3	25%
1.4	10%
1.6	5%
1.8	1%

each instrumental analysis batch, which usually consisted of one or more sample preparation batches) and, (2) sample preparation and field QC samples (which included method blanks, field blanks and blind samples). Laboratory QC results are discussed in this section with respect to these two classifications of QC samples.

4.4.1 Instrumental Analysis Quality Control Samples

The instrumental analysis QC samples were analyzed along with field paint-chip samples to assure acceptable instrument performance during lead determinations. They included daily calibration standards, multiple calibration verification standards, multiple calibration blank samples, and interference check standards. A detailed description of the use, specifications, and frequency of use of these instrumental analysis QC standards was presented under the design elaboration section in section 3.3.2 and Table 3-18. In addition to these QC samples, test solutions, called instrumental detection limit (IDL) standards, were placed in each analysis batch at a minimum rate of five per batch. The IDL standards were prepared at a lead concentration of 0.1 µg/ml. Lead results from analysis of IDL standards were used to calculate an instrumental detection limit (IDL) for each batch. The IDL was calculated as three times the sample standard deviation of the lead results obtained from the IDL standards. All lead results measured at or above the IDL were reported by the laboratory. All lead results measured below the IDL were identified as non-detectable, and reported as "<" a sample-specific detection limit (DL). The DLs were calculated using the IDL and the appropriate sample preparation parameters such as pre-measurement dilution factors and final dilution volume, subsample mass, total sample mass, and collected sample area.

4.4.2 Sample Preparation and Field Quality Control Samples

Sample preparation QC samples were placed in each sample preparation batch to estimate precision and accuracy of laboratory processing of field paint-chip samples. They included method blanks (also called digestion blanks) and two kinds of blind samples as discussed under the design elaboration section in section 3.3.2 and Table 3-17. Method blanks are blank samples processed in a manner identical to field paint-chip samples except no sample matrix is present in the container used for

sample digestion. They are intended to provide information on the potential systematic lead contamination of field paint-chip samples resulting from laboratory processing. Field blanks were collected at a minimum rate of one per housing unit in the field. Field blanks were representative empty sample collection containers shipped from the field to the laboratory with the field paint-chip samples. Results for the sample preparation QC samples and field blanks for all three cities are discussed in the following sections.

4.4.2.1 Method Blanks and Field Blanks

Table 4-31 presents summary statistics for method blanks, field blanks and field paint-chip samples. Included in this table are sample specific detection limit statistics for these samples. Because sample collection and analysis parameters used to calculate lead results varied between samples, detection limits also varied between samples. Sample collection and analysis parameters which varied included the IDL, additional dilution factors, total collected sample mass, subsample mass, and collected sample area. The formulas used to calculated sample specific detection limits and the ranges for these parameters are shown in a footnote to Table 4-31.

Table 4-31 indicates that 16.5% of the 79 method blanks showed detectable levels of lead. All of the detectable levels were very low. In fact, the highest detectable level was smaller than the maximum sample-specific detection limit for the method blanks. Thus, the method blank data indicate that the field paint-chip samples are free from any significant bias caused by contamination during laboratory processing.

As indicated in Table 4-31, 32.2% of the field blanks showed detectable levels of lead. However, when compared to the method blank data, the reported lead value range and maximum is less than those for the method blanks, 2.13 to 8.70 µg/sample for the field blanks compared to 2.13 to 8.91 for the method blanks. These data show that lead levels in field blanks were no higher than in method blanks. Thus, no field contamination problems are indicated.

Field paint-chip sample data in Table 4-31 show that only 4.2% (54) of the 1,290 field samples (100% - 95.8% = 4.2%) were reported as non-detectable. Typical detection limits for the

field paint-chip samples were two orders of magnitude below the federal standards (1.0 mg/cm^2 and 0.5%). For the field samples that had non-detectable levels of lead, the range of the sample specific detection limits was 0.0001 to 0.009 in mg/cm^2 units and 0.0004 to 0.02 in percent by weight units.

Table 4-31. Comparison Summary of Measured Lead Values and Detection Limits for Method Blanks, Field Blanks and Field Paint-Chip Samples in the 39 Sample Preparation Batches Containing Primary Field Paint-Chip Sample Results for the Full and Pilot Studies.

Sample Type	Data Item	No. of Samples	Percentile					Percent ≥DL
			Min	25th	Median	75th	Max	
Method Blank	DL[a] µg/sample	79	2.13	3.25	4.30	5.94	8.91	16.5%
	Reported[b] Lead value µg/sample		2.13	3.47	5.10	6.09	8.91	
Field Blank	DL[a] µg/sample	31	2.13	2.46	3.60	3.60	8.67	32.2%
	Reported[b] Lead value µg/sample		2.13	2.75	3.60	5.95	8.70	
Field Paint-Chip Sample	DL[a] mg/cm^2	1290	0.0001	0.0005	0.0013	0.0030	0.096	95.8%
	Reported[b] Lead value mg/cm^2		0.0001	0.028	0.20	0.62	37.29	
	DL[a] percent by weight		0.0004	0.0010	0.0013	0.0028	0.11	
	Reported[b] Lead value percent by weight		0.0004	0.045	0.20	0.72	34.56	

[a] A sample specific detection limit, calculated using the ICP Instrumental Detection Limit (IDL) as follows:

DL µg/sample = [(IDL)(A)(B)]
DL mg/cm^2 = [(IDL)(A)(B)(C/D)]/[(1000)(E)]
DL percent by weight= [(IDL)(A)(B)(C/D)]/[(10000)(C)]

where:
IDL = 3 times the standard deviation of a minimum of 5 replicate lead results obtained from a 0.1 µg/mL lead standard measured during a given instrumental measurement batch (µg/mL, ranged from 0.0085 to 0.036).
A = final sample volume (250 ml)
B = additional dilution factors used to eliminate any potential interferant present in the sample (mL/mL, ranged from 1 to 100)
C = total collected sample mass (grams, ranged from 0.045 to 25.18)
D = subsample mass (grams, ranged from 0.029 to 0.56)
E = total collected sample area (cm^2, ranged from 19.6 to 35.19)

[b] Detection limit was used for non-detect samples.

4.4.2.2 Blind Samples

A blind sample is a sample submitted for analysis whose composition is known to the submitter but unknown to the analyst. Blind samples were included in the sample stream submitted to the laboratory to test the proficiency of the measurement process and to estimate the accuracy of the lead results obtained from field paint-chip samples. Blind samples were obtained from two sources: (1) National Institute of Standards and Technology (NIST), standard reference material (SRM) No. 1579a (lead-based paint), and (2) paint performance evaluation materials from rounds 01 and 02 prepared for the American Industrial Hygiene Association (AIHA) Environmental Lead Proficiency Analytical Testing (ELPAT) program. Blind samples were included from both of these sources because of concerns that the very high lead concentration in the NIST SRM (11.995%) would not be representative of the actual field paint-chip samples. The concentrations of lead in the ELPAT samples were determined by consensus testing. The consensus value, a mean concentration determined by reference laboratories selected by AIHA to be proficient in the analysis of lead-containing matrices, was used to calculate recoveries for this study. ELPAT samples used in this study included the following:

- Round 2, sample 1 - 0.2007% lead
- Round 2, sample 3 - 0.3809% lead

- Round 1, sample 2 - 0.5568% lead
- Round 1, sample 3 - 0.7026% lead
- Round 2, sample 2 - 3.2180% lead
- Round 2, sample 4 - 9.5536% lead

Additional information on the generation and use of blind samples is presented in Chapter 3.

For this study, planned data quality objectives for lead recoveries from blind samples were set to 75% to 125% of the known lead value. Lead recoveries from the blind samples were plotted on control charts to track the accuracy of laboratory processing. Warning limits were set at 80% and 120% and control limits at 75% and 125%. Duplicate ELPAT samples placed in each batch were used to estimate laboratory processing precision. The estimated precision also was plotted on control charts as a range of duplicate percent by weight lead recoveries calculated as the absolute difference between the lead recoveries of the ELPAT sample duplicates. The warning limit was set at 15% and the control limit at 20%.

Figures 4-30 and 4-31 graphically display the lead recoveries obtained for 39 NIST SRM samples and 77 ELPAT samples, respectively, that were processed among the 39 sample preparation batches containing primary field paint-chip samples for the full and pilot studies. Figure 4-32 graphically displays the range of percent recoveries between duplicate ELPAT samples processed in each sample preparation batch. A single ELPAT sample in one of the sample preparation batches was inadvertently lost during laboratory processing and is not plotted in either of the figures.

Figure 4-30 indicates that the data was in control and met data quality objectives for all NIST SRM samples except for four batches. Similarly, Figure 4-31 shows that the data was in-control and met data quality objectives for all ELPAT blinds except for one ELPAT sample. Figure 4-32 shows in-control data for ELPAT duplicates with one exception caused by the one high lead recovery ELPAT sample shown in Figure 4-31.

The sample preparation batch containing the out-of-control ELPAT sample, with a high lead recovery of 133.4%, had two other blind samples that were in-control: a duplicate ELPAT sample with 94.9% lead recovery and a NIST SRM sample with a 98.7% lead

recovery. These results, when combined with lack of problems with batches processed immediately before and after, suggest that the high ELPAT blind was a random event. However, processing of later batches included four batches with low NIST SRM sample lead recoveries ranging from 48.9% to 73.6%. These low recoveries are clustered together suggesting a potential systematic cause. Eight ELPAT samples processed in the same batches as those containing the low NIST SRM samples were in-control with lead recoveries ranging from 94.2% to 103.1%. A detailed investigation into procedures, personnel and other factors was inconclusive in determining a specific cause for the low NIST SRM sample recoveries in these four batches.

As a result of the four low NIST SRM sample recoveries discussed above, an assessment of the potential effect of the lower NIST SRM recoveries on field paint-chip samples was performed through additional sample preparation and analysis efforts in two sample preparation batches. The first sample preparation batch, labeled batch ZZZ, was entirely dedicated to the assessment of the potential effect of the low recoveries of lead in samples of NIST SRM 1579a. The second sample preparation batch, referred to as batch No. 734, was originally a partial batch containing the remaining samples from Philadelphia that had not been prepared or analyzed when the low NIST SRM samples were

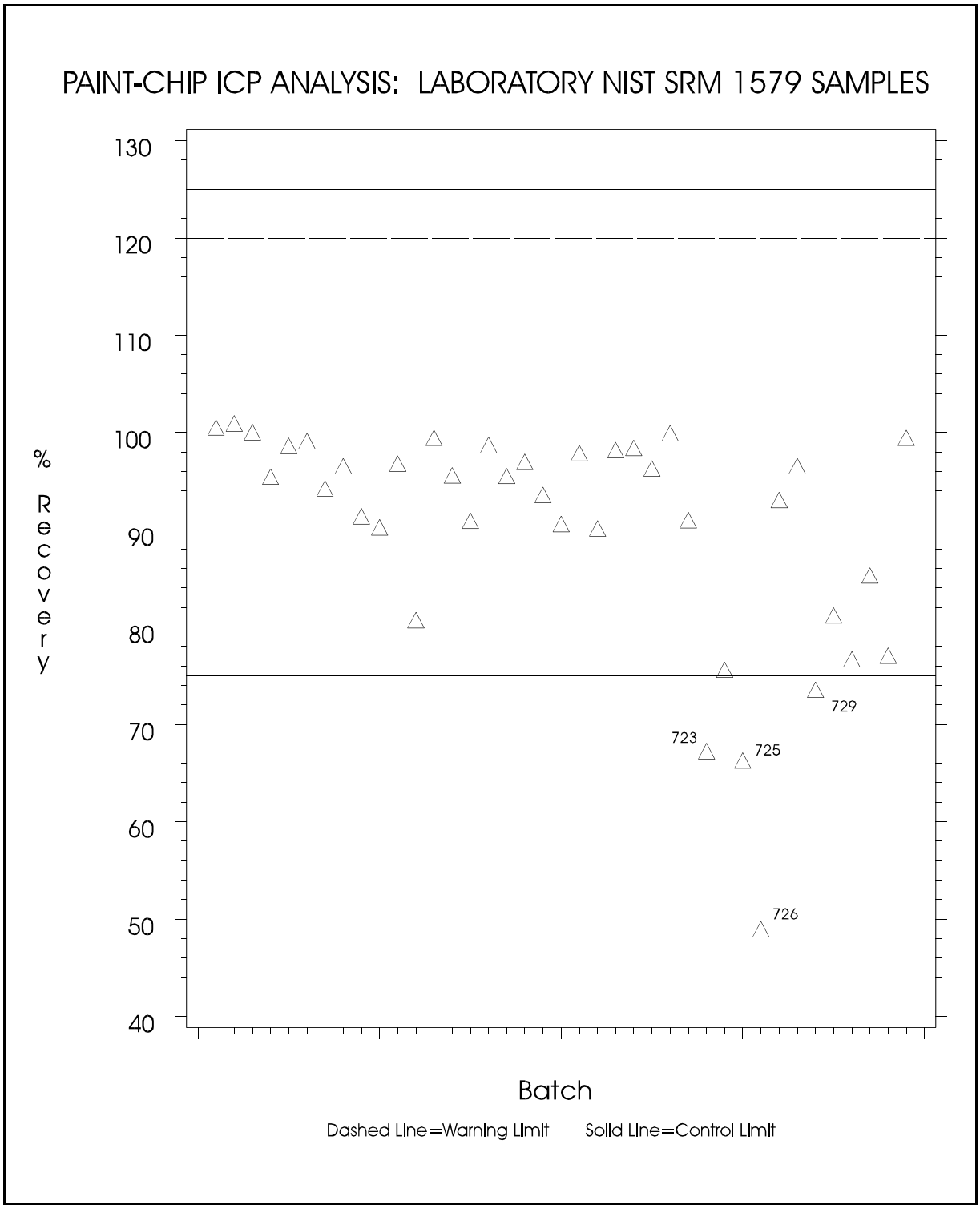

Figure 4-30. Summary of percent lead recoveries for NIST SRM 1579 samples in each sample preparation batch. Sample batch numbers are shown for results beyond the control limits.

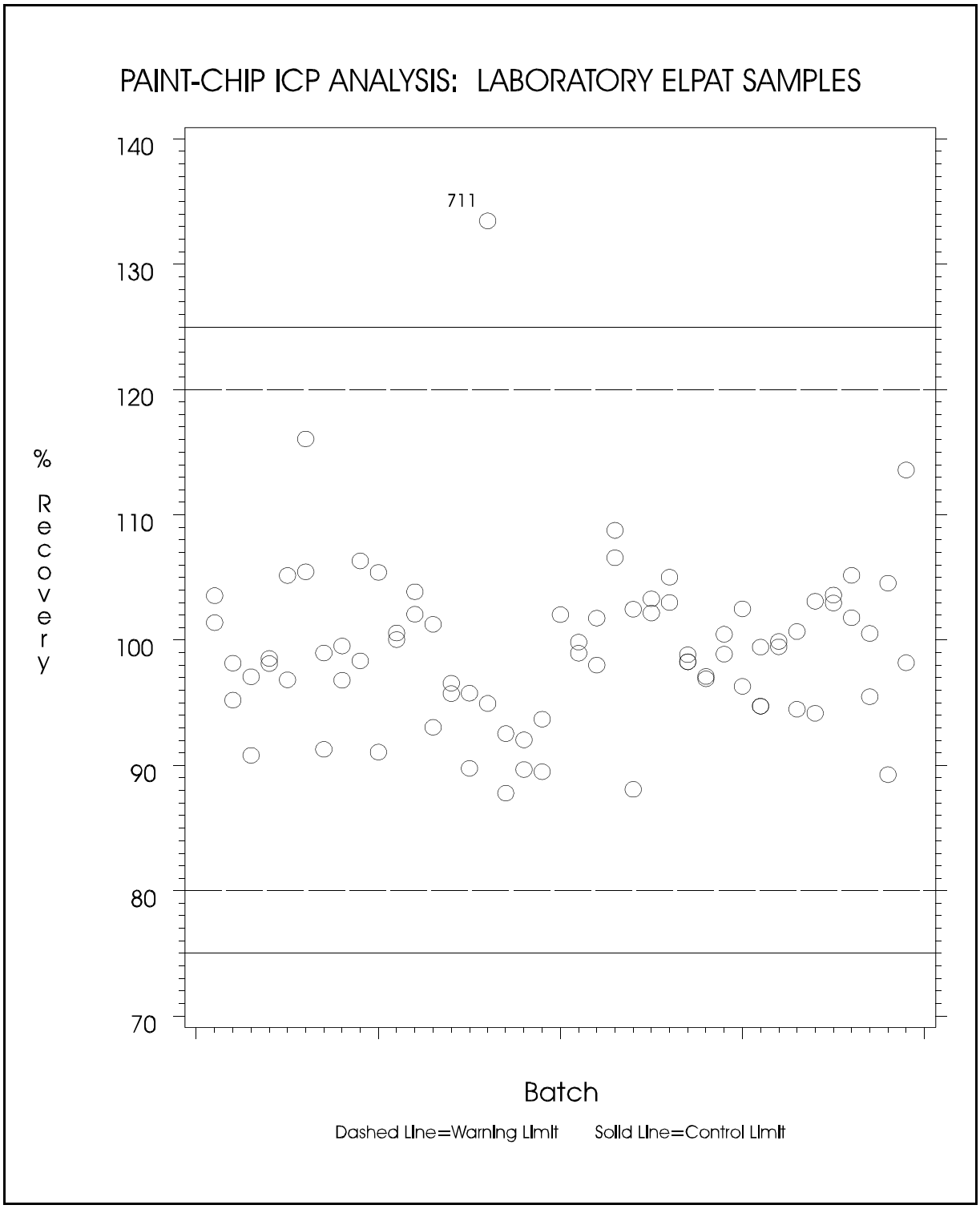

Figure 4-31. Summary of percent lead recoveries for ELPAT samples in each sample preparation batch. Sample batch numbers are shown for results beyond the control limits.

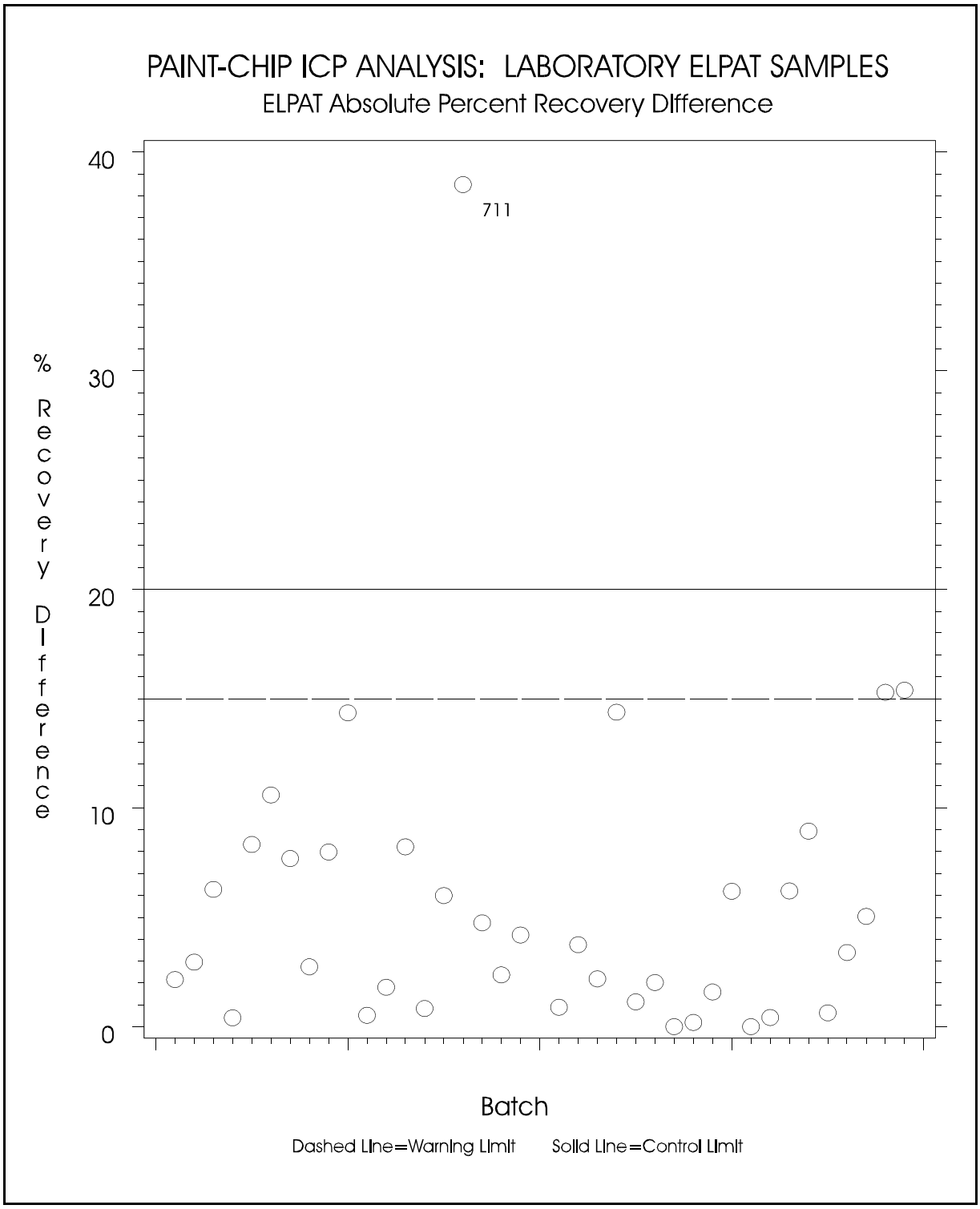

Figure 4-32. Absolute difference between percent lead recoveries for the pair of ELPAT samples in each sample preparation batch. Sample batch numbers are shown for results beyond the control limits.

identified. Extra sample space remaining in this batch was used to include subsamples of previously analyzed Philadelphia samples to supplement data obtained from batch ZZZ. Results from both these batches are discussed in the following two sections.

4.4.2.2.1 Results and Discussion of Investigation Samples in Sample Preparation Batch ZZZ

A test batch, called ZZZ, was assembled, prepared and analyzed to examine the differences in lead concentrations measured for field paint-chip samples analyzed in the four batches with low lead recovery data for the NIST SRM samples shown in Figure 4-30. The design included preparation of a single batch of samples by a single technician. The technician selected for the preparation task was one who performed the majority of the preparations for Denver samples, all of which had in-control NIST SRM sample recoveries.

Three types of samples were included in the design of test batch ZZZ: ELPAT samples, NIST SRM samples and primary field paint-chip samples. The ELPAT and NIST SRM samples were included to demonstrate control with respect to recovery of lead. Six ELPAT samples were included consisting of three duplicate pairs ranging in lead concentration from 0.3809 to 9.5536 percent by weight.

Nine NIST SRM samples were included in test batch ZZZ. These NIST SRM samples were assembled from opened and unopened original NIST SRM bottles and from residual NIST SRM containers used to submit the NIST SRM as blind samples during processing of the batches containing Philadelphia samples. NIST SRM samples from the different sources were included in batch ZZZ to help identify whether the low NIST SRM lead recoveries could have been caused by a physical problem with NIST SRM blind samples, such as inadvertent contamination or dilution of the SRM, as opposed to a laboratory processing problem.

Field paint-chip samples were included in test batch ZZZ to demonstrate the relationship between lead recovery from the NIST SRM samples and lead recovery for field paint-chip samples. Two groups of field paint-chip samples were included in the design: field samples from Philadelphia batch No. 726 containing a low NIST SRM sample recovery and field samples from sample batches having in-control NIST SRM sample recoveries. This design

provided a means of comparing recoveries of NIST SRM samples and field paint-chip samples previously analyzed in two types of batches, assuring that useful comparisons and conclusions could be drawn regardless of the NIST SRM sample status in test batch ZZZ. Field paint-chip samples from batch No. 726 were selected because it had the lowest NIST SRM sample lead recovery at 48.9%.

The selection of samples for test batch ZZZ was limited to those having sufficient sample mass to permit additional subsampling. Additional selection criteria included substrate type and estimated lead level. Field paint-chip samples from sample preparation batches that were in control were selected from Louisville batches which had NIST SRM sample recoveries ranging from 95.5% to 100.9%. Since statistical analysis had been completed on the Louisville sample data at the time of this investigation, these samples were considered to be more expendable than Philadelphia or Denver samples.

Lead recoveries for all ELPAT and NIST SRM samples in test batch ZZZ were in control, ranging from 96.8% to 105.3% and 96.7% to 99.7% for the ELPAT samples and NIST SRM samples, respectively. These results suggest that there were no differences between the different sources of the NIST SRM samples included in batch ZZZ.

Tables 4-32 and 4-33, which contain field paint-chip sample results for test batch ZZZ, show good agreement between batch ZZZ and original lead results for samples in batch No. 726 and the original lead results for matching samples in the Louisville batches. Paired *Student's t* tests, pairing the lead concentration results of the original analyses to those of batch ZZZ analyses, indicate no significant differences between the results for either the mg/cm^2 units or percent by weight units. The variability between the original and ZZZ mg/cm^2 levels on the log scale is estimated as 0.157 for Philadelphia and 0.231 for Louisville (1 standard deviation), using the statistical approach described in section 4.3. As for all laboratory duplicates, variability on the log scale is the same in area units and percent by weight units. The estimates of variability are higher than the overall estimates of analytical measurement variability for these two cities reported in section 4.3 (0.126 for

Philadelphia and 0.118 for Louisville). The difference is statistically significant for Louisville samples but not

statistically significant for the Philadelphia samples based on an F-test. Higher variability was to be expected from test batch ZZZ since it includes the effect of variation between batches, whereas the results for overall analytical measurement error are based on paired subsamples, which were usually analyzed in the same batch. It is concluded that there are no unusual

Table 4-32. Summary of Lead Results for Selected Philadelphia Samples Included in Test Batch ZZZ.

SAMPLE ID	SAMPLE TYPE	LEAD CONCENTRATION RESULTS FOR BATCH ZZZ		ORIGINAL LEAD CONCENTRATION RESULTS		
		Percent by Weight	mg/cm²	BATCH No.	Percent by Weight	mg/cm²
1341	Plaster	0.307	0.376	726	0.274	0.337
1344	Plaster	0.383	0.364	726	0.382	0.363
1345	Plaster	0.420	0.558	726	0.324	0.431
1347	Plaster	0.389	0.508	726	0.356	0.465
1410	Concrete	0.245	0.494	726	0.280	0.564
1411	Concrete	0.153	0.334	726	0.182	0.397
1412	Concrete	0.466	1.014	726	0.364	0.794
1413	Concrete	0.750	1.342	726	0.939	1.680
1414	Concrete	0.366	0.740	726	0.380	0.768
1449	Metal	0.478	0.625	726	0.530	0.693
1534	Metal	0.415	0.544	726	0.596	0.782
1536	Metal	0.110	0.122	726	0.114	0.126
1537	Metal	0.579	0.607	726	0.458	0.481
1539	Metal	0.430	0.516	726	0.507	0.608
1820	Concrete	0.109	0.132	726	0.179	0.217
1824	Plaster	0.149	0.350	726	0.127	0.298
1827	Plaster	0.160	0.272	726	0.118	0.201
AVERAGES		0.348	0.523	-	0.359	0.541

differences between the ZZZ and original results beyond what would be expected from analytical measurement error and batch-to-batch variation. Furthermore, the Philadelphia sample data can be used to draw conclusions related to the potential impact of low NIST SRM sample recoveries on field paint-chip samples. If the original lead recovery of the NIST SRM sample in batch No. 726, at 48.9%, was reflective of lead recoveries from field paint-chip samples, then lead results for field samples in test batch ZZZ would have been expected to be much higher than those in batch No. 726. However, the arithmetic mean lead level from the 17 Philadelphia samples in batch ZZZ is slightly lower than

that obtained from matching samples in batch No. 726 (3.48 mg/g versus 3.59 mg/g, and 0.52 mg/cm^2 versus 0.54 mg/cm^2, for batch

Table 4-33. Summary of Lead Concentration Results for Selected Louisville Samples Included in Test Batch ZZZ.

SAMPLE ID	SAMPLE TYPE[a]	LEAD CONCENTRATION RESULTS FOR BATCH ZZZ		ORIGINAL LEAD CONCENTRATION RESULTS		
		Percent by Weight	mg/cm²	BATCH No.	Percent by Weight	mg/cm²
523	Plaster	3.251	3.922	XDF	4.587	5.533
524	Plaster	0.536	0.499	XDI	0.518	0.482
531	Concrete	0.228	0.241	XDF	0.241	0.256
533	Concrete	0.136	0.224	XDF	0.152	0.251
535DUP	Metal	1.130	0.917	XDJ	1.167	0.910
541	Metal	0.745	0.708	XDF	0.475	0.451
586	Plaster	0.435	0.324	XDJ	0.344	0.256
592	Plaster	3.483	4.360	XDJ	2.670	3.342
593	Plaster	0.129	0.126	XDJ	0.130	0.127
596	Concrete	1.818	1.529	XDJ	3.014	2.534
597	Concrete	0.235	0.243	XDJ	0.223	0.230
604	Metal	0.178	0.207	XDJ	0.165	0.191
606	Metal	0.603	0.503	XDJ	0.853	0.712
607	Metal	0.730	0.731	XDI	0.382	0.382
596DUP	Concrete	3.616	3.161	XDG	2.005	1.788
AVERAGES		1.150	1.180	-	1.128	1.163

ZZZ and batch No. 726, respectively). The low NIST SRM sample recovery of 48.9% clearly indicated that a processing problem with batch No. 726 did occur. However, lead results on the additional aliquots of field paint-chip samples from this Philadelphia batch processed under in-control conditions in batch ZZZ indicate that field sample results were not changed as a result of the difference in control status between the two batches. This suggests that the NIST SRM sample was not particularly representative of the field paint-chip samples contained within the original Philadelphia batch No. 726, and thus, was not an effective measure of the accuracy of lead results for this batch.

Since the results for test batch ZZZ indicated that original field paint-chip sample results in batch No. 726 were not affected by the low NIST SRM sample recovery in this batch, it was concluded that samples in the other three Philadelphia batches with low NIST SRM sample recoveries were likewise not affected. Additional investigations to support this conclusion are discussed in the following section.

4.4.2.2.2 Results and Discussion of Investigative Samples in Sample Preparation Batch No. 734

As discussed previously, the final sample preparation batch, No. 734, from Philadelphia was originally a partial batch containing only those samples needed to complete the remaining samples that had yet to be prepared and analyzed. Because of fixed costs involved with batch processing of samples, a total of 22 samples could be added to batch No. 734 with only minimal additional effort. Therefore, it was decided to use this opportunity to include additional samples in this batch to confirm the conclusion suggested by the results from test batch ZZZ. This conclusion was that the low recoveries of lead in the NIST SRM samples in the four (4) Philadelphia sample batches had apparently <u>not</u> resulted in low recoveries for the actual field paint-chip samples in these batches. Low lead NIST SRM sample recoveries were measured at 67.2%, 66.3%, 48.9%, and 73.6% in Philadelphia batch Nos. 723, 725, 726, and 729, respectively.

The additional 22 samples added to sample batch No. 734 included replicate aliquots of field paint-chip samples from batch Nos. 723, 725, 729, and 733. Since there was a lack of available sample material after completing preparation and analysis of test batch ZZZ, inclusion of additional sample aliquots from batch No. 726 was not possible. Therefore, an alternative batch was selected for inclusion. This was No. 733, which also had a low, but in-control, original NIST SRM lead recovery at 77.1%. Processing of all sample batches in the study included preparation and analysis of four (4) laboratory duplicates per batch. The original laboratory duplicate samples from the four targeted batches (batch Nos. 723, 725, 729, and 733) were selected for batch 734, making the number of additions to batch No. 734 equal to 16. One sample of the four (4) samples from each of the targeted batches was selected at random and processed as a laboratory duplicate in batch No. 734 increasing the total number of sample additions to 20. The remaining two

(2) sample positions within the batch were filled by random selection from those samples left in the targeted batches that had sufficient sample material remaining to permit removal and processing of an additional aliquot.

Table 4-34 shows the reanalysis results. The arithmetic mean lead level of the 22 reanalyses was 0.80 mg/cm^2, as compared to 0.74 mg/cm^2 originally; geometric means were 0.31 mg/cm^2 for the reanalyses and 0.33 mg/cm^2 for the original analyses. Neither difference was statistically significant. The reanalysis median was 0.42 mg/cm^2 as compared to 0.43 mg/cm^2 originally. The conclusion is that there is no significant difference between the original analysis results and the results of reanalyses in batch No. 734.

The variability between original and batch 734 lead levels measured on the log scale (using either mg/cm^2 or percent units) was 0.180, as compared to analytical measurement variability of 0.126 for the Philadelphia samples as a whole. The difference was not statistically significant, based on an F-test. The higher observed variation in the batch 734 results was probably due to inter-batch variability, which was not accounted for in the 0.126 variability estimate.

The batch 734 results confirm that the low recoveries of the NIST SRM samples in batches No. 723, 725, 729 and 733 had no systematic effect on the recoveries of study samples in these batches.

Table 4-34. Batch 734 Reanalyses Compared to Original Results.

SAMPLE ID	LEAD RESULTS FROM BATCH No. 734		LEAD RESULTS FROM ORIGINAL BATCHES		SUBSTRATE TYPE	ORIGINAL BATCH No.
	mg/cm²	PERCENT BY WEIGHT	mg/cm²	PERCENT BY WEIGHT		
1213	5.1174	3.4683	3.6850	2.4975	metal	729
1255	0.0552	0.0117	0.1889	0.0606	plaster	733
1255DUP	0.0951	0.0305	0.1182	0.0379	plaster	733
1318	0.0064	0.0027	0.0039	0.0017	brick	723
1335	0.6028	0.3536	0.4393	0.0577	brick	725
1409	0.6491	0.3401	0.5718	0.2996	concrete	723
1425	1.5011	0.6856	1.2827	0.5859	plaster	725
1425DUP	1.1635	0.5314	1.3653	0.6236	plaster	725
1510	0.5170	0.3267	0.4209	0.2660	concrete	723
1557	0.2565	0.1812	0.3514	0.2482	wood	725
1607	0.5448	0.2093	0.5157	0.1981	plaster	729
1607DUP	0.4349	0.1670	0.5169	0.1985	plaster	729
1643	0.3992	0.1941	0.7093	0.3449	brick	733
1646	0.3997	0.1678	0.3130	0.1314	concrete	733
1736	0.0058	0.0027	0.0078	0.0036	concrete	733
1745	0.6906	0.3401	0.7352	0.3620	plaster	729
1812	0.2409	0.1246	0.2004	0.1037	concrete	723
1812DUP	0.2878	0.1486	0.3019	0.1562	concrete	723
1839	0.0873	0.0717	0.0693	0.0569	plaster	725
1846	0.1386	0.0962	0.1730	0.1202	metal	725
1910	1.8053	1.0886	1.7459	1.0528	concrete	733
1920	2.5121	1.3750	2.6400	1.4449	plaster	729

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Chapter 5 Summary: Analysis of Test Kit Data

- None of the test kits used in this study demonstrated low rates of both false positive and false negative results when compared to laboratory analytical results using the federal thresholds, 1.0 mg/cm^2 and 0.5%.

- The substrate underlying the paint sometimes affected false positive and false negative rates for test kits.

- The probability of a positive classification when the sample's lead level was equal to the federal thresholds varied depending on the kit and substrate. High levels of lead would not always be detected using test kits alone.

- The lead level at which there was a 50% chance of the occurrence of a positive test kit result varied depending on the kit and substrate. In many cases, positive results occurred even when paint with very low lead levels was tested.

5 ANALYSIS OF TEST KIT DATA

This chapter provides analysis results and discussion of test kit performance and addresses the study objective to characterize the relationship between test kit results and the actual lead level in the paint. Section 5.1 presents descriptive statistics on false positive and false negative rates when compared to the ICP measurements classified as positive and negative, by city and substrate and overall, for the federal standards of 1.0 mg/cm^2 and 0.5% by weight. Section 5.2 provides estimates of operating characteristic (OC) curves of the test kits. An OC curve for a test kit shows the probability that the test kit gives a positive reading on paint having true lead concentration at some fixed level. Section 5.3 provides additional analysis results and discussion for the two sodium sulfide type test kits. For only these two kits, the degree of shading for positive results was collected, which may range from light grey to black.

5.1 DESCRIPTIVE STATISTICS ON FALSE POSITIVE AND FALSE NEGATIVE RATES FOR DIFFERENT STANDARDS

The results presented in this section addresses the study objective to characterize the relationship between test kit results and the actual lead level in the paint. As discussed in previous chapters, paint samples were collected at a total of 1,290 sampling locations in the 3 cities. Five of the six test kits were attempted at every sampling location, and results were obtained in all but a few cases. For the Lead Alert: Sanding kit, only a subset of locations in Denver and Philadelphia were tested because of the length of time necessary to complete tests with this kit. Also, data from this kit from Louisville was not included since there was a substantial change in the protocol from the pilot in Louisville to the full study. Simulated "homeowners" were selected with no prior test kit experience to apply the test kits as was discussed in section 3.4.2.3.

Table 5-1 shows the percentages of ICP measurements classified as positive and negative relative to the federal standard of 1.0 mg/cm^2, by city and substrate and overall. An ICP measurement is classified as positive if it equals or exceeds 1.0 mg/cm^2, and is classified as negative if it is less than 1.0 mg/cm^2. Table 5-2 shows the same percentages for the sampling locations where Lead Alert: Sanding testing was performed.

Tables 5-3 and 5-4 present the same information for the alternative standard of 0.5% lead by weight.

Table 5-1. Positive (≥ 1.0 mg/cm^2) and Negative (< 1.0 mg/cm^2) Percentages for ICP Measurements at All Sampling Locations by City and Substrate and Overall.

SUBSTRATE	DENVER			PHILADELPHIA			LOUISVILLE			ALL CITIES		
	N[a]	Pos	Neg	N[a]	Pos	Neg	N[a]	Pos	Neg	N[a]	Pos	Neg
Brick	81	26%	74%	12	0%	100%	N/A	N/A	N/A	93	23%	77%
Concrete	98	19%	81%	120	7%	93%	8	25%	75%	226	13%	87%
Drywall	105	0%	100%	8	0%	100%	11	0%	100%	124	0%	100%
Metal	62	19%	81%	127	25%	75%	28	14%	86%	217	22%	78%
Plaster	101	14%	86%	121	10%	90%	20	35%	65%	242	14%	86%
Wood	303	27%	73%	52	38%	62%	33	61%	39%	388	31%	69%
All	750	20%	80%	440	16%	84%	100	33%	67%	1290	20%	80%

[a] Number of sampling locations.

Table 5-2. Positive (≥ 1.0 mg/cm^2) and Negative (< 1.0 mg/cm^2) Percentages for ICP Measurements at Sampling Locations Where Lead Alert: Sanding Testing Was Performed by City and Substrate and Overall.

SUBSTRATE	DENVER			PHILADELPHIA			BOTH CITIES		
	N[a]	Pos	Neg	N[a]	Pos	Neg	N[a]	Pos	Neg
Brick	23	17%	83%	12	0%	100%	35	11%	89%
Concrete	38	18%	82%	87	9%	91%	125	12%	88%
Drywall	40	0%	100%	8	0%	100%	48	0%	100%
Metal	33	27%	73%	87	32%	68%	120	31%	69%
Plaster	40	10%	90%	78	9%	91%	118	9%	91%
Wood	89	34%	66%	44	38%	62%	133	35%	65%
All	263	21%	79%	316	19%	81%	579	20%	80%

[a] Number of sampling locations.

Table 5-3. Positive (≥ 0.5 %) and Negative (< 0.5 %) Percentages for ICP Measurements at All Sampling Locations by City and Substrate and Overall.

SUBSTRATE	DENVER			PHILADELPHIA			LOUISVILLE			ALL CITIES		
	N[a]	Pos	Neg	N[a]	Pos	Neg	N[a]	Pos	Neg	N[a]	Pos	Neg
Brick	81	28%	72%	12	0%	100%	N/A	N/A	N/A	93	25%	75%
Concrete	98	20%	80%	120	8%	92%	8	37%	63%	226	15%	85%
Drywall	105	10%	90%	8	0%	100%	11	0%	100%	124	8%	92%
Metal	62	39%	61%	127	46%	54%	28	46%	54%	217	44%	56%
Plaster	101	17%	83%	121	10%	90%	20	50%	50%	242	16%	84%
Wood	303	38%	62%	52	67%	33%	33	64%	36%	388	44%	56%
All	750	28%	72%	440	26%	74%	100	47%	53%	1290	29%	71%

[a] Number of sampling locations.

Table 5-4. Positive (≥ 0.5 %) and Negative (< 0.5 %) Percentages for ICP Measurements at Sampling Locations Where Lead Alert: Sanding Testing Was Performed by City and Substrate and Overall.

SUBSTRATE	DENVER			PHILADELPHIA			BOTH CITIES		
	N[a]	Pos	Neg	N[a]	Pos	Neg	N[a]	Pos	Neg
Brick	23	22%	78%	12	0%	100%	35	14%	86%
Concrete	38	18%	82%	87	11%	89%	125	14%	86%
Drywall	40	5%	95%	8	0%	100%	48	4%	96%
Metal	33	45%	55%	87	55%	45%	120	53%	47%
Plaster	40	15%	85%	78	9%	91%	118	11%	89%
Wood	89	47%	53%	44	67%	33%	133	54%	46%
All	263	29%	71%	316	30%	70%	579	30%	70%

[a] Number of sampling locations.

Table 5-1 shows that, overall, Louisville had the highest percentage of positive results relative to the 1.0 mg/cm^2 standard (33%) followed by Denver (20%) and Philadelphia (16%).

There was considerable variation among substrates and sometimes between cities for the same substrate. Out of 124 tests, no positive results were found for drywall. Concrete and brick had much lower positive percentages in Philadelphia than in the other cities. Table 5-2, which displays ICP results from locations where the Lead Alert: Sanding test kit was performed, shows a very similar pattern to the ICP results from all sampling locations shown in Table 5-1. Results for the 0.5% standard are shown in Tables 5-3 and 5-4. Note that the results in Tables 5-3 and 5-4 for all substrate data combined (shown in the "ALL" entry in the tables) show about the same variations between cities and substrates as do Tables 5-1 and 5-2. However, differences can be observed when substrate comparisons are made between the cities. For example, the percentages for metal in Tables 5-1 and 5-2 changed noticeably compared to metal percentages in Tables 5-3 and 5-4. Also, the percentages for wood in Philadelphia are nearly reversed. In general, the positive percentages are higher than for the 1.0 mg/cm^2 standard, reflecting the fact that 0.5% lead is usually a lower level than 1.0 mg/cm^2 (see section 4.1.2). For example, 8% of drywall samples were positive against the 0.5% standard, as compared to 0% for the 1.0 mg/cm^2 standard.

A <u>false negative</u> with a test kit is defined as an ICP measurement of 1.0 mg/cm^2 or greater for which the kit gives a negative result. A <u>false positive</u> is, conversely, an ICP measurement less than 1.0 mg/cm^2 for which the kit gives a positive result. A test kit's false positive <u>rate</u> is the proportion of ICP measurements less than 1.0 mg/cm^2 for which the kit tested positive. A test kit's false negative <u>rate</u> is the proportion of ICP measurements greater than or equal to 1.0 mg/cm^2 for which the kit tested negative. Tables 5-5 through 5-10 give, by city and substrate and overall, the false positive and false negative rates observed in the study for the 1.0 mg/cm^2 standard. Tables 5-11 through 5-16 give the corresponding rates for the 0.5% standard. For the Lead Alert: Sanding and Lead Alert: Coring kits, an additional line is included giving the overall results for all substrates except plaster. Statements for these two test kits will exclude the plaster data.

In most cases, the classification of test kit results

compared to the 1.0 mg/cm^2 standard is similar to the classification compared to the 0.5% by weight standard. However, some differences do exist. This is because the kits vary widely

Table 5-5. False Positive and False Negative Rates for **LeadCheck** by City and Substrate and Overall (1.0 mg/cm^2 Standard).

SUBSTRATE	DENVER		PHILADELPHIA		LOUISVILLE		ALL CITIES	
	FP %	FN %	FP %	FN %	FP %	FN %	FP %	FN %
Brick	52%	5%	58%	N/A	N/A	N/A	53%	5%
Concrete	38%	0%	39%	50%	100%	0.0%	41%	14%
Drywall	23%	N/A	25%	N/A	0%	N/A	21%	N/A
Metal	44%	0%	37%	6%	92%	0.0%	47%	4%
Plaster	62%	0%	35%	33%	92%	0.0%	50%	12%
Wood	58%	1%	63%	10%	38%	0.0%	58%	2%
All	48%	1%	40%	17%	67%	0.0%	46%	6%

Table 5-6. False Positive and False Negative Rates for **Lead Alert: Coring** by City and Substrate and Overall (1.0 mg/cm² Standard).

SUBSTRATE	**DENVER**		**PHILADELPHIA**		**LOUISVILLE**		**ALL CITIES**	
	FP %	**FN %**	**FP %**	**FN %**	**FP %**	**FN %**	**FP %**	**FN %**
Brick	33%	5%	42%	N/A	N/A	N/A	35%	5%
Concrete	10%	5%	10%	88%	50%	0%	11%	28%
Drywall	5%	N/A	0%	N/A	0%	N/A	4%	N/A
Metal	24%	8%	13%	38%	46%	0%	21%	27%
Plaster	2%	79%	6%	92%	23%	86%	5%	85%
Wood	15%	21%	6%	55%	0%	10%	14%	25%
All	14%	21%	10%	57%	25%	24%	13%	32%
All Except Plaster	15%	15%	12%	50%	26%	8%	15%	24%

Table 5-7. False Positive and False Negative Rates for **Lead Alert: Sanding** by City and Substrate and Overall (1.0 mg/cm^2 Standard).

SUBSTRATE	DENVER		PHILADELPHIA		BOTH CITIES	
	FP %	FN %	FP %	FN %	FP %	FN %
Brick	26%	75%	17%	N/A	23%	N/A[a]
Concrete	0%	29%	18%	63%	13%	47%
Drywall	5%	N/A	0%	N/A	4%	N/A
Metal	17%	33%	7%	64%	10%	57%
Plaster	3%	75%	6%	100%	5%	91%
Wood	3%	30%	0%	88%	2%	50%
All	7%	37%	9%	75%	8%	57%
All Except Plaster	8%	34%	11%	71%	9%	53%

[a] Sample size is too small to accurately compute.

Table 5-8. False Positive and False Negative Rates for **Lead Detective** by City and Substrate and Overall (1.0 mg/cm^2 Standard).

SUBSTRATE	**DENVER**		**PHILADELPHIA**		**LOUISVILLE**		**ALL CITIES**	
	FP %	**FN %**	**FP %**	**FN %**	**FP %**	**FN %**	**FP %**	**FN %**
Brick	58%	43%	42%	N/A	N/A	N/A	55%	43%
Concrete	35%	47%	37%	63%	83%	0%	38%	48%
Drywall	30%	N/A	0%	N/A	45%	N/A	29%	N/A
Metal	33%	17%	26%	28%	71%	50%	34%	27%
Plaster	51%	57%	21%	25%	31%	0%	34%	33%
Wood	35%	12%	38%	5%	23%	5%	35%	10%
All	39%	26%	29%	25%	51%	9%	36%	23%

Table 5-9. False Positive and False Negative Rates for **Lead Zone** by City and Substrate and Overall (1.0 mg/cm^2 Standard).

SUBSTRATE	**DENVER**		**PHILADELPHIA**		**LOUISVILLE**		**ALL CITIES**	
	FP %	**FN %**	**FP %**	**FN %**	**FP %**	**FN %**	**FP %**	**FN %**
Brick	40%	5%	25%	N/A	N/A	N/A	38%	5%
Concrete	29%	0.0%	22%	38%	0%	0%	24%	10%
Drywall	26%	N/A	0%	N/A	0%	N/A	22%	N/A
Metal	32%	25%	16%	22%	43%	0%	24%	21%
Plaster	20%	36%	32%	17%	23%	71%	26%	36%
Wood	36%	2%	44%	20%	0%	20%	35%	8%
All	31%	7%	25%	22%	20%	27%	28%	14%

Table 5-10. False Positive and False Negative Rates for **State Sodium Sulfide** by City and Substrate and Overall (1.0 mg/cm² Standard).

SUBSTRATE	**DENVER**		**PHILADELPHIA**		**LOUISVILLE**		**ALL CITIES**	
	FP %	**FN %**	**FP %**	**FN %**	**FP %**	**FN %**	**FP %**	**FN %**
Brick	67%	0%	67%	N/A	N/A	N/A	67%	0%
Concrete	42%	5%	95%	0%	33%	0%	72%	3%
Drywall	45%	N/A	0%	N/A	0%	N/A	38%	N/A
Metal	20%	17%	95%	0%	33%	0%	64%	4%
Plaster	67%	0%	98%	0%	15%	0%	80%	0%
Wood	57%	0%	84%	0%	38%	0%	59%	0%
All	52%	2%	92%	0%	25%	0%	65%	1%

Table 5-11. False Positive and False Negative Rates for **LeadCheck** by City and Substrate and Overall (0.5% Standard).

SUBSTRATE	**DENVER**		**PHILADELPHIA**		**LOUISVILLE**		**ALL CITIES**	
	FP %	**FN %**	**FP %**	**FN %**	**FP %**	**FN %**	**FP %**	**FN %**
Brick	50%	4%	58%	N/A	N/A	N/A	51%	4%
Concrete	37%	0%	39%	50%	100%	0%	40%	15%
Drywall	17%	20%	25%	N/A	0%	N/A	16%	20%
Metal	37%	17%	32%	27%	87%	0%	41%	21%
Plaster	61%	0%	35%	33%	100%	10%	49%	13%
Wood	52%	3%	35%	9%	42%	5%	50%	5%
All	44%	5%	36%	24%	62%	4%	42%	11%

Table 5-12. False Positive and False Negative Rates for **Lead Alert: Coring** by City and Substrate and Overall (0.5% Standard).

SUBSTRATE	**DENVER**		**PHILADELPHIA**		**LOUISVILLE**		**ALL CITIES**	
	FP %	**FN %**	**FP %**	**FN %**	**FP %**	**FN %**	**FP %**	**FN %**
Brick	33%	9%	42%	N/A	N/A	N/A	34%	9%
Concrete	10%	10%	10%	90%	40%	0%	11%	33%
Drywall	3%	80%	0%	N/A	0%	N/A	3%	80%
Metal	16%	29%	12%	59%	20%	8%	14%	45%
Plaster	2%	82%	6%	92%	20%	80%	5%	85%
Wood	6%	24%	6%	71%	0%	14%	6%	32%
All	9%	29%	10%	69%	13%	26%	10%	41%
All Except Plaster	10%	24%	12%	66%	12%	11%	11%	36%

Table 5-13. False Positive and False Negative Rates for **Lead Alert: Sanding** by City and Substrate and Overall (0.5% Standard).

SUBSTRATE	DENVER		PHILADELPHIA		BOTH CITIES	
	FP %	FN %	FP %	FN %	FP %	FN %
Brick	28%	80%	17%	N/A	23%	N/A[a]
Concrete	0%	29%	18%	70%	13%	53%
Drywall	5%	100%	0%	N/A	4%	100%
Metal	6%	40%	8%	77%	7%	68%
Plaster	0%	67%	6%	100%	4%	85%
Wood	4%	50%	0%	93%	3%	68%
All	5%	51%	10%	83%	8%	68%
All Except Plaster	7%	49%	13%	82%	10%	67%

[a] Sample size is too small to accurately compute.

Table 5-14. False Positive and False Negative Rates for **Lead Detective** by City and Substrate and Overall (0.5% Standard).

SUBSTRATE	**DENVER**		**PHILADELPHIA**		**LOUISVILLE**		**ALL CITIES**	
	FP %	**FN %**	**FP %**	**FN %**	**FP %**	**FN %**	**FP %**	**FN %**
Brick	57%	39%	42%	N/A	N/A	N/A	54%	39%
Concrete	35%	45%	36%	60%	80%	0%	37%	45%
Drywall	28%	50%	0%	N/A	46%	N/A	27%	50%
Metal	19%	21%	18%	41%	67%	31%	24%	34%
Plaster	49%	47%	21%	25%	30%	20%	33%	33%
Wood	29%	17%	6%	14%	17%	5%	27%	15%
All	35%	27%	25%	33%	45%	15%	32%	27%

Table 5-15. False Positive and False Negative Rates for **Lead Zone** by City and Substrate and Overall (0.5% Standard).

SUBSTRATE	**DENVER**		**PHILADELPHIA**		**LOUISVILLE**		**ALL CITIES**	
	FP %	**FN %**	**FP %**	**FN %**	**FP %**	**FN %**	**FP %**	**FN %**
Brick	38%	4%	25%	N/A	N/A	N/A	36%	4%
Concrete	28%	0%	23%	50%	0%	33%	24%	18%
Drywall	22%	40%	0%	N/A	0%	N/A	19%	40%
Metal	24%	33%	13%	47%	36%	31%	19%	42%
Plaster	18%	35%	32%	17%	20%	70%	26%	38%
Wood	29%	9%	35%	31%	0%	24%	28%	15%
All	27%	14%	24%	40%	14%	36%	25%	25%

Table 5-16. False Positive and False Negative Rates for **State Sodium Sulfide** by City and Substrate and Overall (0.5% Standard).

SUBSTRATE	**DENVER**		**PHILADELPHIA**		**LOUISVILLE**		**ALL CITIES**	
	FP %	**FN %**	**FP %**	**FN %**	**FP %**	**FN %**	**FP %**	**FN %**
Brick	66%	0%	67%	N/A	N/A	N/A	66%	0%
Concrete	42%	5%	95%	0%	20%	0%	71%	3%
Drywall	42%	30%	0%	N/A	0%	N/A	35%	30%
Metal	11%	33%	93%	0%	20%	31%	58%	13%
Plaster	67%	6%	98%	0%	20%	30%	81%	10%
Wood	50%	1%	71%	0%	42%	5%	51%	1%
All	49%	7%	91%	0%	21%	17%	62%	6%

in their performance against either standard and also because differences do exist between the two standards as noted above (by comparing Tables 5-1 and 5-2 to 5-3 and 5-4). The differences between the 1.0 mg/cm^2 standard and 0.5% standard and other observations from Tables 5-5 through 5-16 are discussed below.

Relative to the 1.0 mg/cm^2 standard, State Sodium Sulfide and LeadCheck have, overall, false negative rates less than 10% and false positive rates of 46% or greater. Lead Zone, Lead Detective and Lead Alert: Coring have rates of both false positive and false negative that range from 14% to 36%. Lead Alert: Sanding has a false positive rate less than 10% but a false negative rate greater than 50%.

Relative to the 0.5% standard, State Sodium Sulfide has a 6% false negative rate, while Lead Alert: Sanding has a 10% false positive rate. False negative rates for LeadCheck and false positive rates for Lead Alert: Coring are just over 10%. Lead Zone and Lead Detective both have false positive and false negative rates ranging from 25% to 32%. These results indicate that no single kit can achieve error rates less than 10% for both types of errors simultaneously.

The manufacturer's substrate interference warning against the use of Lead Alert: Sanding and Lead Alert: Coring on plaster substrates is borne out by the data. Compared to the 1.0 mg/cm^2 standard, individually, the two Lead Alert test kit results showed false negative rates on plaster of 85% or greater. However, results from the other rhodizonate based kit, LeadCheck, did not show unusually high false negative rates on plaster. Compared to the 1.0 mg/cm^2 standard, the false negative rate for the LeadCheck test kit was 12%. False positive and false negative rates relative to the 0.5% by weight standard on plaster were similar.

Some differences in performance between cities are clear from the tables. Results for LeadCheck, Lead Alert: Sanding (excluding Louisville data), and Lead Alert: Coring show much higher false negative rates in Philadelphia than in Denver and Louisville. The results from the State Sodium Sulfide test kit show a much higher false positive rate in Philadelphia than in the other cities. Also, LeadCheck and Lead Detective had results with noticeably higher false positive rates in Louisville than in the other cities. There are many possible explanations for these

differences, including variation in paint, lead levels and substrate composition between cities, and differences between testers.

There are, in many cases, large differences in performance by substrate, which argue for treating substrates separately in the analysis.

In a previous section (section 3.4), test kit protocols for this study were described. Recall that the Louisville protocol for Lead Detective was changed in Denver and Philadelphia to facilitate application of the solution to the painted surface. Tables 5-8 and 5-14 compare the performance of Lead Detective in Louisville and the full study. The differences are not consistent; the Louisville data has the lower false negative rates while the full study data has the lower false positive rates.

The above observations are based on descriptive statistics. Section 5.2 presents more detailed analyses relating the performance of the test kits to the lead level, by means of an operating characteristic curve model.

5.1.1 The Effect of Spatial Variation and Laboratory Error on ICP-Based Classification Rates

The false positive and false negative rates presented in Tables 5-5 through 5-10 are relative to a 1.0 mg/cm^2 standard, using ICP measurements in area units as a substitute for the true lead level. Tables 5-11 through 5-16 present ICP-based classifications relative to a 0.5 percent by weight standard. Under neither standard can laboratory analysis by ICP be regarded as a perfect substitute for the true lead level, because of imprecision from two sources:

(1) Spatial variation in lead levels, due to the use of the test kits near, but not exactly at, the places where primary paint samples were collected for laboratory analysis;

(2) Laboratory error, which encompasses variation due both to the ICP instrument, and to the processing of paint samples prior to instrumental analysis.

Both types of imprecision are discussed extensively in

Chapter 4, where it is shown that spatial variation is the dominant source of imprecision. In this section, the effect of spatial variation and laboratory error on ICP-based classification rates relative to the 1.0 mg/cm^2 and 0.5 percent by weight standards is considered.

The substitution of ICP measurements for the true lead level affects classification rates, because a painted surface with a true lead level that is close to the standard can possibly "switch sides" when the lead level is represented by the ICP measurement of a paint chip sample a small distance away. For example, a positive test kit result, obtained on a painted surface with a true lead level of 0.98 mg/cm^2, is correctly regarded as a false positive relative to the 1.0 mg/cm^2 standard. If an ICP measurement of 1.03 mg/cm^2 was obtained using a paint chip sample collected near, but not exactly at, the place where the test kit was applied, the result would be counted as a true positive if the ICP measurement is substituted for the true lead level. Both the false positive and false negative rates would be underestimated from the effect of this one example.

The potential for misclassification due to substitution of ICP for the true lead level depends on three factors: proximity of the true lead level to the standard, spatial variation in lead levels, and the magnitude of laboratory error. As explained in sections 4.3.1 and 4.3.2, the SD of the natural logarithm of ICP measurements is a valid criterion for measuring the magnitude of the combined effects of spatial variability and laboratory error. Table 4-16 gives SD estimates for laboratory duplicates, by city and substrate, that reflect the impact of laboratory error alone, while Table 4-23 gives SD estimates for field duplicates that reflect both spatial variation and laboratory error.

The distance between field duplicates was approximately 9 inches in Denver and Philadelphia, and 2 inches in Louisville. In the full study, an average distance of approximately 5 inches was maintained between the locations of test kit application and primary paint-chip sample removal. Interpolating between the results given in Tables 4-16 and 4-23 suggests that 0.3 is a plausible estimate of the SD at an average distance of 5 inches for both cities. In the pilot study, an average distance of approximately 9 inches was maintained between the test kit and laboratory sampling locations. A larger SD for Louisville than Denver and Philadelphia may be appropriate, to reflect the

greater potential impact of spatial variation. Using Tables 4-16 and 4-23 to extrapolate a reasonable value for the pilot study SD is difficult, because the 9 inch average distance is much greater than the distance observed for field duplicates. The possibility that there was greater variation between ICP and true lead levels in the pilot study is mitigated by the fact that the pilot study provided only 100 of the 1,290 combined samples.

A simulation study was conducted to determine how the stated false positive and false negative rates changed when random errors were added to the logarithms of the sample ICP values. For the purpose of this exercise, the ICP values were taken as the "true" lead levels, and the sample classification rates were likewise taken as true. Normal random errors, with mean zero and a SD of 0.3, were generated. This SD value is reasonably consistent with the procedures followed in the full study. A total of 1,000 simulations of ICP samples with random errors were generated for each test kit-substrate combination.

Table 5-17 gives the results of the simulation study. The endpoints of the 95% coverage intervals are the 2.5^{th} and 97.5^{th} percentiles of the 1,000 simulated classification rates (false positive and false negative). The means are the averages of the 1,000 simulated rates. By comparing these summary statistics to the sample false positive and false negative rates, insight is gained into the bias and variability implicit in using the sample rates as substitutes for the true rates.

Table 5-17 shows that the false positive rates were little affected when random errors were introduced. The mean simulated values were close to the sample values in every instance, and the 95% coverage intervals exhibit little variability. The false negative rates exhibit small bias in several instances, and had wider 95% coverage intervals than the false negative rates. One reason for the difference is that the sample sizes (number of measurements) for ICP less than 1.0 mg/cm^2 were much larger than for ICP levels above the standard. A small number of ICP switchings affected the false negative rate more than the false positive rate as a result. Because of the smaller sample sizes, the false negatives rates estimated from the data are subject to greater sampling variability than the false positive rates.

Simulations were also conducted in percent by weight units, using the 0.5% federal standard to designate negative and

positive samples.

When normal random errors, with an SD of 0.3, were added to the logarithms of ICP measured in percent by weight units, essentially the same conclusions were reached as for area units. False positive rates were the least affected: in no case did the average of the 1,000 simulated rates differ from the sample false positive rate by more than one percentage point. The 95% coverage intervals also were narrow, the widest being that for LeadCheck on metal (38% to 43%). False negative rates exhibited greater variability in the simulations, but the bias remained small. The largest difference between a sample false negative rate and the average of 1,000 simulated rates was obtained for

Table 5-17. Simulation Study Results of the Effect of Spatial Variation and Laboratory Error in ICP Measurements on Reported False Positive and False Negative Rates (in Percentages).

TEST KIT	SUBSTRATE	FALSE POSITIVE RESULTS			FALSE NEGATIVE RESULTS		
		FP[a]	MEAN[b]	95% INTERVAL[c]	FN[d]	MEAN[e]	95% INTERVAL[c]
LeadCheck	Brick	53	52	51.4, 52.8	5	4	0.0, 4.8
	Concrete	41	40	39.5, 41.3	14	15	12.1, 20.0
	Drywall	21	20	19.7, 21.1	N/A	N/A	N/A
	Metal	47	46	44.7, 47.4	4	5	3.7, 8.0
	Plaster	50	50	49.3, 50.7	12	12	9.1, 16.1
	Wood	58	58	57.0, 59.2	2	3	1.7, 3.5
Lead Alert: coring	Brick	35	34	32.9, 35.2	5	5	0.0, 9.1
	Concrete	11	11	10.7, 12.2	28	32	25.9, 38.7
	Drywall	4	4	3.3, 4.1	N/A	N/A	N/A
	Metal	21	20	18.6, 22.0	27	29	25.5, 33.3
	Plaster	5	5	4.8, 5.7	85	84	81.3, 87.5
	Wood	14	15	13.0, 16.1	25	25	21.7, 27.5
Lead Alert: sanding	Brick	23	23	22.6, 23.3	75	76	75.0, 80.0
	Concrete	13	13	11.9, 14.4	47	52	43.8, 60.0
	Drywall	4	4	4.2, 4.3	N/A	N/A	N/A
	Metal	10	10	7.6, 12.2	57	59	54.1, 62.9
	Plaster	5	5	4.5, 5.6	91	91	87.5, 100
	Wood	2	3	2.2, 5.7	50	52	46.5, 56.9
Lead Detective	Brick	55	55	53.5, 54.9	43	42	38.1, 42.9
	Concrete	38	37	36.4, 38.1	48	47	41.9, 51.7
	Drywall	29	29	27.9, 29.3	N/A	N/A	N/A
	Metal	34	33	31.4, 34.5	27	26	22.2, 30.0
	Plaster	34	34	33.2, 34.7	33	32	26.7, 36.7
	Wood	35	36	34.2, 37.1	10	10	7.5, 12.1

[a] False positive rate reported in this chapter.
[b] Simulation false positive rate.
[c] Simulation 95% coverage interval.
[d] False negative rate reported in this chapter.
[e] Simulation false negative rate.

Table 5-17 (cont). Simulation Study Results of the Effect of Spatial Variation and Laboratory Error in ICP Measurements on Reported False Positive and False Negative Rates (in Percentages).

TEST KIT	SUBSTRATE	FALSE POSITIVE RESULTS			FALSE NEGATIVE RESULTS		
		FP[a]	MEAN[b]	95% INTERVAL[c]	FN[d]	MEAN[e]	95% INTERVAL[c]
Lead Zone	Brick	38	37	35.7, 38.0	5	5	0.0, 9.1
	Concrete	24	25	23.7, 25.3	10	15	7.1, 21.9
	Drywall	22	22	20.8, 22.3	N/A	N/A	N/A
	Metal	24	24	22.5, 25.7	21	23	18.4, 28.0
	Plaster	26	26	25.7, 27.2	36	36	30.3, 40.6
	Wood	35	36	35.0, 37.8	8	9	7.0, 11.7
State Sodium Sulfide	Brick	67	66	65.7, 66.7	0	0	0.0, 0.0
	Concrete	72	72	71.1, 72.1	3	3	2.9, 6.3
	Drywall	38	38	36.4, 37.9	N/A	N/A	N/A
	Metal	64	63	62.3, 64.3	4	4	3.6, 6.0
	Plaster	80	80	79.7, 80.4	0	0	0.0, 0.0
	Wood	59	60	58.9, 61.0	0	0	0.0, 0.0

[a] False positive rate reported in this chapter.
[b] Simulation false positive rate.
[c] Simulation 95% coverage interval.
[d] False negative rate reported in this chapter.
[e] Simulation false negative rate.

LeadCheck on drywall (20% sample, versus 24% simulation). The widest 95% coverage interval was obtained for Lead Detective on drywall (43% to 60%).

None of the simulation results points to an instance where a substantive conclusion about test kit performance would change by taking into account the imprecision due to substituting ICP measurements for the true lead level. This conclusion is robust with respect to the specification of the magnitude of the imprecision. Simulation studies conducted using normal random errors with standard deviations of 0.2 and 0.4, found that the widths of the 95% coverage intervals did increase with the size of the standard deviation, but not to a substantial degree. This was true for ICP measurements both in area and in percent by weight units. Tables 5-5 through 5-16 therefore give an accurate description of how the test kits performed in the study with

respect to the standards of 1.0 mg/cm^2 and 0.5 percent by weight.

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5.2 OPERATING CHARACTERISTIC CURVES FOR LEAD TESTING KITS

In section 5.1, classification results for lead testing kits were presented relative to the 1.0 mg/cm^2 standard. From these results, inferences can be made to situations where the distribution of lead levels is similar to that of the present study. Under different distributions, or in situations where the objective is to describe the performance of a test kit at fixed levels of lead, a more detailed analysis is required.

In this section, analyses of the lead testing kit data are presented in the form of operating characteristic (OC) curves. These analyses address the study objective to characterize the relationship between test kit results and the actual lead level in the paint. The analyses, however, used laboratory ICP measurements as substitutes for the true lead levels in describing the performance of the test kits. This substitution was not perfect, because of (1) spatial variation in lead levels between the locations where the test kits were applied and laboratory paint samples were taken; and (2) various potential sources of laboratory error. Both types of imprecision are explained in section 5.1.1, and are more fully elaborated in Chapter 4. It was found that substitution of ICP measurements for the true lead levels did not adversely affect the analyses. Technical details concerning the estimation of operating characteristic curves with imprecisely measured lead levels are deferred to section 5.2.6.

The purpose of this section is to present estimates of OC curves for each of the six test kits, using ICP measurements in area units (mg/cm^2) as substitutes for the true lead levels in the paint samples. Analyses were conducted in area units for the following reasons:

1. Results for test kits presented in area units are comparable to results for XRF instruments presented in Chapter 6;

2. The inclusion of substrate material in paint samples, which particularly affected both soft and rough substrates such as concrete, brick, and plaster in the study, has the effect of giving percent by weight measurements that are biased low, but imparts negligible bias to area unit measurements.

Results in percent by weight units, oriented to the 0.5 percent by weight standard, are discussed in section 5.2.4.

5.2.1 The Operating Characteristic (OC) Curve.

The OC curve of a lead testing kit describes the probability that the kit gives a positive reading on a paint specimen having true lead concentration at some fixed level. It is a function of the true lead concentration:

$$OC(t) = Prob(test\ kit\ positive \mid true\ Pb = t)$$

Test kit performance was evaluated relative to a standard of 1.0 mg/cm^2 lead on the painted surface. A kit that always gave a negative result at concentrations below the 1.0 mg/cm^2 threshold, and a positive result at those above, would be considered maximally precise. This would be reflected in OC(t) = 0 for t less than 1.0 mg/cm^2, and OC(t) = 1 for t greater than or equal to 1.0 mg/cm^2. As a function of t, this takes the form of a <u>step function</u>, with an infinitely steep jump at t = 1.0 mg/cm^2. Kits that operate with good precision have OC curves that closely resemble this ideal form. This is discussed further in section 5.2.2.4.

The shape of the OC curve indicates how a kit may be expected to perform under actual field conditions. Intuitively, OC(t) should be an increasing function of t: one would not expect a test kit to have a harder time detecting a higher concentration of lead, other factors being equal. A failure of OC(t) to approach zero as t decreases from 1.0 mg/cm^2 may indicate that the kit is prone to give positive readings a certain percentage of the time even if no lead is present. A failure of OC(t) to approach one as t increases from 1.0 mg/cm^2 similarly points to a steady rate of false negatives, no matter how high the lead level in the paint is.

5.2.2 Estimation of the Operating Characteristic Curve

Estimation of OC curves from the study data needed to address the following concerns:

- How, mathematically, should OC curves be represented?

- Should OC curves be developed for a test kit at certain levels of detail, such as by substrate?

Mathematical representation of an OC curve takes the form of a statistical model. Choosing the proper level of detail for analysis is important if practical results are to be obtained. Two aspects of an OC curve that are of particular interest are:

- The 50-percent point of the OC curve (the lead level at which a 50 percent chance of a positive result is obtained);

- The probability of observing a positive result at a true lead level of 1.0 mg/cm^2.

Ideally, the 50-percent point will occur at a value of t just a little less than 1.0 mg/cm^2, with OC(t) at t = 1.0 mg/cm^2 itself close to 100 percent.

The objective in selecting a model was to choose a simple mathematical form that accurately described the performance of the test kits. The use of logistic regression as a modeling technique was initially explored. A description of logistic regression can be found in section 5.2.6.1.1 below. Logistic regression is widely accepted in the statistical community, and it provided a mathematically tractable means for describing test kit performance. The simplicity of the logistic regression model, however, did not allow sufficient flexibility for describing the range of phenomena that were encountered in the study. Specifically, logistic regression does not adequately describe situations where the probability of a positive result increases toward a value less than 1 as the lead level increases, or decreases toward a value greater than 0 as the lead level diminishes.

5.2.2.1 A Model for the Operating Characteristic Curve

An enhancement to the logistic model was developed that did not add substantially to the complexity of the model, but at the same time addressed the deficiencies that were encountered in the simpler model, often resulting in dramatic improvement in the fit of the model to the data. The enhanced logistic model is defined in section 5.2.6.1.2 below. The model has four parameters, denoted a, b, c, and d, which together describe the model completely. The model was fit to the data by substituting

estimated values for these parameters, using the method of nonlinear least squares (NLS) described in section 5.2.6.3.

The enhanced logistic model is described as a function of the natural logarithm of the ICP measurement, rather than the ICP measurement itself. Referring to the logarithm was preferred, because the ICP levels obtained in the study were highly concentrated at lower values. Attributes of the OC curves were more readily apparent when graphed against the logarithm. Further discussion of this issue is presented in section 5.2.6.1.3.

An OC curve estimated with the enhanced logistic model is an increasing function of the lead level that returns a minimum probability of c in the absence of lead, and a maximum probability of $c + d$ when the lead level is infinitely high. Thus, c is interpreted as the <u>baseline probability of positives</u> that can be expected even in the complete absence of lead. The quantity $1 - (c + d)$ is, conversely, the <u>baseline probability of negatives</u> in the presence of maximal lead content. The significance of baseline tendencies is that they point to factors unrelated to the lead content in paint that may affect the performance of a test kit. For example, if a chemical interference with a kit alone accounted for 25 percent of the positive results obtained, c should approximate 0.25.

Parameters a and b also have important interpretations, although not as probabilities. The quantity $-a/b$ is the value of the logarithm of the ICP measurement where the rate of change (the derivative) of the OC curve is greatest. If $a = 0$, this occurs where the logarithm of the ICP measurement is equal to zero, or where the ICP level itself is equal to 1.0 mg/cm^2. A large, positive value of b indicates a sharp rate of change.

If b is very large, approaching an infinite value, the OC curve assumes the shape of a step function, with an abrupt jump occurring at the value $t = -a/b$. Enhanced logistic regression models were fit to the data taking into account the possibility that a step function may provide the optimal solution. An important special case is where b is large (or infinite), $a = 0$ (or small relative to b), $c = 0$, and $d = 1$, which produces the ideal OC curve described previously.

All analyses were conducted using ICP measurements as substitutes for the true lead levels. As noted above, it was not a perfect substitute. section 5.2.6.6 discusses the effect of this substitution on estimation of the enhanced logistic model. A simulation experiment found that the effects of spatial variation and laboratory error, at levels present in the study, did not affect the ability to make inferences from the model to a substantial degree.

5.2.2.2 Graphical Assessment of Estimated OC Curves

In its fullest mathematical generality an OC curve can be any function of the logarithm of lead concentration with values between 0 and 1. It seemed reasonable to restrict this class further to functions that are nondecreasing: higher lead levels should not indicate smaller probabilities of observing a positive test kit result. There are many functions that have this property, and they have a much wider variety of forms than either the simple or the enhanced logistic models can describe, although the enhanced model will often be close.

It is possible to derive an estimate of the OC curve, subject to the constraint that it be nondecreasing, in a way that does not restrict it to a specified functional form. The nonparametric technique known as <u>monotone regression</u> produces estimates that are "best" in the sense that no other nondecreasing function can better fit the data. The estimate can be plotted with the OC curve derived from the enhanced logistic model to give a graphical assessment of how well the model fits the data. Quantities such as probabilities and 50-percent points can be estimated from the monotone regression, and compared with estimates obtained from the enhanced logistic model to assess model fit. Because the monotone regression is a step function, these estimates may not be uniquely defined, in which case the middle of the range of possible candidates is reported. A more detailed description of monotone regression can be found in section 5.2.6.2.

As a formal estimate of the OC curve, a model is preferred to the monotone regression for several reasons. The fact that the monotone regression does not have a simple mathematical description makes it cumbersome for describing the general form of an OC curve in a nonvisual manner. Characteristics described by a set of model parameters are not as easily described by a

monotone regression.

Monotone regression is subject to "endpoint effects" that need to be considered when viewing graphs. If a test kit records a negative result at the smallest ICP measurement, the monotone regression will be equal to zero at that point, regardless of the performance of the test kit otherwise. Similarly, a positive result at the highest value causes the monotone regression to reach 1 at that point. These effects are negligible from a statistical viewpoint, despite their prominence when graphed. section 5.2.6.2 describes how these endpoint effects were handled in the analyses.

Another simple graphical assessment of a model-estimated OC curve is to plot it with a running mean against the log(ICP) measurements. The running mean is obtained at a point log(ICP) = t by averaging zeros (for negatives) and ones (for positives) for a small subset of the data having log(ICP) close to t. Details on how this was carried out in the analyses are provided in section 5.2.6.2. Unlike monotone regression, the running mean is not designed to be a nondecreasing function of the lead level. Its virtue, in fact, resides in its ability to graphically demonstrate where this assumption may be violated. In this case, the running mean appears to diverge from both the model and the monotone regression estimates.

A visual indication of decreasing behavior in the running mean does not automatically imply that the running mean is "right", and the monotone regression and model are "wrong". Some decreasing behavior is to be expected due to sampling variability, although large violations of the nondecreasing assumption may signal unusual sample or test kit characteristics. For this reason, the running mean and monotone regression together were chosen to provide a nonparametric summary of the performance of a test kit.

Like monotone regression, the running mean is subject to endpoint effects that may be visible when graphed. These effects arise because the averages forming the running mean use smaller subsets of data near the smallest and largest ICP measurements. The effect that this "diminished averaging window" has on the running mean is explained more fully in section 5.2.6.2. For the purpose of viewing graphs, endpoint effects are present at the 12 smallest and 12 largest ICP measurements, and must be considered

when making visual inferences.

5.2.2.3 Substrate Effects

The test kits were applied to six different substrate types: brick, concrete, drywall, metal, plaster, and wood. To produce a single OC curve for a given kit makes sense if the behavior of the kit did not depend on the substrate, or if a description of some concept of "average" behavior is desired.

It is clear from the classification results that the performance of the test kits depended on the substrate. Moreover, the instructions for several of the kits listed substrates upon which they were, or were not, intended for application. These factors supported the decision to describe the performance of the test kits by substrate, as opposed to aggregating across substrates.

The data for some substrates posed special problems. For drywall, none of the ICP measurements were at or above the 1.0 mg/cm^2 standard, which made it difficult to infer how the test kits would perform on drywall at that level of lead. On a few other occasions, too few positive results were obtained to make estimation of the OC curve meaningful aside from reporting the low rate of positives itself.

5.2.2.4 Describing Test Kit Performance: Illustrations

Figure 5-1 illustrates the performance of an "ideal" test kit using the graphical assessment described in the previous section. This is a simulated example based on 200 observations. For ease of visual interpretation, the base-10 logarithm ($\log_{10}$) of the ICP measurement was chosen for the horizontal axis of this plot. For example, -1 on the horizontal axis corresponds to an ICP value of 10^{-1}, or 0.1 mg/cm^2. Also shown is the 50-percent point, both in $\log_{10}$ and in area units. Thus, log(1.013) = 0.005 describes a 50-percent point estimated at 1.013 mg/cm^2 in area units, and 0.005 in $\log_{10}$ units.

The solid line shows the estimated enhanced logistic model, the dotted line is the monotone regression, and asterisks are used to plot the running mean.

The following features highlight this as an ideal case:

- The model, monotone regression, and running mean are in close agreement.

- The probability of a positive result approaches zero as the lead level diminishes, and approaches one as the lead level increases.

- The transition from low to high probabilities is sharp, as indicated by the steepness of the plotted curves.

- The transition from low to high probabilities occurs near a lead level of 1.0 mg/cm^2.

The fact that the three curves are in close agreement indicates that the model was an appropriate choice for describing test kit performance. Since the probability of a positive result goes to zero at lower lead levels, the baseline probability of positives is estimated as zero, meaning that the kit did not exhibit a tendency to produce positive results a certain percentage of the time independent of the lead level. Likewise, the baseline probability of negatives is estimated as zero, since the probability of a positive result approaches one at higher lead levels.

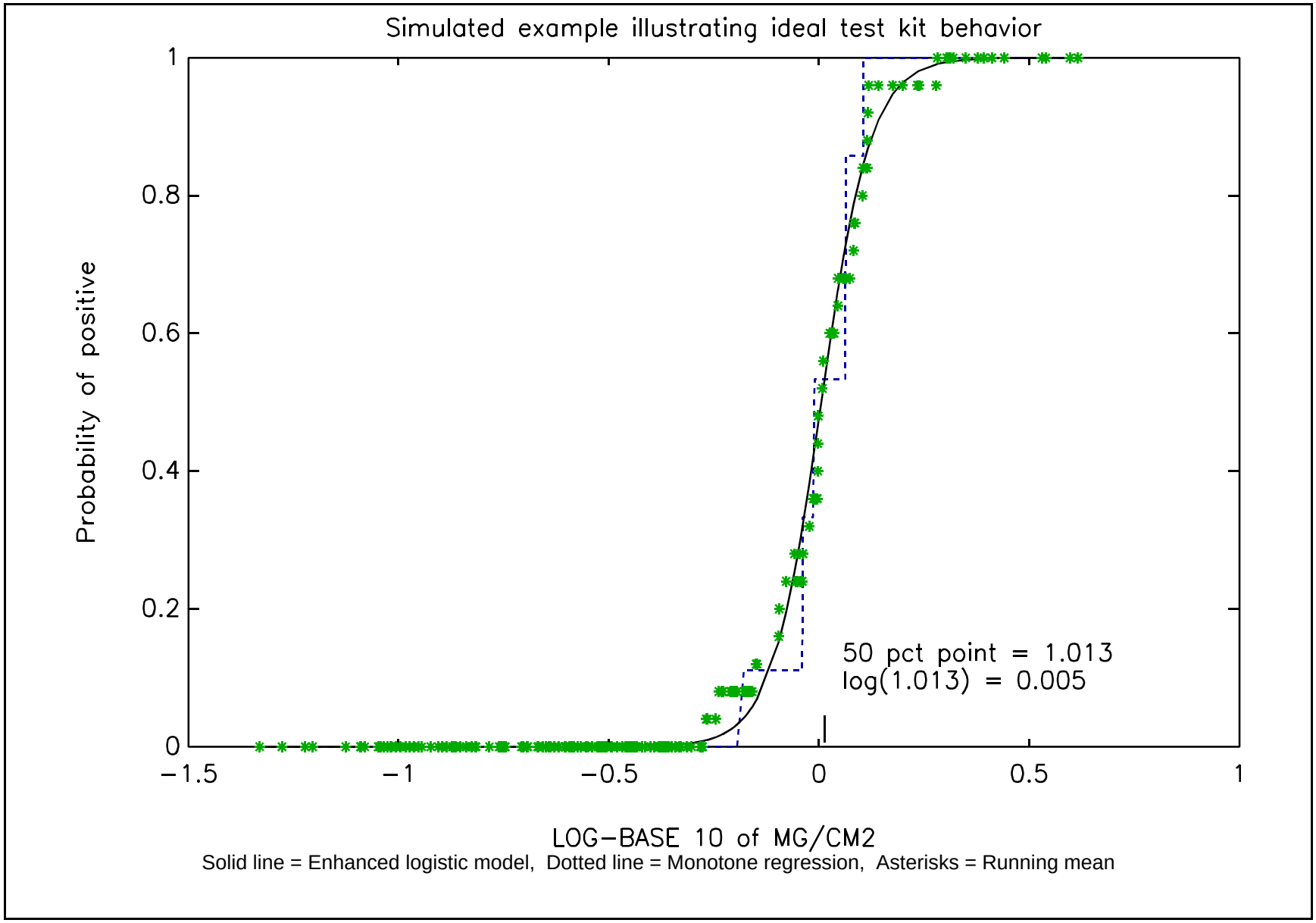

Figure 5-1. Simulated example illustrating ideal test kit behavior.

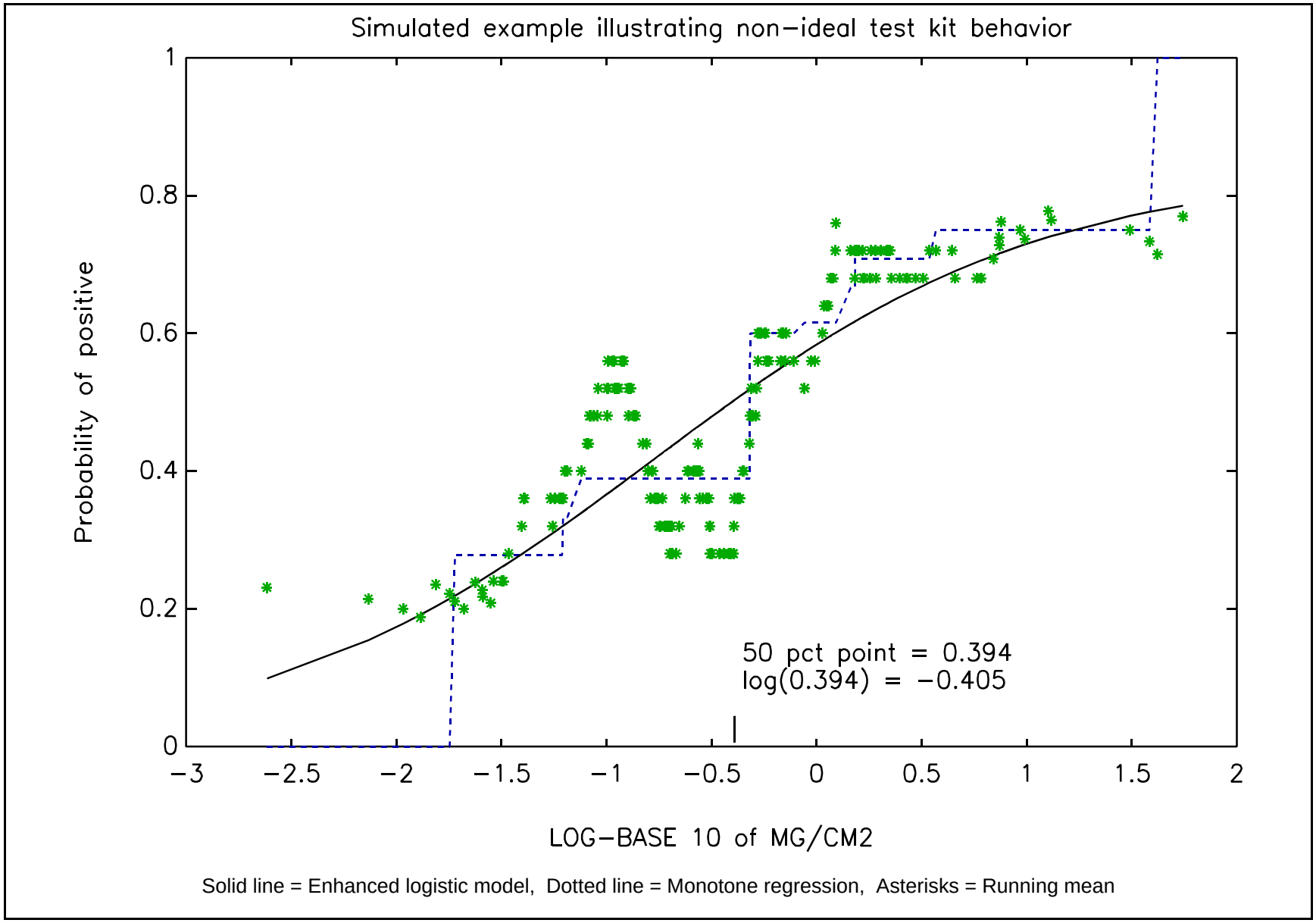

Figure 5-2. Simulated example illustrating non-ideal test kit behavior.

A sharp transition in the plots indicated at the estimated 50-percent point of 1.013 mg/cm^2 suggests that paint with a lead level even a little less than 1.0 mg/cm^2 was highly likely to be classified as negative, and paint even a little above 1.0 mg/cm^2 was highly likely to be classified as positive. This behavior is highly desirable in a test kit, because it suggests that the kit correctly classified both high lead and low lead cases a high percentage of the time.

None of the test kits evaluated in the study were able to emulate the performance of the ideal case in all respects. Figure 5-2 illustrates characteristics of nonoptimal test kit performance that were frequently observed. It also is a simulated example based on 200 observations. The model, monotone regression, and running mean plots appear to be more divergent than in the ideal case; the probability of a positive result does not approach 0 as the lead level decreases and 1 as the lead level increases; the transition from low to high probabilities is not sharp. One reason for the graphical divergence is indicated by a dip in the running mean, the magnitude of which is moderately unlikely as a purely random occurrence. This dip affects the model OC curve overall, making it flatter, but it only imparts a localized effect to the monotone regression.

Endpoint effects in the monotone regression can be seen at both high and low lead levels, for no other reason that the sample with the smallest ICP measurement recorded a negative result, and that with the largest ICP measurement a positive result. A baseline rate of false negatives is estimated at about 20 percent, which suggests that the kit is prone to giving negative readings about one-fifth of the time even at very high lead levels. There is, at the same time, greater than a 50 percent chance of obtaining a positive result at lead levels exceeding 0.394 mg/cm^2, according to the model. The dip in the running mean, however, suggests that the 50-percent point may be even smaller. Positive results may be expected with this hypothetical test kit more than 20 percent of the time for lead levels as low as $10^{-1.5}$ = 0.03 mg/cm^2.

5.2.3 Results of Model Estimation

In the six subsections that follow, OC curves are described for each of the test kits by substrate. Each subsection has a table giving the enhanced logistic model parameters (a,b,c,d);

the 50-percent point denoted Pb(.50); and the estimated probability of a positive test kit result at 1.0 mg/cm^2 for each substrate. The last quantity is also referred to as the threshold probability for ease of exposition.

The estimated model parameters, and all quantities derived from them, are subject to uncertainty due to the fact that they are based on samples. This type of uncertainty, referred to as sampling variability, is quantified by the standard error. For many estimators, a 95 percent confidence interval is formed by adding and subtracting 2 times the standard error from the value of the estimator. Under appropriate conditions, intervals formed in this manner contain the true, but unknown, quantity of interest approximately 95 percent of the time. Confidence intervals can also be derived by methods such as bootstrapping, which is based on simulation and not directly related to the standard error. Like the standard error, a confidence interval measures, in a statistically formal way, the sampling variability implicit in an estimator.

Although the 95 percent confidence level is widely accepted, many in the statistics community recommend a more conservative criterion, such as the 99.7 percent confidence level corresponding to an interval width of 3 times the standard error. There are several reasons why a more conservative criterion may be preferred:

1. Multiple inferences. As the number of confidence intervals simultaneously considered increases, the number of expected instances where the confidence interval fails to cover the quantity of interest increases proportionately. Out of every hundred 95 percent confidence intervals, for instance, 5 failures can be expected. A more conservative criterion can sharply reduce the number of expected failures when multiple inferences are made.

2. Lack of model fit. The models presented in section 5.2, like most statistical models, are approximations. Wider confidence intervals allow greater leeway for effects due to imperfection of the model.

3. Sampling effects. The study data were not, as a matter of necessity, obtained from simple random samples. Clustering by unit, paint type, or the person making test kit result

determinations may cause standard error estimates to be understated. A more conservative criterion allows greater leeway for this effect.

Confidence intervals presented in section 5.2 were obtained either by bootstrapping or through the use of transformations, and are not related in a simple way to standard error estimates. A 95 percent confidence level was used, in spite of the arguments in favor of a more conservative criterion, in order to exhibit the sampling variability of estimates in a conventional manner.

Approximate standard errors are reported in parentheses beneath the parameter estimates in the tables presented below. For 50-percent points and the threshold probabilities, the two numbers in square brackets beneath the estimates are lower and upper endpoints of 95 percent confidence intervals. The construction of approximate standard errors and confidence intervals is explained in section 5.2.6.4. It is emphasized that the model parameter estimates are often highly intercorrelated, which is not apparent in the individual standard error estimates, but for which account was made, either directly through large sample mathematical approximation (asymptotics), or indirectly through bootstrap simulations, in the derivation of reported confidence intervals for 50-percent points and threshold probabilities.

As noted in section 5.2.2.1, model parameters *c* and *d* have simple interpretations relating to the baseline probabilities of positives and negatives. Parameters *a* and *b* are also important in describing the model, but they do not individually have probabilistic interpretations. In fact, relatively large changes in *a* and *b*, arising from spatial variation and laboratory error in ICP measurements or other causes, may produce only small changes in the probabilities calculated from the model, or in other quantities of interest.

In several instances step functions were obtained as model OC curve estimates. This phenomenon is described in sections 5.2.2.1 and 5.2.6.1.2. A step function is represented in the tables by an enhanced logistic regression model with $b = 100$, and *a* chosen so that $-a/100$ gives the logarithm of the changepoint. The estimated parameters in this case are given in bold italics, and reproduce the step function almost exactly.

There are instances where the enhanced logistic model was not applied to a particular substrate. This occurred several times with drywall and plaster when so few positive results were obtained that fitting the model was a futile exercise. Because there were no ICP measurements as large as 1.0 mg/cm^2 on drywall, it was difficult to assess test kit performance at this lead level. Estimates for drywall should therefore be interpreted with caution. To avoid making unsubstantiated inferences about the performance of a test kit on drywall at high levels of lead, the constraint $c + d = 1$ was imposed in estimating the model parameters.

Following the summary tables, plots are presented to allow graphical assessment of the fitted models, as illustrated in section 5.2.2.4. The OC curve estimated from the enhanced logistic model (solid line), the monotone regression (dashed line), and the running mean with a window size of 25 (asterisks) are shown plotted together. An effective way to view these plots is to compare the enhanced logistic model curve to the monotone regression, and the monotone regression to the running mean. The first comparison indicates visually how well the enhanced logistic model fit the data in the class of all nondecreasing functions. The second indicates how well a monotone description of the OC curve was able to fit the data under even the best of circumstances.

5.2.3.1 LeadCheck

Table 5-18 gives the results of fitting enhanced logistic models to the six substrates tested with the LeadCheck kit. Figures 5-3 through 5-8 illustrate the fit of these models to the data. Sections 5.2.3.1.1 through 5.2.3.1.6 discuss the estimated OC curves by substrate. Section 5.2.3.1.7 contains an overall summary for the kit.

5.2.3.1.1 LeadCheck on Brick

There were 93 observations of LeadCheck on brick, of which 35 gave negative and 58 gave positive readings. Figure 5-3 shows that the running mean (asterisks), monotone regression (dotted line), and the model OC curve based on the coefficient values in Table 5-18 agree closely. The enhanced logistic model, which coincided with a simple logistic model in this case, appears to have been an appropriate choice for these data.

The model estimates the threshold probability at .945, with a better than even chance of a positive result for lead levels as low as 0.016 mg/cm^2. These estimates agree closely with those from the monotone regression: .95 for the threshold probability, and 0.006 mg/cm^2 for the 50-percent point. Using the 95% confidence intervals in Table 5-18 to account for sampling variability in these estimates does not change the conclusion that positive results became the more frequent outcome at lead levels below 1.0 mg/cm^2, and predominated near the standard. The high percentage of false positives shown in Table 5-5 (53%) may reflect sensitivity of the kit to levels of lead below 1.0

Table 5-18. LEADCHECK Enhanced Logistic Regressions by Substrate.

SUBSTRATE	NEGATIVE	POSITIVE	MODEL PARAMETERS				Pb(.50) mg/cm²	PROB. AT Pb = 1
			c	d	a	b		
BRICK	35	58	0	.992 (.046)	3.01 (.988)	0.727 (.177)	0.016 [.002, .143]	.945 [.836, .983]
CONCRETE	121	105	.100 (.188)	.900 (.188)	0.624 (.338)	0.513 (.253)	0.191 [.024, .568]	.686 [.591, .768]
DRYWALL	98	26	0	1	-0.045 (.406)	0.338 (.111)	1.14 [.096, 13.5]	.489 [.298, .683]
METAL	92	125	.289 (.051)	.662 (.067)	3.19 (1.71)	3.66 (1.70)	0.340 [.204, .463]	.925 [.826, .969]
PLASTER	109	133	.232 (.239)	.768 (.239)	0.387 (.564)	0.496 (.291)	0.130 [.010, .513]	.690 [.613, .757]
WOOD	116	272	.174 (.092)	.826 (.092)	2.09 (.236)	0.732 (.143)	0.032 [.005, .079]	.909 [.862, .942]

mg/cm^2.

There is no evidence that LeadCheck was prone to high baseline rates of false positives or negatives on brick, since the OC curve levels off near zero at low lead levels, and near one at high lead levels. Positive results were obtained on all 20 samples with ICP measurements above 4.657 mg/cm^2, which again confirms that the test kit was sensitive to lead on brick substrates.

5.2.3.1.2 LeadCheck on Concrete

There were 226 measurements made on concrete, of which 121 gave negative and 105 gave positive results. Figure 5-4 shows that the model OC curve fits the data well relative to the monotone regression and the running mean, except towards the middle of the range (0.01 to 0.1 mg/cm^2) where the running mean flattens out somewhat. Monotone regression, which is more flexible, is able to pick up this effect. Both the model and monotone regression detect a baseline rate of false positives: .100 from the model (the parameter c in Table 5-18) and about half that rate from the monotone regression. All 12 samples with ICP measurements higher than 3.715 mg/cm^2 gave positive results.

The model estimates of the 50-percent point (0.191 mg/cm^2) and threshold probability (.686) are not far from the estimates obtained with monotone regression (0.265 mg/cm^2 and .774 respectively). Both suggest a greater than two thirds chance of observing a positive result at 1.0 mg/cm^2. Both also suggest that there was better than a 50 percent chance of a positive result at lead levels exceeding 0.3 mg/cm^2, which is reflected in the high rate of false positives seen in Table 5-5 (41%). The 95% confidence intervals suggest a 50-percent point as high as 0.568 mg/cm^2, and a threshold probability as low as .591 at 1.0 mg/cm^2.

5.2.3.1.3 LeadCheck on Drywall

There were 124 observations of LeadCheck on drywall, of which 98 gave negative and 26 gave positive readings. Figure 5-5 shows that the running mean has a pronounced dip near 0.1 mg/cm^2 (10^{-1} = 0.1). Since none of the ICP measurements were as high as 1.0 mg/cm^2, it is doubtful that either the model OC curve or the monotone regression accurately describe the performance of the

test kit at this level of lead. The wide confidence interval in Table 5-18 for the 50-percent point reflects this uncertainty.

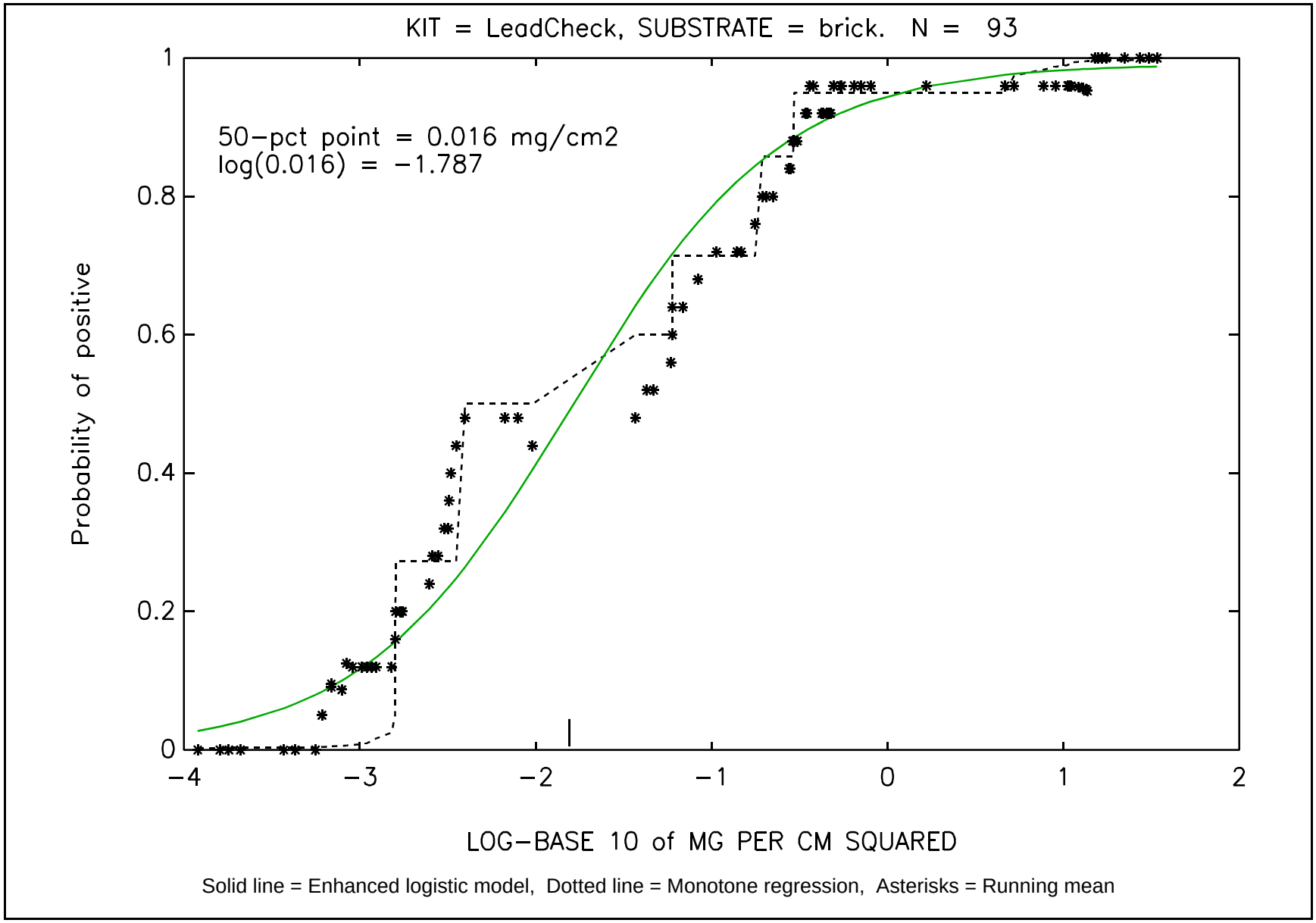

Figure 5-3. Operating characteristic curve for Lead Check on brick.

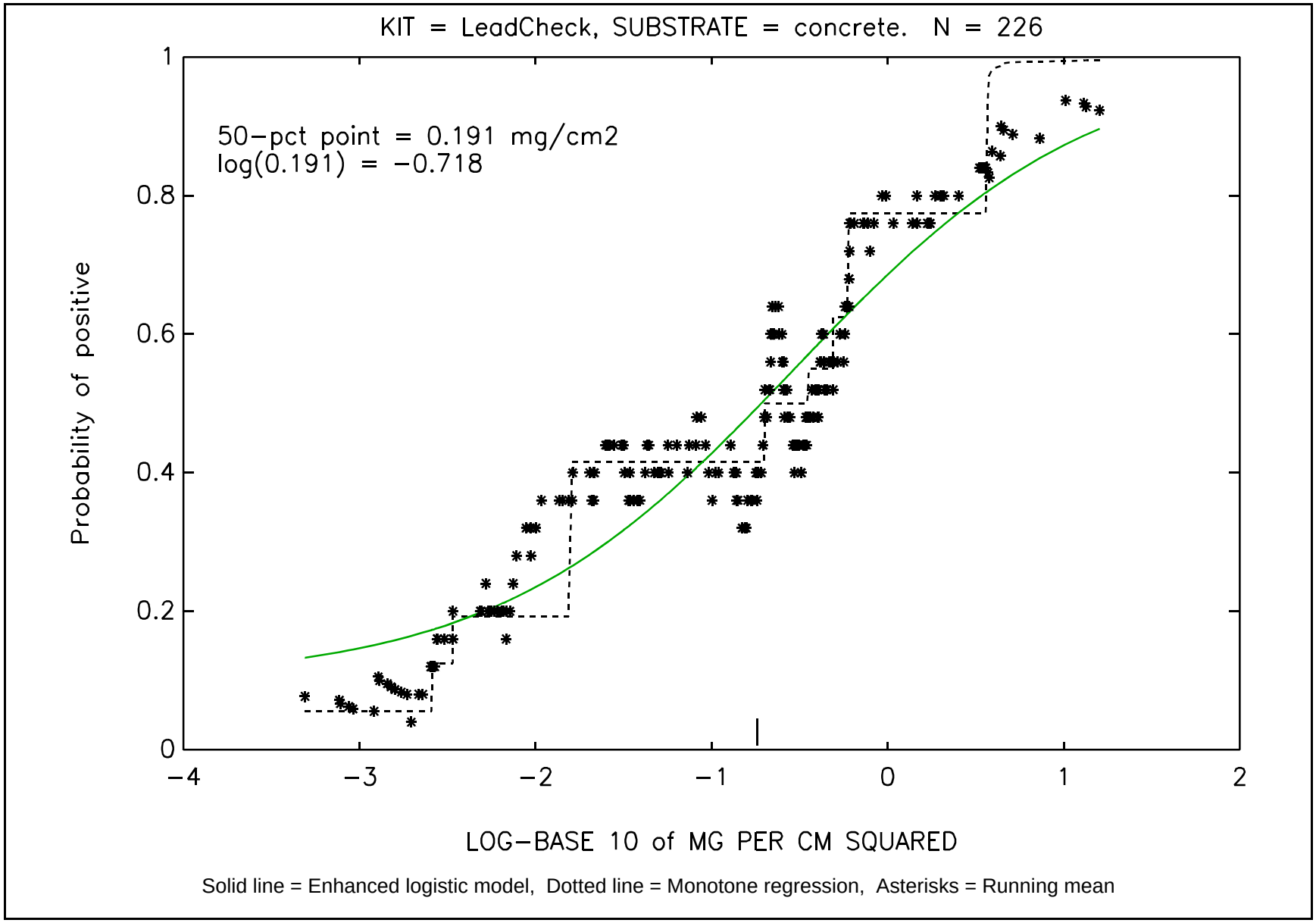

Figure 5-4. Operating characteristic curve for LeadCheck on concrete.

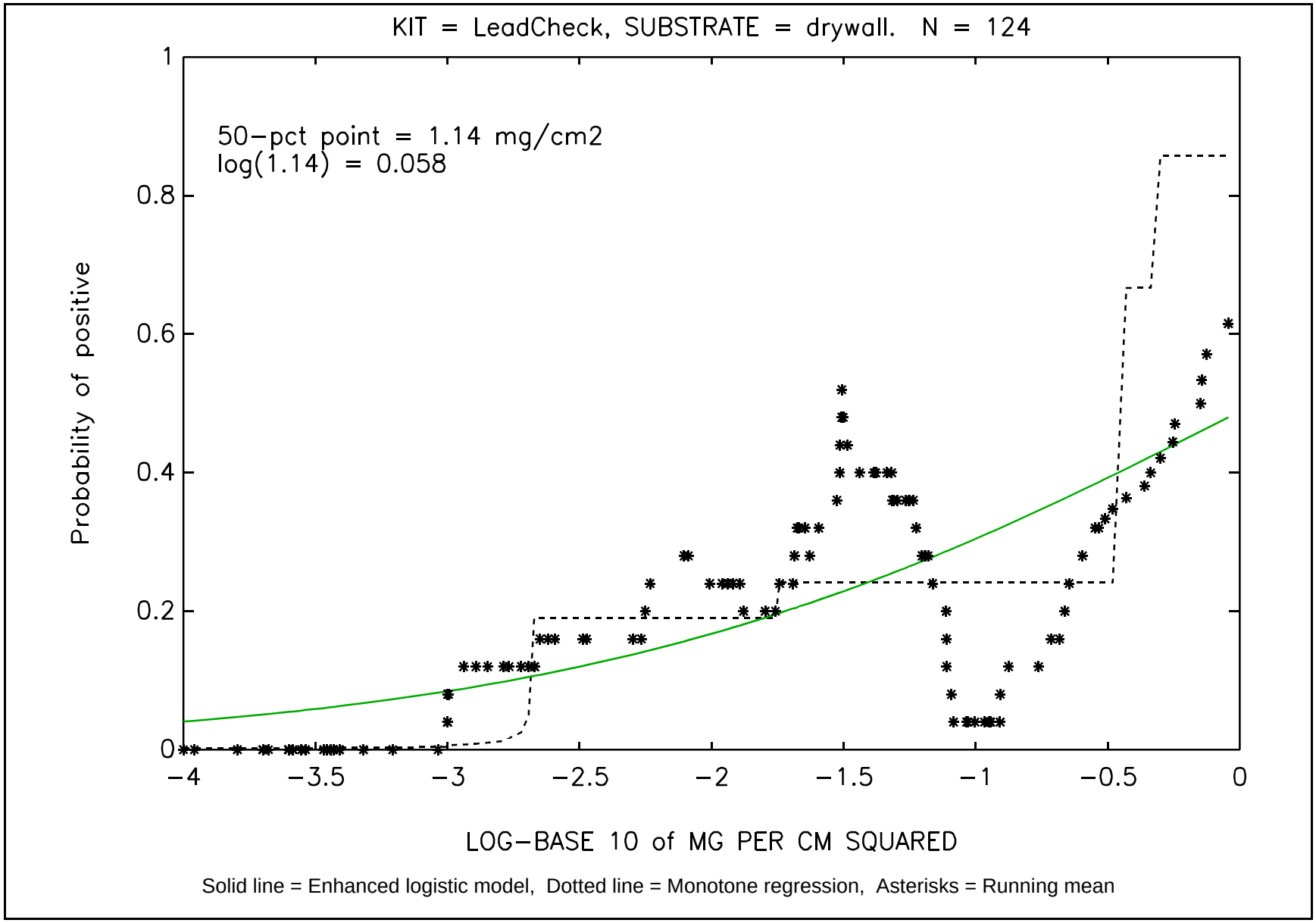

Figure 5-5. Operating characteristic curve for LeadCheck on drywall.

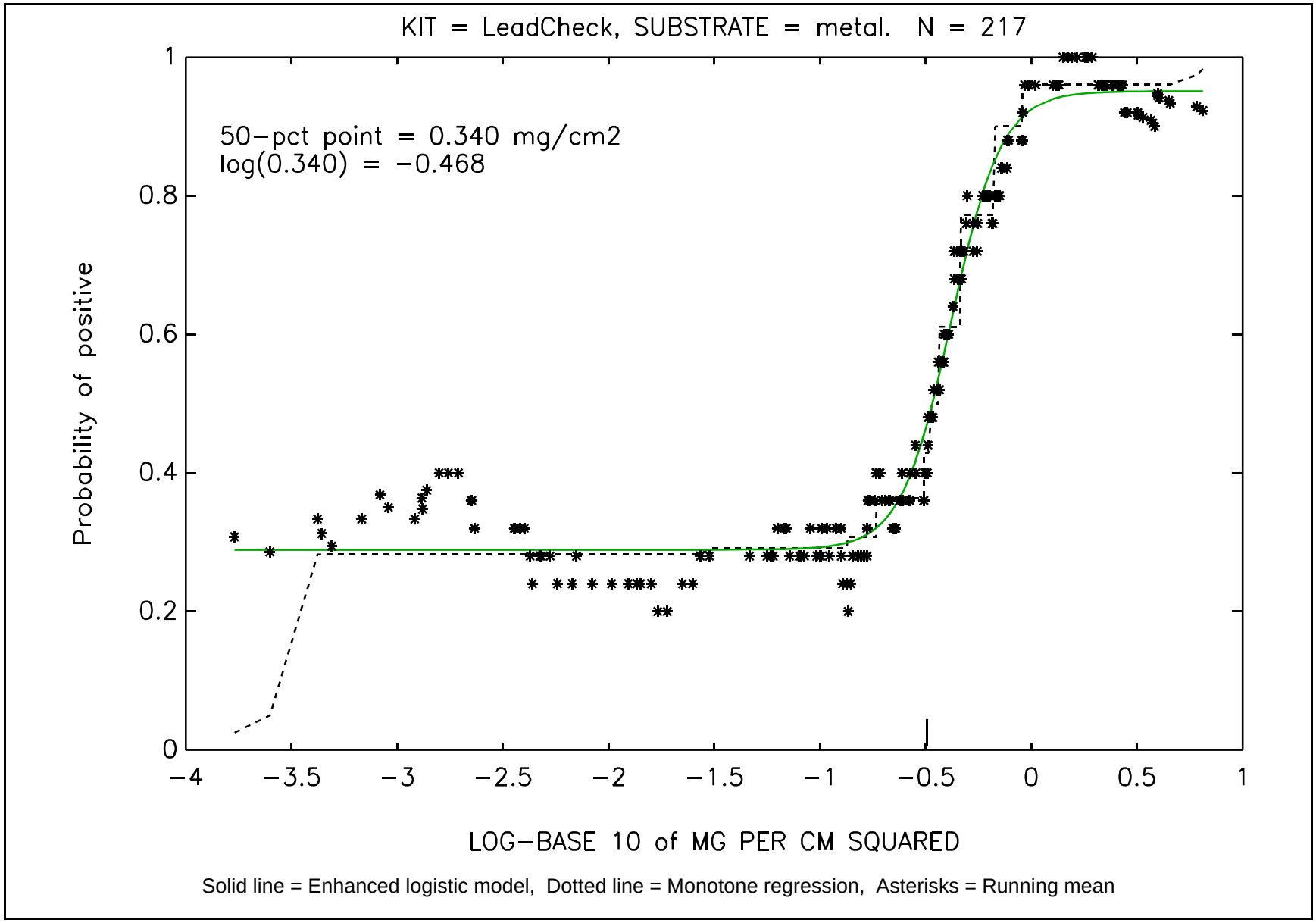

Figure 5-6. Operating characteristic curve for LeadCheck on metal.

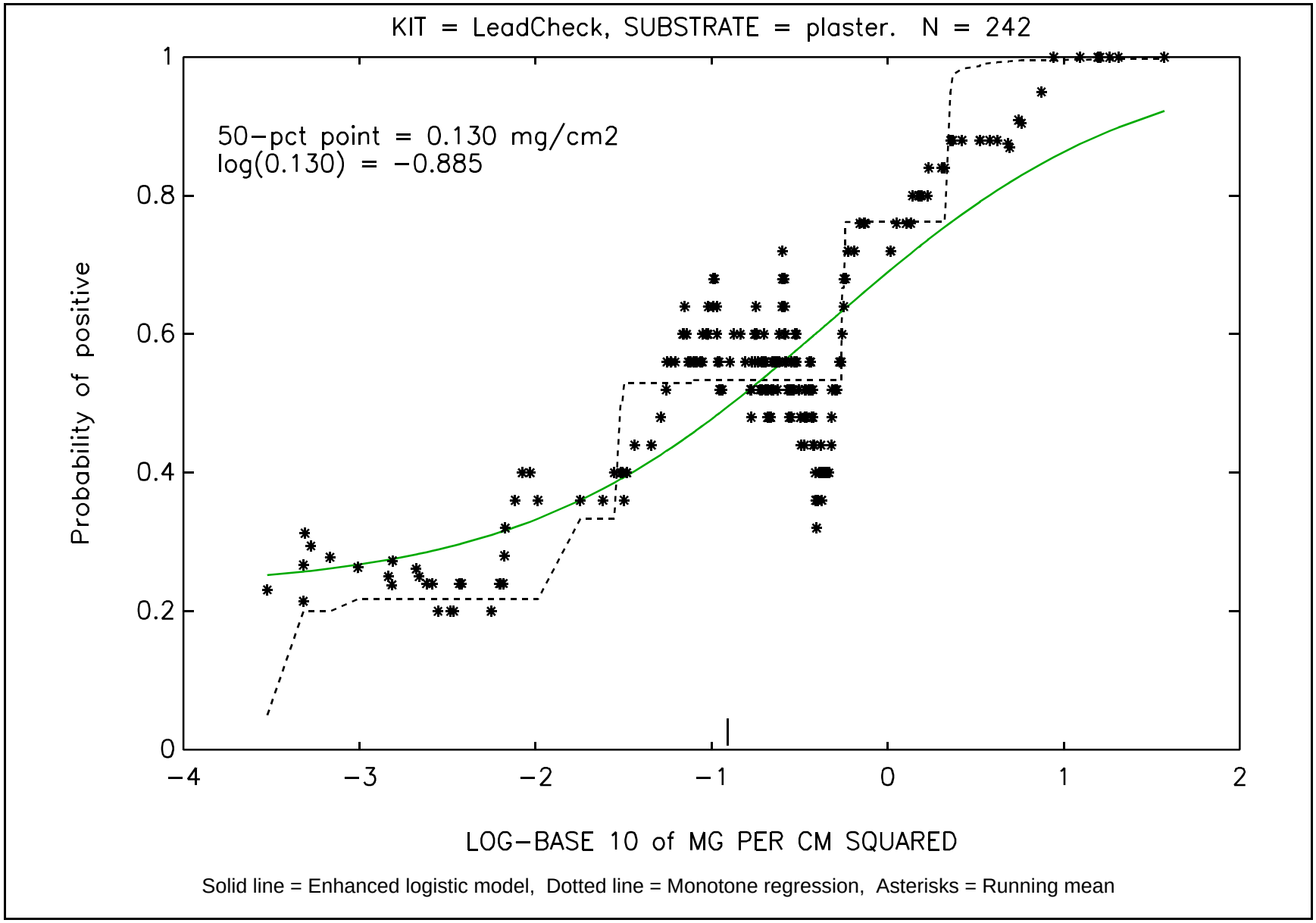

Figure 5-7. Operating characteristic curve for LeadCheck on plaster.

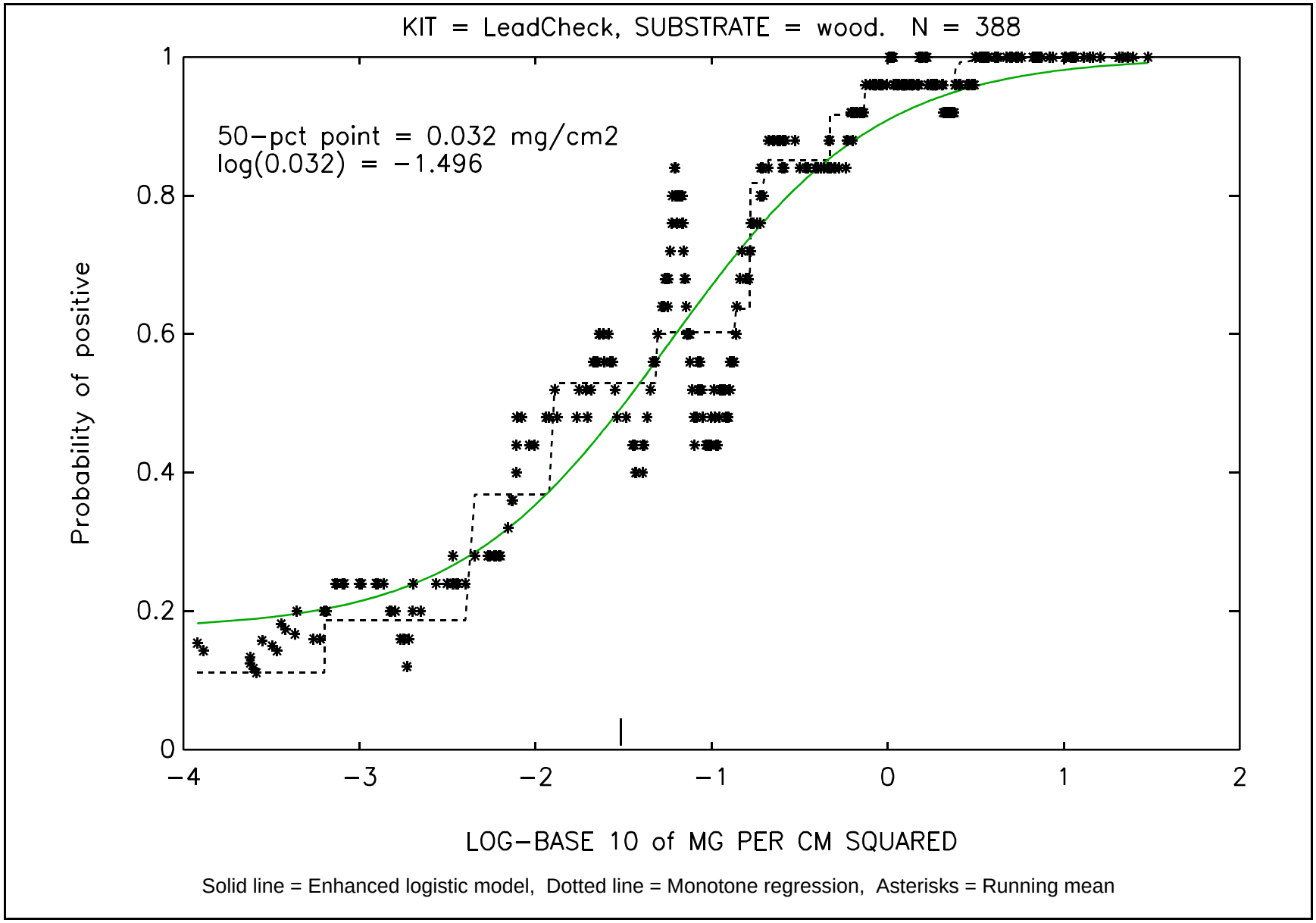

Figure 5-8. Operating characteristic curve for LeadCheck on wood.

5.2.3.1.4 LeadCheck on Metal

There were 217 observations of LeadCheck on metal, of which 92 gave negative and 125 gave positive readings. Figure 5-6 shows close agreement between the running mean, monotone regression, and model OC curve. The baseline rate of false positives is apparently substantial, estimated at .289 with the enhanced logistic model (the parameter *c* in Table 5-18), which agrees with the monotone regression ignoring the endpoint effect. Thus, more than half of the rate of false positives seen in Table 5-5 (47%) may have been due to a factor other than the lead level, such as substrate interference, or the type of paint used.

The threshold probability is estimated at .925 with the model (.961 with monotone regression), and the 50-percent point at 0.340 mg/cm^2 (0.350 mg/cm^2 with monotone regression). The sharp increase in the frequency of positives over a short lead range is reflected in all three plots shown in Figure 5-6. The kit was sensitive to lead at the 1.0 mg/cm^2 standard, and it lost sensitivity rapidly as the level of lead decreased, but with a constant frequency of positives observed at even very low lead levels. Although the model suggests a small baseline probability of negatives (.049), it is not possible to determine if this would occur at lead levels in excess of 10.0 mg/cm^2, since ICP measurements of this magnitude were not obtained on metal in the study.

5.2.3.1.5 LeadCheck on Plaster

There were 242 observations of LeadCheck on plaster, of which 109 gave negative and 133 gave positive readings. Figure 5-7 shows a dip in the running mean toward the center of the lead range that neither the model nor the monotone regression are able to describe. Phenomena of this kind can be expected to occur infrequently as a random event. Pronounced dips affect the fit of the model to the entire data, but have only a localized effect on the monotone regression.

A second important feature seen in Figure 5-7 is that the baseline probability of false positives was apparently substantial: the estimate obtained with the model (.232) appears to be consistent with the monotone regression. This suggests that nearly half of the false positive rate seen in Table 5-5 (50%) was due to non-lead factors. The model and monotone

regression produced similar threshold probability estimates (.690 model versus .761 monotone regression). These estimates seem reliable given the nature of the plots, but the same cannot be said of the 50-percent point estimates, for which the truth may be close to where neither curve fit the data well. Positive results were obtained on all 19 samples with ICP measurements in excess of 2.275 mg/cm^2, which reflects the sensitivity of the kit at higher lead levels.

5.2.3.1.6 LeadCheck on Wood

There were 388 observations of LeadCheck on wood, of which 116 gave negative and 272 gave positive results. Figure 5-8 shows that the running mean, monotone regression, and model OC curve are in agreement, with the exception of the peak-dip phenomenon toward the center of the range that caused some flattening of the curves.

The model OC curve indicates a substantial baseline probability of false positives (.174) that is evident to a lesser extent in the monotone regression. Positive results were obtained on wood with ICP measurements as low as 0.00026 mg/cm^2. Since the overall rate of false positives indicated in Table 5-5 was 58%, it appears that most false positives were due to sensitivity of the kit to low levels of lead rather than to interference from non-lead factors.

The model and the monotone regression estimates of the 50-percent point (0.032 and 0.012 mg/cm^2 respectively) and threshold probability (.909 and .962) also demonstrate the sensitivity of the kit to low lead levels. Although the peak-dip makes it difficult to regard either estimate of the 50-percent point as precise, it is clear that the frequency of positive results near the 1.0 mg/cm^2 standard was high. At higher lead levels the kit gave consistently positive results, which was the observed outcome at all 56 samples with ICP measurements above 2.386 mg/cm^2.

5.2.3.1.7 Summary of Analysis for LeadCheck

The manufacturer's instructions warned about interference with color development of the kit from sulfates in plaster dust, gypsum, and stucco. To help guard against interference, the instructions included a confirmation procedure, which the study

adopted. This procedure, however, was meant to guard against false negatives, as opposed to false positives. A false negatives problem was not observed with LeadCheck, but high rates of false positives were obtained on several substrates. These rates were highest on plaster and metal, and lower but still substantial on concrete and wood.

The manufacturer recommended LeadCheck for painted metal and wood surfaces, and indeed both had greater than 90 percent positive rates at 1.0 mg/cm^2. The same was true for brick. While the rate of positives on metal declined sharply as the level of lead decreased from 1.0 mg/cm^2, it did not fall below 29 percent.

5.2.3.2 Lead Alert: Coring

This section describes the performance of the Lead Alert All-In-One kit using the coring technique. The sanding technique is described in section 5.2.3.3. Table 5-19 gives the results of fitting enhanced logistic models to the substrates tested with Lead Alert using the coring technique. Figures 5-9 through 5-12 illustrate the fit of these models to four of the substrates. Summaries by substrate are given in sections 5.2.3.2.1 through 5.2.3.2.6, and an overall summary is given in section 5.2.3.2.7.

5.2.3.2.1 Lead Alert: Coring on Brick

There were 93 observations of Lead Alert with coring on brick, of which 48 gave negative and 45 gave positive readings. Figure 5-9 shows that the running mean, monotone regression, and model OC curve are in general agreement. The model OC curve has nearly the shape of a step function ($b = 7.36$), influenced primarily by a sharp rise in the rate of positives. The model indicates a high baseline rate of false positives (.200) corresponding to over half of the false positives rate shown in Table 5-6 (35%). Although the 7 samples with ICP measurements less than 0.00056 mg/cm^2 had negative results, a high rate of false positives at low lead levels is nonetheless evident. At high lead levels the model indicates a baseline rate of false negatives estimated at .070, in spite of the fact that all 20 samples with ICP measurements greater than 4.656 mg/cm^2 had positive results. While these two outcomes are not inconsistent with each other, the possibility that the baseline effect is an artifact of the model cannot be dismissed.

The model and monotone regression produce similar estimates of the 50-percent point (0.327 mg/cm^2 versus 0.319 mg/cm^2), which the 95% confidence interval (Table 5-19) indicates was possibly as high as 0.459 mg/cm^2. Estimates of the threshold probability (.928 model, .800 monotone regression) are high. Positive results on brick with the coring technique were obtained on the full range of lead levels represented on this substrate.

Table 5-19. LEAD ALERT: CORING Enhanced Logistic Regressions by Substrate.

SUBSTRATE	NEGATIVE	POSITIVE	MODEL PARAMETERS				Pb(.50) mg/cm²	PROB. AT Pb = 1
			c	d	a	b		
BRICK	48	45	.202 (.059)	.727 (.079)	7.86 (5.85)	7.36 (5.59)	0.327 [.076, .459]	.928 [.735, .984]
CONCRETE	183	43	.098 (.030)	.902 (.030)	-1.46 (.918)	2.05 (.785)	1.84 [.844, 3.49]	.267 [.104, .534]
DRYWALL	119	5	---	---	---	---	---	---
METAL	147	70	.093 (.039)	.632 (.075)	2.24 (1.07)	3.75 (1.87)	0.645 [.476, 1.15]	.664 [.514, .787]
PLASTER	225	16	---	---	---	---	---	---
WOOD	259	128	0	.973 (.074)	0.345 (.333)	1.11 (.173)	0.771 [.257, 1.76]	.569 [.474, .660]

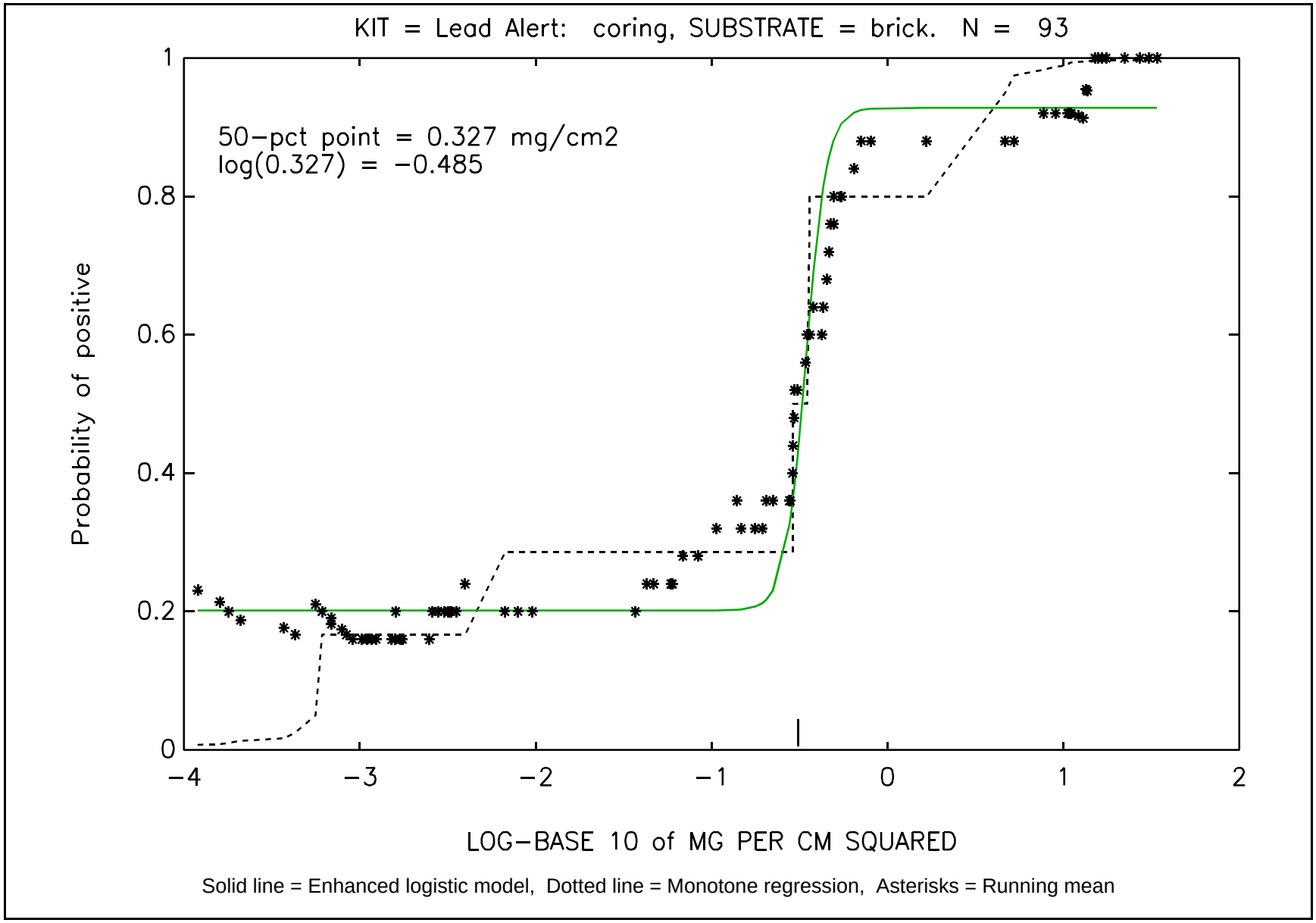

Figure 5-9. Operating characteristic curve for Lead Alert coring on brick.

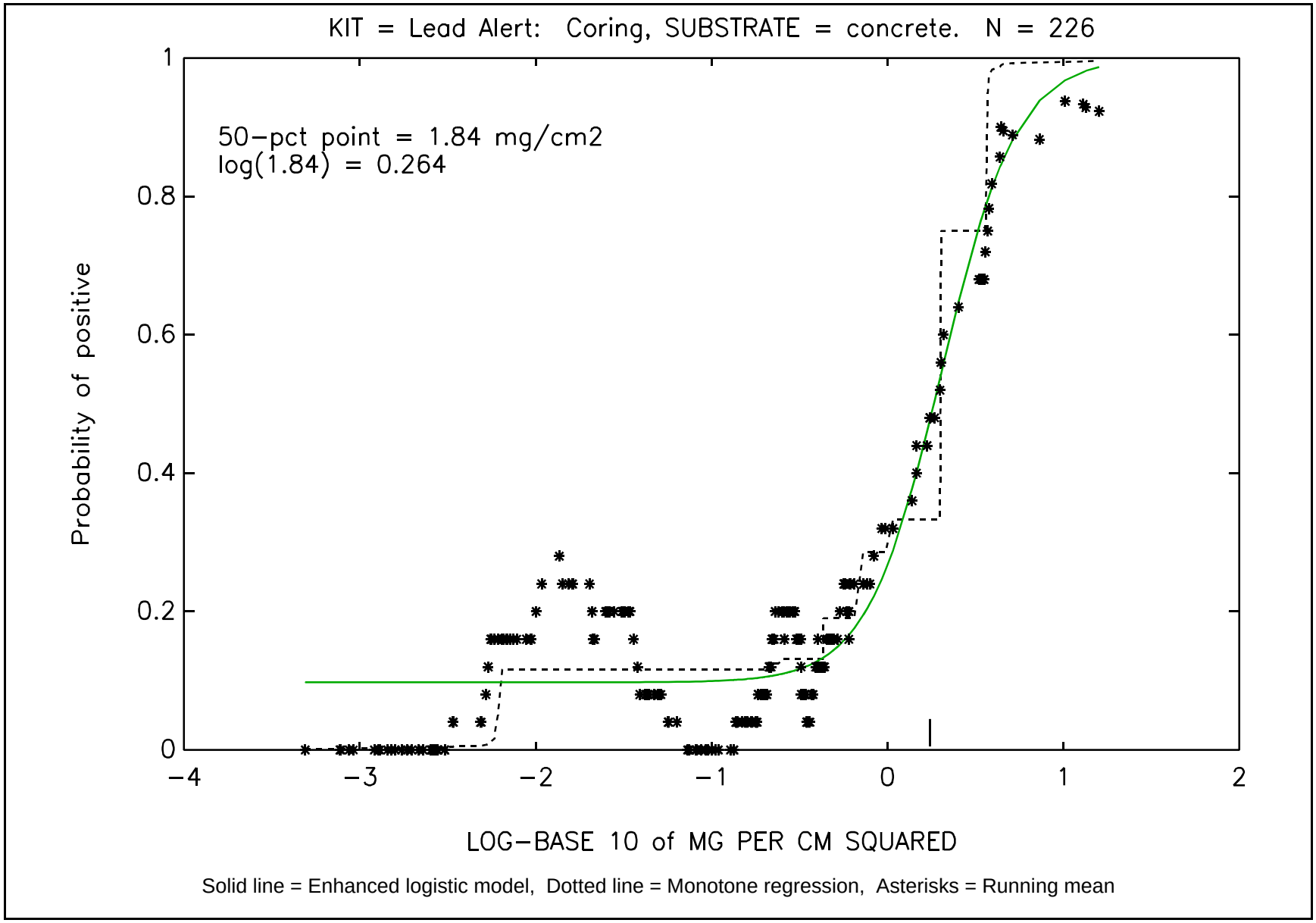

Figure 5-10. Operating characteristic curve for Lead Alert coring on concrete.

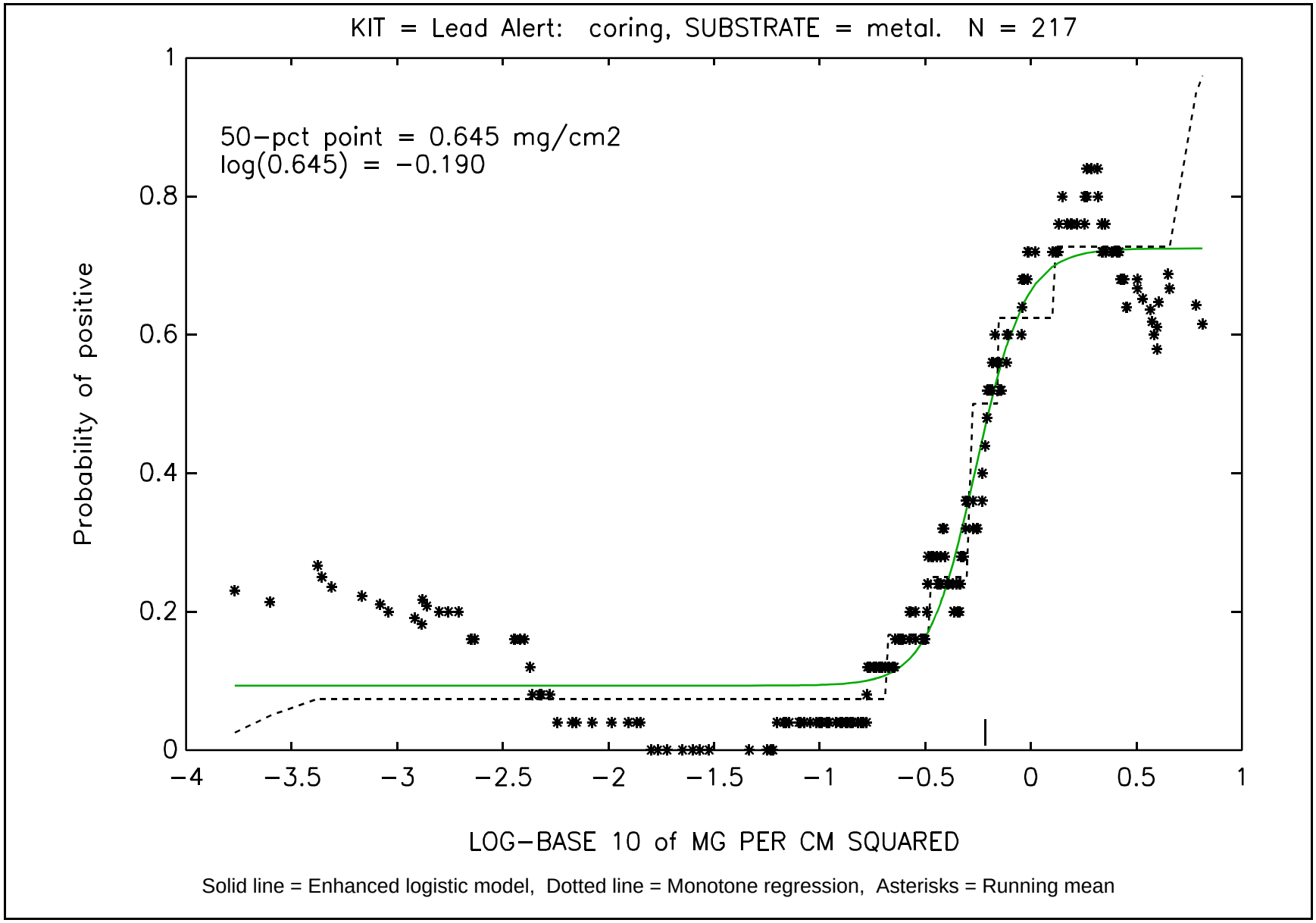

Figure 5-11. Operating characteristic curve for Lead Alert coring on metal.

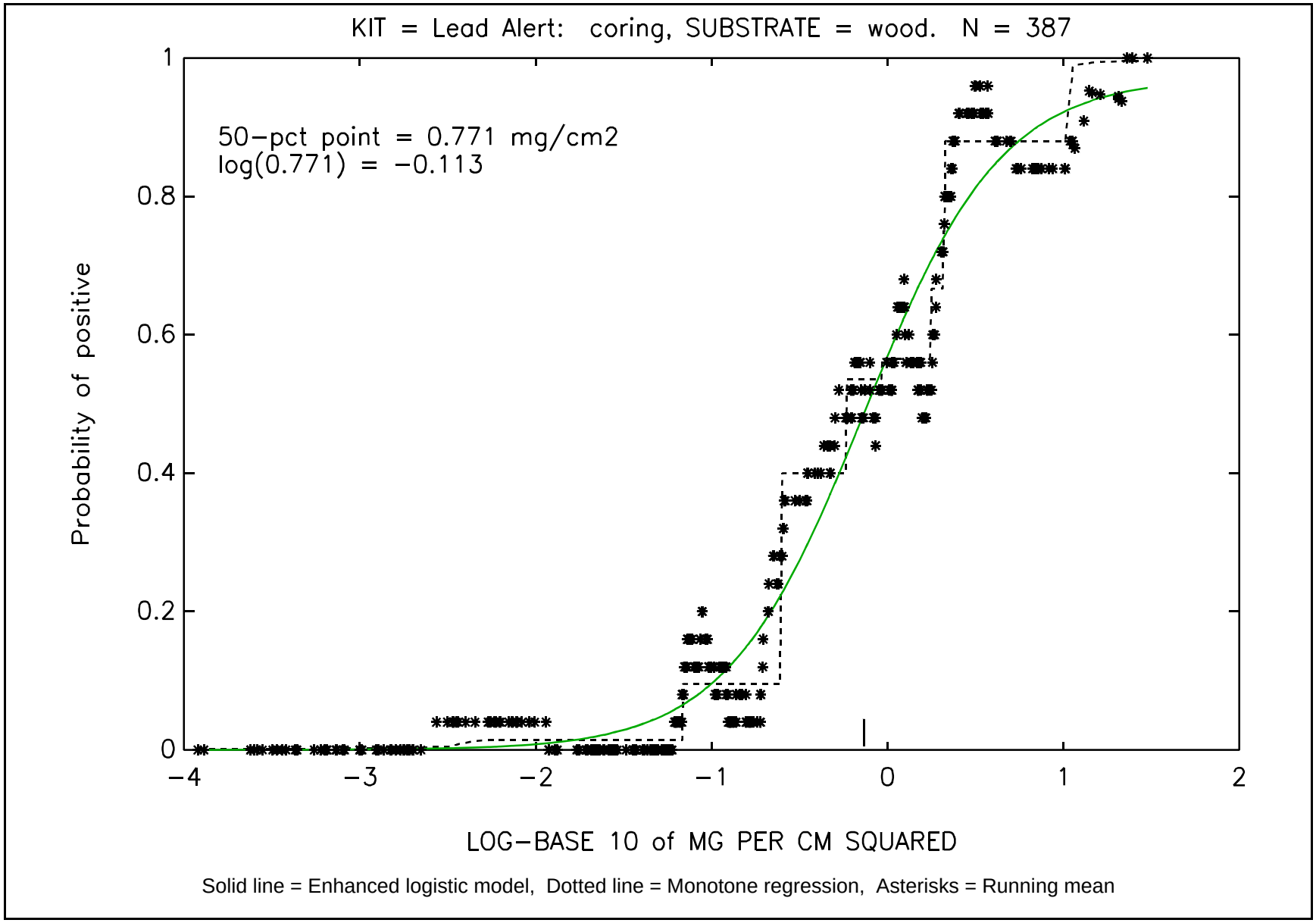

Figure 5-12. Operating characteristic curve for Lead Alert coring on wood.

5.2.3.2.2 Lead Alert: Coring on Concrete

There were 226 observations of Lead Alert with coring on concrete, of which 183 gave negative and 43 gave positive results. The running mean in Figure 5-10 is not an increasing function of the lead level, exhibiting a peak and a dip between 0.01 and 0.1 mg/cm^2. As a result, neither the model OC curve nor the monotone regression fit the data well at lower lead levels. It appears that the likelihood of a positive result may have approached zero as the lead level diminished, contrary to what the model OC curve indicates, since the 37 samples with ICP measurement less than 0.0061 mg/cm^2 all had negative results.

All three plots in Figure 5-10 suggest a threshold probability of less than 50 percent. The model and monotone regression estimates for this probability are .267 and .298 respectively, with 50-percent point estimates of 1.84 mg/cm^2 and 2.00 mg/cm^2. The rate of positive results did, however, increase rapidly as the lead level increased past the 50-percent point. There is no indication that false negatives were a problem at higher lead levels, with the probability of a positive approaching 100 percent with increasing lead levels.

5.2.3.2.3 Lead Alert: Coring on Drywall

There were 124 observations of Lead Alert with coring on drywall, but only 5 of these gave positive results. Table 5-19 therefore does not provide model estimates for drywall, nor are OC curves plotted. The 5 positives were not concentrated at the higher end of the lead range, already restricted to under 1.0 mg/cm^2.

The high rate of negatives is a desirable feature in that all negatives were correct classifications. It also could have resulted if the coring technique penetrated the outer layer of drywall and exposed gypsum, causing interference with the test kit.

5.2.3.2.4 Lead Alert: Coring on Metal

There were 217 observations of Lead Alert with coring on metal, of which 147 gave negative and 70 gave positive results. Figure 5-11 shows that the model OC curve and monotone regression are in close agreement, but that the running mean exhibits

peak-dips that affect the fit of these two curves. The prominent dip in the running mean occurring at a lead level of approximately $10^{-1.5}$ = 0.032 mg/cm^2 influenced estimation of both the model and monotone regression, to the extent that the significant baseline rate of false positives obtained from the model (.093) and monotone regression (ignoring the endpoint effect) may underestimate the true effect.

At higher lead levels the model reflects the substantial frequency of negative results obtained with the test kit at lead levels well above the 1.0 mg/cm^2 standard. This phenomenon is associated with a peak and dip that appears in the running mean at the higher end of measured ICP levels. The 50-percent point estimate of 0.645 mg/cm^2 from the model (0.607 mg/cm^2 from the monotone regression) and the steepness of the curve indicate a sharp drop in the frequency of positive results as the lead level declined from the 1.0 mg/cm^2 standard. At 1.0 mg/cm^2 the estimated probability of a positive result from the model is .664 (.625 from the monotone regression), leveling off at .725 with increasing lead levels.

5.2.3.2.5 Lead Alert: Coring on Plaster

There were 241 observations of Lead Alert with coring on plaster, but only 16 of these gave positive readings. As with drywall, an OC curve was not estimated for this substrate, nor were plots produced, due to the low incidence of positives. Consideration should be given to the possibility that the coring technique exposed substrate to the testing chemical and caused interference with color development of the test. The high rate of false negatives (85%) obtained with this kit on plaster suggests that negative readings were likely regardless of the lead level.

5.2.3.2.6 Lead Alert: Coring on Wood

There were 387 observations of Lead Alert with coring on wood, of which 259 gave negative and 128 gave positive readings. Figure 5-12 shows that the running mean, monotone regression, and model OC curve are in close agreement. The enhanced logistic model appears to give a good approximation to the performance of the test kit on wood.

Unlike other substrates, the estimated chance of observing a

positive result decreases to zero as the lead level diminishes, indicating that substrate interference was not a problem. The 50-percent point is estimated at 0.771 mg/cm^2 with the model (0.583 mg/cm^2 with monotone regression), and the threshold probability at .569 (.565 with monotone regression). Taking into account the 95% confidence interval reported in Table 5-19, the true threshold probability may have been less than one-half, or as large as two-thirds. The frequency of positive results, however, increased sharply as the level of lead increased from the 1.0 mg/cm^2 standard. The false positive (14%) and false negative (25%) rates shown in Table 5-6 reflect rapidly changing sensitivity of the kit near the standard, and the apparent lack of interference from non-lead factors. All 54 samples with ICP measurements less than 0.0059 mg/cm^2 had negative results, and all 15 samples with ICP levels greater than 10.9 mg/cm^2 had positive results.

5.2.3.2.7 Summary of Analysis for Lead Alert: Coring

The manufacturer did not recommend using the coring technique with Lead Alert on plaster, for reasons that may be reflected in the data. The high incidence of negative results for plaster may indicate a problem with substrate interference. The lack of lead levels above 1.0 mg/cm^2 on drywall makes it difficult to determine if the high rate of negative results that was observed was due to substrate interference, or to proper performance of the kit. Metal and brick, by contrast, had baseline rates of false positives that may indicate the existence of factors other than the level of lead in paint that affected performance. A similar observation can be made concerning the baseline rate of false negatives obtained on metal substrates. Baseline tendencies of neither kind were indicated on concrete and wood.

Lead Alert with coring appears to have had the best chance of returning a positive result at the 1.0 mg/cm^2 standard on brick. On concrete and wood the estimated chances are lower, but increase sharply with the lead level from the 1.0 mg/cm^2 standard.

5.2.3.3 Lead Alert: Sanding

This section describes the performance of the Lead Alert kit using the sanding technique. Louisville data were excluded because of noncompliance with the manufacturer's instructions. Table 5-20 gives the results of fitting enhanced logistic models to the substrates tested with Lead Alert using the sanding technique. Figures 5-13 through 5-15 illustrate the fit of these models to the data. Summaries by substrate are given in sections 5.2.3.3.1 through 5.2.3.3.6, and an overall summary is given in section 5.2.3.3.7.

Table 5-20. LEAD ALERT: SANDING Enhanced Logistic Regressions by Substrate.

SUBSTRATE	NEGATIVE	POSITIVE	MODEL PARAMETERS				Pb(.50) mg/cm²	PROB. AT Pb = 1
			c	d	a	b		
BRICK	27	8	---	---	---	---	---	---
CONCRETE	103	22	.122	.378	***24.7***	***100***	undefined [.729, ∞]	.500 [.154, .667]
DRYWALL	46	2	---	---	---	---	---	---
METAL	96	24	.082 (.043)	.348 (.086)	2.15 (4.54)	11.4 (13.0)	undefined [.951, ∞]	.394 [.175, .666]
PLASTER	112	6	---	---	---	---	---	---
WOOD	108	25	.021	.569	***-21.5***	***100***	1.24 [1.24, 10.8]	.021 [.005, .167]

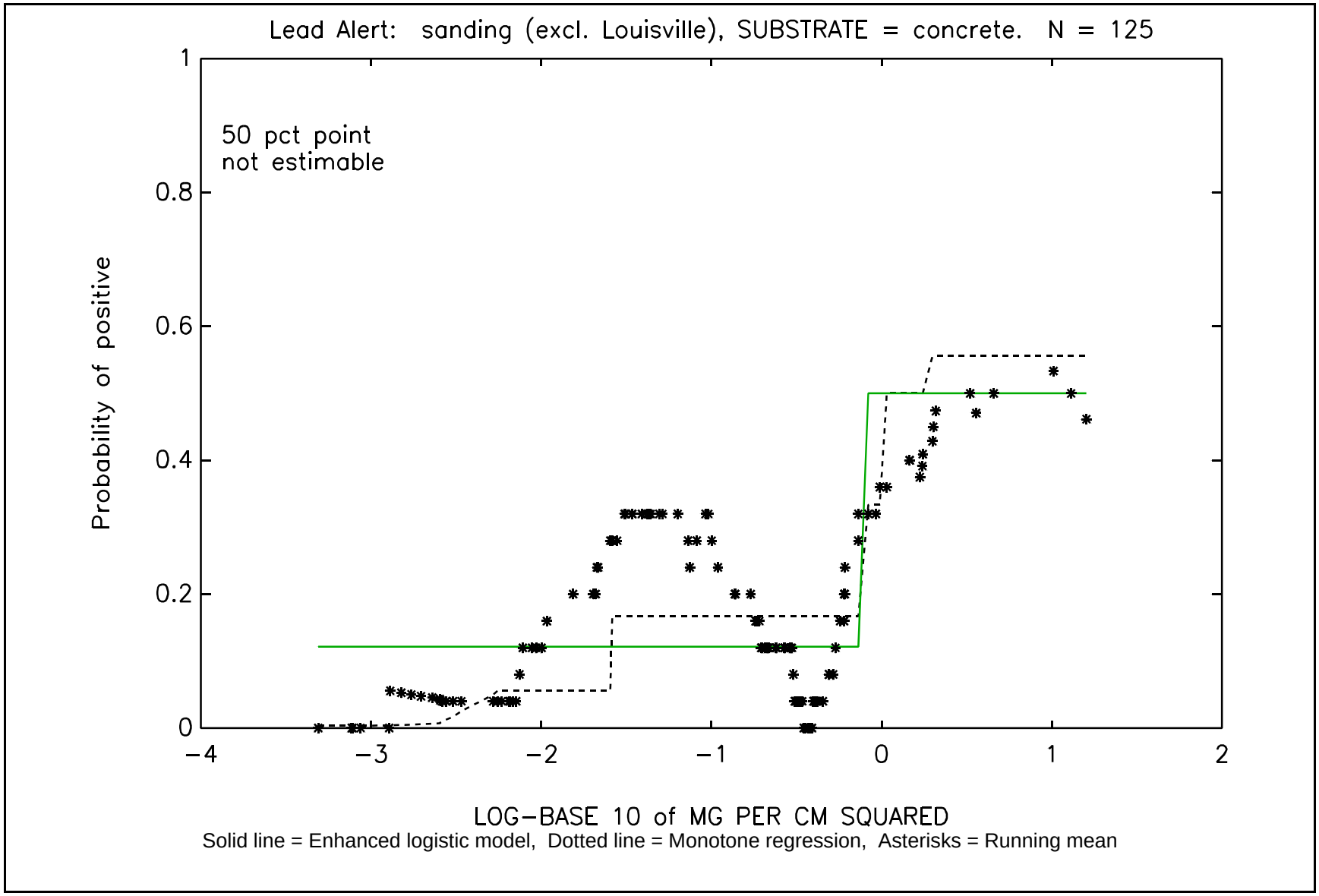

Figure 5-13. Operating characteristic curve for Lead Alert sanding on concrete, excluding Louisville.

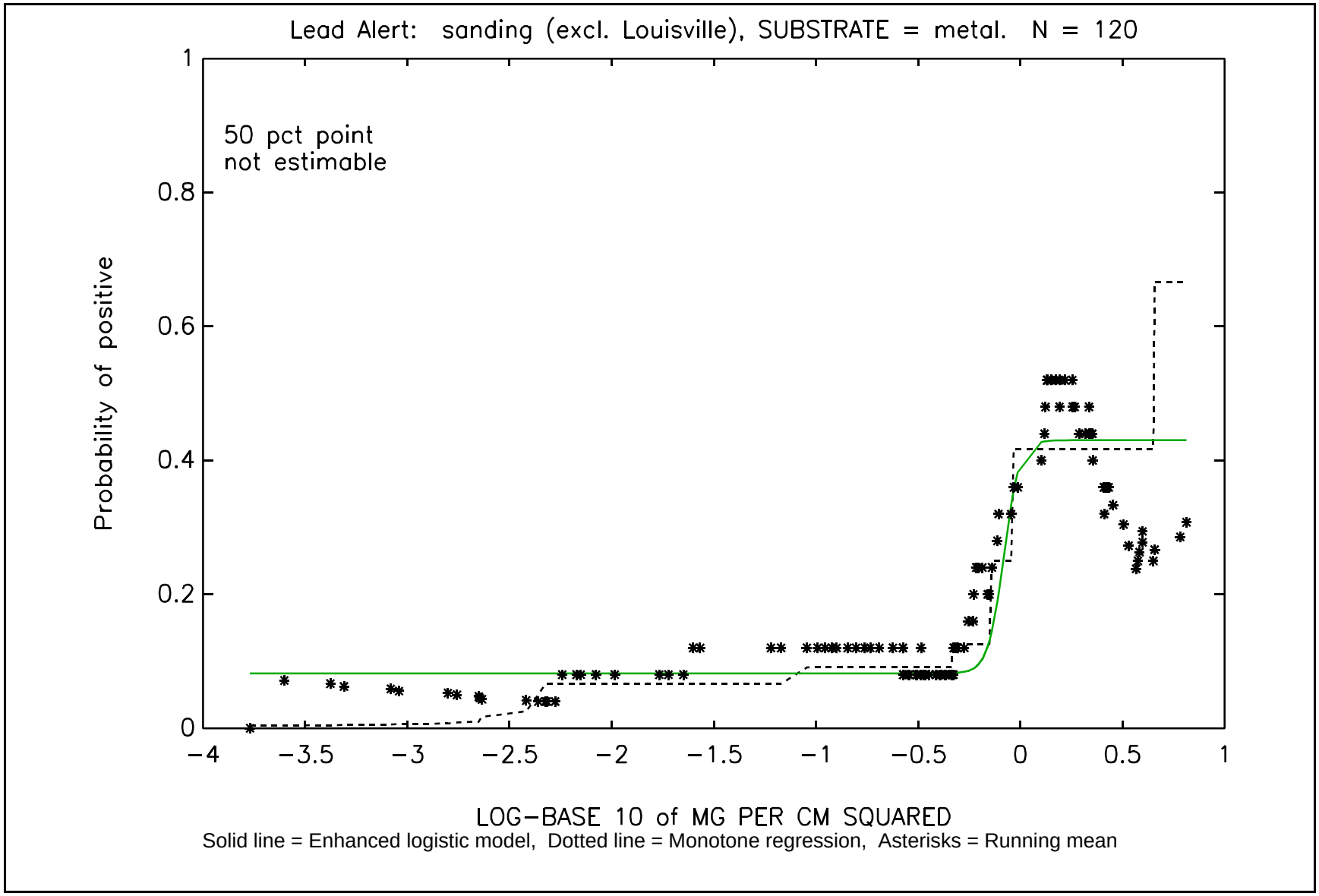

Figure 5-14. Operating characteristic curve for Lead Alert sanding on metal, excluding Louisville.

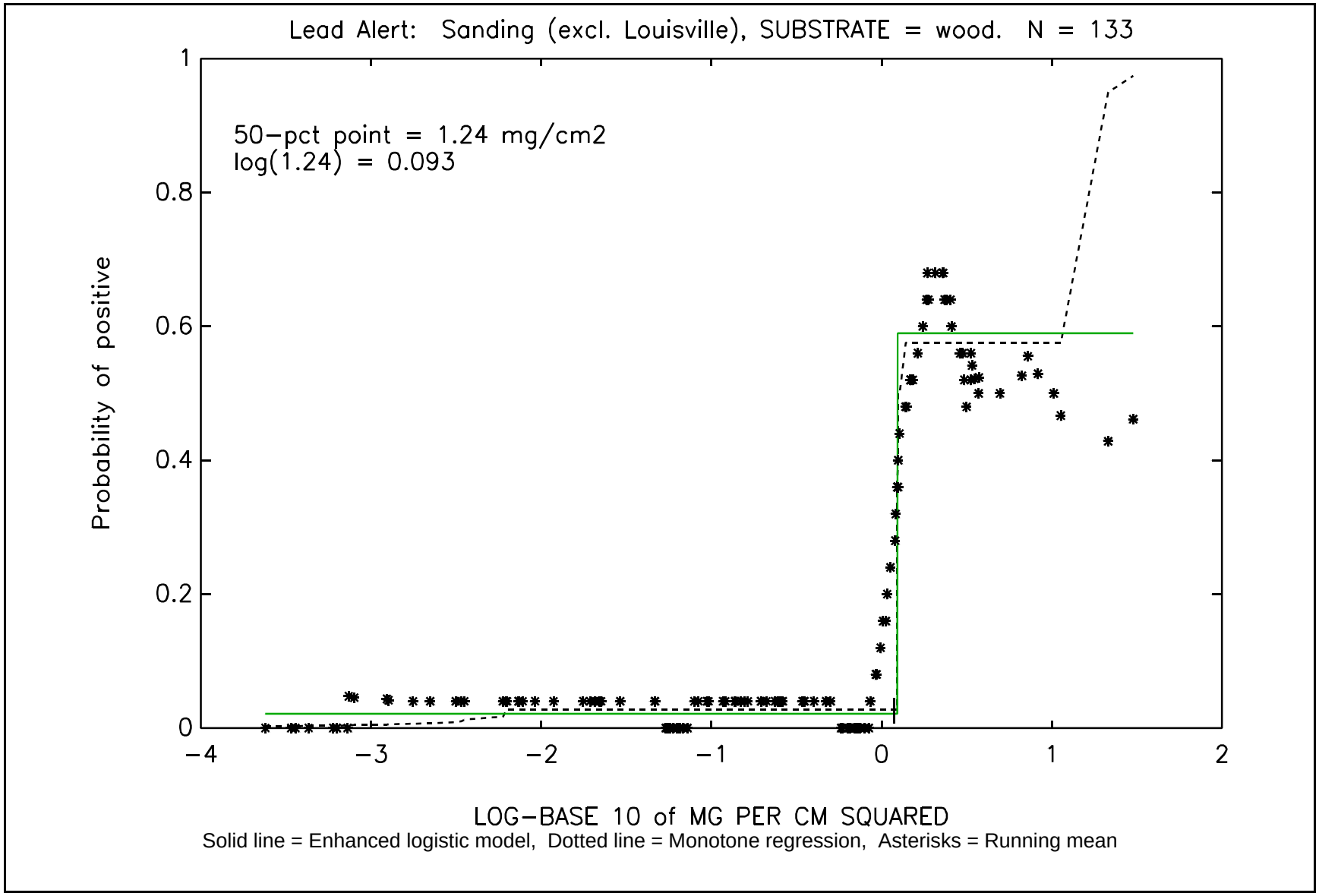

Figure 5-15. Operating characteristic curve for Lead Alert sanding on wood, excluding Louisville.

5.2.3.3.1 Lead Alert: Sanding on Brick

There were only 35 observations of Lead Alert with sanding on brick, of which 8 gave positive readings. It was not possible to estimate an OC curve accurately with so few data (an attempt to do so produced a model that exhibits a decreasing relationship with lead), and for this reason estimates were not obtained for this substrate.

5.2.3.3.2 Lead Alert: Sanding on Concrete

There were 125 observations of Lead Alert with sanding on concrete after the Louisville data were excluded, of which 103 gave positive and 22 gave negative readings. In Figure 5-13 the running mean does not show an increasing relationship, with the result that neither the model OC curve nor the monotone regression fits these data well. In fact, the running mean is similar to that for concrete using the coring technique (Figure 5-10). The sanding data, however, do not indicate sharp responsiveness to even high levels of lead. Perhaps the only conclusion that can be clearly drawn is that there is little evidence that the probability of a positive result exceeded 50 percent at any level of lead. As a result, the false negative rate shown in Table 5-7 is large (47%) relative to the false positive rate (13%).

The model estimate has the form of a step function, as indicated in Table 5-20 with $b = 100$. Both the model and monotone regression confirm that the test kit frequently gave negative results at high lead levels. The changepoint of the step function occurs at 0.78 mg/cm^2.

5.2.3.3.3 Lead Alert: Sanding on Drywall

There were 48 observations of Lead Alert with sanding on drywall excluding the Louisville data, and only two of these gave positive readings. OC curves were therefore not fit to these data. The high rate of negatives may indicate an interference of gypsum with the color development of the kit, or it may indicate that the kit performed as intended, since none of the ICP measurements on drywall exceeded 1.0 mg/cm^2 in the study.

5.2.3.3.4 Lead Alert: Sanding on Metal

There were 120 observations of Lead Alert with sanding on metal after the Louisville data were excluded, of which 96 gave negative and 24 gave positive results. Figure 5-14 shows the running mean, monotone regression, and model OC curve for these data. The peak-dip in the running mean is not highly unusual as a random occurrence, and otherwise the three estimates are in visual agreement. The model is nearly a step function (*b = 11.4*) with a changepoint near 0.8 mg/cm^2. Since the model does not produce probabilities greater than .43, it does not have a 50-percent point, and the monotone regression estimate (4.51 mg/cm^2) cannot be regarded as reliable.

A low rate of positives was obtained at all lead levels, implying a high baseline rate of false negatives. The estimated threshold probabilities from the model (.394) and monotone regression (.417) are below 0.5. Thus, the high rate of false negatives shown in Table 5-7 (56%) was possibly due largely to non-lead factors. As the level of lead diminishes, the model suggests a small baseline rate of false positives, but this is not confirmed in the monotone regression, and the 13 samples with ICP measurements less than 0.0044 mg/cm^2 gave negative results.

5.2.3.3.5 Lead Alert: Sanding on Plaster

There were 118 observations of Lead Alert with sanding on plaster excluding the Louisville data, but only 6 of these gave positive readings. As with brick and drywall, these were judged as too few to provide useful estimates of an OC curve. Like drywall, sanding may have breached substrate that interfered with the color development of the kit. The high rate of false negatives seen in Table 5-7 (91%) suggests that the tendency of the kit to produce negative results on plaster was not limited to paint samples with low lead readings. All 7 samples with ICP measurements greater than 1.534 mg/cm^2, including 2 with ICP measurements greater than 10.0 mg/cm^2, had negative results with this test kit.

5.2.3.3.6 Lead Alert: Sanding on Wood

There were 133 observations of Lead Alert with sanding on wood excluding the Louisville data, of which 108 returned negative and 25 returned positive results. The running mean, monotone regression, and model OC curve are shown in Figure 5-15. The running mean suggests a small probability of a positive

result for lead below 1.0 mg/cm^2, a high probability for lead above 1.0 mg/cm^2, and a sharp transition at 1.0 mg/cm^2. The model takes the form of a step function (*b = 100*), with a changepoint at 1.24 mg/cm^2. Like metal, the running mean decreases somewhat at higher lead levels, but otherwise the three OC curve estimates are in close agreement. The model and the monotone regression both appear to capture the salient features of the performance of this test kit on wood.

At lead levels just below the 1.0 mg/cm^2 standard the estimated probability of a positive result drops nearly to zero. At levels just above the standard, the probability increases to about .59 under both the model and the monotone regression. A high rate of negatives was observed for lead levels as high as 10.0 mg/cm^2. Thus, non-lead factors may explain most of the false negative rate of 50% shown in Table 5-7.

5.2.3.3.7 Summary of Analysis for Lead Alert: Sanding

The manufacturer's instructions warned that chemical interference may occur from gypsum or plaster dust. This may have happened on drywall and plaster samples in Denver and Philadelphia, since very few positive results were observed. The rate of positives was low for all substrates, suggesting a high baseline probability of false negatives for a wide variety of materials. The kit was not prone to giving false positive results as the lead level approached 0.0 mg/cm^2.

Comparing results for the sanding technique with those for the coring technique must take into account the exclusion of the Louisville data from the sanding, but not the coring analyses. When Louisville data were excluded from both and rates of positives and negatives compared on common sites, both coring and sanding exhibited high negative rates on all substrates except brick, where the rate was high for sanding (27 negatives, 8 positives) but not for coring (19 negatives, 16 positives). On plaster and drywall both techniques produced very high negative rates.

5.2.3.4 Lead Detective

Table 5-21 gives the results of fitting enhanced logistic models to the six substrates tested with Lead Detective. Figures 5-16 through 5-21 illustrate the fit of these models to the data.

Sections 5.2.3.4.1 through 5.2.3.4.6 discuss the estimated OC curves by substrate. Section 5.2.3.4.7 contains an overall summary for this test kit.

5.2.3.4.1 Lead Detective on Brick

There were 92 observations of Lead Detective on brick, of which 41 returned negative and 51 returned positive readings. Figure 5-16 shows the running mean, monotone regression, and

Table 5-21. LEAD DETECTIVE Enhanced Logistic Regressions by Substrate.

SUBSTRATE	NEGATIVE	POSATIVE	MODEL PARAMETERS				Pb(.50) mg/cm²	PROB. AT Pb = 1
			c	d	a	b		
BRICK	41	51	.176 (.067)	.631 (.085)	15.8 (7.24)	5.35 (3.17)	0.053 [.003, .095]	.807 [.669, .896]
CONCRETE	137	89	.350	.232	***51.9***	***100***	0.595 [.328, 8.65]	.582 [.390, .714]
DRYWALL	88	36	0	1	-0.686 (.385)	0.049 (.078)	large or nonexistent	.335 [.189, .521]
METAL	123	92	.251 (.054)	.496 (.097)	3.80 (5.49)	6.40 (9.28)	0.553 [.404, .758]	.736 [.601, .838]
PLASTER	149	93	.326 (.108)	.426 (.232)	-0.306 (8.62)	2.87 (2.64)	0.977 [.392, 2.19]	.507 [.365, .778]
WOOD	185	203	.099 (.055)	.902 (.055)	1.10 (.194)	0.814 (.146)	0.198 [.094, .671]	.775 [.709, .830]

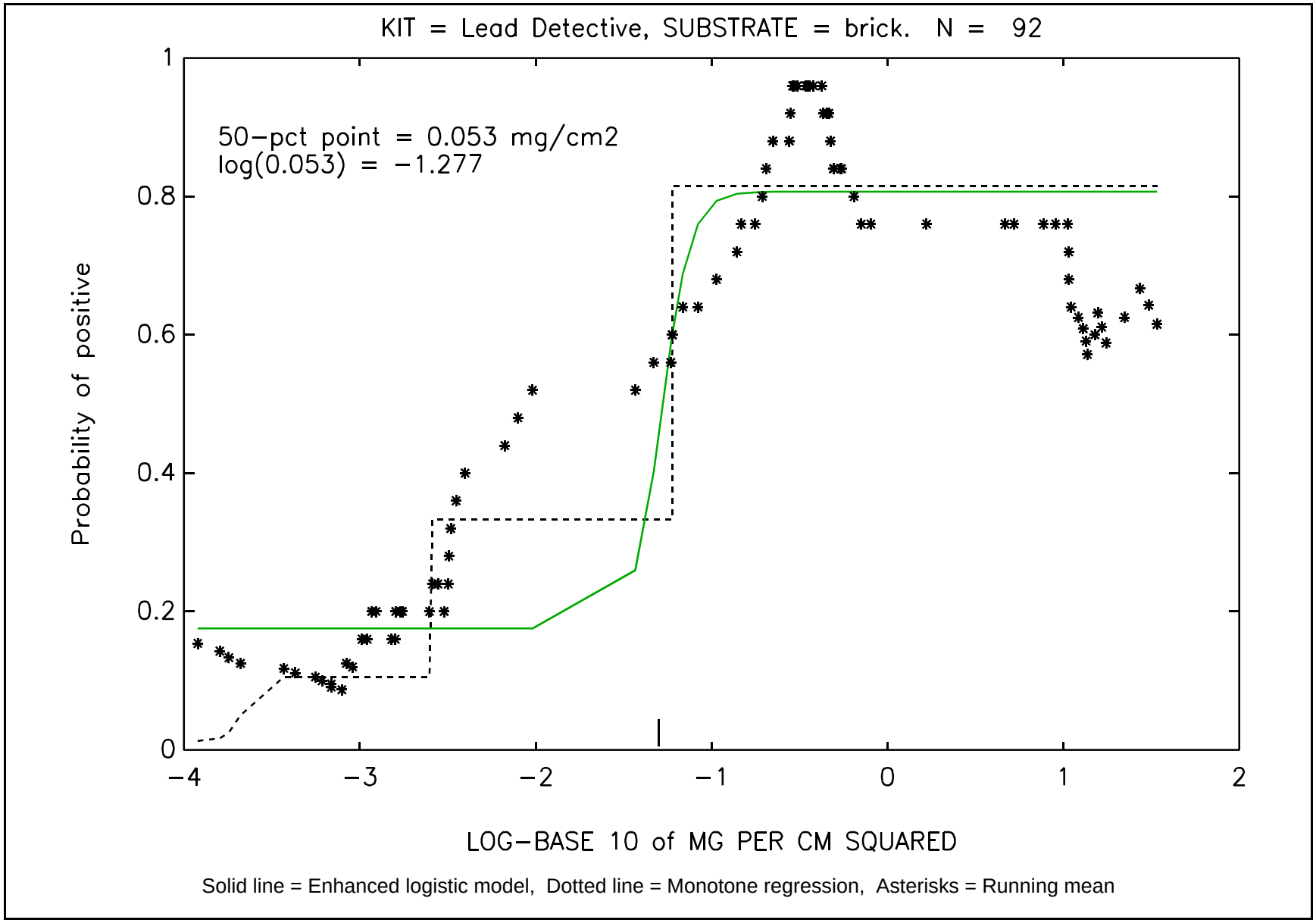

Figure 5-16. Operating characteristic curve for Lead Detective on brick.

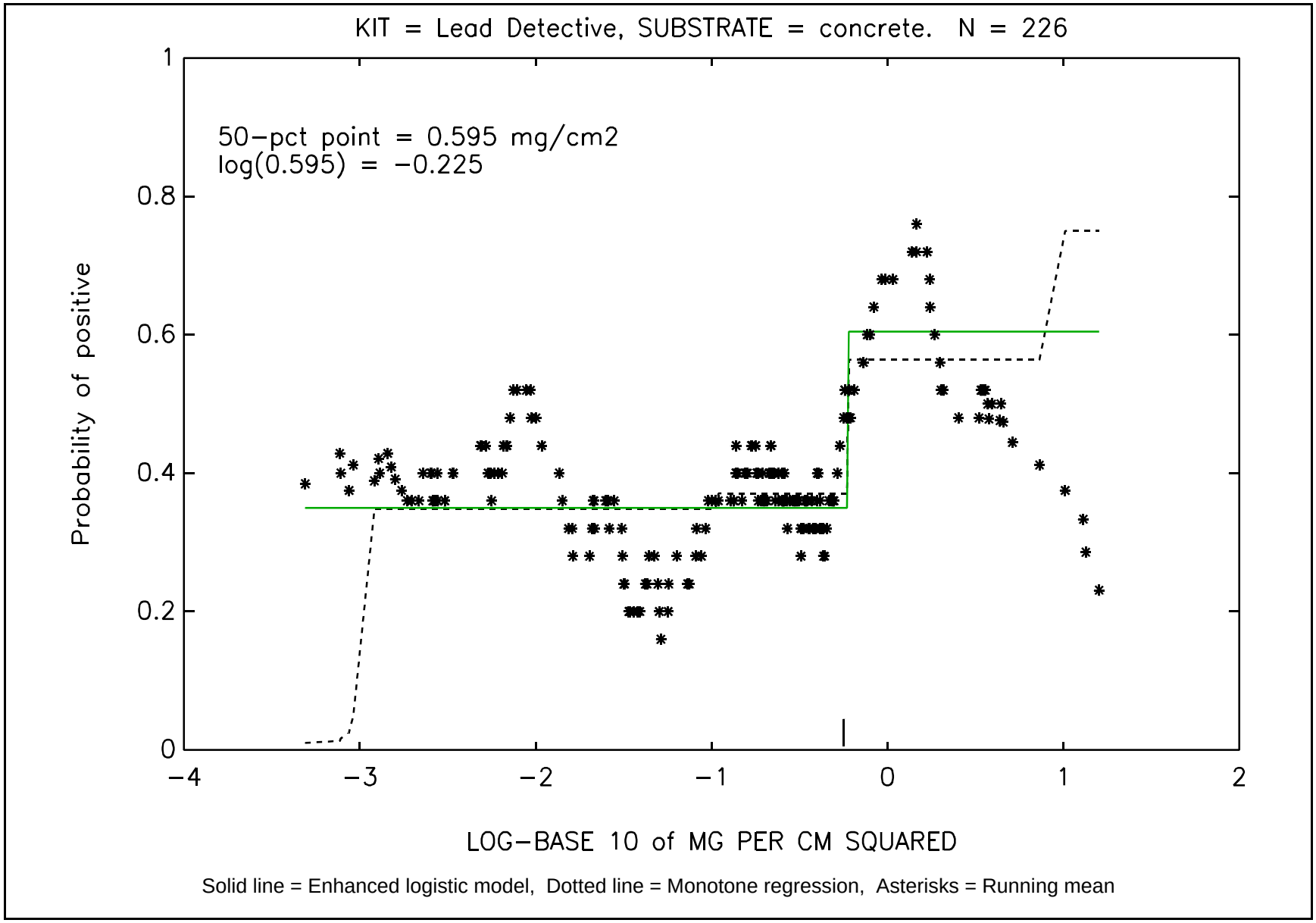

Figure 5-17. Operating characteristic curve for Lead Detective on concrete.

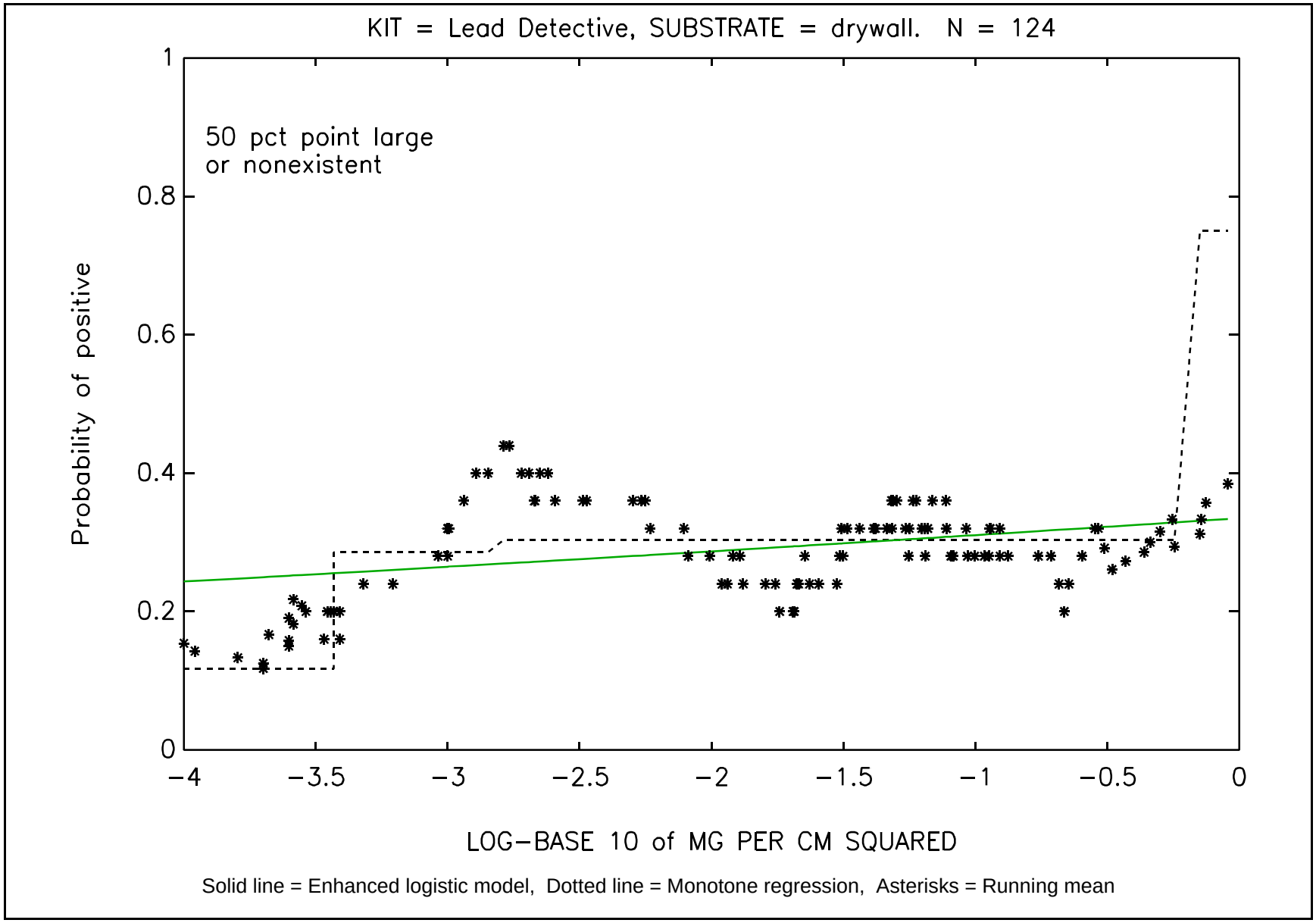

Figure 5-18. Operating characteristic curve for Lead Detective on drywall.

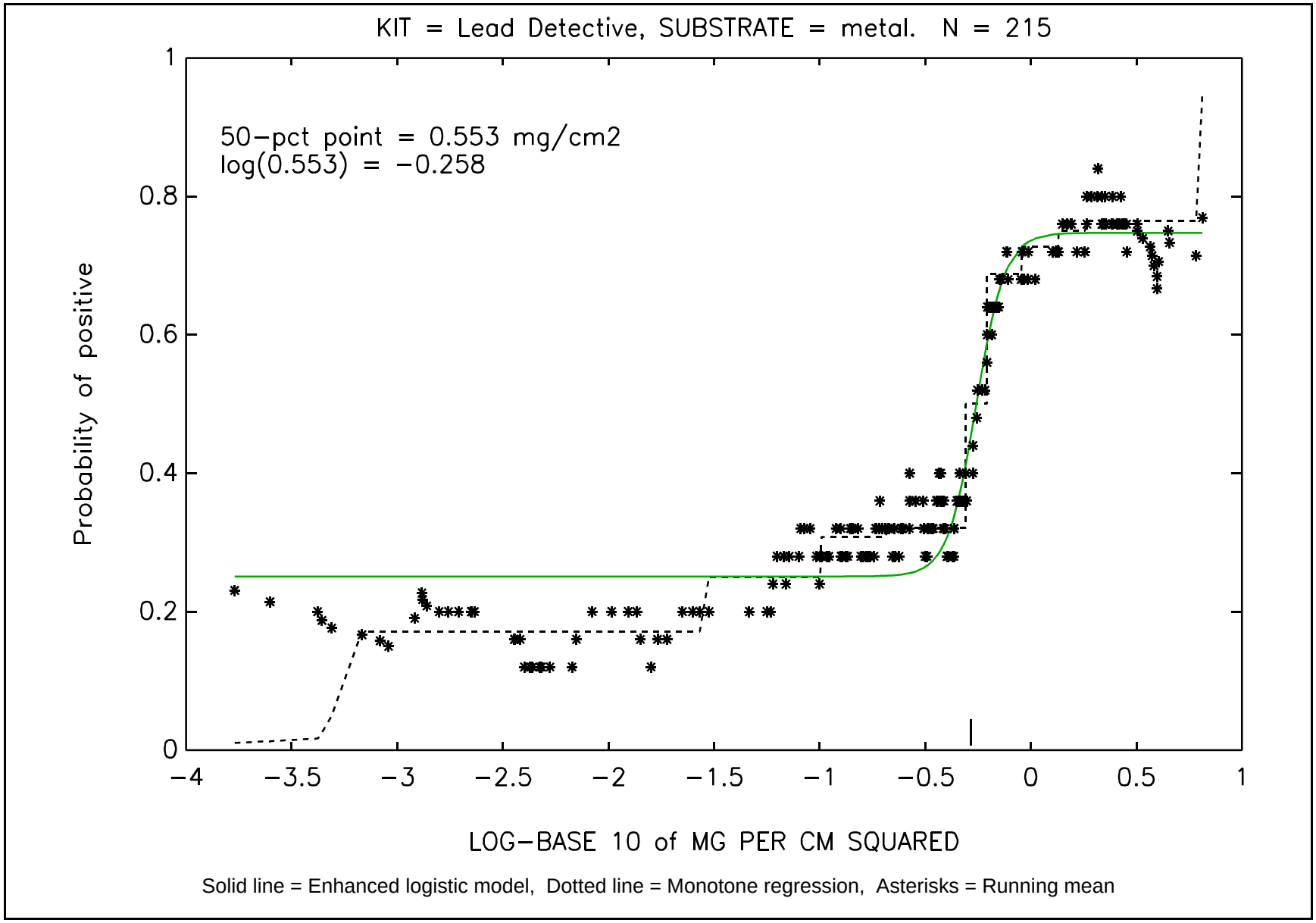

Figure 5-19. Operating characteristic curve for Lead Detective on metal.

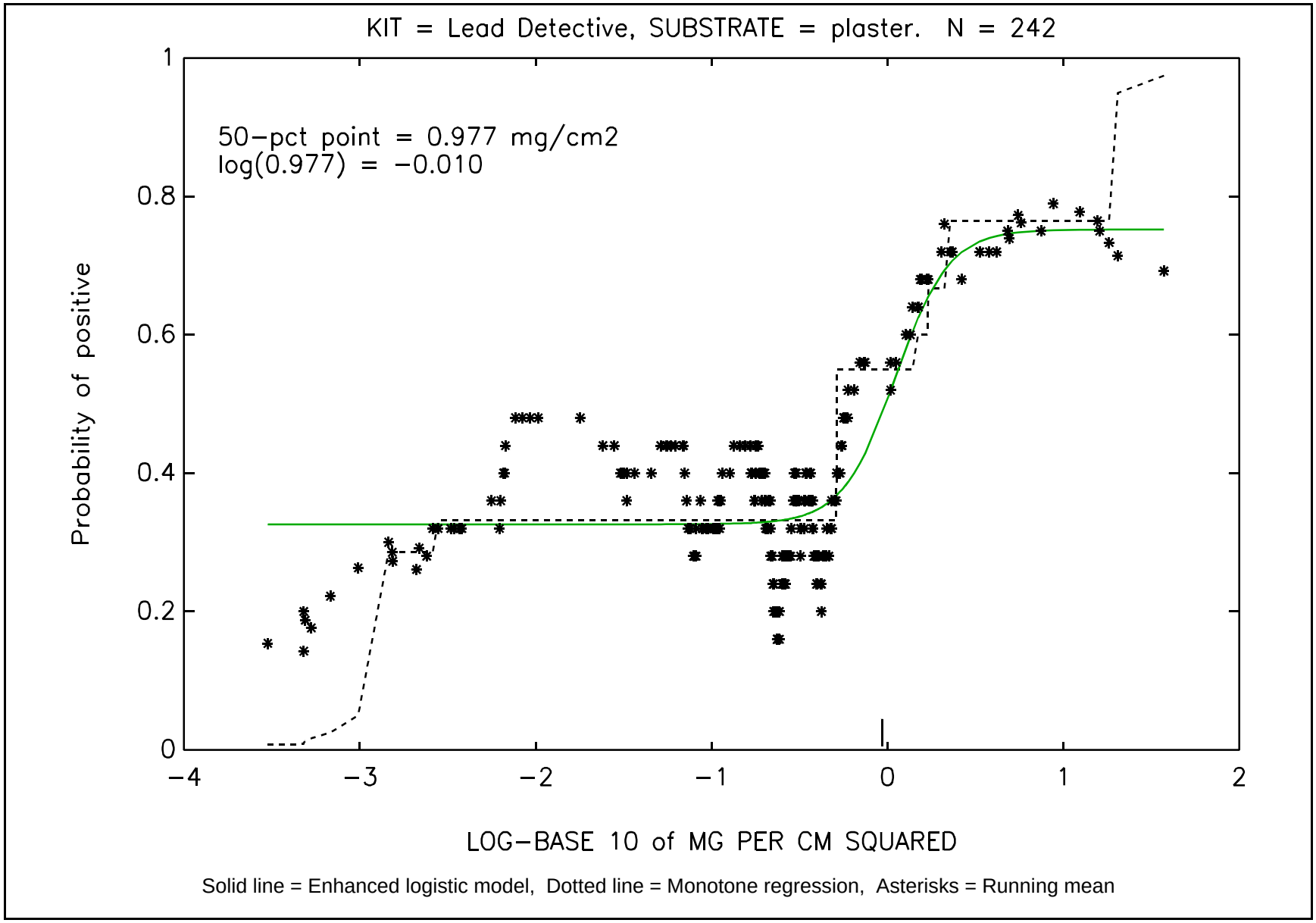

Figure 5-20. Operating characteristic curve for Lead Detective on plaster.

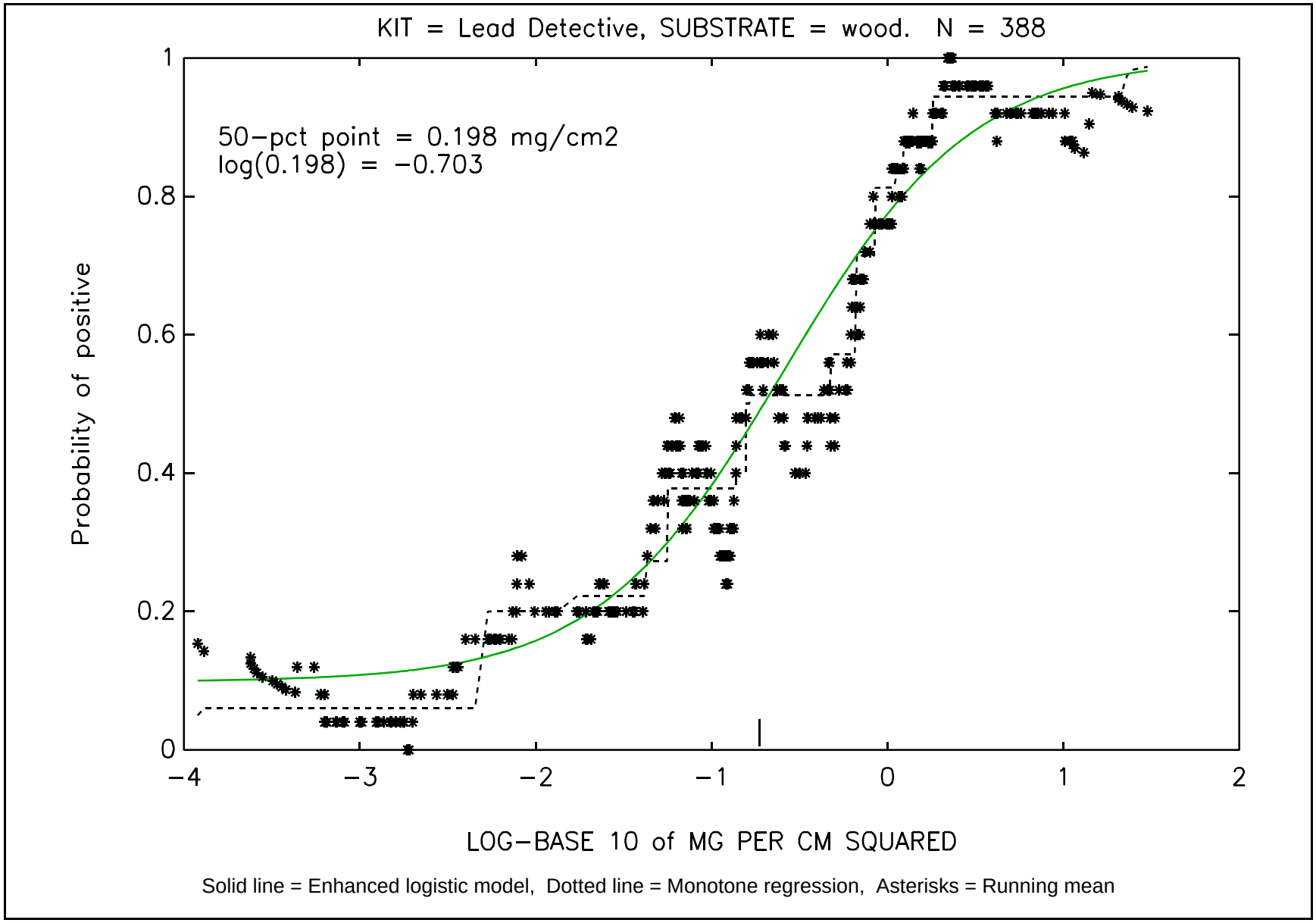

Figure 5-21. Operating characteristic curve for Lead Detective on wood.

model OC curves that were estimated from the data. The peak-dip that appears in the running mean just below 1.0 mg/cm^2 (zero on the log-scale) would not be highly unusual as a random occurrence.

The model nearly has the appearance of a step function ($b = 5.35$), and is similar to the monotone regression for lead levels above 0.01 mg/cm^2.

Both the model and the monotone regression suggest a high baseline rate of false negatives, estimated at about 20 percent. A baseline rate of false positives is estimated at .176 with the model. This false positives estimate appears to be lower, or even zero, in the monotone regression, although it is clear that positive results were frequently obtained at lead levels well below 0.01 mg/cm^2.

The estimated 50-percent points with the model (0.053 mg/cm^2) and monotone regression (0.060 mg/cm^2) are substantially less than the 1.0 mg/cm^2 standard, and the running mean suggests an even lower 50-percent point. The threshold probability (.807 model and .818 monotone regression) is consequently estimated to be high. The high rate of false positives shown in Table 5-8 (55%) may have been due primarily to sensitivity of the kit to low levels of lead, and secondarily to interference from non-lead factors.

5.2.3.4.2 Lead Detective on Concrete

There were 226 observations of Lead Detective on concrete, of which 137 gave negative and 89 gave positive readings. The running mean in Figure 5-17 indicates only a weak relationship between the rate of positives and the level of lead. Likewise, the monotone regression and model are only mildly responsive to the lead level. The overall rate of positives was 39 percent. The model has the form of a step function, with an estimated probability of .350 for lead levels less than 0.595 mg/cm^2, and .582 for lead levels above.

5.2.3.4.3 Lead Detective on Drywall

There were 124 observations of Lead Detective on drywall, of which 88 gave negative and 36 gave positive results. The running mean in Figure 5-18 suggests only a mild responsiveness of the

kit to the level of lead in the paint samples tested, notwithstanding the fact that all of the ICP measurements were below 1.0 mg/cm^2. Due to the small slope coefficient *b* obtained from the model, the estimated 50-percent point is too large (over 1,000 mg/cm^2) to be regarded as meaningful, and it is difficult to tell if a 50 percent chance of a positive result was achieved at any level of lead on drywall. The estimated chance of a positive result remains fixed at about 30 percent for lead at the 1.0 mg/cm^2 standard down to about 0.001 mg/cm^2, below which it tapers off towards zero. The samples with the 15 smallest ICP measurements all had negative results. The rate of false positives shown in Table 5-8 (29%) appears to reflect sensitivity to low levels of lead rather than interference.

5.2.3.4.4 Lead Detective on Metal

There were 215 observations of Lead Detective on metal, of which 123 gave negative and 92 gave positive results. The manufacturer's instructions warned against the use of this kit on painted metal, due to color interference that may lead to false positive readings. Figure 5-19 suggests that this concern may have been valid. The running mean, monotone regression (ignoring the endpoint effects), and model OC curve each exhibit high baseline rates of false positives, but not unlike the graphs for other substrates for which no concern was expressed. A high baseline rate of false negatives may also be indicated.

The model and monotone regression both appear to fit the data well. The baseline probability of false positives is estimated at .251 with the model, but the monotone regression suggests a somewhat lower rate. The 50-percent point is estimated at 0.553 mg/cm^2 both with the model and the monotone regression; the threshold probability at .736 model and .727 monotone regression; and baseline probability of false negatives at about 25 percent. Since the overall rate of false negatives from Table 5-8 is 27%, interference from non-lead factors was possibly a major factor.

Both the model and the monotone regression suggest that the probability of a positive result at the 1.0 mg/cm^2 standard was high, and both captured the sharp decline in the observed frequency of positives as the lead level decreased from the standard. As a result, as much as half of the false positive rate shown in Table 5-8 (34%) may be due to non-lead factors.

5.2.3.4.5 Lead Detective on Plaster

There were 242 observations of Lead Detective on plaster, of which 149 gave negative and 93 gave positive readings. A baseline rate of false positives is estimated at .326 by the model, which agrees with the monotone regression ignoring the effect of the 7 lowest ICP measurements, all of which had negative results. This may explain, to a large extent, the high rate of false positives shown in Table 5-8 (34%).

Plots of the model and the monotone regression estimates (Figure 5-20) suggest that the 50-percent point was near the 1.0 mg/cm^2 standard, although the 95% confidence interval in Table 5-21 could place this point as low as 0.392 mg/cm^2 or as high as 2.19 mg/cm^2. They also give similar estimates of the baseline probability of false negatives, at about 25 percent, or most of the overall rate of false negatives shown in Table 5-8 (33%).

5.2.3.4.6 Lead Detective on Wood

There were 388 observations of Lead Detective on wood, of which 185 gave negative and 203 gave positive readings. Figure 5-21 shows that the running mean, monotone regression, and model OC curve are in agreement. The model estimates the baseline rate of false positives at .099. The high rate of false positives shown in Table 5-8 (35%) was possibly due to sensitivity of the kit to low levels of lead.

The model and the running mean produce similar estimates of the 50-percent point (0.198 mg/cm^2 model and 0.160 mg/cm^2 monotone regression) and threshold probability (.775 and .813, respectively).

5.2.3.4.7 Summary of Analysis for Lead Detective

Lead Detective is a sodium sulfide kit that the manufacturer did not recommend for use on metal, but it was recommended for use on wood, wallboard (drywall) and plaster. A high baseline rate of false positives was observed on metal, but also on concrete, plaster, and to a lesser extent on brick. The probability of a positive result with the kit did not appear to change sharply with the lead level on drywall, concrete, and plaster. A high threshold probability was indicated for brick, metal, and wood.

5.2.3.5 Lead Zone

Table 5-22 gives the results of fitting enhanced logistic models to the six substrates tested with the Lead Zone kit. Figures 5-22 through 5-27 graphically show how well these models fit the data. Sections 5.2.3.5.1 through 5.2.3.5.6 discuss the estimated OC curves by substrate. Section 5.2.3.5.7 contains an

Table 5-22. LEAD ZONE Enhanced Logistic Regressions by Substrate.

SUBSTRATE	NEGATIVE	POSITIVE	MODEL PARAMETERS				Pb(.50) mg/cm²	PROB. AT Pb = 1
			c	d	a	b		
BRICK	46	47	0	1	1.48 (.420)	0.586 (.104)	0.080 [.007, .346]	.815 [.656, .911]
CONCRETE	152	74	.243 (.031)	.757 (.031)	-3.23 (1.50)	7.92 (2.66)	1.38 [.424, 1.74]	.272 [.185, .380]
DRYWALL	96	27	0	1	0.588 (.465)	0.505 (.132)	0.312 [.080, 1.21]	.643 [.416, .820]
METAL	137	79	.169 (.039)	.693 (.099)	0.414 (.791)	2.53 (.784)	0.819 [.469, 1.38]	.586 [.398, .752]
PLASTER	166	76	.134 (.051)	.519 (.111)	1.44 (.911)	1.64 (.598)	0.711 [.342, 2.49]	.553 [.419, .680]
WOOD	182	205	0	1	1.36 (.179)	0.703 (.073)	0.145 [.055, .583]	.795 [.731, .848]

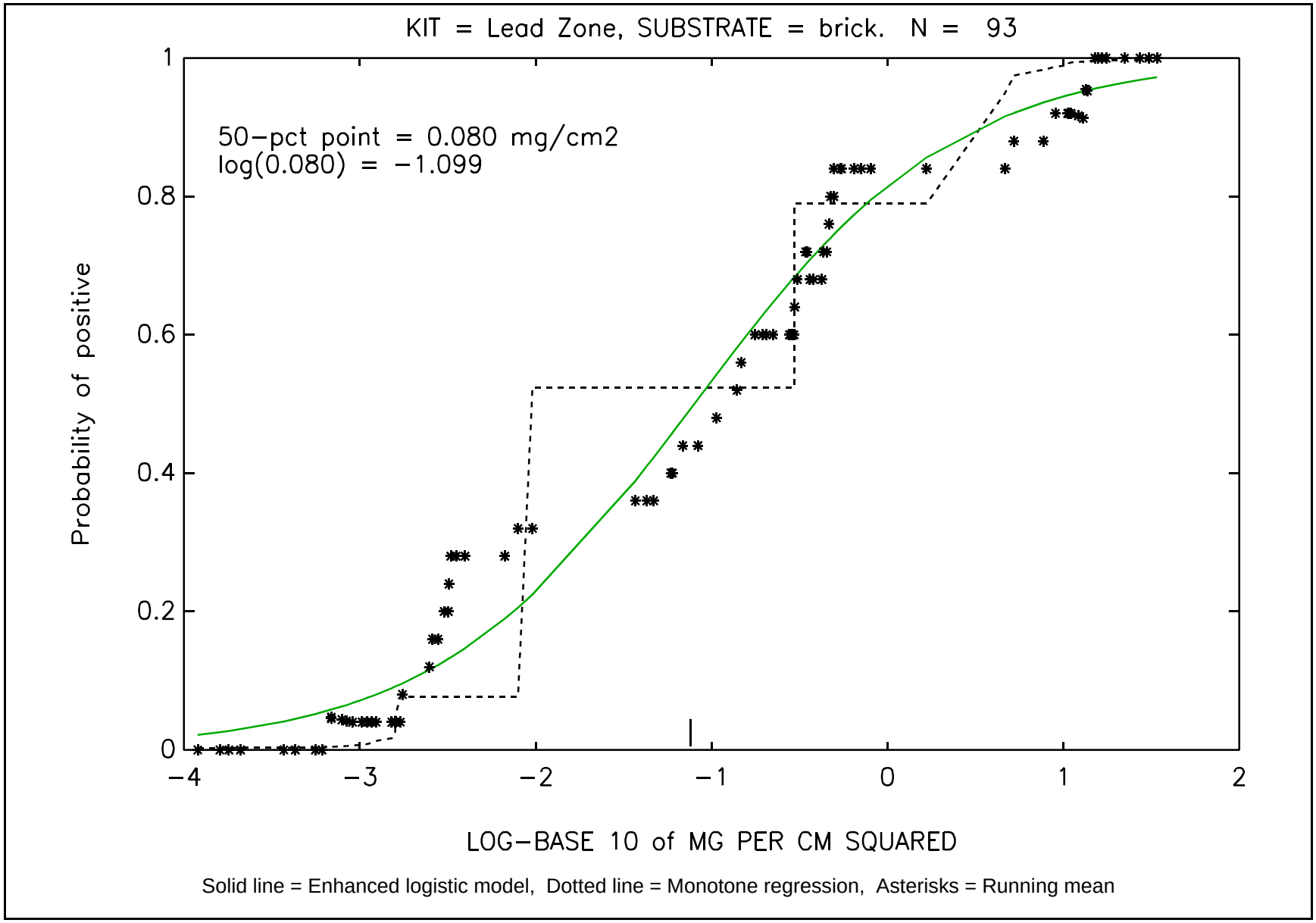

Figure 5-22. Operating characteristic curve for Lead Zone on brick.

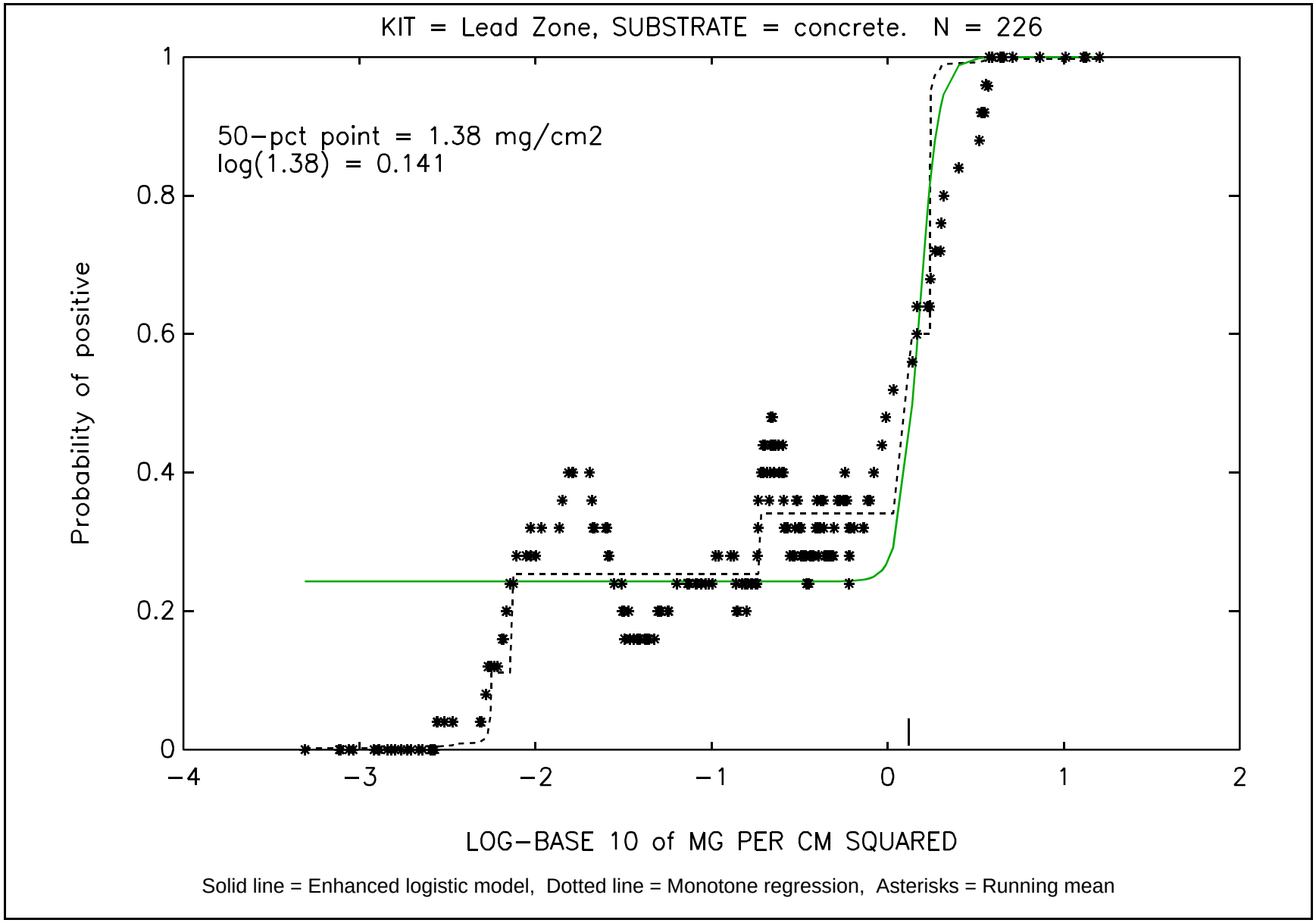

Figure 5-23. Operating characteristic curve for Lead Zone on concrete.

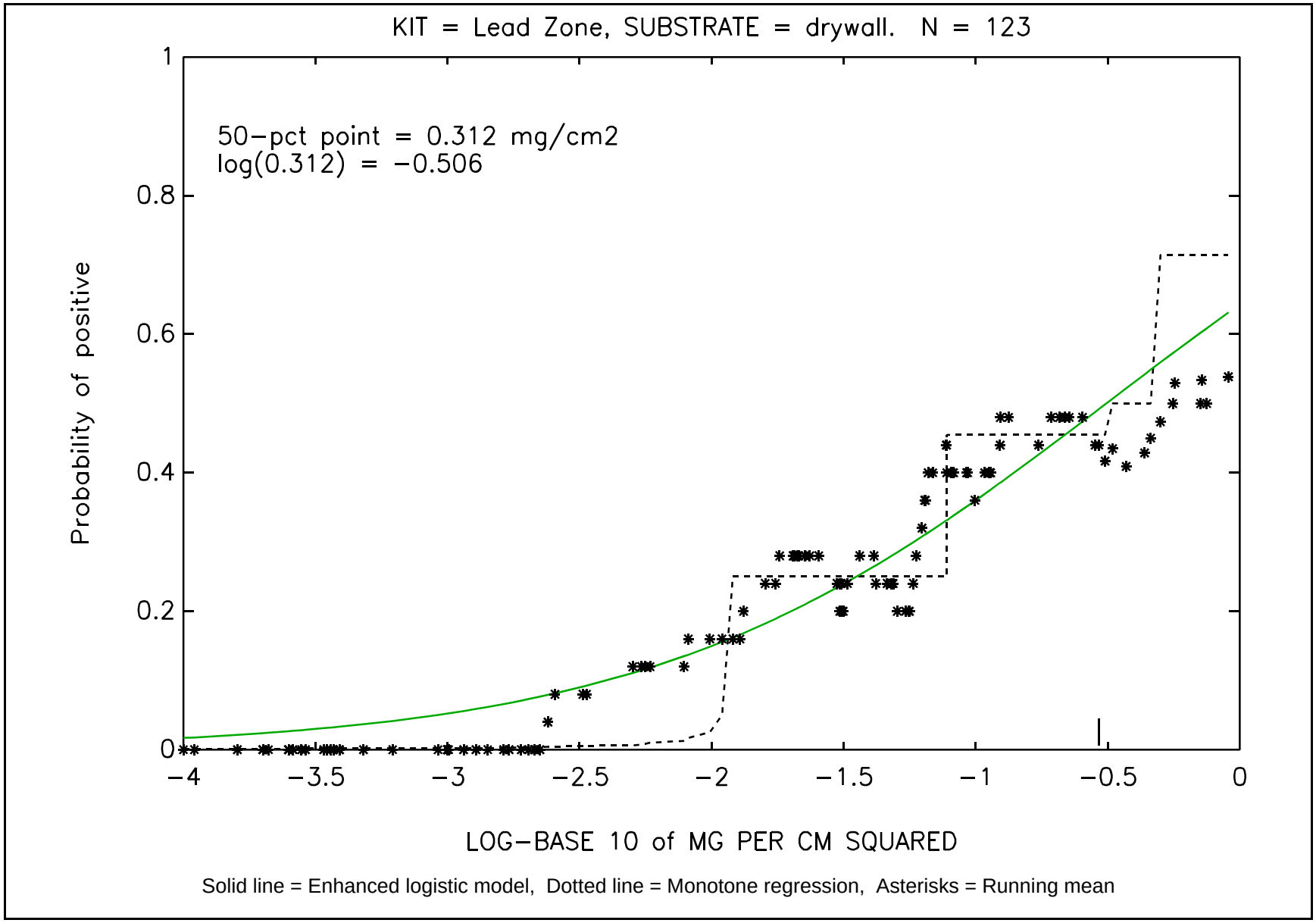

Figure 5-24. Operating characteristic curve for Lead Zone on drywall.

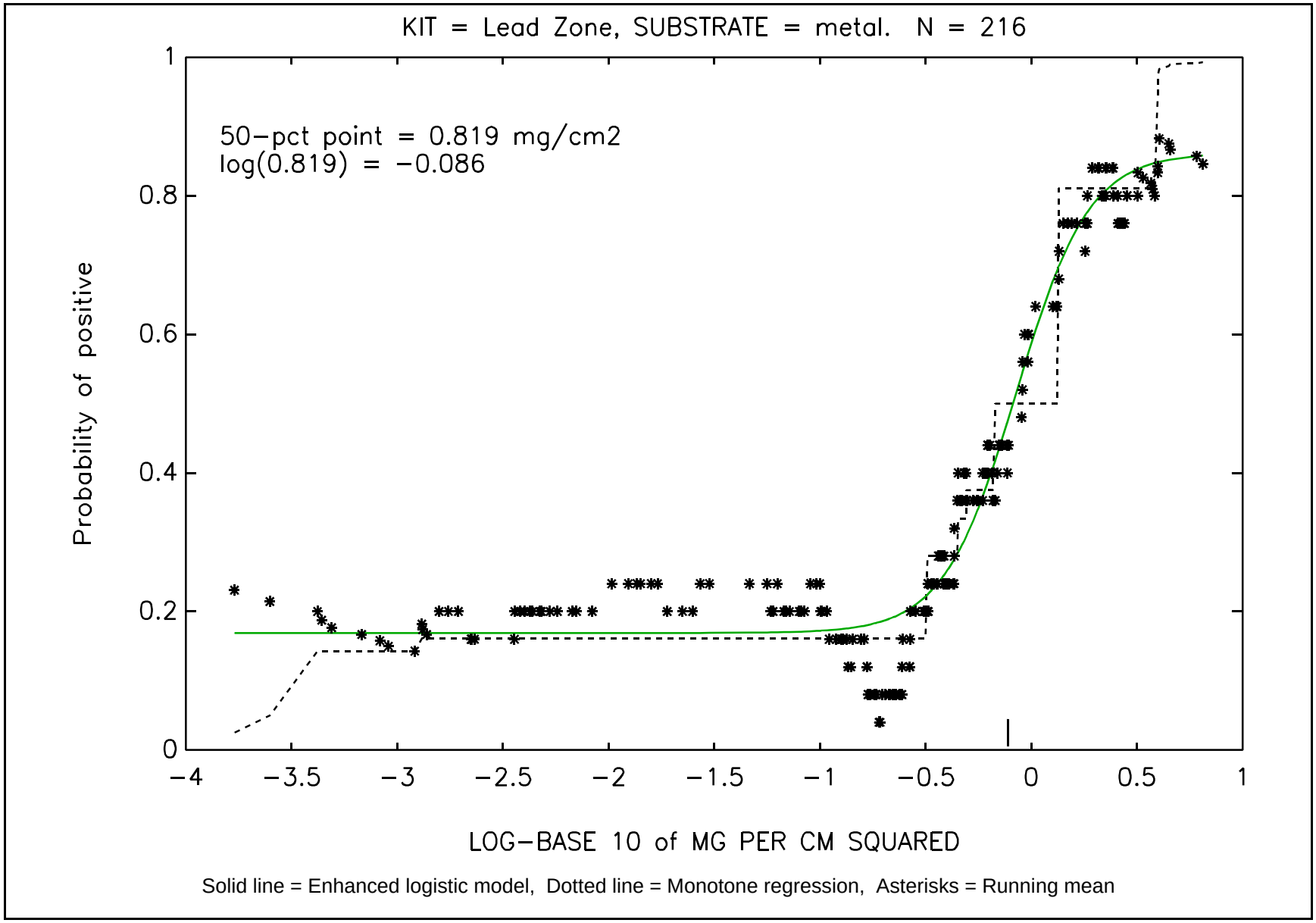

Figure 5-25. Operating characteristic curve for Lead Zone on metal.

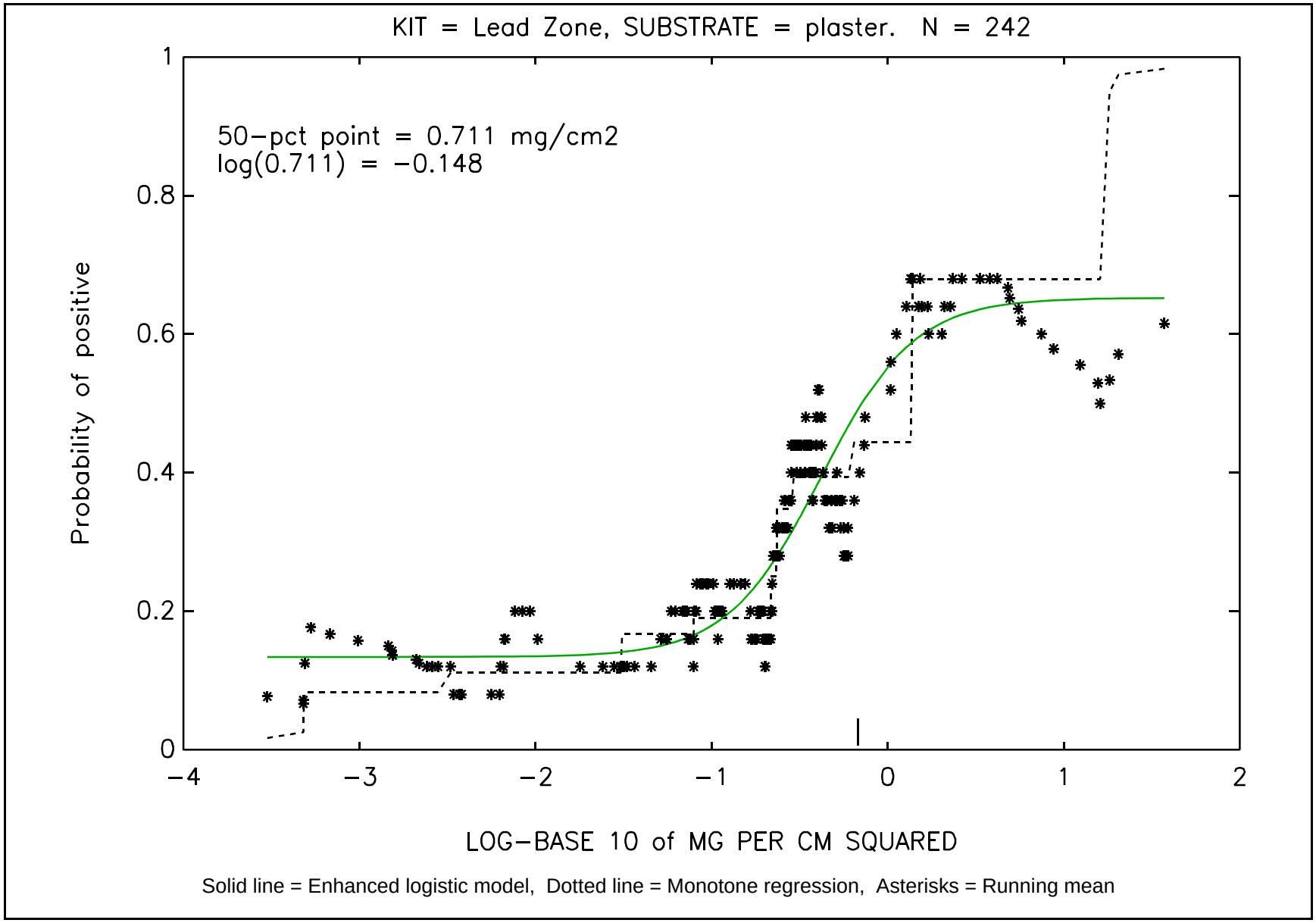

Figure 5-26. Operating characteristic curve for Lead Zone on plaster.

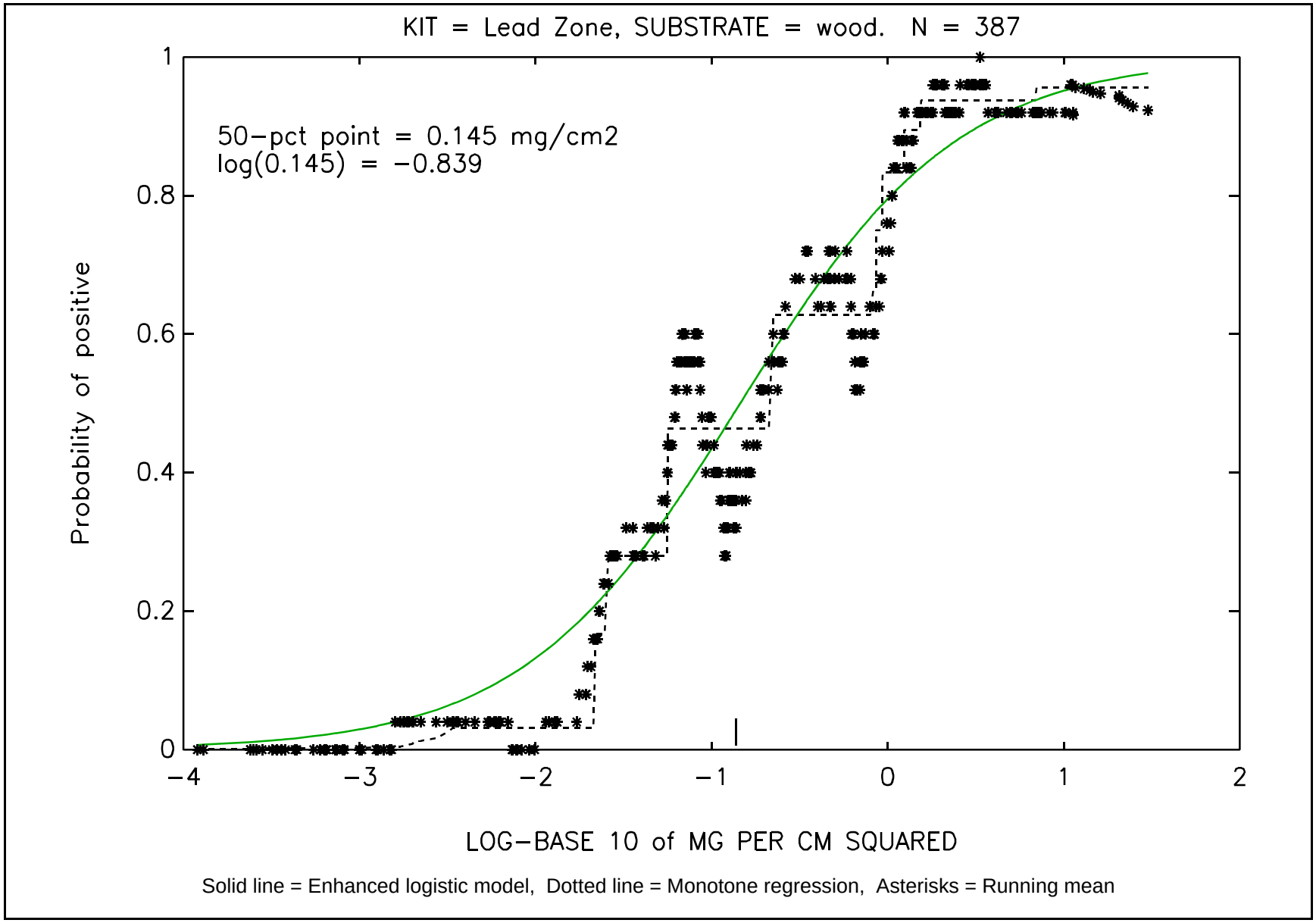

Figure 5-27. Operating characteristic curve for Lead Zone on wood.

overall summary for the test kit.

5.2.3.5.1 Lead Zone on Brick

There were 93 observations of Lead Zone on brick, of which 46 gave negative and 47 gave positive results. Figure 5-22 shows that the model, monotone regression, and model OC curve are in general agreement, although multiple inflection points suggested by the running mean were not picked up by the model. The kit does not appear to have exhibited baseline rates of false positives or false negatives on this substrate. The 20 samples with ICP measurements less than 0.0016 mg/cm^2 all had negative results; the 20 samples with ICP measurements greater than 4.657 mg/cm^2 all had positive results.

The model and monotone regression produce similar estimates of the threshold probability (.815 and .790 respectively). Accounting for sampling variability with a 95% confidence interval does not bring this estimate lower than .656. The 50-percent point estimates from the model (0.080 mg/cm^2) and monotone regression (0.009 mg/cm^2) are consequently low. Even with the 95% confidence interval taken into account, the 50-percent point estimate remains below the 1.0 mg/cm^2 standard. Thus, it is estimated that the chance of obtaining a positive result at the standard was high, at least 50 percent for lead levels greater than one-tenth of the standard. This may explain the high overall rate of false positives shown in Table 5-9 (37%).

5.2.3.5.2 Lead Zone on Concrete

There were 226 observations of Lead Zone on concrete, of which 152 gave negative and 74 gave positive results. The running mean (Figure 5-23) is irregular toward the center of the lead range, a shape which an enhanced logistic regression model cannot fully capture. This accounts for the near step-function form of the model estimate ($b = 7.92$), suggesting a high baseline rate of false positives (.243) not indicated by the data: all 33 samples with ICP levels less than 0.0056 mg/cm^2 gave negative results. Monotone regression appears to have done a better job describing these data. The threshold probability estimated by the model at .272 is close to the estimate obtained from the monotone regression. A baseline rate of false negatives is not indicated: all 23 samples having ICP measurements greater than

1.746 mg/cm^2 gave positive results.

5.2.3.5.3 Lead Zone on Drywall

There were 123 observations of Lead Zone on Drywall, of which 96 gave negative and 27 gave positive results. In spite of therestricted range of lead levels, Figure 5-24 shows that the kit did not appear to exhibit a baseline rate of false positives, and that the probability of a positive result increased with the lead level. All 50 samples with ICP measurements less than 0.011 mg/cm^2 gave negative results.

The model estimates the threshold probability at .643. The 50-percent point estimate at 0.312 mg/cm^2 is in the upper range of the recorded ICP measurements. The estimated 50-percent point from the monotone regression (0.390 mg/cm^2) is similar.

5.2.3.5.4 Lead Zone on Metal

There were 216 observations of Lead Zone on metal, of which 137 gave negative and 79 gave positive results. Figure 5-25 reveals that, except for a dip in the running mean, the running mean and the monotone regression are close, as are the model and the monotone regression. Although the kit was recommended for use on painted metal, it produced the highest baseline rate of false positives on this substrate. Both the model and the monotone regression estimate this rate at about 17 percent, which is more than half of the overall false positive rate shown in Table 5-9 (24%), and may indicate interference from non-lead factors. The moderate baseline rate of false negatives (.138) obtained from the model is mitigated by the fact that all 7 samples with ICP measurements greater than 3.96 mg/cm^2 had positive results. There were no ICP measurements greater than 7.0 mg/cm^2 on metal, which makes it difficult to infer a baseline rate of false negatives at higher lead levels.

The model and monotone regression gave similar threshold probability estimates (.586 and .500 respectively), but given the steepness of the plots near the standard, the 95% confidence interval in Table 5-22 reflect a high degree of variability in these estimates. The estimated 50-percent points (0.819 mg/cm^2 model and 0.948 mg/cm^2 monotone regression) are also similar, and the 95% confidence interval covers the 1.0 mg/cm^2 standard. At the standard the estimated chance of obtaining a positive result

is about a half, with the chance dropping to about .17 rapidly as the lead level decreases from the 1.0 mg/cm^2 standard, and rising above .80 rapidly as the lead level increases from the standard.

5.2.3.5.5 Lead Zone on Plaster

There were 242 observations of Lead Zone on plaster, of which 166 gave negative and 76 gave positive readings. Figure 5-26 shows that the running mean, monotone regression, and model OC curve are in general agreement. The model indicates a substantial baseline rate of false negatives (.347), which is not inconsistent with the fact that the 3 samples with ICP measurements greater than 18.0 mg/cm^2 had positive results. These three samples contributed the endpoint effect that is evident in the monotone regression.

The model and monotone regression produced similar threshold probability estimates (.553 model and .444 monotone regression). The difference in 50-percent point estimates (0.711 mg/cm^2 model, 1.368 mg/cm^2 monotone regression) is not great when sampling variability, as demonstrated in the 95% confidence interval (Table 5-22), is taken into account. The estimated chance of obtaining a positive result at the standard is about .50. The similarity of the overall false positive and false negatives rates shown in Table 5-9 (26% and 36% respectively) reflects the closeness of the 50-percent point to the 1.0 mg/cm^2 standard.

5.2.3.5.6 Lead Zone on Wood

There were 387 observations of Lead Zone on wood, of which 182 gave negative and 205 gave positive results. In Figure 5-27 the running mean exhibits several visible dip-peak pairs, but these would not be unusual as random occurrences. The model seems to adequately describe the data. Neither the model nor the monotone regression indicate baseline rates of false negatives or positives.

The model and the monotone regression suggest high estimated threshold probabilities (.795 and .833). Likewise, the estimated 50-percent points are low (0.145 and 0.218 mg/cm^2). It appears that the chance of a positive result was high at the standard, with a better than a 50 percent chance for lead levels as low as 0.1 mg/cm^2. Thus the overall false positive rate shown in Table 5-9 (35%) is high.

5.2.3.5.7 Summary of Analysis for Lead Zone

The manufacturer's instructions only referred to the application of Lead Zone on wood or metal substrates, but there is no evidence that the test kit performed better on these two substrates than on others. A high baseline rate of false positives was observed on metal, concrete and plaster, but not on wood, brick and drywall. Although the rate of positives changed rapidly near the 1.0 mg/cm^2 standard on metal, concrete and plaster, the probability at the standard may have been no better than 50 percent for these substrates. The kit was most responsive to lead at the 1.0 mg/cm^2 standard on wood and brick, for which approximately an 80 percent chance of observing a positive result was obtained.

5.2.3.6 State Sodium Sulfide

Table 5-23 gives the results of fitting enhanced logistic models to the six substrates tested with the State Sodium Sulfide kit. Figures 5-28 through 5-33 graphically show the fit of these models to the data. Sections 5.2.3.6.1 through 5.2.3.6.6 discuss the estimated OC curves by substrate. Section 5.2.3.6.7 contains an overall summary for the test kit.

5.2.3.6.1 State Sodium Sulfide on Brick

There were 93 observations of the State Sodium Sulfide kit on brick, of which 24 gave negative and 69 gave positive results.

Figure 5-28 shows that the model OC curve fits the data moderately well. A high baseline rates of false positives is evident in all three plots, estimated by the model at .234. The test kit frequently produced positive results at very low lead levels.

Both the model and monotone regression estimate the threshold probability at nearly 100 percent. All 54 samples with ICP measurements greater than 0.06 mg/cm^2 gave positive results, accounting for more than half of the total sample. Conversely, the estimated 50-percent points are low (0.006 mg/cm^2 model and 0.003 mg/cm^2 monotone regression). The inference that the kit was very likely to produce a positive result at lead levels well below the standard does not change by taking into account the 95% confidence intervals shown in Table 5-23. Both non-lead

interference and sensitivity to low levels of lead may have contributed to the high false positive rate of 67% shown in Table 5-10.

5.2.3.6.2 State Sodium Sulfide on Concrete

There were 224 observations of the State Sodium Sulfide kit on concrete, of which 56 gave negative and 168 gave positive results. Figure 5-29 indicates that the kit frequently gave

Table 5-23. STATE SODIUM SULFIDE Enhanced Logistic Regressions by Substrate.

SUBSTRATE	NEGATIVE	POSITIVE	MODEL PARAMETERS				Pb(.50) mg/cm²	PROB. AT Pb = 1
			c	d	a	b		
BRICK	24	69	.234 (.129)	.766 (.129)	4.38 (1.55)	0.976 (.228)	0.006 [.001, .053]	.991 [.844, .999]
CONCRETE	56	168	.411 (.662)	.589 (.662)	2.38 (1.65)	0.901 (2.35)	0.011 [.001, .040]	.950 [.944, .999]
DRYWALL	77	47	0	1	0.765 (.362)	0.300 (.073)	0.078 [.017, .360]	.682 [.510, .816]
METAL	63	154	.142 (.102)	.840 (.161)	3.07 (1.93)	1.29 (1.01)	0.075 [.017, .143]	.944 [.877, .975]
PLASTER	42	200	.259 (.088)	.692 (.100)	5.87 (3.39)	1.74 (.986)	0.024 [.007, .048]	.949 [.811, .988]
WOOD	108	280	.275 (.305)	.725 (.305)	2.60 (1.85)	1.08 (1.53)	0.043 [.005, .100]	.950 [.412, .998]

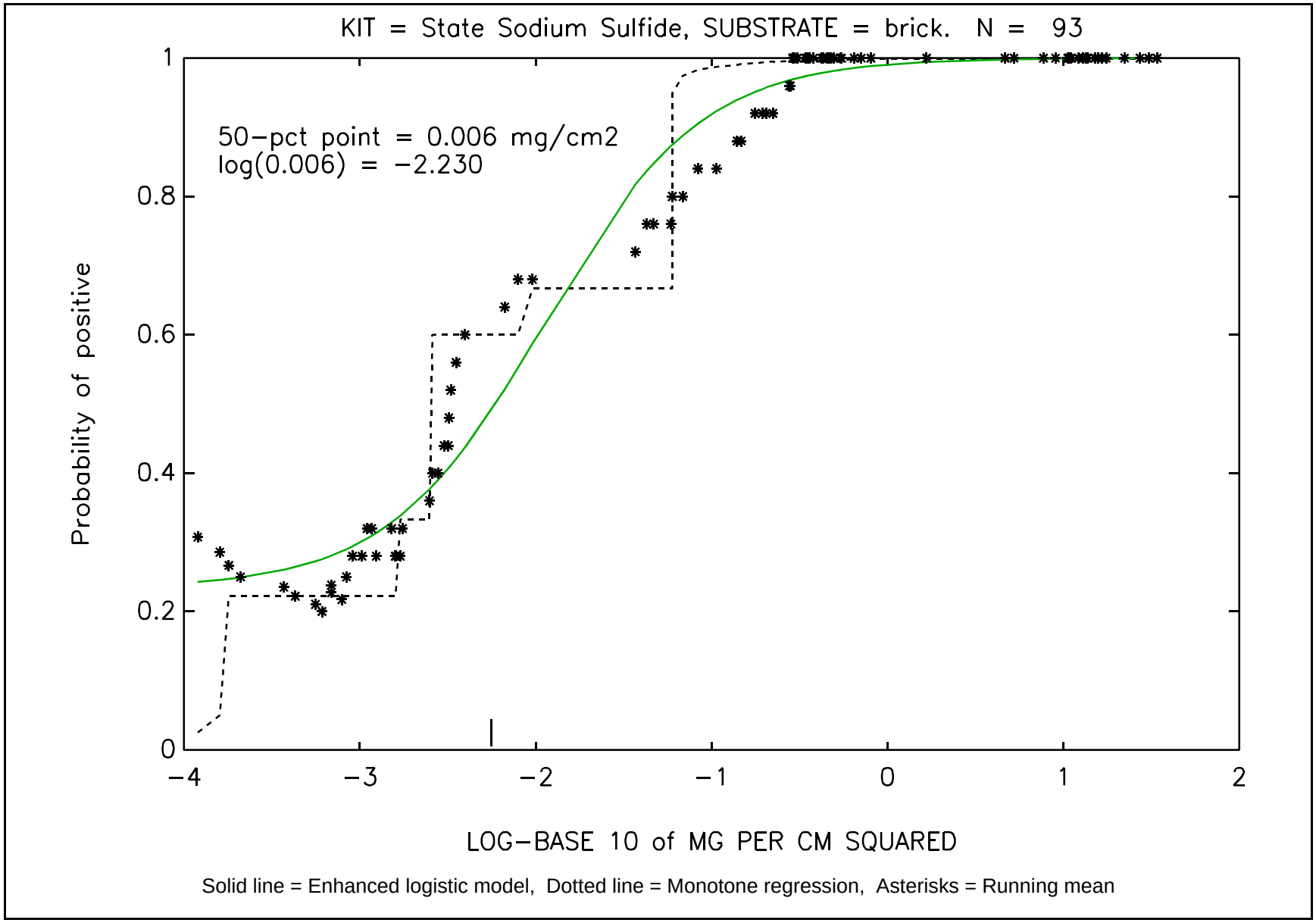

Figure 5-28. Operating characteristic curve for State Sodium Sulfide on brick.

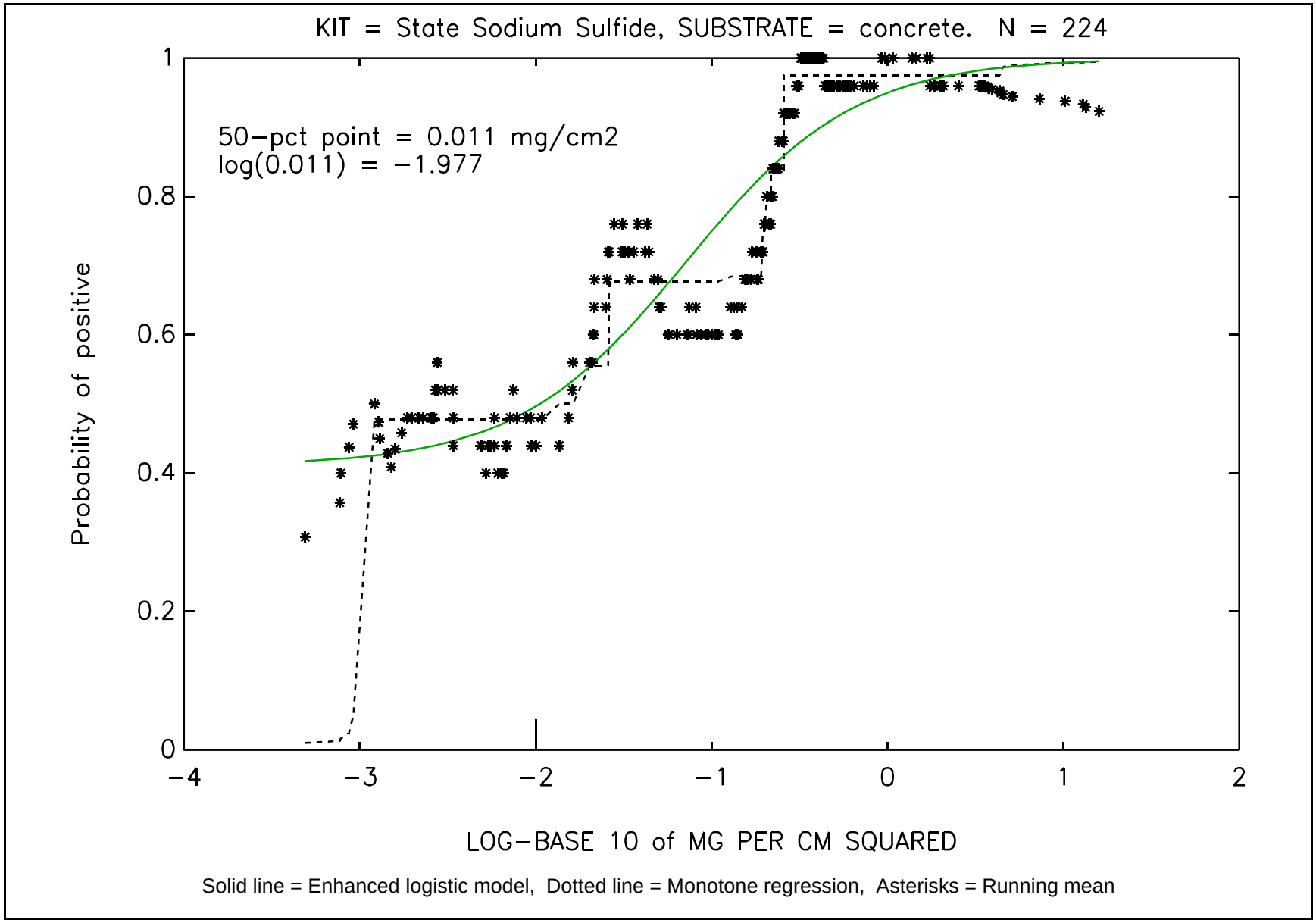

Figure 5-29. Operating characteristic curve for State Sodium Sulfide on concrete.

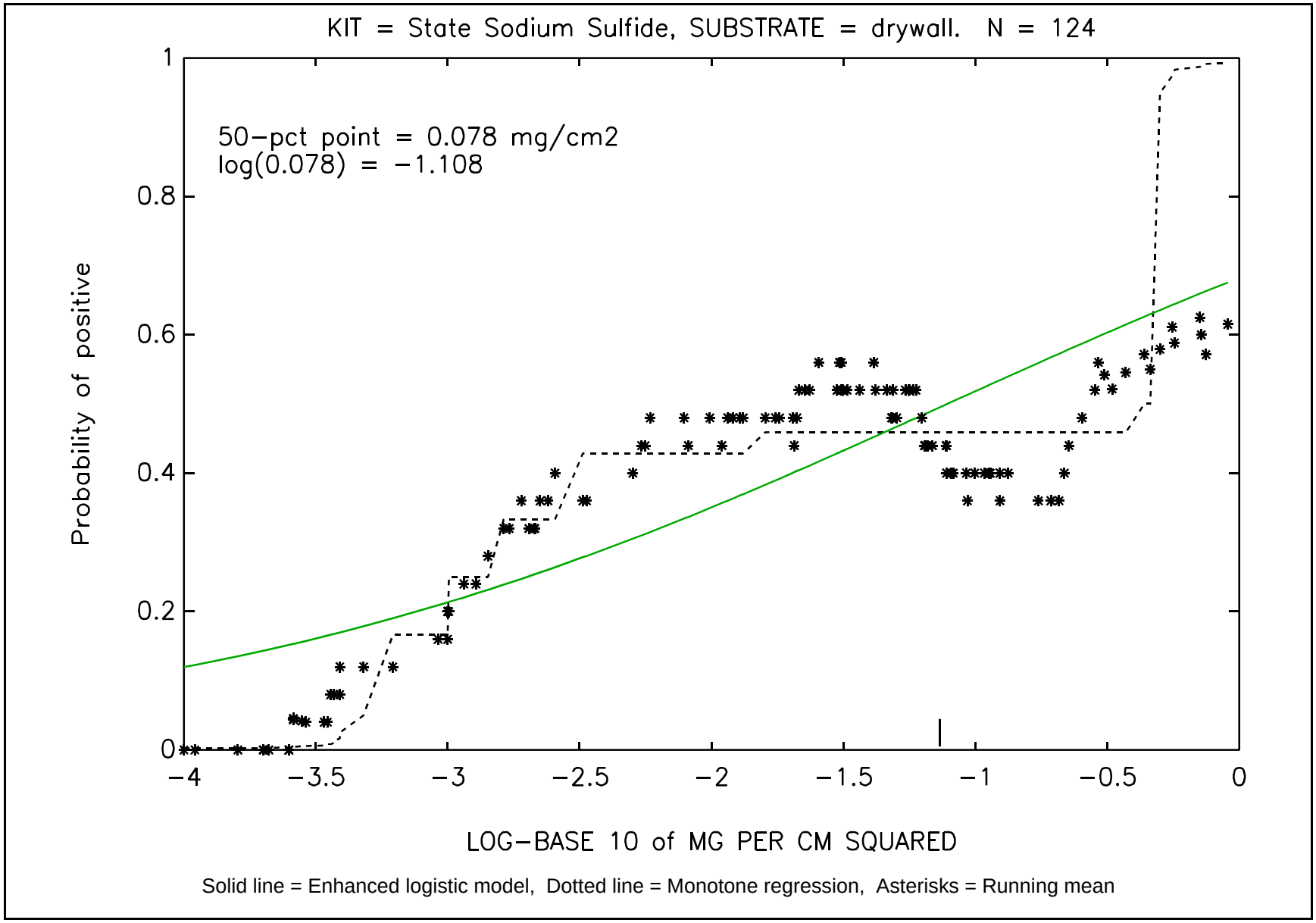

Figure 5-30. Operating characteristic curve for State Sodium Sulfide on drywall.

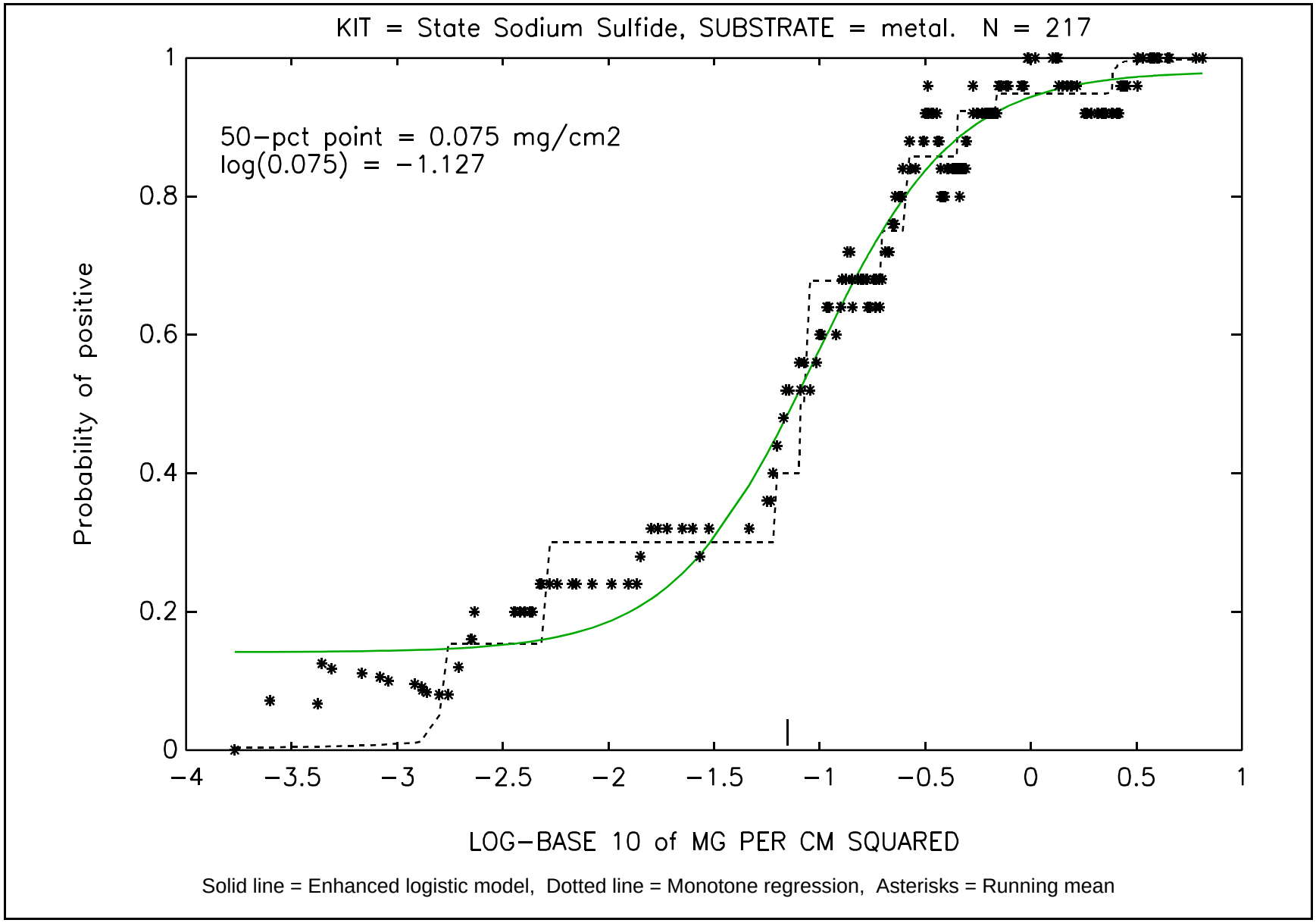

Figure 5-31. Operating characteristic curve for State Sodium Sulfide on metal.

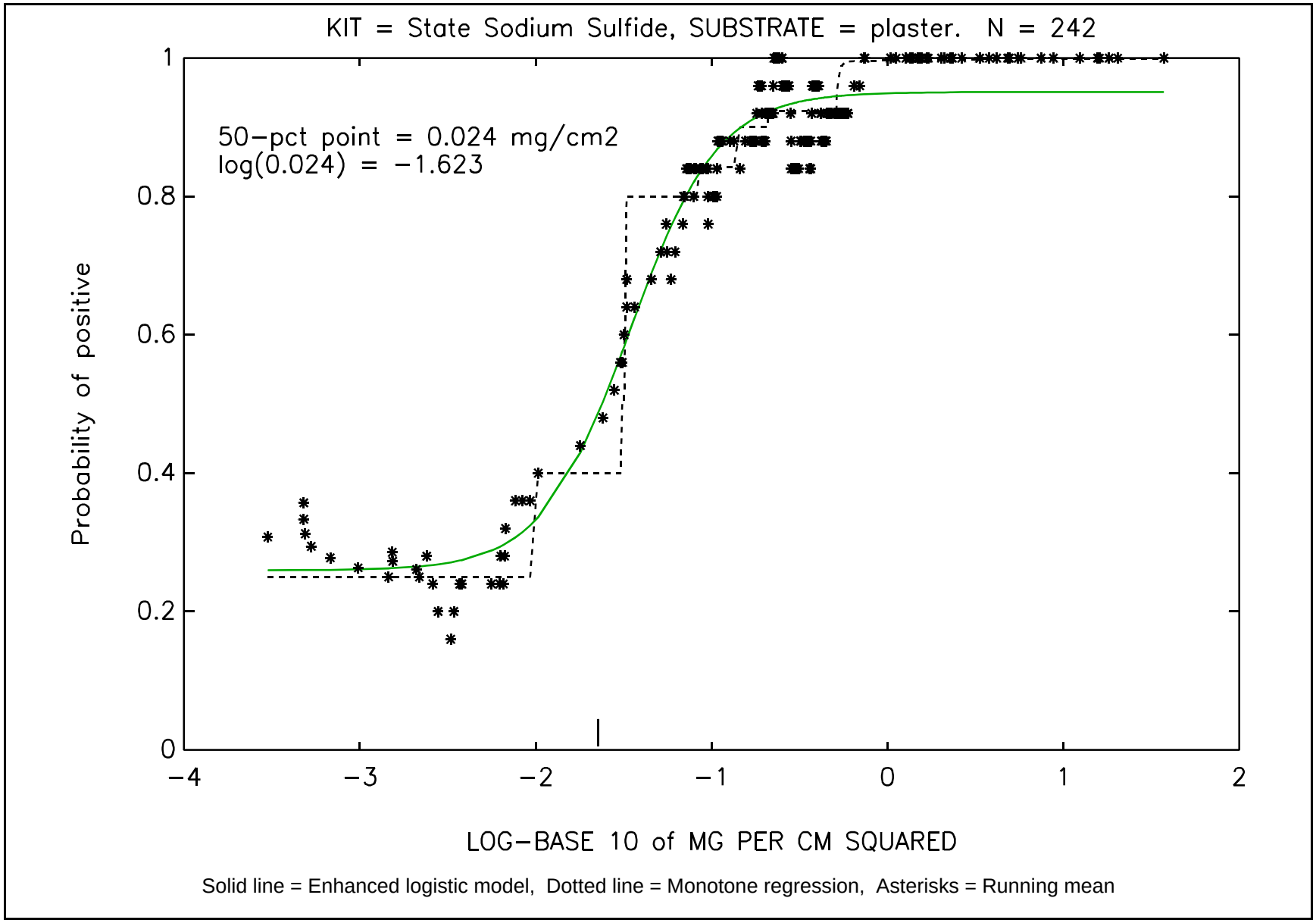

Figure 5-32. Operating characteristic curve for State Sodium Sulfide on plaster.

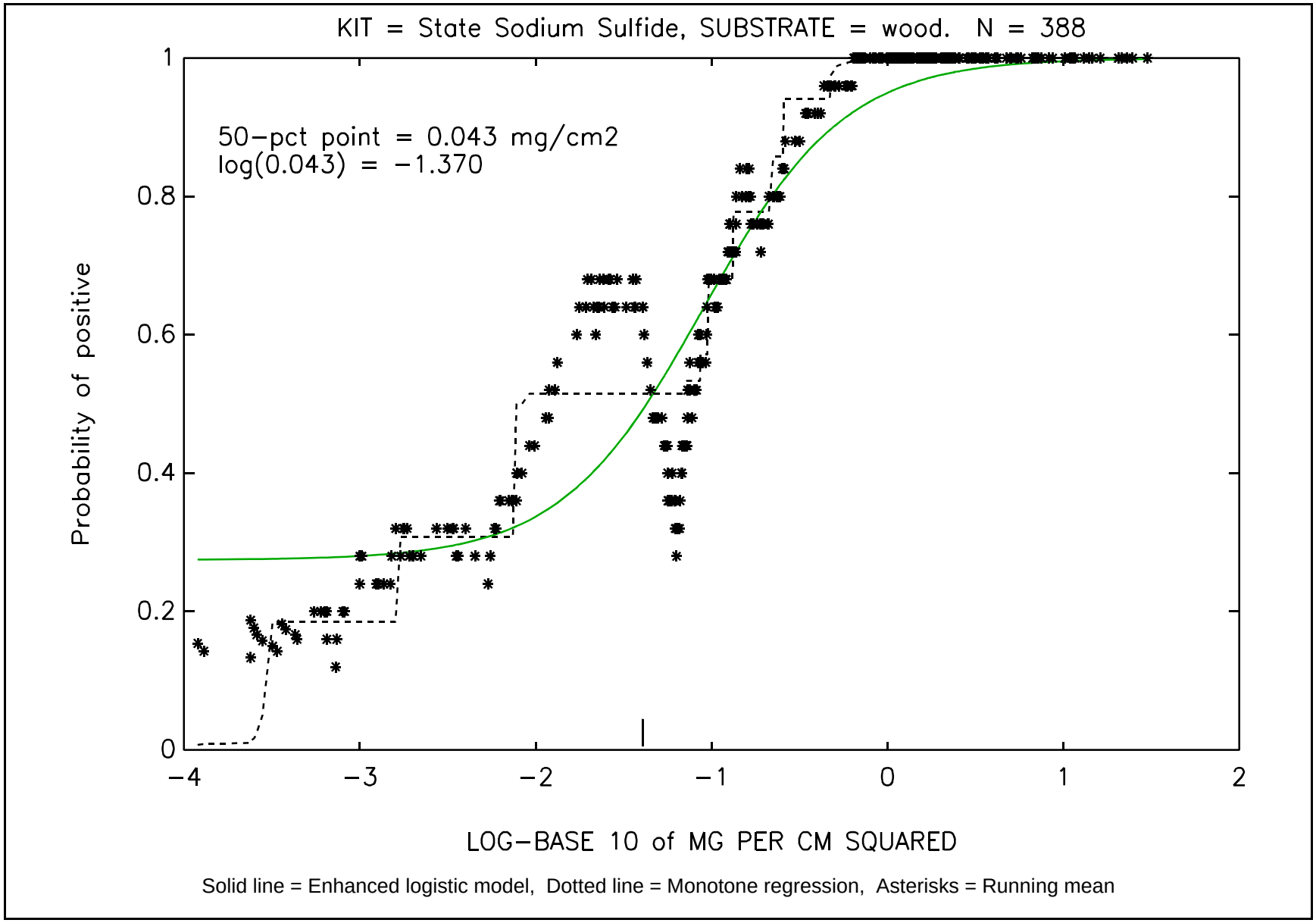

Figure 5-33. Operating characteristic curve for State Sodium Sulfide on wood.

positive results at all lead levels, with a high baseline rate of false positives estimated at .411 with the model.

The model and monotone regression produce high estimates of the threshold probability (.950 and .975), and low estimates of the 50-percent point (0.011 and 0.015 mg/cm^2). The running mean, however, indicates that the 50-percent point could have been as low as 10^{-3} = 0.001 mg/cm^2. The kit had a substantial probability of giving a positive result at any lead level, and at the 1.0 mg/cm^2 standard the result was almost certain to be positive. Thus the high overall rate of false positives shown in Table 5-10 (72%) may reflect both non-lead factors and sensitivity to low levels of lead.

5.2.3.6.3 State Sodium Sulfide on Drywall

There were 124 observations of the State Sodium Sulfide kit on drywall, of which 77 gave negative and 47 gave positive results. Figure 5-30 shows the rate of positives leveling off for lead levels greater than 10^{-2} = 0.01 mg/cm^2, although near the 1.0 mg/cm^2 standard the monotone regression suggests a higher rate.
This is a smoothing effect of the running mean: a smaller smoothing window (not shown) captured the increase in the rate.

Unlike other substrates, there was no indication of a baseline rate of false positives on drywall: the 21 samples with ICP measurements less than 0.0005 mg/cm^2 all gave negative results. The overall false positive rate of 38% shown in Table 5-10 may have been due primarily to sensitivity of the kit to low lead levels.

5.2.3.6.4 State Sodium Sulfide on Metal

There were 217 observations of the State Sodium Sulfide kit on metal, of which 63 gave negative and 154 gave positive results. The removal of the paint from the substrate prior to testing may explain why the monotone regression and running mean (Figure 5-31) do not indicate a high baseline rate of false positives, as was observed with other substrates. The 13 samples with ICP measurements less than 0.0016 mg/cm^2 all gave negative results. The model estimate of .142 for this baseline rate does not appear to be supported by the data. The running mean, monotone regression, and model OC curve are otherwise similar.

The model and monotone regression produce nearly identical, high estimates of the threshold probability (.944 and .949). The estimated 50-percent points are also close (0.075 and 0.083 mg/cm^2), well below the 1.0 mg/cm^2 standard. The latter conclusion is not changed by taking into account the 95% confidence interval derived from the model. While interference from non-lead factors did not appear to be a problem with the State Sodium Sulfide kit on metal, the overall rate of false positives indicated in Table 5-10 (64%) for metal was not much lower than for substrates where interference was possible.

5.2.3.6.5 State Sodium Sulfide on Plaster

There were 242 observations of the State Sodium Sulfide kit on plaster, of which 42 were negative and 200 were positive. Figure 5-32 reveals a high baseline rate of false positives, estimated at about 26 percent by the model.

Both the model and the monotone regression give similar, high estimates of the threshold probability (.949 model and .997 monotone regression), and both give similar estimates of the 50-percent point (0.024 and 0.031 mg/cm^2). Accounting for uncertainty in these estimates with the 95% confidence intervals reported in Table 5-23, does not affect the conclusion that the rate of positives at the 1.0 mg/cm^2 standard was high, and was above 50 percent for lead levels well below the standard. All 47 samples with ICP measurements greater than 0.5149 mg/cm^2 had positive results. The high rate of false positives shown in Table 5-10 (80%) may reflect both interference from non-lead factors and sensitivity of the kit to low levels of lead.

5.2.3.6.6 State Sodium Sulfide on Wood

There were 388 observations of the State Sodium Sulfide kit on wood, of which 108 gave negative and 280 gave positive results. Figure 5-33 reveals a pronounced peak-dip in the running mean between 10^{-2} = 0.01 mg/cm^2 and 10^{-1} = 0.1 mg/cm^2 that adversely affects the fit of the model to the data in the lower lead range. The monotone regression was less affected by this phenomenon. The high baseline rate of false positives estimated from the model (.275) therefore may not precisely describe how the test kit performed. Nonetheless, positive results were obtained on samples with ICP measurements less than 0.001 mg/cm^2. Non-lead factors and/or sensitivity to low levels of lead may

account for the high overall false positive rate (59%) shown in Table 5-10.

Positive results were overwhelmingly prevalent as the lead level approached the 1.0 mg/cm^2 standard. All 158 sample values with ICP measurements greater than 0.4725 mg/cm^2 had positive results with the State Sodium Sulfide test kit.

At higher lead levels the running mean, monotone regression, and model agree more closely. The model and monotone regression give high estimates of the threshold probability (.950 and .999). The running mean indicates that the 50-percent point may have been lower than the model estimate (0.043 mg/cm^2), possibly as low as 0.01 mg/cm^2.

5.2.3.6.7 Summary of Analysis for State Sodium Sulfide

The instructions for State Sodium Sulfide warned about the possibility of obtaining false positive results on painted metal, but this problem was not as evident on metal as it was on brick, concrete, plaster, and wood, which all had high baseline rates of false positives. Removal of the paint from metal substrates prior to testing could be the reason for this. On all substrates except drywall, the probability of a positive result at a lead level of 1.0 mg/cm^2 may have exceeded 90 percent, and positive results were frequent at even much lower levels. The kit did not exhibit a tendency to give false negatives at high levels of lead on any substrate.

5.2.4 Inference in Percent by Weight Units

In section 4.2, it is demonstrated that lead concentration expressed in area units (milligrams lead per centimeter squared) and in percent lead by weight of specimen were closely related in the study, although they are not equivalent. Both units of measure are in the form of ratios, with the estimated mass of lead appearing in both numerators. The denominator of the area units ratio is the *area* of the paint sample analyzed, while that of the percent by weight units ratio is the *mass* of the paint sample analyzed.

As discussed in Chapter 4, measurement of the mass of a paint sample is affected by the inclusion of substrate, which is a source of error that can vary in magnitude across substrate

types, or even within a particular substrate type under varying conditions. Brick, concrete, and (certain) plaster substrates were particularly prone to this problem. The effect of substrate inclusion is to impart a downward bias to the lead level measured in percent by weight units. In contrast, measurement in area units is much less affected by the problem of substrate inclusion.

Table 5-24 gives the results of fitting enhanced logistic regression models to the test kit data using percent by weight units. The confidence intervals for the 50-percent points were obtained by bootstrapping. The confidence intervals for the probability of a positive result at a lead level of 0.5% per weight were obtained from the fitted models, except for Lead Alert: Coring on all substrates; Lead Detective on concrete; and State Sodium Sulfide on concrete, for which confidence intervals were obtained by bootstrapping. The derivation of confidence intervals is explained further in section 5.2.6.4.

Table 4-11 presents regression results demonstrating that the logarithms of lead levels measured in the two units had correlations greater than 0.96 on all substrates except metal, where the correlation was 0.93. As a result, the OC curves derived in section 5.2.3 for area units can be used to give a description of how the test kits performed for lead levels measured in percent by weight units. This is done by transforming area units to percent by weight units using the regression coefficients presented in Table 4-11. Let Y represent the logarithm of lead in percent by weight units; X the logarithm of lead in area units; and let $Y = u + v \cdot X$ be the linear regression function of Y on X, where u is the intercept, and v is the slope. If the OC curve for the enhanced logistic model in area units has parameters (a,b,c,d) in the notation used above, then the OC curve in percent by weight units obtained by the transformation method has parameters (a',b',c',d') computed as follows:

$$a' = a - b \cdot u/v, \quad b' = b/v, \quad c' = c, \quad d' = d.$$

The two methods are illustrated with LeadCheck on wood substrates. Figure 5-34 shows the OC curve obtained by directly fitting the model in percent by weight units, and the curve obtained by the transformation method. Due to the high correlation between the two units, it is clear that little would be lost using either estimate as a substitute for the other.

A less favorable scenario concerns metal substrates, where the correlation between units at .93 was weakest. Direct and transformation method OC curves are plotted for the State Sodium Sulfide kit in Figure 5-35. The source of the discrepancy may be the grouping represented by metal substrates: painted metal surfaces in Denver were frequently encountered outdoors and were weathered, while those in Philadelphia and Louisville were usually found indoors with thick layers of paint. Regressions by city on metal revealed similar slopes (0.84 Denver, 0.82 Philadelphia, and 0.89 Louisville) but very different intercepts

Table 5-24. Results for lead levels in percent by weight units

TEST KIT	SUBSTRATE	Pb(.50)	95% CI for Pb(.50)	PROB 0.5%	95% CI for PROB
LeadCheck	Brick	0.021	[.009, .069]	0.954	[.718, .994]
	Concrete	0.155	[.055, .358]	0.682	[.583, .767]
	Drywall	0.563	[.146, .865]	0.484	[.265, .709]
	Metal	0.324	[.202, .427]	0.618	[.425, .781]
	Plaster	0.141	[.027, .322]	0.681	[.604, .750]
	Wood	0.074	[.030, .133]	0.829	[.767, .877]
Lead Alert: coring	Brick	0.132	[.024, .273]	0.734	[.558, .858]
	Concrete	1.14	[.395, 2.18]	0.227	[.116, .396]
	Metal	1.09	[.660, 3.02]	0.261	[.177, .365]
	Wood	0.968	[.558, 1.60]	0.280	[.201, .376]
Lead Alert: sanding	Concrete	0.877	[.444, 7.35]	0.126	[.122, .529]
	Metal	---	[1.27, 10.5]	0.052	[.004, .161]
	Wood	1.68	[1.55, 8.97]	0.031	[.001, .081]
Lead Detective	Brick	0.014	[.005, .048]	0.801	[.653, .896]
	Concrete	0.326	[.225, 7.38]	0.547	[.393, .690]
	Drywall	---	---	0.309	[.183, .471]
	Metal	0.627	[.380, 1.35]	0.433	[.318, .555]
	Plaster	0.584	[.248, 1.32]	0.464	[.269, .672]
	Wood	0.355	[.228, .750]	0.582	[.497, .663]
Lead Zone	Brick	0.069	[.016, .209]	0.812	[.653, .908]
	Concrete	0.487	[.255, 1.22]	0.507	[.392, .621]
	Drywall	0.354	[.056, 1.41]	0.545	[.353, .725]
	Metal	1.03	[.704, 1.72]	0.194	[.069, .440]
	Plaster	0.437	[.270, 3.04]	0.533	[.396, .665]
	Wood	0.264	[.086, .810]	0.622	[.556, .684]
State Sodium Sulfide	Brick	0.010	[.003, .024]	0.998	[.848, .999]
	Concrete	0.010	[.001, .051]	0.931	[.917, .999]
	Drywall	0.134	[.003, .918]	0.586	[.421, .734]
	Metal	0.082	[.049, .150]	0.829	[.705, .908]

	Plaster	0.021	[.005, .065]	0.909	[.852, .946]
	Wood	0.089	[.022, .182]	0.871	[.782, .926]

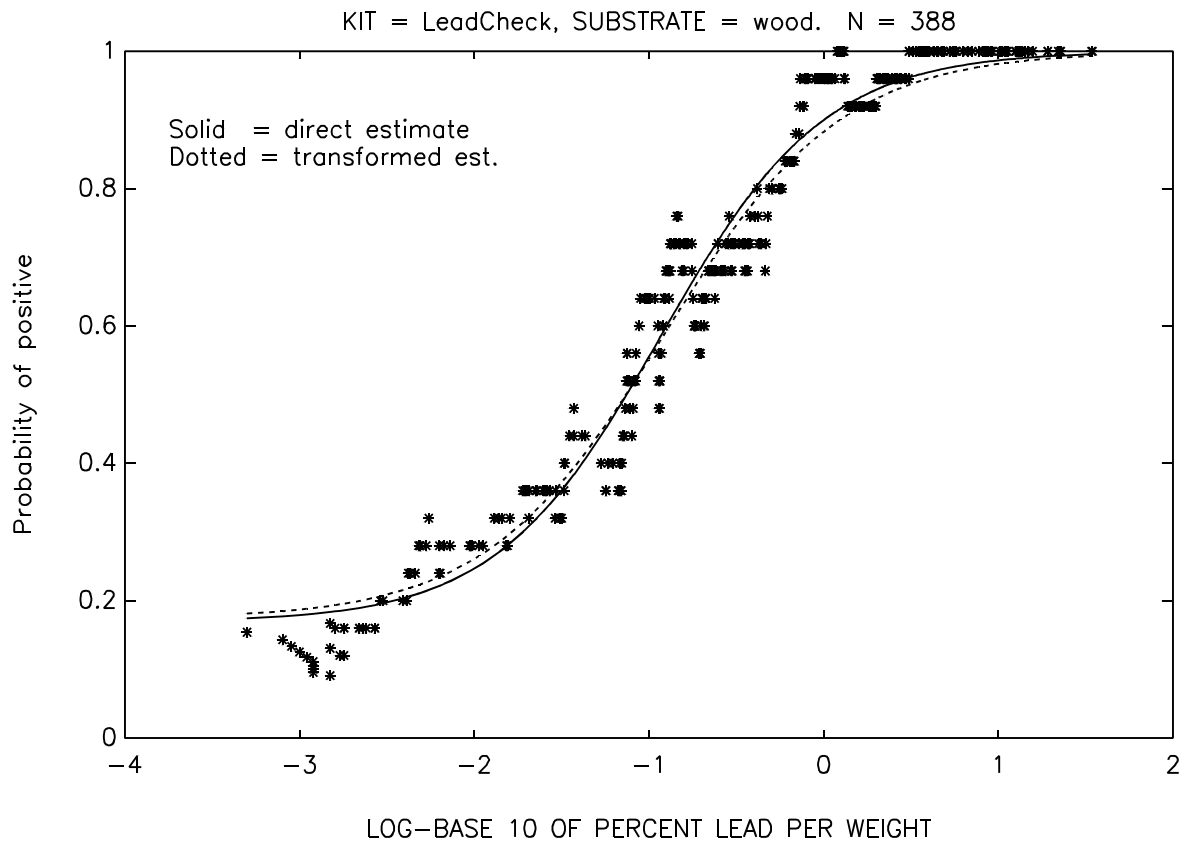

Figure 5-34. Operating characteristic curve for LeadCheck on wood.

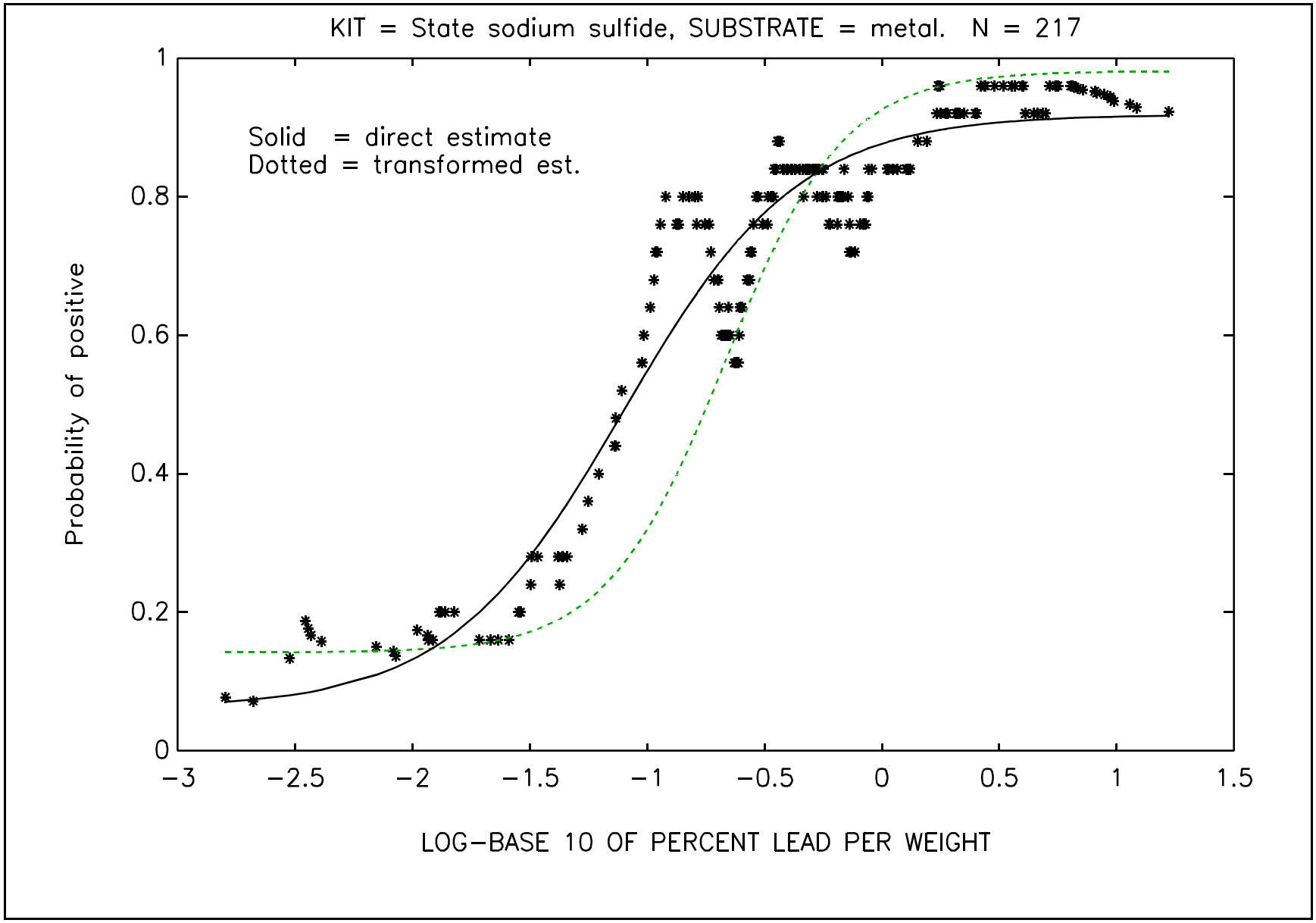

Figure 5-35. Operating characteristic curve for State Sodium Sulfide on metal.

(1.42 Denver, 0.49 Philadelphia, and 0.52 Louisville), confirming that the Denver samples were markedly different from samples taken in Philadelphia and Louisville.

Because of the weaker association between area and percent by weight units on metal substrates, OC curves were estimated for metal by fitting models directly. Plots of these curves are shown in Figures 5-36 through 5-41. Table 5-25 gives the corresponding model parameters. Results presented for percent by weight units are applicable to other situations only insofar that factors such as substrate inclusion and thickness of paint samples are similar to the present study.

5.2.5 Lead Test Kit Performance: Conclusions

The enhanced logistic model described important features of test kit performance, as reflected in the study data, and it did so in a clearly interpretable manner. The analyses demonstrated that no test kit was ideal in all respects, on any substrate. Test kits that were likely to give positive results at high levels of lead were also likely to give positive results at lower levels of lead as well. Other kits frequently gave negative results regardless of the lead level. The failure of any test kit to simultaneously give low false positive and low false negative classification rates, as demonstrated in Tables 5-5 through 5-16, is confirmed with the analyses described in section 5.2.3.

The observed tendency of some OC curves to level off at probabilities greater than zero as the lead level diminished suggests potential interference with the performance of the kits. Possibilities include chemical interferences from the paint or substrate, interferences between the color of the paint and color changes with the kits, and tester effects.

5.2.6 Estimation of OC Curves: Statistical Methodology

In this section, technical details concerning model development are presented, and the impact of spatial variation and laboratory error in ICP measurements on OC curve estimation is considered.

5.2.6.1 Model Selection

A mathematical model for test kit performance should provide a simple, yet accurate description of its important characteristics. To achieve simplicity, certain assumptions

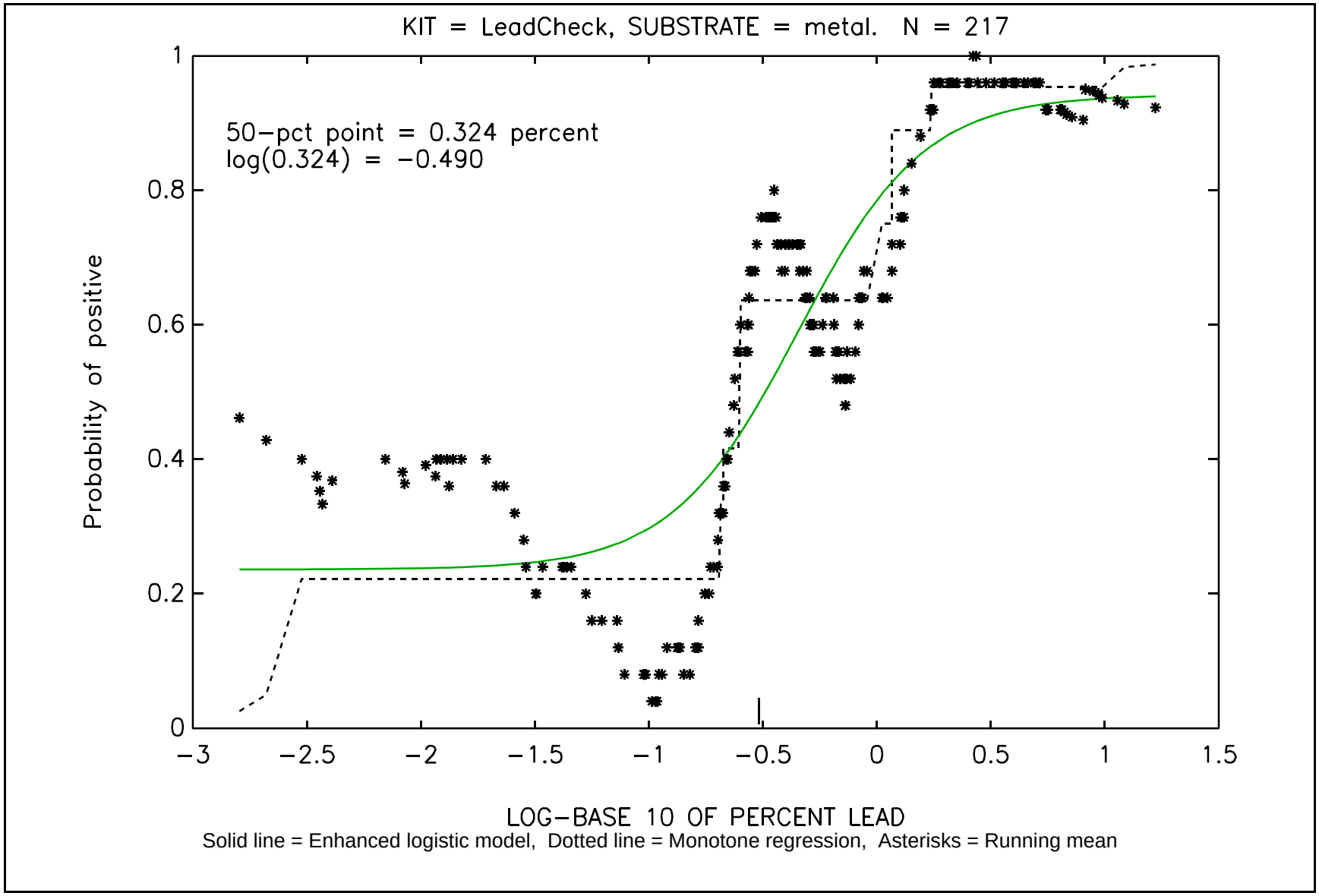

Figure 5-36. Operating characteristic curve for LeadCheck on metal, in weight concentration units.

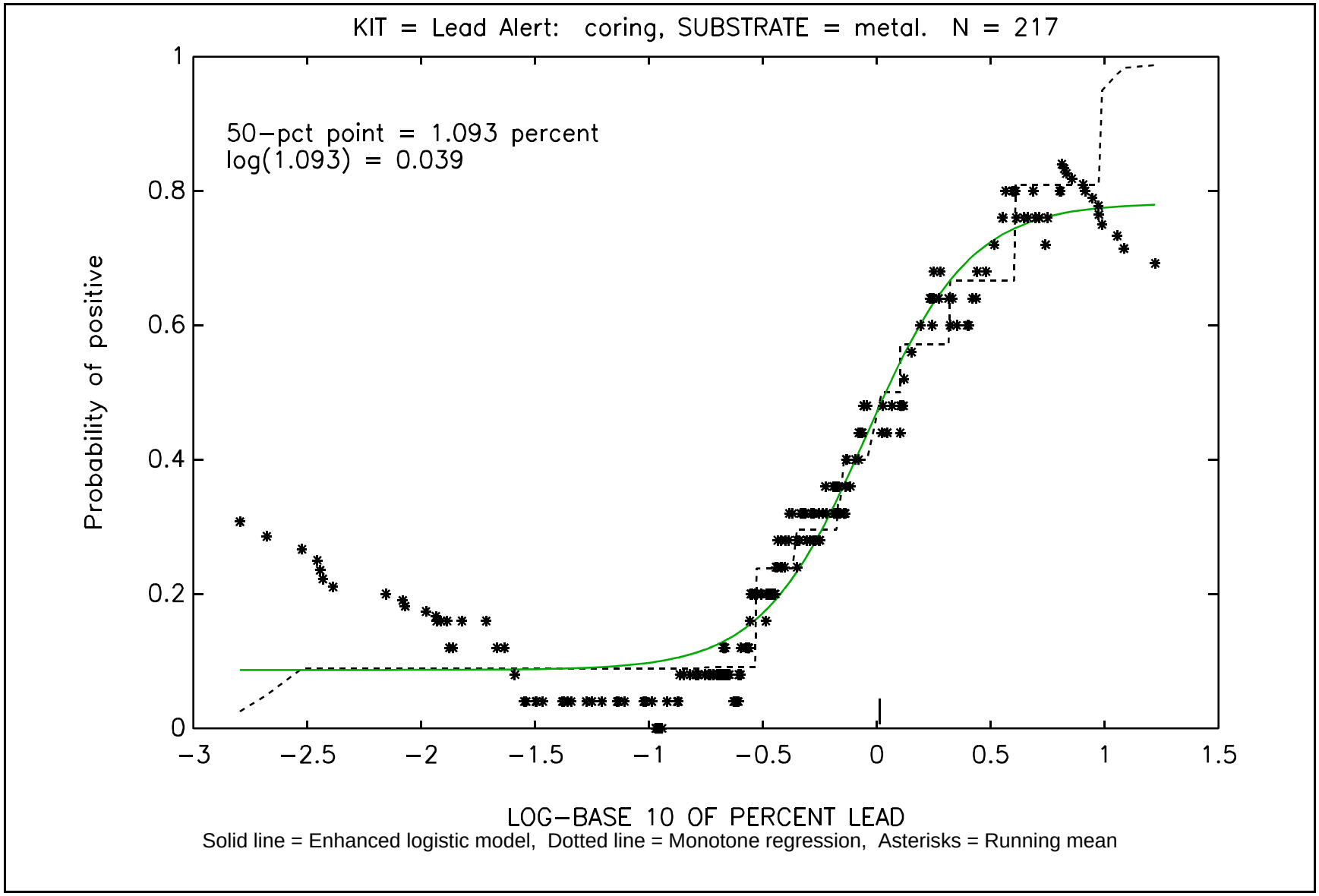

Figure 5-37. Operating characteristic curve for Lead Alert: coring on metal, in weight concentration units.

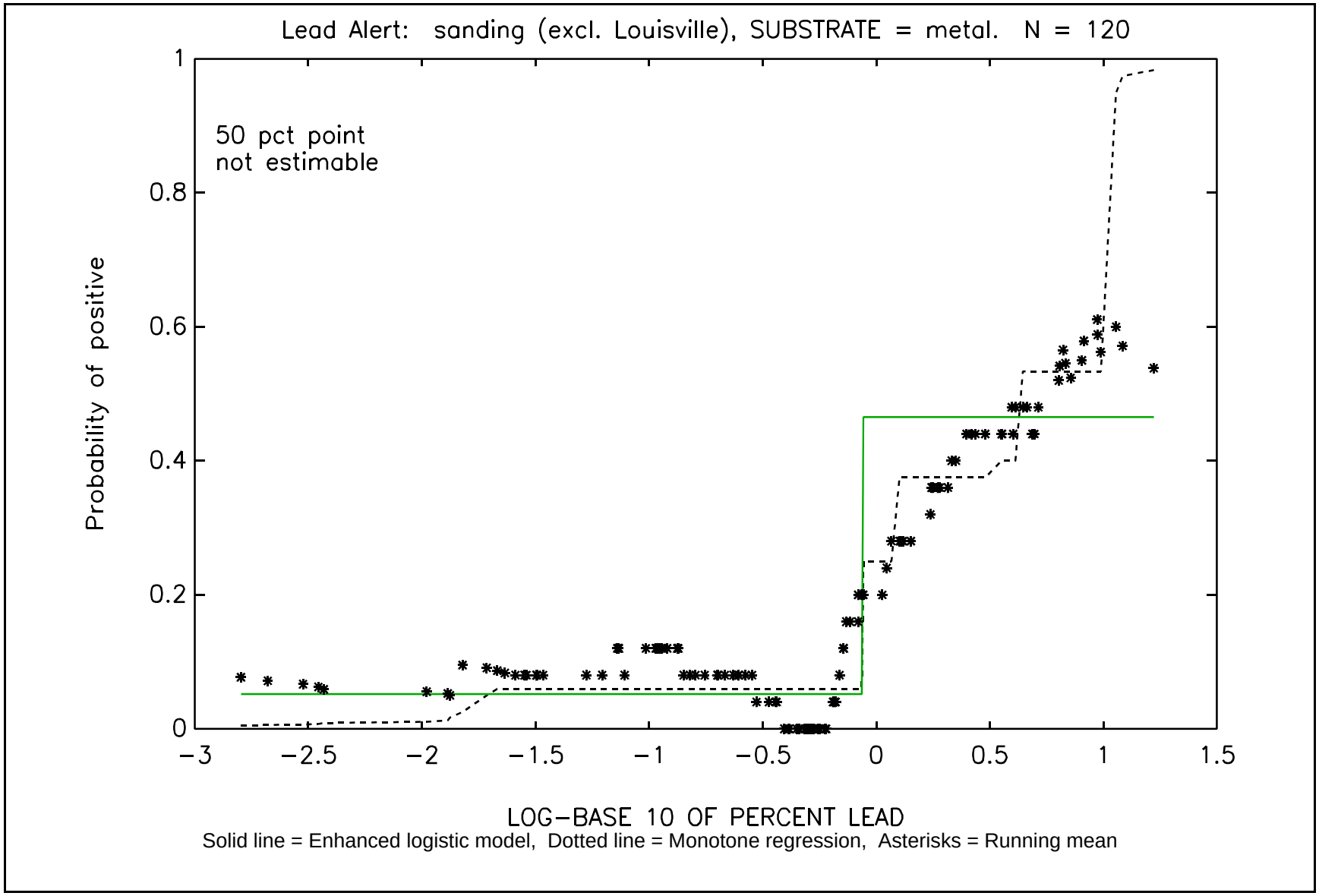

Figure 5-38. Operating characteristic curve for Lead Alert: sanding on metal, in weight concentration units.

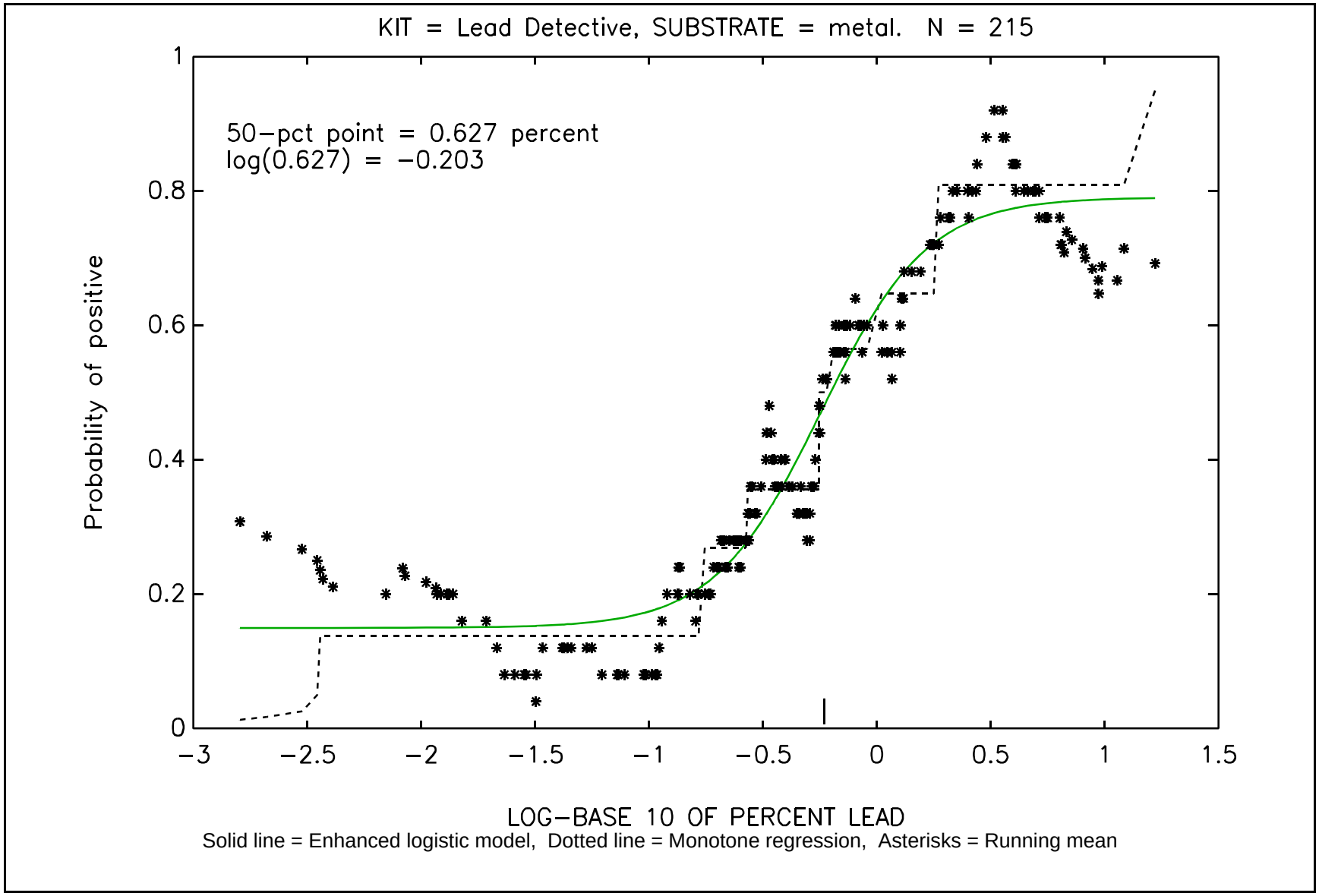

Figure 5-39. Operating characteristic curve for Lead Detective on metal, in weight concentration units.

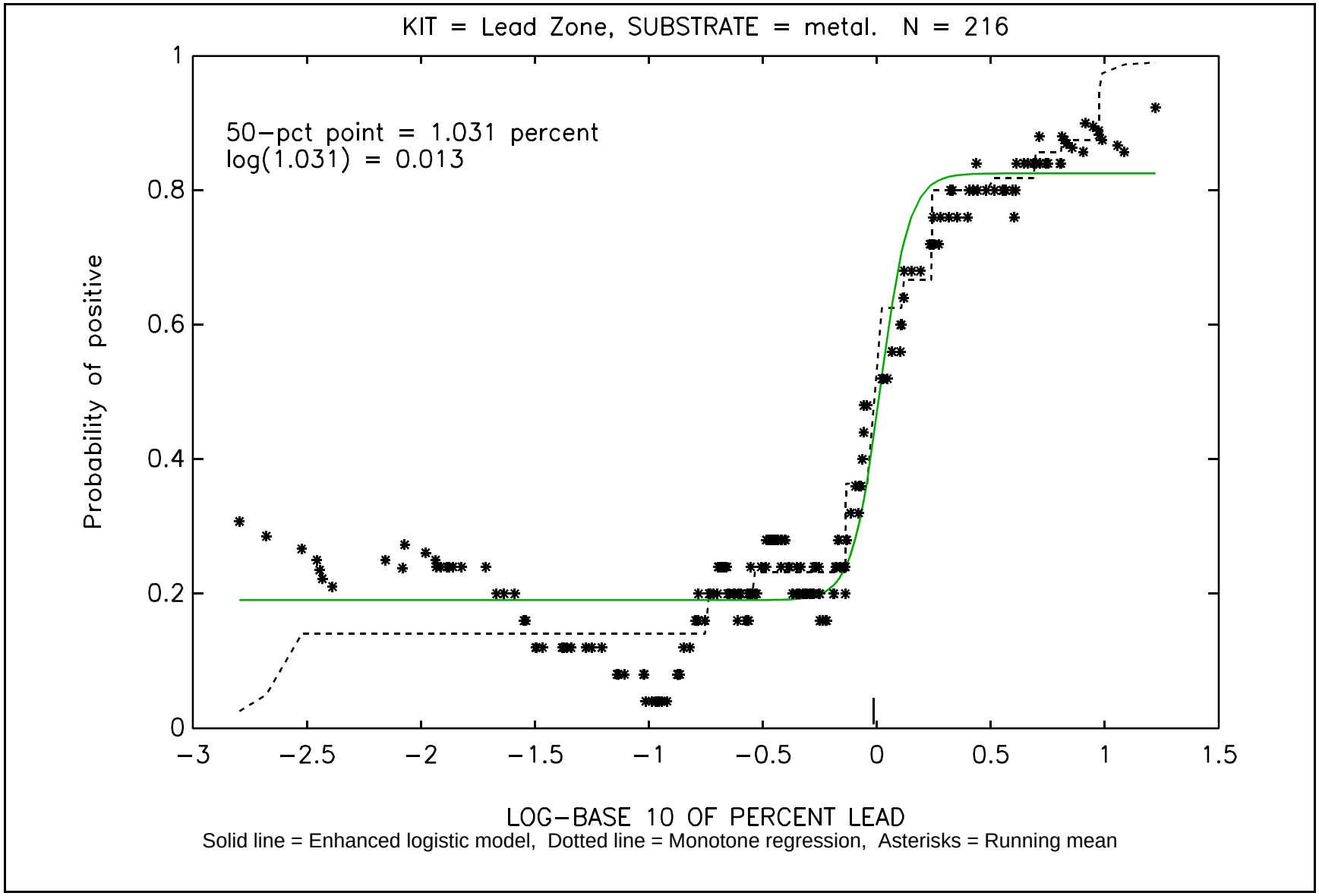

Figure 5-40. Operating characteristic curve for Lead Zone on metal, in weight concentration units.

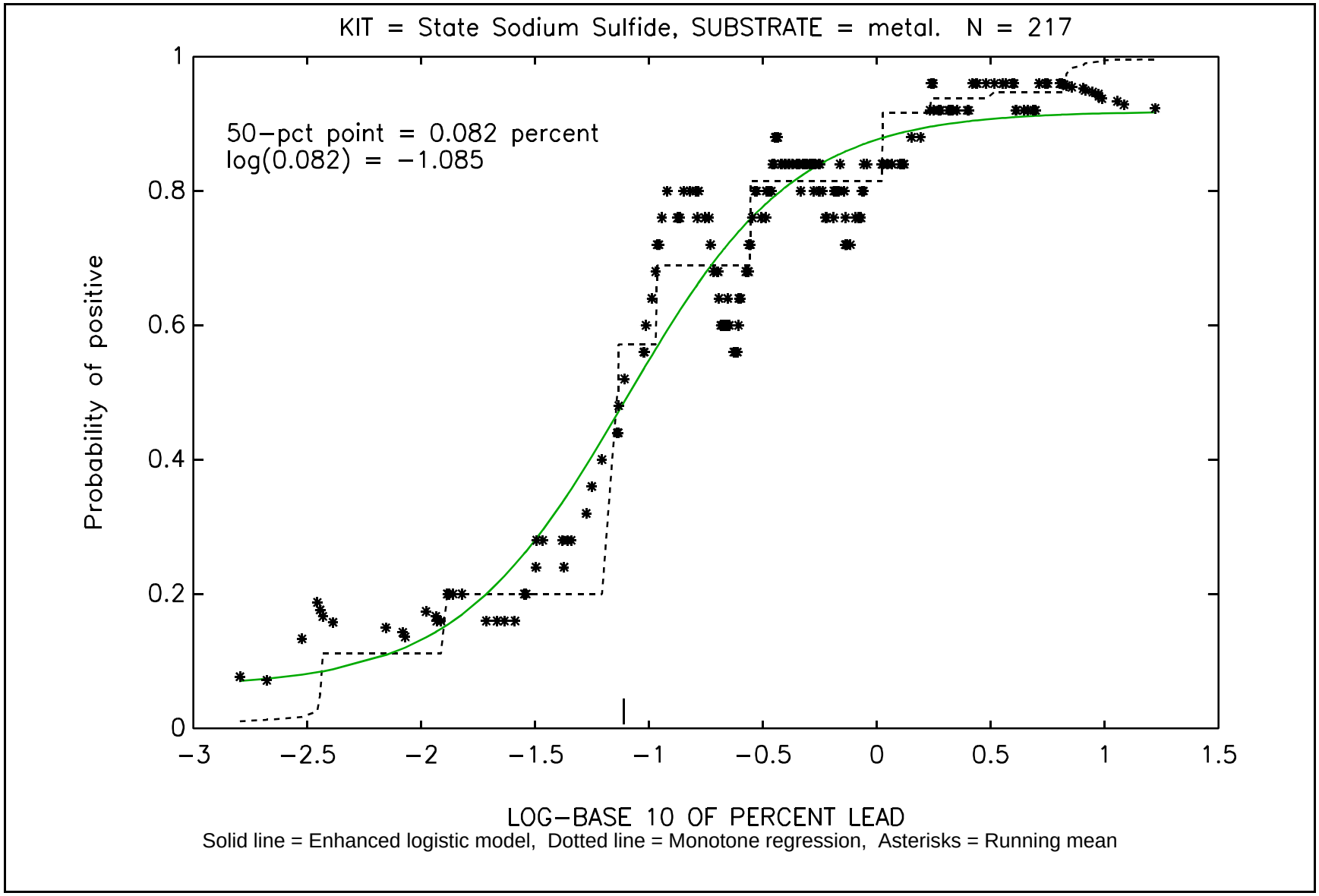

Figure 5-41. Operating characteristic curve for State Sodium Sulfide on metal, in weight concentration units.

Table 5-25. Enhanced Logistic Regressions for Metal in Percent by Weight Units.

TEST KIT	NEG	POS	MODEL PARAMETERS			
			c	d	a	b
LeadCheck	92	125	.236 (.071)	.706 (.089)	1.25 (1.27)	1.57 (.520)
Lead Alert: Coring	147	70	.087 (.036)	.695 (.101)	0.212 (.682)	1.90 (.567)
Lead Alert: Sanding	95	24	.052	.413	***-13.9***	***100***
Lead Detective	123	92	.150 (.057)	.641 (.087)	1.06 (.602)	1.86 (.547)
Lead Zone	137	79	.191 (.154)	.635 (.657)	-0.261 (5.49)	7.00 (96.9)
State Sodium Sulfide	63	154	.062 (.118)	.857 (.198)	2.96 (2.04)	1.17 (.819)

about the data are imposed when a model is selected. The most basic assumption is that an OC curve should be a nondecreasing function of the lead level. This section explains the reasoning behind the selection of the enhanced logistic regression model for describing test kit performance.

5.2.6.1.1 Logistic Regression

Logistic regression uses the following mathematical model for the OC curve:

$$OC(t) = \frac{\exp(a + bt)}{1 + \exp(a + bt)}$$

where a and b are coefficients that are typically estimated from the data. Since this functional form involves only two parameters, the richness of the class of possible curve estimates is somewhat restricted. When appropriate, it is a good model form to use, for several reasons:

- The estimated OC(t) is a strictly increasing function of t, assuming b is positive;
- All aspects of the OC curve, including all probabilities

calculated from it and the 50-percent point, depend solely on the two parameters;

- Maximum likelihood or least squares estimation of the parameters allows the derivation of standard error estimates and confidence intervals for many quantities of interest in relatively straightforward fashion.

It was found that letting t denote the logarithm of the lead level, instead of the lead level itself, improved the analysis for several reasons that are elaborated in section 5.2.6.1.3. All references to logarithms are to natural logarithms (base $e \approx 2.718$) unless otherwise indicated.

5.2.6.1.2 An Enhanced Logistic Regression Model

The class of OC curves--nondecreasing functions of t in the range 0 to 1--is much richer than the class of logistic curves obtained by varying parameters a and b. As a result, there are some phenomena that logistic regression cannot describe very well. For example, if a kit produces a certain percentage of positive or negative readings while remaining responsive to the concentration of lead, its OC curve will remain above zero probability at the lower end of the lead scale, or below a probability of one at the higher end. Both can even occur simultaneously. Behavior of this type was often observed in the study. Logistic regression cannot adequately describe such phenomena without changes to the model.

The following model, while remaining functionally simple, produces nondecreasing OC curves that describe a broader range of phenomena than the logistic regression model:

$$OC(t) = c + d \frac{\exp(a + bt)}{1 + \exp(a + bt)}$$

This is referred to as the enhanced logistic regression model. Note that it has two parameters c and d in addition to those found in the simple logistic model. By setting $c = 0$ and $d = 1$, the simple model is obtained. Practical interpretation of the model parameters is explained in section 5.2.2.1. Again, t refers to the (natural) logarithm of the measured lead level.

The enhanced logistic model does not make provision for the fact that laboratory ICP measurement cannot perfectly measure the level of lead in paint. Detailed consideration of this issue is given in section 5.2.6.6 below. The effect of spatial variation and laboratory error in ICP measurements on the estimated performance of the test kits was demonstrated not to be substantial. Thus, it was possible to make valid inferences from the observable relationship between test kit measurements and the level of lead as measured by ICP.

A special case that merits attention concerns large values of parameter *b*, which governs how sharply the probability of a positive increases from *c* at low levels of lead, to *c* + *d* at higher lead levels. As *b* approaches infinity, the OC curve assumes the shape of a <u>step function</u>, which suggests that the probability of a positive is equal to *c* for all lead levels below a certain value, and *c* + *d* for all lead levels above. The lead level at which the probability changes is called the <u>changepoint</u>. There were three instances where step functions were chosen as estimates of the OC curve. They are indicated in the tables of section 5.2.3 as having *b* = *100*, and *a* equal to minus the changepoint of the step function (in log-units) times 100.

5.2.6.1.3 Modeling Based on Logarithms

The decision to use the logarithm of the lead level, instead of the lead level itself, was based on several factors: (1) the distribution of the logarithms was much closer to symmetric than that of the lead levels themselves; (2) variation in ICP measurements from the true lead levels was easier to handle when logarithms were taken; (3) the natural domain of a logistic or enhanced logistic regression model includes both positive and negative values. Although neither the logistic nor the enhanced logistic regression models requires the taking of logarithms, using the lead levels themselves may fail to capture salient features of test kit performance, and estimates may be strongly influenced by small groups of data at higher lead levels.

A difficulty posed by the taking of logarithms concerns the handling of zero lead level readings. Zeros can be handled in the enhanced logistic regression model by assigning a probability of *c*, which is the lower limit of the OC curve. Similarly, an option is to treat all lead levels below a pre-specified, small value as zeros. In the study no instances of zero ICP readings

were obtained, due to the recording of the detection limit itself when very low readings were observed. The handling of non-detects is discussed in section 5.2.6.5.

5.2.6.2 Nonparametric OC Curve Estimation

Two different nonparametric estimates of the OC curve were used: running means and monotone regression. Both estimates are briefly described in section 5.2.2.2, and were used primarily to graphically assess the fit of the enhanced logistic regression models to the data.

The running mean is obtained at a point log(ICP) = t by averaging zeros (for negatives) and ones (for positives) for a small subset of the data having ICP measurements close to t. It gives a "localized" estimate of the OC curve, and it is not necessarily a nondecreasing function of t. Choosing the subset to have a large amount of data makes the running mean appear smoother than if a small amount of data is used. Some of the resulting smoothness reflects bias, due to the averaging out of informative features. On the other hand, some of the non-smoothness obtained with a smaller averaging subset is due to random variability in the data, which is not informative. It was found that using 25 observations in the averaging window (12 values smaller, 12 values larger, plus one at t itself) achieved a reasonable balance between smoothness and bias for graphical purposes.

Towards the endpoints the two-sided averaging window cannot be used in the manner described. At the eighth smallest ICP measurement, for instance, there are 12 larger, but only 7 smaller ICP measurements. The running average in this instance was calculated by averaging 20 (12 + 7 + 1) ICP measurements in a restricted window. Restricted averaging windows were used to compute the running means at the 12 smallest and 12 largest ICP measurements, which necessarily introduced "endpoint effects". These effects arose because the denominators in the restricted window averages, consisting of the number of observations in the window, decreased as an endpoint was approached. To illustrate, suppose that the samples with the 20 smallest ICP measurements all had negative results, except for the ninth smallest, which had a positive result. The running mean at the eighth smallest measurement is then 1/20 = 0.05, but at the seventh smallest it is 1/19, at the sixth smallest 1/18, and the smallest value it is

1/13, or approximately 0.08. The statistical impact of these endpoint effects is minimal in large samples.

Monotone regression is a nonparametric technique that produces an estimate of the OC curve that is nondecreasing. It is obtained by averaging zeros (negatives) and ones (positives) in a way that a nondecreasing step function is obtained. The resulting function is "best" in that no other nondecreasing function can better fit the data, in either a least squares or a maximum likelihood sense. Monotone regression estimates were obtained using the "pool adjacent violators" (PAV) algorithm to minimize the sum of squared errors. These and other details concerning monotone regression can be found in Barlow, Bartholomew, Bremner and Brunk [8].

Monotone regression is also subject to endpoint effects: if the sample with the highest ICP measurement had a positive test kit result, then the monotone regression will be equal to 1 at that point. Similarly, if the lowest ICP measurement corresponds to a negative result, the monotone regression will be equal to zero at that lead level. The figures presented in section 5.2.3 attenuate endpoint effects. To illustrate, suppose that positive results were obtained corresponding to the 20 largest ICP measurements. The fourteenth largest ICP measurement (denoted t) represents a run of 7 consecutive positives, starting with the 20th largest value. The unattenuated monotone regression estimate at t is equal to 1. The attenuated value is $(.95)^{1/7}$ = .993, which is the 95% lower confidence bound for the probability of a positive result obtained from a sample of size 7 consisting entirely of positives. The attenuation is an increasing function of t, approaching a value of 1 if the run of positives at the upper endpoint is large. A similar attenuation scheme was used at the lower endpoint for runs of negative results. In both cases, attenuation was not allowed to violate the monotonicity of the estimated OC curve.

5.2.6.3 Estimation of Model Parameter and Related Quantities

The coefficients a, b, c and d of the enhanced logistic model were estimated using nonlinear least squares (NLS). Minimization of the sum of squared errors used a Newton-Raphson iterative algorithm. Constraints were imposed to ensure that c

did not become negative, and that $c + d$ did not exceed 1. On drywall, the constraint $c + d = 1$ was imposed, because the low lead levels present on this substrate did not support inferences of how the test kits performed at high lead levels. In several cases where the slope parameter b became large, suggesting a step function, minimization of the sum of squared errors was instead performed over the class of all possible step functions. In no case where a smooth model estimate is reported did a step function achieve a smaller sum of squared errors.

Maximum likelihood (MLE) was also explored as a method for estimating the model parameters. In most cases MLE and NLS estimates agreed closely. In several instances MLE was found to converge to local, nonoptimal solutions, while NLS did not encounter this difficulty. The MLE estimates were also sensitive to the presence of consecutive positive results at the highest ICP measurements, and to consecutive negatives at the lowest ICP values, which compromised the overall fit of the models to the data.

Attributes such as the 50-percent point, or the probability of a positive result at 1.0 mg/cm^2 of lead, were obtained directly from the estimated models, as functions of the parameters. Nonparametric estimates of these attributes were also obtained using monotone regression. The tables in section 5.2.3 report model estimates, with nonparametric estimates introduced in the narratives.

5.2.6.4 Standard Errors and Confidence Intervals

Asymptotically valid standard error estimates for the NLS estimated parameters were obtained using the Hessian matrix and gradient vectors obtained upon convergence of the algorithm. An estimated parameter plus or minus 2 times its standard error constitutes an approximate 95% confidence interval.

It should be noted that the estimated model parameters were often highly intercorrelated, which had the effect of inflating the individual standard error estimates. This indicates a condition where large changes in the model parameters produce visually similar OC curves. Under such circumstances, it is difficult to make meaningful inferences on the individual parameters.

Asymptotic 95% confidence intervals for 50-percent points and threshold probabilities were obtained using linear approximations to these quantities, as functions of the model parameters. The intervals were first derived on a transformed scale, and then transformed back to ensure that the confidence intervals were in the proper range.

The accuracy of asymptotic standard error estimates and confidence intervals was suspect in certain cases. This was particularly true when a step function, or a model with large slope parameter *b* approximating a step function, was obtained, or when the baseline probability parameter *c* was large. For this reason, bootstrap confidence intervals were also obtained for the 50-percent points and the threshold probabilities. The NLS model estimates were used as the basis for generating 1,000 bootstrap samples of test kit results. For the sake of computational efficiency, monotone regression was used to estimate the 50-percent point and detection probability for each bootstrap sample. The 2.5^{th} and 97.5^{th} percentiles of the respective bootstrap quantities were taken as the endpoints of the 95% confidence intervals.

Bootstrap confidence intervals for the 50-percent points were generally wider than the asymptotic intervals. The bootstrap confidence intervals for the threshold probabilities were, by contrast, generally narrower, especially when the estimated probabilities approached 1. For 50-percent points, the bootstrap confidence intervals are reported, except for drywall, where the asymptotic intervals are reported. For threshold probabilities, the asymptotic confidence intervals are reported, except where the model estimate has the form of a step function (Lead Alert: Sanding, on concrete and metal; Lead Detective on concrete), or where the value of the estimated model parameter *c* is greater than 0.3 (Lead Detective on plaster; State Sodium Sulfide on concrete).

The asymptotic confidence intervals for 50-percent points and threshold probabilities accounted for the correlations between estimated model parameters, and as a result they usually suggest less variability than the standard errors of the parameters themselves. The same is true of the bootstrap confidence intervals, for which variability in the OC curves, rather than in a set of model parameters used to represent them, is important. For these reasons, variability in estimated OC

curves is better inferred from the confidence intervals than from standard errors assigned individually to the model parameters.

5.2.6.5 The Treatment of Non-detects

Of the 1,290 sample locations where paint chip samples were analyzed for lead content, 54 (4.2%) had ICP measurements below the detection limit. The following breakdown demonstrates that the non-detects occurred most frequently on drywall substrates:

Substrate	Total	Non-detects	Percent
Brick	93	6	6.5
Concrete	226	10	4.4
Drywall	124	16	12.9
Metal	217	3	1.4
Plaster	242	10	4.1
Wood	388	9	2.3

The enhanced logistic regression model was applied to data using the non-detects, for which the recorded detection limits were taken as the ICP measurements. A detailed explanation of non-detects and the meaning of the detection limit is presented in Chapter 4. In essence, a non-detected ICP measurement represented information that the measured lead level was less than or equal to the detection limit.

The effect of handling non-detects in this manner was explored by comparing test kit modeling results for brick substrates to results obtained by (1) deleting the non-detects from the analysis, and (2) setting the non-detects equal to zero. Although drywall had a higher percentage of non-detects than brick, the absence of ICP measurements above 1.0 mg/cm^2 made it difficult to infer how the test kits would perform at moderate to high lead levels, regardless of how non-detects were used. Brick had the next highest percentage of non-detects, and it represented a wide range of lead levels in the study.

Deletion of non-detects had the greatest impact on the model estimates for the State Sodium Sulfide kit, where the parameter estimates changed from $c = .234$, $d = .766$, $a = 4.382$, $b = .976$ before deletion (93 observations) to $c = .305$, $d = .695$, $a = 4.312$, $b = 1.034$ after deletion (87 observations). The implications of this change for lead levels above 0.01 mg/cm^2 are not substantial. For example, at a lead level of .05 mg/cm^2 the

estimated probability of a positive result decreased from .855 to .841 when the non-detects were deleted. The effect of deletion on models fit to the other four test kits (Lead Alert: Sanding was excluded, as explained in section 5.2.3.3) was smaller.

Deletion was not a preferred option, because of the resulting loss of information. Setting non-detects to zero entails assigning these observations a model probability of *c* in NLS estimation. This had the greatest effect on the model estimates for LeadCheck, where the original parameter estimates of $c = 0$, $d = .988$, $a = 3.005$, $b = .687$ changed to $c = .106$, $d = .870$, $a = 3.404$, $b = .906$. The practical effect of this change is small for lead levels above 0.05 mg/cm^2, for which the estimated probability of a positive was .712 originally, and .685 with the non-detects treated as zeros. The other four test kits exhibited smaller changes when the non-detects were treated as zeros.

The use of the detection limit as the ICP measurement for non-detect paint chip samples, as was done in the analyses presented in section 5.2, allowed these samples to be treated in a manner consistent with their indicated low lead levels. The above exercises demonstrate that estimation of important aspects of test kit performance was not critically affected by this designation.

5.2.6.6 The Impact of Spatial Variation and Laboratory Error in ICP Measurements on Model Estimation

In linear regression, it is well known that regressing onto a variable measured with error produces biased estimates of the regression parameters. The presence of error dulls the apparent relationship between the response variable and the "true" regressor. The same is true in both the simple and enhanced logistic models: measuring the true lead level with error produces biased estimates of the model parameters. The effect of the bias is to produce OC curves that are somewhat flattened out. The larger the errors, the greater this effect becomes.

The true concentrations of lead were not available, since the laboratory measurements, while more precise than other types of measurement, were affected both by laboratory error and spatial variation, and therefore cannot be regarded as perfectly

accurate. Work reported in Chapter 4 established a model in which the standard deviation (SD) of the combined error is proportional to the true lead concentration. Taking logarithms converts this model to one with additive errors. Accounting for laboratory error alone suggested an SD equal to about 0.125. Accounting for spatial variation in addition to laboratory error increased the SD to a magnitude in the range 0.2 to 0.4, using the results reported in Table 4-23.

There is a small volume of statistical literature on estimating logistic regression parameters with errors in the regression variables. Most of this work was conducted within the last ten years, and it is still an active topic of statistical research. Some references on the measurement error problem in logistic regression are listed in Chapter 8. A recent reference is Stefanski and Carroll [9], who used a two-stage estimation procedure based on an adjustment of the regression variables. A slightly different approach was taken in Whittemore and Keller [10], wherein the parameter estimates, as opposed to the variables, were adjusted for the effects of measurement error. The Whittemore-Keller estimator was cited by Stefanski [11] as a refinement of his earlier method.

Although the techniques presented in the literature vary somewhat in their approaches to undoing the bias caused by measurement error, they share certain common features. Perhaps the most important is a recognition that reduction of the bias comes at the price of increasing the standard errors of the estimated parameters. This places the measurement error problem in the framework of a bias-variance tradeoff that is encountered in many different kinds of statistical problems. The practical effect of this tradeoff is to make full bias correction a less than optimal strategy, which is reflected in both the Stefanski-Carroll and the Whittemore-Keller estimators. Generally, the amount of bias reduced increases as the sample size becomes larger. Several researchers have suggested that no bias correction is best in smaller samples.

Experience from simulations on the test kit data suggests that the amount of bias reduction can be 50 percent or less using the Whittemore-Keller estimator with measurement error standard deviations as large as 0.5. Published work suggests that the Stefanski-Carroll estimator should behave in a similar manner. Applying the Whittemore-Keller estimator to the test kit data

usually produced only small changes in the parameter estimates, and even smaller changes in quantities (probabilities, 50-percent points) derived from the estimated models.

The foregoing discussion applies only to the simple logistic model, not to the enhanced model that is more appropriate for use on the test kit data. Correcting for error introduced by the substitution of ICP measurements for true lead levels in the enhanced model does not appear to be straightforward. Still, experience with the Whittemore-Keller estimator in the simple logistic model suggested that the amount of bias correction that the data could support would likewise be small.

To assess measurement error bias in the enhanced logistic model, a small Monte Carlo experiment was conducted on six test kit-substrate combinations. The enhanced logistic model was fit to each set of data (regarded as the zero-measurement error case), and to the data with random normal measurement errors added to the log(ICP) values. Ten simulations were conducted for measurement error standard deviations equal to 0.15, 0.30, and 0.60, and the enhanced model estimates for the simulated data sets were averaged. The differences between the zero-error estimates and the simulation averages gave an indication of the degree of bias resulting from measurement error.

Table 5-26 summarizes the results of this study. It should be noted that what may seem to be large changes in the model parameters a, b, c, and d do not necessarily portend large changes in the probabilities derived from the model, or in the estimated levels of lead that achieve a 50 percent probability of a positive test kit reading. In particular, the estimated probability of a positive reading at a lead concentration equal to 1.0 mg/cm^2 changed very little in all six cases as the

Table 5-26. Results of Monte Carlo experiment to assess the effect of measurement error on enhanced logistic model estimates. Based on 10 simulated normal samples per error level.

KIT	SUB	SAMPLE SIZE	SCENARIO	MODEL				Pb(.50) mg/cm²	PROB AT Pb = 1 mg/cm²
				c	d	a	b		
Lead Check	Concrete	226	Original data	.096	.904	.701	.539	.184	.700
			SD=0.15	.093	.908	.701	.534	.183	.699
			SD=0.30	.107	.893	.680	.556	.189	.700
			SD=0.60	.070	.930	.655	.476	.180	.682
Lead Alert Coring	Brick	93	Original data	.180	.820	1.252	1.383	.293	.818
			SD=0.15	.182	.818	1.194	1.353	.296	.809
			SD=0.30	.177	.823	1.139	1.255	.282	.799
			SD=0.60	.171	.829	.972	1.051	.265	.772
Lead Alert Sanding	Metal	118	Original data	.035	.965	-1.227	.682	5.448	.254
			SD=0.15	.031	.969	-1.212	.635	6.174	.253
			SD=0.30	.031	.954	-1.212	.652	6.852	.251
			SD=0.60	.015	.954	-1.102	.506	12.684	.252
Lead Detective	Wood	388	Original data	.099	.901	1.121	.810	.191	.779
			SD=0.15	.097	.903	1.109	.799	.189	.776
			SD=0.30	.098	.902	1.111	.795	.189	.776
			SD=0.60	.095	.905	1.033	.733	.183	.762
Lead Zone	Plaster	242	Original data	.036	.964	-0.134	.560	1.113	.486
			SD=0.15	.035	.965	-0.130	.563	1.112	.486
			SD=0.30	.018	.980	-0.129	.503	1.217	.477
			SD=0.60	.016	.966	-0.158	.448	1.474	.460
State Sodium Sulfide	Concrete	224	Original data	.410	.590	2.531	.950	.011	.957
			SD=0.15	.403	.597	2.533	.935	.011	.956
			SD=0.30	.327	.673	2.463	.801	.010	.947
			SD=0.60	.318	.682	2.401	.777	.008	.943

measurement error SD was increased to 0.60. In five of the six cases the same conclusion held for the estimated 50 percent point, with the exception of Lead Alert sanding on metal, where the estimate increased from 5.448 to 12.684 mg/cm² as the measurement error SD increased from 0 to 0.6. The effect was

less severe when it is considered that the original estimate was outside of the range of the data, making it unreliable even in the absence of measurement error. An approximate 95% confidence interval, calculated at 0.692 to 42.927 mg/cm^2 from the original data, contained all of the simulation averages.

Again, it is emphasized that a bias correction methodology could not be expected to correct for all of the bias indicated in the experiment. This and the apparently small order of bias suggested by the Monte Carlo experiment for quantities of interest support the conclusion that the use of the enhanced logistic model, without accounting for the effect of error caused by substituting ICP measurements for the true lead levels, allowed valid inferences to be drawn about the sensitivity of the test kits to the true amount of lead present in paint.

5.3 EFFECT OF DARKNESS OF SHADE (GREY-TO-BLACK) ON PERFORMANCE OF THE SULFIDE KITS

For the two sodium sulfide kits, information was collected in the field on the darkness of the positive tests, which may range from light grey to black. In Louisville, the testers were asked to note and record whether the color was grey or black. In Denver and Philadelphia, a series of five boxes shaded from light grey to black was provided on the data collection form for each location, and the testers were asked to mark the box which most closely approximated the test result. The shades were categorized as 1 (light grey) to 5 (black). Thus, the information from Denver and Philadelphia is more accurate and comprehensive than the Louisville information. Therefore, only the Denver and Philadelphia shade information will be analyzed. The results of this analysis address the study objective to characterize the relationship between test kit results and the actual lead level in the paint.

Table 5-27 presents summary statistics for Lead Detective in mg/cm^2, for Denver and Philadelphia combined, by shade category, and for positive and negative results overall. The last line of the table shows the percentile represented by the 1.0 mg/cm^2 federal standard for each category. Table 5-28 shows the same information for State Sodium Sulfide (note that the shade = 5 category is missing for this kit because the tester never selected the darkest shade on the form). Tables 5-29 and 5-30 present summary statistics for Lead Detective and State Sodium

Sulfide in percent lead by weight. Figures 5-42 and 5-43 give a graphical depiction of the distributions by shade category for mg/cm^2 and Figures 5-44 and 5-45 give a graphical depiction of the distributions by shade category for percent lead by weight. The vertical axes are on the natural logarithm scale. Central tendency is represented by the median, and shade = 0 represents

Table 5-27. Summary Statistics for **Lead Detective** in mg/cm^2 for Denver and Philadelphia Combined, by Shade Category, and for Positive and Negative Results Overall.

SUMMARY STATISTICS	KIT RESULT		SHADE CATEGORY				
	Neg	**Pos**	**1**	**2**	**3**	**4**	**5**
N	688	500	127	130	114	53	23
Mean	0.64	1.76	1.33	0.95	1.74	3.66	5.59
Median	0.09	0.41	0.15	0.32	0.65	0.91	2.64
25th percentile	0.008	0.092	0.046	0.088	0.206	0.138	0.659
75th percentile	0.31	1.52	0.51	0.77	2.03	2.41	8.29
Minimum	0.0001	0.0001	0.0001	0.0003	0.0009	0.0015	0.0317
Maximum	34.09	37.29	22.28	13.45	15.09	37.29	24.77
1.0 mg/cm^2 percentile	0.92	0.68	0.87	0.78	0.60	0.53	0.35

Table 5-28. Summary Statistics for **State Sodium Sulfide** in mg/cm^2 for Denver and Philadelphia Combined, by Shade Category, and for Positive and Negative Results Overall.

SUMMARY STATISTICS	KIT RESULT		SHADE CATEGORY				
	Neg	**Pos**	**1**	**2**	**3**	**4**	**5***
N	320	868	419	282	129	28	N/A
Mean	0.07	1.49	0.45	1.92	2.35	8.24	N/A
Median	0.008	0.323	0.190	0.418	1.494	2.483	N/A
25th percentile	0.002	0.106	0.055	0.184	0.620	1.430	N/A
75th percentile	0.07	1.00	0.38	1.41	2.39	15.92	N/A
Minimum	0.0001	0.0002	0.0002	0.0003	0.0025	0.2244	N/A
Maximum	3.77	37.29	30.58	34.09	18.21	37.29	N/A
1.0 mg/cm^2 percentile	0.99	0.75	0.95	0.70	0.35	0.18	N/A

* The State Sodium Sulfide test kit sampler never selected the shade 5 category.

Table 5-29. Summary Statistics for **Lead Detective** in Percent by Weight for Denver and Philadelphia Combined, by Shade Category, and for Positive and Negative Results Overall.

SUMMARY STATISTICS	KIT RESULT		SHADE CATEGORY				
	Neg	**Pos**	**1**	**2**	**3**	**4**	**5**
N	688	500	127	130	114	53	23
Mean	0.421	1.764	0.421	1.024	1.670	3.551	7.097
Median	0.120	0.394	0.120	0.292	0.785	1.510	3.737
25th percentile	0.019	0.124	0.019	0.107	0.222	0.138	1.054
75th percentile	0.277	1.971	0.277	1.284	2.274	4.388	7.073
Minimum	0.0005	0.0007	0.0005	0.0021	0.0007	0.0012	0.019
Maximum	12.161	34.559	12.161	9.685	14.828	34.192	34.559
0.5% by wt percentile	0.86	0.54	0.75	0.65	0.43	0.40	0.22

Table 5-30. Summary Statistics for **State Sodium Sulfide** in Percent by Weight for Denver and Philadelphia Combined, by Shade Category, and for Positive and Negative Results Overall.

SUMMARY STATISTICS	KIT RESULT		SHADE CATEGORY				
	Neg	**Pos**	**1**	**2**	**3**	**4**	**5***
N	320	868	419	282	129	28	N/A
Mean	0.130	1.302	0.397	1.366	2.325	9.371	N/A
Median	0.026	0.280	0.179	0.379	1.605	4.593	N/A
25th percentile	0.004	0.120	0.067	0.154	0.617	1.756	N/A
75th percentile	0.111	1.120	0.309	1.535	3.531	16.199	N/A
Minimum	0.0005	0.0006	0.0006	0.0011	0.0022	0.403	N/A
Maximum	6.491	34.559	9.402	14.828	19.153	34.559	N/A
0.5% by wt percentile	0.96	0.64	0.87	0.56	0.22	0.11	N/A

* The State Sodium Sulfide test kit sampler never selected the shade 5 category.

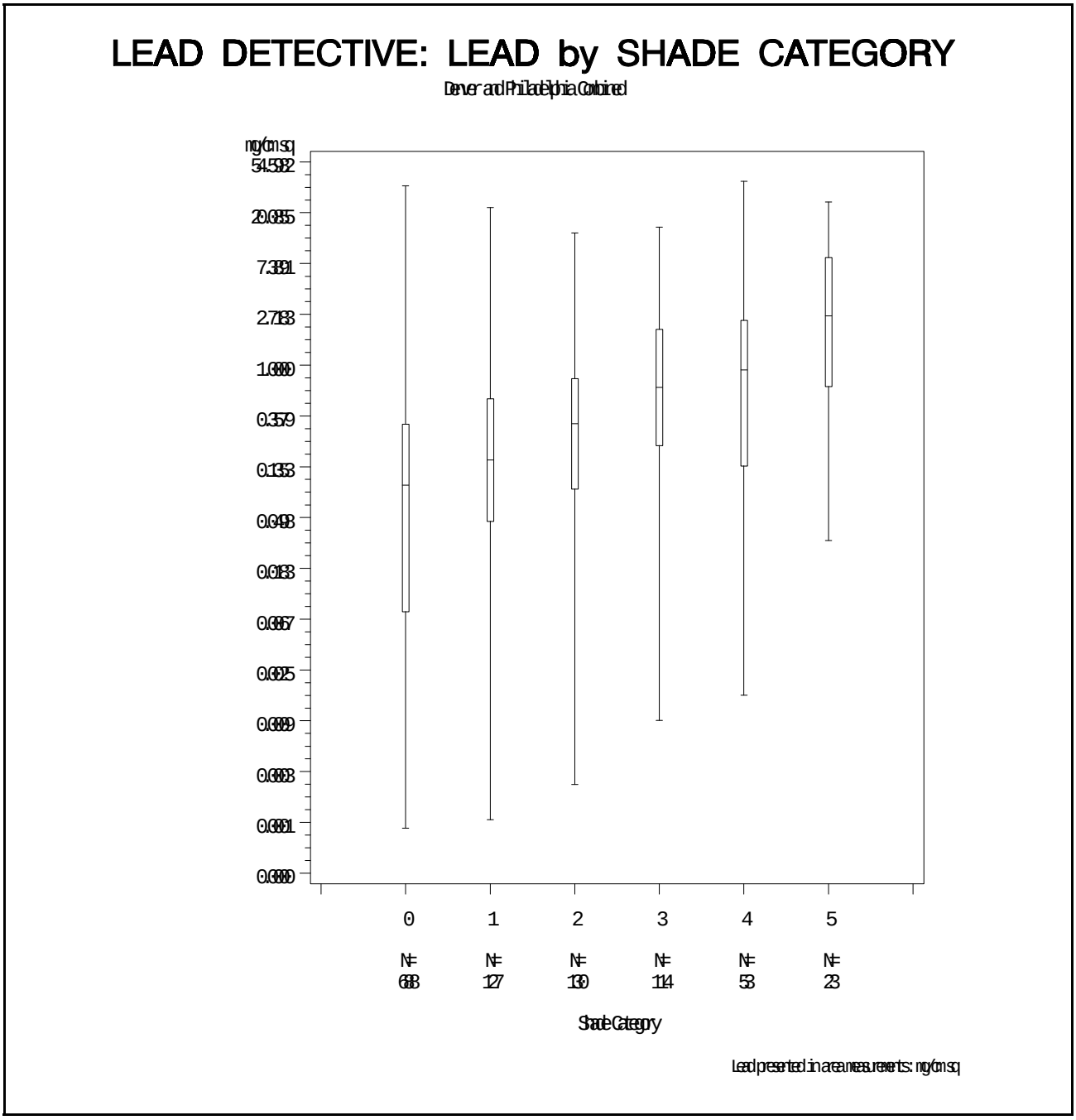

Figure 5-42: The bottom and top edges of the box are located at the sample 25th and the 75th percentiles. The center horizontal line in the box is drawn at the sample 50th percentile (median). The vertical line extending above and below the box is drawn to the maximum and minimum sample values. Shade categories are tabulated from reported test kit results. The "0" (zero) category represents the reported negative results. Shade categories "1" through "5" are categories of positive results. Since positive results are indicated by a color change, categories "1" through "5" are a gradation of color changes with category "1" being the lightest color change (i.e., light grey) and "5" being the strongest color change (i.e., black). The sample sizes appear below the shade category labels.

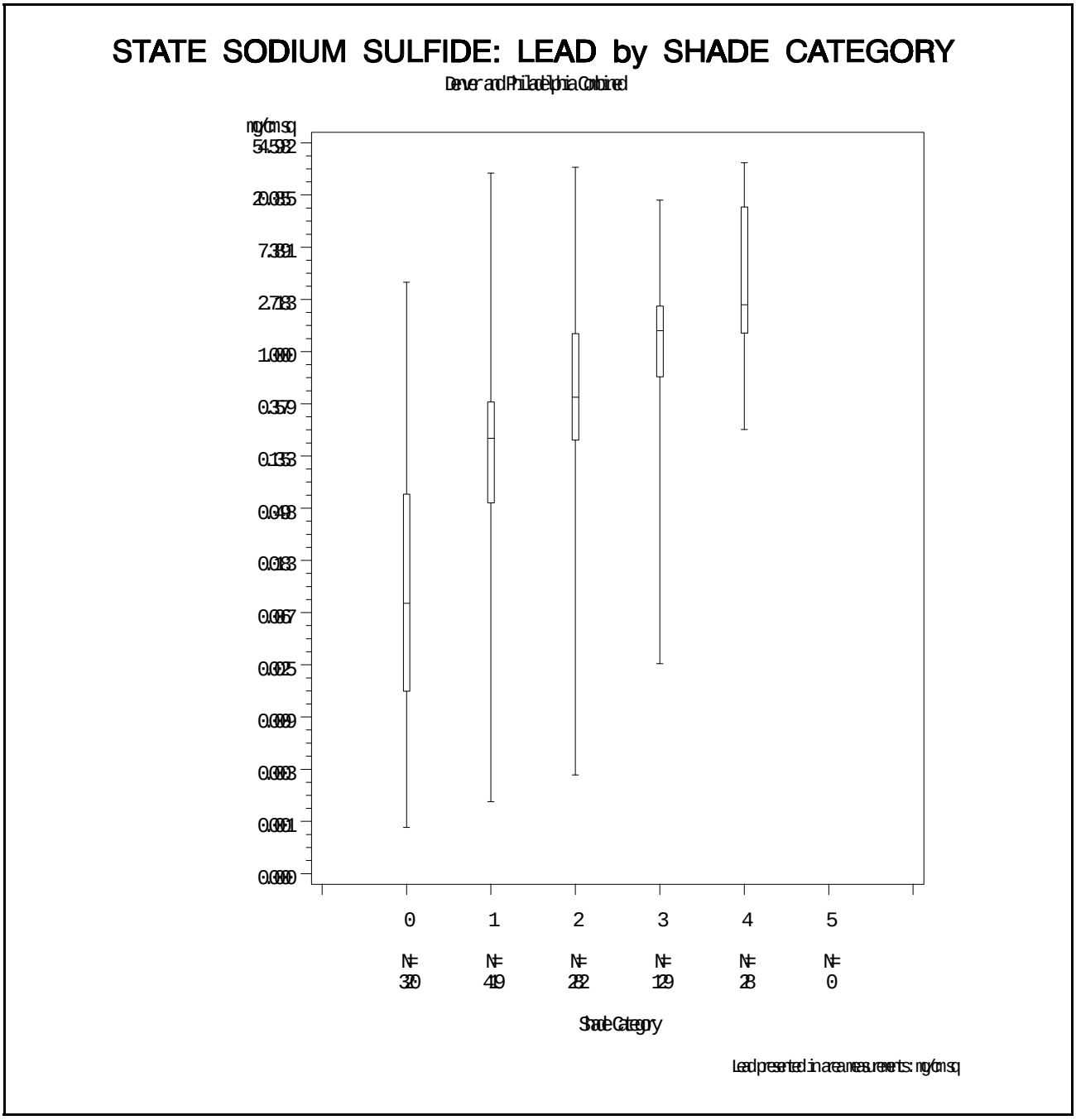

Figure 5-43: The bottom and top edges of the box are located at the sample 25th and the 75th percentiles. The center horizontal line in the box is drawn at the sample 50th percentile (median). The vertical line extending above and below the box is drawn to the maximum and minimum sample values. Shade categories are tabulated from reported test kit results. The "0" (zero) category represents the reported negative results. Shade categories "1" through "5" are categories of positive results. Since positive results are indicated by a color change, categories "1" through "5" are a gradation of color changes with category "1" being the lightest color change (i.e., light grey) and "5" being the strongest color change (i.e., black). The sample sizes appear below the shade category labels.

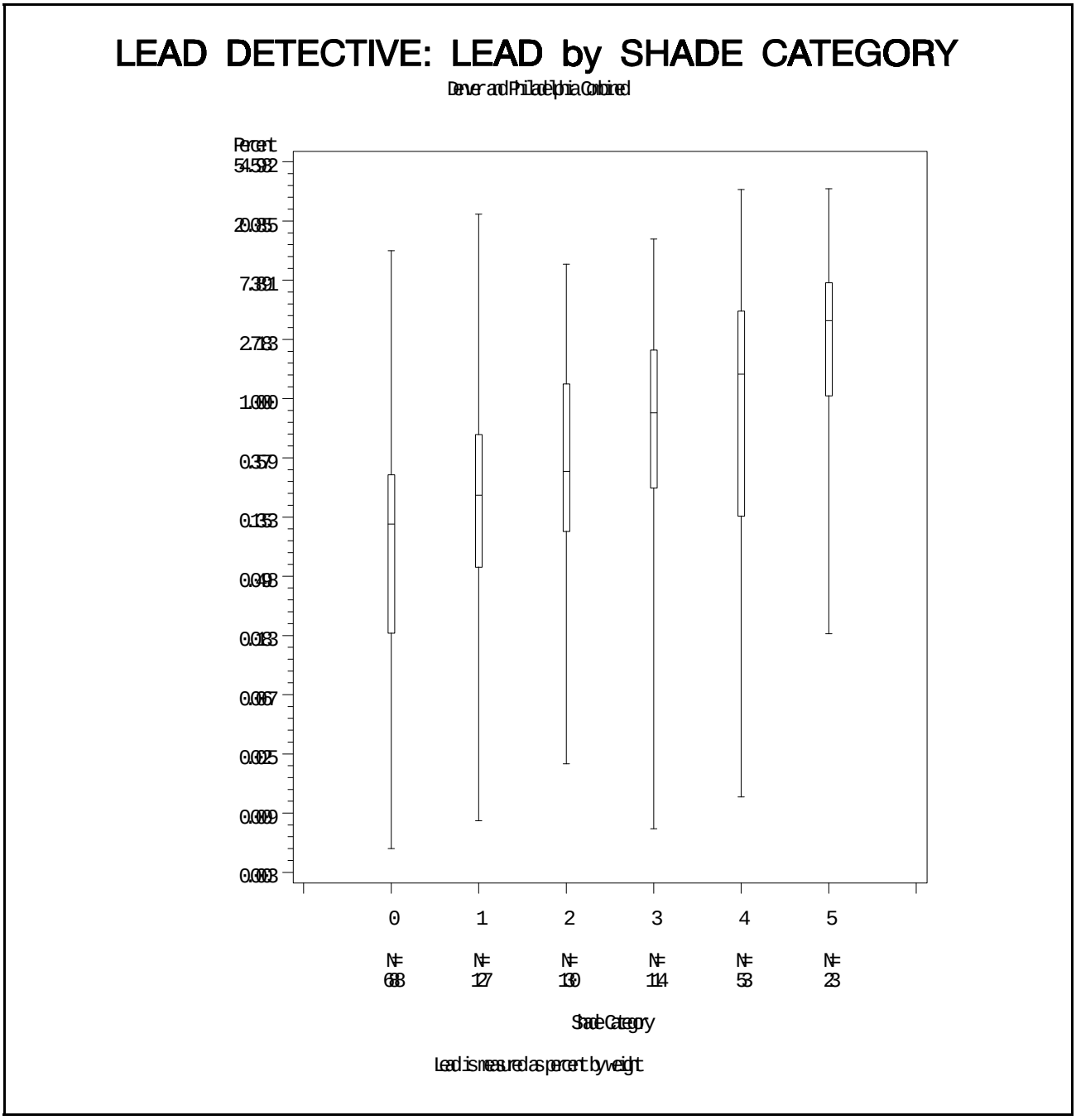

Figure 5-44: The bottom and top edges of the box are located at the sample 25th and the 75th percentiles. The center horizontal line in the box is drawn at the sample 50th percentile (median). The vertical line extending above and below the box is drawn to the maximum and minimum sample values. Shade categories are tabulated from reported test kit results. The "0" (zero) category represents the reported negative results. Shade categories "1" through "5" are categories of positive results. Since positive results are indicated by a color change, categories "1" through "5" are a gradation of color changes with category "1" being the lightest color change (i.e., light grey) and "5" being the strongest color change (i.e., black). The sample sizes appear below the shade category labels.

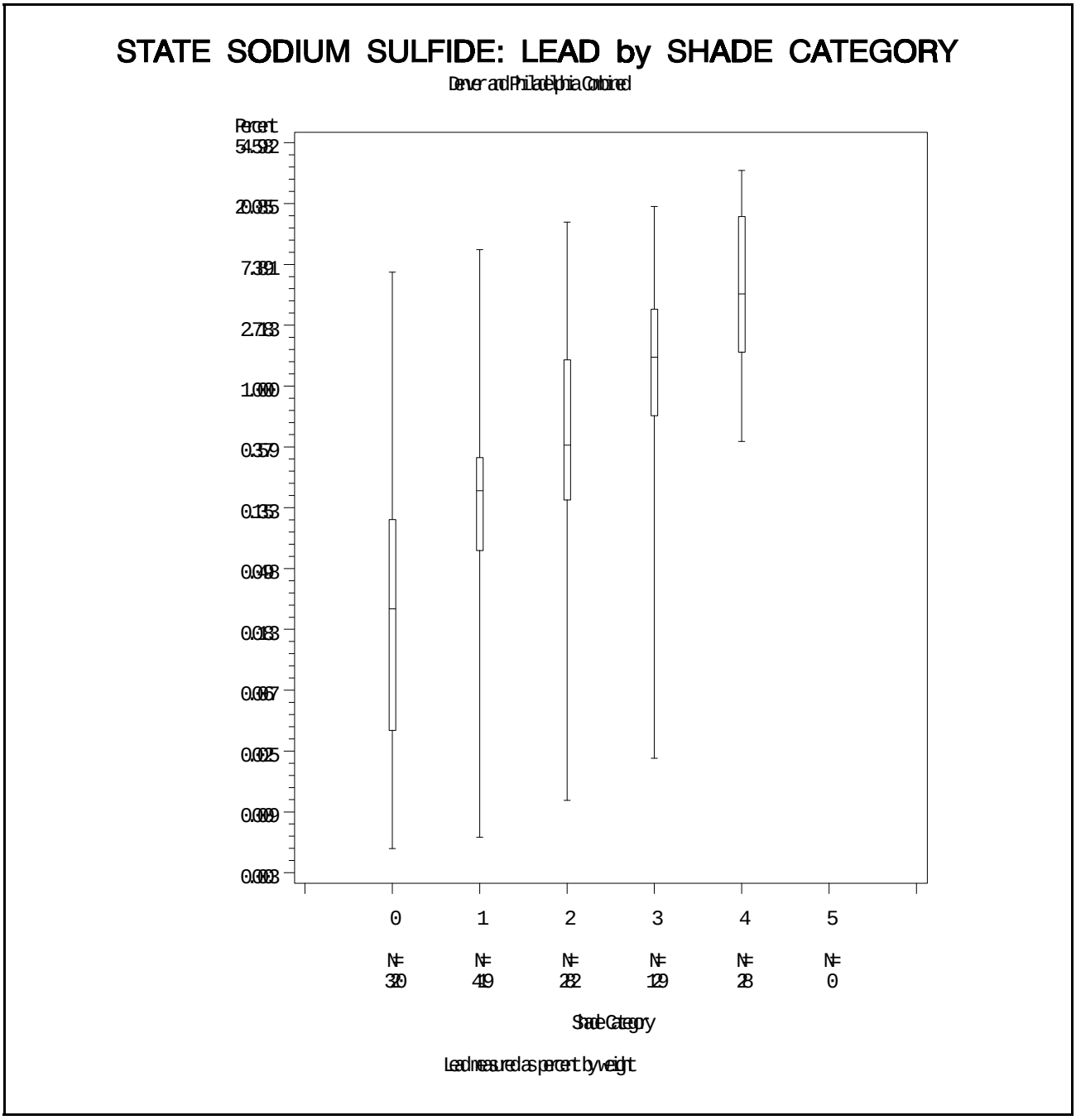

Figure 5-45: The bottom and top edges of the box are located at the sample 25th and the 75th percentiles. The center horizontal line in the box is drawn at the sample 50th percentile (median). The vertical line extending above and below the box is drawn to the maximum and minimum sample values. Shade categories are tabulated from reported test kit results. The "0" (zero) category represents the reported negative results. Shade categories "1" through "5" are categories of positive results. Since positive results are indicated by a color change, categories "1" through "5" are a gradation of color changes with category "1" being the lightest color change (i.e., light grey) and "5" being the strongest color change (i.e., black). The sample sizes appear below the shade category labels.

the negatives for each kit.

Comparisons between the results for lead measured in area units (Tables 5-27 and 5-28 and Figures 5-42 and 5-43) to the results for lead measured as a percent by weight (Tables 5-29 and 5-30 and Figures 5-44 and 5-45) show many similarities. For example, for Lead Detective, the means and medians are similar when comparing the shade category results. Likewise, the means and medians are similar for the State kit. Several other observations can be made based on the tables and figures and are given below.

The median lead level for both kits increases as the shade darkens from 1 to 5. The median level for the negative results is also lower than for shade = 1. A similar pattern is shown by the means, although shade = 2 for Lead Detective has a lower mean than shade = 1. Thus, it is clear that, on average, the darker shades on the sulfide kits <u>do</u> represent higher levels of lead in the paint.

The maximum values of the negative results of the two test kits are very different. The maximum value of the negative results for the State test kit is 3.77 mg/cm^2 while 34.09 mg/cm^2 is the maximum negative result for the Lead Detective test kit.

When one focuses on the extremes of the distribution, the relationship between lead level and shade is much less clear. The minimum lead level represented by each shade category does not change appreciably until the darkest shade (5 for Lead Detective, 4 for State Sodium Sulfide) is reached. Also, the percentile represented by 1.0 mg/cm^2 is at least 35% for all shade categories except shade = 4 for State Sodium Sulfide. This means that even the darker shades for these kits have a significant false positive problem. Likewise, the maximum lead level appears unrelated to the shade.

The operating characteristics of the kits would be changed dramatically if one or more of the lighter shades were counted as negative. For the State kit, if one were to count shade = 1 as a negative rather than as a positive, the false positive rate for the kit would be reduced from 67% (Denver and Philadelphia combined) to 26%, while the false negative rate would be increased from 1% to 11%. However, this would then make the State kit unreliable as a negative screen, while the false

positive rate remains too high for use as a positive screen. If one were to treat both shade = 1 and shade = 2 as negatives, the false positive rate declines to 6% and the false negative rate increases to 50%, making the kit effective as a positive screen. To make Lead Detective effective as a positive screen, shade = 1, 2 or 3 must be treated as negative, resulting in false positive rate of 6% and a false negative rate of 67%.

Another possibility is to treat one or more of the lighter shades as inconclusive subject to laboratory confirmation. For the State kit, treating shade = 1 and shade = 2 as inconclusive would give a kit with low rates of both false positive (6%) and false negative (1%), at the expense of a very high rate of inconclusive (59%). For Lead Detective the false negative rate cannot be reduced below 26%. This approach is not useful for testing because the error rates are not greatly reduced and the number of inconclusive results is large.

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Chapter 6 Summary: Analysis of XRF Testing Data

- **Most K-shell instruments exhibited relatively high variability, even for paint with low levels of lead. The amount of variability was sometimes related to the level of lead in the sample.**

- **Biases of most K-shell instruments were strongly substrate dependent.**

- **With the exception of the XL prototype, test results using L-shell instruments exhibited large negative biases at the 1.0 mg/cm^2 threshold. However, test results using L-shell instruments were less variable than results obtained using K-shell instruments.**

- **The XL results showed smaller biases at 1.0 mg/cm^2 than results of the other L-shell instruments, but still showed large negative biases at higher lead levels.**

- **Substrate correction obtained using readings for NIST SRM paint films placed on test location areas scraped bare of paint reduced bias for results using the Microlead I and the XK-3, and for the MAP-3 K-shell instrument results on metal and wood substrates. The already low bias of the Lead Analyzer's K-shell results was unchanged.**

- **With the exception of the XK-3 and the MAP-3 on some substrates, substrate correction using readings for NIST SRM paint films placed on control blocks of substrate materials brought to the site was not effective in reducing biases of K-shell readings attributable to substrates.**

Chapter 6 Summary: Analysis of XRF Testing Data (continued)

- **Despite the generally high variability and bias of their results, K-shell XRF instruments reliably classified the paint samples in this study using the federal threshold of 1.0 mg/cm^2, with laboratory confirmation of XRF readings between 0.4 and 1.6 mg/cm^2 and correction of biases attributable to substrates as needed.**

- **When the laboratory confirmation range was narrowed to 0.7 to 1.3 mg/cm^2, thereby substantially reducing the inconclusive percentages, the K-shell instruments continued to reliably classify paint samples in this study.**

- **Without a laboratory confirmation range, the K-shell instruments' performance differed when classifying paint samples in this study using the federal threshold of 1.0 mg/cm^2.**

- **With the exception of the XL, L-shell instruments performed poorly when classifying paint using the 1.0 mg/cm^2 threshold, because of a high rate of false negative results.**

- **Although the XL prototype had a lower rate of false negative results than the other L-shell instruments, it still exhibited false negative results at very high lead levels.**

- **Generally, a single XRF reading at one point of an architectural component provided almost as much accuracy as an average of three XRF readings at the same point.**

6 ANALYSIS OF XRF TESTING DATA

Analysis results and discussion of X-Ray Fluorescence (XRF) instrument performance are provided in this chapter to address the following study objectives:

- to characterize the performance (precision and accuracy) or portable XRF instruments under field conditions
- to evaluate the effect on XRF performance of interference from material (the substrate) underlying the paint
- to investigate XRF measurements that were very different than their corresponding laboratory results
- to evaluate field quality assurance and control methods.

There were six XRF instruments that were evaluated in this study. These were the Lead Analyzer manufactured by TN Technologies, Inc.; the MAP-3 manufactured by Scitec Corporation; the Microlead I (ML I) revision 4 manufactured by Warrington, Inc.; the X-MET 880 manufactured by Outokumpu Electronics; the XK-3 manufactured by Princeton Gamma-Tech, Inc.; and the XL prototype manufactured by Niton Corporation.

This chapter provides a description of the various XRF data that were collected and the statistical analysis methods used to analyze these data. Results of the statistical analysis are presented. Section 6.4 is the heart of the chapter. Readers interested primarily in bias and precision estimates for XRF instruments may proceed directly to that section.

The seven sections of the chapter are as follows. Section 6.1 describes the data that were collected and defines the variables used in the statistical analyses. Section 6.2 provides descriptive statistics for the standard and control data collected in Louisville, Denver, and Philadelphia. Section 6.3 provides a description of the methodology used to detect outliers with a discussion of the results. Section 6.4 presents parametric estimates of the accuracy of XRF instruments including estimates of XRF instrument bias and precision. This section also compares results for a single XRF reading versus an average of three readings and examines the difference between readings corrected for substrate interference versus those that were not. Presented next in section 6.5 is a comparison of different types of XRF measurements (single reading versus average and corrected versus uncorrected) using classification analysis results,

including estimates of the effect that ICP measurement error has on these classification results. Estimates of false positive and false negative rates when compared to the laboratory result classified against the 1.0 mg/cm^2 federal standard are presented. Section 6.6 presents a discussion on effects related to changing from one substrate to another. Section 6.7 provides descriptive statistics for the "special" and non-standard data collected in Louisville, Denver, and Philadelphia.

6.1 DATA DESCRIPTION

The descriptive statistics provided in this section address the following study objectives:

- to characterize the performance (precision and accuracy) or portable XRF instruments under field conditions
- to evaluate the effect on XRF performance of interference from material (the substrate) underlying the paint
- to evaluate field quality assurance and control methods.

For the lead-based paint measurement study, data in the form of XRF readings were collected at locations in three cities as was described in previous chapters. The Lead Analyzer, MAP-3, Microlead I revision 4, X-MET 880, XK-3, and XL were used in the full study in Denver and Philadelphia. The XL instruments used in this study were prototype models. Four of the six instrument types were used in the pilot study in Louisville: the MAP-3, Microlead I revision 4, X-MET 880, and XK-3. Data were first collected at a site in Louisville from March 30 through April 2, 1993. Since this was a pilot study, a relatively small amount of data was collected and analyzed. The results of the analysis provided information for planning the data collection for the full study. As a result, the data collection protocol was modified after the pilot study and before the full study work began. The full study was completed in Denver in August, 1993 followed by XRF data collection in Philadelphia in October, 1993.

The datasets used for the statistical analyses consisted of different subsets of the full dataset. This section describes the data and how they were categorized to form the analysis datasets. To understand the analysis dataset descriptions, a brief description of how the data were collected is necessary. This is particularly true since the data collection protocol used in the pilot study differed from that used in the full study,

creating many types of data. Some data collected in the pilot study are not directly comparable to data collected in the full study. However, many of the data are directly comparable and are included in the analysis datasets.

The XRF instruments differed in the way results are displayed. Two XRF instruments truncated readings, four XRF instruments reported negative readings, and one instrument, the Microlead I revision 4, reported readings as running averages. (The *Microlead I revision 4 Instruction Guide* [12] provides a description of how the Microlead I displayed these averages). The XK-3 and the XL instruments truncated readings at high lead levels; the XK-3 truncated at 10.0 mg/cm^2 and the XL truncated at 5.0 mg/cm^2. The four instruments that reported negative readings were the Lead Analyzer, MAP-3, Microlead I revision 4, and XK-3 instruments. The XL instrument truncated all negative readings at 0.00 cm/mg^2. The X-MET 880 did not report negative values. The X-MET 880 used in Louisville reported zero values, but the instrument used in Denver and Philadelphia did not report zero results. Hence, it was unclear whether this instrument truncated at zero. For some analyses presented in subsequent sections of this report, truncated readings were excluded. Methodology descriptions will indicate when this occurs.

As stated above, the Microlead I revision 4 reported readings as running averages. For this XRF instrument only, multiple readings were obtained with a single depression of the trigger. Nominal 15-second readings were displayed and recorded by the instrument in succession as running averages. Most of the time for this study, three readings were required as described in the study testing protocol. For this case, the trigger on the instrument was depressed until three successive readings were displayed and recorded. The first displayed reading was a single reading rather than an average. The reading displayed second was regarded as the average of the first two readings and the reading displayed third was regarded as the average of all three readings. For some sampling locations in Louisville, four readings were required (see chapter 3, section 5.2.7). For this case, the trigger on the instrument was held until four successive readings were displayed and recorded. The first three readings were as described above. The reading displayed fourth was regarded as the average of all four readings. These readings were always reduced to single readings prior to data analysis using the following formulas:

$$\begin{aligned} \textit{Fourth reading} &= 4*4^{th}\textit{display} - 3*3^{rd}\textit{display} \\ \textit{Third reading} &= 3*3^{rd}\textit{display} - 2*2^{nd}\textit{display} \\ \textit{Second reading} &= 2*2^{nd}\textit{display} - 1^{st}\textit{display} \\ \textit{First reading} &= 1^{st}\textit{display} \end{aligned}$$

Several specific types of XRF data were collected. All of these types of data are XRF readings that can be categorized as one of the following four data types:

- standard,
- control,
- special, and
- non-standard.

Differences and similarities between the pilot study data and the full study data along with descriptions of the field and analysis data are provided in the following five sections. Sections 6.1.1 through 6.1.4 provide descriptions of the standard, control, special, and non-standard data, respectively. Section 6.1.5 describes the analysis datasets.

6.1.1 Standard Data Description

Standard data refers to data collected using the standard data collection protocol and constitutes the bulk of the XRF data. There are two types of standard readings, each being a single nominal 15-second reading[1]. A more detailed discussion of nominal reading times was presented in chapter 3. One type of standard reading is the nominal 15-second reading taken on the painted surface of the sampling location. The other type of standard reading is the nominal 15-second XRF reading taken on the bare substrate covered with a red (1.02 mg/cm^2) NIST SRM film. A set of standard readings is defined to be six nominal 15-second readings taken at the same sampling location: three on the painted surface and three on the bare substrate covered by the red NIST SRM film. These will be referred to as the first, second, and third standard paint readings and the first, second, and third red NIST SRM readings. Note that the numbered order is

[1]Nominal reading time is an XRF instrument surface exposure and X-ray data collection time that is based on a new, non-decayed, radiation source. Nominal reading times used in this study were 15 seconds, 60 seconds, and 240 seconds.

the order in which the readings were actually taken.

Standard readings were taken from August 4 through August 19, 1993 in Denver and October 11 through October 25, 1993 in Philadelphia with all six XRF instrument types. Standard readings were taken from March 30 through April 2, 1993 with the Microlead I revision 4 and XK-3 in Louisville. The readings taken by the MAP-3 and X-MET 880 in Louisville are not defined as standard readings. The MAP-3 data that were collected in Louisville are not standard readings because, according to the pilot study data collection protocol, nominal 60-second readings were taken at each sampling location instead of nominal 15-second readings. The X-MET 880 in Louisville made nominal 15-second readings; however, the instrument in the pilot study, with a cadmium (Cd^{109}) source, behaved differently from the instrument used in the full study which had a curium (Cm^{244}) source. The reason for the observed differences between the X-MET 880 instruments is unknown; however, it seems plausible that different sources could give different results. Section 6.4 of this chapter provides a comparison of the results from the two X-MET 880 instruments. As a result of defining the pilot data for the MAP-3 and the X-MET 880 as non-standard, limited analyses were performed on these data.

For each of the six XRF instrument types given above, at least two individual, distinct, instruments were used in this study. Some instruments were represented by several individual instruments. For example, five distinct Microlead I revision 4 instruments were used. Table 6-1 summarizes the individual XRF instrument usage. For each individual XRF instrument, the dates on which standard readings were taken, the number of houses or units tested, and the total number of sampling locations tested are shown in Table 6-1. The "Standard Reading Total" values are shown in the right-most column in Table 6-1. The totals in this column were computed by combining the values in the "Total Number of Locations" column for all XRF instruments of the same type. As previously discussed, the 100 locations where readings were taken by the MAP-3 and X-MET 880 in Louisville are not included in these totals. Therefore, the right-most column shows the number of sampling locations at which a set of standard readings was made for each instrument type. Finally, since the total number of sampling locations used in the pilot study and the full study combined was 1,290, it can be observed in Table 6-1 that some instrument types provided two sets of standard readings for

most sampling locations.

A factor in selecting where to place the sampling locations within a dwelling and city was the targeted number of sampling locations per substrate (Table 4-2). The targeted number of sampling locations for each substrate in Denver and Philadelphia was predetermined during the design stage of this study. These target numbers dictated how many sampling location templates were drawn on each of the six substrates. However, the targeted numbers were not obtained due to factors such as the absence of a particular substrate or the inaccessibility of the painted surface (Table 4-1). Table 6-2 provides the actual number of sampling locations per substrate for each address. Table 6-3 provides the targeted numbers and summarizes the numbers that were actually obtained.

Table 6-1. Individual XRF Instrument Usage for All Sampling Locations.

XRF TYPE	XRF CODE No.[a]	CITY	1993 TESTING DATES (mm/dd)	NUMBER OF UNITS OR HOUSES	TOTAL NUMBER OF LOCATIONS	STANDARD READING TOTAL
Lead Analyzer	01	Denver	08/04-08/14	15[b]	1,031	1,190
		Philadelphia	10/11-10/21			
	02	Philadelphia	10/21-10/25	3[c]	159	
MAP-3	13	Louisville	03/31-04/01	4	100[d]	2,380
	10	Denver	08/04-08/21	18	1,190	
		Philadelphia	10/06-10/25			
	11	Denver	08/04-08/21	10	750	
	12[e]	Philadelphia	10/06-10/25	8	440	
Microlead I	24	Louisville	03/31-04/01	4	100	2,480
	20	Denver	08/07-08/18	18	1,190	
		Philadelphia	10/11-10/25			
	21	Denver	08/09-08/10	6	430	
		Denver	08/16			
		Denver	08/18-08/19			
		Philadelphia	10/15			
	22	Denver	08/11-08/14	5	375	
		Denver	08/17			
	23	Philadelphia	10/11-10/14	7	385	
	23	Philadelphia	10/18-10/25			
X-MET 880	51	Louisville	03/29-03/30	4	100[d]	1,190
	50	Denver	08/04-08/14	18	1,190	
		Philadelphia	10/11-10/25			
XK-3	31	Louisville	03/29-03/30	4	100	2,480
		Denver	08/06-08/16	10	750	
	30	Denver	08/05-08/16	18	1,190	
		Philadelphia	10/11-10/25			
	32	Philadelphia	10/11-10/25	8	440	
XL	40	Denver	08/10-08/14	5	375	1,190
	41	Denver	08/16-08/20	5	375	
	42	Philadelphia	10/11-10/25	8	440	

[a] XRF code numbers are used to distinguish individual instruments.
[b] Includes the first 6 sampling locations tested on October 21.
[c] Excludes 6 sampling locations from the unit tested on October 21.
[d] Not included in the Standard Reading Total.
[e] The instruments with XRF code nos. 11 and 12 were the same instrument, however, factory maintenance was performed on this instrument after testing in Denver was completed and before testing in Philadelphia began.

Table 6-2. Number of Sampling locations per Substrate by Dwelling.

CITY	DWELLING	YEAR BUILT	SAMPLING LOCATIONS PER SUBSTRATE					
			Brick	Concrete	Drywall	Metal	Plaster	Wood
Louisville	1	1937	0	4	4	17	9	16
	2	1937	0	4	7	11	11	17
Denver	1	1943	20	4	4	4	15	28
	2	1948	0	1	0	10	20	44
	3	1952	0	2	8	2	22	41
	4	1905	0	15	25	3	10	22
	5	1949	3	0	20	6	0	46
	6	1948	0	6	18	8	0	43
	7	1952	0	10	16	12	0	37
	8	1890	21	15	1	6	13	19
	9	1949	21	1	0	9	21	23
	10	1947	16	44	13	2	0	0
Philadelphia	1	1942	2	15	2	16	11	9
	2	1942	2	15	2	12	12	12
	3	1942	0	15	2	16	13	9
	4	1942	0	15	2	19	13	6
	5	1942	2	15	0	16	18	4
	6	1942	2	15	0	16	18	4
	7	1942	2	15	0	16	18	4
	8	1942	2	15	0	16	18	4

6.1.2 Control Data Description

Control data refers to XRF readings taken on a standardized block of substrate, called a "control block", placed on top of a twelve-inch styrofoam cube. The control blocks and styrofoam cube were provided to the operators of the XRF instruments at

each dwelling[2].

[2]Dwelling: In Louisville, a dwelling is defined as a building within which two units were tested. In Denver, a dwelling is defined as a house. In Philadelphia, a dwelling is defined as a unit.

Table 6-3. Target and Actual Number of Sampling locations per Substrate by City.

CITY		BRICK	CONCRETE	DRYWALL	METAL	PLASTER	WOOD
Louisville	Target	na	na	na	na	na	na
	Actual	0	8	11	28	20	33
Denver	Target	80	170	170	60	100	170
	Actual	81	98	105	62	101	303
Philadelphia	Target	8	48	8	127	120	128
	Actual	12	120	8	127	121	52
Total	Target	88	238	198	207	240	318
	Actual	93	226	124	217	242	388

The control blocks were made of unpainted substrate materials and were composed of brick, concrete, drywall, metal, plaster, or wood. A control reading is an XRF reading taken on a control block placed on top of the styrofoam cube.

Three basic types of control readings are common to both the pilot and full studies:

- Beginning Control
- Continuing Control
- Ending Control

In Louisville, two dwellings were tested, in Denver there were ten dwellings, and in Philadelphia there were eight dwellings. Beginning and ending control readings were made before and after testing in each dwelling, respectively. Beginning and ending control readings were made on all six substrates. The number and duration of the nominal reading times used in the pilot study differed from those used in the full study as did the way NIST SRM films were used. For the full study, the yellow (3.53 mg/cm^2) and red (1.02 mg/cm^2) NIST SRM films were separately placed on the control blocks prior to taking XRF readings and, also, readings were taken on the bare (uncovered) control blocks. For the pilot study, the red (1.02 mg/cm^2) NIST SRM film was placed on the control blocks prior to taking XRF readings and, for concrete control blocks only, another set of readings were taken with the yellow (3.53 mg/cm^2)

NIST SRM film covering the control block. Whenever beginning or ending control readings were taken, a series of readings, defined as the design-specified number of control readings per control block, were taken. Table 6-4 shows the design-specified number of readings per control block along with the reading duration time and the number of readings per surface type, for both the pilot and full studies.

Continuing control readings were done after completing XRF testing on one substrate and prior to testing on the next substrate. Whenever continuing control readings were taken, readings were first taken on the substrate that corresponded to the substrate of the last sampling location. Next, readings were taken on the substrate that corresponded to the substrate of the next sampling location. How many and which control blocks were used depended upon the order in which the testing was done on the substrates and which substrates were present in a dwelling. Therefore, it is necessary to describe how substrates were tested in the pilot and full studies.

Tables 6-5 and 6-6 provide the number and order in which testing was done on the substrates for the pilot study and the full study, respectively. The order of testing on substrates changed after the pilot study was completed. In Louisville, brick was missing, so only five of the six substrates were present. For the pilot study, the substrates, listed in order of testing, were: wood, drywall, plaster, concrete, and metal. Continuing control readings were made, once before and once after testing on wood, drywall, plaster, concrete, and metal so that continuing control readings were taken ten times as shown in Table 6-5.

For the full study, the order of substrate testing was: metal, wood, brick, drywall, concrete, and plaster. In contrast to the pilot study, the substrate on which testing was to begin varied. Also, some dwellings in the full study were missing one or two substrates. This information is provided in Table 6-6. Differences in how substrates were tested in the pilot study and the full study may be observed by comparing differences between Tables 6-5 and 6-6. The order of testing is different as is the substrate on which testing began; for the pilot study, it was always wood and for the full study, it varied, as stated above.

At the beginning of the day, continuing control readings

were taken before testing began on the control block that matched the substrate scheduled to be tested first. The next continuing control readings were taken after testing of all sampling

Table 6-4. Beginning and Ending Control Block Data Descriptions for Each XRF Instrument Type.

XRF TYPE	PILOT or FULL STUDY	NUMBER OF READINGS/ CONTROL BLOCK	NOMINAL READING TIME IN SECONDS	NUMBER OF READINGS/ SURFACE TYPE
Lead Analyzer	pilot	na	na	na
	full	9	15	3/yellow NIST SRM 3/red NIST SRM 3/bare
MAP-3	pilot	3 or 6[a]	60	3/red NIST SRM 3/yellow NIST SRM[a]
	full	9	15	3/yellow NIST SRM 3/red NIST SRM 3/bare
Microlead I[b]	pilot	9 or 18[a]	15	9/red NIST SRM 9/yellow NIST SRM[a]
	full	9	15	3/yellow NIST SRM 3/red NIST SRM 3/bare
X-MET 880	pilot	9 or 18[a]	15	9/red NIST SRM 9/yellow NIST SRM[a]
	full	9	15	3/yellow NIST SRM 3/red NIST SRM 3/bare
XK-3	pilot	9 or 18[a]	15	9/red NIST SRM 9/yellow NIST SRM[a]
	full	9	15	3/yellow NIST SRM 3/red NIST SRM 3/bare
XL	pilot	na	na	na
	full	9	15	3/yellow NIST SRM 3/red NIST SRM 3/bare

na not applicable, no reading definition.
[a] Additional readings were taken if the control block was concrete.
[b] Readings were reported as running averages.

locations of a given substrate had been completed. Subsequent continuing control readings were first taken on the control block that matched the previously tested substrate followed by continuing control readings taken on the control block that matched the substrate scheduled to be tested next. The last

continuing control readings at a dwelling unit were taken on the control block that matched the substrate that was tested last and were taken before the ending control readings were taken.

Table 6-5. Continuing Control Reading Summary for the Pilot Study (Louisville).

CITY	DWELLING	ORDER OF SUBSTRATES						NUMBER OF SUBSTRATE CHANGES	NO. OF TIMES READINGS TAKEN
		W	D	P	B	C	M		
Louisville	1	x	•	•		•	•	4	10
	2	x	•	•		•	•	4	10

• Indicates substrates present in this dwelling.
x Indicates the substrate was present in this dwelling and testing began with this substrate.

Whenever continuing control readings were taken, a series of readings, defined as the design specified number of continuing control readings per control block, were taken. Table 6-7 shows the design-specified number of readings per control block along with the reading duration time and the number of readings per surface type, for both the pilot and full studies and all XRF instruments.

As an example, dwelling 2 in Denver had sampling locations composed of wood followed by concrete, plaster, and metal. Thus, eight series of continuing control readings were taken in this dwelling as follows. First, a series of continuing control readings were taken on the wood control block after beginning control readings were completed but before testing began on the substrate scheduled to be tested first, which was wood. The next series of continuing control readings were again taken on the wood control block after all testing on the wood substrates in the dwelling was completed.

A series of continuing control readings were taken next on the concrete control block, before testing on any concrete sampling locations began. Similarly, another series of readings were taken on the concrete control block followed by a series on the plaster control block. The sixth series of readings were taken on the plaster control block followed by a series on the metal control block. Finally, after all testing on the sampling locations had been completed in the dwelling, the last series of continuing control readings were taken on the metal control block since the last sampling location that was tested was metal. All testing in the dwelling was completed when the ending control

readings were taken.

Table 6-6. Continuing Control Reading Summary for the Full Study (Denver and Philadelphia).

CITY	DWELLING	ORDER OF SUBSTRATES						NUMBER OF SUBSTRATE CHANGES	NO. OF TIMES READINGS TAKEN
		M	D	W	B	C	P		
Denver	1	•	•	•	x	•	•	5	12
	2	•		x		•	•	3	8
	3	•	x	•		•	•	4	10
	4	•	•	•		x	•	4	10
	5	x	•	•	•			3	8
	6	x	•	•		•		3	8
	7	•	•	x		•		3	8
	8	•		•	x	•	•	4	10
	9	x		•	•	•	•	4	10
	10	•	x		•	•		3	8
Philadelphia	1	x	•	•	•	•	•	5	12
	2	•	•	x	•	•	•	5	12
	3	•	x	•		•	•	4	10
	4	•	•	•		x	•	4	10
	5	•		•	•	•	x	4	10
	6	x		•	•	•	•	4	10
	7	•		•	x	•	•	4	10
	8	•		•	•	x	•	4	10

• Indicates substrates present in this dwelling.
x Indicates the substrate was present in this dwelling and testing began with this substrate.

6.1.3 Special Data Description

Special data refers to certain XRF readings taken according to a data collection protocol other than the standard data collection protocol. "Special" readings differ from standard readings because they were taken on the bare substrate without any NIST SRM film covering it or were taken with a reading time greater than nominal 15-second readings. "Special" data were used to examine bias relative to reading time and surface

coating. "Special" data were analyzed, for example, to determine if a three 15-second reading average or a single nominal

Table 6-7. Continuing Control Block Data Descriptions for Each XRF Instrument Type.

XRF TYPE	PILOT OR FULL STUDY	NUMBER OF READINGS/ CONTROL BLOCK	NOMINAL READING TIME IN SECONDS	NUMBER OF READINGS/ SURFACE TYPE
Lead Analyzer	pilot	na	na	na
	full	9	15	3/yellow NIST SRM 3/red NIST SRM 3/bare
MAP-3	pilot	1 or 2[a]	60	1/red NIST SRM 1/yellow NIST SRM[a]
	full	9	15	3/yellow NIST SRM 3/red NIST SRM 3/bare
Microlead I[b]	pilot	3 or 6[a]	15	3/red NIST SRM 3/yellow NIST SRM[a]
	full	9	15	3/yellow NIST SRM 3/red NIST SRM 3/bare
X-MET 880	pilot	3 or 6[a]	15	3/red NIST SRM 3/yellow NIST SRM[a]
	full	9	15	3/yellow NIST SRM 3/red NIST SRM 3/bare
XK-3	pilot	3 or 6[a]	15	3/red NIST SRM 3/yellow NIST SRM[a]
	full	9	15	3/yellow NIST SRM 3/red NIST SRM 3/bare
XL	pilot	na	na	na
	full	9	15	3/yellow NIST SRM 3/red NIST SRM 3/bare

na not applicable, no reading definition.
[a] Additional readings were taken if the control block was concrete.
[b] Readings are reported as running averages.

60-second reading had the smaller bias or if the bias decreased when readings were taken on bare substrate without a NIST SRM

film covering.

The two types of "special" data are "special" readings and "special-special" readings. These readings were taken at sampling locations designated as "special" and "special-special" locations. Twenty-five percent of the sampling locations within a dwelling, rounded up to the nearest whole number, were randomly selected and designated as "special" locations. Next, twenty-five percent of the "special" locations within a dwelling, again rounded up to the nearest whole number, were randomly selected and designated as "special-special" locations. Table 6-8 provides the number of sampling locations, "special" locations, and "special-special" locations per dwelling. For the pilot and full studies, a total of 328 sampling locations were designated as special.

Once testing began, at each sampling location designated as a "special" location, standard data were first collected followed by "special" data collection. Likewise, at each sampling location designated a "special-special" location, standard data were first collected followed by "special" data collection followed by "special-special" data collection. The data collection protocol for "special" and "special-special" locations depended on the XRF instrument type and whether data were being collected for the pilot study or the full study. The pilot study "special" data collection protocol differs from that of the full study in how many XRF readings were taken and the duration of each XRF reading.

Also, the MAP-3 "special" data collection protocol differed from the other five XRF instrument types. These differences are shown in Table 6-9 which provides a summary of "special" data by XRF instrument type.

Not shown in Table 6-9 are the dates of "special" data collection. For the pilot study, if any additional readings were to be made such as "special" readings, the readings were made immediately following the standard readings. This procedure was used by all four participating XRF instruments. The same procedure was used in the full study for five of the six XRF instrument types. The exception was the MAP-3. The MAP-3 was used to collect "special" data on days other than those when standard data was collected by the MAP-3. "Special" data were collected with the MAP-3 instruments at houses in Denver on

August 17 through 21 and in Philadelphia on October 6 through October 11, 1993.

Another difference for the MAP-3 was the additional "special-special" readings, collected only by the MAP-3. At sampling locations designated "special-special" locations, the

Table 6-8. The Number of Dwellings, the Number of Sampling Locations, Special, and "Special-special" Locations per Dwelling, and the Total Number of "Special" Locations per City in Louisville, Denver, and Philadelphia.

City	No. of Dwellings[a]	No. of Sampling Locations	No. of "Special" Locations	No. of "Special-special" Locations	Total No. of "Special" Locations
Louisville	2	50	13	na	26
Denver	10	75	19	5	190
Philadelphia	8	55	14	4	112

na not applicable, no reading definition.
[a] In Louisville, a dwelling is defined to be a building within which two units were tested. In Denver, a dwelling is defined to be a house. In Philadelphia, a dwelling is defined to be a unit.

MAP-3 instruments took two additional readings at that same sampling location. "Special-special" data consisted of one nominal 240-second reading taken on the painted surface of the sampling location and one nominal 240-second reading taken on the bare substrate covered by the red (1.02 mg/cm^2) NIST SRM film.

6.1.4 Non-standard Data Description

Non-standard data refers to data collected using non-standard data collection procedures. Non-standard data are XRF readings that were taken for the pilot study that are not directly comparable with data collected in the full study and consist of too few data to make parameter estimations using model-based procedures. There are four types of non-standard data as listed below:

- XRF readings made by the X-MET 880 in Louisville.
- XRF readings made by the MAP-3 in Louisville.
- Variability XRF readings taken on the sampling locations that followed a change in substrate in Louisville. (See chapter 3, section 5.2.4 for a detailed explanation of variability XRF readings).
- XRF readings taken on the bare concrete substrates covered by the yellow (3.53 mg/cm^2) NIST SRM film in Louisville.

The MAP-3 data that were collected in Louisville are

non-standard readings because, in accordance with the pilot study data collection protocol, nominal 60-second readings were taken at each sampling location instead of nominal 15-second readings.

Table 6-9. Special Data Descriptions for Each XRF Instrument Type.

XRF Type	Pilot or Full Study	Number of Readings/ Sampling Location	Nominal Reading Time in Seconds	Number of Readings/ Surface Type
Lead Analyzer	pilot	na	na	na
	full	3	15	3/bare
MAP-3	pilot	6	15	3/paint 3/red NIST SRM
	full	3	60[a]	1/paint 1/red NIST SRM 1/bare
Microlead I[b]	pilot	8	15	4/paint 4/red NIST SRM
	full	3	15	3/bare
X-MET 880	pilot	2	60	1/paint 1/red NIST SRM
	full	3	15	3/bare
XK-3	pilot	8	15	4/paint 4/red NIST SRM
	full	3	15	3/bare
XL	pilot	na	na	na
	full	3	15	3/bare

na not applicable, no reading definition.
[a] References 60-second readings as "confirm mode".
[b] Readings are reported as running averages.

Since standard data are nominal 15-second readings, these data are classified non-standard.

The X-MET 880 data that were collected in Louisville are non-standard readings because of observable differences between the instrument used in the pilot study and the instrument used in the full study and also because of the small sample size for the pilot study. Comparisons of the X-MET 880 data collected in Louisville with data collected in the full study indicated that the distributional properties of the two data sets were different. This suggests analyzing the data from the two X-MET 880 instruments separately. But due to the small sample sizes in the pilot study, particularly the small number of sampling locations for most substrates, these data are classified

non-standard. Sufficient data, however, were collected from locations composed of metal and wood by the X-MET 880 in Louisville to allow limited analyses to be performed. These data were analyzed separately from the Denver and Philadelphia data, and are presented in the subsequent sections that contain analysis of the X-MET 880 data.

Also classified as non-standard data are the so-called "variability" XRF readings taken on sampling locations following a change in substrate. In Louisville, all four participating XRF instruments were used to take five additional sets of readings on the first sampling location after a change in substrate occurred using the same data collection protocol as was used when taking the first set of readings at that same location. Since these data were not collected in the full study, these data were classified non-standard.

The last type of data defined as non-standard are the three additional nominal 15-second readings taken over bare concrete covered with the yellow (3.53 mg/cm^2) NIST SRM film. These data were collected at only eight sampling locations in the pilot study. Since these data were not collected in the full study, and because of the small sample size, these data were classified non-standard.

6.1.5 Data Description Summary and Analysis Dataset Descriptions

The XRF data are classified into one of the four categories given below:

- Standard
- Control
- Special
- Non-standard

A set of standard readings consists of six nominal 15-second readings taken at the same sampling location, three of which were taken on the painted surface and three on the bare substrate covered by the red (1.02 mg/cm^2) NIST SRM film. These will be referred to as the first, second, and third standard paint readings and the first, second, and third red NIST SRM film readings. Control data consists of XRF readings taken on six standardized substrates at each dwelling. "Special" data are XRF

readings that were taken at randomly selected sampling locations using a data collection protocol different from what was used to collect standard readings. "Special" readings are different from standard readings because either they were taken on the bare substrate of the sampling location without the NIST SRM film or they were taken with a nominal reading time greater than 15 seconds. Non-standard data are XRF readings that were taken for the pilot study in Louisville that are not directly comparable with data collected in Denver and Philadelphia. Sections 6.1.1 through 6.1.4 above provide detailed descriptions of the standard, control, special, and non-standard data categories, respectively.

Summary statistics will be provided in section 6.2 for all of the data (XRF readings) that were collected in both the pilot study and the full study.

6.1.5.1 Analysis Dataset Descriptions

6.1.5.1.1 XRF Instrument Operators

Nineteen individual distinct XRF instruments were used in this study in Louisville, Denver, and Philadelphia. Recall that XRF data were collected in Louisville during March and April, 1993, in Denver in August, 1993 and in Philadelphia in October, 1993. Table 6-10 summarizes the individual XRF instrument usage for each combination of individual operator and XRF instrument. Nineteen XRF instruments were operated by fourteen individuals who are coded with the letters "A" through "N".

6.1.5.1.2 XRF Data Analysis Variables

Most of the results described in this chapter are from the analysis of standard data and control data. The primary emphasis of this report is the analysis of the first XRF reading taken on the painted surface of each sampling location. However, to fully understand the behavior of the XRF instruments many other variables were analyzed and are presented in this report. They are listed and defined below.

<u>First Paint Reading</u> - the first standard paint reading.

<u>Paint Average</u> - the arithmetic mean of the first, second, and third standard readings taken on the paint computed for each XRF

instrument at each sampling location.

Red NIST SRM Average - the arithmetic mean of the standard first, second, and third red NIST SRM readings. Standard red NIST SRM readings were taken at each sampling location on the bare substrate area covered by the red NIST SRM film. A red NIST SRM average was computed at each sampling location for each XRF instrument.

Table 6-10. Individual XRF Instrument Operator by Instrument Usage in Louisville, Denver, and Philadelphia.

XRF Type	XRF Code No.[a]	City	1993 Testing Dates (mm/dd)	Operator Code Letter[b]
Lead Analyzer	01	Denver	08/04-08/14	A
		Philadelphia	10/11-10/21	
	02	Philadelphia	10/21-10/25	A
MAP-3	13	Louisville	03/31-04/01	B
	10	Denver	08/04-08/21	C
		Philadelphia	10/06-10/25	
	11	Denver	08/04-08/21	D
	12	Philadelphia	10/06-10/25	E
Microlead I	24	Louisville	03/31-04/01	F
	20	Denver	08/07-08/18	G
		Philadelphia	10/11-10/25	
	21	Denver	08/09-08/10	E
		Denver	08/16	
		Denver	08/18-08/19	
	22	Denver	08/11-08/14	E
		Denver	08/17	
	23	Philadelphia	10/11-10/14	H
	21	Philadelphia	10/15	
	23	Philadelphia	10/18-10/25	
X-MET 880	51	Louisville	03/29-03/30	I
	50	Denver	08/04-08/09	J
		Denver	08/10-08/14	K
		Philadelphia	10/11-10/25	
XK-3	31	Louisville	03/29-03/30	L
		Denver	08/06-08/16	
	30	Denver	08/05-08/16	M
		Philadelphia	10/11-10/25	
	32	Philadelphia	10/11-10/24	N
		Philadelphia	10/25	M
XL	40	Denver	08/10-08/14	J
	41	Denver	08/16-08/17	J
		Denver	08/20	

		Denver	08/18-08/19	K
	42	Philadelphia	10/11-10/25	J

[a] XRF code numbers are used to distinguish individual instruments.
[b] The XRF instrument operators are coded with the letters "A" through "N".

Control Average - for each substrate, the arithmetic mean of all beginning and ending control readings taken within a dwelling. A dwelling contains the centralized location where control readings were taken. In Louisville, a dwelling was a building within which two units were tested. In Denver, a dwelling was a house and in Philadelphia, a dwelling was a unit. A control average was computed for all XRF instruments in each dwelling.

Red NIST SRM Dwelling Average - for each substrate, the arithmetic mean of all of the first, second, and third standard red NIST SRM readings taken within a dwelling. For example, for Denver, a red NIST SRM dwelling average would be computed from 225 readings (75 sampling locations per dwelling times three standard red NIST SRM readings per sampling location). A red NIST SRM dwelling average was computed for all XRF instruments in each dwelling.

First Paint Control Corrected - the first standard paint reading corrected for substrate bias. The correction value was the appropriate control average minus 1.02 mg/cm^2. The value 1.02 mg/cm^2 is subtracted from the control average to compensate for the red (1.02 mg/cm^2) NIST SRM film that was placed over the control block prior to taking readings on the control block. An appropriate control average is computed from readings taken on the same substrate and in the same dwelling as the first standard paint reading. The subtrahend (the control average minus 1.02 mg/cm^2) corrects the first standard paint reading for substrate bias. First paint control corrected values were computed for all XRF instruments for each sampling location. The values were computed as follows.

$$\left\{\begin{matrix}\textit{first paint}\\ \textit{control corrected}\end{matrix}\right\} = \left\{\begin{matrix}\textit{first standard}\\ \textit{paint reading}\end{matrix}\right\} - \left\{\begin{matrix}\textit{control}\\ \textit{average}\end{matrix} - 1.02\right\}$$

Average Paint Control Corrected - the paint average corrected for substrate bias. The correction value was the appropriate control average minus 1.02 mg/cm^2. The value 1.02 mg/cm^2 is subtracted from the control average to compensate for the red (1.02 mg/cm^2) NIST SRM film that was placed over the control block prior to taking readings on the control block. An appropriate control

average is computed from readings taken on the same substrate and in the same dwelling as the paint average. The subtrahend (the control average minus 1.02 mg/cm^2) corrects the paint average for substrate bias. Average paint control corrected values were computed for all XRF instruments for each sampling location. The values were computed as follows.

$$\left\{\begin{matrix}\textit{paint average}\\ \textit{control corrected}\end{matrix}\right\} = \left\{\begin{matrix}\textit{paint}\\ \textit{average}\end{matrix}\right\} - \left\{\begin{matrix}\textit{control}\\ \textit{average}\end{matrix} - 1.02\right\}$$

<u>First Paint Fully Corrected</u> - the first standard paint reading corrected for substrate bias using an alternative correction value. The correction value was the standard red NIST SRM average from the same sampling location minus 1.02 mg/cm^2. The value 1.02 mg/cm^2 is subtracted from the standard red NIST SRM average to compensate for the red (1.02 mg/cm^2) NIST SRM film that was placed over the bare substrate area of the sampling location prior to taking the readings. The subtrahend (the red NIST SRM average minus 1.02 mg/cm^2) corrects the first paint reading for substrate bias. First paint fully corrected values were computed for all XRF instruments for each sampling location. The values were computed as follows.

$$\left\{\begin{matrix}\textit{first paint}\\ \textit{fully corrected}\end{matrix}\right\} = \left\{\begin{matrix}\textit{first standard}\\ \textit{paint reading}\end{matrix}\right\} - \left\{\begin{matrix}\textit{red NIST}\\ \textit{SRM average}\end{matrix} - 1.02\right\}$$

<u>Paint Average Fully Corrected</u> - the paint average corrected for substrate bias using the correction value described in the previous paragraph. That is, the correction value was the standard red NIST SRM average from the same sampling location minus 1.02 mg/cm^2. Paint average fully corrected values were computed for all XRF instruments for each sampling location. The values were computed as follows.

$$\left\{\begin{matrix}\textit{paint average}\\ \textit{fully corrected}\end{matrix}\right\} = \left\{\begin{matrix}\textit{paint}\\ \textit{average}\end{matrix}\right\} - \left\{\begin{matrix}\textit{red NIST}\\ \textit{SRM average}\end{matrix} - 1.02\right\}$$

<u>First Paint Red NIST SRM Average Corrected</u> - the first standard paint reading corrected for substrate bias using a third method. The correction value was the appropriate red NIST SRM dwelling average minus 1.02 mg/cm^2. The value 1.02 mg/cm^2 is subtracted from the red NIST SRM dwelling average to compensate for the red (1.02 mg/cm^2) NIST SRM film that was placed over the bare

substrate area of the sampling location prior to taking the readings. An appropriate red NIST SRM dwelling average is computed from all red NIST SRM readings taken in the same dwelling as the standard first paint reading. The subtrahend (the red NIST SRM dwelling average minus 1.02 mg/cm^2) corrects the first paint reading for substrate bias. First paint red NIST SRM average corrected values were computed for all XRF instruments for each sampling location. The values were computed as follows.

$$\left\{\begin{matrix}\textit{first paint}\\ \textit{red NIST}\\ \textit{average corrected}\end{matrix}\right\} = \left\{\begin{matrix}\textit{first standard}\\ \textit{paint reading}\end{matrix}\right\} - \left\{\begin{matrix}\textit{red NIST SRM}\\ \textit{dwelling average}\end{matrix} - 1.02\right\}$$

Paint Average Red NIST SRM Average Corrected - the paint average corrected for substrate bias using the correction value described in the previous paragraph. That is, the correction value was the appropriate red NIST SRM dwelling average minus 1.02 mg/cm^2. Paint average red NIST SRM average corrected values were computed for all XRF instruments for each sampling location. The values were computed as follows.

$$\left\{\begin{matrix} \textit{paint average} \\ \textit{red NIST} \\ \textit{average corrected} \end{matrix}\right\} = \left\{\begin{matrix} \textit{paint} \\ \textit{average} \end{matrix}\right\} - \left\{\begin{matrix} \textit{red NIST SRM} \\ \textit{dwelling average} \end{matrix} - 1.02\right\}$$

6.2 DESCRIPTIVE STATISTICS FOR STANDARD AND CONTROL DATA

The first section of this chapter described XRF data and categorized the data as standard and control data. Summary statistics that include the number of readings, mean, median, maximum, minimum, 25th percentile, and 75th percentile are presented in this section for each data category and address the following study objectives:

- to characterize the performance (precision and accuracy) or portable XRF instruments under field conditions
- to evaluate the effect on XRF performance of interference from material (the substrate) underlying the paint
- to investigate XRF measurements that were very different than their corresponding laboratory results
- to evaluate field quality assurance and control methods.

Due to the large number of tables presented in this section, most tables are not intermingled with text, but instead, tables referenced in a given subsection that provide summary statistics are generally placed after the text for that subsection.

The XRF instrument data were classified into eight XRF categories for this analysis. The categories are the six XRF instrument types plus two additional categories. The six XRF instrument types are: Lead Analyzer, MAP-3, Microlead I (ML I), X-MET 880, XK-3, and XL. The two additional categories are the L-shell readings provided by the Lead Analyzer and the MAP-3 instruments. The Lead Analyzer and the MAP-3 instruments are capable of reporting both K-shell and L-shell readings. The distributional properties or characteristics of the K-shell readings and L-shell readings are very different as will be shown

in subsequent sections. Therefore, for these analyses, eight distinct XRF classifications were analyzed as if they were separate XRF instruments as follows:

- Lead Analyzer K-shell
- Lead Analyzer L-shell
- MAP-3 K-shell
- MAP-3 L-shell
- Microlead I revision 4 (K-shell)
- X-MET 880 (L-shell)
- XK-3 (K-shell)
- XL (L-Shell)

The statistics presented in this section apply only to the set of sampling locations tested in this study. A set of locations with significantly different lead levels than the tested locations might behave differently, even if the same instruments were used.

Data for the Lead Analyzer are complete (i.e., no missing data). The other five XRF instruments have a combined total of 62 missing readings at various sampling locations. A brief summary of the missing readings for each XRF classification is provided in Table 6-11. The missing data values are due to one of the following two reasons:

- XRF instrument's probe could not access sampling location, or
- the XRF instrument was unable to compute and display a reading taken on a metal substrate.

6.2.1 Summary Statistics for Standard Data

This section provides summary statistics for the standard data that were collected. Tables 6-12 through 6-14 provide summary statistics for the first, second and third standard paint readings and also summary statistics for the 1,290 primary ICP measurements in mg/cm^2 units. Tables 6-15 though 6-17 provide summary statistics for the first, second, and third standard red (1.02 mg/cm^2) NIST SRM readings, respectively. These six readings were defined as standard data in the first section of this chapter.

The "Number of Readings" given in Table 6-13 is the same as

the corresponding value given in Table 6-1 in the first section

Table 6-11. Missing First Standard Paint Readings.

XRF DEVICE	# OF MISSING READINGS	REASON
MAP-3	19	Inaccessible sampling location.
Microlead I	5	Inaccessible sampling location.
X-MET 880	2	Inaccessible sampling location.
	14	XRF unable to read on bare metal.
XK-3	2	Inaccessible sampling location.
XL	1	Inaccessible sampling location.

of this chapter under the "Standard Reading Total" column, except for missing data shown in Table 6-11. Out of the total of 1,290 sampling locations in the study, 100 sampling locations were in Louisville, 750 sampling locations were in Denver, and 440 sampling locations were in Philadelphia. Some "Number of Readings" values in the tables are greater than 1,290 because two each of the MAP-3, Microlead I, and XK-3 instruments were used in Denver and Philadelphia. By contrast, only one reading was taken at each sampling location with the Lead Analyzer, X-MET 880, and XL.

For comparison purposes, summary statistics for all of the 1,290 primary ICP measurements in mg/cm^2 units are provided in Tables 6-12 through 6-17. If an XRF instrument were used once at each of the 1,290 sample locations, then the mean of the XRF results and the laboratory ICP mean would estimate the same true level of lead. However, none of the instruments provided a single set of results at all 1,290 sampling locations. For example, the Lead Analyzer provided results for Denver and Philadelphia only. Nevertheless, the laboratory ICP results are useful as a relative guide for examining instrument behavior.

The mean of the 1,290 ICP measurements was 1.171 mg/cm^2. The largest XRF reading mean was 1.585 mg/cm^2 (the second paint reading average computed from readings taken by the XK-3) which was 35% greater than the laboratory mean. Similarly, the smallest XRF reading mean was 0.114 mg/cm^2 (third paint reading mean of the Lead Analyzer L-shell) which was 90% less than the laboratory mean. Comparisons of Tables 6-12, 6-13, and 6-14 show small differences between the first, second, and third standard

paint readings. It is clear that the K-shell readings on paint were much higher than the L-shell readings, although the medians for the XL were comparable to those for the Lead Analyzer and MAP-3 K-shell instruments.

Results for the standard red NIST SRM readings are shown in Tables 6-15, 6-16, and 6-17. Red NIST SRM K-shell arithmetic averages were consistently higher than those taken by the L-shell instruments except for the MAP-3 which had K-shell and L-shell readings that were very similar. On the red NIST SRM, the L-shell readings were on average very close to the true lead level (1.02 mg/cm^2). K-shell arithmetic averages were all greater than the red NIST SRM value of 1.02 mg/cm^2.

The Microlead I in these tables stands out as consistently having the largest maximum and smallest minimum. Three of these six maximum and minimum readings are attributable to one Microlead I instrument: the two maximum values in Table 6-15 (27.500 mg/cm^2) and Table 6-17 (48.500 mg/cm^2) and the minimum value in Table 6-16 (-21.100 mg/cm^2) were made by the same instrument at the same brick sampling location in Denver.

Also shown in the tables are results that illustrate how the XRF instruments present their data. Two XRF instrument types truncate readings and four XRF instrument types report negative readings. The XK-3 and the XL instruments truncated readings at fixed reading levels; the XK-3 truncated at 10.0 mg/cm^2 and the XL truncated at 5.0 mg/cm^2. The XL truncated negative readings at 0.00 mg/cm^2. The maximum values in Tables 6-12 through 6-14 show effects of truncation for the XK-3 and XL, since the maximum reported result from the XK-3 was 10.0 mg/cm^2 and from the XL was 5.0 mg/cm^2. The minimum values in these tables confirm that four instruments display negative readings. The minimum values for the Lead Analyzer, MAP-3, Microlead I, and XK-3 instruments were all negative. The minimum value reported by the XL was 0.00 cm/mg^2. The X-MET 880 had a positive minimum.

Table 6-12. Summary Statistics of Lead Measured in mg/cm^2 Units of the First Paint Reading (Standard Data) for All XRF Instrument Types and the Laboratory Results From All 1,290 Sampling Locations.

DATA SOURCE	NUMBER OF READINGS	ARITHMETIC MEAN	MAXIMUM	MINIMUM	25th PERCENTILE	MEDIAN	75TH PERCENTILE
Lead Analyzer K-shell	1,190	1.020	22.500	-0.240	0.050	0.200	0.600
Lead Analyzer L-shell	1,190	0.114	3.240	-0.053	0.004	0.036	0.097
MAP-3 K-shell	2,367	0.903	30.437	-4.439	-0.348	0.184	0.899
MAP-3 L-shell	2,367	0.152	7.953	-1.275	-0.119	-0.049	0.137
Microlead I	2,475	1.327	22.800	-2.600	0.000	0.500	1.400
X-MET 880	1,174	0.156	3.649	0.010	0.040	0.063	0.130
XK-3	2,478	1.571	10.000	-1.200	0.300	1.000	2.000
XL	1,189	0.408	5.000	0.000	0.000	0.100	0.300
Laboratory	1,290	1.171	37.290	0.000	0.028	0.196	0.619

Table 6-13. Summary Statistics of Lead Measured in mg/cm^2 Units of the Second Paint Reading (Standard Data) for All XRF Instrument Types and the Laboratory Results From All 1,290 Sampling Locations.

DATA SOURCE	NUMBER OF READINGS	ARITHMETIC MEAN	MAXIMUM	MINIMUM	25th PERCENTILE	MEDIAN	75TH PERCENTILE
Lead Analyzer K-shell	1,190	1.024	26.400	-0.300	0.040	0.200	0.700
Lead Analyzer L-shell	1,190	0.116	3.110	-0.052	0.004	0.036	0.096
MAP-3 K-shell	2,367	0.904	29.695	-3.619	-0.403	0.175	0.890
MAP-3 L-shell	2,367	0.156	7.835	-1.660	-0.119	-0.048	0.142
Microlead I	2,475	1.350	22.700	-7.400	0.000	0.500	1.400
X-MET 880	1,174	0.156	3.648	0.016	0.041	0.063	0.130
XK-3	2,478	1.585	10.000	-1.100	0.300	1.000	2.000
XL	1,189	0.409	5.000	0.000	0.000	0.100	0.300
Laboratory	1,290	1.171	37.290	0.000	0.028	0.196	0.619

Table 6-14. Summary Statistics of Lead Measured in mg/cm^2 Units of the Third Paint Reading (Standard Data) for All XRF Instrument Types and the Laboratory Results From All 1,290 Sampling Locations.

DATA SOURCE	NUMBER OF READINGS	ARITHMETIC MEAN	MAXIMUM	MINIMUM	25th PERCENTILE	MEDIAN	75TH PERCENTILE
Lead Analyzer K-shell	1,190	1.029	23.400	-0.280	0.040	0.200	0.700
Lead Analyzer L-shell	1,190	0.117	3.280	-0.052	0.004	0.036	0.098
MAP-3 K-shell	2,367	0.895	29.693	-3.285	-0.408	0.157	0.897
MAP-3 L-shell	2,367	0.156	7.527	-1.229	-0.119	-0.050	0.141
Microlead I	2,475	1.334	22.400	-2.300	0.000	0.500	1.400
X-MET 880	1,174	0.156	3.695	0.016	0.040	0.063	0.130
XK-3	2,478	1.580	10.000	-1.100	0.300	1.000	2.000
XL	1,189	0.411	5.000	0.000	0.100	0.200	0.400
Laboratory	1,290	1.171	37.290	0.000	0.028	0.196	0.619

Table 6-15. Summary Statistics of Lead Measured in mg/cm^2 Units of the First Red (1.02 mg/cm^2) NIST SRM Reading (Standard Data) for All XRF Instrument Types.

XRF TYPE	NUMBER OF READINGS	ARITHMETIC MEAN	MAXIMUM	MINIMUM	25th PERCENTILE	MEDIAN	75TH PERCENTILE
Lead Analyzer K-shell	1,190	1.107	4.400	0.000	0.900	1.100	1.200
Lead Analyzer L-shell	1,190	1.004	1.290	0.010	0.980	1.020	1.050
MAP-3 K-shell	2,369	1.217	5.513	-1.725	0.856	1.168	1.540
MAP-3 L-shell	2,369	1.200	1.941	-0.983	1.126	1.206	1.286
Microlead I	2,475	1.372	27.500	-4.900	0.800	1.200	1.700
X-MET 880	1,188	1.091	2.019	0.891	1.039	1.088	1.127
XK-3	2,478	1.680	5.500	0.000	1.200	1.600	2.100
XL	1,189	1.017	2.200	0.600	0.900	1.000	1.100

Table 6-16. Summary Statistics of Lead Measured in mg/cm^2 Units of the Second Red (1.02 mg/cm^2) NIST SRM Reading (Standard Data) for All XRF Instrument Types.

XRF TYPE	NUMBER OF READINGS	ARITHMETIC MEAN	MAXIMUM	MINIMUM	25th PERCENTILE	MEDIAN	75TH PERCENTILE
Lead Analyzer K-shell	1,190	1.107	3.800	0.030	0.900	1.100	1.200
Lead Analyzer L-shell	1,190	1.008	1.320	0.057	0.980	1.020	1.050
MAP-3 K-shell	2,369	1.195	5.686	-1.818	0.843	1.171	1.527
MAP-3 L-shell	2,369	1.212	1.950	-0.404	1.135	1.211	1.290
Microlead I	2,475	1.373	7.900	-21.100	0.800	1.200	1.700
X-MET 880	1,188	1.088	1.971	0.880	1.039	1.084	1.120
XK-3	2,478	1.693	5.700	0.000	1.200	1.600	2.100
XL	1,189	1.018	2.000	0.600	0.900	1.000	1.100

Table 6-17. Summary Statistics of Lead Measured in mg/cm^2 Units of the Third Red (1.02 mg/cm^2) NIST SRM Reading (Standard Data) for All XRF Instrument Types.

XRF TYPE	NUMBER OF READINGS	ARITHMETIC MEAN	MAXIMUM	MINIMUM	25th PERCENTILE	MEDIAN	75TH PERCENTILE
Lead Analyzer K-shell	1,190	1.095	4.100	-0.140	0.900	1.100	1.200
Lead Analyzer L-shell	1,190	1.006	1.300	-0.002	0.980	1.020	1.050
MAP-3 K-shell	2,368	1.203	5.581	-1.704	0.832	1.167	1.521
MAP-3 L-shell	2,368	1.209	2.644	-0.411	1.126	1.211	1.293
Microlead I	2,475	1.387	48.500	-4.400	0.900	1.200	1.700
X-MET 880	1,188	1.085	1.998	0.903	1.035	1.081	1.118
XK-3	2,478	1.679	6.100	-1.600	1.200	1.600	2.100
XL	1,187	1.017	1.500	0.600	0.900	1.000	1.100

6.2.2 Summary Statistics for Control Data

This section provides summary statistics for the control data that were collected by the eight classifications of XRF instruments. Tables 6-18 through 6-26 provide summary statistics for each of the nine control block readings taken on the red NIST SRM film in Louisville. Table 6-27 provides summary statistics for control block readings taken with the yellow NIST SRM film covering the concrete control blocks only, in Louisville. Except for the MAP-3, each XRF instrument took nine beginning and ending control block readings and three continuing control block readings on the red and yellow NIST SRM film. The MAP-3 took three beginning and ending control block readings and only one continuing control block reading.

Tables 6-28, 6-29, and 6-30 provide summary statistics for the first, second, and third beginning, continuing, and ending control block readings taken using the yellow NIST SRM film in the full study (Denver and Philadelphia), respectively. In these tables, the number of beginning and ending control block readings for the MAP-3 instruments is higher than for the Microlead I, XK-3, and other XRF instruments because of additional control block readings made on the "special" data collection dates. The number of continuing control block readings is not similarly higher because continuing control block readings were not made by the MAP-3 instruments on the "special" data collection dates. Tables 6-31, 6-32, and 6-33 provide summary statistics for the control block readings taken on the red NIST SRM film in the full study. Tables 6-34, 6-35, and 6-36 provide summary statistics for the control block readings taken on the bare control blocks in the full study.

The tables displaying control reading results for the full study (Tables 6-28 through 6-33) show a high degree of consistency between the first, second, and third readings in most cases. On the yellow NIST SRM with true lead level 3.53 mg/cm^2 the instruments show only small biases, with the exception of the Lead Analyzer L-shell (bias approximately -0.8 mg/cm^2), the Microlead I (bias approximately +0.5), and the XK-3 (bias approximately +0.9 mg/cm^2).

On the red NIST SRM, small positive biases are evident, with three exceptions. The MAP-3 K-shell shows a small negative bias, while the Microlead I and the XK-3 have larger positive biases

(approximately 0.6 mg/cm^2 and 0.8 mg/cm^2, respectively). On the bare control blocks, the Lead Analyzer (K- and L-shells) and the X-MET 880 have small biases. The Microlead I, the XK-3, and the XL have positive biases, while the MAP-3 K-shell has a negative bias. The MAP-3 L-shell has a negative bias, also, though smaller than the MAP-3 K-shell bias.

Table 6-18. Summary Statistics of Lead Measured in mg/cm^2 Units of the First Red (1.02 mg/cm^2) NIST SRM Readings Taken on All Six Control Blocks From Louisville Only.

XRF TYPE	CONTROL TYPE	NUMBER OF READINGS	ARITHMETIC MEAN	MAXIMUM	MINIMUM	25th PERCENTILE	MEDIAN	75TH PERCENTILE
MAP-3 K-shell	Beginning	12	1.162	1.522	0.782	1.084	1.148	1.257
	Continuing	20	0.957	1.497	0.095	0.737	1.019	1.254
	Ending	12	1.088	1.415	0.500	0.951	1.104	1.302
MAP-3 L-shell	Beginning	12	1.377	1.568	1.165	1.302	1.409	1.434
	Continuing	20	1.395	1.688	1.159	1.264	1.397	1.476
	Ending	12	1.341	1.452	1.153	1.297	1.349	1.423
Microlead I	Beginning	12	1.017	1.600	0.60	0.650	0.900	1.400
	Continuing	17	1.041	2.000	0.300	0.700	0.900	1.300
	Ending	12	1.208	2.200	0.800	0.900	1.000	1.500
X-MET 880	Beginning	12	1.025	1.145	0.892	0.963	1.017	1.107
	Continuing	16	1.119	1.255	0.937	1.054	1.142	1.197
	Ending	12	1.071	1.240	0.937	1.006	1.044	1.130
XK-3	Beginning	12	1.250	2.700	0.200	0.800	1.100	1.550
	Continuing	17	1.135	2.200	0.000	0.800	1.100	1.500
	Ending	12	0.833	2.200	-0.600	0.200	0.900	1.300

Table 6-19. Summary Statistics of Lead Measured in mg/cm^2 Units of the Second Red (1.02 mg/cm^2) NIST SRM Readings Taken on All Six Control Blocks From Louisville Only.

XRF TYPE	CONTROL TYPE	NUMBER OF READINGS	ARITHMETIC MEAN	MAXIMUM	MINIMUM	25th PERCENTILE	MEDIAN	75TH PERCENTILE
MAP-3 K-shell	Beginning	12	1.145	1.912	0.749	0.967	1.122	1.228
	Continuing	0	na	na	na	na	na	na
	Ending	12	1.065	1.585	0.369	0.833	1.163	1.246
MAP-3 L-shell	Beginning	12	1.361	1.550	1.196	1.273	1.366	1.433
	Continuing	0	na	na	na	na	na	na
	Ending	12	1.322	1.474	1.153	1.289	1.330	1.351
Microlead I	Beginning	12	0.917	1.300	0.600	0.750	0.950	1.000
	Continuing	17	1.041	1.500	0.400	0.700	1.000	1.400
	Ending	12	1.142	1.900	0.400	0.700	1.100	1.550
X-MET 880	Beginning	12	1.060	1.230	0.904	0.960	1.069	1.138
	Continuing	16	1.128	1.270	0.865	1.087	1.150	1.224
	Ending	12	1.137	1.371	1.020	1.057	1.110	1.188
XK-3	Beginning	12	1.075	2.000	0.300	0.750	1.050	1.300
	Continuing	17	1.176	2.500	0.400	0.700	1.100	1.300
	Ending	12	1.108	2.200	0.400	0.600	1.050	1.450

Table 6-20. Summary Statistics of Lead Measured in mg/cm^2 Units of the Third Red (1.02 mg/cm^2) NIST SRM Readings Taken on All Six Control Blocks From Louisville Only.

XRF TYPE	CONTROL TYPE	NUMBER OF READINGS	ARITHMETIC MEAN	MAXIMUM	MINIMUM	25th PERCENTILE	MEDIAN	75TH PERCENTILE
MAP-3 K-shell	Beginning	12	1.174	1.660	0.842	1.025	1.141	1.274
	Continuing	0	na	na	na	na	na	na
	Ending	12	1.010	1.828	0.496	0.618	1.022	1.224
MAP-3 L-shell	Beginning	12	1.355	1.491	1.175	1.273	1.366	1.429
	Continuing	0	na	na	na	na	na	na
	Ending	12	1.318	1.449	1.164	1.272	1.337	1.381
Microlead I	Beginning	12	1.042	1.800	0.400	0.700	1.000	1.350
	Continuing	17	1.182	2.000	0.600	0.900	1.200	1.400
	Ending	12	0.850	1.900	-0.300	0.550	0.900	1.000
X-MET 880	Beginning	12	1.071	1.248	0.908	0.986	1.031	1.196
	Continuing	16	1.083	1.449	0.838	0.991	1.080	1.164
	Ending	12	1.135	1.267	0.941	1.097	1.135	1.197
XK-3	Beginning	12	1.225	2.400	0.500	0.650	1.150	1.700
	Continuing	17	1.206	2.700	0.700	0.800	1.000	1.300
	Ending	12	1.225	2.000	0.400	0.800	1.200	1.650

Table 6-21. Summary Statistics of Lead Measured in mg/cm^2 Units of the Fourth Red (1.02 mg/cm^2) NIST SRM Readings Taken on All Six Control Blocks From Louisville Only.

XRF TYPE	CONTROL TYPE	NUMBER OF READINGS	ARITHMETIC MEAN	MAXIMUM	MINIMUM	25th PERCENTILE	MEDIAN	75TH PERCENTILE
MAP-3 K-shell	Beginning	0	na	na	na	na	na	na
	Ending	0	na	na	na	na	na	na
MAP-3 L-shell	Beginning	0	na	na	na	na	na	na
	Ending	0	na	na	na	na	na	na
Microlead I	Beginning	12	1.083	1.700	0.600	0.800	1.000	1.400
	Ending	12	1.067	1.900	0.300	0.700	1.000	1.500
X-MET 880	Beginning	12	1.083	1.234	0.867	1.013	1.094	1.174
	Ending	12	1.141	1.651	0.899	1.054	1.153	1.175
XK-3	Beginning	12	1.108	2.200	0.300	0.750	1.000	1.250
	Ending	12	1.275	2.600	0.600	0.700	1.150	1.500

Table 6-22. Summary Statistics of Lead Measured in mg/cm^2 Units of the Fifth Red (1.02 mg/cm^2) NIST SRM Readings Taken on All Six Control Blocks From Louisville Only.

XRF TYPE	CONTROL TYPE	NUMBER OF READINGS	ARITHMETIC MEAN	MAXIMUM	MINIMUM	25th PERCENTILE	MEDIAN	75TH PERCENTILE
MAP-3 K-shell	Beginning	0	na	na	na	na	na	na
	Ending	0	na	na	na	na	na	na
MAP-3 L-shell	Beginning	0	na	na	na	na	na	na
	Ending	0	na	na	na	na	na	na
Microlead I	Beginning	12	1.033	1.700	0.600	0.800	0.850	1.350
	Ending	12	1.167	1.900	0.200	0.700	1.100	1.850
X-MET 880	Beginning	12	1.058	1.300	0.990	0.998	1.015	1.068
	Ending	12	1.141	1.334	1.014	1.090	1.129	1.178
XK-3	Beginning	12	1.200	2.100	0.600	0.950	1.050	1.350
	Ending	12	1.175	2.300	0.500	0.900	1.050	1.350

Table 6-23. Summary Statistics of Lead Measured in mg/cm^2 Units of the Sixth Red (1.02 mg/cm^2) NIST SRM Readings taken on All Six Control Blocks From Louisville Only.

XRF TYPE	CONTROL TYPE	NUMBER OF READINGS	ARITHMETIC MEAN	MAXIMUM	MINIMUM	25th PERCENTILE	MEDIAN	75TH PERCENTILE
MAP-3 K-shell	Beginning	0	na	na	na	na	na	na
	Ending	0	na	na	na	na	na	na
MAP-3 L-shell	Beginning	0	na	na	na	na	na	na
	Ending	0	na	na	na	na	na	na
Microlead I	Beginning	12	0.883	1.400	0.400	0.650	0.950	1.050
	Ending	12	1.117	1.900	0.500	0.700	1.150	1.450
X-MET 880	Beginning	12	1.070	1.291	0.8520	0.977	1.074	1.159
	Ending	12	1.139	1.315	0.901	1.058	1.151	1.233
XK-3	Beginning	12	1.217	1.900	0.400	0.700	1.300	1.600
	Ending	12	1.158	2.300	0.500	0.750	0.950	1.550

Table 6-24. Summary Statistics of Lead Measured in mg/cm^2 Units of the Seventh Red (1.02 mg/cm^2) NIST SRM Readings Taken on All Six Control Blocks From Louisville Only.

XRF TYPE	CONTROL TYPE	NUMBER OF READINGS	ARITHMETIC MEAN	MAXIMUM	MINIMUM	25th PERCENTILE	MEDIAN	75TH PERCENTILE
MAP-3 K-shell	Beginning	0	na	na	na	na	na	na
	Ending	0	na	na	na	na	na	na
MAP-3 L-shell	Beginning	0	na	na	na	na	na	na
	Ending	0	na	na	na	na	na	na
Microlead I	Beginning	12	0.917	1.200	0.600	0.700	0.850	1.150
	Ending	12	0.825	1.700	0.100	0.550	0.750	1.100
X-MET 880	Beginning	12	1.118	1.284	0.931	1.031	1.119	1.229
	Ending	12	1.175	1.369	1.005	1.113	1.164	1.232
XK-3	Beginning	12	1.183	2.300	0.400	0.850	1.050	1.350
	Ending	12	1.192	2.200	0.300	0.750	1.100	1.600

Table 6-25. Summary Statistics of Lead Measured in mg/cm^2 Units of the Eighth Red (1.02 mg/cm^2) NIST SRM Readings Taken on All Six Control Blocks From Louisville Only.

XRF TYPE	CONTROL TYPE	NUMBER OF READINGS	ARITHMETIC MEAN	MAXIMUM	MINIMUM	25th PERCENTILE	MEDIAN	75TH PERCENTILE
MAP-3 K-shell	Beginning	0	na	na	na	na	na	na
	Ending	0	na	na	na	na	na	na
MAP-3 L-shell	Beginning	0	na	na	na	na	na	na
	Ending	0	na	na	na	na	na	na
Microlead I	Beginning	12	1.033	1.500	0.700	0.900	1.000	1.150
	Ending	12	1.158	2.000	0.700	0.800	1.100	1.400
X-MET 880	Beginning	12	1.064	1.199	0.793	0.979	1.120	1.146
	Ending	12	1.414	4.840	0.960	1.005	1.054	1.275
XK-3	Beginning	12	1.333	2.200	0.500	1.100	1.300	1.550
	Ending	12	1.067	2.600	0.500	0.750	0.900	1.100

Table 6-26. Summary Statistics of Lead Measured in mg/cm^2 Units of the Ninth Red (1.02 mg/cm^2) NIST SRM Readings taken on All Six Control Blocks From Louisville Only.

XRF TYPE	CONTROL TYPE	NUMBER OF READINGS	ARITHMETIC MEAN	MAXIMUM	MINIMUM	25th PERCENTILE	MEDIAN	75TH PERCENTILE
MAP-3 K-shell	Beginning	0	na	na	na	na	na	na
	Ending	0	na	na	na	na	na	na
MAP-3 L-shell	Beginning	0	na	na	na	na	na	na
	Ending	0	na	na	na	na	na	na
Microlead I	Beginning	12	1.025	1.500	0.500	0.800	1.050	1.250
	Ending	12	0.967	1.600	0.300	0.550	1.050	1.300
X-MET 880	Beginning	12	1.086	1.312	0.951	1.025	1.072	1.123
	Ending	12	1.130	1.293	0.948	1.096	1.137	1.183
XK-3	Beginning	12	1.150	2.400	0.000	0.800	1.050	1.350
	Ending	12	1.175	2.100	0.600	0.750	1.100	1.450

Table 6-27. Summary Statistics of Lead Measured in mg/cm^2 Units of the Yellow (3.53 mg/cm^2) NIST SRM Readings Taken on the Concrete Control Block in Louisville Only.

XRF TYPE	CONTROL TYPE	NUMBER OF READINGS	ARITHMETIC MEAN	MAXIMUM	MINIMUM	25th PERCENTILE	MEDIAN	75TH PERCENTILE
MAP-3 K-shell	Beginning	6	3.684	4.002	3.476	3.558	3.592	3.885
	Continuing	4	3.563	3.908	3.078	3.335	3.634	3.792
	Ending	6	3.763	4.032	3.349	3.568	3.812	4.004
MAP-3 L-shell	Beginning	6	4.391	4.696	4.104	4.249	4.423	4.449
	Continuing	4	4.691	4.968	4.173	4.479	4.812	4.904
	Ending	6	4.064	4.333	3.818	3.963	4.044	4.179
Microlead I	Beginning	18	3.483	4.000	1.700	3.300	3.600	3.800
	Continuing	12	3.650	4.200	3.400	3.400	3.600	3.800
	Ending	18	3.500	3.900	3.000	3.300	3.550	3.800
X-MET 880	Beginning	18	3.852	4.254	3.452	3.650	3.825	4.073
	Continuing	12	4.046	4.269	3.787	3.938	4.017	4.202
	Ending	18	4.097	4.280	3.907	4.054	4.097	4.165
XK-3	Beginning	18	2.978	3.500	2.400	2.800	3.000	3.200
	Continuing	12	2.983	3.500	2.500	2.800	3.000	3.200
	Ending	18	2.933	3.500	2.200	2.700	2.950	3.300

Table 6-28. Summary Statistics of Lead Measured in mg/cm^2 Units of the First Yellow (3.53 mg/cm^2) NIST SRM Readings Taken on All Six Control Blocks From Denver and Philadelphia Only.

XRF TYPE	CONTROL TYPE	NUMBER OF READINGS	ARITHMETIC MEAN	MAXIMUM	MINIMUM	25th PERCENTILE	MEDIAN	75TH PERCENTILE
Lead Analyzer K-shell	Beginning	126	3.690	5.000	2.800	3.400	3.700	3.900
	Continuing	177	3.749	5.100	2.400	3.400	3.700	4.000
	Ending	120	3.734	4.700	2.700	3.500	3.700	4.000
Lead Analyzer L-shell	Beginning	126	2.726	2.930	2.280	2.700	2.740	2.790
	Continuing	177	2.710	2.910	1.590	2.680	2.730	2.770
	Ending	120	2.715	2.860	2.540	2.675	2.720	2.760
MAP-3 K-shell	Beginning	396	3.283	4.580	-0.138	2.975	3.362	3.652
	Continuing	355	3.263	4.746	-1.519	3.009	3.345	3.614
	Ending	396	3.254	4.434	0.149	2.971	3.317	3.613
MAP-3 L-shell	Beginning	396	3.343	3.808	-0.410	3.233	3.353	3.462
	Continuing	355	3.298	3.976	-0.403	3.184	3.328	3.466
	Ending	396	3.325	3.860	1.170	3.193	3.334	3.478
Microlead I	Beginning	228	3.724	10.600	-0.190	3.200	3.600	4.000
	Continuing	355	3.912	10.400	-0.300	3.300	3.700	4.200
	Ending	228	3.776	10.200	1.400	3.100	3.600	4.100
X-MET 880	Beginning	114	3.711	4.172	3.309	3.614	3.710	3.820
	Continuing	179	3.685	4.100	3.185	3.575	3.677	3.819
	Ending	114	3.720	5.712	3.197	3.590	3.693	3.874
XK-3	Beginning	216	4.444	7.100	2.200	3.900	4.300	4.950
	Continuing	354	4.548	8.700	2.800	4.100	4.400	5.000
	Ending	216	4.378	9.000	1.900	3.900	4.200	4.750

XL	Beginning	114	3.228	4.000	2.100	2.800	3.400	3.600
	Continuing	198	3.359	4.300	1.600	3.100	3.500	3.700
	Ending	114	3.580	4.300	2.100	3.400	3.600	3.800

Table 6-29. Summary Statistics of Lead Measured in mg/cm^2 Units of the Second Yellow (3.53 mg/cm^2) NIST SRM Readings Taken on All Six Control Blocks From Denver and Philadelphia Only.

XRF TYPE	CONTROL TYPE	NUMBER OF READINGS	ARITHMETIC MEAN	MAXIMUM	MINIMUM	25th PERCENTILE	MEDIAN	75TH PERCENTILE
Lead Analyzer K-shell	Beginning	126	3.656	5.000	2.700	3.400	3.700	3.900
	Continuing	177	3.740	4.900	2.600	3.400	3.800	4.000
	Ending	120	3.709	4.900	2.800	3.500	3.700	3.900
Lead Analyzer L-shell	Beginning	126	2.712	2.930	2.010	2.670	2.730	2.770
	Continuing	177	2.710	2.900	1.560	2.680	2.720	2.780
	Ending	120	2.723	2.880	2.500	2.685	2.730	2.770
MAP-3 K-shell	Beginning	396	3.280	4.565	1.380	3.014	3.343	3.632
	Continuing	354	3.280	4.832	0.217	2.983	3.318	3.616
	Ending	396	3.267	4.943	-0.088	3.002	3.305	3.563
MAP-3 L-shell	Beginning	396	3.335	3.859	2.044	3.198	3.345	3.473
	Continuing	355	3.299	3.765	-0.404	3.190	3.325	3.457
	Ending	396	3.311	3.839	1.162	3.190	3.324	3.462
Microlead I	Beginning	228	4.025	10.800	1.900	3.400	3.900	4.200
	Continuing	355	4.071	11.000	-0.900	3.400	3.900	4.300
	Ending	228	4.008	11.200	2.600	3.500	3.700	4.150
X-MET 880	Beginning	114	3.696	4.081	3.334	3.598	3.698	3.816
	Continuing	179	3.666	4.075	3.165	3.537	3.656	3.808
	Ending	114	3.711	5.691	3.194	3.578	3.700	3.850

XK-3	Beginning	216	4.431	6.700	2.000	4.000	4.300	4.900
	Continuing	354	4.531	6.800	2.700	4.100	4.400	5.000
	Ending	216	4.408	6.800	3.100	4.000	4.250	4.800
XL	Beginning	114	3.251	4.200	2.200	2.900	3.300	3.600
	Continuing	198	3.371	4.700	1.700	3.100	3.500	3.700
	Ending	114	3.542	4.500	2.600	3.300	3.600	3.700

Table 6-30. Summary Statistics of Lead Measured in mg/cm^2 Units of the Third Yellow (3.53 mg/cm^2) NIST SRM Readings Taken on All Six Control Blocks From Denver and Philadelphia Only.

XRF TYPE	CONTROL TYPE	NUMBER OF READINGS	ARITHMETIC MEAN	MAXIMUM	MINIMUM	25th PERCENTILE	MEDIAN	75TH PERCENTILE
Lead Analyzer K-shell	Beginning	126	3.641	4.800	2.900	3.300	3.600	3.900
	Continuing	177	3.724	5.000	2.700	3.500	3.700	4.000
	Ending	120	3.703	4.900	2.800	3.500	3.700	3.900
Lead Analyzer L-shell	Beginning	126	2.724	2.930	2.310	2.670	2.745	2.780
	Continuing	177	2.711	2.930	1.560	2.680	2.730	2.770
	Ending	120	2.719	2.890	2.520	2.675	2.730	2.770
MAP-3 K-shell	Beginning	396	3.258	5.154	1.168	2.938	3.291	3.642
	Continuing	355	3.289	4.985	0.473	3.015	3.317	3.592
	Ending	396	3.262	4.675	-0.106	3.012	3.287	3.581
MAP-3 L-shell	Beginning	396	3.342	3.888	2.742	3.208	3.341	3.490
	Continuing	355	3.325	3.890	-0.078	3.193	3.334	3.473
	Ending	396	3.322	3.881	1.085	3.187	3.338	3.471
Microlead I	Beginning	228	3.954	11.000	1.7000	3.400	3.800	4.100
	Continuing	355	4.072	11.200	-0.900	3.400	3.800	4.300
	Ending	228	3.983	10.400	2.500	3.300	3.800	4.300

X-MET 880	Beginning	114	3.676	4.072	3.305	3.567	3.664	3.807
	Continuing	179	3.660	4.203	2.718	3.551	3.654	3.805
	Ending	114	3.700	5.557	3.214	3.548	3.689	3.845
XK-3	Beginning	216	4.325	7.100	2.100	3.900	4.200	4.850
	Continuing	354	4.461	6.500	2.300	4.000	4.400	4.900
	Ending	216	4.410	8.100	3.000	4.000	4.300	4.700
XL	Beginning	114	3.268	4.200	2.100	2.900	3.400	3.600
	Continuing	198	3.339	4.300	1.600	3.000	3.400	3.700
	Ending	114	3.546	4.300	2.600	3.400	3.600	3.800

Table 6-31. Summary Statistics of Lead Measured in mg/cm^2 Units of the First Red (1.02 mg/cm^2) NIST SRM Reading (Control Blocks) From Denver and Philadelphia Only.

XRF TYPE	CONTROL TYPE	NUMBER OF READINGS	ARITHMETIC MEAN	MAXIMUM	MINIMUM	25th PERCENTILE	MEDIAN	75TH PERCENTILE
Lead Analyzer K-shell	Beginning	126	1.083	1.700	0.100	0.900	1.100	1.200
	Continuing	177	1.106	1.900	0.700	1.000	1.100	1.200
	Ending	120	1.066	1.600	0.500	0.900	1.100	1.200
Lead Analyzer L-shell	Beginning	126	1.070	1.900	0.900	1.040	1.070	1.100
	Continuing	177	1.052	1.160	0.590	1.020	1.050	1.080
	Ending	120	1.059	1.150	0.950	1.040	1.060	1.080
MAP-3 K-shell	Beginning	396	0.907	2.623	-1.027	0.645	0.985	1.264
	Continuing	355	0.934	2.192	-0.840	0.656	0.964	1.250
	Ending	396	0.887	2.882	-3.373	0.611	0.920	1.201
MAP-3 L-shell	Beginning	396	1.160	1.455	0.802	1.102	1.158	1.221
	Continuing	355	1.149	1.449	-0.799	1.092	1.156	1.220
	Ending	396	1.151	3.052	-0.406	1.094	1.150	1.211

Microlead I	Beginning	228	1.432	7.600	-0.200	0.900	1.200	1.650
	Continuing	355	1.555	7.300	-0.900	0.900	1.400	1.800
	Ending	228	1.508	7.700	-0.900	0.850	1.300	1.700
X-MET 880	Beginning	113	1.074	1.205	0.971	1.042	1.073	1.097
	Continuing	179	1.073	1.170	0.991	1.039	1.074	1.104
	Ending	114	1.080	1.629	0.982	1.037	1.081	1.108
XK-3	Beginning	216	1.900	3.600	0.600	1.450	1.800	2.400
	Continuing	354	1.901	4.000	0.400	1.500	1.800	2.400
	Ending	216	1.906	7.300	0.600	1.400	1.800	2.400
XL	Beginning	114	1.049	1.200	0.800	1.000	1.100	1.100
	Continuing	198	1.063	1.300	0.600	1.000	1.100	1.100
	Ending	114	1.099	1.300	0.800	1.100	1.100	1.200

Table 6-32. Summary Statistics of Lead Measured in mg/cm^2 Units of the Second Red (1.02 mg/cm^2) NIST SRM Readings Taken on All Six Control Blocks From Denver and Philadelphia Only.

XRF TYPE	CONTROL TYPE	NUMBER OF READINGS	ARITHMETIC MEAN	MAXIMUM	MINIMUM	25th PERCENTILE	MEDIAN	75TH PERCENTILE
Lead Analyzer K-shell	Beginning	126	1.065	1.600	0.500	0.900	1.100	1.200
	Continuing	177	1.095	1.700	0.500	0.900	1.100	1.200
	Ending	120	1.110	1.500	0.600	1.000	1.100	1.300
Lead Analyzer L-shell	Beginning	126	1.055	1.170	0.750	1.030	1.060	1.090
	Continuing	177	1.054	1.150	0.610	1.030	1.050	1.090
	Ending	120	1.064	1.140	0.970	1.040	1.060	1.090
MAP-3 K-shell	Beginning	396	0.851	2.315	-1.111	0.557	0.922	1.182
	Continuing	355	0.888	2.254	-0.748	0.672	0.937	1.197
	Ending	396	0.912	2.782	-0.942	0.647	0.936	1.243

MAP-3 L-shell	Beginning	396	1.159	1.413	0.901	1.097	1.164	1.220
	Continuing	355	1.147	1.407	0.871	1.092	1.145	1.210
	Ending	396	1.153	2.798	-0.414	1.096	1.158	1.213
Microlead I	Beginning	228	1.661	8.300	-1.000	1.050	1.400	1.900
	Continuing	355	1.695	7.200	-1.500	1.000	1.400	2.000
	Ending	228	1.667	7.700	-1.500	1.000	1.4000	1.950
X-MET 880	Beginning	114	1.070	1.196	0.993	1.036	1.069	1.095
	Continuing	179	1.066	1.186	0.961	1.038	1.068	1.095
	Ending	114	1.073	1.548	0.960	1.031	1.069	1.104
XK-3	Beginning	216	1.772	3.900	0.400	1.300	1.700	2.100
	Continuing	354	1.877	4.600	0.400	1.400	1.800	2.300
	Ending	216	1.744	4.100	0.300	1.300	1.700	2.200
XL	Beginning	114	1.046	1.200	0.800	1.000	1.100	1.100
	Continuing	198	1.060	1.300	0.700	1.000	1.100	1.100
	Ending	114	1.089	1.300	0.800	1.100	1.100	1.100

Table 6-33. Summary Statistics of Lead Measured in mg/cm² Units of the Third Red (1.02 mg/cm²) NIST SRM Readings Taken on All Six Control Blocks From Denver and Philadelphia Only.

XRF TYPE	CONTROL TYPE	NUMBER OF READINGS	ARITHMETIC MEAN	MAXIMUM	MINIMUM	25th PERCENTILE	MEDIAN	75TH PERCENTILE
Lead Analyzer K-shell	Beginning	126	1.085	1.900	0.600	0.900	1.100	1.200
	Continuing	177	1.085	1.800	0.500	0.900	1.100	1.200
	Ending	120	1.102	1.600	0.500	0.950	1.100	1.300
Lead Analyzer L-shell	Beginning	126	1.058	1.160	0.830	1.030	1.065	1.090
	Continuing	177	1.054	1.170	0.590	1.030	1.050	1.080
	Ending	120	1.058	1.140	0.960	1.040	1.060	1.080

MAP-3 K-shell	Beginning	396	0.855	2.494	-0.899	0.565	0.927	1.177
	Continuing	355	0.966	2.877	-0.991	0.706	1.011	1.233
	Ending	396	0.917	2.602	-0.515	0.657	0.941	1.214
MAP-3 L-shell	Beginning	396	1.156	1.930	0.173	1.102	1.169	1.218
	Continuing	355	1.148	1.450	0.869	1.089	1.147	1.204
	Ending	396	1.151	3.122	-0.151	1.095	1.156	1.216
Microlead I	Beginning	228	1.595	8.000	-1.000	0.900	1.300	1.800
	Continuing	355	1.670	7.700	-1.900	1.000	1.400	2.000
	Ending	228	1.621	7.700	-2.400	1.000	1.400	2.000
X-MET 880	Beginning	114	1.067	1.182	0.978	1.036	1.066	1.098
	Continuing	179	1.066	1.177	0.970	1.031	1.067	1.094
	Ending	114	1.071	1.593	0.972	1.036	1.064	1.099
XK-3	Beginning	216	1.742	5.000	0.400	1.300	1.600	2.100
	Continuing	354	1.753	3.700	0.600	1.300	1.700	2.200
	Ending	216	1.739	3.400	0.400	1.300	1.600	2.200
XL	Beginning	114	1.045	1.200	0.800	1.000	1.100	1.100
	Continuing	198	1.062	1.300	0.600	1.000	1.100	1.100
	Ending	114	1.093	1.200	0.900	1.100	1.100	1.100

Table 6-34. Summary Statistics of Lead Measured in mg/cm² Units of the First Bare Substrate Readings Taken on All Six Control Blocks From Denver and Philadelphia Only.

XRF TYPE	CONTROL TYPE	NUMBER OF READINGS	ARITHMETIC MEAN	MAXIMUM	MINIMUM	25th PERCENTILE	MEDIAN	75TH PERCENTILE
Lead Analyzer K-shell	Beginning	126	-0.003	0.400	-0.368	-0.080	-0.004	0.050
	Continuing	177	-0.002	0.200	-0.400	-0.050	0.000	0.060
	Ending	120	-0.009	0.300	-0.459	-0.070	-0.006	0.040

Lead Analyzer L-shell	Beginning	126	0.001	0.063	-0.049	-0.006	0.001	0.010
	Continuing	177	-0.002	0.064	-0.054	-0.008	0.000	0.008
	Ending	120	0.000	0.055	-0.048	-0.008	0.001	0.010
MAP-3 K-shell	Beginning	396	-0.733	0.932	-3.300	-1.386	-0.458	-0.035
	Continuing	355	-0.626	0.773	-3.240	-1.255	-0.389	-0.006
	Ending	396	-0.650	1.834	-4.356	-1.156	-0.447	0.009
MAP-3 L-shell	Beginning	396	-0.184	0.342	-1.146	-0.216	-0.177	-0.137
	Continuing	355	-0.171	0.181	-1.086	-0.202	-0.166	-0.126
	Ending	396	-0.169	1.332	-1.128	-0.200	-0.163	-0.117
Microlead I	Beginning	228	0.393	6.800	-1.500	-0.200	0.200	0.600
	Continuing	355	0.570	8.600	-1.300	-0.100	0.300	0.700
	Ending	228	0.486	6.800	-1.700	-0.100	0.200	0.700
X-MET 880	Beginning	95	0.035	0.094	0.021	0.030	0.034	0.037
	Continuing	141	0.036	0.096	0.020	0.028	0.034	0.037
	Ending	95	0.036	0.095	0.021	0.030	0.034	0.038
XK-3	Beginning	216	0.728	2.300	-0.800	0.400	0.600	1.100
	Continuing	354	0.711	2.600	-1.000	0.300	0.700	1.100
	Ending	216	0.692	3.000	-1.100	0.300	0.600	1.100
XL	Beginning	114	0.281	2.400	0.000	0.000	0.100	0.200
	Continuing	198	0.266	3.500	0.000	0.000	0.000	0.200
	Ending	114	0.175	1.500	0.000	0.000	0.000	0.200

Table 6-35. Summary Statistics of Lead Measured in mg/cm^2 Units of the Second Bare Substrate Readings Taken on All Six Control Blocks From Denver and Philadelphia Only.

XRF TYPE	CONTROL TYPE	NUMBER OF READINGS	ARITHMETIC MEAN	MAXIMUM	MINIMUM	25th PERCENTILE	MEDIAN	75TH PERCENTILE

Lead Analyzer K-shell	Beginning	126	0.009	0.400	-0.300	-0.500	0.000	0.050
	Continuing	177	-0.008	0.300	-0.300	-0.050	0.000	0.030
	Ending	120	-0.004	0.300	-0.300	-0.050	-0.001	0.050
Lead Analyzer L-shell	Beginning	126	0.001	0.068	-0.047	-0.007	0.001	0.009
	Continuing	177	-0.002	0.060	-0.052	-0.008	0.000	0.008
	Ending	120	0.001	0.071	-0.051	-0.008	0.001	0.011
MAP-3 K-shell	Beginning	396	-0.765	2.135	-3.970	-1.475	-0.511	0.005
	Continuing	355	-0.630	1.420	-3.279	-1.167	-0.405	0.056
	Ending	396	-0.657	0.722	-4.343	-1.275	-0.344	-0.001
MAP-3 L-shell	Beginning	396	-0.193	0.268	-1.943	-0.230	-0.175	-0.139
	Continuing	355	-0.170	0.160	-1.198	-0.204	-0.161	-0.124
	Ending	396	-0.169	1.181	-2.193	-0.203	-0.160	-0.116
Microlead I	Beginning	228	0.562	7.300	-1.300	0.000	0.200	0.800
	Continuing	355	0.698	7.500	-1.600	0.000	0.300	1.000
	Ending	228	0.641	7.000	-1.300	0.000	0.300	0.800
X-MET 880	Beginning	95	0.035	0.095	0.021	0.030	0.034	0.037
	Continuing	141	0.036	0.092	0.022	0.029	0.034	0.037
	Ending	95	0.035	0.091	0.023	0.030	0.033	0.037
XK-3	Beginning	216	0.635	2.100	-1.100	0.200	0.600	1.000
	Continuing	354	0.728	2.600	-0.900	0.300	0.700	1.200
	Ending	216	0.702	3.700	-0.800	0.300	0.600	1.100
XL	Beginning	114	0.282	2.500	0.000	0.000	0.100	0.300
	Continuing	198	0.269	3.700	0.000	0.000	0.100	0.200
	Ending	114	0.167	1.500	0.000	0.000	0.100	0.100

Table 6-36. Summary Statistics of Lead Measured in mg/cm^2 Units of the Third Bare Substrate Readings Taken on All Six Control Blocks From Denver and Philadelphia Only.

XRF TYPE	CONTROL TYPE	NUMBER OF READINGS	ARITHMETIC MEAN	MAXIMUM	MINIMUM	25th PERCENTILE	MEDIAN	75TH PERCENTILE
Lead Analyzer K-shell	Beginning	126	0.009	0.300	-0.400	-0.050	0.000	0.060
	Continuing	177	0.005	0.300	-0.400	-0.040	0.000	0.060
	Ending	120	-0.013	0.400	-0.300	-0.080	-0.001	0.035
Lead Analyzer L-shell	Beginning	126	0.001	0.070	-0.047	-0.007	0.000	0.009
	Continuing	177	-0.001	0.061	-0.053	-0.009	0.000	0.008
	Ending	120	0.000	0.059	-0.052	-0.007	0.001	0.010
MAP-3 K-shell	Beginning	396	-0.669	2.725	-3.344	-1.323	-0.445	-0.019
	Continuing	355	-0.648	1.130	-3.566	-1.309	-0.377	0.010
	Ending	396	-0.589	1.502	-3.519	-1.115	-0.366	0.003
MAP-3 L-shell	Beginning	396	-0.182	0.253	-0.571	-0.224	-0.178	-0.139
	Continuing	355	-0.169	0.258	-0.901	-0.204	-0.163	-0.125
	Ending	396	-0.172	2.193	-2.323	-0.197	-0.160	-0.112
Microlead I	Beginning	228	0.504	7.500	-1.500	-0.15	0.200	0.700
	Continuing	355	0.667	7.800	-1.400	-0.100	0.300	0.900
	Ending	228	0.591	6.900	-1.200	-0.100	0.300	0.900
X-MET 880	Beginning	95	0.035	0.094	0.022	0.030	0.033	0.037
	Continuing	141	0.035	0.095	0.020	0.028	0.033	0.037
	Ending	95	0.036	0.096	0.022	0.030	0.034	0.037
XK-3	Beginning	216	0.643	2.300	-1.000	0.200	0.500	1.000
	Continuing	353	0.680	2.000	-0.700	0.300	0.600	1.100
	Ending	216	0.619	2.800	-0.900	0.200	0.600	1.000
XL	Beginning	114	0.309	2.500	0.000	0.000	0.100	0.300
	Continuing	198	0.269	4.000	0.000	0.000	0.000	0.200

	Ending	114	0.168	1.900	0.000	0.000	0.000	0.100

6.3 DATA OUTLIERS 6-53
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6.3 DATA OUTLIERS

The primary data examined for outliers were the standard first paint readings. Standard paint readings are defined in the first section of this chapter. The identification of outlier data in the standard first paint readings used a procedure based on nonparametric regression techniques. A discussion of this procedure is given below.

The purpose of outlier designation was to identify data that might adversely affect estimation of the relationship between XRF and the level of lead in paint. This effort addressed the following study objectives:

- to characterize the performance (precision and accuracy) of portable XRF instruments under field conditions

- to investigate XRF measurements that were very different than their corresponding laboratory results.

Note that it is not suggested that an outlier represents bad data: it may reflect behavior of an XRF instrument under unusual circumstances, or that cannot be described in a uniform way. Failure to remove outliers from an analysis may produce results that are descriptive of neither the circumstances leading to the outliers, nor the typical performance of the instrument. If a standard first paint reading was identified as an outlier, it was excluded from the analyses presented in the next section of this chapter.

Combined, over 15,000 XRF first standard paint readings were taken with different instruments and on different substrates. A total of 84 outliers were identified in the standard first paint readings from seven of the eight XRF instrument classifications in Denver and Philadelphia. Outlier identification methodology was not applied to the eighth instrument, the XL, because of the truncation of its readings at 0.0 mg/cm^2 and at 5.0 mg/cm^2. All other standard first paint readings from the other seven XRF instruments were examined for outliers, resulting in the identification of 84 standard first paint readings designated as outliers from 41 sampling locations in Denver and Philadelphia. Summary statistics and other descriptions of these outliers are given in section 6.3.2 below.

6.3.1 Outlier Identification Methodology

In this section an objective criterion for defining outliers is described. Its use led to the identification of a very small set of data that were excluded from model estimation. Since the aim was to identify data that could distort the estimated relationship between XRF readings and the level of lead in paint, the outlier criterion used the observable XRF relationship compared to the laboratory ICP result measured in mg/cm^2 as its basis.

6.3.1.1 Basic Assumptions Concerning the XRF Response to Lead

In the following section of this chapter (6.4) a detailed model relating XRF readings to the true (but unknown) lead level is developed. The model suggests that, on average, XRF readings respond linearly to the lead level, with variability that increases with the lead level according to a simple functional form. The same should also be true, approximately, for the observable XRF-ICP relationship. One way to define an outlier would be to fit a regression model where the mean XRF reading is a linear function of ICP, with a standard deviation having the form specified in the model. An observation that deviated too far from the mean, as measured by its standardized residual, would be flagged as an outlier. What "too far" precisely constitutes can be judged from the distribution of the standardized residuals, which are approximately standard normal random variables, and the size of the sample. Standardized residuals that are smaller than about -3.5 or larger than 3.5 are typical candidates for outlier designation.

Applying a strictly specified model to the data to distinguish outliers requires an accurate model. Observations may be flagged as outliers if the model does not fit the data very well over all or part of its range. But the purpose of developing an outlier criterion was to identify a small set of data that were unusual with respect to the rest, not to eliminate data that violated a set of assumptions that may have, in particular instances, even been wrong.

Still, the identification of unusual XRF measurements must take their relationship to ICP into account. A less strict set of assumptions about this relationship than those expressed in a

formal model was used in the development of an outlier criterion. These assumptions are the following:

(1) On average, the relationship between XRF readings and the true lead level (represented by the laboratory ICP result) is *nondecreasing*, and not restricted to being strictly linear;

(2) The standard deviation of XRF measurements at a fixed lead level (represented by the laboratory ICP result) is also a nondecreasing function of the lead level (or ICP), but not restricted to a particular functional form;

(3) The standardized residuals, formed by subtracting the mean response from XRF measurements and dividing by the standard deviation, are approximately independent, standard normal random variables.

Assumptions (1) and (2) include the model that is developed in the following section, but are very broad and include a wide range of other possibilities as well. For example, if the mean XRF response to lead is linear up to a certain point and then becomes constant, its overall response is not linear, but it is still nondecreasing. Scatter plots of XRF readings and laboratory ICP results suggested that both of these assumptions were appropriate for describing the relationship, although with possible exceptions. Higher levels of lead may have been associated with older paint under multiple layers, which may be more difficult to detect than lower levels of lead occurring in newer paint, particularly with an L-shell instrument.

Assumption (3) is needed to describe, probabilistically, the range of plausible values that a standardized residual can take. It appeared to be a reasonable assumption judging from histograms of standardized residuals that were inspected.

6.3.1.2 Derivation of Nonparametric Standardized Residuals

Deriving the standardized residuals for an XRF-ICP relationship requires the estimation of two quantities: the mean XRF response as a function of ICP, and the standard deviation (SD) of XRF measurements as a function of ICP. In both cases nondecreasing functions of ICP were required. Estimation of both components used a technique known as *monotone regression*, which

is described in section 6.4.7.3.3.

Subtracting the monotone regression estimated response from an XRF measurement, and dividing by the estimated SD, yields a quantity that will be referred to as a *nonparametric standardized residual*. In essence, nonparametric standardized residuals are a representation of XRF readings that are free of dependence on the lead level, as measured by ICP. They show no trend when plotted against ICP, and they do not exhibit increasing variability when plotted against ICP. These attributes facilitated the designation of outliers in a way that did not require further reference to the lead level.

The eight XRF instrument types evaluated in the study were categorized into 12 *field classifications* that were distinguished within an instrument type by the shell (K or L) that was used, and possibly by which of two individual machines was used. As stated above, the XL was omitted from outlier analysis due to its heavy lower truncation at 0.0 mg/cm^2 and upper truncation at 5.0 mg/cm^2. Since the performance of an XRF instrument was found to vary significantly with the substrate, of which six were identified in the study, separate outlier determinations were made on 66 (11 times 6) XRF-ICP relationships. This was done by first deriving nonparametric standardized residuals separately for each field classification-substrate combination. The Louisville pilot data were excluded from this analysis.

6.3.1.4 An Outlier Criterion

How large in absolute value should a nonparametric standardized residual be in order for it to be designated as an outlier? Referring to a probability table of the standard normal distribution gave an answer to this question in a manner that was objective, while ensuring the loss of very little data, knowing that large residuals can be expected to arise randomly in large samples.

The outlier criterion was developed in two stages. The first stage used a rule that designated a nonparametric standardized residual as an outlier if its absolute value exceeded a cutoff value. To illustrate, suppose that a sample

has *N* observations. The quantity *Z(N,90)* is defined to have the following property: the largest absolute value of *N* standard normal random variables is less than *Z(N,90)* with a probability of 90 percent. The larger the value of *N* is, the larger *Z(N,90)* is as well. For *N* = 93, which is typical of brick substrate analyses, *Z(N,90)* = 3.21 is the cutoff value. For *N* = 356, which is typical of wood analyses, *Z(N,90)* = 3.62 is the cutoff. Applying the *Z(N,90)* criterion to 13,990 observations on 11 field classifications (excluding the XL) led to the designation of 64, or less than one half of one percent of standard first paint readings as outliers.

A number of nonparametric standardized residuals barely failed to meet the *Z(N,90)* criterion, which is not unusual. A small number of these, however, had the property that they were present on several instruments of the same shell type. This may signal the location itself as an anomaly, and justify the use of a weaker outlier designation criterion. A weaker second stage outlier criterion, using a *Z(N,50)* instead of a *Z(N,90)* cutoff value, was applied if either of two conditions were met:

(1) At least one other instrument of the same shell type exceeded the *Z(N,90)* cutoff value; or

(2) At least two other instruments of the same shell type exceeded the *Z(N,50)* cutoff value.

Applying this secondary outlier criterion led to the designation of 20 additional XRF readings as outliers. In total, 84 readings were designated as outliers, which was slightly more than one half of one percent of the entire sample.

6.3.2 Outlier Data

Tables 6-37 and 6-38 provide a listing of all 84 standard first paint readings identified as outliers from Denver and Philadelphia, respectively. The identification numbers appearing twice in the tables are due to different XRF instrument field classifications that produced an outlier for the first paint reading at the same sample location. The column headings in these tables identify the sample identification number, XRF instrument, substrate, dwelling, first paint reading, and the corresponding laboratory result in mg/cm^2 for those standard

first paint readings identified as outliers. Table 6-39 presents summary statistics for the 84 outlier first paint readings and their corresponding laboratory results categorized by instrument type. Table 6-40 provides the same information except categorized by shell (radiation type). Table 6-41 provides the frequency and percent of occurrence of outliers for each substrate categorized by XRF instrument shell. Finally, Table 6-42 provides the frequency and percentage of unique sampling locations from which readings identified as outliers were taken. The values in Table 6-41 describe the occurrence of first paint reading outliers for all XRF instruments. Table 6-42 shows the frequency and percentage of sampling locations with at least one outlier, by substrate and shell.

Table 6-37. Listing of Standard First Reading Outliers From Denver.

ID NUMBER	XRF INSTRUMENT	SUBSTRATE	DWELLING	STANDARD FIRST PAINT	LABORATORY RESULT
80014	Lead Analyzer K-shell	Wood	1	0.800	0.00631
80014	MAP-3 K-shell (I)	Wood	1	1.869	0.00631
80038	MAP-3 L-shell (II)	Brick	1	-1.275	0.00159
80058	MAP-3 L-shell (II)	Concrete	1	-1.109	0.00087
80075	X-MET 880	Plaster	1	0.106	0.00633
80207	Lead Analyzer K-shell	Wood	3	1.700	0.26298
80207	MAP-3 K-shell (I)	Wood	3	4.230	0.26298
80207	Microlead I (I)	Wood	3	3.400	0.26298
80207	Microlead I (II)	Wood	3	5.100	0.26298
80213	XK-3 (I)	Wood	3	3.800	0.21029
80218	Lead Analyzer K-shell	Wood	3	1.000	0.03731
80218	MAP-3 K-shell (I)	Wood	3	2.907	0.03731
80218	MAP-3 K-shell (II)	Wood	3	3.271	0.03731
80218	Microlead I (I)	Wood	3	4.800	0.03731
80218	Microlead I (II)	Wood	3	4.700	0.03731
80227	MAP-3 L-shell (I)	Drywall	3	0.040	0.05038
80227	X-MET 880	Drywall	3	0.142	0.05038
80260	Lead Analyzer K-shell	Plaster	3	1.200	0.02799
80260	Microlead I (I)	Plaster	3	2.500	0.02799
80260	Microlead I (II)	Plaster	3	2.700	0.02799
80262	Lead Analyzer K-shell	Plaster	3	1.700	0.07583
80262	MAP-3 K-shell (I)	Plaster	3	2.688	0.07583
80262	Microlead I (I)	Plaster	3	3.400	0.07583
80262	Microlead I (II)	Plaster	3	3.900	0.07583
80311	Lead Analyzer K-shell	Wood	4	14.700	5.75144
80311	MAP-3 K-shell (I)	Wood	4	16.570	5.75144
80323	MAP-3 K-shell (II)	Wood	4	2.665	0.00987
80323	Microlead I (I)	Wood	4	2.400	0.00987
80332	MAP-3 K-shell (II)	Drywall	4	3.332	0.00224
80332	MAP-3 L-shell (II)	Drywall	4	1.158	0.00224
80332	XK-3 (I)	Drywall	4	1.000	0.00224
80343	Lead Analyzer K-shell	Drywall	4	0.900	0.00028
80343	MAP-3 K-shell (I)	Drywall	4	1.002	0.00028
80343	Microlead I (I)	Drywall	4	1.900	0.00028
80343	Microlead I (II)	Drywall	4	1.900	0.00028
80345	Lead Analyzer K-shell	Drywall	4	1.400	0.04183
80345	MAP-3 K-shell (I)	Drywall	4	3.152	0.04183
80345	MAP-3 K-shell (II)	Drywall	4	2.246	0.04183
80345	Microlead I (I)	Drywall	4	4.100	0.04183
80345	Microlead I (II)	Drywall	4	3.700	0.04183
80345	XK-3 (II)	Drywall	4	1.600	0.04183
80407	XK-3 (I)	Metal	5	4.000	0.00044
80518	X-MET 880	Wood	6	0.173	0.00596
80541	MAP-3 K-shell (II)	Wood	6	-4.439	0.00063
80653	MAP-3 L-shell (II)	Wood	7	-0.901	0.02828
80720	MAP-3 K-shell (I)	Wood	8	5.781	1.04388
80720	MAP-3 K-shell (II)	Wood	8	5.321	1.04388
80720	Microlead I (I)	Wood	8	6.900	1.04388
80720	Microlead I (II)	Wood	8	7.100	1.04388
80750	XK-3 (I)	Brick	8	7.600	0.00353
80773	Lead Analyzer K-shell	Plaster	8	0.500	1.03873
80777	XK-3 (II)	Plaster	8	5.300	1.12200
80908	Lead Analyzer L-shell	Brick	10	0.960	1.66695
80908	MAP-3 L-shell (I)	Brick	10	1.728	1.66695
80908	X-MET 880	Brick	10	1.112	1.66695
80935	X-MET 880	Drywall	10	0.113	0.00128
80938	MAP-3 L-shell (I)	Concrete	10	0.203	0.00049

Table 6-38. Listing of Standard First Reading Outliers From Philadelphia.

ID NUMBER	XRF INSTRUMENT	SUBSTRATE	DWELLING	STANDARD FIRST PAINT	LABORATORY RESULT
81210	MAP-3 L-shell (I)	Metal	11	0.780	0.00482
81210	MAP-3 L-shell (II)	Metal	11	1.176	0.00482
81234	Lead Analyzer L-shell	Concrete	11	0.143	0.18387
81234	MAP-3 L-shell (I)	Concrete	11	0.315	0.18387
81234	X-MET 880	Concrete	11	0.136	0.18387
81255	Lead Analyzer K-shell	Plaster	11	0.900	0.05526
81316	Lead Analyzer L-shell	Wood	12	0.530	0.29780
81316	X-MET 880	Wood	12	0.454	0.29780
81340	X-MET 880	Plaster	12	0.160	0.30272
81342	X-MET 880	Plaster	12	0.139	0.23624
81348	Lead Analyzer K-shell	Metal	12	6.700	1.80351
81348	MAP-3 K-shell (I)	Metal	12	6.644	1.80351
81348	MAP-3 K-shell (II)	Metal	12	7.260	1.80351
81348	MAP-3 L-shell (II)	Metal	12	2.029	1.80351
81348	Microlead I (I)	Metal	12	7.000	1.80351
81348	Microlead I (II)	Metal	12	7.900	1.80351
81350	MAP-3 L-shell (I)	Metal	12	0.696	0.00673
81350	MAP-3 L-shell (II)	Metal	12	0.858	0.00673
81355	MAP-3 L-shell (I)	Metal	12	0.600	0.00359
81355	MAP-3 L-shell (II)	Metal	12	0.961	0.00359
81710	Microlead I (I)	Metal	16	5.300	0.33784
81723	Microlead I (I)	Wood	16	5.300	0.00921
81840	XK-3 (II)	Metal	17	3.200	0.26598
81944	MAP-3 K-shell (II)	Metal	18	-1.375	0.09021
81953	Microlead I (I)	Metal	18	6.100	0.10001

Some observations made from these tables are discussed below.

- For all first standard paint readings from all XRF instruments combined and within each substrate, readings on drywall contributed the greatest percentage to the outlier list. Sixteen out of a total 113 (14%) drywall readings from Denver and Philadelphia were identified as outliers. Nine percent (17 out of 189) of the metal readings in Denver and Philadelphia were identified as outliers followed by 7% of the 355 wood readings, 6% of the 222 plaster readings, 5% of the 93 brick readings, and 3% of the 218 concrete readings.

- Three sampling locations in Denver in dwelling number four accounted for thirteen of the sixteen total drywall outliers. Laboratory results for these three samples ranged from 0.00028 mg/cm^2 to 0.04183 mg/cm^2. These thirteen outliers resulted from readings that overestimated the actual lead level, and ranged from 0.900 to 4.100 mg/cm^2.

Twelve of the thirteen outliers were attributable to K-shell XRF readings.

Table 6-39. Summary Statistics for Outlier Data Points in the XRF Standard First Paint Readings and Their Associated Laboratory ICP Value Categorized by Instrument.

INSTRUMENT	SAMPLE SIZE	DATA SOURCE	MEAN	MAXIMUM	MINIMUM	25th PERCENTILE	MEDIAN	75TH PERCENTILE
Lead Analyzer K-shell	11	XRF	2.864	14.700	0.500	0.900	1.200	1.700
		Lab	0.827	5.751	0.000	0.028	0.055	1.039
Lead Analyzer L-shell	3	XRF	0.544	0.960	0.143	0.143	0.530	0.960
		Lab	0.716	1.667	0.184	0.184	0.298	1.667
MAP-3 K-shell	17	XRF	3.713	16.570	-4.439	2.246	3.152	5.321
		Lab	0.709	5.751	0.000	0.010	0.042	1.044
MAP-3 L-shell	15	XRF	0.484	2.029	-1.275	0.040	0.696	1.158
		Lab	0.251	1.804	0.000	0.002	0.005	0.050
Microlead I	20	XRF	4.505	7.900	1.900	3.050	4.400	5.700
		Lab	0.352	1.804	0.000	0.028	0.059	0.300
X-MET 880	11	XRF	0.250	1.112	0.055	0.113	0.142	0.173
		Lab	0.250	1.667	0.000	0.003	0.050	0.298
XK-3	7	XRF	3.786	7.600	1.000	1.600	3.800	5.300
		Lab	0.235	1.122	0.000	0.002	0.042	0.266

Table 6-40. Summary Statistics for Outlier Data Points in the XRF Standard First Paint Readings and Their Associated Laboratory ICP Value Categorized by Shell.

SHELL	SAMPLE SIZE	DATA SOURCE	MEAN	MAXIMUM	MINIMUM	25th PERCENTILE	MEDIAN	75TH PERCENTILE
K-shell	55	XRF	3.840	16.570	-4.439	1.700	3.332	5.300
		Lab	0.543	5.75	0.000	0.010	0.042	0.338
L-shell	29	XRF	0.402	2.029	-1.275	0.136	0.203	0.858
		Lab	0.299	1.804	0.000	0.004	0.007	0.236

Table 6-41. Frequency and Percent of First Standard Paint Readings Identified as Outliers per Substrate Categorized by Shell.

SHELL	STATISTIC	SUBSTRATE					
		Brick	Concrete	Drywall	Metal	Plaster	Wood
K-shell	Frequency	1	0	12	10	10	22
	Percent	1.1	0.0	10.7	5.3	4.5	6.2
L-shell	Frequency	4	7	4	7	3	4
	Percent	4.3	3.2	3.6	3.7	1.5	1.1
Combined	Frequency	5	7	16	17	13	26
	Percent	5.4	3.2	13.0	9.0	5.9	7.3

Table 6-42. Frequency and Percentage of Unique Sampling Locations From Which Standard First Paint Readings Were Taken and Identified as Outliers for Each Substrate and Categorized by Shell.

SHELL	STATISTIC	SUBSTRATE					
		Brick	Concrete	Drywall	Metal	Plaster	Wood
K-shell	Frequency	1	0	3	6	5	9
	Percent	1.1	0.0	2.7	3.2	2.3	2.5
L-shell	Frequency	2	4	3	4	3	3
	Percent	2.2	1.8	2.7	2.1	1.4	0.8
Combined	Frequency	3	4	5	9	8	12
	Percent	3.2	1.8	4.5	4.8	3.6	3.4

- Percentages of outliers from L-shell instruments from each substrate were similar. The percent of outliers from K-shell instruments from each substrate was more variable ranging from 0.0% (concrete) to 10.7% (drywall).

- Sixty-five percent of the standard first paint readings identified as outliers were taken by XRF instruments reporting K-shell results. The mean of these readings was 3.8 mg/cm^2. The mean of the outlier readings taken using L-shell instruments was significantly less with a reported value of 0.4 mg/cm^2.

- All 84 of the outliers were taken from 41 unique sampling locations. The K-shell instruments produced 55 outliers and the L-shell instruments produced 29 outliers.

- The 55 outliers attributable to the K-shell instruments occurred at 24 unique sampling locations. The 29 outliers produced by the L-shell instruments were taken from 19 unique sampling locations.

- Readings taken on only two sampling locations produced outliers from both K-shell and L-shell instruments. There were seven sampling locations for which four or more XRF instruments had outliers.

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6.4 ESTIMATION OF THE ACCURACY OF XRF MEASUREMENTS

The two fundamental aspects of XRF measurement accuracy are bias and variability in using XRF readings to infer the true level of lead in paint. Bias refers to a systematic tendency of the instrument to either underestimate or overestimate the true lead level. Variability refers to the fluctuation that the instrument exhibits in producing measurements on surfaces with the same level of lead in paint. The purpose of this section is to present estimates of the bias and standard deviation (a measure of variability) for the six XRF instruments considered in the study. This section addresses the following study objectives:

- To characterize the performance (precision and accuracy) of portable XRF instruments under field conditions;

- To evaluate the effect on XRF performance of interference from the material or substrate underlying the paint.

Designing analyses of the XRF data to meet these objectives raised a number of complex statistical issues. Laboratory ICP measurements were used as substitutes for the true lead levels in paint at the locations where XRF measurements were made. This substitution was prone to error from two sources:

(1) Spatial variation, which is a consequence of the fact that XRF measurements could not be made at exactly the same locations where paint specimens were collected for laboratory analysis;

(2) Laboratory error, which encompasses variation due both to the ICP instrument, and to the processing of paint samples prior to instrumental analysis.

Assessing the relationship of XRF measurements to the true levels of lead in paint, with only imperfect knowledge of the true lead levels, is a statistical estimation problem for which standard or elementary techniques are not designed. In addition, XRF instruments were evaluated in the full study under conditions where machines, operators, and other factors that may have affected measurements varied.

An attempt has been made to keep the narrative of this

chapter at an intuitive level, while recognizing that the issues raised do not always lend themselves to a terse or elementary treatment. For this reason, many of the technical details underlying the analyses have been placed in section 6.4.8 at the end of this chapter. The reader may find it helpful to refer to the more detailed treatment of statistical issues given there if the motivation or terminology for the analyses is not clear.

The organization of this section is as follows. Section 6.4.1 lays out the objectives of XRF data analysis in general terms. section 6.4.2 describes, in nontechnical terms, the methodology used to describe the relationship between XRF measurements and the level of lead, accounting for spatial variation and laboratory error in ICP measurements. From this relationship estimates of the bias and standard deviation of XRF readings at various levels of lead were obtained.

Section 6.4.3 describes the data that were used in the analyses, and explains why certain data from the pilot study, and data designated as outliers, were excluded.

Section 6.4.4 is a detailed narrative, by XRF instrument and substrate, of the XRF-true lead relationships estimated from the data. Estimates of model parameters, and of XRF bias and standard deviation at lead levels of 0.0 mg/cm^2 and 1.0 mg/cm^2, are presented. Eight measurement regimes derived from six distinct instruments are considered in separate subsections:

6.4.4.1 Lead Analyzer K-shell
6.4.4.2 Lead Analyzer L-shell
6.4.4.3 MAP-3 K-shell
6.4.4.4 MAP-3 L-shell
6.4.4.5 Microlead (ML) I
6.4.4.6 X-MET 880
6.4.4.7 XK-3
6.4.4.8 XL

The Microlead I and XK-3 are solely K-shell instruments, and the X-MET 880 and XL (as tested) are solely L-shell instruments, so that results on four instruments per shell are described. The highest level of aggregation attempted within each instrument type was that of substrate, six of which were represented in the study: brick, concrete, drywall, metal, plaster, and wood. Section 6.4.4 thus describes 48 "aggregate" analyses, with

additional analyses included at finer levels of detail where appropriate. At the end of each section a summary is provided to describe features of instrument performance that generalized across substrates.

In the analyses presented in section 6.4.4, the representative XRF measurement is taken to be the first nominal 15-second reading. Since three consecutive nominal 15-second readings were made in the full study, the question of whether to use the average of the three readings as the representative measurement was of interest. Section 6.4.5 gives a detailed explanation of why the use of the average did not result in a substantial improvement over the use of the first reading alone.

With the exception of the Lead Analyzer K-shell, the XRF instruments that were evaluated were prone to exhibit significant bias either generally, or under certain conditions. Section 6.4.6 considers the efficacy of correcting XRF readings for bias, using readings on NIST SRM films over control blocks, and over the substrate at sampled locations with paint removed.

Section 6.4.7 states conclusions in more detail and provides a summary for the entire section. Section 6.4.8 elaborates on methodological issues, including the development of the XRF measurement model.

6.4.1 Objectives of Data Analysis

The central focus of this chapter is to determine how accurately the XRF instruments measured the amount of lead in paint. As stated above, XRF accuracy consists of two components: bias and variability. Bias is quantified by the long-run average (or expected value) of XRF readings at a particular level of lead, minus the true level of lead. For example, if an instrument produced a large number of readings on the red NIST SRM film, which has a lead level of 1.02 mg/cm^2, and the average of these readings was 0.89 mg/cm^2, the estimated bias would be 0.89 - 1.02 = -0.13 mg/cm^2 at a true lead level of 1.02 mg/cm^2. The bias may change with the lead level, which was found to be true for all L-shell instruments, and possibly certain K-shell instruments as well.

Variability is quantified by the standard deviation (SD) of the readings obtained with an XRF instrument, at a fixed level of

lead. An instrument that is unbiased but has a large SD is not necessarily better than a biased instrument with a small SD. Like bias, the SD of an instrument was often found to vary with the lead level.

The bias and variability of an XRF instrument did not usually lend themselves to being resolved in a meaningful way to a single set of fixed numbers. These quantities often varied, not only with the level of lead, but also with the particular machine used, with the person operating the machine, and with other circumstances of measurement.

Estimation of the bias and variability of an XRF instrument from a long series of observations at fixed lead levels, on painted surfaces under field conditions, could not be done with the full study data. The lead levels obtained in the field samples were not known in advance, making replication of this kind impossible. The primary objective of the analyses presented in this chapter was to describe how an XRF instrument performed at various lead levels by means of a statistical model. From the model it was possible to obtain estimates of the bias and SD at a given level of lead in paint. Standard errors for estimates derived from the model were also estimated, which facilitates the derivation of approximate confidence intervals and hypothesis testing.

Two levels of lead were of particular interest: 0.0 mg/cm^2 (the absence of lead in paint), and 1.0 mg/cm^2. Estimates of the bias and standard deviation at these two levels of lead were compared to control block summary statistics for readings made on bare substrate, and on red NIST SRM film placed over bare substrate.

The model used for this purpose is discussed in the following section. Alternatives to the use of a model, including nonparametric estimation, were not capable of making inferences about the XRF-true lead relationship based on the observable XRF-ICP relationship, accounting for imprecision due to using ICP measurements as substitutes for the true lead levels. In spite of this limitation, nonparametric methods were used to assess important aspects of model fit, and are discussed in the narrative.

6.4.2 The XRF Measurement Model

A statistical model was developed to describe the relationship of XRF instrument readings to the true lead levels, recognizing that the relationship cannot be perfectly descriptive, due to the presence of factors such as instrumental error. The model served two purposes: it gave estimates of quantities related to XRF instrument performance at various lead levels, and it gave an overall description of how XRF and lead levels were related.

The selection of an appropriate model was complicated by the fact that the true lead levels in the field samples were not known. Since the combined effect of spatial variation and laboratory error in ICP measurements was usually small relative to the range of lead levels represented in a sample, deducing basic model attributes from the observable XRF-ICP relationship appeared to be reasonable. A model for describing XRF performance without taking this type of imprecision into account was developed first. Nonparametric estimates were also derived, permitting an assessment of model attributes using graphical tools. Finally, provision for the substitution of ICP measurements for true lead levels was made, giving the *XRF measurement model*, upon which subsequent analyses, including those presented in section 6.4.4, were based.

6.4.2.1 Basic Model Attributes

The top graph of Figure 6-1 is a scatterplot of XRF versus ICP measurements for one of the K-shell instruments evaluated in the full study. The 294 readings represented in the scatterplot were made by one operator, using one machine, on one substrate type, in one city. This scatterplot illustrates several important attributes of the XRF-ICP relationship, and perhaps of the XRF-true lead relationship as well:

1. An essentially linear relationship between XRF and ICP measurements is evident.
2. The variability of XRF values increases as the ICP level increases (a condition known as *heteroscedasticity*, or nonconstant variance).
3. The distribution of ICP measurements is heavily weighted towards lower values, which might suggest a logarithmic transformation of both the XRF and ICP

measurements to preserve linearity; but,

4. A logarithmic transformation of XRF measurements is not possible, because zero and even negative values are present, which in part explains why they appear, in this example, to be nearly unbiased when the ICP level approaches zero, while at the same time exhibiting variance that remains substantial.

It is interesting to note that of the 294 ICP measurements, which ranged from nearly 0.0 mg/cm^2 to over 30 mg/cm^2, 54 (nearly one-fifth) were less than 0.01 mg/cm^2. The bottom plot of Figure 6-1 reveals more detailed information about the distribution of XRF readings at low levels of lead. It is a histogram, showing how XRF readings corresponding to the 54 ICP measurements less

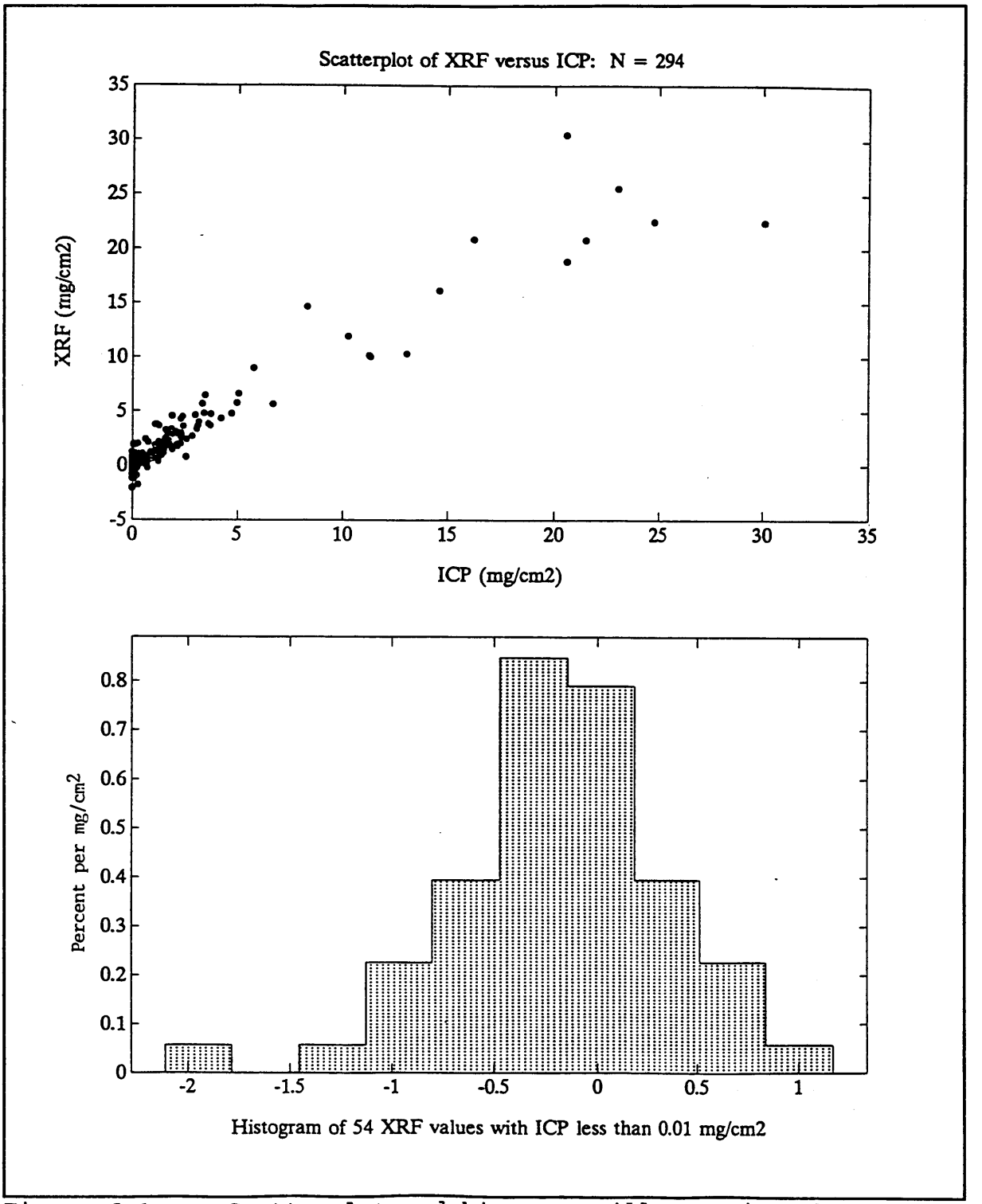


Figure 6-1. Scatterplot and histogram illustrating the XRF-ICP relationship.

than 0.01 mg/cm^2 were distributed across 10 equally spaced subintervals. The shape of the histogram suggests a normal curve, and it reaffirms that negative XRF readings with this instrument were not uncommon.

In section 6.4.8.2.1 a simple model is described that captures the attributes of the above example. There are two components to the model: a *response component* that mathematically describes the mean XRF reading at a particular level of lead, and a *SD component* that describes the variation in XRF readings as a function of the lead level. These two components are determined from the four model parameters, denoted a, b, c and d. The XRF response is a linear function of the lead level, given by $a + b\cdot(\mathrm{Pb})$, where Pb refers to the lead level in mg/cm^2, and is measured by ICP. The SD is a nonlinear function of the lead level, given by the quantity $[c + b\cdot(\mathrm{Pb})^2]^{1/2}$. As the lead level increases, the SD more closely resembles a linear function. The four model parameters were estimated from the data using maximum likelihood under an assumption that XRF readings are normally distributed at fixed lead levels.

The model is essentially a weighted regression, with a weight function (given by the reciprocal of the squared SD) that is also estimated from the data. Large, apparently influential observations that have correspondingly large SD estimates were assigned smaller weights in the regression. Since most of the lead levels were clustered at lower values where the SD was also smallest, these data usually had the greatest influence in determining the model estimates.

6.4.2.2 Nonparametric Estimation

It was also possible to derive estimates of the response and SD of XRF readings that did not rely on the development of a statistical model. These nonparametric estimates assumed only that the mean XRF reading was a nondecreasing function of the lead level, and that its standard deviation was also nondecreasing. Monotone regression was used to produce estimates meeting both conditions. It was also used to develop an outlier criterion for the XRF data. Monotone regression, and its use in describing XRF performance, is described in section 6.4.8.2.3.

If the model described in section 6.4.2.1 is an appropriate choice for the data, the monotone regression and model estimates

should be similar, although they cannot be identical. One respect in which these two estimates always differ is that the model produces smooth, continuous functions as estimates of the response and SD components, while monotone regression produces "step functions" that are not smooth in appearance. Monotone regression was used primarily as a graphical tool for evaluating model performance, and for estimating the performance of an instrument where model estimates were not reliable.

Unlike the model, there is no clear way to adapt the nonparametric estimates to account for spatial variation and laboratory error in ICP measurements. Monotone regression estimates were based on the *observable* XRF-ICP relationship, which again is not the same as the *unobservable* relationship of XRF readings to the true lead level. Where the model was clearly inadequate for describing critical aspects of XRF performance, the nonparametric estimates may be preferred in spite of this shortcoming. One case where this may be true concerns the XL instrument, which is described in section 6.4.4.8.

6.4.2.3 Model Versus Nonparametric Estimation: Illustration

Figure 6-2 shows graphically how the model and nonparametric estimates compare, using the example illustrated in Figure 6-1. In the top graph, the estimated model response function $a + b\cdot(\mathrm{Pb})$, with ICP representing the lead level Pb, is plotted as a solid line, where $a = -.027$, and $b = 1.235$. The nonparametric (monotone regression) estimate is plotted with dashed lines. Individual data are plotted with large dots. At ICP = .008 mg/cm^2 (the largest ICP measurement less than .01 mg/cm^2), the model estimate of the bias is $-.027 + 1.235\cdot(.008) = -.017$ mg/cm^2, compared to a nonparametric estimate of -.182 mg/cm^2. In absolute terms, the two estimates of the bias are close, with a difference of .165 mg/cm^2. At ICP = 1.0 mg/cm^2 the model estimate is 1.208 mg/cm^2, compared to a nonparametric estimate of 1.275 mg/cm^2, again reflecting the close agreement between the model and nonparametric response function estimates.

The bottom graph shows the SD estimated by the model (solid line) and by the nonparametric technique (dotted line). Agreement between the two estimates is close for lead levels smaller than about 5.0 mg/cm^2. The model SD is the square root

of the quantity $c + d \cdot (\mathrm{Pb})^2$. Substituting $c = .309$ and $d = .116$, at ICP = .008 mg/cm^2 the model SD estimate is given by the square root of $.309 + .116 \cdot (.008)^2$, or .556 mg/cm^2. At ICP = 1.0 mg/cm^2 the estimate is the square root of $.309 + .116 \cdot (1.0)^2 = .652$ mg/cm^2. These are compared to nonparametric estimates of .464 mg/cm^2 and .634 mg/cm^2 respectively.

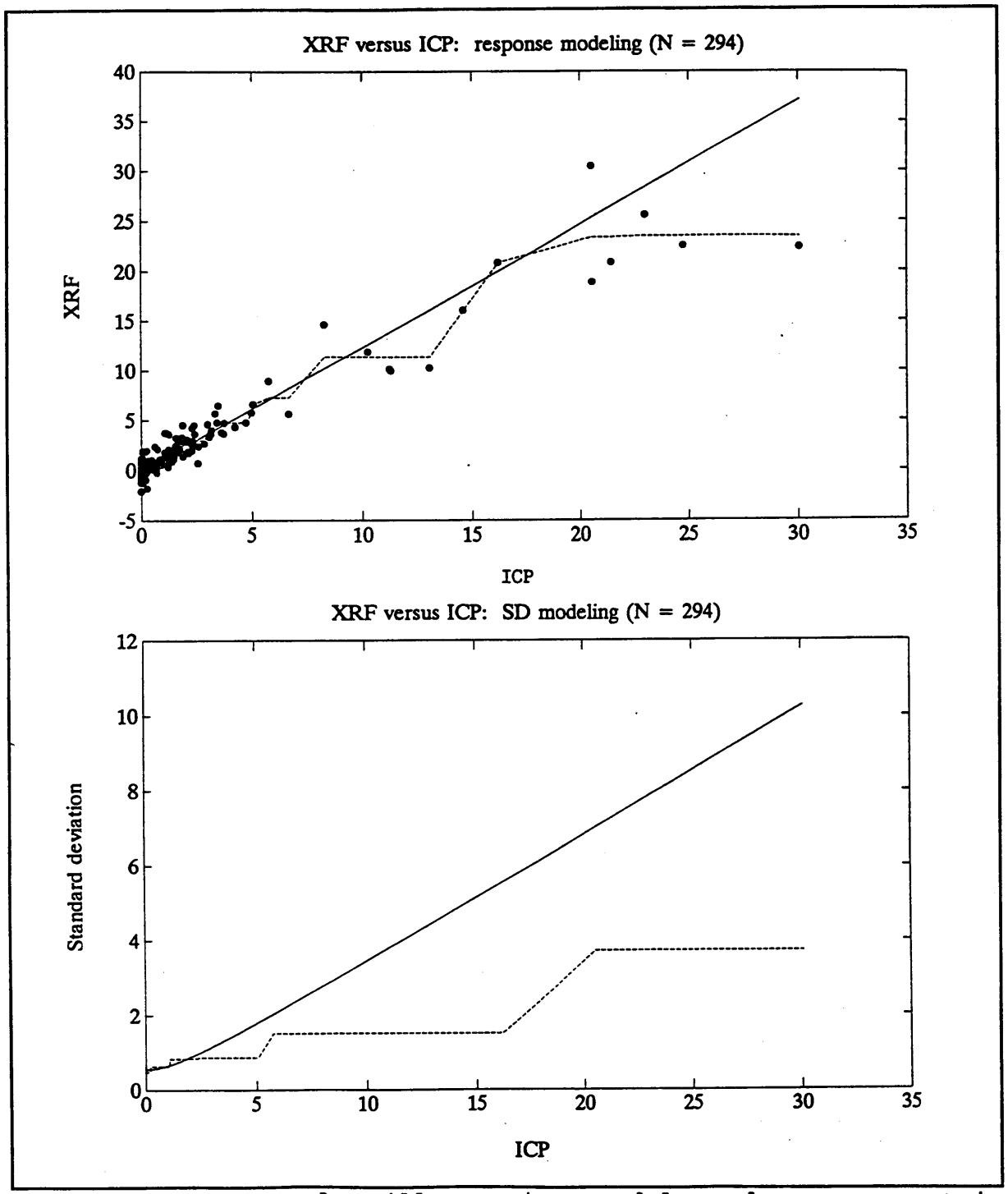


Figure 6-2. Example illustrating model and non-parametric estimation of XRF performance. Solid lines are model estimates. Dashed lines are nonparametric (monotone regression) estimates.

In section 6.4.4, the question of model fit is considered separately for each instrument-substrate combination, using both model-based and nonparametric estimation. In most instances the model fits the data well, possibly by restricting the analysis to data in a limited ICP range where the model appears to capture the important aspects of XRF performance.

6.4.2.4 Accounting for Spatial Variation and Laboratory Error in ICP Measurements

In the above example, no provision was made for the imprecise substitution of ICP measurements for true lead levels. The underlying assumption is that if a model having the stated form accurately describes the XRF-ICP relationship, then a model of a similar form is appropriate for the XRF-true lead relationship. This assumption is valid provided that the effect caused by the substitution was small relative to the range of lead levels represented in the data.

The XRF measurement model has the same form as the model described in section 6.4.2.1, except that it makes provision for the combined effect of spatial variation and laboratory error in ICP measurements. The rationale for and development of the XRF measurement model is given in sections 6.4.8.2.4 through 6.4.8.2.6.

6.4.2.5 Interpretation and Comparison of Model Estimates

All four parameters of the XRF measurement model have important meanings in describing the performance of an XRF instrument:

- Parameter *a* is the intercept, and is compared to a value of 0.0 to determine if the instrument produced unbiased readings in the absence of lead;

- Parameter *b* is the slope, and is compared to a value of 1.0 to determine if the instrument responded in a proportionate way to changes in the lead level;

- Parameter *c* is the variance (standard deviation squared) of XRF readings at a lead level of 0.0 mg/cm^2;

- Parameter *d* measures the homogeneity of variance as the lead level increases, and is compared to a value of 0.0 to determine if the variability of XRF measurements remained constant as the lead level changed.

The slope coefficient *b* measures the change in the average XRF reading resulting from a change of 1.0 mg/cm^2 in the lead level. An instrument is *proportionately responsive* to the lead level if *b* is equal to 1.0, it is *under-responsive* if *b* is less than 1.0, and it is *over-responsive* if *b* is greater than 1.0. Only instruments that are proportionately responsive to the lead level and have intercept coefficients equal to 0.0 give unbiased readings at all lead levels. Under-responsive instruments with *a* approximately equal to 0.0, which was typical of the L-shell instruments in the full study, have negative biases that become more pronounced as the lead level increases. The K-shell instruments were generally proportionately responsive, with biases approximately equal to the intercept coefficients.

Hypothesis testing can be performed by constructing confidence intervals with the parameter estimates and their estimated standard errors. Adding and subtracting 2 times the standard error corresponds to a confidence level of about 95 percent. Although use of the 95 percent confidence level is widely accepted, many in the statistics community recommend a more conservative criterion, such as the 99.7 percent confidence level corresponding to an interval width of 3 times the standard error. There are several reasons why a more conservative criterion may be preferred:

1. Multiple inferences. As the number of confidence intervals simultaneously considered increases, the number of expected instances where the confidence interval fails to cover the quantity of interest increases proportionately. Out of every hundred 95 percent confidence intervals, for instance, 5 failures can be expected. A more conservative criterion can sharply reduce the number of expected failures when multiple inferences are made.

2. Lack of model fit. The models presented in section 6.4, like most statistical models, are approximatations. Wider confidence intervals allow greater leeway for effects due to imperfection of the model.

3. Sampling effects. The study data were not, as a matter of necessity, obtained from simple random samples. Clustering by unit, paint type, machine, or operator may cause standard error estimates to be understated. A more conservative criterion allows greater leeway for this effect.

To illustrate, suppose that an estimated model has b = 1.25 with a standard error = 0.08. A 95 percent confidence interval is given by 1.25 ± 2·(.08), or 1.09 to 1.41. Using a width of 3 standard errors gives an interval of 1.01 to 1.49. Neither of these two intervals contains 1.0, leading to the conclusion that the instrument was marginally over-responsive to changes in the lead level. Use of the wider confidence interval gives additional leeway for the factors mentioned above. In marginal cases such as the present example, even the failure of the wider confidence interval to contain 1.0 should not be regarded as strong evidence that the instrument is inherently over-responsive.

Another kind of comparison that is of interest concerns estimates derived from different groups of data. Where the XRF measurement model was fit to nonoverlapping groups of data delineated by machine, operator, or other factors of interest, a comparison of the estimated model parameters could be made to infer whether or not significant differences were evident. This was be done by computing a chi-square statistic based on joint differences of the four estimated model parameters a, b, c, and d. It is possible to limit the chi-square statistic to certain parameters, such as a and b dealing with the response function. The chi-square statistic has the same number of degrees of freedom as the number of parameters used in its calculation.

Statistical tests were used in a limited way to assess differences between models that were estimated on distinct groups of data defined by a certain factor, such as machine or city. The use of statistical testing for this purpose should not be regarded as a panacea for disposing of what are, in actuality, very complex issues. The models do not explain the data perfectly, and effects due to machines, operators, and cities were confounded with lead levels to various degrees. These reasons alone make statistically significant results all but certain with large samples. Conclusions regarding significant results should take the magnitude of the effect into account, and seek confirmation from other sources, such as the control block

data.

6.4.2.6 Comparison to Control Block Data

Summaries of control block data are presented along with the results of model estimation, by instrument and substrate type, in section 6.4.4. Estimates of the bias and SD are given for the first nominal 15-second readings made on bare substrate (0.0 mg/cm^2), on red NIST SRM film (1.02 mg/cm^2), and on yellow NIST SRM film (3.53 mg/cm^2). Because a large number of readings were made for each of these fixed, precisely measured lead levels, it was not necessary to use a model in order to obtain estimates. The SD estimates reported are sample standard deviations, calculated both separately by machine, and combined over groups of machines where appropriate. The bias estimates reported are sample average readings minus the lead level of the film (or bare substrate) used.

6.4.3 Data Used in Analyses

The ICP measurement of the primary paint-chip sample, expressed in area (mg/cm^2) units, was used to represent the true lead level of the field sample. The representative XRF measurement used in the analyses presented in section 6.4.4 was the first nominal 15-second reading. The use of the average of three successive readings as the representative XRF measurement is discussed in section 6.4.5, and the use of the first reading corrected for bias is discussed in section 6.4.6. Data from the full study (Denver and Philadelphia) were the primary focus of the analyses.

6.4.3.1 Use of the Louisville Pilot Data

Data from the Louisville pilot study consisted of readings taken with a limited set of instruments: X-MET 880, XK-3, MAP-3, and Microlead I. The X-MET 880 in Louisville used a Cd^{109} source with a strength of 5 mC dated August 1992, while the X-MET 880 in Denver and Philadelphia used a Cm^{244} source with a strength of 100 mC dated September 1991. The MAP-3 in Louisville performed one standard measurement using a nominal 60-second reading, while the MAP-3 in Denver and Philadelphia performed three standard measurements using nominal 15-second readings. Also, the MAP-3 in Louisville truncated its readings at 0.0 mg/cm^2, unlike the Denver and Philadelphia MAP-3 machines, which gave negative

readings for both the K- and L-shells. Hence, only the Microlead I and XK-3 were comparable between the full and pilot studies.

Given the small sample sizes obtained from the pilot study when substrate detail was considered (100 observations per instrument, 33 on wood being the largest substrate sample size), the gain from combining the pilot data with the full study data was not great, with the possible exception of wood substrate analyses. Pilot study data were combined with the full study data for certain Microlead I and XK-3 analyses. X-MET 880 analyses on metal, plaster and wood for the pilot data were conducted to illustrate their differences from the full study data.

6.4.3.2 Outliers

Section 6.3 describes the methodology used for identifying outliers in the XRF data. Data identified as outliers were omitted from the analyses reported in section 6.4. Only 84 observations were designated as outliers, which was slightly more than one half of one percent of the data available for analysis. Other "borderline cases" were occasionally identified in the analyses reported in section 6.4.4. Since outliers are not cut-and-dried phenomena, discretion was used in deciding whether borderline cases should be used in reported results.

6.4.3.3 The Treatment of Non-detects

Of the 1,290 sample locations where paint chip samples were analyzed for lead content, 54 (4.2%) had ICP measurements below the detection limit. Drywall samples were the most affected, with 16 out of 124 (12.9%) samples classified as non-detects.

For an ICP measurement classified as a non-detect, the lead level of the sample is estimated to be no greater than the recorded detection limit. A detailed explanation of non-detects and the meaning of the detection limit is presented in Chapter 4.

Analyses of the field sample data reported in section 6.4 used the detection limit as the ICP measurement in cases where non-detects were encountered. Since the lead level of a non-detect paint chip sample is indicated to be very low, the choice of the detection limit, or any other small value consistent with the designation of the sample as a non-detect, had a negligible effect on estimates relating the performance of an XRF instrument to the lead level.

6.4.3.4 Control Block Data

Beginning, ending, and continuing control block readings were made on bare substrate (0.0 mg/cm^2), and on red (1.02 mg/cm^2) and yellow (3.53 mg/cm^2) NIST SRM films placed over the substrate. Studying the performance of an instrument on the control blocks could, in principle, give an idea of how the instrument responded to different levels of lead, with full knowledge of the actual lead levels.

Summary statistics for the control block data are included as part of the analyses, although with several caveats. The control block data reflected *instrumental sources of variability* only, while in practice other sources of variability affected XRF performance to a greater degree. The level of operator intervention needed to make XRF readings differed between the field samples and the control blocks. On the field samples, the XRF instruments had to be repositioned from location to location, at differing heights, angles, and surface textures. These factors, which were relatively constant between successive control block readings, may represent contributions to the variability of field sample data that are not present in control block data.

The control block data were also used in attempting to correct XRF field sample measurements for bias. The efficacy of bias correction is discussed in section 6.4.6.

6.4.4 XRF Estimation: Presentation of Results

This section presents estimates pertaining to the accuracy of XRF measurements that were derived for each of the six substrates encountered within each of the eight instrument classes evaluated. Each of the 48 narratives is organized as follows:

- Sample breakdown by machine, operator, and city, and the identification of outliers;
- Matched pair analysis for instruments having two field classifications;
- Graphical evaluation of response and SD modeling, which does not account for the imprecise substitution of ICP measurements for true lead levels;
- Presentation of XRF measurement model estimates, with comparison to estimates based on control blocks.

Graphical information is presented in the same format used in Figure 6-2, consisting of separate plots for the response and SD functions. In the top plot, the response function estimated by the model (the mean XRF measurement at a fixed ICP level) is graphed as a solid line. The dashed line is the monotone regression of XRF on ICP measurements, which does not depend on a particular model form. The data are scatterplotted with large dots. In the bottom plot, the SD of XRF measurements, estimated by the model as a function of the ICP measurement, is graphed as a solid line, and a nonparametric SD estimate based on monotone smoothing of squared residuals is graphed as a dashed line.

As stated above, the assumption underlying the use of these plots for diagnostic purposes is that basic issues of model fit can be addressed with the observable XRF-ICP relationship, notwithstanding the fact that the unobservable XRF-true lead relationship is the subject of interest. This assumption appears to be reasonable.

Results are presented, where possible, for subgroupings that are homogeneous with respect to factors (such as operators and machines), provided that there are at least 25 observations in the subgrouping. Machines are referred to by XRF code numbers. Human operators are referred to by operator code letters.

Standard errors of estimates from the XRF measurement model

and from the control block data are shown in parentheses beneath the estimates. Beginning, end of day, and continuing control block readings were used. The first of three nominal 15-second control block readings was taken as the representative measurement, in order to facilitate comparison with the field sample data. Section 6.4.2.5 explains how model estimates can be used to draw conclusions about the performance of an instrument. The propriety of pooling data across factors was generally assumed, even in light of statistically significant differences, unless a distorted picture of how the instrument can be expected to perform would emerge as a result.

Duplicate sets of readings were made with the MAP-3 (K- and L-shells), Microlead I, and XK-3 instruments at all sample locations in the full study. The term *field classification* is used to describe one full set of such readings across all sample locations. The analysis of field classified data, including the sign tests and Fisher's exact test that were used, is described in section 6.4.8.3.

Results of statistical tests are presented in the form of p-values, expressed as percentages. For example, a p-value of 0.01 percent is the same as 0.0001.

6.4.4.1 Results for Lead Analyzer K-shell

Data for the Lead Analyzer K-shell were obtained in Denver and Philadelphia (not Louisville). Two machines (1 and 2) were used by the one operator (A) of this instrument type. Machine 1 was used in both Denver and Philadelphia, and Machine 2 was used in Philadelphia only. It is not possible to attribute effects to the operator, but comparisons between cities and between instruments within operator can be made for some substrates.

6.4.4.1.1 Lead Analyzer K-shell on Brick

There were 93 observations of the Lead Analyzer K-shell on brick, none of which were designated as outliers. Readings on Machine 1 were made 87 times, 81 in Denver and 6 in Philadelphia. Readings on Machine 2 were made 6 times, all in Philadelphia. There were too few data to meaningfully fit the XRF measurement model to Machine 2 or to Philadelphia data separately.

Figure 6-3 shows the response and SD components of the XRF

data fit to ICP measurements, before accounting for the combined effect of spatial variation and laboratory error. The ICP range is heavily clustered toward smaller values, with values above 5.0 mg/cm^2 somewhat separated, and possibly following a different relationship. The nonparametric response flattens out at about 5.0 mg/cm^2, and the model may not be reliable beyond that point. Since there were no locations with ICP measurements between 0.8042 mg/cm^2 and 4.6567 mg/cm^2 that gave usable data, it is impossible to tell where or how the relationship may have changed. Inference at the 1.0 mg/cm^2 lead level must therefore be approached with caution. Both SD estimates appear to capture the nonconstant XRF variability, and the model suggests higher variability as the lead level increases.

Table 6-43 gives the results of fitting the XRF measurement model, taking into account the imprecise substitution of ICP measurements for true lead levels. The fit to the 81 readings of Machine 1 in Denver may give the best indication of how well the Lead Analyzer K-shell performed on brick. The addition of the six Philadelphia readings degraded the fit somewhat, possibly due to the fact that three of these had high XRF readings in the low ICP range.

Comparing bias and SD estimates at 0.0 mg/cm^2 and at 1.0 mg/cm^2 to the control block summary reported in Table 6-44 suggests that, for Machine 1, variability in addition to machine error was exhibited in the field sample measurements as the lead level increased. Bias estimates differ markedly at about 1.0 mg/cm^2, with a downward bias suggested by the model that is not evident in the control block data. But this may be due to poor model fit at that lead level.

Restricting the analysis to ICP measurements less than 1.0 mg/cm^2 reduced the estimated bias to about -0.09 (0.08) mg/cm^2, which is not statistically significant and is more in line with the control block estimates. The estimated SD, however, was reduced to 0.109, which does not agree with the control block

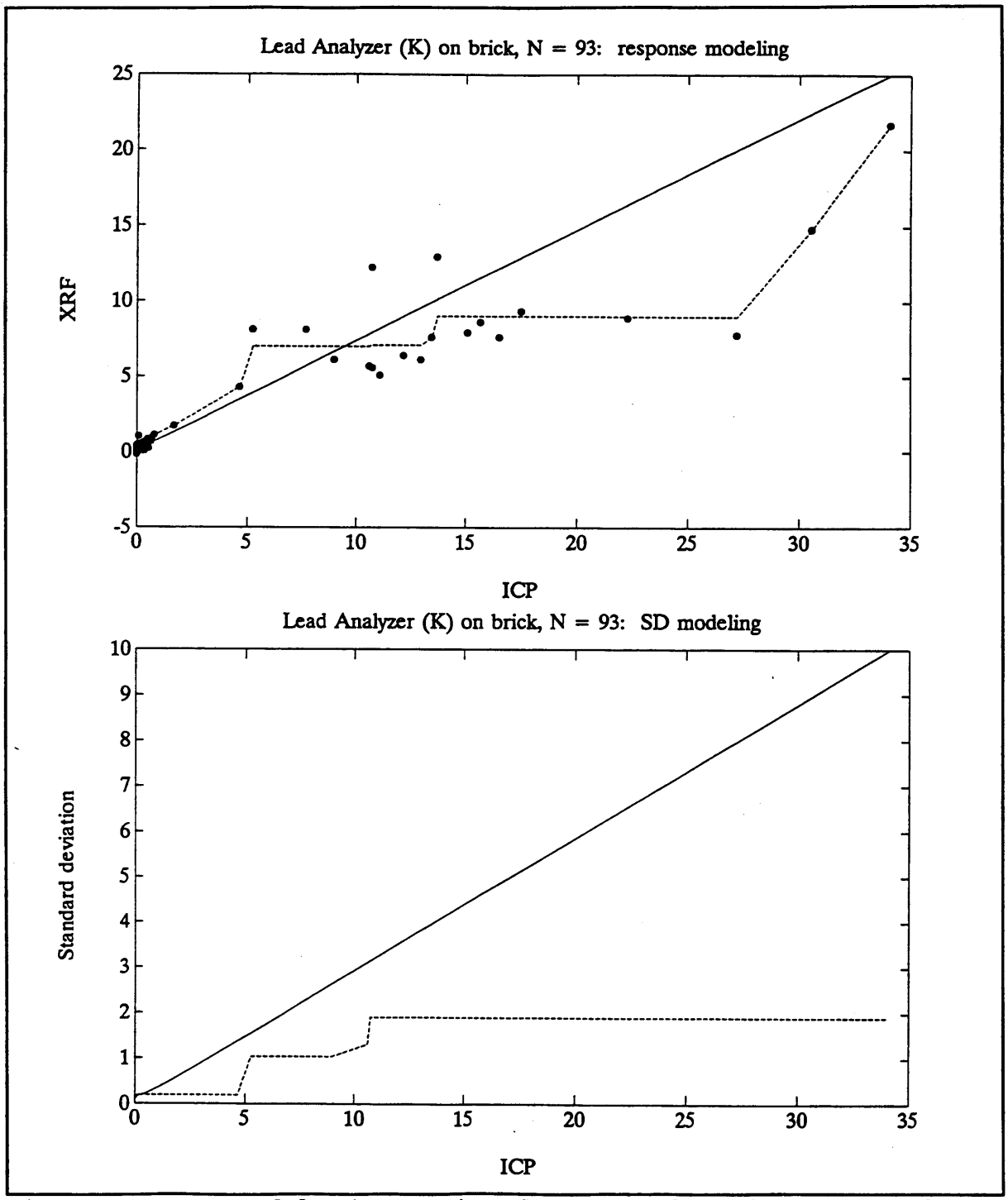


Figure 6-3. Model Diagnostic Plots, Lead Analyzer K-shell on brick. Solid lines are model estimates. Dashed lines are nonparametric (monotone regression) estimates.

Table 6-43. Lead Analyzer K-shell on Brick: Model Estimates.

DEVICE	SAMPLE SIZE	MODEL PARAMETERS				Pb=0.0 mg/cm²		Pb=1.0 mg/cm²	
		a	b	c	d	BIAS	SD	BIAS	SD
Machine 1, Denver	81	0.055 (.017)	0.720 (.053)	0.012 (.003)	0.030 (.019)	0.055 (.017)	0.107	-0.225 (.049)	0.204
Machine 1, Denver and Philadelphia	87	0.080 (.024)	0.702 (.055)	0.028 (.006)	0.023 (.017)	0.080 (.024)	0.167	-0.219 (.050)	0.226
Machines 1 & 2	93	0.084 (.023)	0.703 (.055)	0.030 (.006)	0.026 (.017)	0.084 (.023)	0.173	-0.213 (.051)	0.229

Table 6-44. Lead Analyzer K-shell on Brick: Control Block Summary.

DEVICE	SAMPLE SIZE	BARE (0.0 mg/cm²)		RED NIST SRM (1.02 mg/cm²)		YELLOW NIST SRM (3.53 mg/cm²)	
		BIAS	SD	BIAS	SD	BIAS	SD
Machine 1	51	0.044 (.015)	0.108	0.080 (.028)	0.197	0.107 (.087)	0.476
Machine 2	12	0.071 (.036)	0.126	0.080 (.104)	0.362	0.087 (.076)	0.262
Machines 1 and 2	63	0.049 (.014)	0.111	0.080 (.030)	0.235	0.103 (.056)	0.445

data.

6.4.4.1.2 Lead Analyzer K-shell on Concrete

There were 218 observations of the Lead Analyzer K-shell on concrete, none of which were designated as outliers. Readings on Machine 1 were made 179 times, 98 in Denver and 81 in Philadelphia. Readings on Machine 2 were made 39 times, all in Philadelphia.

Figure 6-4 shows the response and SD components of the model as fit to ICP measurements before provision for the combined effect of spatial variation and laboratory error. The model fit appears to be good with respect to both estimated components, especially for ICP measurements less than about 4.0 mg/cm^2. Table 6-45 gives the results of fitting the XRF measurement model to the data under various subgroupings, and pooled. Table 6-46 reports control block results by machine, and pooled.

There is little indication of either city or machine effects. Chi-square statistics on the four parameters (4 degrees of freedom) had p-values in excess of 10 percent comparing Denver and Philadelphia within Machine 1, and Machines 1 and 2 within Philadelphia. The pooled results (N = 218) indicate how the instrument performed overall. The control block summary likewise does not show significant machine effects.

Neither the model nor the control blocks indicate serious bias. At the 0.0 mg/cm^2 lead level, the model SD (0.114 mg/cm^2) and bare control block SD (0.111 mg/cm^2) are very close, but the model SD estimates are larger at the 1.0 mg/cm^2 lead level. This may reflect non-instrumental sources of variability present in the field sample data, but not in the control blocks.

6.4.4.1.3 Lead Analyzer K-shell on Drywall

There were 113 observations of the Lead Analyzer K-shell on drywall, 2 of which were designated as outliers (80343 and 80345), leaving 111 observations used in estimation. All readings were made by Machine 1, 103 in Denver and 8 in Philadelphia. Testing machine or city effects by fitting separate models was not possible.

Figure 6-5 shows the response and SD components of the

estimated model before provision for the combined effect of spatial variation and laboratory error in ICP measurements. Both appear to agree with the nonparametric estimates reasonably well.

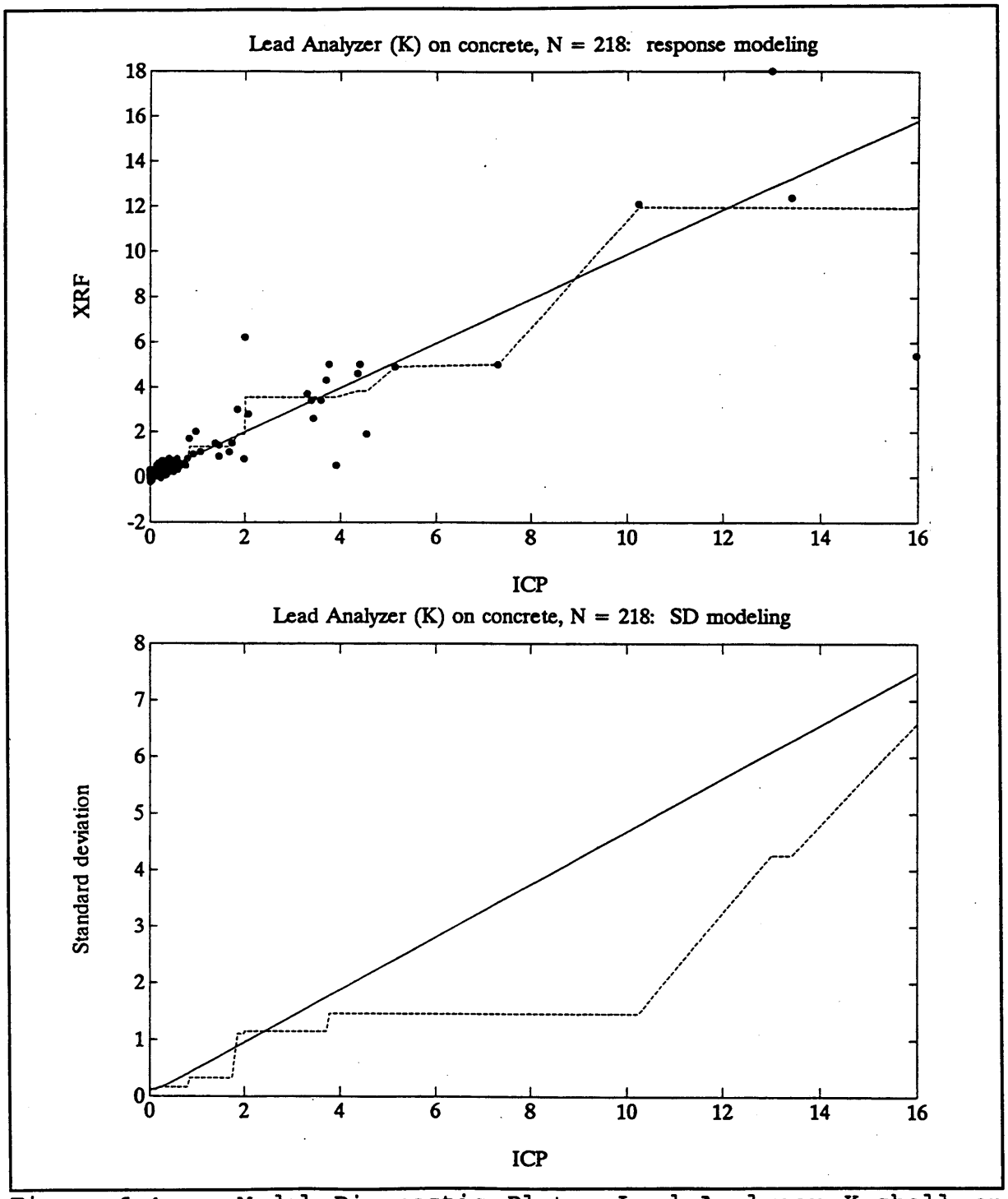


Figure 6-4. Model Diagnostic Plots, Lead Analyzer K-shell on concrete. Solid lines are model estimates. Dashed lines are nonparametric (monotone regression) estimates.

Table 6-45. Lead Analyzer K-shell on Concrete: Model Estimates.

DEVICE	SAMPLE SIZE	MODEL PARAMETERS				Pb=0.0 mg/cm²		Pb=1.0 mg/cm²	
		a	b	c	d	BIAS	SD	BIAS	SD
Machine 1, Denver	98	0.017 (.015)	1.066 (.102)	0.014 (.003)	0.183 (.070)	0.017 (.015)	0.118	.083 (.100)	0.444
Machine 1, Philadelphia	81	-0.007 (.021)	0.964 (.076)	0.011 (.003)	0.087 (.041)	-0.007 (.021)	0.105	-0.043 (.070)	0.313
Machine 1, Denver and Philadelphia	179	0.010 (.012)	0.974 (.058)	0.013 (.002)	0.121 (.033)	0.010 (.012)	0.113	-0.016 (.053)	0.366
Machine 2, Philadelphia	39	0.066 (.036)	0.865 (.147)	0.016 (.007)	0.092 (.090)	0.066 (.036)	0.127	-0.069 (.122)	0.328
Machines 1 & 2	218	0.017 (.012)	0.972 (.054)	0.013 (.002)	0.124 (.032)	0.017 (.012)	0.114	-0.011 (.049)	0.371

Table 6-46. Lead Analyzer K-shell on Concrete: Control Block Summary.

DEVICE	SAMPLE SIZE	BARE (0.0 mg/cm²)		RED NIST SRM (1.02 mg/cm²)		YELLOW NIST SRM (3.53 mg/cm²)	
		BIAS	SD	BIAS	SD	BIAS	SD
Machine 1	65	-0.013 (.014)	0.110	0.045 (.029)	0.233	0.194 (.054)	0.432
Machine 2	12	-0.011 (.032)	0.112	0.155 (.072)	0.249	0.162 (.161)	0.558
Machines 1 and 2	77	-0.013 (.013)	0.111	0.062 (.027)	0.235	0.189 (.052)	0.453

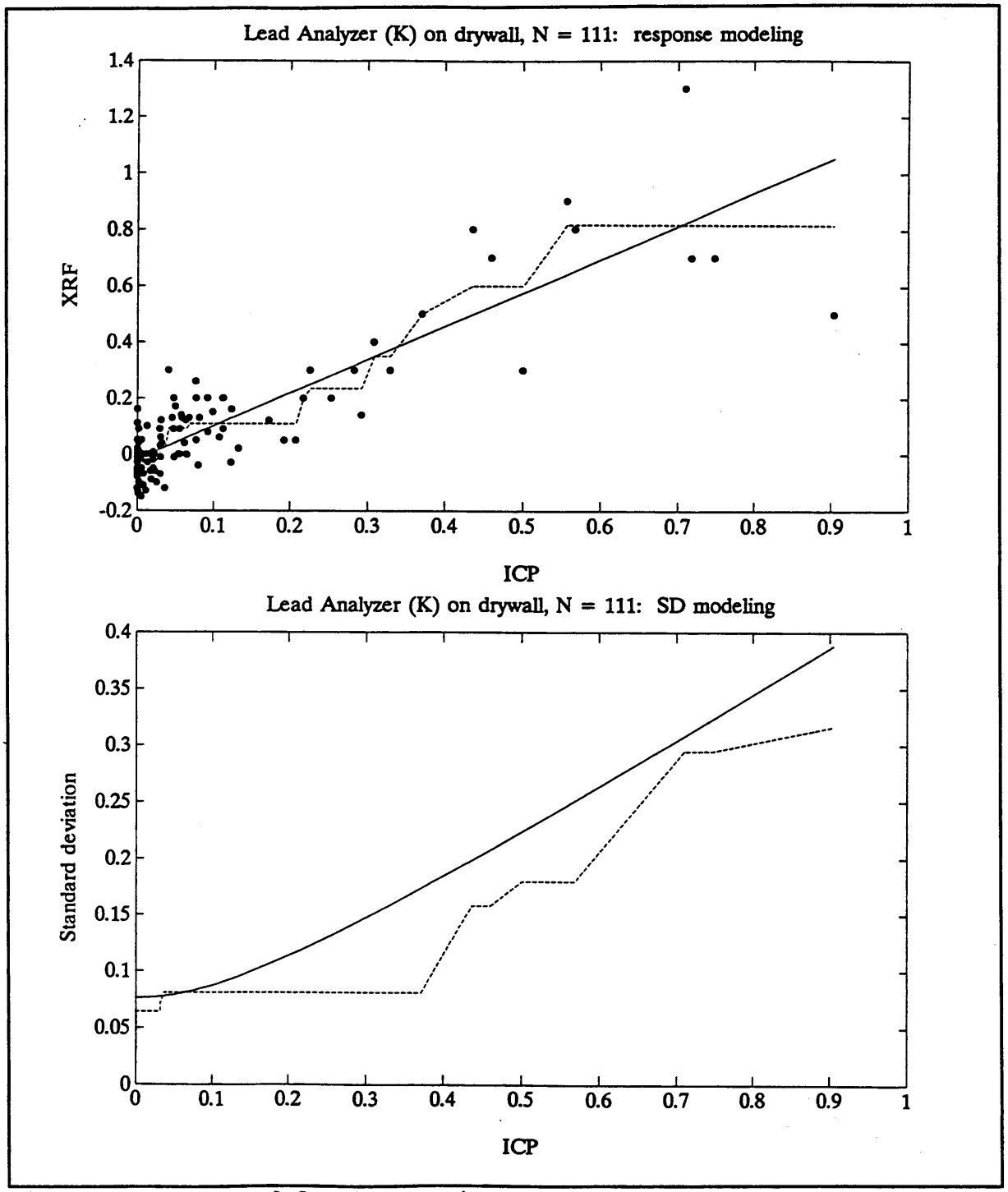


Figure 6-5. Model Diagnostic Plots, Lead Analyzer K-shell on drywall. Solid lines are model estimates. Dashed lines are nonparametric (monotone regression) estimates.

It should be noted that all ICP measurements were less than 1.0 mg/cm^2, making it difficult to infer XRF performance at that lead level.

Table 6-47 gives the results of fitting the XRF measurement model to only the Denver data, and to data from both cities combined. The inclusion of the 8 Philadelphia observations did not greatly change the estimated quantities. Control block results are given in Table 6-48. Neither the models nor the control block data suggest that the instrument was prone to bias at lead levels as high as 1.0 mg/cm^2. At 3.53 mg/cm^2 the instrument exhibited small, positive bias on the control blocks. Comparing SD estimates confirms a pattern seen across all substrates with this instrument where the model and control block SD estimates agree closely at 0.0 mg/cm^2, but with the model suggesting higher variability as the lead level increases.

6.4.4.1.4 Lead Analyzer K-shell on Metal

There were 189 observations of the Lead Analyzer K-shell on metal, one of which was designated as an outlier (81348), leaving 188 observations for analysis. Readings on Machine 1 were made 140 times, 62 in Denver and 78 in Philadelphia. All 48 of the Machine 2 readings were made in Philadelphia.

Figure 6-6 shows the response and SD components of the estimated model before provision for the combined effect of spatial variation and laboratory error in ICP measurements. Readings corresponding to the 9 largest ICP measurements are below the response line, which indicates that the true response may have "flattened out" as the lead level increased. The nonparametric estimated response also confirms this. Both the response and the SD components seem to fit the data well for ICP measurements as large as 2.0 mg/cm^2.

Table 6-49 gives the results of fitting XRF measurement models to the data. Comparing model parameter estimates for Denver and Philadelphia within Machine 1 produced a chi-square statistic with a p-value between 1 percent and 2 percent. The difference appears to arise mainly in the intercept term *a*, suggesting that Machine 1 read systematically higher in Denver than in Philadelphia. But city and ICP levels were not properly crossed: all sites with ICP measurements greater than 2.5 mg/cm^2 where Machine 1 was used were in Philadelphia. It is therefore

hard to attach much significance to this result.

Table 6-47. Lead Analyzer K-shell on Drywall: Model Estimates.

DEVICE	SAMPLE SIZE	MODEL PARAMETERS				Pb=0.0 mg/cm²		Pb=1.0 mg/cm²	
		a	b	c	d	BIAS	SD	BIAS	SD
Machine 1, Denver	103	-0.014 (.010)	1.169 (.114)	0.006 (.001)	0.113 (.077)	-0.014 (.010)	0.077	0.155 (.113)	0.345
Machine 1, Denver and Philadelphia	111	-0.018 (.009)	1.196 (.115)	0.006 (.001)	0.120 (.081)	-0.018 (.009)	0.076	0.178 (.110)	0.354

Table 6-48. Lead Analyzer K-shell on Drywall: Control Block Summary.

DEVICE	SAMPLE SIZE	BARE (0.0 mg/cm²)		RED NIST SRM (1.02 mg/cm²)		YELLOW NIST SRM (3.53 mg/cm²)	
		BIAS	SD	BIAS	SD	BIAS	SD
Machine 1	57	-0.016 (.009)	0.069	0.073 (.026)	0.193	0.202 (.054)	0.406
Machine 1	6	0.023 (.018)	0.045	-0.037 (.070)	0.172	0.137 (.176)	0.432
Machines 1 and 2	63	-0.012 (.008)	0.067	0.063 (.024)	0.191	0.195 (.051)	0.408

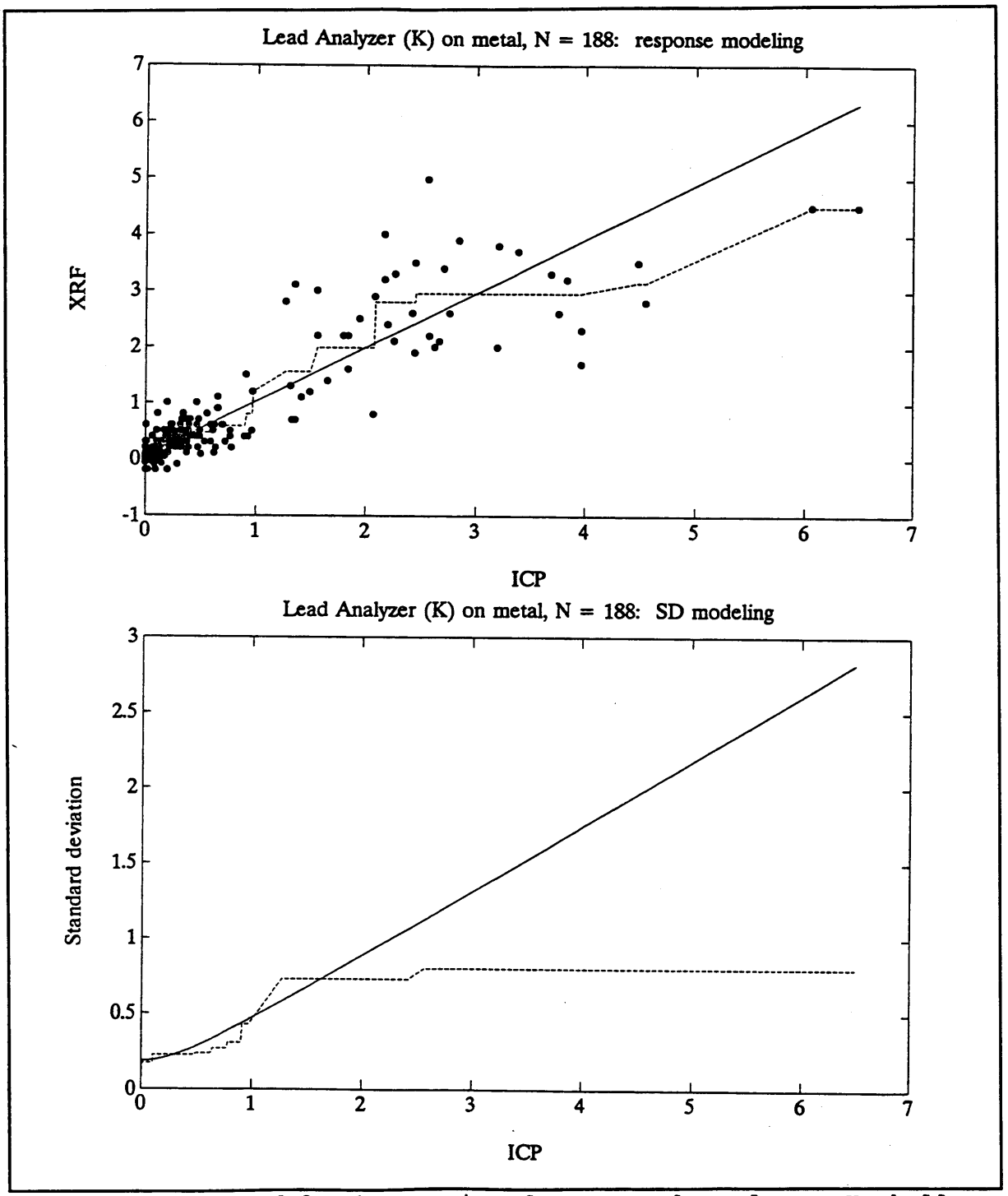


Figure 6-6. Model Diagnostic Plots, Lead Analyzer K-shell on metal. Solid lines are model estimates. Dashed lines are nonparametric (monotone regression) estimates.

Table 6-49. Lead Analyzer K-shell on Metal: Model Estimates.

DEVICE	SAMPLE SIZE	MODEL PARAMETERS				Pb=0.0 mg/cm²		Pb=1.0 mg/cm²	
		a	b	c	d	BIAS	SD	BIAS	SD
Machine 1, Denver	62	0.107 (.027)	1.139 (.157)	0.026 (.007)	0.335 (.173)	0.107 (.027)	0.160	0.246 (.150)	0.601
Machine 1, Philadelphia	78	-0.000 (.033)	0.999 (.069)	0.021 (.006)	0.114 (.034)	-0.000 (.033)	0.145	-0.001 (.150)	0.367
Machine 1, Denver and Philadelphia	140	0.075 (.022)	0.963 (.060)	0.029 (.006)	0.149 (.040)	0.075 (.022)	0.169	0.037 (.054)	0.421
Machine 2, Philadelphia	48	0.096 (.045)	0.752 (.071)	0.058 (.013)	-0-	0.096 (.045)	0.242	-0.152 (.059)	0.242
Machines 1 and 2	188	0.063 (.021)	0.958 (.055)	0.034 (.006)	0.132 (.035)	0.063 (.021)	0.183	0.020 (.047)	0.406

Table 6-50. Lead Analyzer K-shell on Metal: Control Block Summary.

DEVICE	SAMPLE SIZE	BARE (0.0 mg/cm²)		RED NIST SRM (1.02 mg/cm²)		YELLOW NIST SRM (3.53 mg/cm²)	
		BIAS	SD	BIAS	SD	BIAS	SD
Machine 1	65	0.011 (.018)	0.145	0.129 (.028)	0.226	0.245 (.053)	0.429
Machine 2	12	-0.141 (.057)	0.197	0.022 (.056)	0.193	0.003 (.123)	0.425
Machines 1 and 2	77	-0.013 (.018)	0.154	0.112 (.025)	0.221	0.208 (.049)	0.429

Machines 1 and 2 also differed within Philadelphia, primarily with respect to the variance parameters *c* and *d*, but Machine 2 observations were also more concentrated toward lower ICP measurements. Fitting models with ICP measurements restricted to less than 1.0 mg/cm^2 gave a chi-square statistic with a p-value of about 3 percent comparing the two machines. This moderately significant result does not point to a large machine difference, however, and pooling across factors did not appear to be problematical.

Table 6-50 summarizes the control block data. Apparent differences between the two machines must take into account the small sample size (N = 12) for Machine 2, and the consequently large standard errors. Pooled and within-machine SD estimates at 0.0 mg/cm^2 show agreement between control blocks and model estimates, but the model indicates additional variability as the lead level increases.

6.4.4.1.5 Lead Analyzer K-shell on Plaster

There were 222 observations of the Lead Analyzer K-shell on plaster, 4 of which were removed as outliers (80260, 80262, 80773, and 81255), leaving 218 observations for analysis. Readings were made with Machine 1 164 times, 98 in Denver and 66 in Philadelphia. Readings with Machine 2 were made 54 times, all in Philadelphia.

Figure 6-7 shows the response and SD components of the estimated model before provision for the combined effect of spatial variation and laboratory error in ICP measurements. It should be noted that the reading at the highest ICP measurement was not unduly influential, because the large estimated SD weighed it down in model estimation. The model appears to fit the data well, except at the observation having the highest ICP measurement.

Table 6-51 gives the results of fitting XRF measurement models to the data. City effects within Machine 1 are indicated primarily in the SD estimates, with Philadelphia estimates lower than those for Denver. The p-value of the corresponding chi-square statistic is about 0.5 percent. Results for the pooled city data give an indication of how the instrument performed under a broader range of conditions. Comparing Machines 1 and 2 on the Philadelphia data produced a chi-square statistic with a

p-value greater than 10 percent. There is little to suggest that a machine effect exists in these data.

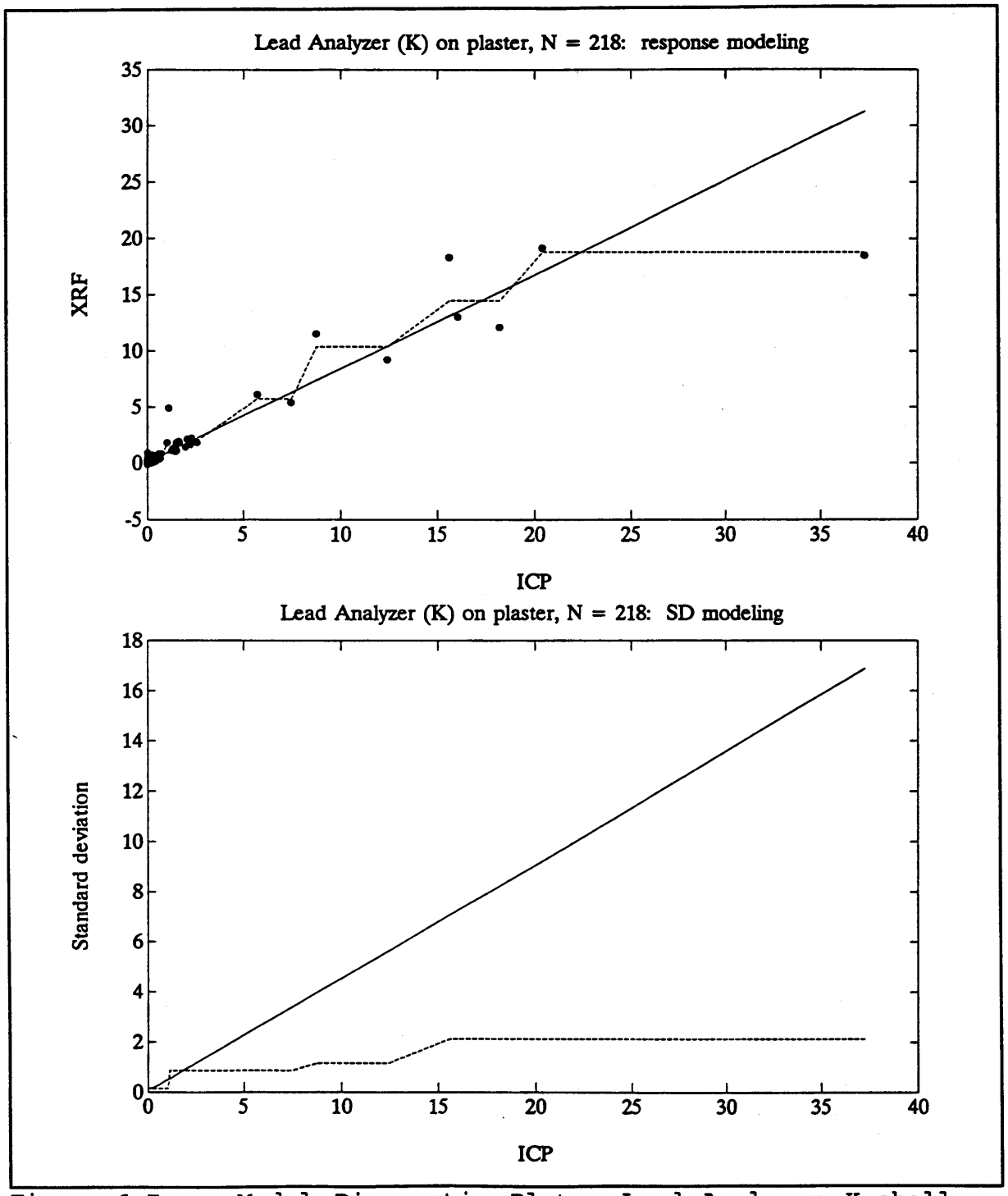


Figure 6-7. Model Diagnostic Plots, Lead Analyzer K-shell on plaster. Solid lines are model estimates. Dashed lines are nonparametric (monotone regression) estimates.

Table 6-51. Lead Analyzer K-shell on Plaster: Model Estimates.

DEVICE	SAMPLE SIZE	MODEL PARAMETERS				Pb=0.0 mg/cm²		Pb=1.0 mg/cm²	
		a	b	c	d	BIAS	SD	BIAS	SD
Machine 1, Denver	98	0.038 (.020)	0.903 (.085)	0.024 (.004)	0.082 (.043)	0.038 (.020)	0.156	-0.060 (.077)	0.326
Machine 1, Philadelphia	66	-0.045 (.032)	0.968 (.087)	0.011 (.005)	0.016 (.032)	-0.045 (.032)	0.107	-0.077 (.062)	0.167
Machine 1, Denver and Philadelphia	164	0.022 (.016)	0.862 (.053)	0.020 (.003)	0.038 (.019)	0.022 (.016)	0.142	-0.116 (.045)	0.241
Machine 2, Philadelphia	54	0.060 (.030)	0.839 (.094)	0.015 (.005)	0.048 (.040)	0.060 (.030)	0.123	-0.101 (.075)	0.250
Machines 1 and 2	218	0.030 (.014)	0.861 (.045)	0.019 (.002)	0.037 (.016)	0.030 (.014)	0.139	-0.109 (.038)	0.238

Table 6-52. Lead Analyzer K-shell on Plaster: Control Block Summary.

DEVICE	SAMPLE SIZE	BARE (0.0 mg/cm²)		RED NIST SRM (1.02 mg/cm²)		YELLOW NIST SRM (3.53 mg/cm²)	
		BIAS	SD	BIAS	SD	BIAS	SD
Machine 1	57	-0.023 (.012)	0.088	0.040 (.026)	0.193	0.212 (.060)	0.456
Machine 2	12	-0.043 (.034)	0.119	0.080 (.065)	0.226	0.387 (.080)	0.276
Machines 1 and 2	69	-0.027 (.011)	0.093	0.047 (.024)	0.198	0.242 (.052)	0.432

Table 6-52 gives the control block data summary. Positive bias is detectable at 3.53 mg/cm^2, but not at lower levels. With model parameter *b* = 0.861 (.045), the estimated bias from the model is negative at higher lead levels.

Both the model and control block SD estimates suggest greater variability as the lead level increases.

6.4.4.1.6 Lead Analyzer K-shell on Wood

There were 355 observations of the Lead Analyzer K-shell on wood, 4 of which were designated as outliers (80014, 80207, 80218, and 80311) and removed, leaving 351 observations for analysis. Readings on Machine 1 were made 339 times, 299 in Denver and 40 in Philadelphia. Readings on Machine 2 were made 12 times, all in Philadelphia. There were too few Machine 2 data for separate model fitting.

Figure 6-8 shows the response and SD components of the estimated model before provision for the combined effect of spatial variation and laboratory error in ICP measurements. The nonparametric estimate indicates a flatter response than the model, and the problem appears to reside in the model estimate. This occurred because of the tight clustering of ICP measurements at very low levels, where XRF variability was also low. These readings essentially determined the model response, because readings at higher ICP levels were less numerous, and the nonconstant SD suggested in the bottom frame of Figure 6-8 caused the higher ICP observations to be downweighted.

Table 6-53 gives the results of fitting XRF measurement models. The slope parameters (*b*) are all significantly greater than 1, reflecting what is apparent in Figure 6-8. They imply that the XRF measurements become increasingly positively biased as the lead level increases. The same can be seen in the control block data summary shown in Table 6-54, although not to the same extent.

A chi-square test of the Denver versus the Philadelphia parameter estimates on Machine 1 was highly significant (p-value less than 0.01 percent), due mainly to the difference in the estimates of *c*: 0.007 (.001) Denver versus 0.001 (.001) Philadelphia. Restricting the test to response function parameters *a* and *b*, however, gave an insignificant result. The

two cities had different ranges of ICP measurements, which may partly explain the disagreement in the model parameters. Almost no change resulted when Machine 2 data were pooled with those for

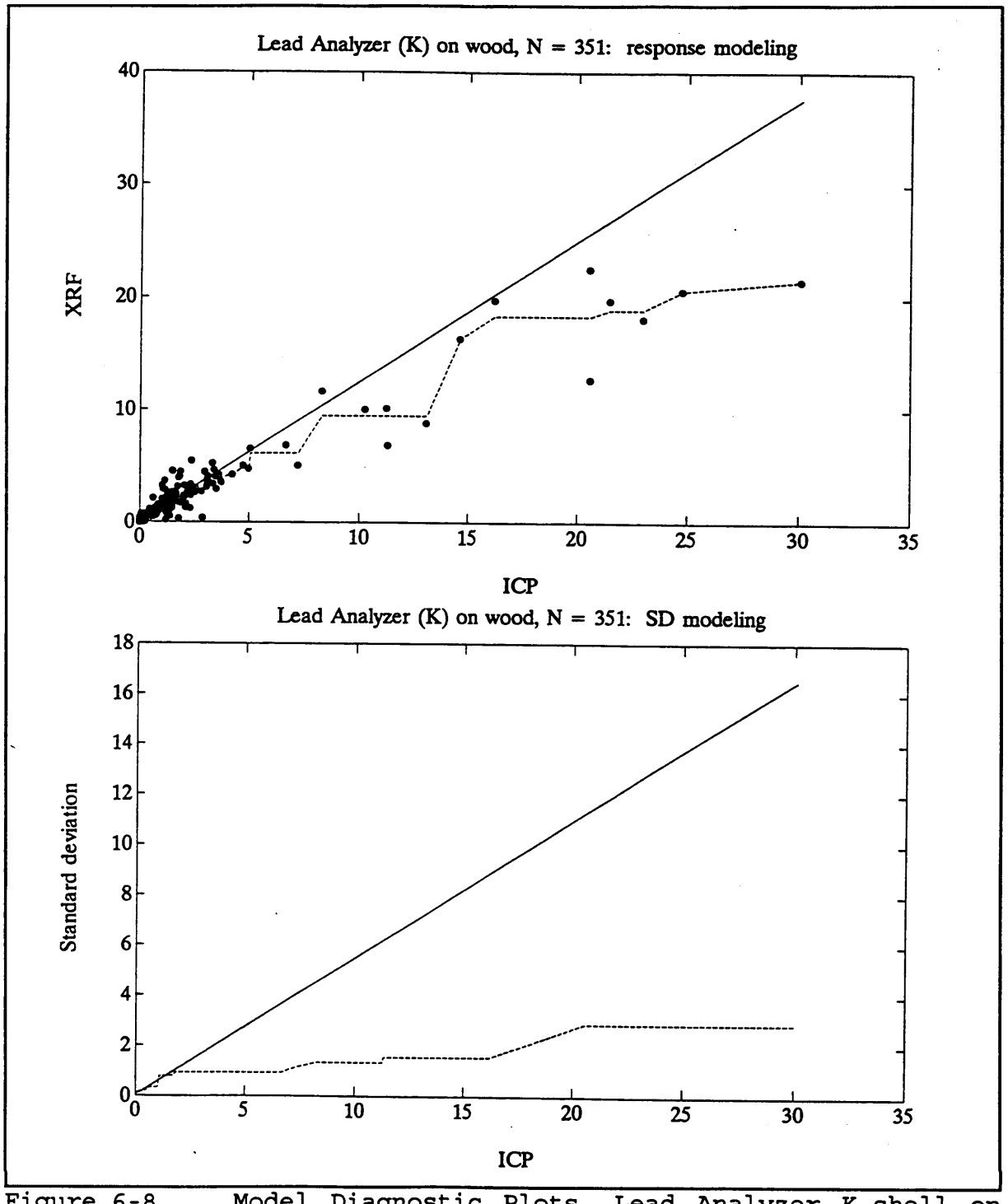


Figure 6-8. Model Diagnostic Plots, Lead Analyzer K-shell on wood. Solid lines are model estimates. Dashed lines are nonparametric (monotone regression) estimates.

Table 6-53. Lead Analyzer K-shell on Wood: Model Estimates.

DEVICE	SAMPLE SIZE	MODEL PARAMETERS				Pb=0.0 mg/cm²		Pb=1.0 mg/cm²	
		a	b	c	d	BIAS	SD	BIAS	SD
Machine 1, Denver	299	0.013 (.007)	1.250 (.053)	0.007 (.001)	0.198 (.042)	0.013 (.007)	0.083	0.263 (.051)	0.452
Machine 1, Philadelphia	40	0.013 (.016)	1.359 (.085)	0.001 (.001)	0.157 (.053)	0.013 (.016)	0.030	0.372 (.080)	0.398
Machine 1, combined	339	0.013 (.007)	1.269 (.046)	0.006 (.001)	0.187 (.035)	0.013 (.007)	0.082	0.282 (.044)	0.440
Machines 1 and 2	351	0.013 (.007)	1.266 (.044)	0.007 (.001)	0.180 (.033)	0.013 (.007)	0.080	0.278 (.042)	0.432

Table 6-54. Lead Analyzer K-shell on Wood: Control Block Summary.

DEVICE	SAMPLE SIZE	BARE (0.0 mg/cm²)		RED NIST SRM (1.02 mg/cm²)		YELLOW NIST SRM (3.53 mg/cm²)	
		BIAS	SD	BIAS	SD	BIAS	SD
Machine 1	63	-0.001 (.004)	0.031	0.034 (.022)	0.177	0.219 (.058)	0.459
Machine 2	12	-0.002 (.011)	0.037	0.072 (.050)	0.173	0.270 (.128)	0.443
Machines 1 and 2	75	-0.001 (.004)	0.032	0.040 (.020)	0.176	0.227 (.053)	0.456

Machine 1. The pooled estimates (N = 351) indicate how this instrument performed under a broader range of conditions on wood substrates.

6.4.4.1.7 Lead Analyzer K-shell: Summary of Analysis

The Lead Analyzer K-shell exhibited little difference in performance across substrates. For lead levels in the 0.0 mg/cm^2 to 1.0 mg/cm^2 range, the bias was minimal. At the 0.0 mg/cm^2 lead level the SD estimates from the model and the control block data were similar. At higher lead levels, the estimates from the model generally exceeded those from the control block data, which may reflect the influence of non-instrumental sources of variation. Differences between the two machines used in the full study did not appear to be an important factor. City effects were harder to detect, because the distributions of ICP measurements in Denver and Philadelphia field samples were markedly different. There were no indications of city effects, however, to indicate that the data should not be pooled across cities.

6.4.4.2 Results for Lead Analyzer L-shell

Readings for the Lead Analyzer L-shell were taken with the same instruments as the K-shell, described in the previous section. Two machines (1 and 2) were used by one operator (A). Machine 1 was used in both Denver and Philadelphia; Machine 2 was used in Philadelphia only. Machine and city effects could be analyzed on some substrates, but it was not possible to attribute effects to the operator.

6.4.4.2.1 Lead Analyzer L-shell on Brick

There were 93 observations of the Lead Analyzer L-shell on brick, one of which was designated as an outlier (80908) and removed from the analysis. Readings on Machine 1 were made 86 times, 80 in Denver and 6 in Philadelphia. Readings on Machine 2 were made only 6 times, all in Philadelphia.

Figure 6-9 shows response and SD components of the estimated model before provision for the combined effect of spatial variation and laboratory error in ICP measurements. There were no samples with ICP measurements between 0.8042 mg/cm^2 and 4.6567 mg/cm^2, and most of the data were at lower ICP levels. The

model, especially the response, does not appear to be valid across the entire ICP range. The only XRF readings above 1.0 mg/cm^2 occurred at the two highest ICP measurements.

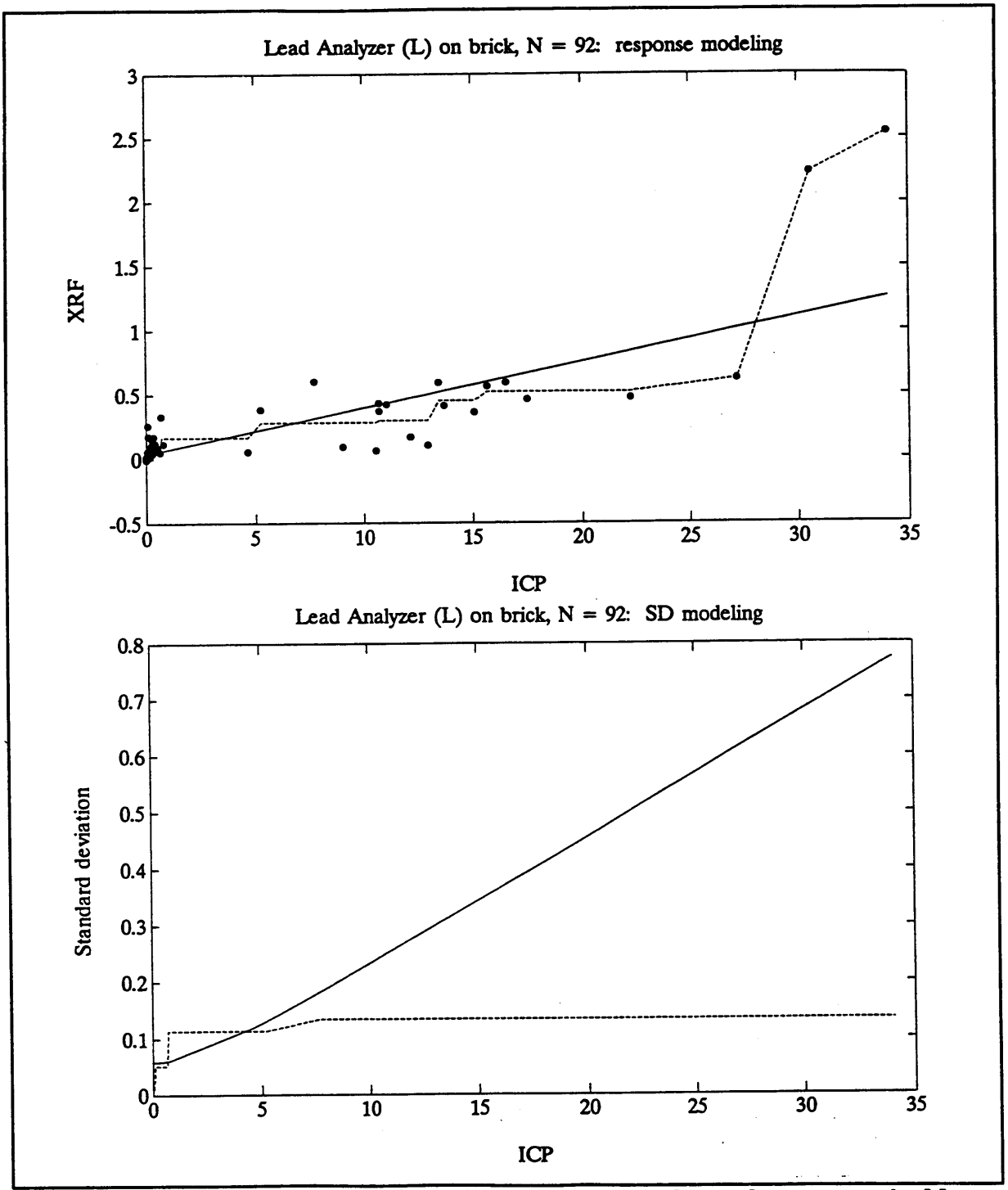


Figure 6-9. Model Diagnostic Plots, Lead Analyzer L-shell on brick. Solid lines are model estimates. Dashed lines are nonparametric (monotone regression) estimates.

Table 6-55 gives the results of fitting XRF measurement models to the data. The inclusion of Philadelphia data did not seem to affect the results significantly. Restriction of the analysis to ICP measurements less than 1.0 mg/cm^2 (a loss of 20 observations) produced a large difference in the estimated model parameters, with the slope *b* increasing from 0.036 (.006) on the full data to 0.226 (.031) on the restricted data. The latter suggests a "one fifth the actual lead level" response that was seen in many of the L-shell analyses. Consequently, bias was not a problem at 0.0 mg/cm^2 but was very prominent at 1.0 mg/cm^2.

Table 6-56 presents a summary of the control block data, where the situation was very different. The bias was small and did not appear to change greatly up to 1.02 mg/cm^2, but became prominent at 3.53 mg/cm^2, although not of the magnitude suggested by the model. The field sample data exhibited more variability near 0.0 mg/cm^2, but limitation of the analysis to ICP measurements less than 1.0 mg/cm^2 makes it difficult to compare model estimates and control block summary statistics at higher lead levels.

6.4.4.2.2 Lead Analyzer L-shell on Concrete

There were 218 observations of the Lead Analyzer L-shell on concrete, one of which was designated as an outlier (81234), leaving 217 observations for analysis. Readings on Machine 1 were made 178 times, 98 in Denver and 80 in Philadelphia. Readings on Machine 2 were made 39 times, all in Philadelphia.

Figure 6-10 shows the response and SD components of the estimated model before provision for the combined effect of spatial variation and laboratory error in ICP measurements. The linear response does not appear to have global validity in describing the XRF-ICP relationship, although it may be reasonable for ICP measurements no larger than about 1.5 mg/cm^2. Restriction to ICP levels less than 1.0 mg/cm^2, however, changed both of the estimated model components very little, due to the downweighting implied by the SD component at high ICP measurements. Figure 6-11 shows the ICP-restricted model components. The nonparametric estimate suggests that the response is possibly nonlinear at lower lead levels. In any case, the XRF readings appear to show no responsiveness to increases in the lead level for ICP measurements greater than 2.0 mg/cm^2. Only one XRF reading exceeded 1.0 mg/cm^2, and this

occurred at the lower end of the ICP range represented in the data.

Table 6-55. Lead Analyzer L-shell on Brick: Model Estimates.

DEVICE	SAMPLE SIZE	MODEL PARAMETERS				Pb=0.0 mg/cm²		Pb=1.0 mg/cm²	
		a	b	c	d	BIAS	SD	BIAS	SD
Machine 1, Denver	80	0.028 (.005)	0.039 (.007)	0.001 (.0003)	0.0004 (.0002)	0.028 (.005)	0.038	-0.932 (.007)	0.044
Machine 1, Denver and Philadelphia	86	0.035 (.007)	0.036 (.006)	0.003 (.0006)	0.0004 (.0002)	0.035 (.007)	0.057	-0.928 (.008)	0.060
Machines 1 and 2	92	0.038 (.007)	0.036 (.006)	0.001 (.001)	0.0004 (.0002)	0.038 (.007)	0.032	-0.926 (.008)	0.037
Machines 1 and 2 (ICP < 1)	72	0.009 (.007)	0.226 (.031)	0.018 (.0004)	-0-	0.009 (.007)	0.043	-0.765 (.030)	0.043

Table 6-56. Lead Analyzer L-shell on Brick: Control Block Summary.

DEVICE	SAMPLE SIZE	BARE (0.0 mg/cm²)		RED NIST SRM (1.02 mg/cm²)		YELLOW NIST SRM (3.53 mg/cm²)	
		BIAS	SD	BIAS	SD	BIAS	SD
Machine 1	51	0.024 (.003)	0.019	0.031 (.006)	0.042	-0.841 (.008)	0.060
Machine 2	12	0.02 (.003)	0.011	-0.021 (.013)	0.044	-0.931 (.017)	0.060
Machines 1 and 2	63	0.019 (.002)	0.018	0.021 (.005)	0.043	-0.858 (.008)	0.060

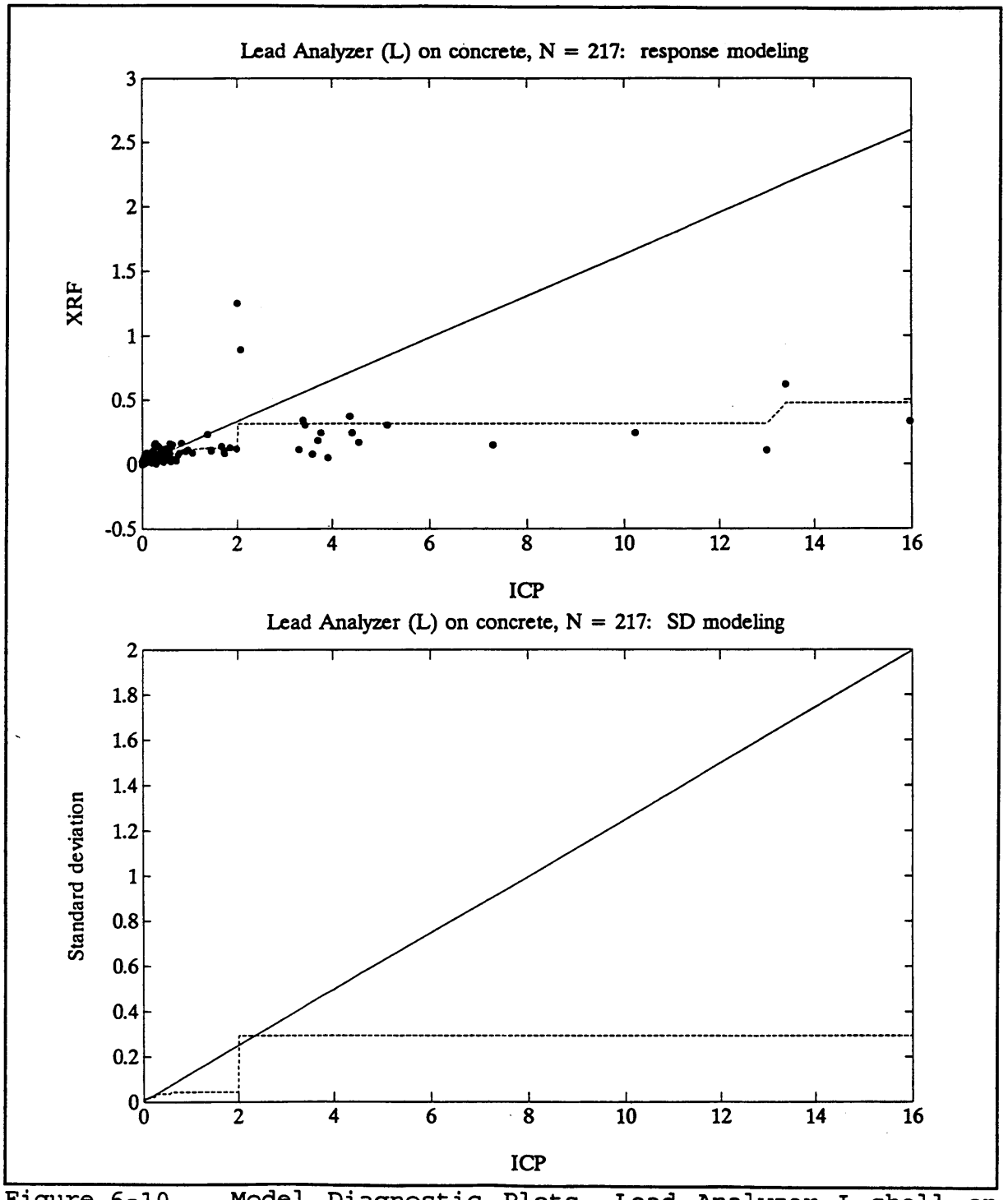


Figure 6-10. Model Diagnostic Plots, Lead Analyzer L-shell on concrete. Solid lines are model estimates. Dashed lines are nonparametric (monotone regression) estimates.

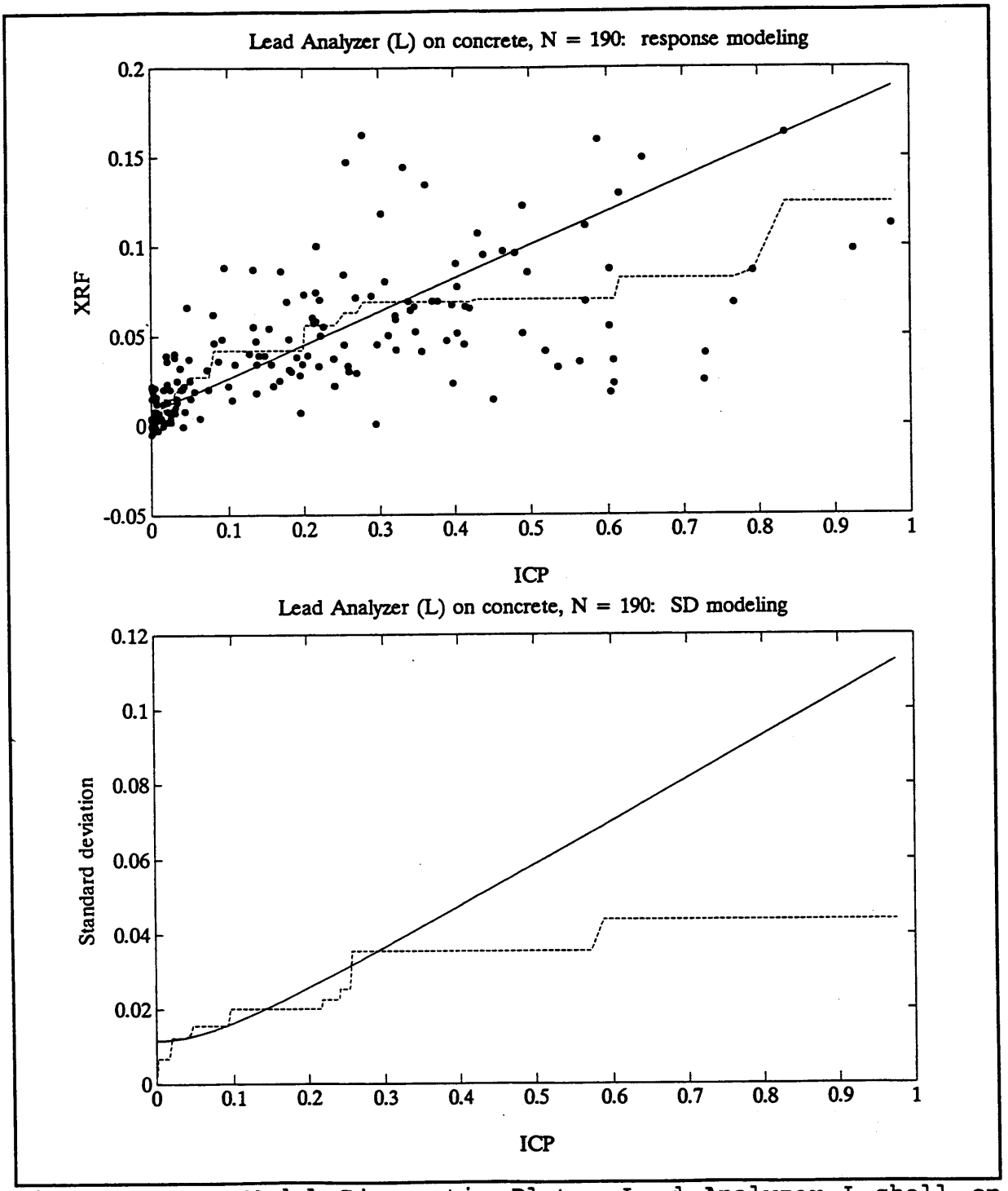


Figure 6-11. Model Diagnostic Plots, Lead Analyzer L-shell on drywall. Solid lines are model estimates. Dashed lines are nonparametric (monotone regression) estimates.

Table 6-57 gives the results of fitting XRF measurement models. Differences between cities (Denver versus Philadelphia within Machine 1) and machines (Machines 1 versus 2 within Philadelphia) were discerned with chi-square tests, both having p-values less than 0.01 percent. In spite of possible confounding with the lead level, and poor model fit, a similar city effect was seen on other substrates, notably metal and plaster.

The control block data (Table 6-58) did not bear out machine differences to nearly the same degree. While the control block data exhibited little bias at 1.02 mg/cm^2, the field sample data and model estimates derived from them suggest otherwise. One conclusion that can be drawn is that the Lead Analyzer L-shell performed differently on the painted samples than on the control blocks.

6.4.4.2.3 Lead Analyzer L-shell on Drywall

There were 113 observations of the Lead Analyzer L-shell on drywall, none of which were designated as outliers. All of the readings were made by Machine 1: 105 in Denver and 8 in Philadelphia.

Figure 6-12 shows the response and SD components of the estimated model before provision for the combined effect of spatial variation and laboratory error in ICP measurements. Both appear to fit the data well, although the limited ICP range (no ICP measurements greater than 1.0 mg/cm^2 were observed on drywall) makes this conclusion hard to extrapolate to higher lead levels.

Table 6-59 gives the results of fitting XRF measurement models to the full data, and to Denver data alone. Inclusion of the Philadelphia data degraded the fit of the model to a small degree.

Estimates at the 1.0 mg/cm^2 lead level are presented, but none of the ICP measurements in the full study were as large as this. Bias and SD estimates from the model agree with those from the control blocks (Table 6-60) at 0.0 mg/cm^2, but usage of the instrument on painted surfaces under field conditions would not appear likely to replicate the bias figures reported for the control block data at higher lead levels.

Table 6-57. Lead Analyzer L-shell on Concrete: Model Estimates.

DEVICE	SAMPLE SIZE	MODEL PARAMETERS				Pb=0.0 mg/cm²		Pb=1.0 mg/cm²	
		a	b	c	d	BIAS	SD	BIAS	SD
Machine 1, Denver	98	0.0003 (.001)	0.297 (.036)	0.00001 (.00001)	0.049 (.014)	0.0003 (.001)	0.003	-0.702 (.032)	0.222
Machine 1, Philadelphia	80	0.023 (.003)	0.109 (.013)	0.0002 (.0001)	0.003 (.001)	0.023 (.003)	0.014	-0.869 (.010)	0.056
Machine 1, combined	178	0.008 (.002)	0.174 (.015)	0.0001 (.0001)	0.012 (.003)	0.008 (.002)	0.011	-0.818 (.014)	0.109
Machine 2, Philadelphia	39	0.016 (.004)	0.049 (.012)	0.0002 (.0001)	0.0003 (.0004)	0.016 (.004)	0.014	-0.935 (.009)	0.021
Machines 1 and 2	217	0.009 (.001)	0.152 (.012)	0.0001 (.0002)	0.010 (.002)	0.009 (.001)	0.011	-0.840 (.011)	0.099

Table 6-58. Lead Analyzer L-shell on Concrete: Control Block Summary.

DEVICE	SAMPLE SIZE	BARE (0.0 mg/cm²)		RED NIST SRM (1.02 mg/cm²)		YELLOW NIST SRM (3.53 mg/cm²)	
		BIAS	SD	BIAS	SD	BIAS	SD
Machine 1	65	0.003 (.001)	0.011	0.055 (.014)	0.109	-0.812 (.009)	0.075
Machine 2	12	-0.010 (.002)	0.006	-0.012 (.006)	0.020	-0.911 (.012)	0.043
Machines 1 and 2	77	0.001 (.001)	0.010	0.045 (.012)	0.101	-0.827 (.008)	0.071

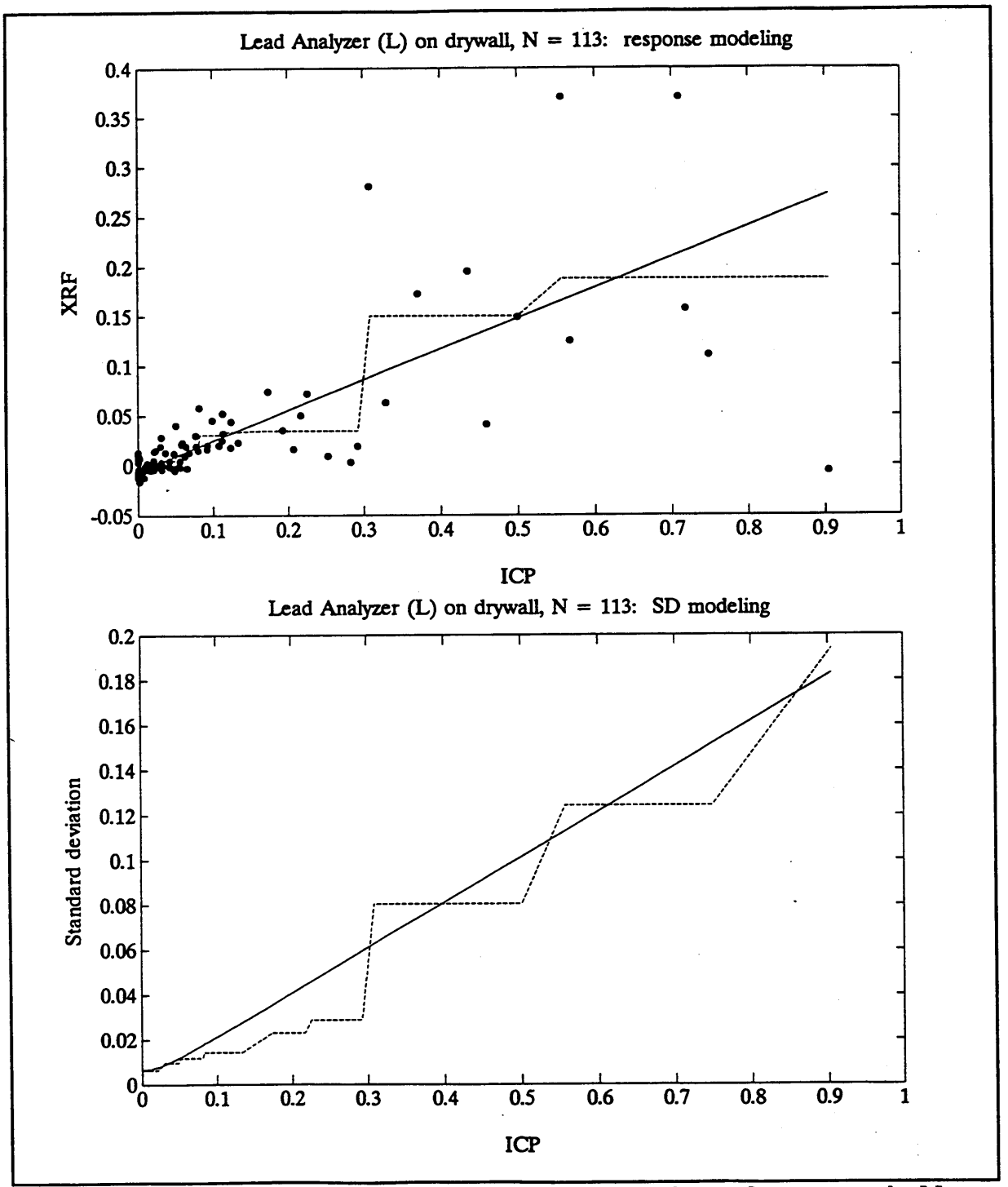


Figure 6-12. Model Diagnostic Plots, Lead Analyzer L-shell on metal. Solid lines are model estimates. Dashed lines are nonparametric (monotone regression) estimates.

Table 6-59. Lead Analyzer L-shell on Drywall: Model Estimates.

DEVICE	SAMPLE SIZE	MODEL PARAMETERS				Pb=0.0 mg/cm²		Pb=1.0 mg/cm²	
		a	b	c	d	BIAS	SD	BIAS	SD
Machine 1, Denver	105	-0.008 (.0004)	0.353 (.028)	0.0000 (.0000)	0.040 (.009)	-0.008 (.0004)	0.002	-0.655 (.028)	0.200
Machine 1, Denver and Philadelphia	113	-0.006 (.001)	0.302 (.029)	0.0000 (.0000)	0.029 (.008)	-0.006 (.001)	0.006	-0.704 (.029)	0.172

Table 6-60. Lead Analyzer L-shell on Drywall: Control Block Summary.

DEVICE	SAMPLE SIZE	BARE (0.0 mg/cm²)		RED NIST SRM (1.02 mg/cm²)		YELLOW NIST SRM (3.53 mg/cm²)	
		BIAS	SD	BIAS	SD	BIAS	SD
Machine 1	57	-0.003 (.001)	0.009	0.058 (.005)	0.038	-0.765 (.007)	0.053
Machine 1	6	-0.015 (.002)	0.004	-0.002 (.007)	0.017	-0.872 (.018)	0.043
Machines 1 and 2	63	-0.004 (.001)	0.009	0.052 (.005)	0.037	-0.775 (.007)	0.053

6.4.4.2.4 Lead Analyzer L-shell on Metal

There were 189 observations of the Lead Analyzer L-shell on metal, none of which were designated as outliers. Readings on Machine 1 were made 141 times, 62 in Denver and 79 in Philadelphia. All 48 readings on Machine 2 were made in Philadelphia.

Figure 6-13 shows the response and SD components of the estimated model before provision for the combined effect of spatial variation and laboratory error in ICP measurements. The model and nonparametric responses appear to agree, especially for ICP measurements below 2.0 mg/cm^2. Most of the readings above 2.0 mg/cm^2 were made with Machine 1.

Table 6-61 gives the results of fitting XRF measurement models. The higher slopes obtained on Denver (0.499) versus Philadelphia (0.198) data on Machine 1 are noteworthy because of the similarity to what was seen on concrete and plaster. Comparing Machines 1 and 2 on the Philadelphia data with the ICP range restriction on both models did not produce a significant chi-square statistic when only the response parameters *a* and *b* were considered.

Table 6-62 gives the control block data summary, which indicates little bias for lead levels as high as 1.02 mg/cm^2. Unlike other substrates, Figure 6-13 does indicate that a number of field sample XRF readings exceeded 1.0 mg/cm^2 where the ICP measurement did as well, but the predominant indication is to the contrary. The higher SD estimates from the model at 1.0 mg/cm^2 suggest non-instrumental sources of variability that may have degraded both the response and the SD.

6.4.4.2.5 Lead Analyzer L-shell on Plaster

There were 222 observations of the Lead Analyzer L-shell on plaster, none of which were designated as outliers. Readings on Machine 1 were made 168 times, 101 in Denver and 67 in Philadelphia. Readings on Machine 2 were made 54 times, all in Philadelphia.

The top of Figure 6-14 is a scatterplot of XRF and ICP measurements for all data. In no case was an XRF reading as large as 1.0 mg/cm^2 observed. The 9 XRF measurements with ICP

measurements larger than 5.0 mg/cm^2 are seen to be very low. The same unit in Denver, nearly one hundred years old, produced these readings. The bottom scatterplot restricts ICP measurements to

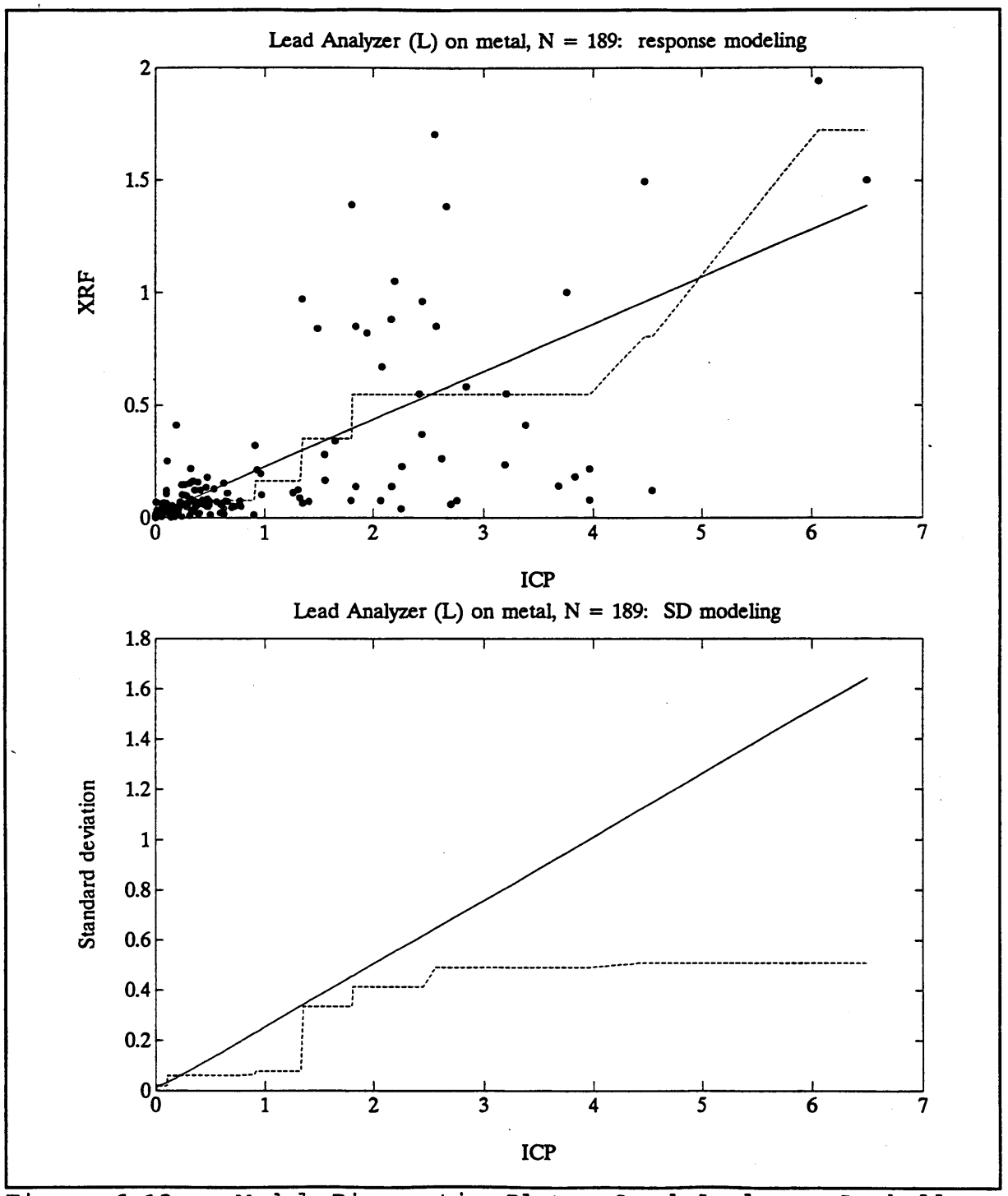


Figure 6-13. Model Diagnostic Plots, Lead Analyzer L-shell on plaster. Solid lines are model estimates. Dashed lines are nonparametric (monotone regression) estimates.

Table 6-61. Lead Analyzer L-shell on Metal: Model Estimates.

DEVICE	SAMPLE SIZE	MODEL PARAMETERS				Pb=0.0 mg/cm²		Pb=1.0 mg/cm²	
		a	b	c	d	BIAS	SD	BIAS	SD
Machine 1, Denver	62	0.007 (.002)	0.499 (.087)	0.00009 (.000)	0.281 (.043)	0.004 (.002)	0.010	-0.493 (.087)	0.530
Machine 1, Philadelphia	79	0.014 (.002)	0.198 (.022)	0.00002 (.00002)	0.029 (.006)	0.014 (.002)	0.004	-0.788 (.022)	0.169
Machine 1, combined	141	0.014 (.002)	0.230 (.024)	0.0002 (.0001)	0.043 (.008)	0.014 (.002)	0.015	-0.757 (.023)	0.209
Machine 1 (ICP < 1)	101	0.013 (.002)	0.264 (.036)	0.0002 (.0001)	0.054 (.015)	0.013 (.002)	0.014	-0.724 (.030)	0.232
Machine 2, Phila. (ICP < 1)	44	0.019 (.008)	0.103 (.038)	0.0002 (.0002)	0.013 (.005)	0.019 (.008)	0.014	-0.878 (.030)	0.113
Machines 1 and 2 (ICP < 1)	145	0.013 (.002)	0.196 (.023)	0.0002 (.00006)	0.032 (.007)	0.013 (.002)	0.015	-0.790 (.022)	0.179

Table 6-62. Lead Analyzer L-shell on Metal: Control Block Summary.

DEVICE	SAMPLE SIZE	BARE (0.0 mg/cm²)		RED NIST SRM (1.02 mg/cm²)		YELLOW NIST SRM (3.53 mg/cm²)	
		BIAS	SD	BIAS	SD	BIAS	SD
Machine 1	65	0.005 (.000)	0.004	0.041 (.008)	0.068	-0.817 (.021)	0.169
Machine 2	12	-0.001 (.002)	0.007	-0.011 (.006)	0.020	-0.904 (.013)	0.044

Machines 1 and 2	77	0.004 (.000)	0.004	0.033 (.007)	0.063	-0.831 (.018)	0.157

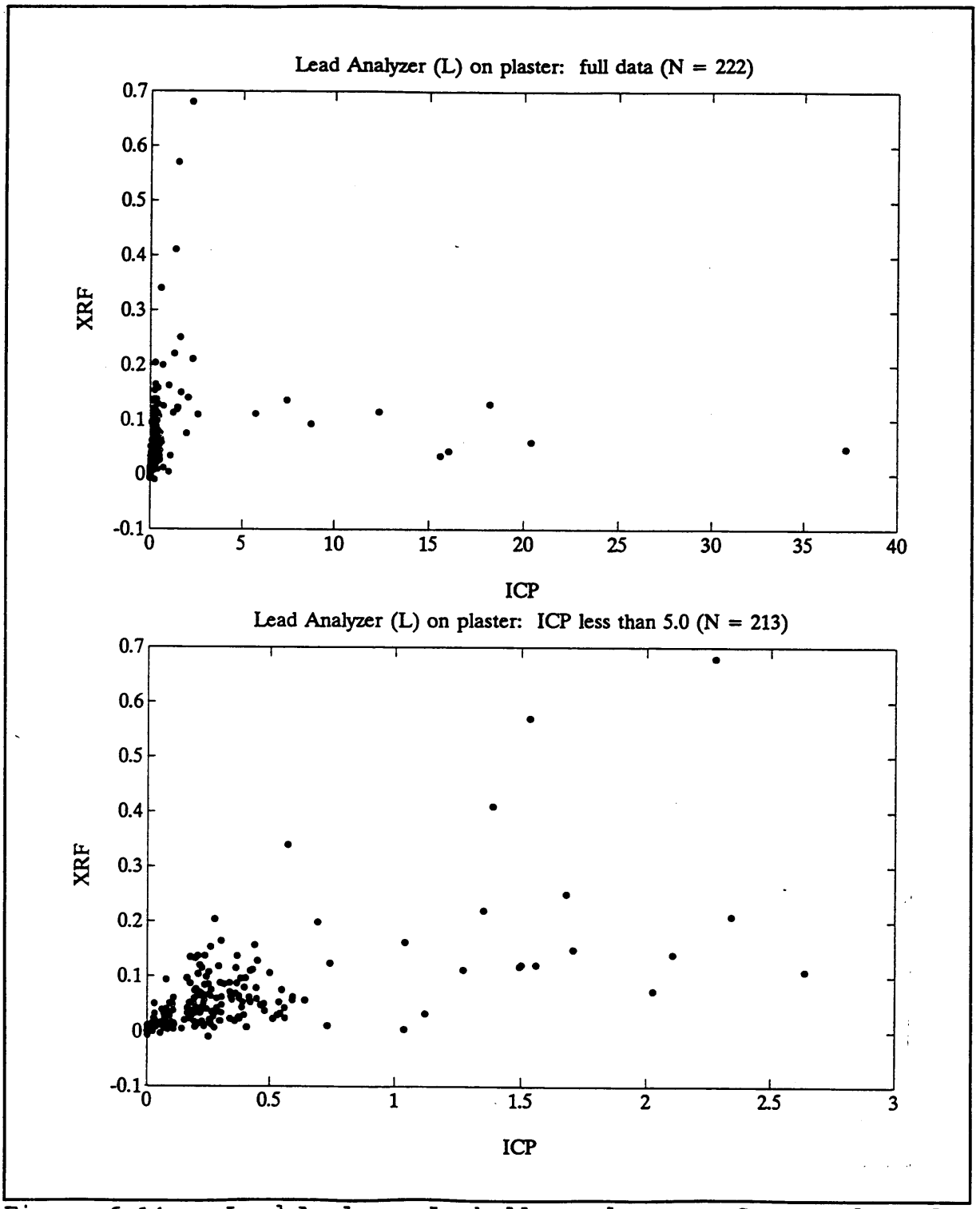


Figure 6-14. Lead Analyzer L-shell on plaster: Scatterplots for the full and restricted ICP ranges.

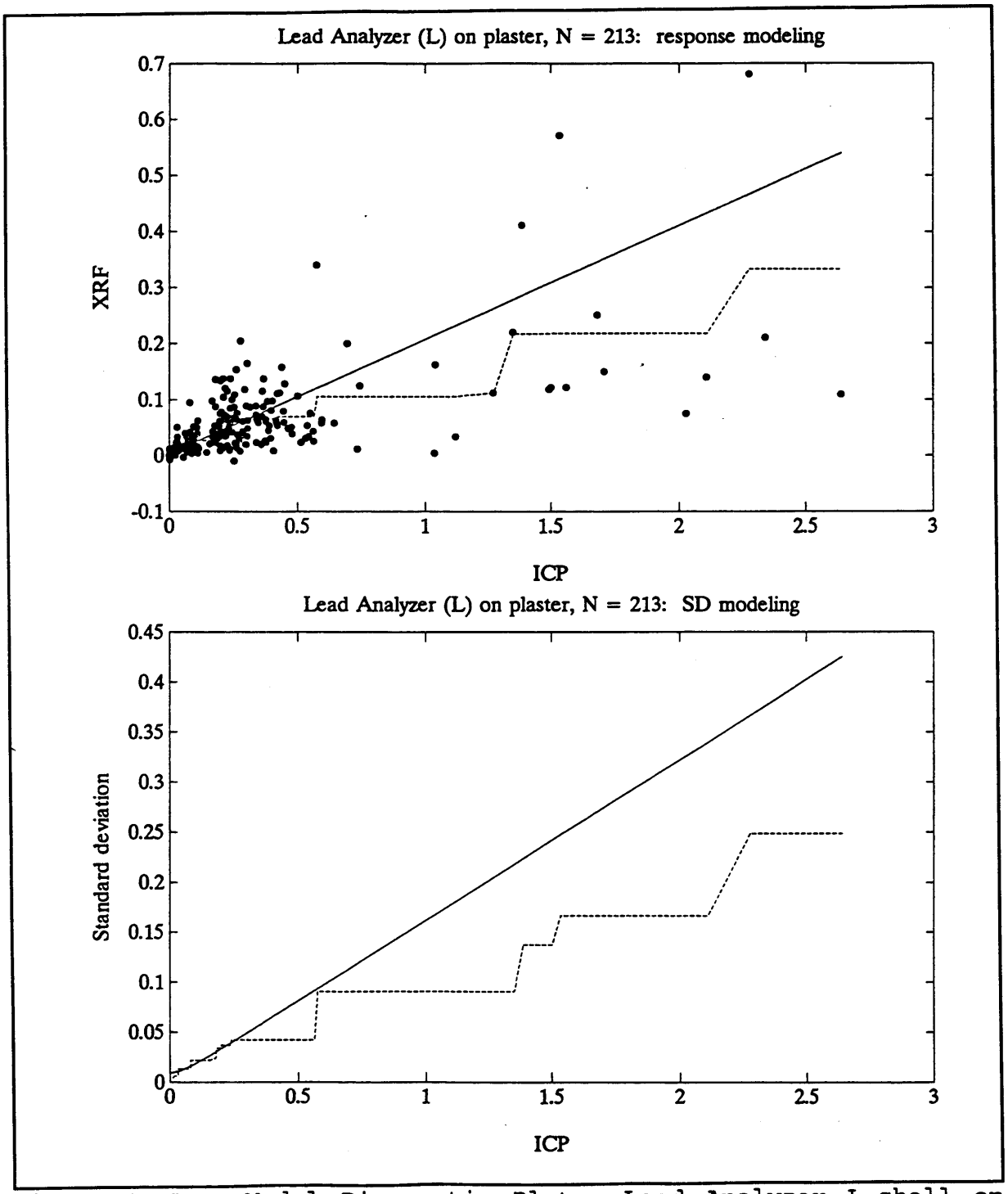


Figure 6-15. Model Diagnostic Plots, Lead Analyzer L-shell on plaster. Solid lines are model estimates. Dashed lines are nonparametric (monotone regression) estimates.

less than 5.0 mg/cm^2, and it shows an increasing relationship. Applying this restriction to model estimation better described the performance of the instrument at 0.0 mg/cm^2 and at 1.0 mg/cm^2. Figure 6-15 shows response and SD components of the estimated model before provision for the combined effect of spatial variation and laboratory error in ICP measurements. There were no field samples on plaster with ICP readings between 3.0 mg/cm^2 and 5.0 mg/cm^2, which explains the labeling of the horizontal axes. Both model components appear to agree with their nonparametric analogues, primarily at the lower end of the ICP scale.

Table 6-63 gives the results of fitting XRF measurement models. A city effect is apparent on Machine 1, with Denver having a slope of 0.313 (.031) and Philadelphia a slope of 0.087 (.020). The resulting chi-square has a p-value of less than 0.01 percent. This same type of city effect (larger Denver slope) was also seen on metal and concrete. An effect due to machines within Philadelphia can also be discerned with a chi-square test (p-value less than 1 percent), which mainly reflects the influence of parameters *a* and *c*, relating to instrument performance at 0.0 mg/cm^2. Figure 6-16, which shows scatterplots for the two machines using the Philadelphia data only, reveals different performance characteristics corresponding to the machines.

Table 6-64 gives the control block data summary. Bias and SD estimates varied noticeably between the control block and the model estimates. The control block data summary suggests little bias at 1.02 mg/cm^2, contrary to what the model estimates imply.

6.4.4.2.6 Lead Analyzer L-shell on Wood

There were 355 observations of the Lead Analyzer L-shell on wood, one of which was designated as an outlier (81316) and removed from the analysis. Readings on Machine 1 were made 342 times, 303 in Denver and 39 in Philadelphia. Readings on Machine 2 were made 12 times, all in Philadelphia. There were too few Machine 2 data for separate model fitting.

Figure 6-17 shows the response and SD components of the estimated model before provision for the combined effect of spatial variation and laboratory error in ICP measurements. The response function does not agree very well with the nonparametric

estimate. The instrument appears to have responded to increases in the lead level only up to a certain point, above which the response flattened out. XRF readings less than 1.0 mg/cm^2 were

Table 6-63. Lead Analyzer L-shell on Plaster: Model Estimates.

DEVICE	SAMPLE SIZE	MODEL PARAMETERS				Pb=0.0 mg/cm²		Pb=1.0 mg/cm²	
		a	b	c	d	BIAS	SD	BIAS	SD
Machine 1, Denver (ICP < 5)	92	-0.001 (.001)	0.313 (.031)	0.0000 (.0000)	0.046 (.010)	-0.001 (.001)	0.004	-0.688 (.031)	0.215
Machine 1, Phila. (ICP < 5)	67	0.038 (.008)	0.087 (.020)	0.001 (.0003)	0.001 (.001)	0.038 (.008)	0.032	-0.876 (.020)	0.044
Machine 1, combined (ICP < 5)	159	0.000 (.001)	0.243 (.017)	0.0000 (.0000)	0.025 (.004)	0.000 (.001)	0.005	-0.756 (.016)	0.159
Machine 2 (ICP < 5)	54	0.017 (.004)	0.065 (.013)	0.0002 (.0001)	0.001 (.001)	0.017 (.004)	0.014	-0.918 (.011)	0.035
Machines 1 and 2 (ICP < 5)	213	0.002 (.001)	0.201 (.014)	0.0001 (.0000)	0.019 (.003)	0.002 (.001)	0.008	-0.797 (.014)	0.138

Table 6-64. Lead Analyzer L-shell on Plaster: Control Block Summary.

DEVICE	SAMPLE SIZE	BARE (0.0 mg/cm²)		RED NIST SRM (1.02 mg/cm²)		YELLOW NIST SRM (3.53 mg/cm²)	
		BIAS	SD	BIAS	SD	BIAS	SD
Machine 1	57	0.005 (.001)	0.009	0.051 (.005)	0.036	-0.792 (.010)	0.073
Machine 2	12	-0.008 (.001)	0.005	0.001 (.006)	0.022	-0.919 (.012)	0.042
Machines 1 and 2	69	0.003 (.001)	0.009	0.042 (.004)	0.034	-0.814 (.008)	0.069

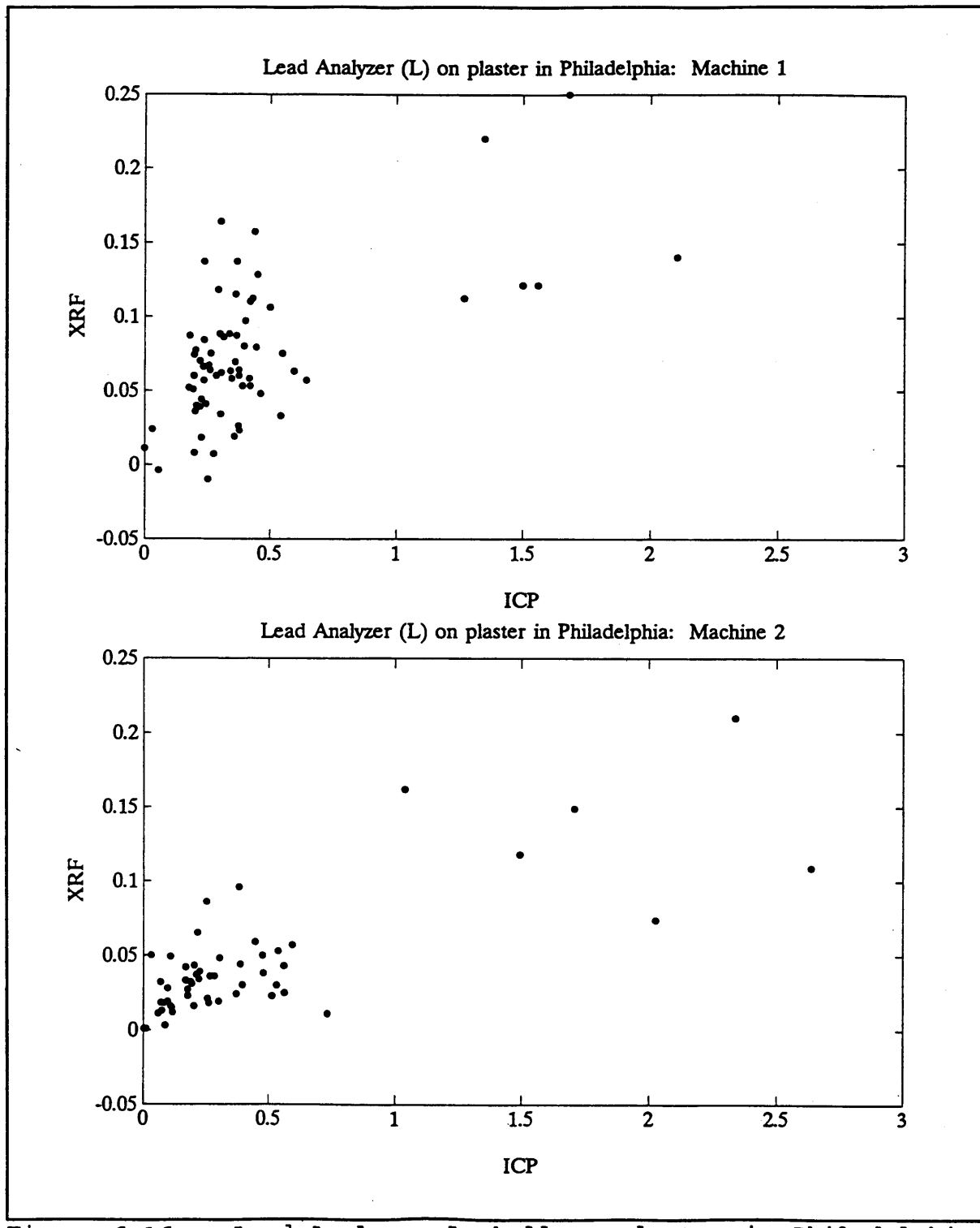


Figure 6-16. Lead Analyzer L-shell on plaster in Philadelphia: Machine 1 vs Machine 2.

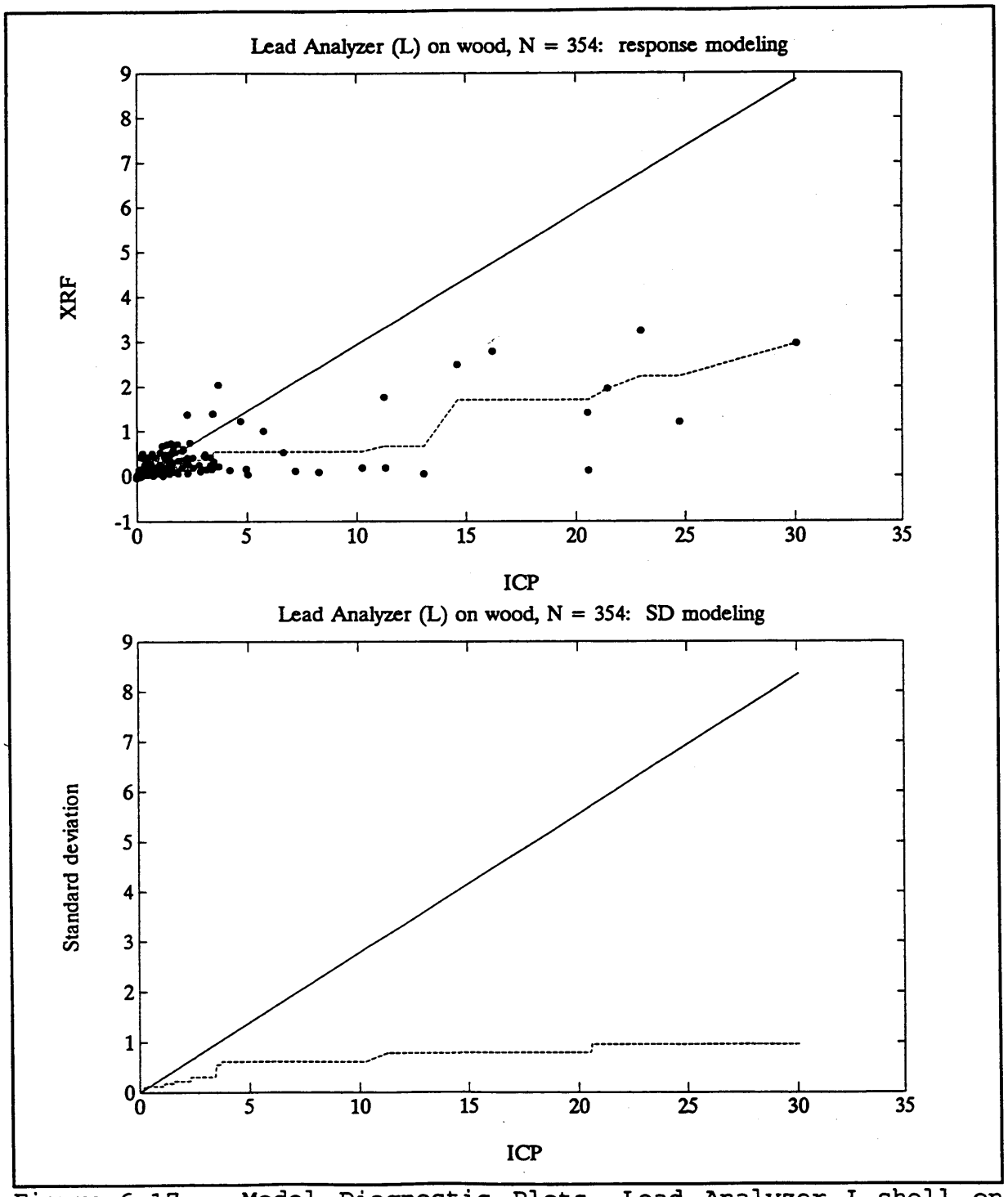


Figure 6-17. Model Diagnostic Plots, Lead Analyzer L-shell on wood. Solid lines are model estimates. Dashed lines are nonparametric (monotone regression) estimates.

obtained at lead levels higher than 10.0 mg/cm^2. Restriction of the ICP range to under 5.0 mg/cm^2 did a better job of capturing the limited responsiveness.

Table 6-65 gives the results of fitting XRF measurement models to the data. A strong city effect was evident within Machine 1, and as seen with several other substrates, a higher slope coefficient *b* was obtained for Denver compared to Philadelphia data. The difference is highly statistically significant, and hard to regard as spurious. Readings obtained on the field samples in both cities, however, showed much less responsiveness to the lead level than seen on the control blocks (Table 6-66), where the estimated bias is remarkably low at 1.02 mg/cm^2.

6.4.4.2.7 Lead Analyzer L-shell: Summary of Analysis

The Lead Analyzer L-shell did not differ markedly in its performance across substrates, which was reflected both in the field sample and control block data. The performance of this instrument on the field samples, however, diverged from its performance on the control blocks in important respects. The control block data found little or no bias at lead levels as high as 1.0 mg/cm^2, but the model estimates suggest that the instrument is under-responsive to the lead level, ensuring that its readings became increasingly negatively biased as the lead level increased. Readings less than 1.0 mg/cm^2 were obtained on field samples with ICP measurements greater than 10.0 mg/cm^2 on all substrates except drywall and metal, for which no such ICP measurements were obtained in the full study. At the 3.53 mg/cm^2 lead level, the control block data also suggest that the instrument exhibited substantial negative bias, but of a lower magnitude than estimated with the models.

The fact that samples with higher lead levels more often occurred in Denver than in Philadelphia may explain the intercity differences that appear on four of the six substrates with the model. Results for pooled city data give an indication of how the instrument performed under a broader range of conditions than were present in any one of the two cities alone.

6.4.4.3 Results for MAP-3 K-shell

MAP-3 readings were taken by three different machines, each

with a different operator. The machines were grouped into two field classifications (Class I and Class II), so that each sampled location had two MAP-3 readings. Class I represented an

Table 6-65. Lead Analyzer L-shell on Wood: Model Estimates.

DEVICE	SAMPLE SIZE	MODEL PARAMETERS				Pb=0.0 mg/cm²		Pb=1.0 mg/cm²	
		a	b	c	d	BIAS	SD	BIAS	SD
Machine 1, Denver (ICP < 5)	287	-0.020 (.001)	0.303 (.018)	0.0001 (.0000)	0.029 (.005)	-0.020 (.001)	0.011	-0.717 (.017)	0.171
Machine 1, Phila. (ICP < 5)	38	0.034 (.010)	0.089 (.017)	0.003 (.001)	0.001 (.001)	0.034 (.016)	0.055	-0.877 (.017)	0.063
Machine 1, combined (ICP < 5)	325	-0.019 (.001)	0.289 (.017)	0.0002 (.0000)	0.035 (.005)	-0.019 (.001)	0.012	-0.730 (.017)	0.188
Machines 1 and 2 (ICP < 5)	337	-0.019 (.001)	0.279 (.016)	0.0002 (.0000)	0.034 (.005)	-0.019 (.001)	0.012	-0.740 (.016)	0.184

Table 6-66. Lead Analyzer L-shell on Wood: Control Block Summary.

DEVICE	SAMPLE SIZE	BARE (0.0 mg/cm²)		RED NIST SRM (1.02 mg/cm²)		YELLOW NIST SRM (3.53 mg/cm²)	
		BIAS	SD	BIAS	SD	BIAS	SD
Machine 1	63	-0.020 (.003)	0.020	0.055 (.008)	0.065	-0.764 (.019)	0.151
Machine 2	12	-0.042 (.003)	0.009	-0.000 (.006)	0.020	-0.844 (.013)	0.045
Machines 1 and 2	75	-0.023 (.002)	0.019	0.046 (.007)	0.061	-0.777 (.016)	0.140

entire set of readings across all locations, as did Class II. The machines and their operators are designated as follows:

Machine 10 (Operator C): Denver and Philadelphia, Class I
Machine 11 (Operator D): Denver, Class II
Machine 12 (Operator E): Philadelphia, Class II

Data from the Louisville pilot study were not used, because the nominal time of measurement was different, and because the instrument in the pilot study did not display negative values, unlike the full study. Data from the full study using nominal 60 second ("specials") and 240 second ("special-specials") reading also were not used in the present analysis.

Machine 10 was the only Class I instrument: it was used at all Denver and Philadelphia locations. Machines 11 and 12 together comprised Class II, which represented a second set of measurements at the same locations where Machine 10 was used. The issue of statistical dependence between Machines 10 and 11, and between Machines 10 and 12 thus arises, as explained in section 6.4.8.3. On the other hand, repeated measurement at the same locations affords the use of matched pairs methods, which in many ways are preferable as a means of testing for machine or operator effects.

Separating operator from machine effects was, however, impossible because of the association of machines with operators. Reference to a machine should always be understood to be a reference to a machine-operator pair, unless indicated otherwise.

6.4.4.3.1 MAP-3 K-shell on Brick

There were 185 observations of the MAP-3 K-shell on brick, none of which were designated as outliers. For Class I, readings on Machine 10 were made 93 times: 81 in Denver and 12 in Philadelphia. For Class II, readings on Machine 11 (Denver) were made 80 times, and on Machine 12 (Philadelphia) 12 times.

<u>Matched pairs analysis</u>: Two matched pairs comparisons were made on the data:

(1) Machines 10 and 11 were compared on the 80 Denver sites where both made readings. There were no cases where the two machines read exactly the same. On 43 occasions Machine 10

read higher than Machine 11, which is not an unusual occurrence under a 50-50 chance hypothesis.

(2) Machines 10 and 12 were compared on 12 common Philadelphia sites. There were no cases of equality, and 6 of the 12 sites had higher Machine 10 readings. This is not unusual if either machine has a 50 percent chance of reading higher than the other. Thus, machine effects are not indicated.

Figure 6-18 shows the response and SD components of the estimated model before provision for the combined effect of spatial variation and laboratory error in ICP measurements. The nonparametric response function (dashed line) indicates a flattening out at higher lead levels. Since there was a large gap in ICP measurements between 0.8042 mg/cm^2 and 4.6567 mg/cm^2 where a change in the relationship may have occurred, it is difficult to accurately predict how the instrument would perform at 1.0 mg/cm^2. The SD component appears to agree with the nonparametric estimate reasonably well for ICP measurements below 5.0 mg/cm^2.

Table 6-67 gives the results of fitting XRF measurement models to Machines 10, 11, and pooled. There were too few data to fit a separate model to the Machine 12 data. Pooling took into account the dependence between Class I and Class II instruments, and the standard errors indicated are conservative estimates. Both machines exhibited high negative bias even in the absence of lead. The control block data summary (Table 6-68) also appears to confirm this. The SD estimates at 0.0 mg/cm^2 from the model and the control blocks are close, but two of the machines even exhibited decreasing variability on the control blocks with increasing lead levels.

6.4.4.3.2 MAP-3 K-shell on Concrete

There were 436 observations of the MAP-3 K-shell on concrete, none of which were designated as outliers. For Class I, readings on Machine 10 were made 218 times: 98 in Denver and 120 in Philadelphia. For Class II, readings were made on Machine 11 (Denver) 98 times, and on Machine 12 (Philadelphia) 120 times.

<u>Matched pairs analysis</u>: Two matched pairs comparisons were made on the data:

(1) Machines 10 and 11 were compared on the 98 common Denver sites. There were no cases where the two machines read exactly the same. On 54 of the sites Machine 10 read higher than Machine 11, and the resulting sign test indicates that this is not unusual under a 50-50 chance hypothesis.

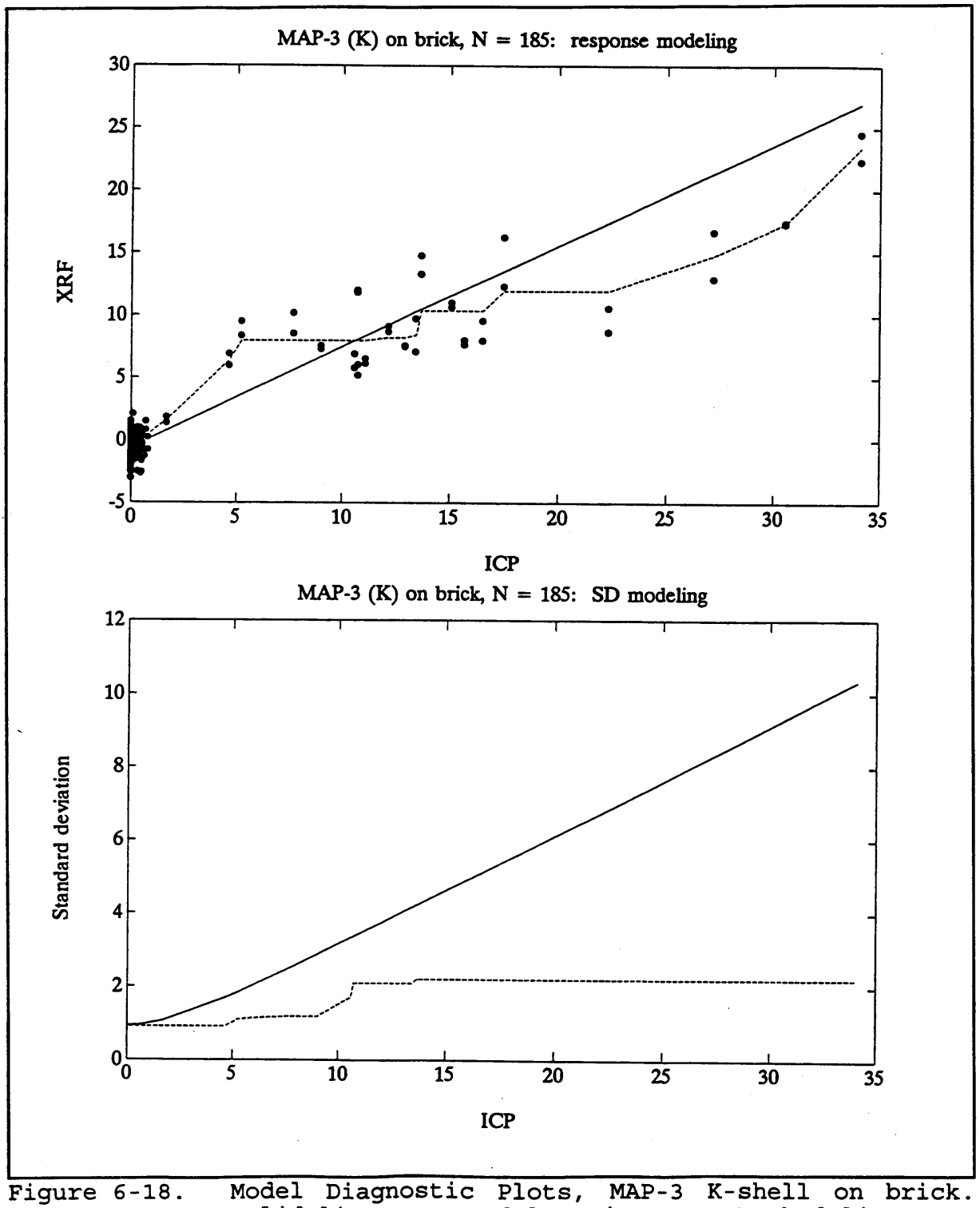


Figure 6-18. Model Diagnostic Plots, MAP-3 K-shell on brick. Solid lines are model estimates. Dashed lines are nonparametric (monotone regression) estimates.

Table 6-67. MAP-3 K-shell on Brick: Model Estimates.

DEVICE	SAMPLE SIZE	MODEL PARAMETERS				Pb=0.0 mg/cm²		Pb=1.0 mg/cm²	
		a	b	c	d	BIAS	SD	BIAS	SD
Machine 10	93	-0.554 (.092)	0.818 (.065)	0.581 (.098)	0.013 (.022)	-0.554 (.092)	0.762	-0.733 (0.080)	0.771
Machine 11	80	-0.616 (.134)	0.769 (.063)	1.025 (.191)	0.011 (.019)	-0.616 (.134)	1.012	-0.847 (.120)	1.018
All Machines (10, 11, and 12)	185	-0.599 (.079)	0.797 (.045)	0.857 (.103)	0.012 (.014)	-0.599 (.079)	0.926	-0.802 (.081)	0.932

Table 6-68. MAP-3 K-shell on Brick: Control Block Summary.

DEVICE	SAMPLE SIZE	BARE (0.0 mg/cm²)		RED NIST SRM (1.02 mg/cm²)		YELLOW NIST SRM (3.53 mg/cm²)	
		BIAS	SD	BIAS	SD	BIAS	SD
Machine 10	60	-0.931 (.085)	0.655	0.075 (.077)	0.600	-0.019 (.065)	0.507
Machine 11	30	-1.494 (.166)	0.909	-0.224 (.119)	0.651	-0.254 (.087)	0.474
Machine 12	30	-1.377 (.110)	0.603	-0.104 (.110)	0.604	-0.372 (.196)	1.073
Machines 10, 11, and 12	120	-1.183 (.065)	0.715	-0.045 (.056)	0.614	-0.166 (.063)	0.686

(2) Machines 10 and 12 were compared on the 120 common Philadelphia sites. There were no cases of equal measurements, and on 80 sites Machine 10 read higher than Machine 12. This is not compatible with the 50-50 chance hypothesis: the p-value of the sign test is about 0.04 percent, suggesting that Machine 12 read systematically lower than Machine 10, and possibly lower than Machine 11 as well.

Figure 6-19 shows the response and SD components of the estimated model before provision for the combined effect of spatial variation and laboratory error in ICP measurements. Both components appear to be in agreement with the nonparametric estimates.

Table 6-69 gives the results of fitting XRF measurement models to the data. Machines 11 and 12 have very similar model parameter estimates that are not statistically significantly different when compared with a chi-square test: the p-value exceeds 40 percent. Splitting Machine 10 into Denver and Philadelphia data was revealing, because the difference in intercepts (-0.779 Denver and -0.413 Philadelphia) may be the reason behind the significant sign test described in (2) above. The resulting chi-square statistic has a p-value of about 0.5 percent. In other words, the effect may be due to factors associated with the city, instead of the machine.

Table 6-70 gives the control block data summary. Bias in the control block data at 0.0 mg/cm^2 was higher than indicated in the model fits controlling for city and machine. The bias declined on the control blocks at the two higher lead levels, and to a much lesser extent in the models. SD estimates from the model are higher than on the control blocks, which may indicate the presence of non-instrumental sources of variability.

6.4.4.3.3 MAP-3 K-shell on Drywall

There were 226 observations of the MAP-3 K-shell on drywall, 4 of which were designated as outliers (Machine 10: 80343, 80345; Machine 11: 80332, 80345), leaving 222 observations for analysis. For Class I, readings on Machine 10 were made 111 times: 103 in Denver and 8 in Philadelphia. For Class II, readings on Machine 11 (Denver) were made 103 times and on Machine 12 (Philadelphia) 8 times.

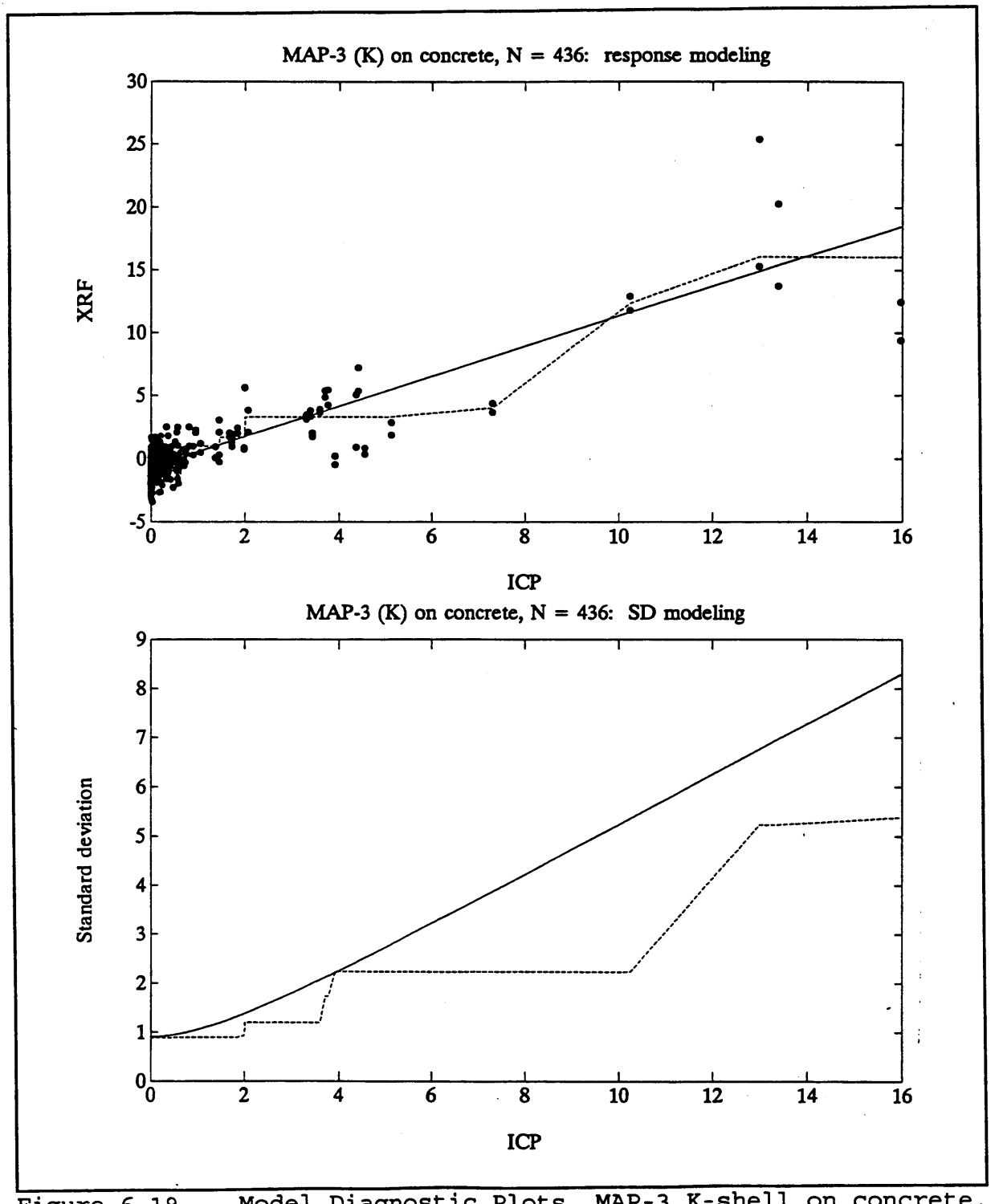


Figure 6-19. Model Diagnostic Plots, MAP-3 K-shell on concrete. Solid lines are model estimates. Dashed lines are nonparametric (monotone regression) estimates.

Table 6-69. MAP-3 K-shell on Concrete: Model Estimates.

DEVICE	SAMPLE SIZE	MODEL PARAMETERS				Pb=0.0 mg/cm²		Pb=1.0 mg/cm²	
		a	b	c	d	BIAS	SD	BIAS	SD
Machine 10, Denver	98	-0.779 (.083)	1.225 (.161)	0.506 (.082)	0.308 (.152)	-0.779 (.083)	0.711	-0.554 (.150)	0.902
Machine 10, Philadelphia	120	-0.413 (.089)	1.156 (.150)	0.575 (.076)	-0-	-0.413 (.089)	0.758	-0.257 (.150)	0.758
Machine 10, combined	218	-0.590 (.062)	1.264 (.123)	0.563 (.060)	0.229 (.097)	-0.590 (.062)	0.751	-0.325 (.108)	0.890
Machine 11	98	-0.722 (.124)	1.106 (.152)	1.163 (.192)	0.178 (.126)	-0.722 (.124)	1.078	-0.616 (.165)	1.158
Machine 12	120	-0.766 (.115)	1.253 (.192)	0.974 (.127)	-0-	-0.766 (.115)	0.987	-0.513 (.151)	0.987
Machines 10, 11, and 12	436	-0.661 (.072)	1.212 (.123)	0.807 (.085)	0.182 (.094)	-0.661 (.072)	0.899	-0.449 (.097)	0.995

Table 6-70. MAP-3 K-shell on Concrete: Control Block Summary.

DEVICE	SAMPLE SIZE	BARE (0.0 mg/cm²)		RED NIST SRM (1.02 mg/cm²)		YELLOW NIST SRM (3.53 mg/cm²)	
		BIAS	SD	BIAS	SD	BIAS	SD
Machine 10	72	-1.008 (.076)	0.643	-0.097 (.075)	0.633	-0.149 (.057)	0.486
Machine 11	38	-1.366 (.091)	0.564	-0.174 (.103)	0.636	-0.085 (.086)	0.533
Machine 12	34	-1.409 (.125)	0.726	-0.373 (.132)	0.771	-0.116 (.081)	0.472
Machines 10, 11, and 12	144	-1.197 (.054)	0.644	-0.183 (.056)	0.669	-0.124 (.041)	0.496

Matched pairs analysis: Two matched pairs comparisons were made on the data:

(1) Machines 10 and 11 were compared on 102 Denver sites of common measurement. On 43 sites Machine 10 read higher than Machine 11 (there were no ties), which is a plausible outcome under a 50-50 chance hypothesis. But the differences were correlated with the ICP measurement (the Spearman rank correlation is 0.3), which suggests that Machine 10 read higher than Machine 11 as the lead level increased.

(2) Machines 10 and 11 were compared on 8 common Philadelphia sites, of which 2 had a higher Machine 10 reading (there were no ties). The p-value is 28.8 percent, again a plausible outcome under a 50-50 chance hypothesis.

Figure 6-20 shows the response and SD components of the estimated model before provision for the combined effect of spatial variation and laboratory error in ICP measurements. Both components appear to fit the data reasonably well, and both agree with the nonparametric estimates. Since no ICP measurement exceeded 1.0 mg/cm^2 on drywall, extrapolation of the model to higher lead levels appears to be tenuous.

Table 6-71 gives the results of fitting XRF measurement models to Machines 10 and 11 separately, and pooled. The slope parameters *b* of the two machines (1.267 Machine 10 versus 0.378 Machine 11) confirm what was found in the matched pairs analysis, and explain the bias estimates at 1.0 mg/cm^2. A machine effect may have been present, in which case the pooled estimates would not be indicative of how a particular machine performed.

Table 6-72 gives the control block data summary. There is no apparent difference between Machines 10 and 11, contrary to what is suggested in the field sample data (i.e., data from readings on painted housing components). Since operator intervention differed somewhat between usage of the instrument in the field samples and on the control blocks, the possibility of assigning what appears to be machine effects to the operator needs to be considered. Machine 10 exhibited greater similarity in performance on the control blocks and on painted samples than did the other instruments. Bias is not indicated as a significant phenomenon except at higher levels of lead.

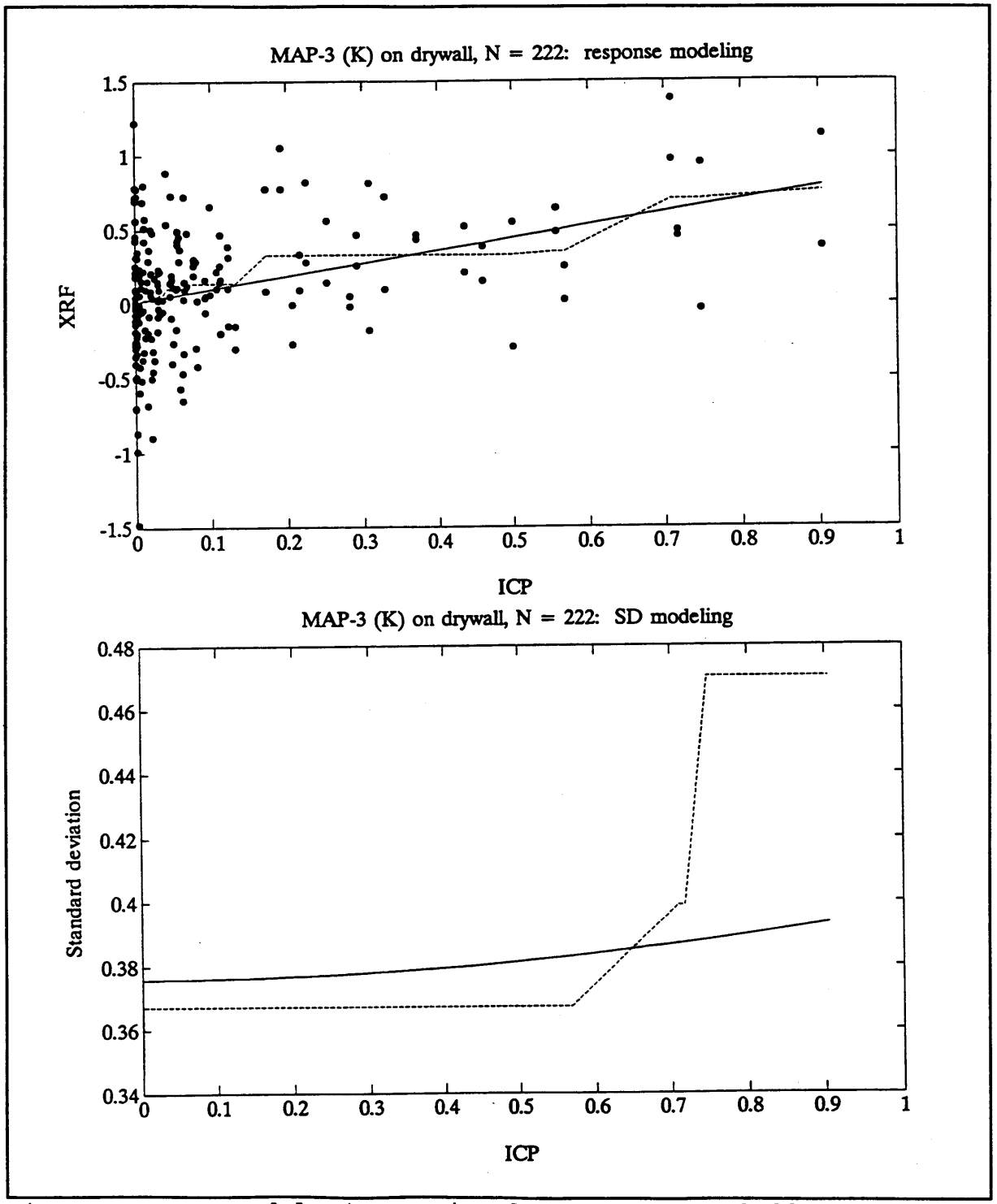


Figure 6-20. Model Diagnostic Plots, MAP-3 K-shell on drywall. Solid lines are model estimates. Dashed lines are nonparametric (monotone regression) estimates.

Table 6-71. MAP-3 K-shell on Drywall: Model Estimates.

DEVICE	SAMPLE SIZE	MODEL PARAMETERS				Pb=0.0 mg/cm²		Pb=1.0 mg/cm²	
		a	b	c	d	BIAS	SD	BIAS	SD
Machine 10	111	-0.058 (.036)	1.267 (.215)	0.105 (.015)	-0-	-0.058 (.036)	0.324	0.209 (.199)	0.324
Machine 11	103	0.112 (.044)	0.388 (.212)	0.144 (.021)	-0-	0.112 (.044)	0.380	-0.500 (.193)	0.380
All Machines (10, 11, and 12)	222	0.014 (.040)	0.863 (.209)	0.141 (.018)	-0-	0.014 (.040)	0.375	-0.123 (.042)	0.375

Table 6-72. MAP-3 K-shell on Drywall: Control Block Summary.

DEVICE	SAMPLE SIZE	BARE (0.0 mg/cm²)		RED NIST SRM (1.02 mg/cm²)		YELLOW NIST SRM (3.53 mg/cm²)	
		BIAS	SD	BIAS	SD	BIAS	SD
Machine 10	60	-0.082 (.030)	0.235	0.008 (.040)	0.307	-0.247 (.046)	0.353
Machine 11	34	-0.115 (.056)	0.324	0.015 (.050)	0.293	-0.147 (.060)	0.348
Machine 12	26	-0.115 (.058)	0.297	0.134 (.086)	0.439	-0.244 (.082)	0.419
Machines 10, 11, and 12	120	-0.099 (.025)	0.276	0.037 (.031)	0.336	-0.218 (.033)	0.367

6.4.4.3.4 MAP-3 K-shell on Metal

There were 378 observations of the MAP-3 K-shell on metal, 3 of which were designated as outliers (Machine 10: 81348; Machine 12: 81348, 81944), leaving 375 observations for analysis. For Class I, readings on Machine 10 were made 188 times: 62 in Denver and 126 in Philadelphia. For Class II, readings were made on Machine 11 (Denver) 62 times and on Machine 12 (Philadelphia) 125 times.

Matched pairs analysis: Two matched pairs comparisons were made on the data:

(1) Machines 10 and 11 were compared on 62 Denver sites where readings on both were made. There were no cases of tied values, and on 34 sites Machine 10 had a higher reading than Machine 11. This is very plausible under a 50-50 chance hypothesis. The differences were not highly correlated with the ICP measurement.

(2) Machines 10 and 12 were compared on 125 Philadelphia sites of common measurement. On 66 of these sites Machine 10 had the higher reading, with no tied measurements, which likewise is a plausible outcome assuming no machine effects. The sign test therefore does not indicate that a machine effect was present.

Figure 6-21 shows the response and SD components of the estimated model before provision for the combined effect of spatial variation and laboratory error in ICP measurements. Both seem to explain the data in an adequate fashion in the lower ICP range, although flattening of the nonparametric response is apparent at higher ICP measurements. The scatterplot in the first graph reveals an observation at ICP = 2.63 mg/cm^2 and XRF = -1.253 mg/cm^2 that is unusual with respect to the general pattern of the data. This observation had sample ID number 81636, and its XRF reading was made by Machine 12. Although this observation did not meet the outlier criterion developed in section 6.3, it barely failed to do so. This observation was deleted from subsequent analyses.

Table 6-73 gives the results of fitting XRF measurement models to the field sample data. There is a moderate indication of a city effect comparing Denver to Philadelphia on Machine 10

(the chi-square p-value is approximately 0.25 percent).

Table 6-74 gives the control block data summary. Estimates

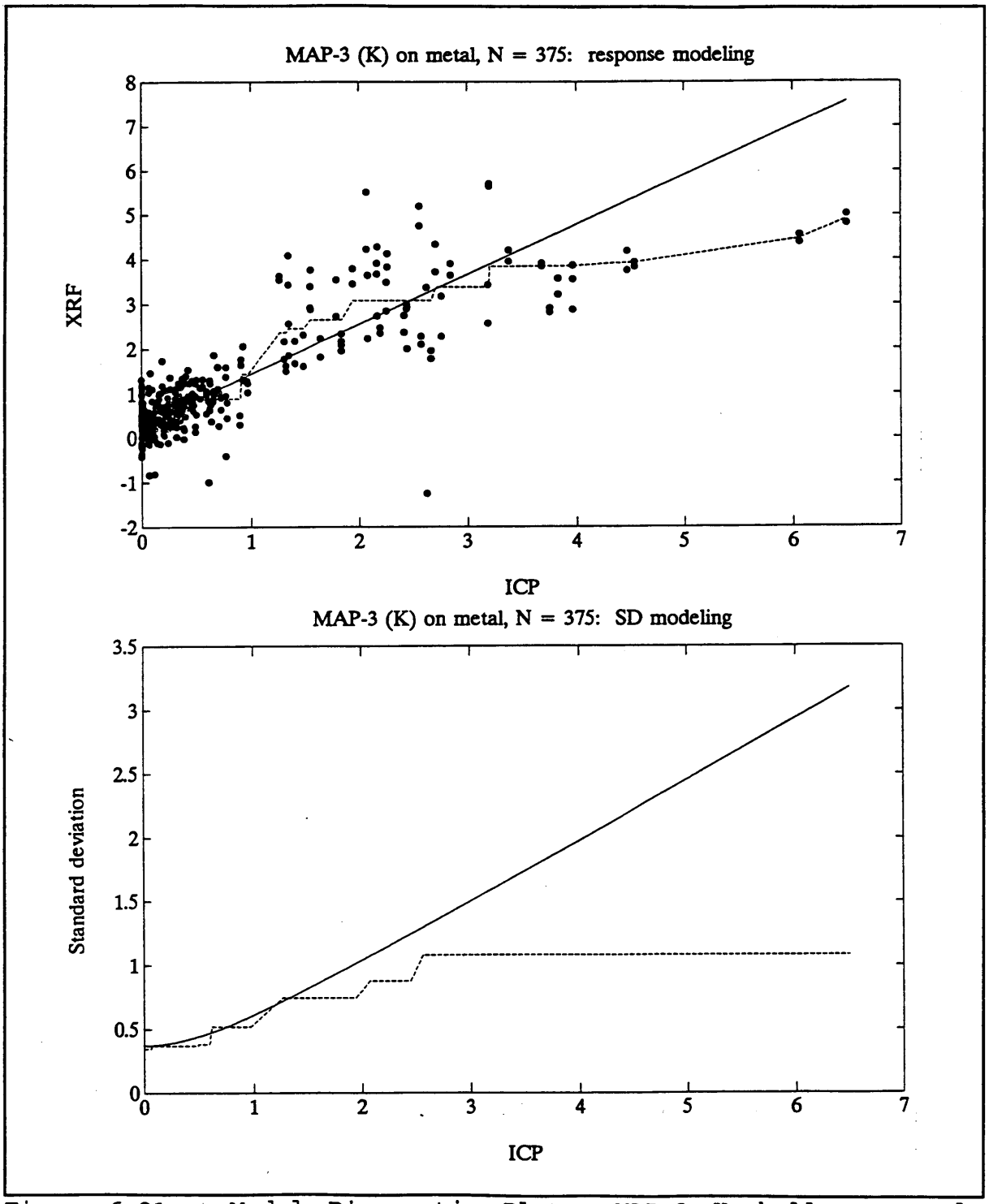


Figure 6-21. Model Diagnostic Plots, MAP-3 K-shell on metal. Solid lines are model estimates. Dashed lines are nonparametric (monotone regression) estimates.

Table 6-73. MAP-3 K-shell on Metal: Model Estimates.

DEVICE	SAMPLE SIZE	MODEL PARAMETERS				Pb=0.0 mg/cm²		Pb=1.0 mg/cm²	
		a	b	c	d	BIAS	SD	BIAS	SD
Machine 10, Denver	62	0.395 (.060)	1.388 (.158)	0.149 (.031)	0.188 (.126)	0.395 (.060)	0.386	0.783 (.147)	0.581
Machine 10, Philadelphia	125	0.233 (.040)	1.162 (.077)	0.069 (.014)	0.124 (.044)	0.233 (.040)	0.264	0.395 (.059)	0.440
Machine 10, combined	188	0.311 (.034)	1.144 (.064)	0.109 (.014)	0.122 (.038)	0.311 (.034)	0.330	0.455 (.057)	0.481
Machine 11	62	0.381 (.068)	1.285 (.113)	0.198 (.040)	-0-	0.381 (.068)	0.445	0.666 (.110)	0.445
Machine 12	125	0.292 (.055)	1.024 (.101)	0.140 (.030)	0.245 (.085)	0.292 (.055)	0.374	0.316 (.098)	0.620
All Machines (10, 11, and 12)	374	0.328 (.039)	1.098 (.071)	0.140 (.019)	0.159 (.049)	0.328 (.039)	0.374	0.421 (.052)	0.547

Table 6-74. MAP-3 K-shell on Metal: Control Block Summary.

DEVICE	SAMPLE SIZE	BARE (0.0 mg/cm²)		RED NIST SRM (1.02 mg/cm²)		YELLOW NIST SRM (3.53 mg/cm²)	
		BIAS	SD	BIAS	SD	BIAS	SD
Machine 10	74	0.245 (.025)	0.213	0.153 (.026)	0.228	-0.149 (.036)	0.301
Machine 11	40	0.177 (.032)	0.204	0.262 (.045)	0.283	-0.244 (.106)	0.668
Machine 12	34	0.254 (.037)	0.215	0.138 (.041)	0.242	-0.257 (.091)	0.531
Machines 10, 11, and 12	148	0.229 (.017)	0.211	0.179 (.020)	0.247	-0.199 (.039)	0.479

of the SD at 1.02 mg/cm^2 are lower than the model would indicate, which was possibly due to non-instrumental factors that affected variability in instrument readings. Bias estimates at 0.0 mg/cm^2 are similar between the control block and field sample data, but decreasing in the control blocks to a noticeable degree with increasing lead levels, which is not seen in the model estimates.

6.4.4.3.5 MAP-3 K-shell on Plaster

There were 444 observations of the MAP-3 K-shell on plaster, one of which was designated as an outlier (Machine 10: 80262) and removed, leaving 443 observations for analysis. For Class I, readings on Machine 10 were made 221 times: 100 in Denver and 121 in Philadelphia. For Class II, readings on Machine 11 (Denver) were made 101 times, and on Machine 12 (Philadelphia) 121 times.

Matched pairs analysis: Two matched pairs comparisons can be made with the data:

(1) Machines 10 and 11 were compared on the 100 Denver sites where readings on both were available. On 67 of these, Machine 10 read higher than Machine 11 (no ties). The resulting p-value of the sign test is less than 0.2 percent, suggesting that Machine 10 read higher than Machine 11. It should be noted, moreover, that Machine 10 read higher than Machine 11 on each of the 11 sites having the highest ICP measurements, 9 of which were greater than 5.0 mg/cm^2.

(2) Machines 10 and 12 were compared on the 121 Philadelphia sites of common measurement. On 69 of these Machine 10 had the larger reading (no ties), which is not unusual under a 50-50 chance hypotheses, where 60.5 such occurrences are expected. It should be noted that the largest ICP measurement of these sites was only 2.64 mg/cm^2, so that the possibility of an effect at higher lead levels cannot be ruled out.

Figure 6-22 shows the response and SD components of the estimated model before provision for the combined effect of spatial variation and laboratory error in ICP measurements. The components agree reasonably well with nonparametric estimates at lower lead levels. Usage of the model to infer instrument performance characteristics at lower lead levels appears to be justified.

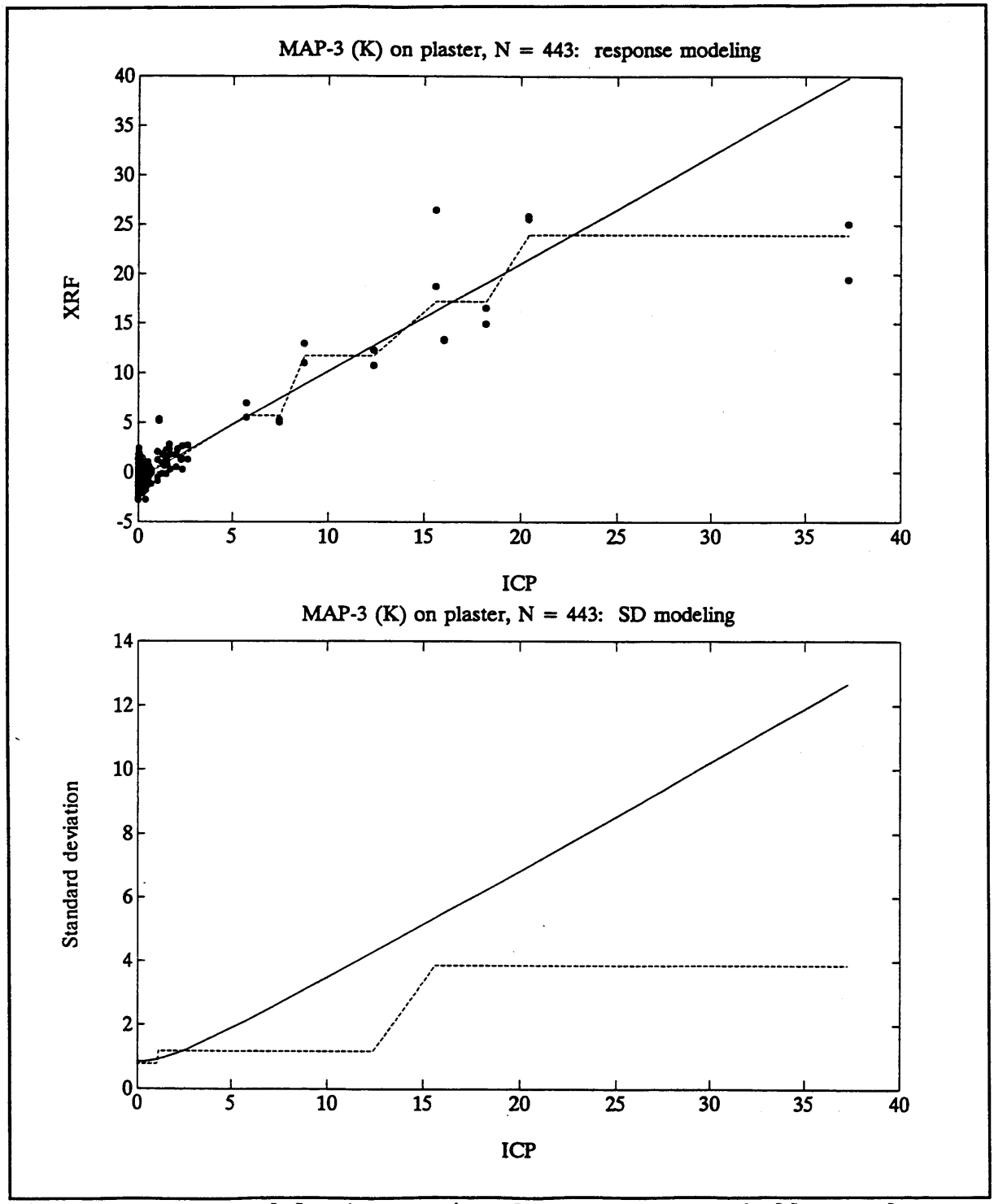


Figure 6-22. Model Diagnostic Plots, MAP-3 K-shell on plaster. Solid lines are model estimates. Dashed lines are nonparametric (monotone regression) estimates.

Table 6-75 gives the results of fitting XRF measurement models to the data. City effects within Machine 10 are indicated especially in the intercept terms (-0.421 Denver versus -0.825 Philadelphia), and the resulting chi-square test comparing the four parameters is highly significant. Machine 11, which was used only in Denver, also has a higher intercept term (-0.550) than Machine 12 (-0.975), which was used only in Philadelphia. The highest ICP readings were obtained in Denver, in a building that was nearly a century old. This may explain both the city effect and the machine effect (within Denver) that was exhibited only on field samples with high ICP measurements.

Table 6-76 gives a summary of the control block data. Bias at 1.02 mg/cm^2 and at 3.53 mg/cm^2 appeared to be similar, but was much larger at 0.0 mg/cm^2. Bias inferred from the model is likewise negative and significant, but not reflecting the same level of discrepancy between low and high lead levels seen in the control block data. The SD estimates do not appear to increase on the control blocks with the lead level. The SD estimates from the model are larger, which may reflect non-instrumental sources of variability.

6.4.4.3.6 MAP-3 K-shell on Wood

There were 698 observations of the MAP-3 K-shell on wood, 9 of which were designated as outliers (Machine 10: 80014, 80207, 80218, 80311, 80720; Machine 11: 80218, 80323, 80541, 80720), leaving 689 observations for analysis. For Class I, readings on Machine 10 were made 344 times: 292 in Denver and 52 in Philadelphia. For Class II, readings on Machine 11 (Denver) were made 293 times, and on Machine 12 (Philadelphia) 52 times.

Matched pairs analysis: Two matched pairs comparisons were made on the data:

(1) Machines 10 and 11 were compared on the 289 Denver sites where paired readings were available. On 153 occasions Machine 10 had the higher reading, with one tie. Under a 50-50 hypothesis this is a very plausible outcome. There is no indication that the differences between Machine 10 and 11 readings were related to the ICP measurement, and therefore no machine effect is apparent.

(2) Machines 10 and 12 were compared on the 52 Philadelphia

sites of common measurement. On 24 occasions Machine 10 had the higher reading, with no ties. This is a very plausible outcome if no machine effect is assumed, and there is no

Table 6-75. MAP-3 K-shell on Plaster: Model Estimates.

DEVICE	SAMPLE SIZE	MODEL PARAMETERS				Pb=0.0 mg/cm²		Pb=1.0 mg/cm²	
		a	b	c	d	BIAS	SD	BIAS	SD
Machine 10, Denver	100	-0.421 (.092)	1.153 (.149)	0.675 (.113)	0.175 (.157)	-0.421 (.092)	0.821	-0.268 (.149)	0.922
Machine 10, Philadelphia	121	-0.825 (.077)	1.404 (.157)	0.312 (.045)	0.066 (.088)	-0.825 (.077)	0.559	-0.421 (.114)	0.615
Machine 10, combined	221	-0.602 (.058)	1.163 (.105)	0.493 (.052)	0.117 (.074)	-0.602 (.058)	0.702	-0.438 (.090)	0.781
Machine 11	101	-0.550 (.115)	1.041 (.119)	1.098 (.175)	0.046 (.057)	-0.550 (.115)	1.048	-0.509 (.139)	1.070
Machine 12	121	-0.975 (.099)	1.266 (.179)	0.568 (.083)	0.043 (.132)	-0.975 (.099)	0.754	-0.709 (.160)	0.782
All Machines (10, 11, and 12)	443	-0.684 (.065)	1.137 (.102)	0.657 (.069)	0.094 (.067)	-0.684 (.065)	0.811	-0.547 (.091)	0.867

Table 6-76. MAP-3 K-shell on Plaster: Control Block Summary.

DEVICE	SAMPLE SIZE	BARE (0.0 mg/cm²)		RED NIST SRM (1.02 mg/cm²)		YELLOW NIST SRM (3.53 mg/cm²)	
		BIAS	SD	BIAS	SD	BIAS	SD
Machine 10	68	-1.295 (.070)	0.579	-0.296 (.073)	0.600	-0.360 (.064)	0.530
Machine 11	34	-1.431 (.149)	0.871	-0.289 (.081)	0.472	-0.354 (.094)	0.548
Machine 12	34	-1.500 (.118)	0.688	-0.570 (.101)	0.591	-0.402 (.091)	0.532
Machines 10, 11, and 12	136	-1.380 (.059)	0.689	-0.636 (.049)	0.569	-0.369 (.046)	0.535

indication that the differences were related to the measured lead levels.

Figure 6-23 shows the response and SD components of the estimated model before provision for the combined effect of spatial variation and laboratory error in ICP measurements. The nonparametric response estimate suggests a flattening out at higher lead levels, but the model appears to fit the data well in the lower ICP range. The same appears to be true for the estimated SD.

Table 6-77 gives the results of fitting XRF measurement models to the field sample data. Comparing Denver and Philadelphia within Machine 10 reveals an apparent city effect (the chi-square p-value is less than 0.01 percent), seen especially in the estimated slope parameters (1.275 in Denver versus 1.948 in Philadelphia). But the maximum ICP measurement for the Philadelphia sites was only 7.2333 mg/cm^2, which makes the comparison questionable.

Table 6-78 gives a summary of the control block data. Machine effects were not evident, and the bias at 0.0 mg/cm^2 was lower on wood than on other substrates. This agrees with the model estimates, but the bias inferred from the model increases and becomes positive at 1.0 mg/cm^2. SD estimates from the model are larger than those obtained from the control block data, suggesting the possibility of non-instrumental sources of variability.

6.4.4.3.7 MAP-3 K-shell: Summary of Analysis

The MAP-3 K-shell performed differently according to the substrate. On brick, concrete and plaster, the instrument exhibited substantial negative bias on the control blocks at 0.0 mg/cm^2, which became less pronounced as the lead level increased. Negative bias was prominent on these three substrates in the field sample data as well, but the bias did not follow the pattern seen on the control blocks. The instrument exhibited positive bias on metal in the field sample data, which was also reflected in the control block data except at the 3.53 mg/cm^2 lead level. The low order of bias exhibited on wood and drywall on the field samples was matched on the control blocks at 0.0 mg/cm^2, but not at higher lead levels for wood.

SD estimates from the models typically exceeded those obtained from control block data, especially with increasing lead levels. Larger SD estimates from the model suggest the presence

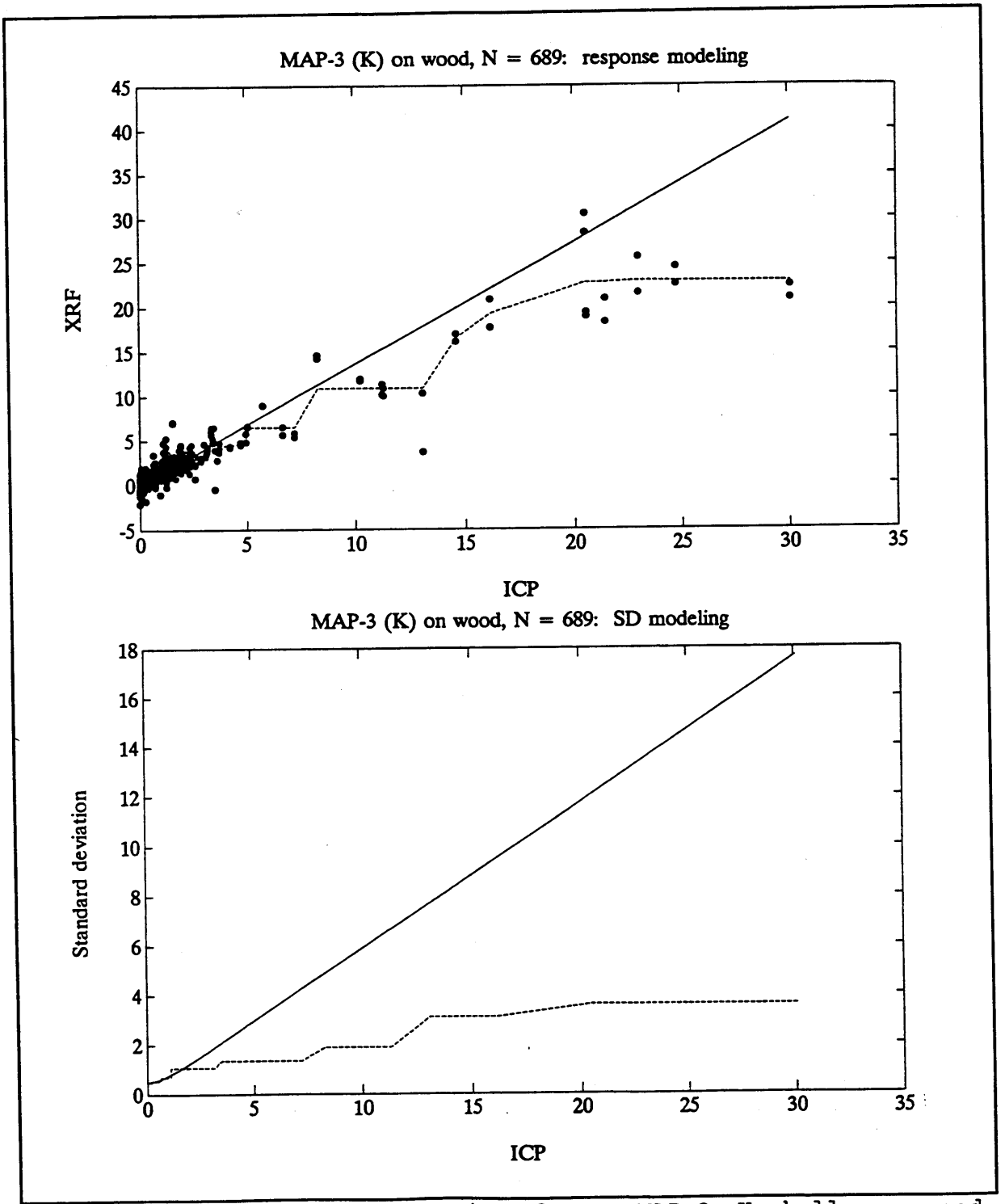


Figure 6-23. Model Diagnostic Plots, MAP-3 K-shell on wood. Solid lines are model estimates. Dashed lines are nonparametric (monotone regression) estimates.

Table 6-77. MAP-3 K-shell on Wood: Model Estimates.

DEVICE	SAMPLE SIZE	MODEL PARAMETERS				Pb=0.0 mg/cm²		Pb=1.0 mg/cm²	
		a	b	c	d	BIAS	SD	BIAS	SD
Machine 10, Denver	292	-0.023 (.034)	1.275 (.063)	0.208 (.021)	0.089 (.036)	-0.023 (.034)	0.456	0.252 (.058)	0.545
Machine 10, Philadelphia	52	-0.273 (.089)	1.948 (.170)	0.102 (.039)	0.460 (.174)	-0.273 (.089)	0.320	0.675 (.136)	0.750
Machine 10	344	-0.045 (.033)	1.427 (.065)	0.202 (.020)	0.194 (.050)	-0.044 (.033)	0.449	0.383 (.058)	0.629
Machine 11	292	-0.039 (.035)	1.256 (.052)	0.279 (.027)	0.002 (.028)	-0.039 (.035)	0.528	0.217 (.050)	0.530
Machine 12	52	-0.246 (.140)	1.792 (.230)	0.276 (.088)	0.721 (.250)	-0.246 (.140)	0.525	0.546 (.174)	0.999
All Machines (10, 11, and 12)	689	-0.052 (.036)	1.410 (.063)	0.239 (.025)	0.203 (.051)	-0.052 (.036)	0.488	0.358 (.057)	0.665

Table 6-78. MAP-3 K-shell on Wood: Control Block Summary.

DEVICE	SAMPLE SIZE	BARE (0.0 mg/cm²)		RED NIST SRM (1.02 mg/cm²)		YELLOW NIST SRM (3.53 mg/cm²)	
		BIAS	SD	BIAS	SD	BIAS	SD
Machine 10	72	-0.270 (.027)	0.228	-0.139 (.029)	0.250	-0.402 (.046)	0.390
Machine 11	38	-0.327 (.044)	0.269	-0.168 (.033)	0.202	-0.340 (.056)	0.348
Machine 12	34	-0.203 (.041)	0.242	-0.107 (.043)	0.250	-0.471 (.115)	0.670
Machines 10, 11, and 12	144	-0.269 (.020)	0.243	-0.139 (.020)	0.238	-0.402 (.038)	0.462

of non-instrumental factors that may have contributed to the variability of readings with this instrument.

City effects were indicated, especially at high lead levels. Effects attributed to machines, or more properly to machine-operator pairings since a different operator used each of the three machines in the study, can be discerned on certain substrates (concrete, drywall, and plaster) with the sign test. Machine effects are less strongly indicated in both the model estimates and the control block data summaries.

6.4.4.4 Results for MAP-3 L-shell

Data for the MAP-3 were taken with three different instruments, each with a different operator. The machines and their operators are designated as follows:

Machine 10 (Operator C): Denver and Philadelphia, Class I
Machine 11 (Operator D): Denver, Class II
Machine 12 (Operator E): Philadelphia, Class II

Data from the Louisville pilot study were not used because the time of measurement was different, and because the MAP-3 in Louisville did not produce negative readings, unlike the full study. Data from the full study using 60 second ("specials") and 240 second ("special-specials") readings were also not used in the analyses reported in this section.

There were two different field classifications for this instrument, which are designated here as Class I and Class II. Machine 10 was the only Class I instrument: it was used at all Denver and Philadelphia locations. Machines 11 and 12 together comprised Class II, which represented a second set of measurements at the same locations where Machine 10 was used.

Separating operator and machine effects was not possible because of the association of machines with operators. Reference to a machine should always be understood to be a reference to a machine-operator pair, unless indicated otherwise.

6.4.4.4.1 MAP-3 L-shell on Brick

There were 185 observations of the MAP-3 L-shell on brick, 2 of which were designated as outliers (Machine 10: 80908; Machine

11: 80038), leaving 183 observations in the analysis. For Class I, readings on Machine 10 were made 92 times: 80 in Denver and 12 in Philadelphia. For Class II, readings on Machine 11 (Denver) were made 79 times and on Machine 12 (Philadelphia) 12 times.

Matched pairs analysis: Two matched pairs comparisons were made on the data:

(1) Machines 10 and 11 were compared on the 78 Denver sites where both made readings. On 57 of these sites Machine 10 had a higher reading than Machine 11, with no ties. Machine 10 read higher an unusually large number of times: the sign test has a p-value of less than 0.01 percent. The Spearman rank correlation of the differences with the ICP measurements at the 78 sites was 0.311, which suggests that the effect was greater at higher lead levels.

(2) Machines 10 and 12 were compared on the 12 Philadelphia sites of common measurement. On 11 of these sites Machine 10 had a higher reading than Machine 12. This is very unlikely under a 50-50 chance hypothesis: the p-value of the sign test is 0.64 percent.

Figure 6-24 shows the response and SD components of the estimated model before provision for the combined effect of spatial variation and laboratory error in ICP measurements. The model appears to fit the data reasonably well, especially at lower lead levels. XRF readings less than 1.0 mg/cm^2 were observed 7 times on field samples with ICP measurements greater than 10.0 mg/cm^2.

Table 6-79 gives the results of fitting XRF measurement models to the data. Pooling across instruments may not be advisable due to the results of the matched pairs analysis. Table 6-80 gives a summary of the control block data. It is interesting to note that bias estimates in Tables 6-79 and 6-80 for Machine 10 are higher than those for Machines 11 and 12, which is consistent with the results of the matched pairs analysis. But there is very little congruity between the two tables, which is a common pattern across the range of L-shell instruments. The control block data suggest that the instrument on average read even higher than the true lead level at 1.02 mg/cm^2, which is not inferred from the field sample data. Bias

in the control block results showed a negative-positive-negative pattern as the lead level increased, which may suggest a nonlinear responsiveness to lead. The performance of the instrument on painted samples was very nonresponsive to changes in the lead level.

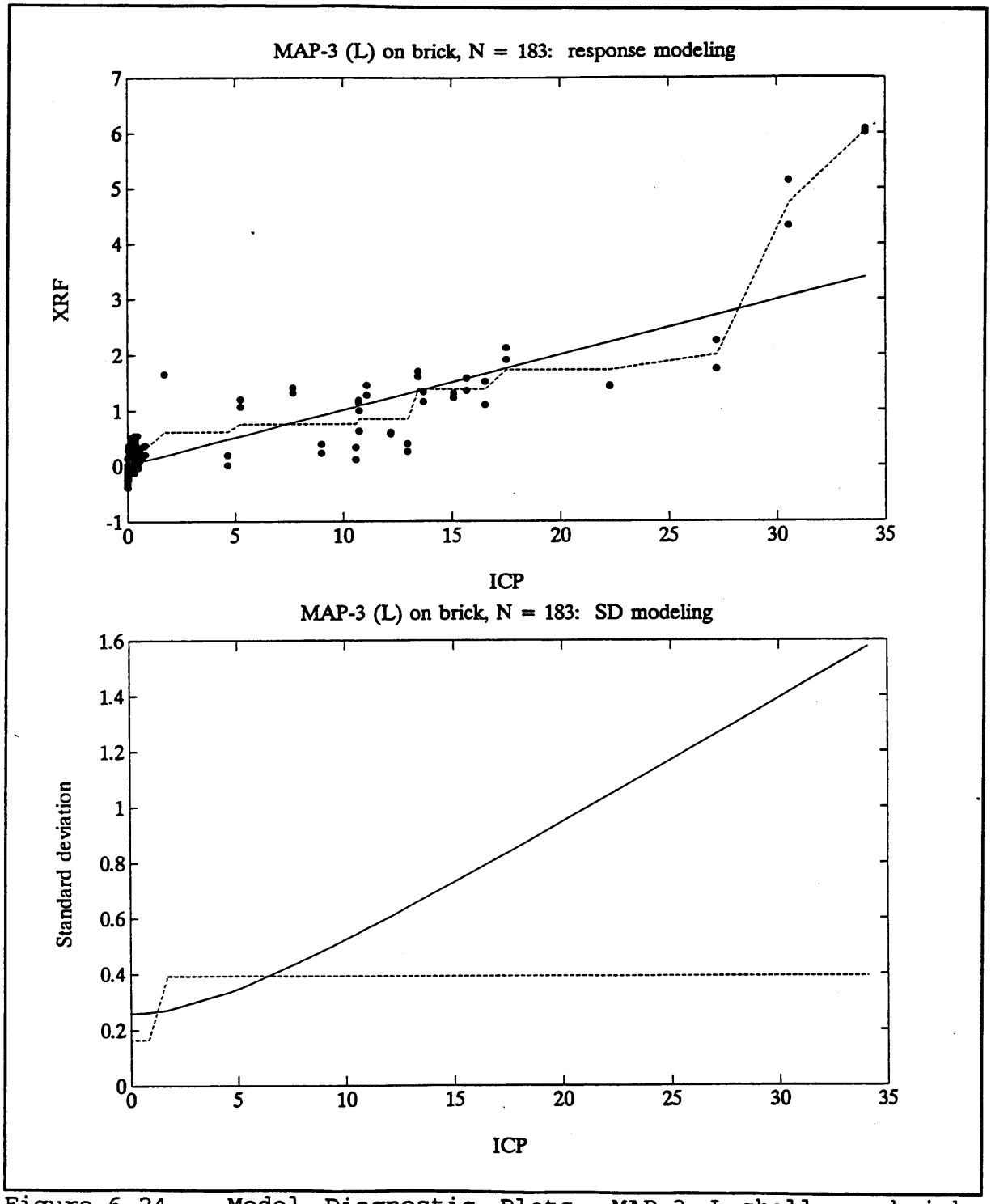


Figure 6-24. Model Diagnostic Plots, MAP-3 L-shell on brick. Solid lines are model estimates. Dashed lines are nonparametric (monotone regression) estimates.

Table 6-79. MAP-3 L-shell on Brick: Model Estimates.

DEVICE	SAMPLE SIZE	MODEL PARAMETERS				Pb=0.0 mg/cm²		Pb=1.0 mg/cm²	
		a	b	c	d	BIAS	SD	BIAS	SD
Machine 10, Denver	80	0.060 (.031)	0.098 (.012)	0.056 (.010)	0.0009 (.001)	0.060 (.031)	0.237	-0.842 (.030)	0.239
Machine 10, combined	92	0.034 (.028)	0.102 (.012)	0.056 (.010)	0.0009 (.001)	0.034 (.028)	0.237	-0.864 (.228)	0.239
Machine 11	79	0.025 (.030)	0.112 (.019)	0.048 (.010)	0.004 (.002)	0.025 (.030)	0.219	-0.863 (.027)	0.228
All Machines (10, 11, and 12)	183	0.012 (.029)	0.109 (.016)	0.055 (.010)	0.003 (.002)	0.012 (.029)	0.235	-0.880 (.029)	0.241

Table 6-80. MAP-3 L-shell on Brick: Control Block Summary.

DEVICE	SAMPLE SIZE	BARE (0.0 mg/cm²)		RED NIST SRM (1.02 mg/cm²)		YELLOW NIST SRM (3.53 mg/cm²)	
		BIAS	SD	BIAS	SD	BIAS	SD
Machine 10	60	-0.173 (.009)	0.069	0.197 (.011)	0.087	-0.142 (.018)	0.142
Machine 11	30	-0.205 (.023)	0.124	0.188 (.015)	0.081	-0.165 (.035)	0.189
Machine 12	30	-0.250 (.010)	0.055	0.104 (.025)	0.139	-0.223 (.132)	0.723
Machines 10, 11, and 12	120	-0.200 (.008)	0.083	0.172 (.009)	0.101	-0.168 (.035)	0.386

6.4.4.4.2 MAP-3 L-shell on Concrete

There were 436 observations of the MAP-3 L-shell on concrete, 3 of which were designated as outliers (Machine 10: 80058; Machine 11: 80938; Machine 12: 81234), leaving 433 observations for analysis. For Class I, readings on Machine 10 were made 216 times: 97 in Denver and 119 in Philadelphia. For Class II, readings on Machine 11 (Denver) were made 97 times and on Machine 12 (Philadelphia) 120 times.

Matched pairs analysis: Two matched pairs comparisons were made on the data:

(1) Machines 10 and 11 were compared on 96 Denver sites of common measurement. On 67 of these sites Machine 10 had the larger reading, with no ties. This result is very unlikely under a 50-50 chance hypothesis: the sign test has a p-value of about 0.01 percent. It thus appears that Machine 10 tended to give higher readings than Machine 11. There was a moderate rank correlation of 0.25 between the difference in readings and the ICP measurements, suggesting that the difference became larger as the lead level increased.

(2) Machines 10 and 12 were compared on 119 Philadelphia sites of common measurement. On 100 of these sites Machine 10 had the larger reading, again with no ties. Such an occurrence is practically impossible under a 50-50 chance hypothesis. It appears that Machine 10 read systematically higher than Machine 12, with little association between the difference in measurements and the lead level.

Figure 6-25 shows the response and SD components of the estimated model before provision for the combined effect of spatial variation and laboratory error in ICP measurements. The response does not agree well with the nonparametric estimate for ICP levels above 2.0 mg/cm^2. The SD component appears to agree with the nonparametric estimate if attention is restricted to lower ICP levels. XRF readings less than 1.0 mg/cm^2 were observed at ICP levels higher than 10.0 mg/cm^2. Below 10.0 mg/cm^2, almost all XRF readings were less than 1.0 mg/cm^2.

Table 6-81 gives the results of fitting XRF measurement models to the data. A city effect appears to exist, which can be

seen by comparing Denver and Philadelphia within Machine 10. It was especially apparent in the estimated slope parameters (0.373 Denver versus 0.167 Philadelphia), and it is notable that Machine

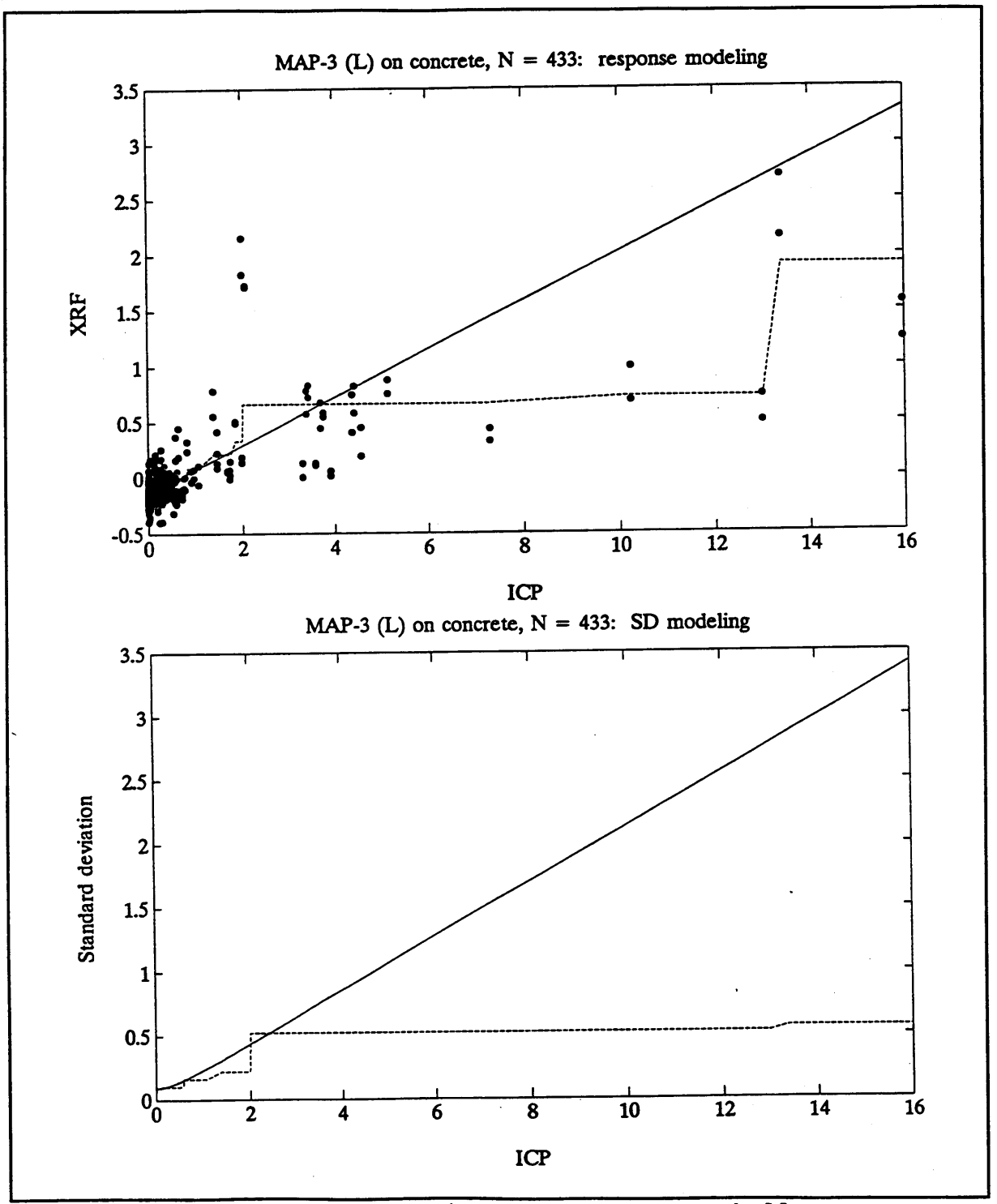


Figure 6-25. Model Diagnostic Plots, MAP-3 L-shell on concrete. Solid lines are model estimates. Dashed lines are nonparametric (monotone regression) estimates.

Table 6-81. MAP-3 L-shell on Concrete: Model Estimates.

DEVICE	SAMPLE SIZE	MODEL PARAMETERS				Pb=0.0 mg/cm²		Pb=1.0 mg/cm²	
		a	b	c	d	BIAS	SD	BIAS	SD
Machine 10, Denver	97	-0.098 (.013)	0.373 (.066)	0.009 (.002)	0.055 (.026)	-0.098 (.013)	0.095	-0.725 (.060)	0.253
Machine 10, Philadelphia	119	-0.130 (.001)	0.167 (.025)	0.004 (.001)	0.004 (.002)	-0.130 (.001)	0.063	-0.963 (.022)	0.089
Machine 10, combined	216	-0.117 (.008)	0.225 (.025)	0.008 (.001)	0.018 (.006)	-0.117 (.008)	0.087	-0.892 (.022)	0.161
Machine 11	97	-0.130 (.012)	0.319 (.059)	0.009 (.002)	0.044 (.021)	-0.130 (.012)	0.094	-0.812 (.055)	0.230
Machine 12	120	-0.195 (.010)	0.138 (.023)	0.006 (.001)	0.003 (.003)	-0.195 (.010)	0.075	-1.057 (.018)	0.093
All Machines (10, 11, and 12)	433	-0.141 (.008)	0.201 (.025)	0.008 (.001)	0.019 (.007)	-0.141 (.008)	0.090	-0.940 (.022)	0.163

Table 6-82. MAP-3 L-shell on Concrete: Control Block Summary.

DEVICE	SAMPLE SIZE	BARE (0.0 mg/cm²)		RED NIST SRM (1.02 mg/cm²)		YELLOW NIST SRM (3.53 mg/cm²)	
		BIAS	SD	BIAS	SD	BIAS	SD
Machine 10	72	-0.193 (.006)	0.047	0.202 (.009)	0.077	-0.060 (.016)	0.138
Machine 11	38	-0.229 (.027)	0.166	0.178 (.009)	0.058	-0.131 (.029)	0.181
Machine 12	34	-0.249 (.010)	0.057	0.141 (.016)	0.093	-0.084 (.041)	0.241
Machines 10, 11, and 12	144	-0.216 (.008)	0.096	0.181 (.006)	0.077	-0.085 (.015)	0.178

11 (Denver) versus Machine 12 (Philadelphia) also shows that Denver had the higher slope. This pattern was common across the L-shell instruments on certain substrates, in particular brick.

Table 6-82 gives a summary of the control block data. The estimates of bias suggest that the instrument was able to accurately measure the level of lead, even overestimating the lead level at 1.02 mg/cm^2. No such performance was observed on the field samples, where under-responsiveness to the level of lead in paint was the rule. The negative-positive-negative pattern in the bias estimates appearing in the control block summary may suggest nonlinear responsiveness to lead.

6.4.4.4.3 MAP-3 L-shell on Drywall

There were 226 observations of the MAP-3 L-shell on drywall, 2 of which were designated as outliers (Machine 10: 80227; Machine 11: 80332), leaving 224 observations for analysis. For Class I, readings on Machine 10 were made 112 times: 104 in Denver and 8 in Philadelphia. For Class II, readings on Machine 11 (Denver) were made 104 times, and on Machine 12 (Philadelphia) 8 times.

Matched pairs analysis: Two matched pairs comparisons were made on the data:

(1) Machines 10 and 11 were compared on the 103 Denver sites of common measurement. On only 31 occasions did Machine 10 read higher than Machine 11, with no ties. The sign test has a p-value of less than 0.1 percent, which suggests a tendency for Machine 10 to read lower than Machine 11. There was no apparent relationship of this tendency to the ICP measurement, but no ICP measurements in excess of 1.0 mg/cm^2 were available on drywall.

(2) Machines 10 and 12 were compared on the 8 Philadelphia sites of common measurement: Machine 10 had the higher reading on 3 occasions, with no ties. There is no indication that the two machines systematically differed in their readings.

Figure 6-26 shows the response and SD components of the estimated model before provision for the combined effect of spatial variation and laboratory error in ICP measurements. Both seem to provide a reasonable fit, and are close to the

nonparametric estimates. Extrapolation to a lead level as high as 1.0 mg/cm^2 is, however, difficult due to the restricted range of ICP measurements represented in the data.

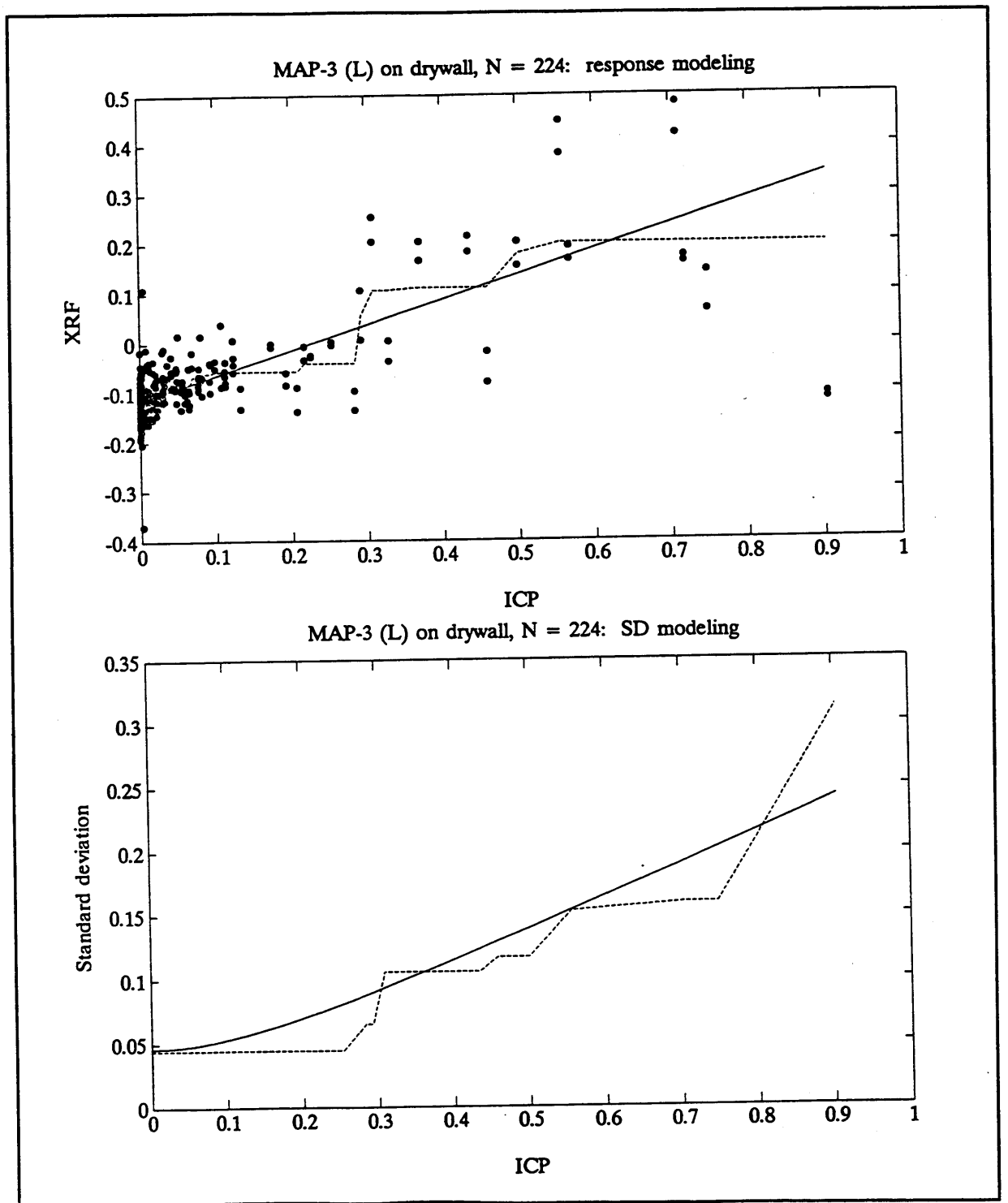


Figure 6-26. Model Diagnostic Plots, MAP-3 L-shell on drywall. Solid lines are model estimates. Dashed lines are nonparametric (monotone regression) estimates.

Table 6-83 gives the results of fitting XRF measurement models to the data. It was not possible to detect instrument or city effects given the limited data at factor level detail, but the inclusion of the Philadelphia data did not appear to affect the results substantially.

Table 6-84 gives a summary of the control block data. Bias estimates at 0.0 mg/cm^2 agree between the control block and model results. The larger model SD estimates may reflect non-instrumental sources of variability. At higher lead levels, however, the agreement in bias estimates no longer holds. The negative-positive-negative pattern seen in the control block bias as the lead level increases suggests a nonlinear responsiveness to lead.

6.4.4.4.4 MAP-3 L-shell on Metal

There were 378 observations of the MAP-3 L-shell on metal, of which 7 were designated as outliers (Machine 10: 81210, 81350, 81355; Machine 12: 81210, 81348, 81350, 81355), leaving 371 observations for analysis. For Class I, readings on Machine 10 were made 186 times: 62 in Denver and 124 in Philadelphia. For Class II, readings on Machine 11 (Denver) were made 62 times, and on Machine 12 (Philadelphia) 123 times.

Matched pairs analysis: Two matched pairs comparisons were made on the data:

(1) Machines 10 and 11 were compared on the 62 Denver sites where both made readings. Machine 10 had a higher reading than Machine 11 on 39 of these sites, with one tie. The resulting sign test has a p-value of about 5 percent, which suggest that Machine 10 may be prone to reading higher than Machine 11.

(2) Machines 10 and 12 were compared on the 123 Philadelphia sites common to both. Machine 10 had the higher reading 60 times, which is plausible under a 50-50 chance hypothesis.

Figure 6-27 shows the response and SD components of the estimated model before provision for the combined effect of spatial variation and laboratory error in ICP measurements. Both seem to fit the data reasonably well, and agree with the nonparametric estimates especially in the lower ICP range.

Table 6-83. MAP-3 L-shell on Drywall: Model Estimates.

DEVICE	SAMPLE SIZE	MODEL PARAMETERS				Pb=0.0 mg/cm²		Pb=1.0 mg/cm²	
		a	b	c	d	BIAS	SD	BIAS	SD
Machine 10	112	-0.123 (.005)	0.508 (.067)	0.002 (.0002)	0.064 (.024)	-0.123 (.005)	0.039	-0.615 (.064)	0.255
Machine 11	104	-0.097 (.005)	0.441 (.066)	0.002 (.0003)	0.059 (.023)	-0.097 (.005)	0.041	-0.656 (.063)	0.247
All Machines (10, 11, and 12)	224	-0.115 (.005)	0.498 (.060)	0.002 (.0003)	0.059 (.021)	-0.115 (.005)	0.046	-0.616 (.058)	0.247

Table 6-84. MAP-3 L-shell on Drywall: Control Block Summary.

DEVICE	SAMPLE SIZE	BARE (0.0 mg/cm²)		RED NIST SRM (1.02 mg/cm²)		YELLOW NIST SRM (3.53 mg/cm²)	
		BIAS	SD	BIAS	SD	BIAS	SD
Machine 10	60	-0.120 (.003)	0.022	0.151 (.008)	0.065	-0.173 (.022)	0.169
Machine 11	34	-0.096 (.004)	0.023	0.169 (.011)	0.066	-0.215 (.021)	0.122
Machine 12	26	-0.125 (.004)	0.020	0.234 (.073)	0.374	-0.149 (.035)	0.177
Machines 10, 11, and 12	120	-0.114 (.002)	0.022	0.174 (.017)	0.182	-0.180 (.015)	0.159

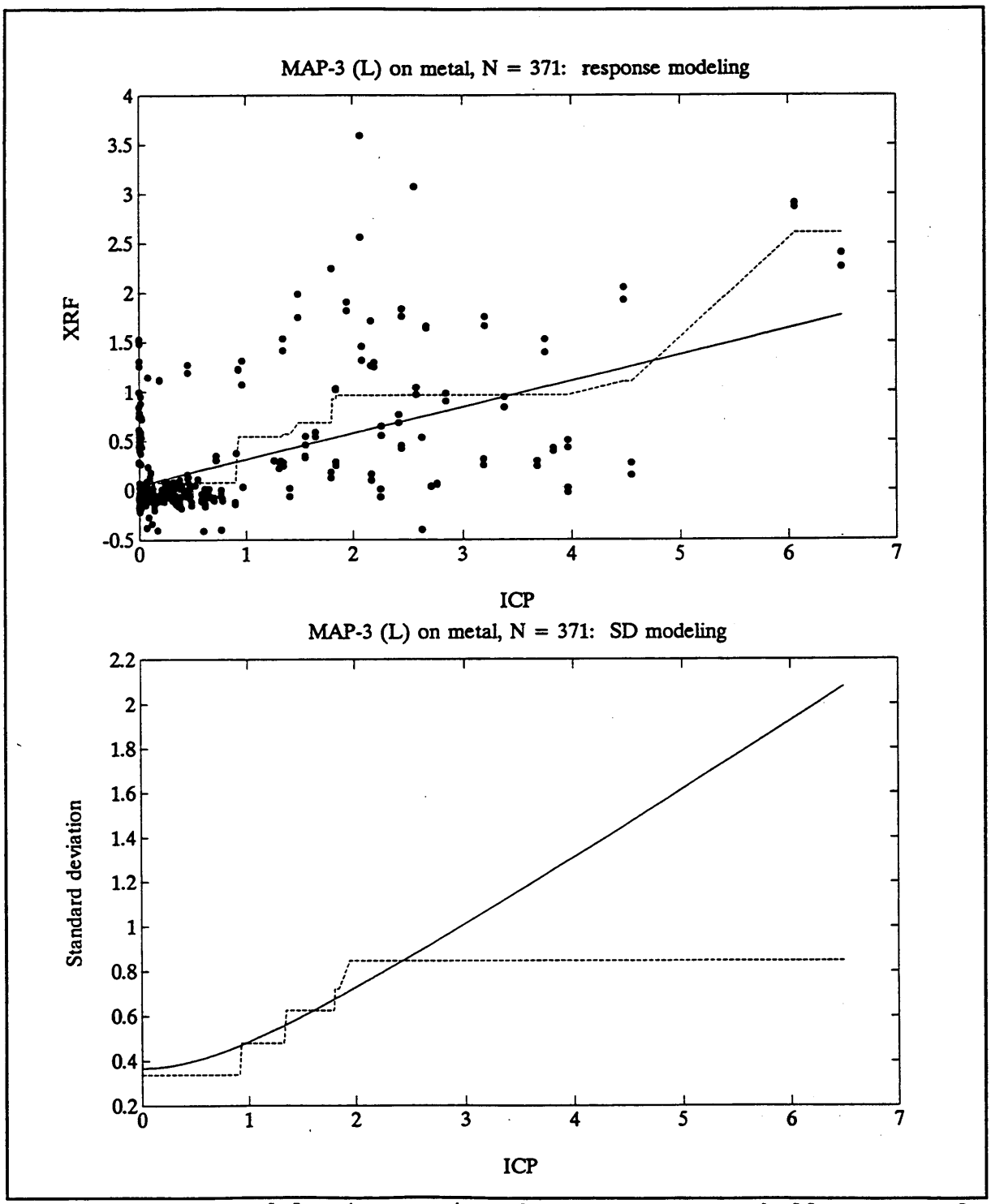


Figure 6-27. Model Diagnostic Plots, MAP-3 L-shell on metal. Solid lines are model estimates. Dashed lines are nonparametric (monotone regression) estimates.

Table 6-85 gives the results of fitting XRF measurement models to the data. Comparing Denver and Philadelphia within Machine 10 revealed a city effect much like the one seen on other substrates with this instrument. The slope for Denver (0.457) was higher than that for Philadelphia (0.256), and a chi-square test on all 4 model parameters has a p-value of less than 0.01 percent.

Pooling data across the two cities did not appear to be advisable. Comparing Machines 10 and 11 within Denver revealed a difference in the intercept terms that may explain the mildly significant sign test result.

Table 6-86 gives a summary of the control block data. It does not reveal major differences in the bias between the machines. A negative-positive-negative pattern in the bias, common to all substrates, is apparent as the lead level increases. Little bias is suggested at 1.02 mg/cm^2, which is a very different conclusion than drawn from the field sample data. The field sample performance of the instrument was much less responsive to changes in the lead level than indicated by the control block data summary.

6.4.4.4.5 MAP-3 L-shell on Plaster

There were 444 observations of the MAP-3 L-shell on plaster, none of which were designated as outliers. For Class I, readings on Machine 10 were made 222 times: 101 in Denver and 121 in Philadelphia. For Class II, readings on Machine 11 (Denver) were made 101 times, and on Machine 12 (Philadelphia) 121 times.

<u>Matched pairs analysis</u>: Two matched pairs comparisons were made on the data:

(1) Machines 10 and 11 were compared on the 101 Denver sites where both made readings. Machine 10 read higher than Machine 11 at 66 of these sites, with no ties. The resulting sign test has a p-value of about 0.3 percent, suggesting that Machine 10 read higher than Machine 11. At the 10 sites where the ICP measurement was greater than 2.0 mg/cm^2, Machine 10 had the higher reading 9 times, which suggests that the machine effect became prominent at higher lead levels.

(2) Machines 10 and 12 were compared on the 121 Philadelphia sites of common measurement. Machine 10 read higher than Machine 12 100 times, with one tie, which is a highly

Table 6-85. MAP-3 L-shell on Metal: Model Estimates.

DEVICE	SAMPLE SIZE	MODEL PARAMETERS				Pb=0.0 mg/cm²		Pb=1.0 mg/cm²	
		a	b	c	d	BIAS	SD	BIAS	SD
Machine 10, Denver	62	0.309 (.077)	0.457 (.138)	0.250 (.051)	0.125 (.088)	0.309 (.077)	0.500	-0.234 (.126)	0.613
Machine 10, Philadelphia	124	-0.101 (.020)	0.256 (.041)	0.014 (.003)	0.047 (.010)	-0.101 (.020)	0.119	-0.845 (.031)	0.247
Machine 10, combined	186	0.054 (.038)	0.284 (.056)	0.145 (.019)	0.080 (.025)	0.054 (.038)	0.381	-0.662 (.047)	0.475
Machine 11	62	0.252 (.067)	0.458 (.123)	0.191 (.040)	0.100 (.073)	0.252 (.067)	0.437	-0.290 (.111)	0.540
Machine 12	123	-0.109 (.025)	0.240 (.043)	0.027 (.005)	0.042 (.011)	-0.109 (.025)	0.165	-0.868 (.032)	0.263
All Machines (10, 11, and 12)	371	0.044 (.037)	0.269 (.055)	0.133 (.018)	0.076 (.024)	0.044 (.037)	0.364	-0.687 (.045)	0.467

Table 6-86. MAP-3 L-shell on Metal: Control Block Summary.

DEVICE	SAMPLE SIZE	BARE (0.0 mg/cm²)		RED NIST SRM (1.02 mg/cm²)		YELLOW NIST SRM (3.53 mg/cm²)	
		BIAS	SD	BIAS	SD	BIAS	SD
Machine 10	74	-0.124 (.005)	0.042	0.040 (.007)	0.063	-0.384 (.017)	0.144
Machine 11	40	-0.155 (.026)	0.165	0.073 (.010)	0.063	-0.416 (.092)	0.581
Machine 12	34	-0.119 (.006)	0.035	-0.002 (.011)	0.064	-0.427 (.074)	0.431
Machines 10, 11, and 12	148	-0.131 (.008)	0.092	0.039 (.005)	0.063	-0.402 (.031)	0.379

significant result under the sign test. In particular, all 12 sites where the ICP measurement was greater than 1.0 mg/cm^2 had higher Machine 10 readings.

Figure 6-28 shows the response and SD components of the estimated model before provision for the combined effect of spatial variation and laboratory error in ICP measurements. There were no XRF readings above 1.0 mg/cm^2 in spite of the presence of very high ICP measurements. It is clear that the observations with ICP levels greater than 5.0 mg/cm^2 were not well described by this model. These data were obtained from the same century-old unit in Denver. Restriction of the data to ICP levels less than 5.0 mg/cm^2 is suggested if inference to lower lead levels is desired. Figure 6-29 shows the model components for the restricted data, and the fit appears to be reasonable. There were no field samples with ICP levels between 3.0 mg/cm^2 and 5.0 mg/cm^2 on plaster, which explains the labeling of the horizontal axes.

Table 6-87 gives the results of fitting XRF measurement models to the data. The differences between cities (within Machine 10) and between machines are seen primarily in the slope parameters *b*, which measure how responsive the instrument was to changes in the lead level. Comparing Denver and Philadelphia within Machine 10 gave a chi-square statistic with a p-value of less than 0.5 percent, which is highly significant. Again, slope parameters obtained with Denver data were higher than those for Philadelphia data, a pattern that was seen across substrates.

Table 6-88 gives a summary of the control block data. Differences between machines were not evident, and bias was not nearly of the magnitude seen in the model estimates obtained from the field sample data. A negative-positive-negative pattern in the bias as the lead level increases, seen in all substrates, may indicate a nonlinear response.

6.4.4.4.6 MAP-3 L-shell on Wood

There were 698 observations of the MAP-3 L-shell on wood, one of which was designated as an outlier (Machine 11: 80653), leaving 697 observations for analysis. For Class I, readings on Machine 10 were made 349 times, 297 in Denver and 52 in Philadelphia. For Class II, readings on Machine 11 (Denver) were made 296 times, and on Machine 12 (Philadelphia) 52 times.

Matched pairs analysis: Two matched pairs comparisons were made on the data:

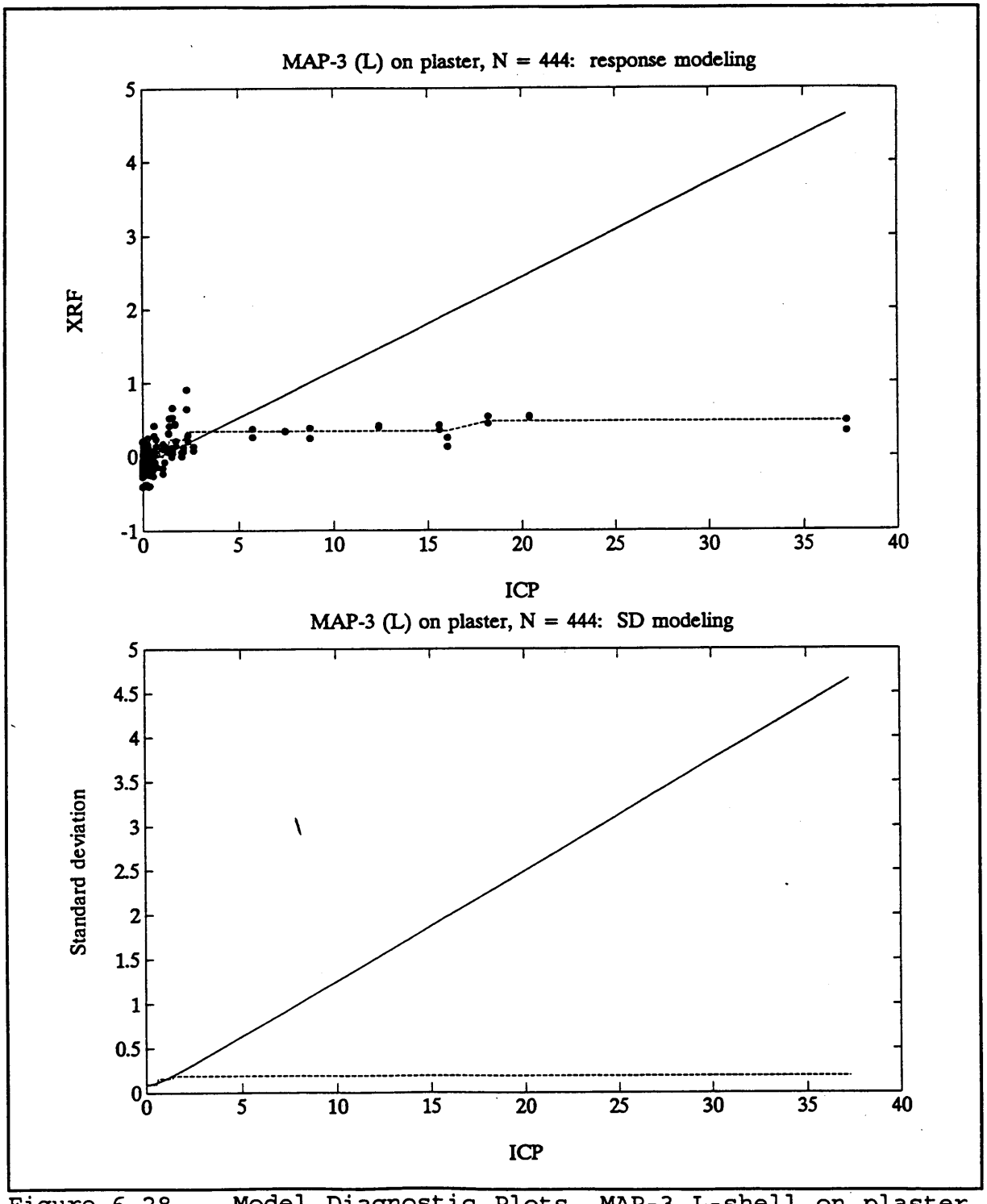


Figure 6-28. Model Diagnostic Plots, MAP-3 L-shell on plaster. Solid lines are model estimates. Dashed lines are nonparametric (monotone regression) estimates.

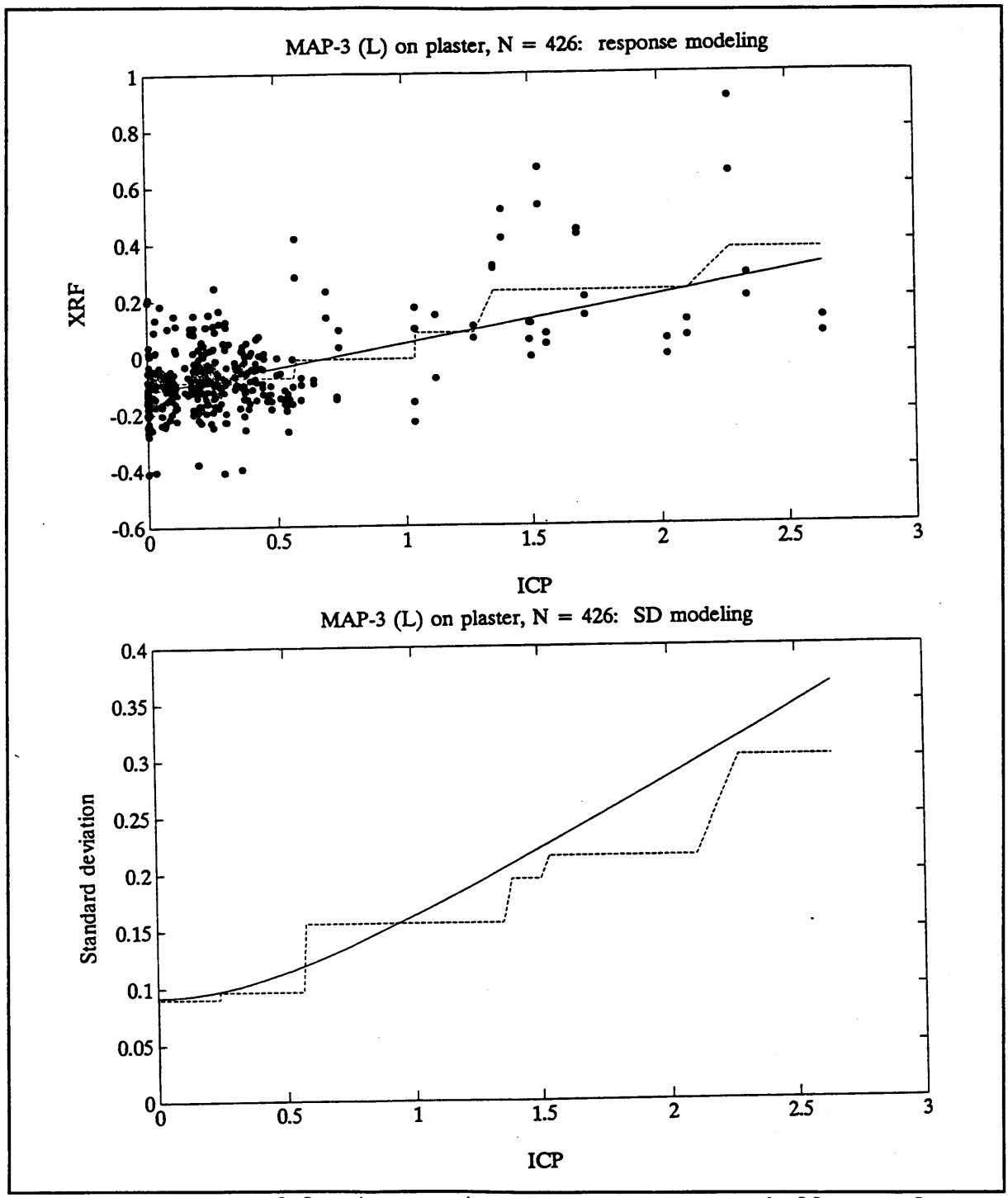


Figure 6-29. Model Diagnostic Plots, MAP-3 L-shell on plaster with ICP restricted to less than 3.0 mg/cm². Solid lines are model estimates. Dashed lines are nonparametric (monotone regression) estimates.

Table 6-87. MAP-3 L-shell on Plaster: Model Estimates.

DEVICE	SAMPLE SIZE	MODEL PARAMETERS				Pb=0.0 mg/cm²		Pb=1.0 mg/cm²	
		a	b	c	d	BIAS	SD	BIAS	SD
Machine 10, Denver (ICP < 5)	92	-0.123 (.013)	0.416 (.079)	0.007 (.002)	0.070 (.044)	-0.123 (.013)	0.084	-0.708 (.072)	0.276
Machine 10, Philadelphia (ICP < 5)	121	-0.108 (.011)	0.144 (.027)	0.005 (.001)	0.005 (.004)	-0.108 (.011)	0.073	-0.964 (.020)	0.100
Machine 10, combined (ICP < 5)	213	-0.112 (.009)	0.200 (.030)	0.007 (.001)	0.019 (.008)	-0.112 (.009)	0.081	-0.911 (.025)	0.161
Machine 11 (ICP < 5)	92	-0.112 (.014)	0.270 (.064)	0.010 (.002)	0.024 (.015)	-0.112 (.014)	0.100	-0.842 (.057)	0.185
Machine 12 (ICP < 5)	121	-0.180 (.011)	0.170 (.026)	0.000 (.001)	0.004 (.003)	-0.180 (.011)	0.077	-1.010 (.019)	0.099
All Machines (10, 11, and 12) (ICP < 5)	426	-0.123 (.010)	0.169 (.029)	0.008 (.001)	0.015 (.007)	-0.123 (.010)	0.091	-0.955 (.024)	0.152

Table 6-88. MAP-3 L-shell on Plaster: Control Block Summary.

DEVICE	SAMPLE SIZE	BARE (0.0 mg/cm²)		RED NIST SRM (1.02 mg/cm²)		YELLOW NIST SRM (3.53 mg/cm²)	
		BIAS	SD	BIAS	SD	BIAS	SD
Machine 10	68	-0.177 (.066)	0.046	0.190 (.009)	0.074	-0.121 (.021)	0.173
Machine 11	34	-0.198 (.017)	0.097	0.188 (.015)	0.087	-0.185 (.037)	0.218
Machine 12	34	-0.186 (.049)	0.284	0.146 (.012)	0.068	-0.125 (.034)	0.200
Machines 10, 11, and 12	136	-0.184 (.013)	0.153	0.178 (.007)	0.076	-0.138 (.016)	0.192

(1) Machines 10 and 11 were compared on the 295 Denver sites of common measurement. Machine 10 had the higher reading 157 times, with 2 ties, which is a plausible outcome under a 50-50 chance hypothesis.

(2) Machines 10 and 12 were compared on the 52 Philadelphia sites of common measurement. Machine 10 had the higher reading 26 times, with no ties, again a plausible outcome if no machine effect is present.

Figure 6-30 shows the response and SD components of the estimated model before provision for the combined effect of spatial variation and laboratory error in ICP measurements. The nonparametric response estimate is clearly flatter than the model estimate at higher lead levels. XRF readings below 1.0 mg/cm^2 were observed at ICP levels higher than 20.0 mg/cm^2. Restriction of the analysis to data with ICP measurements less than 5.0 mg/cm^2 should better describe the performance of the instrument at lower lead levels. Figure 6-31 shows the estimated model components on the restricted data, and the fit appears to be adequate for inferring instrument performance.

Table 6-89 gives the results of fitting XRF measurement models to the data. Comparing parameter estimates for different instruments within the same city did not show evidence of a machine effect, as the matched pairs analysis indicated. There did appear to be a city effect, with Denver having higher slope estimates (parameter *b*) than Philadelphia, a pattern that was seen across substrates. Comparing the two cities within Machine 10 gave a chi-square statistic with a p-value of less than 0.01 percent. Results for the combined city data give an indication of how the instrument performed on wood substrates under a broader range of conditions.

Table 6-90 gives a summary of the control block data, and the conclusions are the same as with the other substrates. The control block data gave a picture of instrument performance that was not duplicated on the field samples. The negative-positive-negative pattern in the bias as the lead level increases may be indicative of a nonlinear response.

6.4.4.4.7 MAP-3 L-shell: Summary of Analysis

Like other L-shell instruments, the MAP-3 L-shell did not

perform the same on painted samples as on the control blocks. Bias was of a much lower order of magnitude on the control blocks, and all substrates showed a negative-positive-negative

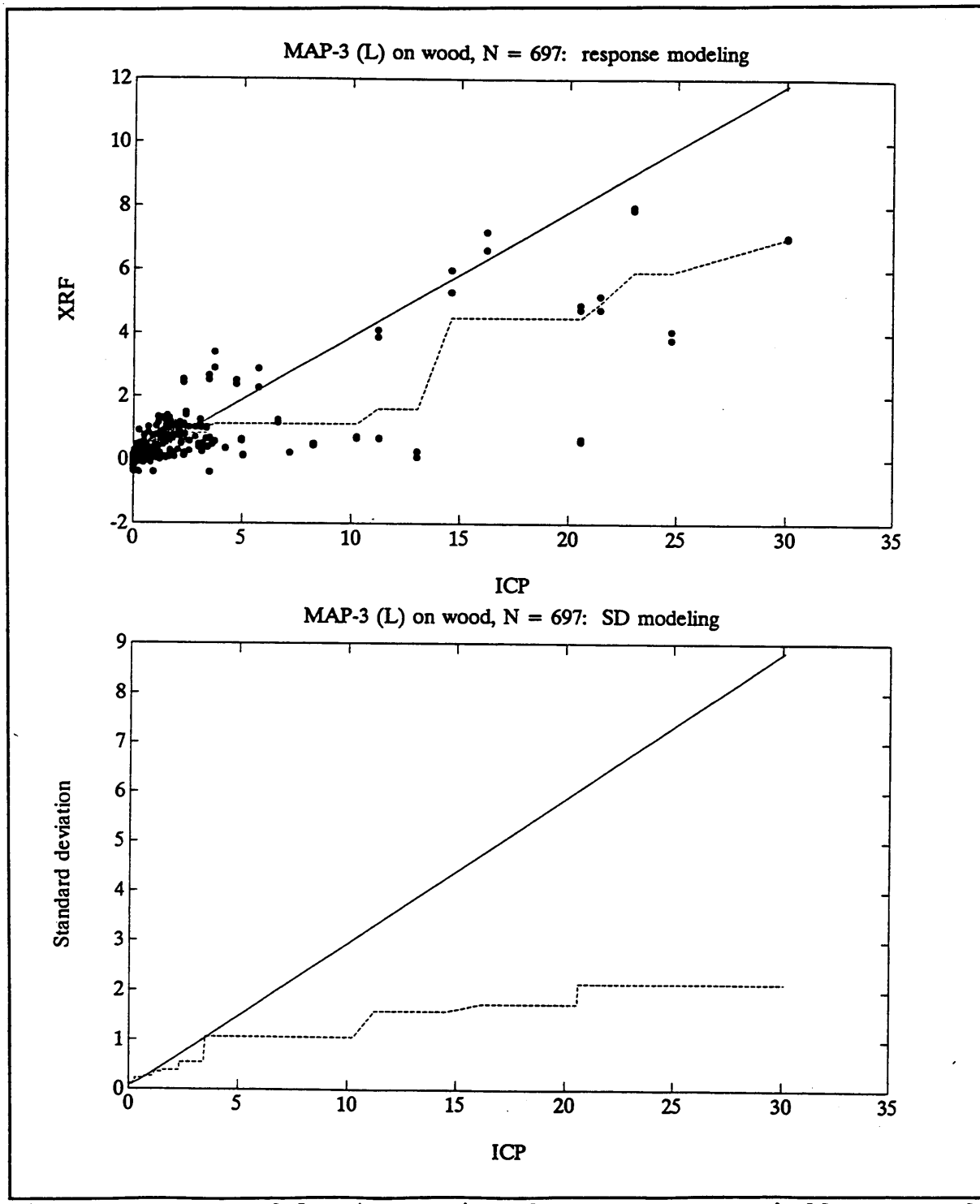


Figure 6-30. Model Diagnostic Plots, MAP-3 L-shell on wood. Solid lines are model estimates. Dashed lines are nonparametric (monotone regression) estimates.

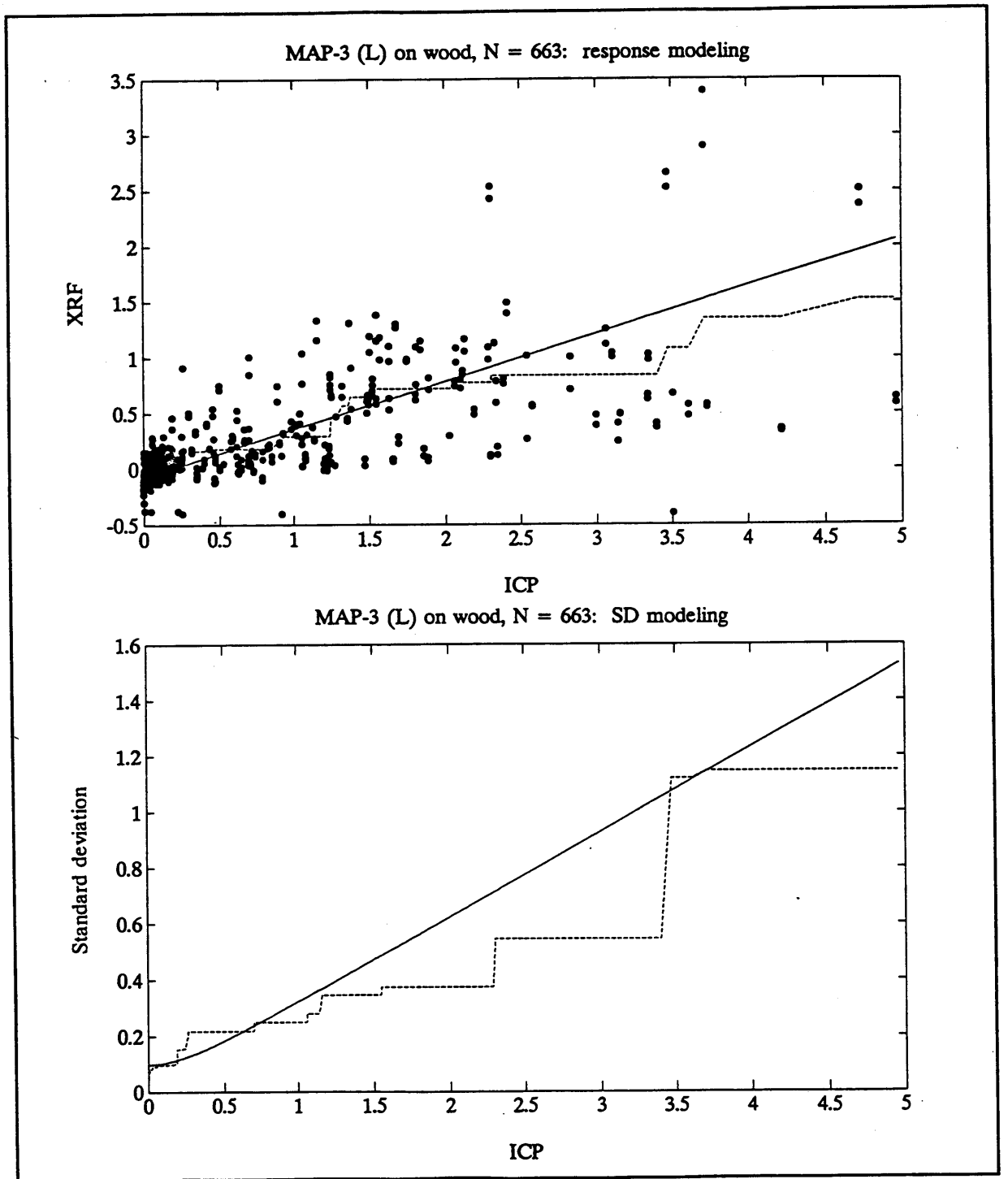


Figure 6-31. Model Diagnostic Plots, MAP-3 L-shell on wood with ICP restricted to less than 5.0 mg/cm². Solid lines are model estimates. Dashed lines are nonparametric (monotone regression) estimates.

Table 6-89. MAP-3 L-shell on Wood: Model Estimates.

DEVICE	SAMPLE SIZE	MODEL PARAMETERS				Pb=0.0 mg/cm²		Pb=1.0 mg/cm²	
		a	b	c	d	BIAS	SD	BIAS	SD
Machine 10, Denver (ICP < 5)	281	-0.087 (.008)	0.542 (.038)	0.009 (.001)	0.076 (.018)	-0.087 (.008)	0.092	-0.545 (.035)	0.292
Machine 10, Philadelphia (ICP < 5)	51	-0.067 (.032)	0.213 (.036)	0.019 (.005)	0.004 (.004)	-0.067 (.032)	0.138	-0.854 (.025)	0.152
Machine 10, combined (ICP < 5)	332	-0.084 (.008)	0.454 (.031)	0.009 (.001)	0.071 (.015)	-0.084 (.008)	0.095	-0.630 (.028)	0.282
Machine 11 (ICP < 5)	280	-0.074 (.007)	0.467 (.032)	0.007 (.001)	0.056 (.014)	-0.074 (.007)	0.086	-0.607 (.030)	0.253
Machine 12 (ICP < 5)	51	-0.051 (.036)	0.165 (.047)	0.022 (.006)	0.015 (.007)	-0.051 (.036)	0.147	-0.886 (.032)	0.192
All Machines (10, 11, and 12) (ICP < 5)	663	-0.079 (.008)	0.425 (.030)	0.008 (.001)	0.068 (.014)	-0.078 (.008)	0.092	-0.653 (.027)	0.276

Table 6-90. MAP-3 L-shell on Wood: Control Block Summary.

DEVICE	SAMPLE SIZE	BARE (0.0 mg/cm²)		RED NIST SRM (1.02 mg/cm²)		YELLOW NIST SRM (3.53 mg/cm²)	
		BIAS	SD	BIAS	SD	BIAS	SD
Machine 10	72	-0.180 (.002)	0.020	0.103 (.009)	0.073	-0.239 (.021)	0.180
Machine 11	38	-0.161 (.004)	0.024	0.120 (.008)	0.052	-0.298 (.039)	0.242
Machine 12	34	-0.171 (.003)	0.018	0.042 (.057)	0.335	-0.279 (.115)	0.671
Machines 10, 11, and 12	144	-0.216 (.008)	0.020	0.181 (.006)	0.172	-0.085 (.015)	0.370

pattern in the bias with increasing lead levels, which may indicate a nonlinear response to the lead level. The usage of the instrument on the field samples exhibited much less responsiveness to changes in the lead level.

The instrument frequently made readings below 1.0 mg/cm^2 when the ICP measurement was higher than 10.0 mg/cm^2. This occurred on all substrates except drywall and metal, for which no field samples with ICP measurements greater than 10.0 mg/cm^2 were present in the full study. On plaster, all MAP-3 L-shell readings were less than 1.0 mg/cm^2 on the 9 samples with ICP measurements greater than 5.0 mg/cm^2.

Both machine (or operator) and city effects were evident in the field sample data. City effects were indicated, since the slope parameter *b* estimated for Denver was about twice as large as it was for Philadelphia. This suggests that the instrument was less responsive to changes in the lead level in Philadelphia compared to Denver, which may be indicative of factors such as paint mass or paint thickness in the field samples. Machine effects were reflected in the intercept parameter *a*, except for plaster, where the slope parameter explains the difference between Machines 10 and 11 restricted to the same Denver sites. Differences in both intercepts and slopes were indicated between cities on metal substrates, which may reflect differences in building materials, age and thickness of paint, or other factors that distinguished cities in the study.

6.4.4.5 Results for Microlead I

The Microlead I revision 4 (also referred to as the ML I) is a K-shell instrument. Data were obtained on 5 machines by 4 different operators, from both the full and pilot studies. These are described as follows:

Machine 20 (Operator G)	Denver and Philadelphia, Class I
Machine 21 (Operators E, H)	Denver and Philadelphia Class II
Machine 22 (Operator E)	Denver only, Class II
Machine 23 (Operator H)	Philadelphia only, Class II
Machine 24 (Operator F)	Louisville only,

unclassed

The partial crossing of operators, cities, and instruments allowed limited assessment of these factors controlling for the others, depending on the availability of data. Field classifications (Class I and Class II) applied to only the full study, where paired comparisons were made, and the problem of statistical dependence arose. Cities are sometimes referred to by their first letters for the sake of brevity.

6.4.4.5.1 Microlead I on Brick

There were 186 observations of the Microlead I on brick, none of which were designated as outliers. The breakdown by machines, operators, and cities was as follows:

Machine 20:	Operator G	93 total (81 D, 93 P)
Machine 21:	Operator E	58 total (all D)
	Operator H	2 total (all P)
Machine 22:	Operator E	23 total (all D)
Machine 23:	Operator H	10 total (all P)

No observations on brick substrates were obtained in the Louisville pilot study.

Matched pairs analysis: A number of sign tests were performed on matched pairs:

(1) Machine 20 (Operator G) was compared to Machine 21 (Operator E) on 58 Denver sites of common measurement. Machine 20 gave a lower reading 37 times, with 3 ties. The sign test has a p-value of about 1.5 percent, indicating a possible machine or operator effect. Machine 20 gave a lower reading on all 21 samples with ICP measurements greater than 1.0 mg/cm^2, which indicates that the effect was manifest at higher lead levels.

(2) Machine 20 (Operator G) was compared to Machine 22 (Operator E) on 23 Denver sites of common measurement. Machine 20 gave a higher reading 7 times, with 2 ties. This is a plausible result with the sign test (p-value in excess of 10 percent). Since the highest ICP measurement was only 0.0079 mg/cm^2, it cannot be determined if this conclusion held at higher lead levels.

(3) Machine 20 (Operator G) was compared to Machine 23 (Operator H) on 10 Philadelphia sites of common measurement. Machine 20 gave a higher reading 6 times, with 1 tie. This again is a plausible result with the sign test. But since the highest ICP measurement was 0.7094 mg/cm^2, it is not known if an effect at higher lead levels may have existed.

Operator E using Machines 21 and 22 permits comparison of the two machines with a Fisher's exact test using the results of (2) and (3). The test does not find that the pattern of positive and negative differences with respect to Machine 21 is significantly different from that of Machine 22. A machine effect controlling for operator and city was therefore not found.

Figure 6-32 shows the response and SD components of the estimated model before provision for the combined effect of spatial variation and laboratory error in ICP measurements. The model does not appear to fit the data well over the entire ICP range, and restriction to ICP measurements less than 1.0 mg/cm^2 may be more revealing. This is shown in Figure 6-33. The model response is very flat, and the nonparametric estimate even more so. For ICP measurements less than 1.0 mg/cm^2, there was little response to change in the lead level, but at higher lead levels (greater than 5.0 mg/cm^2) the response was obviously higher. What happened in between is hard to tell because of a lack of data.

Fitting the XRF measurement model to the data was inappropriate, given the lack of responsiveness at lower lead levels and the poor model fit at higher lead levels. Performance at 0.0 mg/cm^2 was inferred by taking averages and standard deviations for ICP measurements less than 0.02 mg/cm^2, and at 1.0 mg/cm^2 for ICP between 0.02 mg/cm^2 and 1.0 mg/cm^2. Results are reported in Table 6-91. Table 6-92 is a summary of the control block data.

There is some agreement between the control block results and the models in the SD estimates, but little else. Table 6-92 suggests large positive bias for Machines 21, 22 and 23 that diminished as the lead level increases, and a negative bias for Machine 20 that became worse as the lead level increased. A similar pattern was not seen on the field samples.

6.4.4.5.2 Microlead I on Concrete

There were 444 observations of the Microlead I on concrete, none of which were designated as outliers. The breakdown by machines, operators, and cities is as follows:

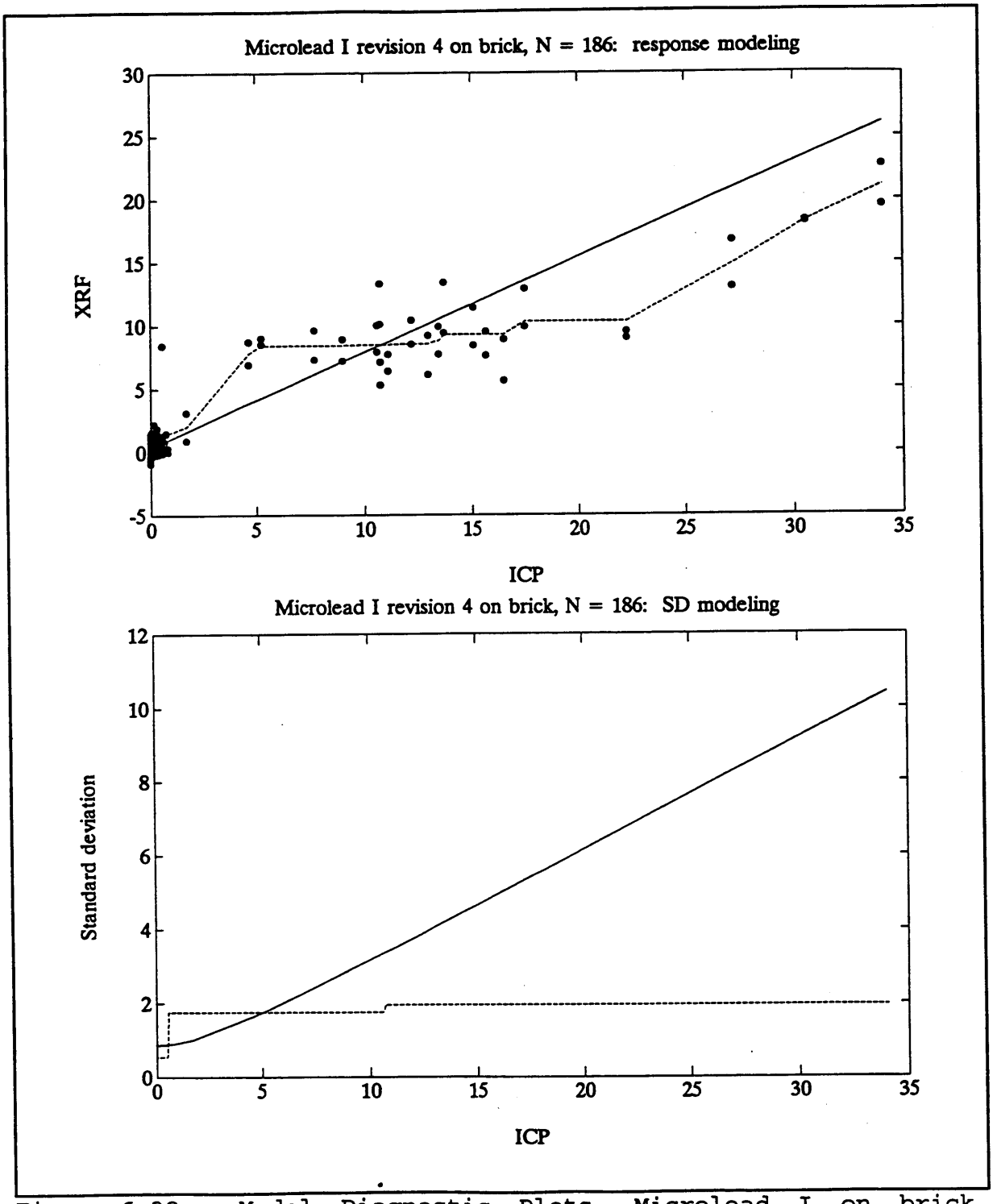


Figure 6-32. Model Diagnostic Plots, Microlead I on brick. Solid lines are model estimates. Dashed lines are nonparametric (monotone regression) estimates.

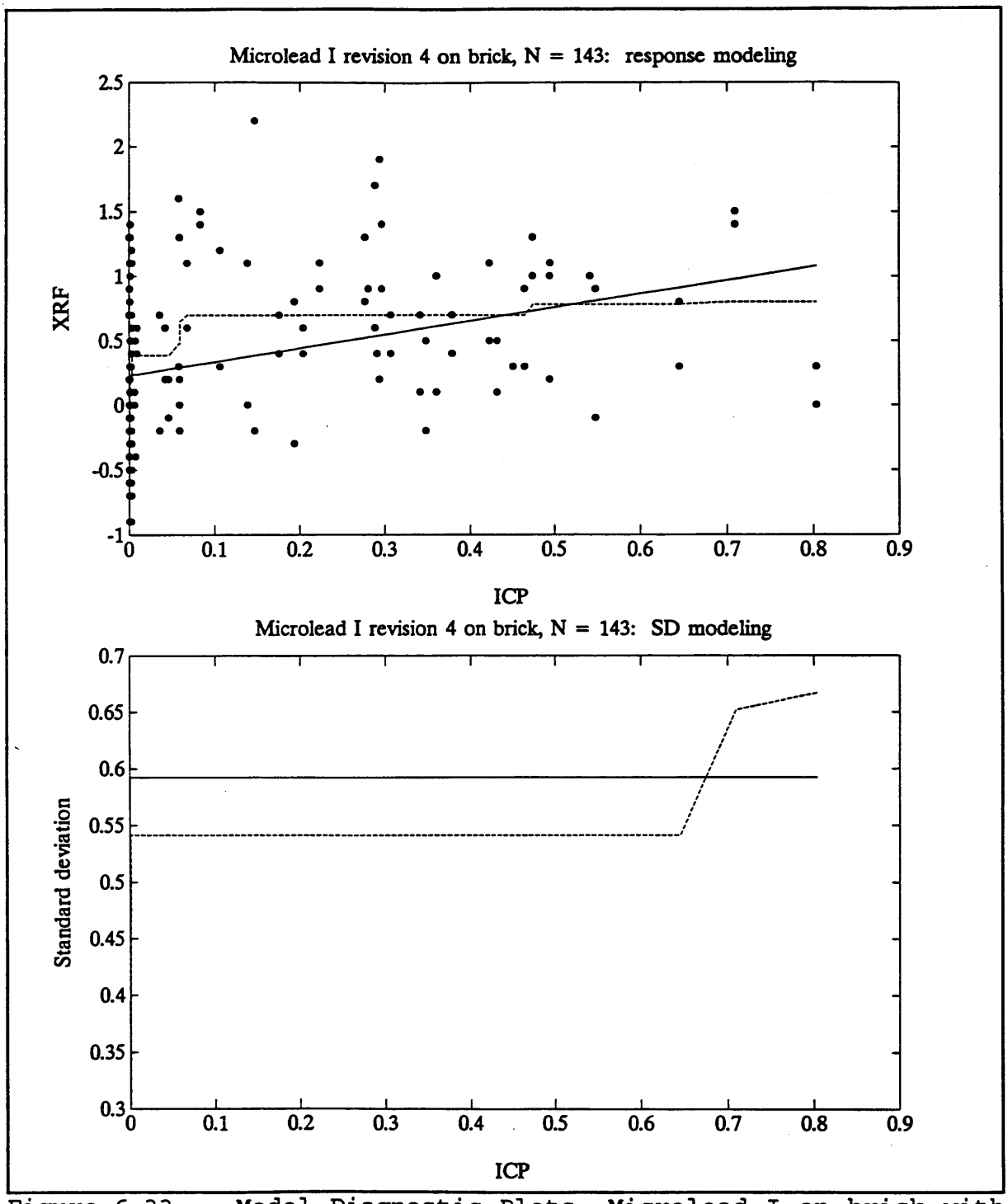


Figure 6-33. Model Diagnostic Plots, Microlead I on brick with ICP restricted to less than 0.9 mg/cm². Solid lines are model estimates. Dashed lines are nonparametric (monotone regression) estimates.

Table 6-91. Microlead I on Brick: Model Estimates.

DEVICE	SAMPLE SIZE	MODEL PARAMETERS				Pb=0.0 mg/cm²		Pb=1.0 mg/cm²	
		a	b	c	d	BIAS	SD	BIAS	SD
Machine 20 (ICP < 1)	71	--	--	--	--	0.077 (.102)	0.593	-0.366 (.084)	0.509
Machine 21 (ICP < 1)	38	--	--	--	--	0.133 (.294)	0.720	-0.300 (.104)	0.586
Machine 22 (ICP < 1)	23	--	--	--	--	0.030 (.110)	0.530	-	-
All Machines (20, 21, 22, and 23) (ICP < 1)	143	--	--	--	--	0.103 (.101)	0.583	-0.333 (.154)	0.552

Table 6-92. Microlead I on Brick: Control Block Summary.

DEVICE	SAMPLE SIZE	BARE (0.0 mg/cm²)		RED NIST SRM (1.02 mg/cm²)		YELLOW NIST SRM (3.53 mg/cm²)	
		BIAS	SD	BIAS	SD	BIAS	SD
Machine 20	60	-0.103 (.084)	0.653	-0.307 (.079)	0.613	-0.707 (.096)	0.740
Machine 21	21	0.509 (.098)	0.449	0.537 (.086)	0.396	0.208 (.129)	0.591
Machine 22	14	0.407 (.204)	0.763	0.380 (.156)	0.584	0.134 (.176)	0.659
Machine 23	26	0.469 (.055)	0.280	0.415 (.051)	0.258	-0.080 (.072)	0.365
Machines 21, 22, and 23	61	0.469 (.062)	0.484	0.449 (.051)	0.399	0.068 (.067)	0.525

Machine 20:	Operator G	218	total (98 D, 120 P)
Machine 21:	Operator E	67	total (all D)
	Operator H	15	total (all P)
Machine 22:	Operator E	31	total (all D)
Machine 23:	Operator H	105	total (all P)
Machine 24:	Operator F	8	total (all L)

Matched pairs analysis: A number of sign tests were performed on matched pairs:

(1) Machine 20 (Operator G) was compared to Machine 21 (Operator E) on 67 Denver sites of common measurement. Machine 20 gave a higher reading 8 times, with 3 ties. The sign test has a p-value of less than 0.01 percent, strongly indicating a possible machine or operator effect where Machine 20 read lower than Machine 21.

(2) Machine 20 (Operator G) was compared to Machine 21 (Operator H) on 15 Philadelphia sites of common measurement. Machine 20 gave a higher reading 11 times, with no ties. This is a plausible result with the sign test (p-value in excess of 10 percent).

(3) Machine 20 (Operator G) was compared to Machine 22 (Operator E) on 31 Denver sites of common measurement. Machine 20 gave a higher reading only 5 times, with 3 ties. This result is highly significant with the sign test, and suggests that Machine 20 gave lower readings than Machine 22.

(4) Machine 20 (Operator G) was compared to Machine 23 (Operator H) on 105 Philadelphia sites of common measurement. Machine 20 gave a higher reading 73 times, with 7 ties, which is a highly significant result with the sign test (p-value of about 0.01 percent), suggesting that Machine 20 tended to give higher readings than Machine 23. Again, it must be emphasized that this result cannot separate the effect of the operator from that of the machine.

The two comparisons of Machine 20 with Machine 21 under different operators for Machine 21 suggests either an operator or a city effect. The Fisher's exact test on the 2 by 2 table based on the information in (1) and (2) was used to explore this further. The test has a p-value of less than 0.01 percent. The

possibility that what are indicated as machine effects are in fact due to operators or cities therefore cannot be ruled out. Given the strong possibility of effects of either kind, the small quantity of Louisville data was not used in the analysis.

Figure 6-34 shows the response and SD components of the estimated model before provision for the combined effect of spatial variation and laboratory error in ICP measurements. The fit appears to be reasonable for the purposes of making inference at lower lead levels.

Table 6-93 gives the results of fitting XRF measurement models. The slopes (parameter *b*) of the estimated models are all similar, but differences are evident in the intercepts (parameter *a*), which appears to account for machine or operator effects found in the matched pairs analysis. Comparing Denver and Philadelphia within Machine 20 indicated a city effect with a highly significant chi-square p-value (less than .01 percent). The difference in the intercepts, -0.030 (.065) in Denver and 0.589 (.064) in Philadelphia, appeared to be the reason. The ordering of machines suggested by the sign tests, Machine 20 less than Machine 21 in Denver, and less than Machine 22 but greater than Machine 23 in Philadelphia, was also reflected in the intercepts.

Table 6-94 gives a summary of the control block data. Machine 22 stood out as giving the highest readings, followed by 20, 23, and 21. Estimates of the SD increased with the lead level, as did the model estimates. Non-instrumental sources of variability may be reflected in the higher SD estimates obtained with the model.

6.4.4.5.3 Microlead I on Drywall

There were 237 observations of the Microlead I on concrete, 4 of which were designated as outliers (Machines 20 and 21: 80343 and 80345), leaving 233 observations for analysis. The breakdown by machines, operators, and cities was as follows:

Machine 20:	Operator G	111 total (103 D, 8 P)
Machine 21:	Operator E	32 total (all D)
Machine 22:	Operator E	71 total (all D)
Machine 23:	Operator H	8 total (all P)
Machine 24:	Operator F	11 total (all L)

Matched pairs analysis: A number of sign tests were performed on matched pairs:

(1) Machine 20 (Operator G) was compared to Machine 21 (Operator E) on 32 Denver sites of common measurement. Machine 20

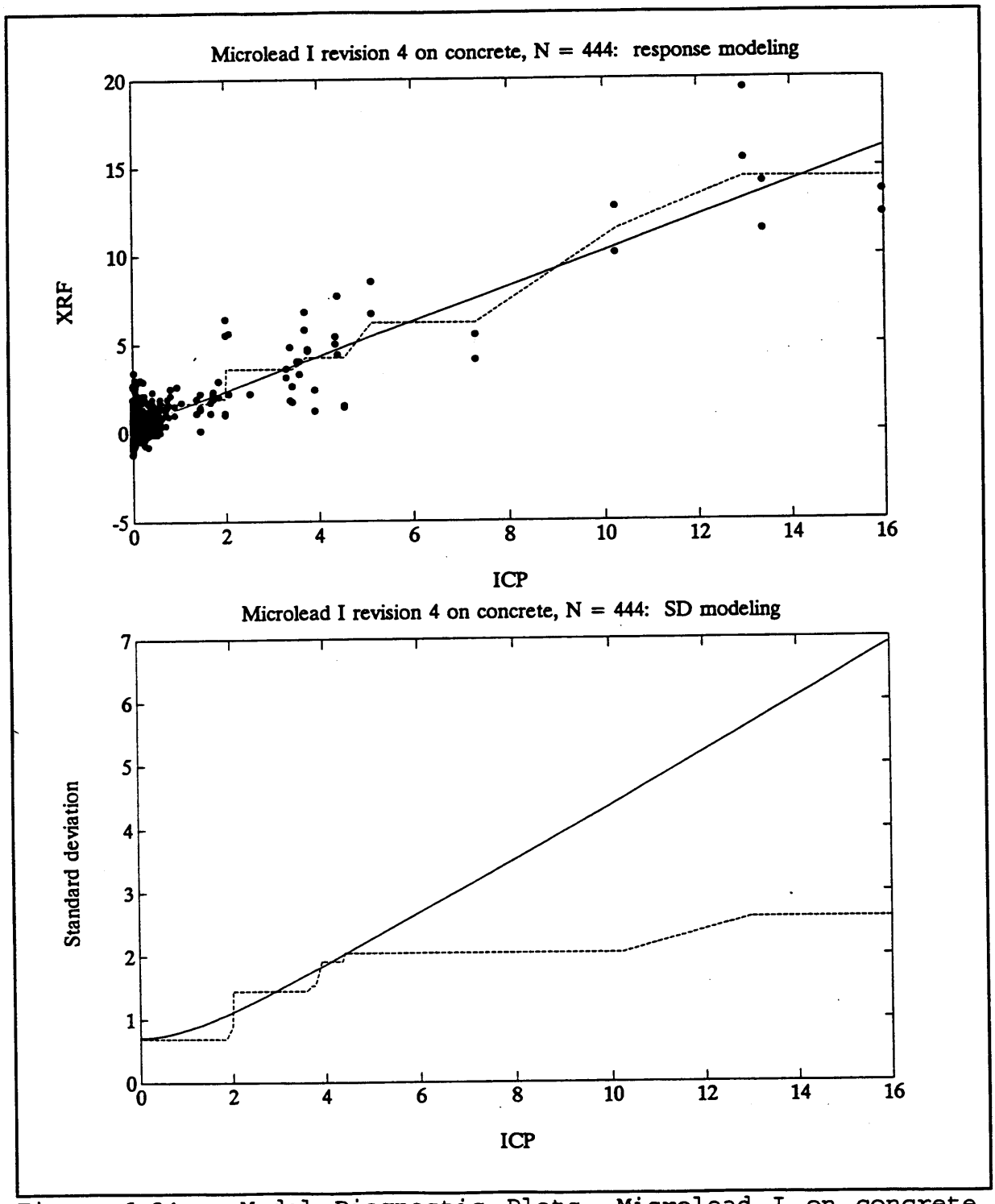


Figure 6-34. Model Diagnostic Plots, Microlead I on concrete. Solid lines are model estimates. Dashed lines are nonparametric (monotone regression) estimates.

Table 6-93. Microlead I on Concrete: Model Estimates.

DEVICE	SAMPLE SIZE	MODEL PARAMETERS				Pb=0.0 mg/cm²		Pb=1.0 mg/cm²	
		a	b	c	d	BIAS	SD	BIAS	SD
Machine 20, Denver	98	-0.030 (.060)	1.022 (.126)	0.313 (.051)	0.188 (.104)	-0.030 (.066)	0.559	0.008 (.100)	0.708
Machine 20, Philadelphia	120	0.589 (.064)	0.868 (.110)	0.295 (.039)	-0-	0.589 (.064)	0.543	0.457 (.100)	0.543
Machine 20, combined	218	0.283 (.051)	1.094 (.106)	0.375 (.041)	0.143 (.071)	0.283 (.051)	0.613	0.377 (.092)	0.720
Machine 21	82	0.670 (.093)	0.925 (.109)	0.524 (.094)	0.111 (.068)	0.670 (.093)	0.724	0.595 (.121)	0.790
Machine 22	31	0.892 (.251)	1.337 (.455)	1.547 (.423)	0.166 (.455)	0.892 (.251)	1.244	1.230 (.415)	1.309
Machine 23	105	0.110 (.070)	1.043 (.158)	0.233 (.033)	-0-	0.110 (.070)	0.483	0.152 (.116)	0.483

Table 6-94. Microlead I on Concrete: Control Block Summary.

DEVICE	SAMPLE SIZE	BARE (0.0 mg/cm²)		RED NIST SRM (1.02 mg/cm²)		YELLOW NIST SRM (3.53 mg/cm²)	
		BIAS	SD	BIAS	SD	BIAS	SD
Machine 20	72	0.636 (.060)	0.513	0.822 (.074)	0.629	0.314 (.072)	0.612
Machine 21	24	0.145 (.070)	0.344	0.247 (.092)	0.451	0.004 (.222)	1.090
Machine 22	18	1.428 (.117)	0.494	1.586 (.160)	0.677	1.237 (.174)	0.736
Machine 23	30	0.397 (.057)	0.314	0.537 (.074)	0.407	-0.020 (.072)	0.396
Machines 21, 22, and 23	72	0.571 (.044)	0.376	0.702 (.059)	0.500	0.302 (.091)	0.772

gave a higher reading 11 times, with 1 tie. This result is plausible under a hypothesis of no machine or operator effects.

(2) Machine 20 (Operator G) was compared to Machine 22 (Operator E) on 71 Denver sites of common measurement. Machine 20 gave a higher reading only 5 times, with no ties. The resulting sign test is very highly significant (p-value less than 0.01 percent), suggesting that Machine 20 systematically read lower than Machine 22.

(3) Machine 20 (Operator G) was compared to Machine 23 (Operator H) on 8 Philadelphia sites of common measurement. Machine 20 gave a higher reading 3 times, with 2 ties. This is not an unusual outcome if no machine or operator effects were to exist. The highest ICP measurement for these 8 sites was only 0.0079 mg/cm^2.

One Fisher's exact test is of interest. It is possible to compare Machines 21 and 22 within operator (E) and city (Denver) using the information in (1) and (2) above. The resulting p-value for the 2 by 2 table is about 0.1 percent, which suggests that Machine 22 read higher than Machine 21, or that site-specific effects within Denver were present.

Figure 6-35 shows the response and SD components of the estimated model before provision for the combined effect of spatial variation and laboratory error in ICP measurements. The model response appears to agree with the nonparametric estimate, and there is no indication that variability increases with the lead level, although the absence of ICP readings greater than 1.0 mg/cm^2 on drywall must be noted.

Table 6-95 gives the results of fitting XRF measurement models. Although the slope parameters appear to differ greatly, account needs to be taken of the standard errors. For Machine 22, the slope of 2.128 has a standard error of 0.548, and a 95 percent confidence interval created by subtracting and adding three (or even two) times the standard error places it in the range of the other estimates. The difference between Machines 20 and 22 that gave the highly significant sign test was reflected in the intercepts: 0.004 for Machine 20 and 0.658 for Machine 22.

Table 6-96 gives a summary of the control block data. A similar effect is seen for Machines 20 and 22: Machine 22 had the higher bias at 0.0 mg/cm^2, and the difference between them is

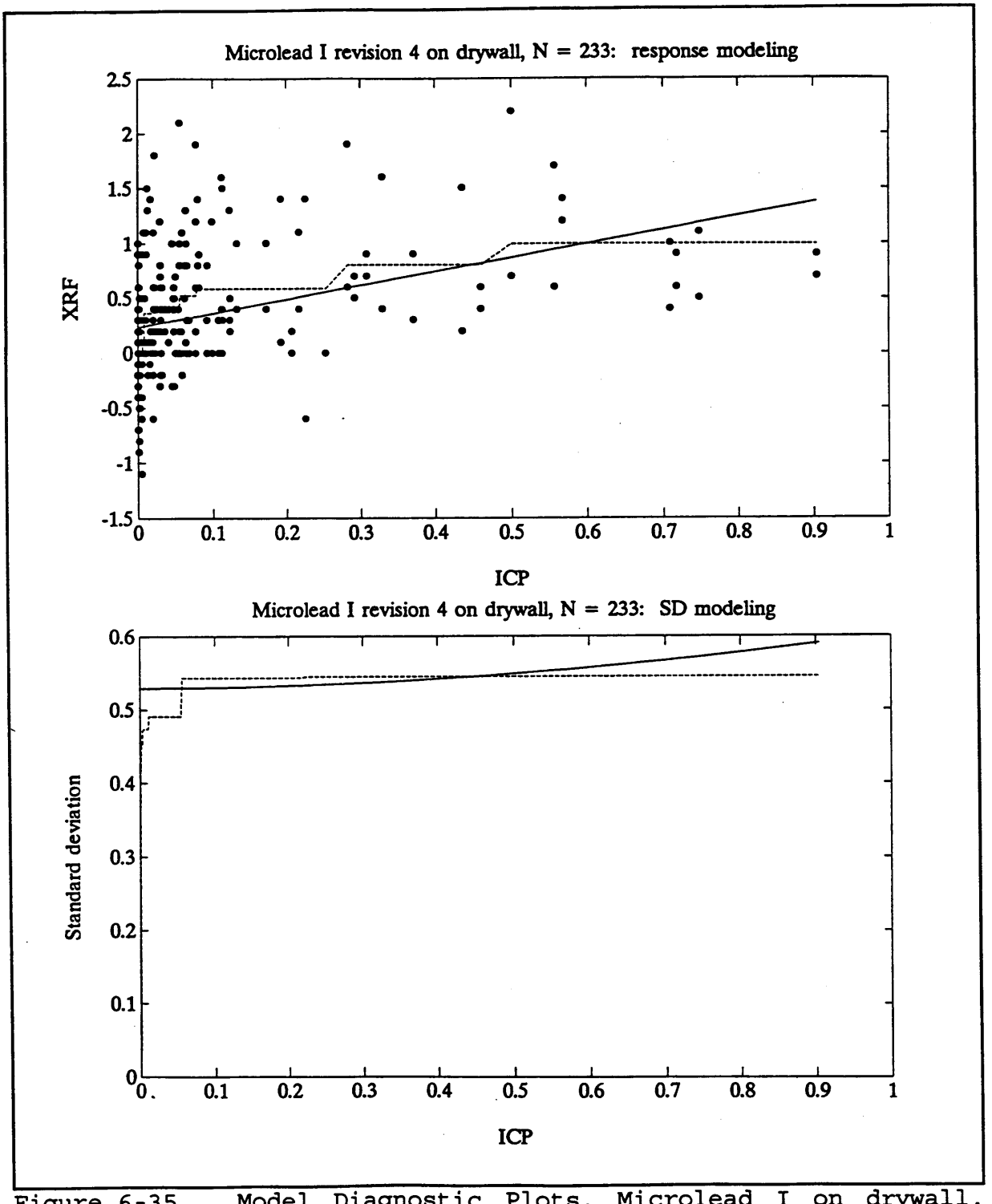


Figure 6-35. Model Diagnostic Plots, Microlead I on drywall. Solid lines are model estimates. Dashed lines are nonparametric (monotone regression) estimates.

Table 6-95. Microlead I on Drywall: Model Estimates.

DEVICE	SAMPLE SIZE	MODEL PARAMETERS				Pb=0.0 mg/cm²		Pb=1.0 mg/cm²	
		a	b	c	d	BIAS	SD	BIAS	SD
Machine 20	111	0.004 (.039)	1.179 (.225)	0.119 (.017)	-0-	0.004 (.039)	0.345	0.183 (.208)	0.345
Machine 21	32	0.202 (.074)	0.959 (.223)	0.123 (.031)	-0-	0.202 (.074)	0.351	0.162 (.193)	0.351
Machine 22	71	0.658 (.077)	2.128 (.548)	0.285 (.049)	-0-	0.658 (.077)	0.534	1.787 (.509)	0.534
Machines 20, 21, 23, and 24	162	0.023 (.031)	1.194 (.175)	0.115 (.013)	-0-	0.023 (.031)	0.338	0.217 (.162)	0.338

Table 6-96. Microlead I on Drywall: Control Block Summary.

DEVICE	SAMPLE SIZE	BARE (0.0 mg/cm²)		RED NIST SRM (1.02 mg/cm²)		YELLOW NIST SRM (3.53 mg/cm²)	
		BIAS	SD	BIAS	SD	BIAS	SD
Machine 20	60	-0.615 (.054)	0.418	-0.558 (.056)	0.435	-0.718 (.046)	0.356
Machine 21	16	0.138 (.055)	0.219	0.099 (.086)	0.345	0.107 (.099)	0.396
Machine 22	20	-0.055 (.087)	0.387	0.180 (.056)	0.251	0.105 (.097)	0.432
Machine 23	24	0.017 (.044)	0.214	0.076 (.059)	0.287	0.245 (.052)	0.254
Machines 21, 22, and 23	60	0.025 (.037)	0.285	0.117 (.038)	0.292	0.162 (.046)	0.360

about 0.6. But an even larger effect for Machines 20 and 21 would have been anticipated, and the biases generally did not agree with the model estimates. This follows not only from the model, but is borne out graphically as well, suggesting that the instruments performed differently on the field samples than on the control blocks. SD estimates from both sets of data were fairly close.

6.4.4.5.4 Microlead I on Metal

There were 406 observations of the Microlead I on metal, 4 of which were designated as outliers (Machine 20: 81348, 81710, 81953; Machine 23: 81348), leaving 402 observations for analysis. The breakdown by machines, operators, and cities was as follows:

Machine 20:	Operator G	186 total (62 D, 124 P)
Machine 21:	Operator E	35 total (all D)
	Operator H	16 total (all P)
Machine 22:	Operator E	27 total (all D)
Machine 23:	Operator H	110 total (all P)
Machine 24:	Operator F	28 total (all L)

<u>Matched pairs analysis</u>: A number of sign tests were performed on matched pairs:

(1) Machine 20 (Operator G) was compared to Machine 21 (Operator E) on 35 Denver sites of common measurement. Machine 20 gave a higher reading 32 times, with no ties. The sign test has a p-value of less than 0.01 percent, suggesting that Machine 20 read higher than Machine 21 in Denver with these operators.

(2) Machine 20 (Operator G) was compared to Machine 21 (Operator H) on 15 Philadelphia sites of common measurement. Machine 20 gave a higher reading 14 times, with no ties. The sign test has a p-value of about 0.05 percent, again suggesting a significant effect.

(3) Machine 20 (Operator G) was compared to Machine 22 (Operator E) on 27 Denver sites of common measurement. Machine 20 gave a higher reading 6 times, with 3 ties. The sign test has a p-value of about 2.5 percent, which suggests that Machine 20 may have been prone to reading lower than Machine 22 under these measurement circumstances.

(4) Machine 20 (Operator G) was compared to Machine 23 (Operator H) on 109 sites of common measurement. Machine 20 gave a higher reading 84 times, with 6 ties. The sign test has a p-value of less than 0.01 percent, strongly suggesting that an effect existed.

Several Fisher's exact tests are of note. Comparing Machines 21 and 22 for Operator E in Denver using the information in (1) and (3) gives a 2 by 2 table with a p-value of less than 0.01 percent. It appears that Machine 21 read lower than Machine 22. Comparing Operator E and Operator H within Machine 21 using the information in (1) and (2) gives a Fisher's exact test with a p-value of nearly 100 percent. While these tests do not constitute evidence that the operators affected the performance of this machine, the sample sizes were also small.

Figure 6-36 shows the response and SD components of the estimated model before provision for the combined effect of spatial variation and laboratory error in ICP measurements. The response seems to be well modeled for ICP levels less than about 3.0 mg/cm^2, above which the nonparametric estimate suggests a flattening of the response. The SD also seems to be well modeled up to about 2.0 mg/cm^2, where it closely agrees with the nonparametric estimate.

Table 6-97 gives the results of fitting XRF measurement models. The ordering of the machines suggested by the sign tests is evident in intercept terms: Machine 22 highest (1.080), followed by Machine 20 (0.351), Machine 21 (-0.381) and Machine 23 (0.415). The slopes are all similar, and not far from 1.0, which was typical for the K-shell instruments evaluated. Splitting Machine 20 readings by city revealed a significant difference, reflected primarily in the intercepts: 0.607 (.085) in Denver, and 0.207 (.083) in Philadelphia. This particular machine was suspected of having experienced a problem on metal control blocks in Philadelphia, and the apparent city effect may simply be a reflection of the same problem.

Table 6-98 gives a summary of the control block data. Machine 20, which as noted above experienced performance problems on the control blocks, had results that are clearly out of line with the other machines. The bias estimates again suggest that Machine 22 read higher than Machines 21 or 23. Only Machine 23, however, showed agreement with the model estimates of bias. The

model SD estimates were higher than for the control blocks, which may suggest non-instrumental sources of variability.

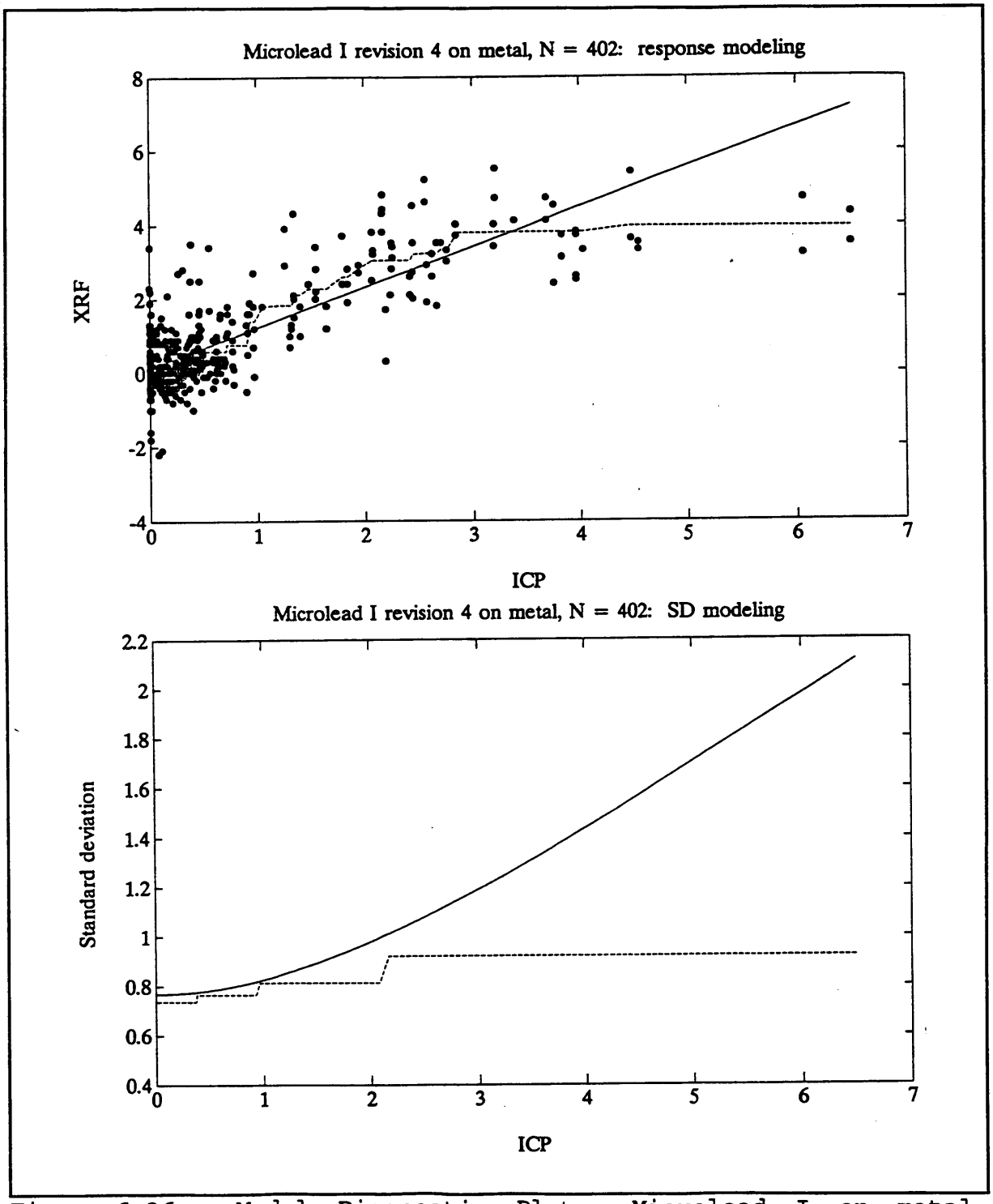


Figure 6-36. Model Diagnostic Plots, Microlead I on metal. Solid lines are model estimates. Dashed lines are nonparametric (monotone regression) estimates.

Table 6-97. Microlead I on Metal: Model Estimates.

DEVICE	SAMPLE SIZE	MODEL PARAMETERS				Pb=0.0 mg/cm²		Pb=1.0 mg/cm²	
		a	b	c	d	BIAS	SD	BIAS	SD
Machine 20, Denver	62	0.607 (.085)	1.158 (.131)	0.305 (.059)	-0-	0.607 (.085)	0.552	0.765 (.120)	0.552
Machine 20, Philadelphia	124	0.207 (.083)	1.074 (.067)	0.473 (.071)	-0-	0.207 (.083)	0.688	0.281 (.070)	0.688
Machine 20, combined	186	0.351 (.060)	1.100 (.075)	0.380 (.053)	0.088 (.050)	0.351 (.060)	0.617	0.451 (.065)	0.684
Machine 21	51	-0.381 (.130)	1.207 (.199)	0.536 (.117)	0.089 (.091)	-0.381 (.130)	0.732	-0.174 (.169)	0.790
Machine 22	71	1.080 (.175)	1.281 (.271)	0.653 (.184)	-0-	1.080 (.175)	0.808	1.361 (.255)	0.808
Machine 23	110	-0.415 (.057)	1.241 (.093)	0.134 (.030)	0.163 (.067)	-0.415 (.057)	0.366	-0.174 (.072)	0.545
Machine 24	28	0.217 (.182)	0.950 (.202)	0.523 (.145)	-0-	0.217 (.182)	0.723	0.167 (.164)	0.723

Table 6-98. Microlead I on Metal: Control Block Summary.

DEVICE	SAMPLE SIZE	BARE (0.0 mg/cm²)		RED NIST SRM (1.02 mg/cm²)		YELLOW NIST SRM (3.53 mg/cm²)	
		BIAS	SD	BIAS	SD	BIAS	SD
Machine 20	74	2.247 (.278)	2.390	1.995 (.249)	2.142	1.773 (.266)	2.291
Machine 21	24	-0.817 (.052)	0.257	-0.841 (.043)	0.213	-0.455 (.056)	0.275
Machine 22	21	-0.348 (.072)	0.330	0.266 (.128)	0.588	0.441 (.251)	1.152
Machine 23	30	-0.430 (.040)	0.222	-0.397 (.042)	0.232	0.017 (.051)	0.280
Machines 21, 22, and 23	75	-0.336 (.031)	0.266	-0.353 (.042)	0.363	-0.015 (.075)	0.652

6.4.4.5.5 Microlead I on Plaster

There were 463 observations of the Microlead I on plaster, 4 of which were designated as outliers (Machines 20 and 22: 80260 and 80262), leaving 459 observations for analysis. The breakdown by machines, operators, and cities was as follows:

Machine 20:	Operator G	220	total (99 D, 121 P)
Machine 21:	Operator E	54	total (all D)
	Operator H	18	total (all P)
Machine 22:	Operator E	44	total (all D)
Machine 23:	Operator H	103	total (all P)
Machine 24:	Operator F	20	total (all L)

<u>Matched pairs analysis</u>: A number of sign tests were performed on matched pairs:

(1) Machine 20 (Operator G) was compared to Machine 21 (Operator E) on 54 Denver sites of common measurement. Machine 20 gave a higher reading 24 times, with 5 ties. This would be a very plausible result if no machine or operator effects were present.

(2) Machine 20 (Operator G) was compared to Machine 21 (Operator H) on 18 Philadelphia sites of common measurement. Machine 20 gave a higher reading only 3 times, with 1 tie. This is a moderately unlikely result under a 50-50 chance hypothesis: the p-value of the sign test is approximately 1.56 percent.

(3) Machine 20 (Operator G) was compared to Machine 22 (Operator E) on 46 sites of common measurement. Machine 20 gave a higher reading 24 times, with 2 ties, which is a likely result if there were no machine or operator effects.

(4) Machine 20 (Operator G) was compared to Machine 23 (Operator H) on 103 sites of common measurement. Machine 20 gave a higher reading 28 times, with 10 ties. The sign test has a p-value of about 0.02 percent, which suggests that Machine 20 was prone to reading less than Machine 23, or that the effect may be due to the difference in operators.

Comparing Machines 21 and 22 for Operator E using results from (1) and (3) does not give a significant Fisher's exact test,

nor does comparison of Machines 21 and 23 for Operator H using (2) and (4). A comparison of Operator E and Operator H within Machine 21 using results from (1) and (2) has a p-value of about 2 percent, which is moderately significant, but may suggest city as opposed to operator effects.

Figure 6-37 shows the response and SD components of the estimated model before provision for the combined effect of spatial variation and laboratory error in ICP measurements. The agreement between the model and the nonparametric estimates appears to be good at ICP levels less than about 20.0 mg/cm^2.

Table 6-99 gives the results of fitting XRF measurement models to the data. Although the machine slopes (parameter *b*) range from 0.765 to 1.211, the large standard errors suggest that this may not be a true distinguishing feature. The intercepts (parameter *a*) show Machine 23 as the only standout, and appear to confirm the sign test result noted above. Overall, however, the intercepts do not vary greatly across machines. A city effect was not prominent on this substrate. Splitting Machine 20 into Denver and Philadelphia and fitting separate models to each gave intercept estimates of -0.092 (.072) for Denver and 0.013 (.055) for Philadelphia.

Table 6-100 gives a summary of the control block data. Machine 21 stands out as having given lower readings than the other machines, and its corresponding bias estimates are consistent with those obtained from the model. The high positive bias for Machine 22 suggested in the control block data, however, was not replicated in the field sample data. Machine 22, unlike the other machines, also gave markedly different SD estimates in the two sets of results, in contrast to the other machines considered.

6.4.4.5.6 Microlead I on Wood

There were 739 observations of the Microlead I on wood, 8 of which were designated as outliers (Machines 20 and 21: 80207 and 80218; Machines 20 and 22: 80720; Machine 20 only: 80323 and 81723), leaving 731 observations for analysis. The breakdown by machines, operators, and cities was as follows:

Machine 20:	Operator G	348 total (297 D, 51 P)
Machine 21:	Operator E	126 total (all D)

	Operator H	4	total (all P)
Machine 22:	Operator E	172	total (all D)
Machine 23:	Operator H	48	total (all P)
Machine 24:	Operator F	33	total (all L)

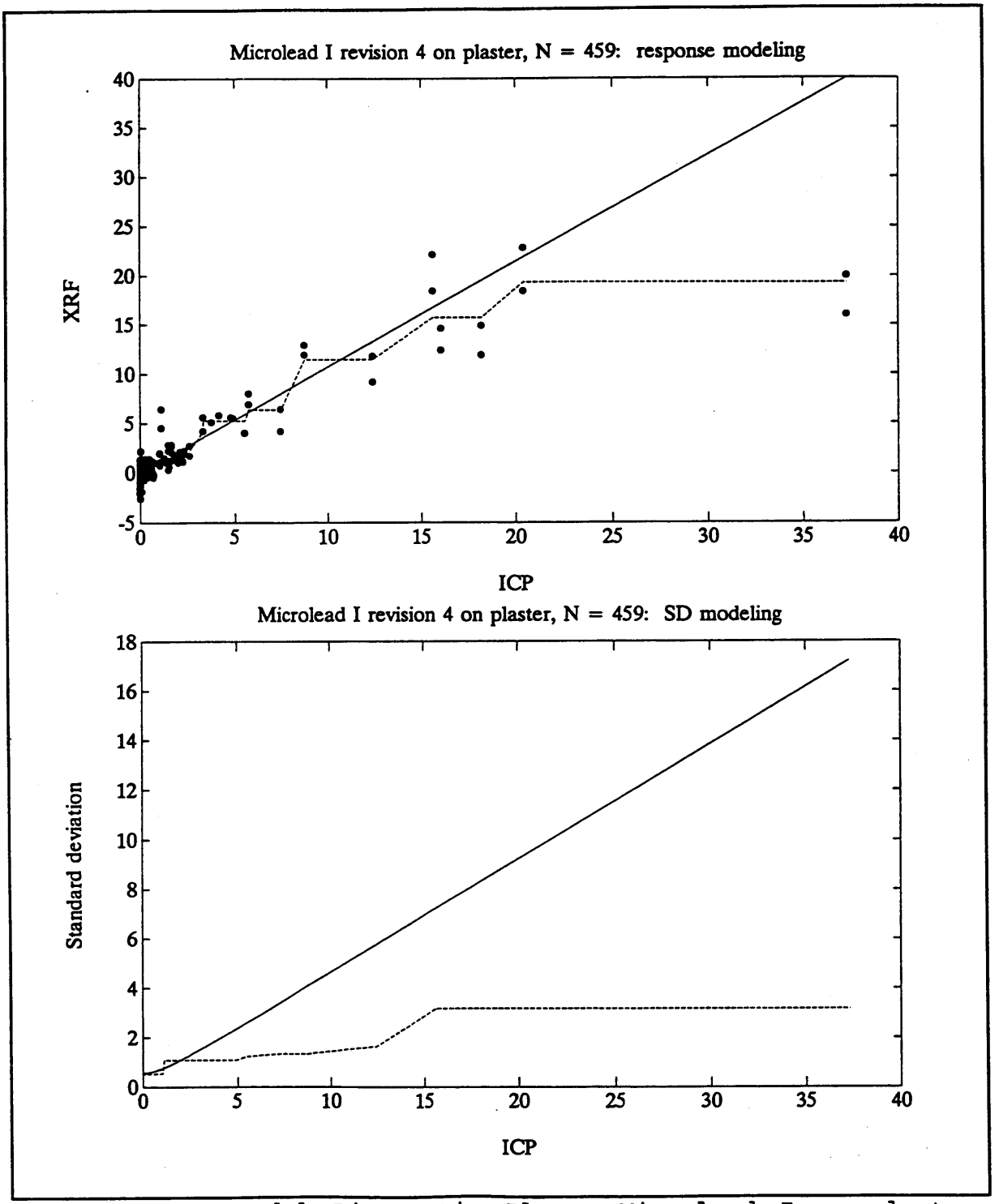


Figure 6-37. Model Diagnostic Plots, Microlead I on plaster. Solid lines are model estimates. Dashed lines are nonparametric (monotone regression) estimates.

Table 6-99. Microlead I on Plaster: Model Estimates.

DEVICE	SAMPLE SIZE	MODEL PARAMETERS				Pb=0.0 mg/cm²		Pb=1.0 mg/cm²	
		a	b	c	d	BIAS	SD	BIAS	SD
Machine 20, Denver	99	-0.092 (.072)	0.950 (.124)	0.396 (.064)	0.123 (.084)	-0.092 (.072)	0.630	-0.141 (.121)	0.721
Machine 20, Philadelphia	121	0.013 (.055)	0.903 (.117)	0.152 (.023)	0.058 (.059)	0.013 (.055)	0.390	-0.084 (.110)	0.458
Machine 20, combined	220	-0.043 (.043)	0.945 (.083)	0.260 (.027)	0.081 (.042)	-0.043 (.043)	0.510	-0.098 (.070)	0.584
Machine 21	72	-0.035 (.097)	1.211 (.147)	0.435 (.089)	0.216 (.133)	-0.035 (.097)	0.660	0.177 (.139)	0.807
Machine 22	44	-0.081 (.167)	0.765 (.480)	1.026 (.214)	-0-	-0.081 (.167)	1.013	-0.316 (.433)	1.013
Machine 23	103	0.217 (.069)	0.793 (.181)	0.137 (.026)	0.145 (.116)	0.217 (.069)	0.370	0.010 (.131)	0.531
Machines 20, 21, 23, and 24	415	0.010 (.049)	1.068 (.086)	0.265 (.034)	0.118 (.049)	0.010 (.049)	0.550	0.061 (.073)	0.641

Table 6-100. Microlead I on Plaster: Control Block Summary.

DEVICE	SAMPLE SIZE	BARE (0.0 mg/cm²)		RED NIST SRM (1.02 mg/cm²)		YELLOW NIST SRM (3.53 mg/cm²)	
		BIAS	SD	BIAS	SD	BIAS	SD
Machine 20	66	0.512 (.062)	0.505	0.510 (.085)	0.687	0.258 (.077)	0.625
Machine 21	20	0.055 (.148)	0.663	0.090 (.120)	0.535	-0.240 (.132)	0.588
Machine 22	16	1.125 (.125)	0.501	1.018 (.158)	0.632	1.214 (.150)	0.601
Machine 23	30	0.350 (.060)	0.327	0.287 (.054)	0.298	0.170 (.065)	0.356
Machines 21, 22, and 23	66	0.448 (.060)	0.491	0.404 (.058)	0.472	0.299 (.061)	0.500

Matched pairs analysis: A number of sign tests were performed on matched pairs:

(1) Machine 20 (Operator G) was compared to Machine 21 (Operator E) on 126 Denver sites of common measurement. Machine 20 gave a higher reading 29 times, with 7 ties. The probability that this could happen under a 50-50 chance hypothesis is nearly zero, suggesting that Machine 20 was prone to reading lower than Machine 21 with these operators.

(2) Machine 20 (Operator G) was compared to Machine 21 (Operator H) on 4 Philadelphia sites of common measurement. Machine 20 gave a higher reading 0 times, with no ties. The sample size is too small, however, to draw conclusions from this, except to note that it is consistent with the finding in (1).

(3) Machine 20 (Operator G) was compared to Machine 22 (Operator E) on 171 Denver sites of common measurement. Machine 20 gave a higher reading only 41 times, with 8 ties. The sign test has a p-value that is nearly zero percent, strongly suggesting the existence of an effect due to machine or operator.

(4) Machine 20 (Operator G) was compared to Machine 23 (Operator H) on 47 Philadelphia sites of common measurement. Machine 20 gave a higher reading only 11 times, with no ties. The sign test has a p-value of about 0.02 percent, which suggests that Machine 20 gave lower readings than Machine 23 with these operators.

None of the Fisher's exact tests for operator effects within machines, or machine effects within operators give significant results.

Figure 6-38 shows the response and SD components of the estimated model before provision for the combined effect of spatial variation and laboratory error in ICP measurements. The model appears to agree with the nonparametric estimates for ICP levels less than about 5.0 mg/cm^2, beyond which the nonparametric response estimate suggests a flattening out. The model should be adequate for drawing inferences about instrument performance at lower lead levels, where greater interest is focused.

Table 6-101 gives the results of fitting XRF measurement models to the data. The differences between machines, in particular those indicated in the matched pairs analysis, are

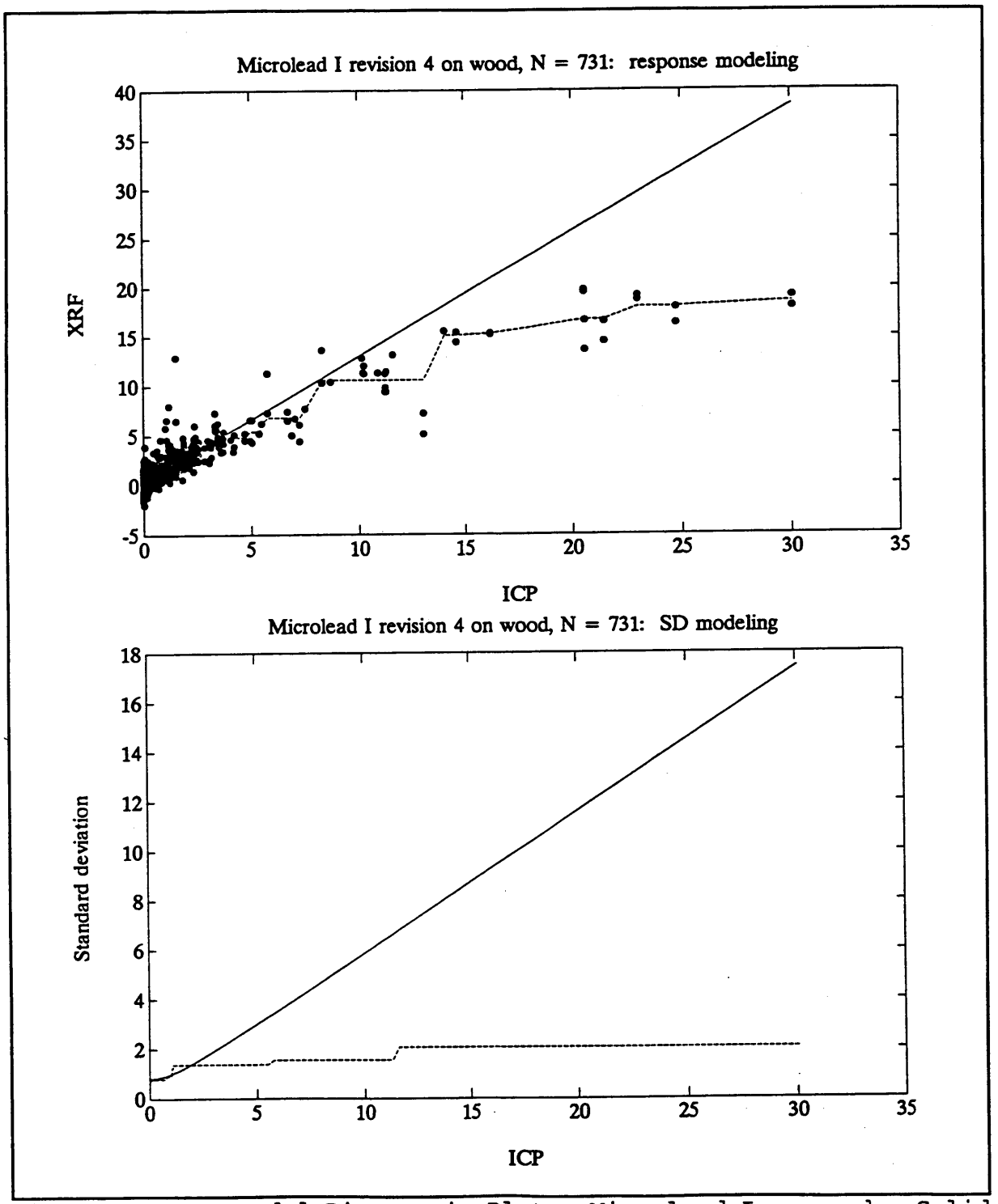


Figure 6-38. Model Diagnostic Plots, Microlead I on wood. Solid lines are model estimates. Dashed lines are nonparametric (monotone regression) estimates.

Table 6-101. Microlead I on Wood: Model Estimates.

DEVICE	SAMPLE SIZE	MODEL PARAMETERS				Pb=0.0 mg/cm²		Pb=1.0 mg/cm²	
		a	b	c	d	BIAS	SD	BIAS	SD
Machine 20, Denver	297	0.074 (.048)	1.106 (.059)	0.427 (.044)	0.047 (.027)	0.074 (.048)	0.654	0.180 (.055)	0.689
Machine 20, Philadelphia	51	0.042 (.238)	1.441 (.228)	1.113 (.243)	-0-	0.042 (.238)	1.055	0.483 (.230)	1.055
Machine 20, combined	348	0.001 (.045)	1.424 (.087)	0.389 (.040)	0.448 (.105)	0.001 (.045)	0.624	0.425 (.078)	0.915
Machine 21	130	0.505 (.078)	1.391 (.121)	0.439 (.067)	0.135 (.073)	0.505 (.078)	0.663	0.896 (.105)	0.758
Machine 22	172	0.601 (.075)	1.139 (.073)	0.689 (.085)	0.036 (.030)	0.601 (.075)	0.831	0.740 (.081)	0.851
Machine 23	48	0.329 (.132)	1.415 (.160)	0.250 (.069)	0.053 (.054)	0.329 (.132)	0.500	0.743 (.104)	0.550
Machine 24	33	0.341 (.161)	1.059 (.061)	0.334 (.138)	-0-	0.341 (.161)	0.578	0.400 (.139)	0.578

Table 6-102. Microlead I on Wood: Control Block Summary.

DEVICE	SAMPLE SIZE	BARE (0.0 mg/cm²)		RED NIST SRM (1.02 mg/cm²)		YELLOW NIST SRM (3.53 mg/cm²)	
		BIAS	SD	BIAS	SD	BIAS	SD
Machine 20	72	1.571 (.245)	2.076	1.472 (.266)	2.254	0.966 (.222)	1.886
Machine 21	22	-0.218 (.050)	0.236	-0.052 (.053)	0.248	-0.098 (.063)	0.343
Machine 22	22	0.482 (.065)	0.307	0.539 (.101)	0.473	0.552 (.220)	1.030
Machine 23	30	0.170 (.045)	0.248	0.097 (.042)	0.229	0.407 (.048)	0.260
Machines 21, 22, and 23	74	0.147 (.031)	0.264	0.184 (.038)	0.325	0.300 (.070)	0.606

reflected in the intercept terms (parameter *a*). Model estimates for Machine 24 (Louisville pilot) are much in line with the estimates for Machines 21 and 23.

Table 6-102 gives a summary of the control block data. As for metal, results for Machine 20 on wood may reflect a problem that does not accurately indicate its performance. Of the remainder,

Machine 21 stands out as having given low readings, and Machine 22 stands out as having given high readings. Machine 21 in particular did not exhibit comparable performance on the control blocks and on the field samples. Machine 22 had the highest SD estimates (excluding Machine 20), which is a pattern that appeared across substrates in both the control blocks and the model estimates.

6.4.4.5.7 Microlead I: Summary of Analysis

The multiplicity of machines, operators and cities made it difficult to fully assess their effects, which is necessary if a description of how the Microlead I performed on painted surfaces under practical conditions is desired. An attempt to do so using matched pairs, Fisher's exact tests, and tests on estimated model parameters revealed effects on every substrate, with the possible exception of brick. Effects distinguishing Denver and Philadelphia with the same operator using the same machine surfaced on concrete and metal. Effects due to machine (or operator) found Machine 22 reading higher than the others on drywall, metal and wood.

Machine 20 read lower than the others on concrete, drywall and wood. Machine 22 readings were also the most variable on several substrates, and this tendency was further confirmed in the control block data.

Results from both the field sample and control block data suggest that the instrument was proportionately responsive (*b* approximately equal to 1.0) to the lead level. These data also suggest bias that was prominent and variable across machines. This bias appeared to be an "add on" effect that might be removed through subtraction if its numerical value were known. But the bias levels did not appear to agree between the control blocks and the field sample data. Machine 22 on plaster, for instance,

had a bias estimated at 1.125 mg/cm^2 at a lead level of 0.0 mg/cm^2 in the control blocks, but the same estimate from the model was -0.081 mg/cm^2. SD estimates were closer in line, suggesting only a small to moderate increase in variability with the use of the instrument on the field samples, due possibly to non-instrumental factors.

6.4.4.6 Results for X-MET 880

The X-MET 880 is an L-shell instrument that was used in both the pilot and full studies. In the pilot, however, a different and newer radioactive source was used, as explained in section 6.4.1.1.1, which raised the issue of comparability with the full study. For this reason the Louisville pilot data were not combined with those from the full study. Separate model estimates were obtained for the pilot data on metal, and nonparametric estimates were derived for plaster and wood in order to bear out the difference in the performance of the X-MET 880 between the pilot and full studies.

Only one machine was used in the full study, designated as Machine 50. Two operators used the instrument in the full study. Operator K used the machine in Denver and in Philadelphia, and Operator J used the machine in Denver only. A different machine, Machine 51, was used in the pilot study, and it had a different operator (I).

6.4.4.6.1 X-MET 880 on Brick

There were 93 observations of the X-MET 880 on brick, one of which was designated as an outlier (80908), leaving 92 observations for analysis. Operator K made 69 of these readings, 57 in Denver and 12 in Philadelphia. Operator J made 23 readings, all in Denver.

Figure 6-39 shows the response and SD components of the estimated model before provision for the combined effect of spatial variation and laboratory error in ICP measurements. There is, in reality, very little response of the instrument to changes in the ICP measurement, as the narrow range of the vertical scale of the top graph alone would indicate. None of the readings with the instrument exceeded 1.0 mg/cm^2. The nonparametric estimate is itself flat for ICP measurements as high as 10.0 mg/cm^2, and it is clear that there was little or

nothing to be gained by fitting a detailed model to these data.

Table 6-103 gives a statistical summary of the field sample data that helps to shed light on instrument performance at lead levels of 0.0 mg/cm^2 and 1.0 mg/cm^2. Estimates at 0.0 mg/cm^2 are the average and SD of the XRF reading for ICP measurements less

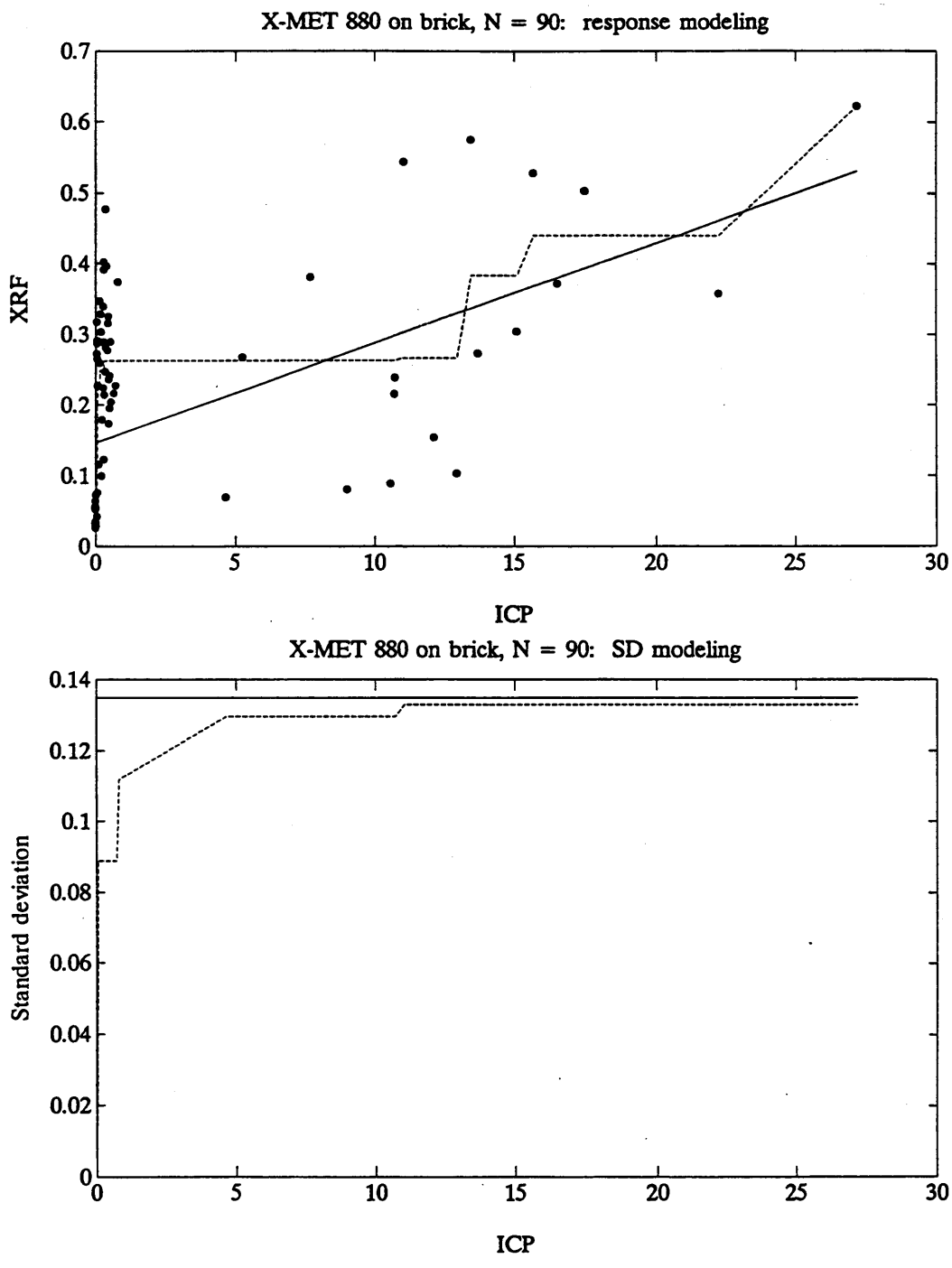


Figure 6-39. Model Diagnostic Plots, X-MET 880 on brick. Solid lines are model estimates. Dashed lines are nonparametric (monotone regression) estimates.

Table 6-103. X-MET 880 on Brick: Model Estimates.

DEVICE	SAMPLE SIZE	MODEL PARAMETERS				Pb=0.0 mg/cm²		Pb=1.0 mg/cm²	
		a	b	c	d	BIAS	SD	BIAS	SD
Machine 50, Operator K	49	--	--	--	--	0.041 (.005)	0.017	-0.741 (.015)	0.094
Machine 50, Operator J	23	--	--	--	--	0.030 (.0003)	0.002	--	--
Machine 50, combined	72	--	--	--	--	0.033 (.002)	0.011	-0.741 (.015)	0.094

Table 6-104. X-MET 880 on Brick: Control Block Summary.

DEVICE	SAMPLE SIZE	BARE (0.0 mg/cm²)		RED NIST SRM (1.02 mg/cm²)		YELLOW NIST SRM (3.53 mg/cm²)	
		BIAS	SD	BIAS	SD	BIAS	SD
Machine 50	72	0.052 (.003)	0.023	0.082 (.005)	0.042	0.298 (.021)	0.165

than 0.05 mg/cm^2, and those at 1.0 mg/cm^2 are for ICP measurements between 0.05 mg/cm^2 and 1.0 mg/cm^2. Strong negative bias with increasing lead levels is indicated, which agrees with Figure 6-39. An increase in variability with the lead level is also indicated for Operator K, but since Operator J made no readings at ICP measurements above 0.0079 mg/cm^2, a similar conclusion cannot be drawn for this operator.

Table 6-104 gives a summary of the control block data on the same machine. Increasing SD estimates with the lead level were apparent, but the bias figures suggest that the instrument overestimated the true level of lead, completely contrary to what was found on the field samples with this instrument.

6.4.4.6.2 X-MET 880 on Concrete

There were 218 observations of the X-MET 880 on concrete, 3 of which were designated as outliers (80938, 80945 and 81234), leaving 215 observations for analysis. Operator K made 188 of these readings, 69 in Denver and 119 in Philadelphia. Operator J made 27 readings, all in Denver.

Figure 6-40 shows the response and SD components of the estimated model before provision for the combined effect of spatial variation and laboratory error in ICP measurements. The nonparametric response estimate indicates a flattening effect for ICP levels greater than about 2.0 mg/cm^2. Only one XRF reading exceeded 1.0 mg/cm^2, and this was not at a higher ICP measurement. The SD estimates are in close agreement up to this point as well. Fitting the model only to data where the ICP measurement is less than 2.0 mg/cm^2 appears reasonable if inference to lower lead levels is desired. Figure 6-41 shows the model components on the restricted ICP range.

Table 6-105 gives the results of fitting XRF measurement models to the data. It should be noted that a significant city effect was apparent in the Operator K readings: the slope for the Denver data was 0.221 (.036) and for the Philadelphia data 0.034 (.006). The chi-square test has a p-value of less than 0.01 percent. This type of city effect, where the Denver slope was higher than for Philadelphia, appeared not only on several other substrates with the X-MET 880 but was observed on all of the L-shell instruments evaluated. But even with the higher slope in Denver, the instrument was under-responsive to changes

in the lead level, and became progressively more negatively biased as the lead level increased.

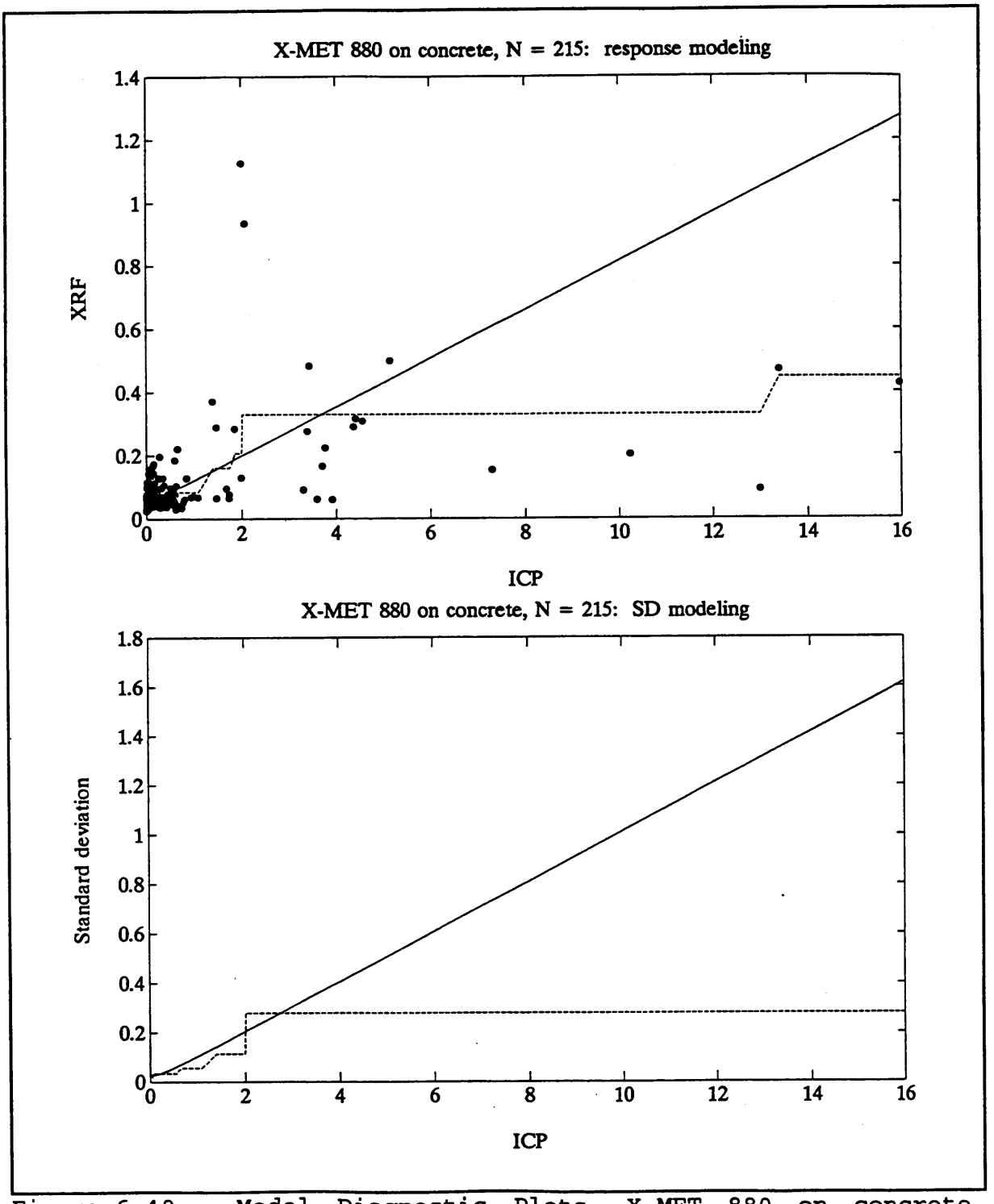


Figure 6-40. Model Diagnostic Plots, X-MET 880 on concrete. Solid lines are model estimates. Dashed lines are nonparametric (monotone regression) estimates.

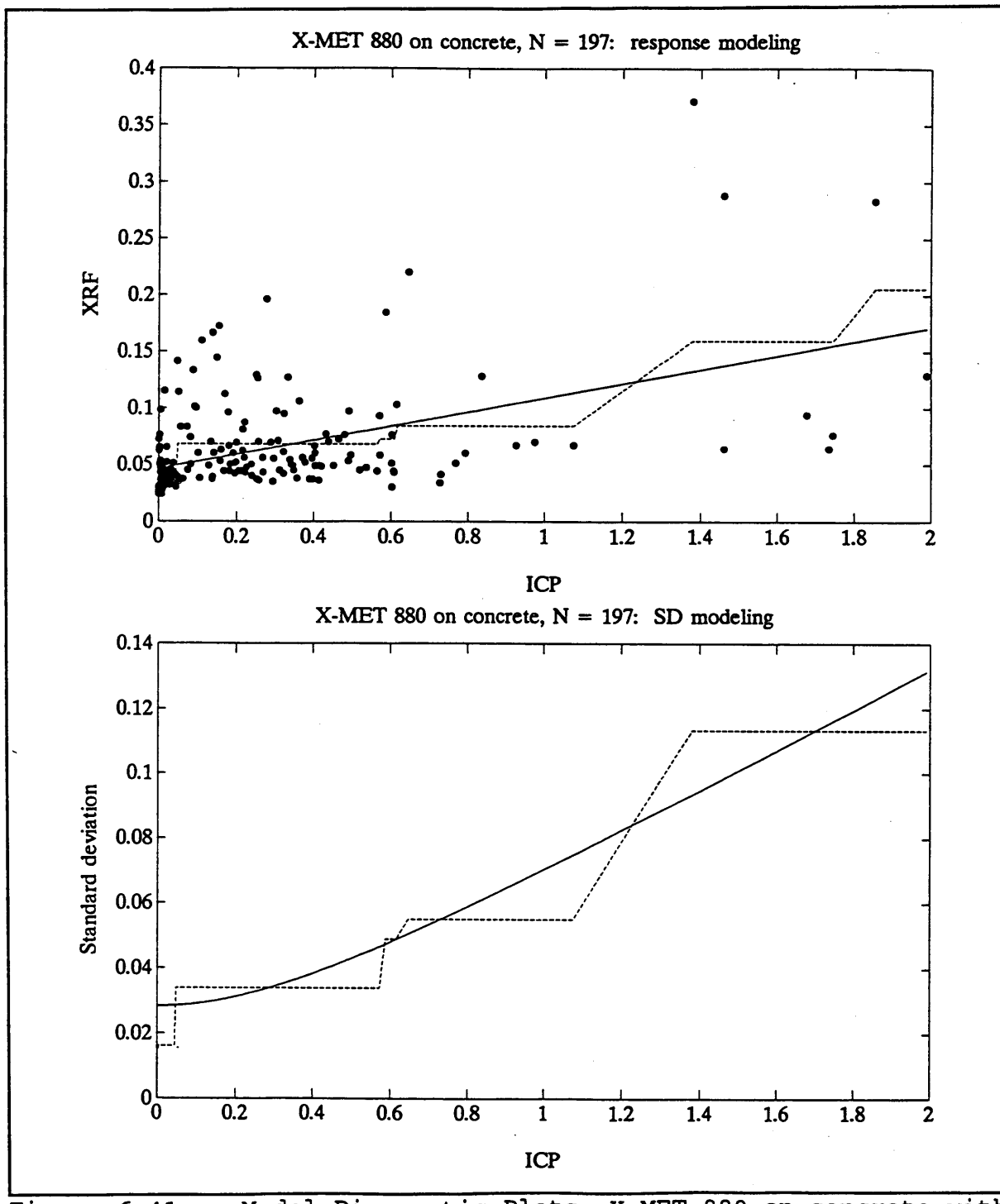


Figure 6-41. Model Diagnostic Plots, X-MET 880 on concrete with ICP restricted to less than 2.0 mg/cm². Solid lines are model estimates. Dashed lines are nonparametric (monotone regression) estimates.

Table 6-105. X-MET 880 on Concrete: Model Estimates.

DEVICE	SAMPLE SIZE	MODEL PARAMETERS				Pb=0.0 mg/cm²		Pb=1.0 mg/cm²	
		a	b	c	d	BIAS	SD	BIAS	SD
Machine 50, Operator K, Denver (ICP < 2)	56	0.049 (.006)	0.221 (.036)	0.001 (.0002)	-0-	0.049 (.006)	0.035	-0.730 (.034)	0.035
Machine 50, Operator K, Phila. (ICP < 2)	117	0.042 (.002)	0.034 (.006)	0.0001 (.0000)	0.0008 (.0003)	0.042 (.002)	0.011	-0.924 (.005)	0.030
Machine 50, Operator K (ICP < 2)	173	0.046 (.003)	0.055 (.016)	0.001 (.0001)	0.003 (.002)	0.046 (.003)	0.027	-0.899 (.013)	0.058
Machine 50, combined (ICP < 2)	197	0.045 (.003)	0.064 (.013)	0.001 (.0001)	0.004 (.002)	0.045 (.003)	0.027	-0.890 (.011)	0.067

Table 6-106. X-MET 880 on Concrete: Control Block Summary.

DEVICE	SAMPLE SIZE	BARE (0.0 mg/cm²)		RED NIST SRM (1.02 mg/cm²)		YELLOW NIST SRM (3.53 mg/cm²)	
		BIAS	SD	BIAS	SD	BIAS	SD
Machine 50	72	0.025 (.000)	0.002	0.053 (.005)	0.039	0.177 (.016)	0.135

Table 6-106 gives a summary of the control block data. A bias problem, to the extent that one exists, was one of overestimating the true lead level. Thus the instrument performed very differently on the control blocks and on the field samples. The SD estimates were comparable at 1.0 mg/cm^2. The higher model estimates at 0.0 mg/cm^2 reflect additional variability, due possibly to non-instrumental factors.

6.4.4.6.3 X-MET 880 on Drywall

There were 113 observations of the X-MET 880 on drywall, of which 2 were designated as outliers (80227 and 80935), leaving 111 observations for analysis. Operator K made 37 of these readings, 29 in Denver and 8 in Philadelphia. Operator J made 74 readings, all in Denver.

Figure 6-42 shows the response and SD components of the estimated model before provision for the combined effect of spatial variation and laboratory error in ICP measurements. Both appear to agree reasonably well with the nonparametric estimates. No ICP measurement in excess of 1.0 mg/cm^2 was observed on drywall on the field samples.

Table 6-107 gives the results of fitting XRF measurement models to the data. There were insufficient data to give separate results by city, but it is clear that the slope obtained for Operator K (0.245) reflected a predominance of Denver data. Bias became substantially negative as the lead level increased because the slopes are much less than 1.0. Table 6-108 gives a summary of the control block data, which even shows a positive bias at 3.53 mg/cm^2. Performance of the instrument on control blocks was not reflected in the field sample data.

6.4.4.6.4 X-MET 880 on Metal

There were 175 observations of the X-MET 880 on metal, none of which were designated as outliers. Operator K made 160 readings, 38 in Denver and 122 in Philadelphia. Operator J made 15 readings, all in Denver.

Figure 6-43 shows the response and SD components of the estimated model before provision for the combined effect of spatial variation and laboratory error in ICP measurements. Although there were instances where the XRF reading was close to

the ICP measurement at higher levels, this was more the exception

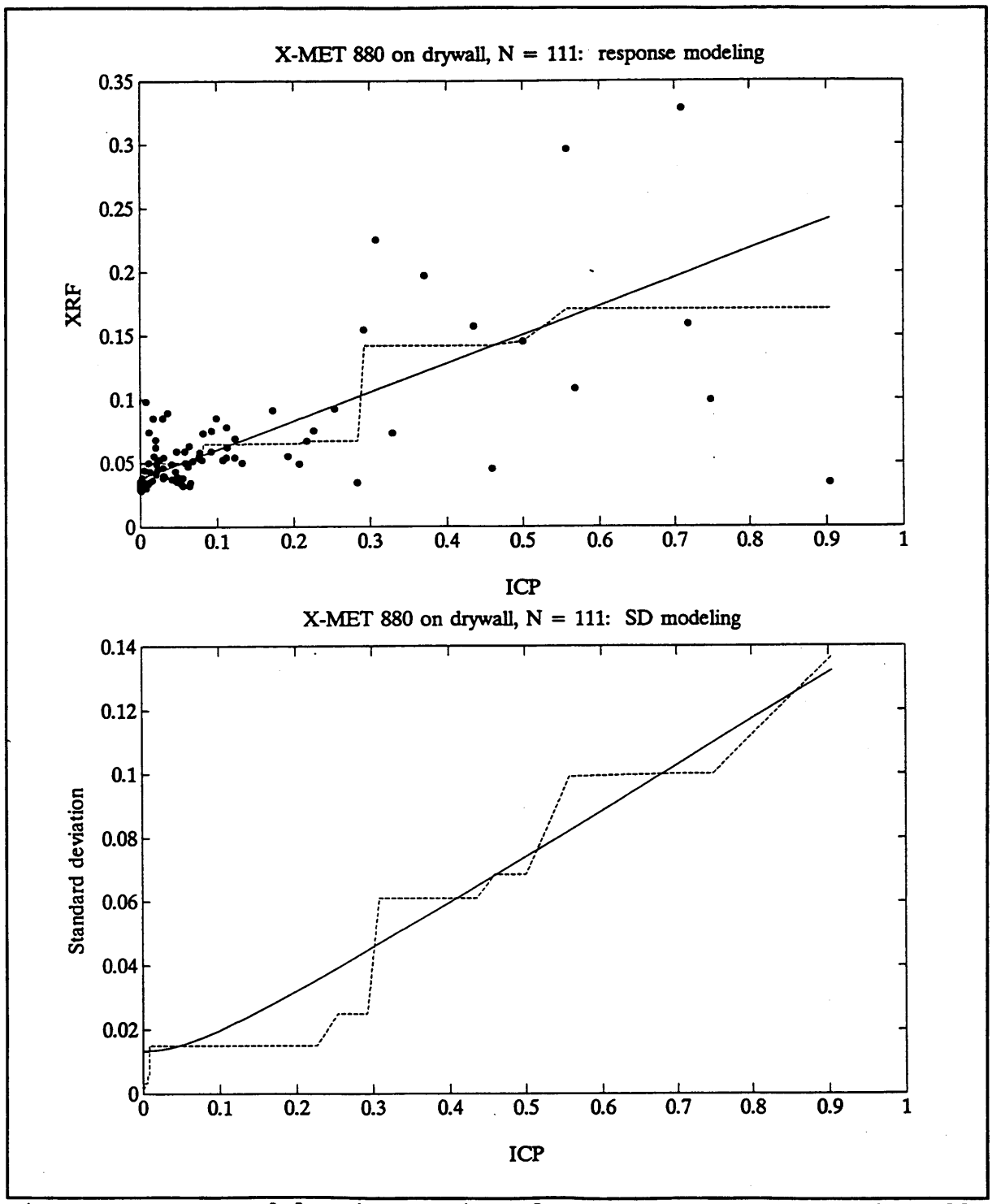


Figure 6-42. Model Diagnostic Plots, X-MET 880 on drywall. Solid lines are model estimates. Dashed lines are nonparametric (monotone regression) estimates.

Table 6-107. X-MET 880 on Drywall: Model Estimates.

DEVICE	SAMPLE SIZE	MODEL PARAMETERS				Pb=0.0 mg/cm²		Pb=1.0 mg/cm²	
		a	b	c	d	BIAS	SD	BIAS	SD
Machine 50, Operator K	37	0.040 (.003)	0.245 (.044)	0.0002 (.0001)	0.007 (.007)	0.040 (.003)	0.014	-0.714 (.042)	0.082
Machine 50, Operator J	74	0.036 (.002)	0.211 (.038)	0.0002 (.0001)	0.021 (.009)	0.036 (.002)	0.013	-0.753 (.037)	0.147
Machine 50, combined	111	0.038 (.002)	0.223 (.031)	0.0002 (.0003)	0.018 (.006)	0.038 (.002)	0.013	-0.739 (.030)	0.133

Table 6-108. X-MET 880 on Drywall: Control Block Summary.

DEVICE	SAMPLE SIZE	BARE (0.0 mg/cm²)		RED NIST SRM (1.02 mg/cm²)		YELLOW NIST SRM (3.53 mg/cm²)	
		BIAS	SD	BIAS	SD	BIAS	SD
Machine 50	60	0.035 (.000)	0.002	0.041 (.005)	0.040	0.260 (.016)	0.125

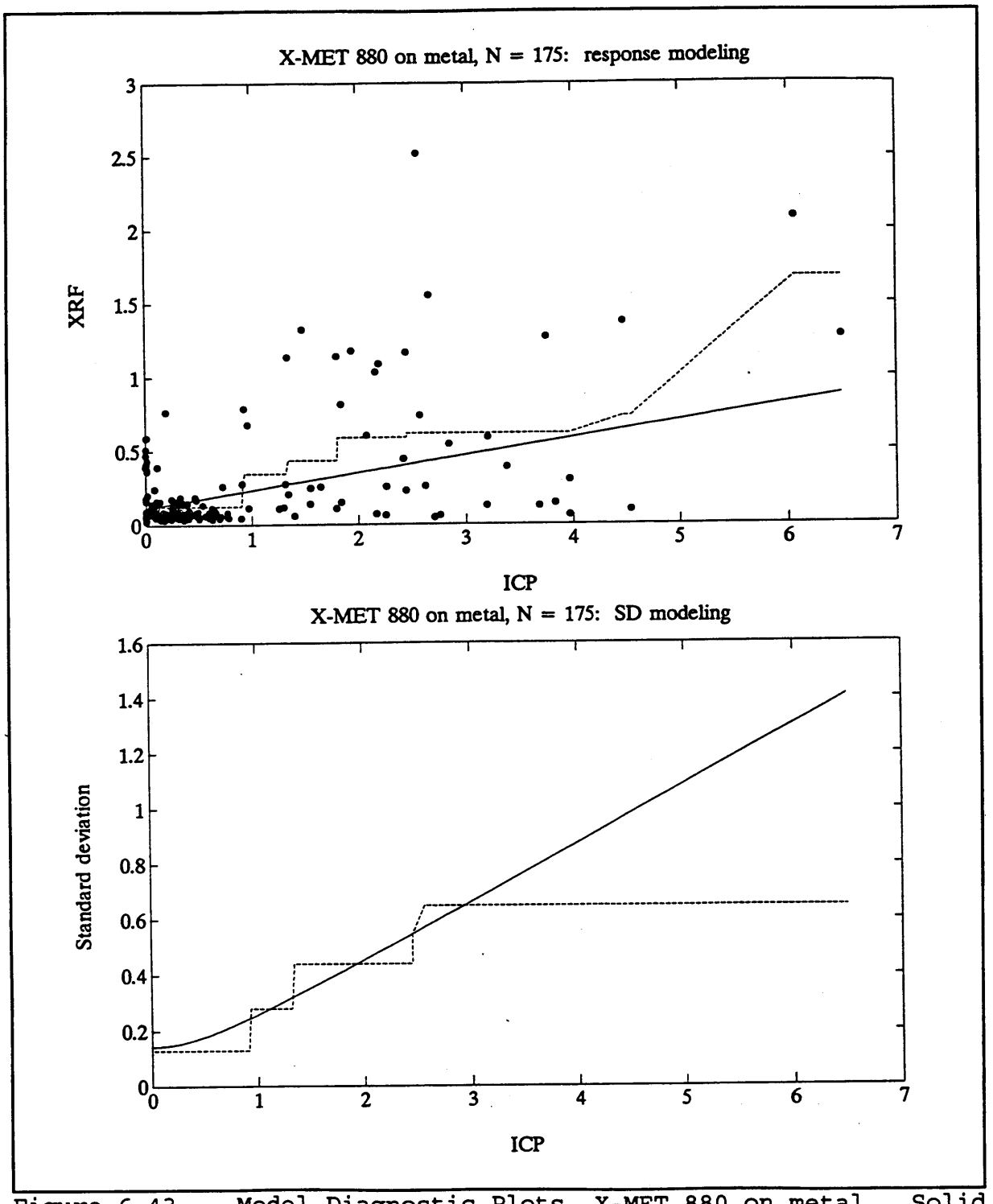


Figure 6-43. Model Diagnostic Plots, X-MET 880 on metal. Solid lines are model estimates. Dashed lines are nonparametric (monotone regression) estimates.

than the rule. The model appears to fit the data well relative to the nonparametric estimates. In contrast, Figure 6-44 shows that the pilot data exhibited more responsiveness to the lead level, especially in the lower ICP range.

Table 6-109 gives the results of fitting XRF measurement models to the data. The model was also fit to the Louisville pilot data. The Louisville model reveals the highest slope of any model fit to data from the X-MET 880 (0.795), accounting for its relatively small bias at 1.0 mg/cm^2 (-0.126).

Separate city estimation for Operator K revealed a city effect that was common across many of the L-shell analyses. The slope in Denver was higher (0.286 versus 0.103), as was the intercept (0.207 versus 0.060). A chi-square test on the 4 estimated model parameters has a p-value of less than 0.01 percent. Figure 6-45 reveals the dichotomy in performance, restricted to a narrow ICP range of under 0.5 mg/cm^2. The Philadelphia data were essentially oblivious to changes in the lead level. The slope in the Denver model was larger, yet there was still substantial bias at higher lead levels.

Since there are no control block data for the X-MET 880 on bare metal, Table 6-110 presents a summary for red and yellow NIST SRM only. As with other substrates, almost no bias was indicated at either of the two lead levels, which did not correspond to the performance of the instrument on the field samples.

6.4.4.6.5 X-MET 880 on Plaster

There were 222 observations of the X-MET 880 on plaster, 3 of which were designated as outliers (80075, 81340 and 81342), leaving 219 observations for analysis. Operator K made 173 of these readings, 54 in Denver and 119 in Philadelphia. Operator J made 46 readings, all in Denver.

Figure 6-46 shows a scatterplot of the XRF readings over the entire ICP range, and over the range restricted to ICP measurements less than 5.0 mg/cm^2. There were no ICP readings between 3.0 mg/cm^2 and 5.0 mg/cm^2. Restricting the ICP range for model fitting was reasonable for inference at lower lead levels in this case. It is worth noting that in no instance was an XRF reading above 1.0 mg/cm^2 obtained.

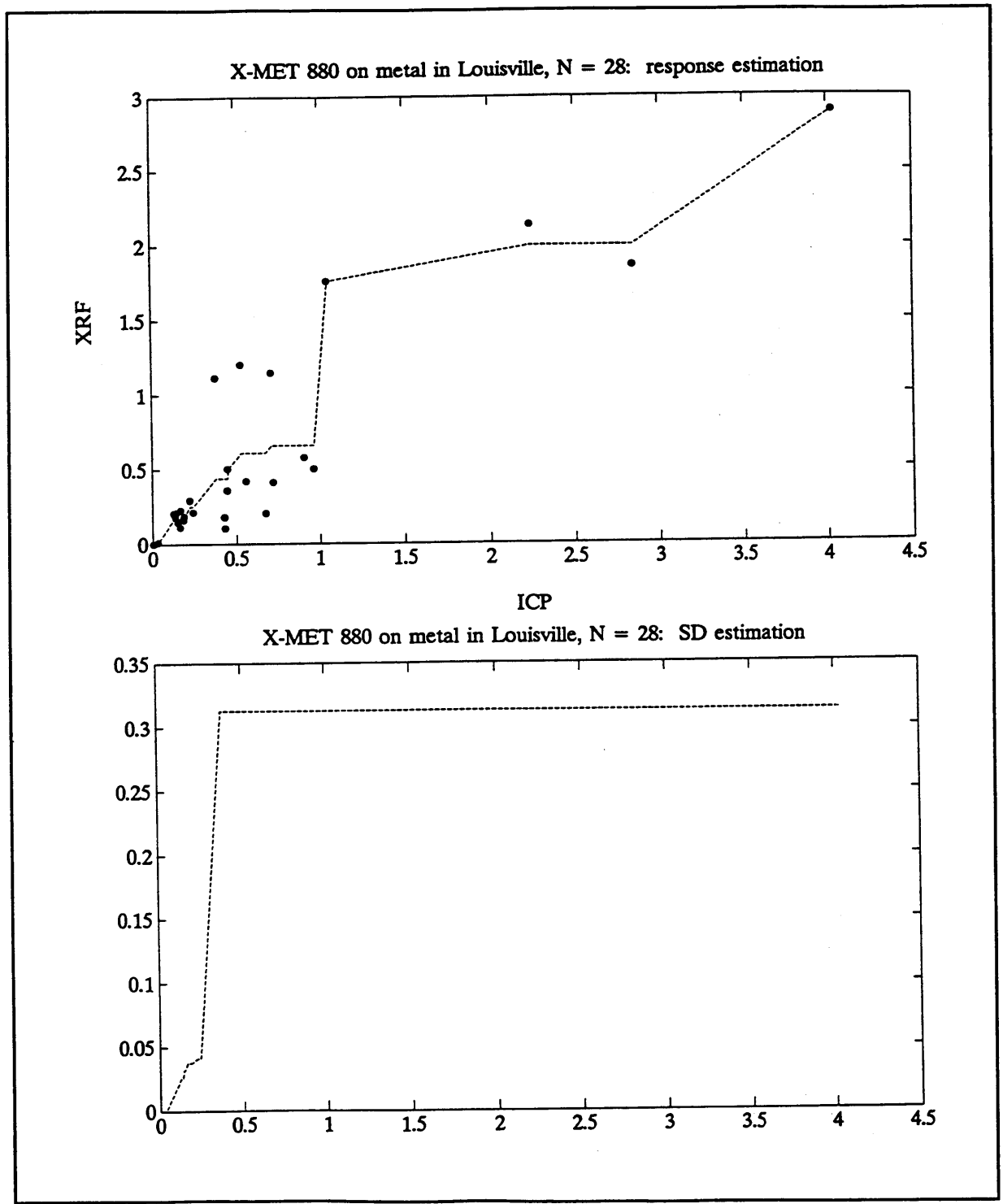


Figure 6-44. Nonparametric estimates for X-MET 880 on Metal in Louisville. Dashed lines are nonparametric (monotone regression) estimates.

Table 6-109. X-MET 880 on Metal: Model Estimates.

DEVICE	SAMPLE SIZE	MODEL PARAMETERS				Pb=0.0 mg/cm²		Pb=1.0 mg/cm²	
		a	b	c	d	BIAS	SD	BIAS	SD
Machine 50, Operator K, Denver	38	0.207 (.038)	0.286 (.096)	0.038 (.010)	0.050 (.037)	0.207 (.038)	0.195	-0.507 (.088)	0.296
Machine 50, Operator K, Philadelphia	122	0.060 (.008)	0.103 (.023)	0.001 (.0003)	0.020 (.004)	0.060 (.008)	0.038	-0.837 (.018)	0.145
Machine 50, Operator K	160	0.101 (.018)	0.122 (.033)	0.017 (.003)	0.036 (.008)	0.101 (.018)	0.131	-0.777 (.027)	0.230
Machine 50, combined	175	0.112 (.017)	0.120 (.032)	0.020 (.003)	0.037 (.008)	0.112 (.017)	0.141	-0.769 (.026)	0.238
Machine 51, Operator I	28	0.080 (.079)	0.795 (.114)	0.077 (.027)	-0-	0.080 (.079)	0.278	-0.126 (.082)	0.278

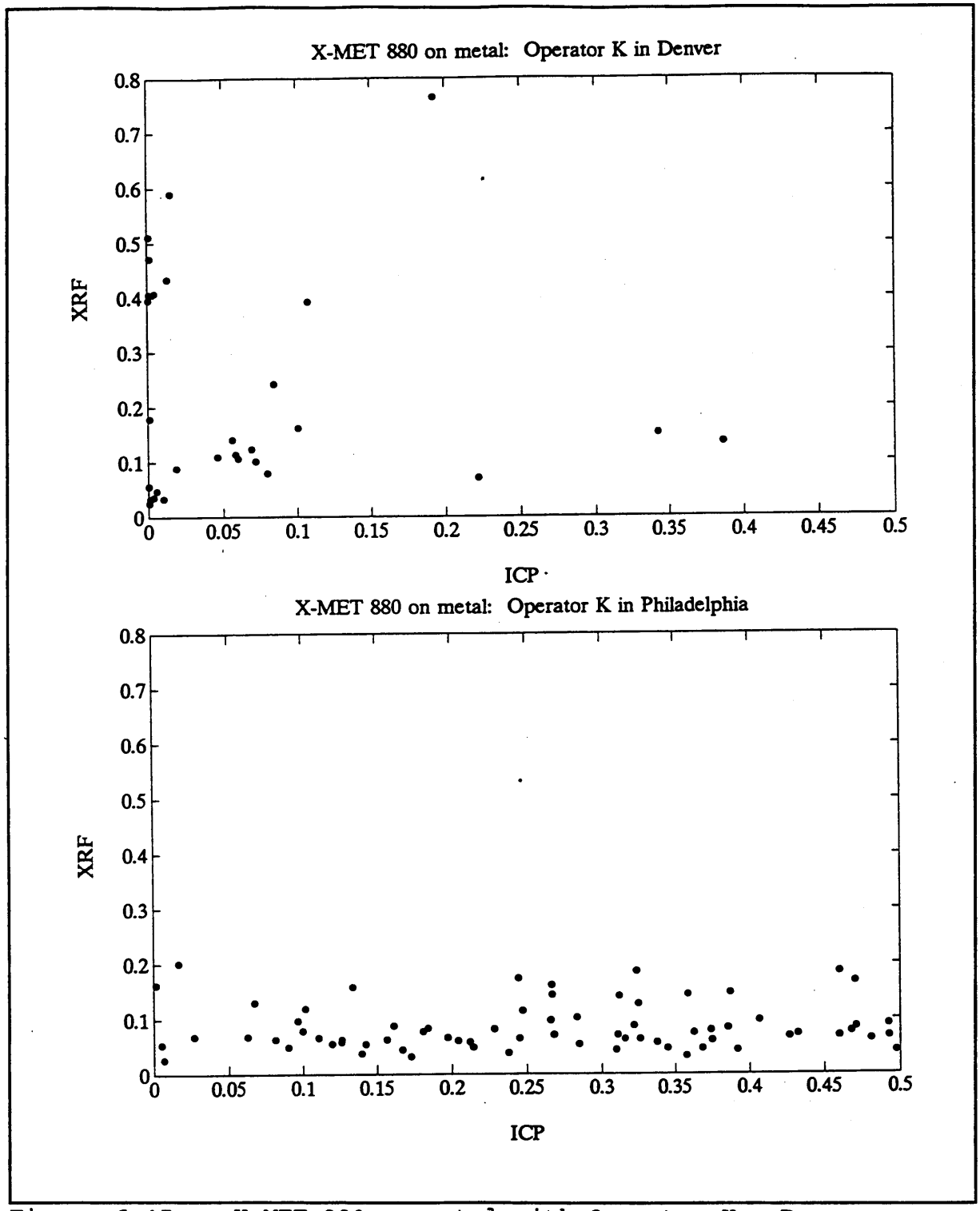


Figure 6-45. X-MET 880 on metal with Operator K: Denver versus Philadelphia.

Table 6-110. X-MET 880 on Metal: Control Block Summary.

DEVICE	SAMPLE SIZE	BARE (0.0 mg/cm²)		RED NIST SRM (1.02 mg/cm²)		YELLOW NIST SRM (3.53 mg/cm²)	
		BIAS	SD	BIAS	SD	BIAS	SD
Machine 50	77	--	--	0.054 (.005)	0.046	0.037 (.021)	0.183

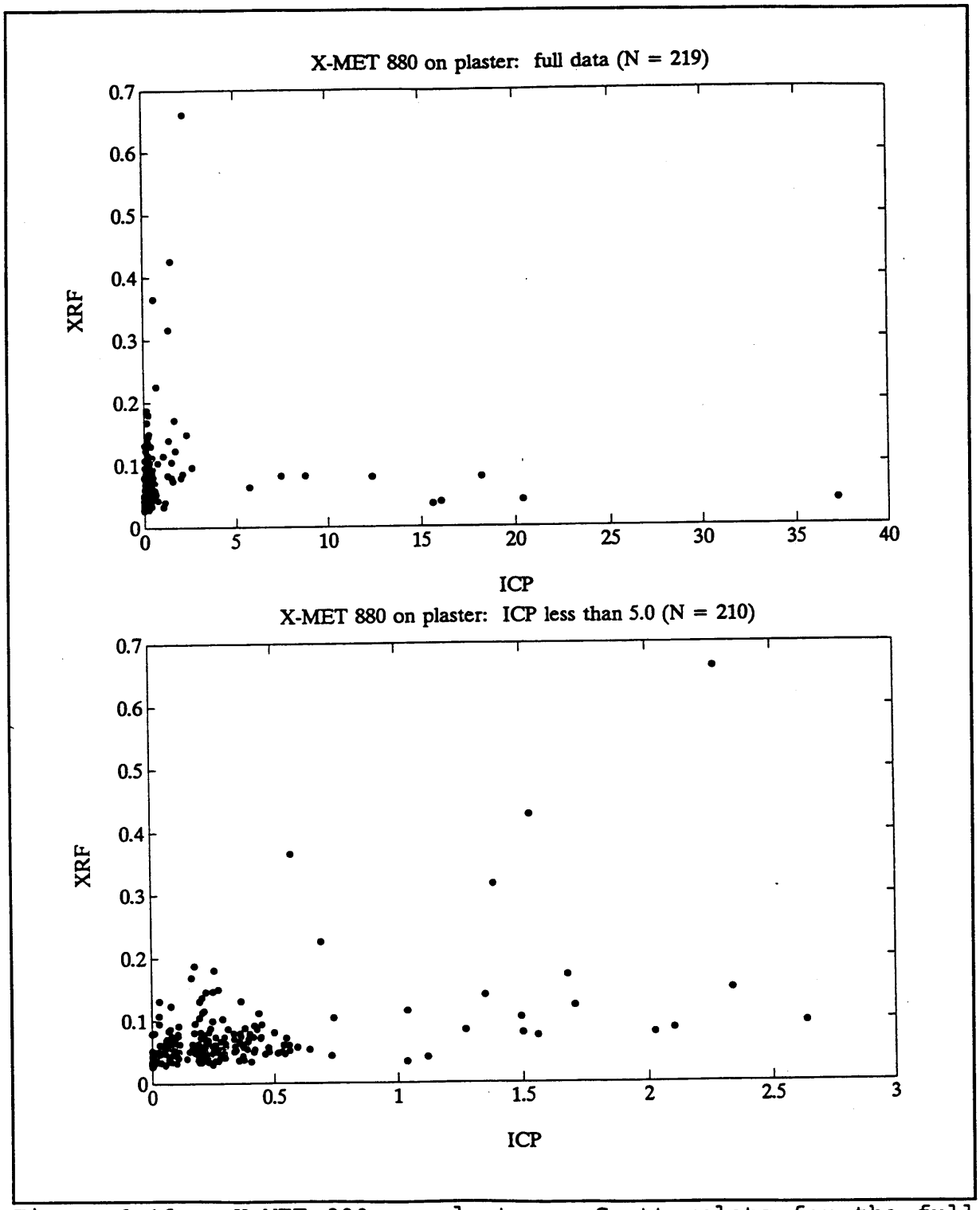


Figure 6-46. X-MET 880 on plaster: Scatterplots for the full and restricted ICP ranges.

Figure 6-47 shows the response and SD components of the estimated model before provision for the combined effect of spatial variation and laboratory error in ICP measurements. Both appear to be reasonably close to the nonparametric estimates, supporting the use of the model for making inferences at lower lead levels.

Figure 6-48 gives nonparametric graphical estimates for the Louisville pilot data, using Machine 51 and a different radioactive source. It is difficult to tell how this machine would have performed at a lead level of 1.0 mg/cm^2, due to a lack of data at lead levels near 1.0 mg/cm^2. Unlike the full study, there were no instances in the pilot where a sample with an ICP measurement in excess of 1.0 mg/cm^2 was classified by the X-MET 880 as having a lead level below 1.0 mg/cm^2.

Table 6-111 gives the results of fitting XRF measurement models to the data. Separate models fit to the Operator K readings by city yielded, as with other substrates and L-shell instruments, evidence of a city effect, with Denver having the higher of the two slopes (*b*). The difference in parameters is highly significant under a chi-square test. But the Operator J readings, which were made only in Denver, had a higher slope than the Denver readings made by Operator K (0.373 versus 0.160), which was marginally significant given the size of the standard errors of these estimates. In all cases the tendency was for the instrument to underestimate the true lead level to a substantial degree in the full study.

Table 6-112 gives a summary of the control block data. The SD estimates are close to those obtained from fitting the model to the combined field sample data. The control block data did not reflect the bias of the model estimates, however. At 3.53 mg/cm^2 of lead, the control block data even suggested a positive bias.

6.4.4.6.6 X-MET 880 on Wood

There were 353 observations of the X-MET 880 on wood, 2 of which were designated as outliers (80518 and 81316), leaving 351 observations for analysis. Operator K made 172 of these readings, 121 in Denver and 51 in Philadelphia. Operator J made the remaining 179 readings, all in Denver.

Figure 6-49 shows the response and SD components of the estimated model before provision for the combined effect of spatial variation and laboratory error in ICP measurements. The

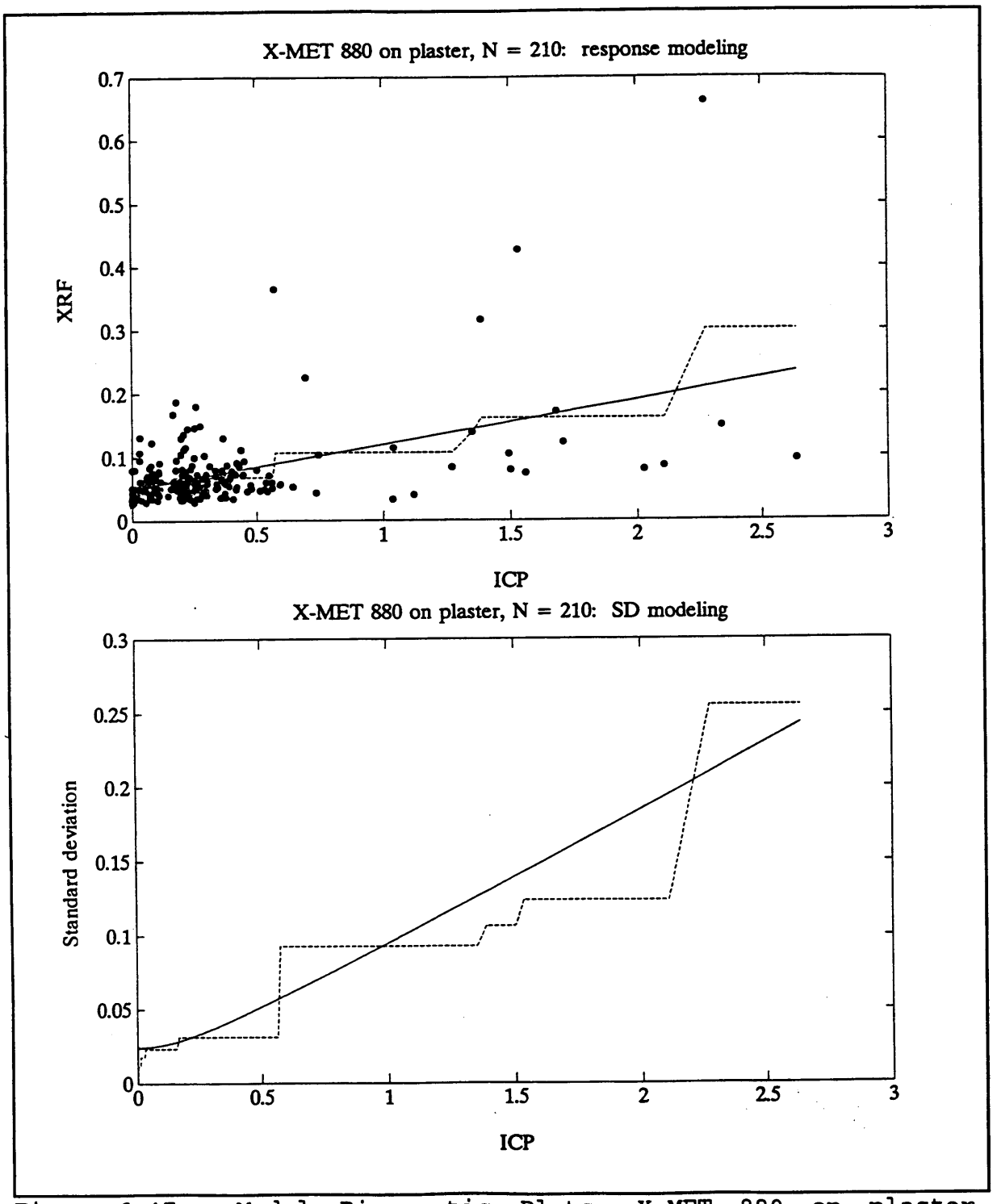


Figure 6-47. Model Diagnostic Plots, X-MET 880 on plaster. Solid lines are model estimates. Dashed lines are nonparametric (monotone regression) estimates.

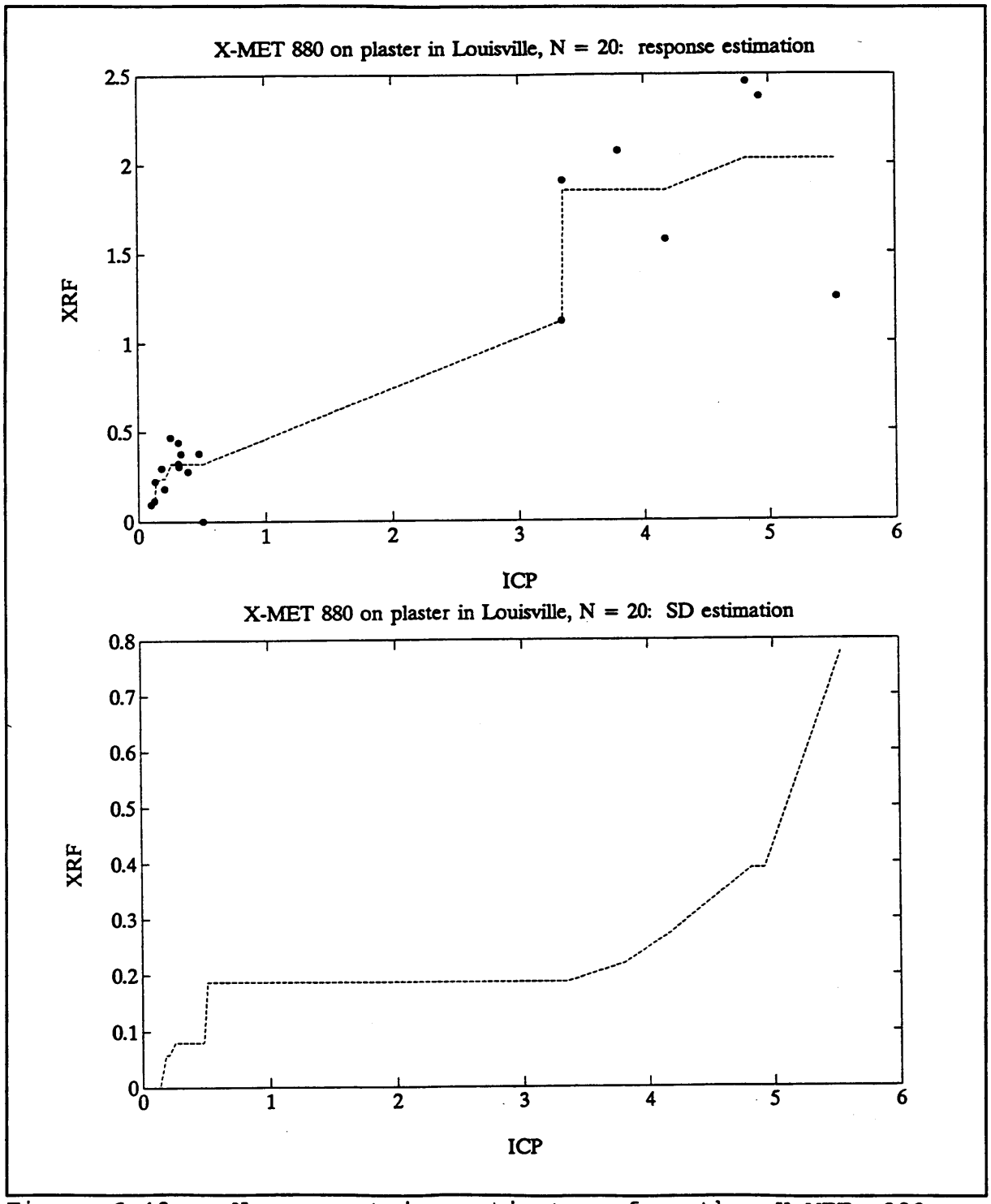


Figure 6-48. Nonparametric estimates for the X-MET 880 on Plaster. Dashed lines are nonparametric (monotone regression) estimates.

Table 6-111. X-MET 880 on Plaster: Model Estimates.

DEVICE	SAMPLE SIZE	MODEL PARAMETERS				Pb=0.0 mg/cm²		Pb=1.0 mg/cm²	
		a	b	c	d	BIAS	SD	BIAS	SD
Machine 50, Operator K, Denver (ICP<5)	45	0.038 (.004)	0.160 (.040)	0.0001 (.0001)	0.028 (.009)	0.038 (.004)	0.009	-0.803 (.038)	0.168
Machine 50, Operator K, Phila. (ICP<5)	119	0.055 (.003)	0.027 (.004)	0.0004 (.0001)	-0-	0.055 (.003)	0.021	-0.918 (.004)	0.021
Machine 50, Operator K (ICP<5)	164	0.053 (.003)	0.037 (.011)	0.0005 (.0001)	0.002 (.0009)	0.053 (.003)	0.022	-0.910 (.008)	0.050
Machine 50, Operator J (ICP<5)	46	0.036 (.003)	0.373 (.079)	0.0001 (.0001)	0.103 (.044)	0.036 (.003)	0.010	-0.591 (.070)	0.321
Machine 50, combined (ICP<5)	210	0.048 (.003)	0.072 (.013)	0.0005 (.0001)	0.006 (.002)	0.048 (.002)	0.022	-0.880 (.011)	0.083

Table 6-112. X-MET 880 on Plaster: Control Block Summary.

DEVICE	SAMPLE SIZE	BARE (0.0 mg/cm²)		RED NIST SRM (1.02 mg/cm²)		YELLOW NIST SRM (3.53 mg/cm²)	
		BIAS	SD	BIAS	SD	BIAS	SD
Machine 50	65	0.033 (.000)	0.002	0.041 (.010)	0.082	0.247 (.036)	0.291

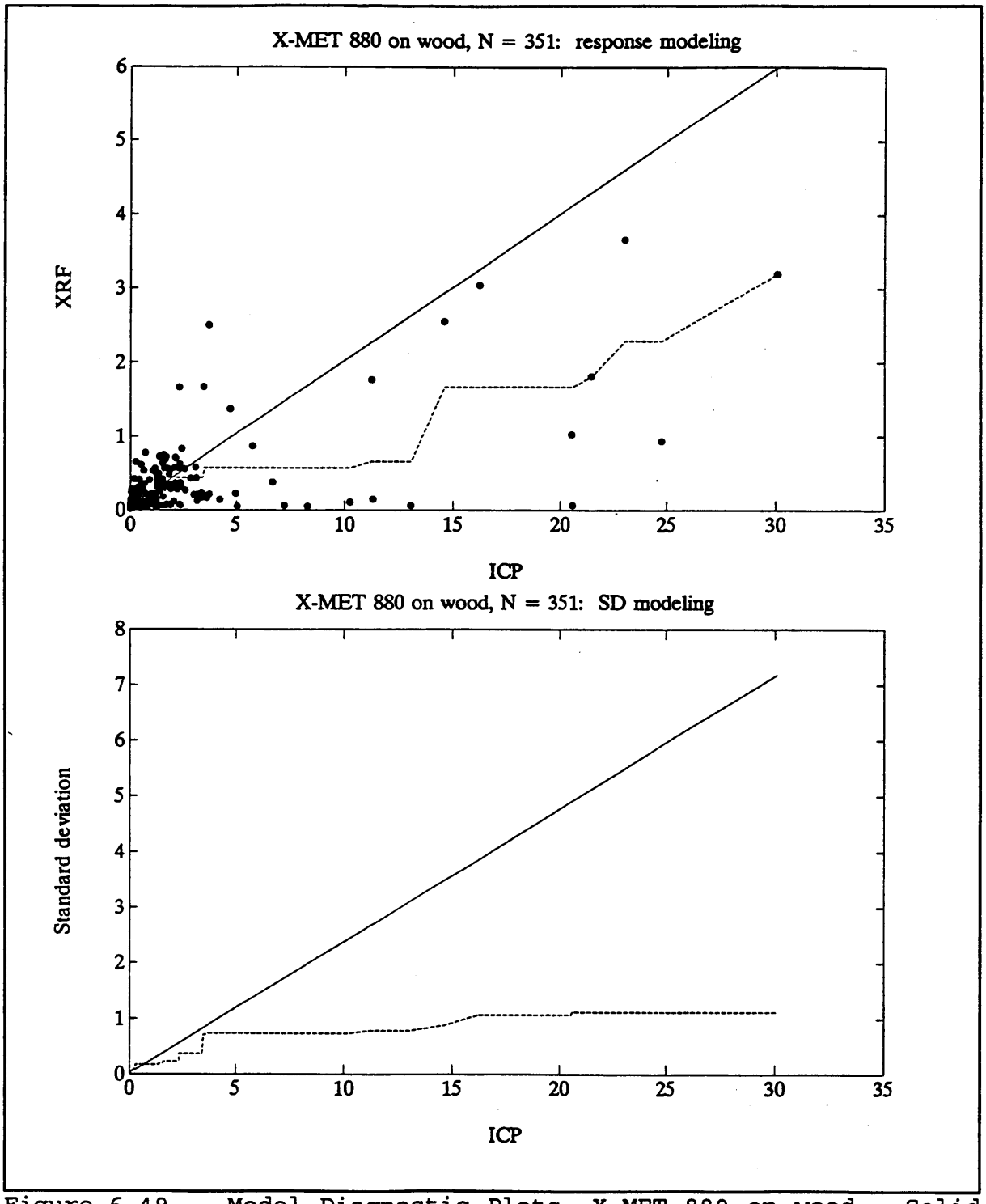


Figure 6-49. Model Diagnostic Plots, X-MET 880 on wood. Solid lines are model estimates. Dashed lines are nonparametric (monotone regression) estimates.

model fails to explain the data well at ICP levels above 5.0 mg/cm^2, which was true, generally, across the L-shell instruments. Restriction to a lower ICP range was necessary for the model to produce reasonable estimates of instrument performance at lower lead levels. Although XRF readings above 1.0 mg/cm^2 were obtained on a number of sampled locations, a reading of this magnitude was not assured, even with lead levels as high as 10.0 mg/cm^2.

Figure 6-50 shows nonparametric estimates for the Louisville pilot data. A cluster of zero readings at the lower ICP scale made it difficult to fit the XRF measurement model to these data. Compared to Machine 50 in the full study, Machine 51 in the pilot was clearly more responsive to the level of lead in paint, and it never gave a reading below 1.0 mg/cm^2 when the ICP measurement exceeded this level. The response is clearly nonlinear and gradually flattens out at higher ICP levels, but the bias remains relatively small for ICP levels as large as 4.0 mg/cm^2.

Table 6-113 gives the results of fitting XRF measurement models to the data, with restriction to ICP levels less than 5.0 mg/cm^2. These results repeat patterns seen across substrates with the X-MET 880, and across L-shell instruments as well. Denver data, for both Operators K and J, gave higher slope estimates than Philadelphia data.

Comparing Denver and Philadelphia within Operator K by means of a chi-square test gives a p-value of about 0.5 percent. The slope estimate of 0.408 (.051) obtained for Operator J readings is higher than the 0.209 (.023) obtained for Operator K readings in Denver, a highly significant difference. Both city and operator effects were therefore indicated by the estimated models.

Table 6-114 gives a summary of the control block data. Bias is not indicated as a problem on the control blocks. The SD estimates are lower in the control blocks compared to those derived from the model, which may reflect non-instrumental sources of variability.

6.4.4.6.7 X-MET 880: Summary of Analysis

The X-MET 880 performed like other L-shell instruments that were evaluated in the full study. Under even the best of

circumstances, responsiveness of the instrument to changes in the lead level fell short, which explains the increase in the bias as the lead level increased. This behavior was not reflected in the

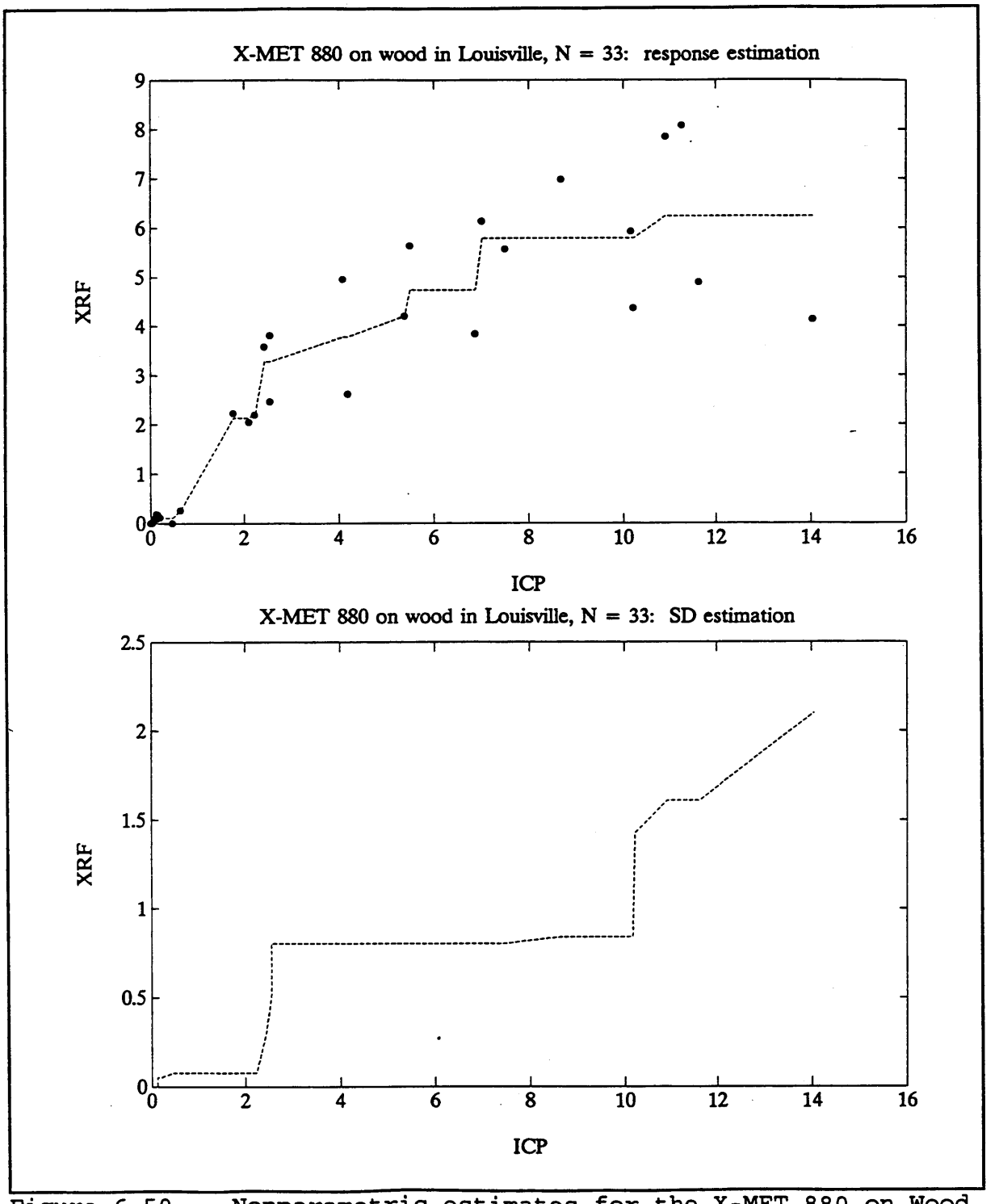


Figure 6-50. Nonparametric estimates for the X-MET 880 on Wood. Dashed lines are nonparametric (monotone regression) estimates.

Table 6-113. X-MET 880 on Wood: Model Estimates.

DEVICE	SAMPLE SIZE	MODEL PARAMETERS				Pb=0.0 mg/cm²		Pb=1.0 mg/cm²	
		a	b	c	d	BIAS	SD	BIAS	SD
Machine 50, Operator K, Denver (ICP<5)	113	0.034 (.002)	0.209 (.023)	0.0002 (.0001)	0.019 (.005)	0.034 (.002)	0.013	-0.758 (.022)	0.137
Machine 50, Operator K, Phila. (ICP<5)	50	0.042 (.006)	0.078 (.020)	0.0005 (.00001)	0.014 (.004)	0.042 (.006)	0.023	-0.879 (.019)	0.122
Machine 50, Operator K (ICP<5)	163	0.035 (.002)	0.163 (.018)	0.0002 (.0001)	0.023 (.005)	0.035 (.002)	0.013	-0.802 (.017)	0.152
Machine 50, Operator J (ICP<5)	171	0.044 (.003)	0.408 (.051)	0.0007 (.002)	0.177 (.045)	0.044 (.003)	0.026	-0.547 (.050)	0.422
Machine 50, Operators K and J (ICP<5)	334	0.042 (.003)	0.259 (.025)	0.0006 (.0001)	0.083 (.014)	0.042 (.003)	0.024	-0.699 (.024)	0.289

Table 6-114. X-MET 880 on Wood: Control Block Summary.

DEVICE	SAMPLE SIZE	BARE (0.0 mg/cm²)		RED NIST SRM (1.02 mg/cm²)		YELLOW NIST SRM (3.53 mg/cm²)	
		BIAS	SD	BIAS	SD	BIAS	SD
Machine 50	72	0.035 (.000)	0.004	0.060 (.006)	0.047	0.061 (.022)	0.185

control blocks, which implies that their usefulness as a baseline for practical measurement situations was questionable. On all substrates except wood and metal, the control blocks even showed a positive bias at a lead level of 3.53 mg/cm^2.

Several factors affected the performance of the X-MET 880 on the field samples. City effects, consistent across substrates, suggest that the XRF readings were affected by factors aside from the true lead level, the generic substrate type (wood, plaster, etc.), operator effects, and instrumental error. An effect due to operators was also evident.

The performance of the X-MET 880 in the pilot study, using a different machine, operator, and radioactive source, was markedly different from the full study. The instrument responded more sharply at lower lead levels, its bias was lower, and it was more certain to give readings above 1.0 mg/cm^2 in the presence of high lead levels.

6.4.4.7 Results for XK-3

The XK-3 is a K-shell instrument that was used in both the pilot and full studies. In the full study there were two field classifications (Class I and Class II), allowing paired comparisons to be made. Three different machines and three different operators were used, which are described as follows:

Machine 30 (Operator M)	Denver (I) and Philadelphia(II)
Machine 31 (Operator L)	Denver (II) and Louisville
Machine 32 (Operators M & N)	Philadelphia (II)

The XK-3 only read a maximum of 10 mg/cm^2: values potentially higher than this were truncated to the maximum value. To avoid problems with truncation, analyses were conducted on restricted ICP ranges as necessary to eliminate or reduce the frequency of these readings. The partial crossing of machines, operators, and cities allowed limited assessment of these factors controlling for the others, depending on the availability of data. Cities are at times referred to by their first letters for the sake of brevity.

6.4.4.7.1 XK-3 on Brick

There were 186 observations of the XK-3 on brick, one of which was designated as an outlier (Machine 30: 80750), leaving 185 observations for analysis. Breakdown of the data by machines, cities and operators was as follows:

Machine 30:	Operator M	92 total (80 D, 12 P)
Machine 31:	Operator L	81 total (all D)
Machine 32:	Operator M	2 total (all P)
	Operator N	10 total (all P)

No observations on brick substrates were obtained in the Louisville pilot study. Upper truncation of the XRF readings at 10.0 mg/cm^2 was observed at 9 locations, the lowest ICP measurement of which was 10.7203 mg/cm^2.

<u>Matched pairs analysis</u>: Several sign tests were performed on matched pairs arising from the field classifications:

(1) Machine 30 (Operator M) was compared to Machine 31 (Operator L) on 80 Denver sites of common measurement. Machine 30 gave a higher reading 53 times, with 5 ties. The sign test has a p-value of about 0.04 percent, suggesting a significant effect where Machine 30 reads higher than Machine 31. The difference in measurements has a Spearman rank correlation of about -0.3 with the ICP measurement, suggesting that the effect was seen at lower lead levels.

(2) Machine 30 (Operator M) was compared to Machine 32 (Operator N) on 10 Philadelphia sites of common measurement. Machine 30 gave a higher reading only 1 time, with no ties. The sign test has a p-value of about 2 percent, making this a moderately strong indication that a machine effect existed.

(3) Machine 30 (Operator M) was compared to Machine 32 (Operator M) on 2 Philadelphia sites of common measurement. On both sites Machine 30 gave a higher reading. The sample size was not large enough to reach conclusions about machine effects, but the result was moderately incompatible with the sign test in (2), with the same machines but a different Machine 31 operator. The Fisher's exact test has a p-value of 4.4 percent. An effect, if one existed, may be due to the operator.

To avoid the truncation problem in model fitting, only data

for which the ICP measurement is less than 10.0 mg/cm^2 were used, which resulted in the loss of 32 observations. Figure 6-51 shows the response and SD components of the estimated model before provision for the combined effect of spatial variation and laboratory error in ICP measurements. The nonparametric response

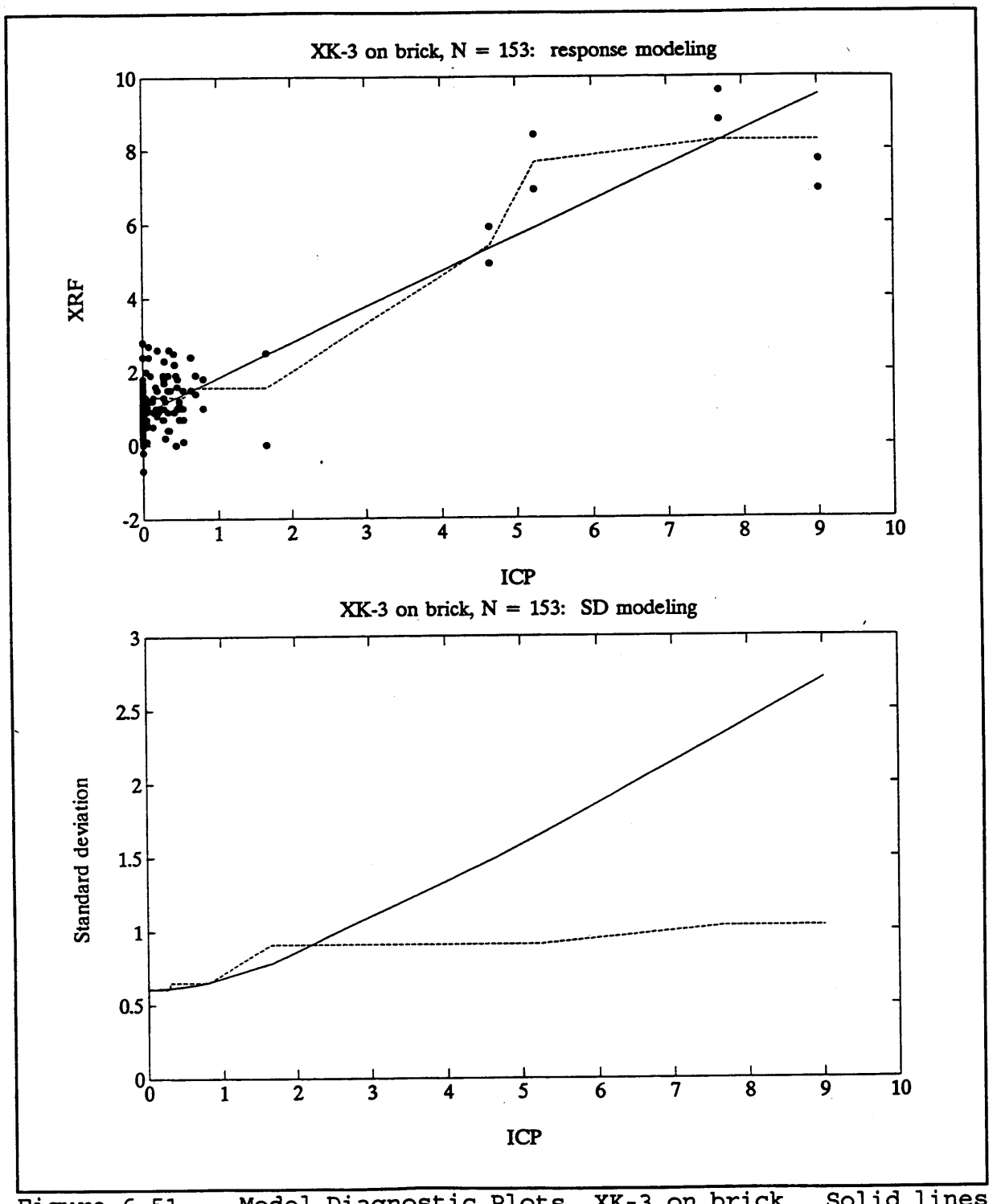


Figure 6-51. Model Diagnostic Plots, XK-3 on brick. Solid lines are model estimates. Dashed lines are nonparametric (monotone regression) estimates.

is much flatter in the lower ICP range than the model would indicate. Further restriction to ICP measurements below 1.0 mg/cm^2 would appear to better describe instrument performance at lower lead levels.

Table 6-115 gives the results of fitting XRF measurement models for Machines 30, 31, and combined. The intercept estimates for Machine 30 (1.001) and Machine 31 (0.472) bear out the machine effect detected with the sign test (1). Table 6-116 gives a summary of the control block data. Machine 30 read higher than Machine 31 on the control blocks, as it did on the field samples, but Machine 32 had still higher readings. Strong positive bias, and a slope of about 1.0 in response to changes in the lead level, are indicated in both sets of results.

6.4.4.7.2 XK-3 on Concrete

There were 444 observations of the XK-3 on concrete, none of which were designated as outliers. Breakdown of the data by machines, cities and operators was as follows:

Machine 30:	Operator M	218 total (98 D, 120 P)
Machine 31:	Operator L	106 total (98 D, 8 L)
Machine 32:	Operator M	15 total (all P)
	Operator N	105 total (all P)

Upper truncation of the XRF readings at 10.0 mg/cm^2 was observed at 5 locations, the lowest ICP measurement of which was 10.2428 mg/cm^2.

<u>Matched pairs analysis</u>: Several sign tests were performed on matched pairs arising from the field classifications:

(1) Machine 30 (Operator M) was compared to Machine 31 (Operator L) on 98 Denver sites of common measurement. Machine 30 gave a higher reading 53 times, with 10 ties. The sign test has a p-value of about 7 percent, which does not suggest a significant effect. All ICP measurements were below 0.7 mg/cm^2 in this comparison.

(2) Machine 30 (Operator M) was compared to Machine 32 (Operator N) on 105 Philadelphia sites of common measurement. Machine 30 gave a higher reading only 17 times, with 1 tie. The sign test has a p-value of less than 0.01 percent, making

this strong evidence that Machine 30 read less than Machine 32, or that an operator effect existed.

Table 6-115. XK-3 on Brick: Model Estimates.

DEVICE	SAMPLE SIZE	MODEL PARAMETERS				Pb=0.0 mg/cm²		Pb=1.0 mg/cm²	
		a	b	c	d	BIAS	SD	BIAS	SD
Machine 30 (ICP<1)	71	1.001 (.090)	1.328 (.355)	0.350 (.060)	-0-	1.001 (.090)	0.591	1.329 (.320)	0.591
Machine 31 (ICP<1)	60	0.472 (.056)	1.181 (.242)	0.102 (.022)	-0-	0.472 (.056)	0.320	0.653 (.210)	0.320
Machines 30, 31, and 32 (ICP<1)	143	0.861 (.064)	1.016 (.251)	0.359 (.043)	-0-	0.861 (.064)	0.600	0.877 (.218)	0.600

Table 6-116. XK-3 on Brick: Control Block Summary.

DEVICE	SAMPLE SIZE	BARE (0.0 mg/cm²)		RED NIST SRM (1.02 mg/cm²)		YELLOW NIST SRM (3.53 mg/cm²)	
		BIAS	SD	BIAS	SD	BIAS	SD
Machine 30	58	0.912 (.044)	0.335	1.099 (.060)	0.460	0.370 (.089)	0.678
Machine 31	30	0.733 (.049)	0.271	0.710 (.040)	0.217	0.290 (.069)	0.377
Machine 32	28	1.339 (.072)	0.382	1.501 (.085)	0.451	1.256 (.127)	0.671
Machines 30, 31, and 32	116	0.969 (.031)	0.332	1.096 (.038)	0.409	0.563 (.057)	0.613

(3) Machine 30 (Operator M) was compared to Machine 32 (Operator M) on 15 Philadelphia sites of common measurement. Machine 30 gave a higher reading 10 times, with 1 tie. This is not strong evidence of a machine effect under the sign test.

An operator effect may be suggested by the results of (2) and (3), since a Fisher's exact test has a p-value of less than 0.01 percent. Thus, the difference between Machines 30 and 32 may reflect operator as opposed to machine effects.

Figure 6-52 shows the response and SD components of the estimated model before provision for the combined effect of spatial variation and laboratory error in ICP measurements. Only data for ICP levels less than 10.0 mg/cm^2 were used in order to avoid the problem with upper truncation. The nonparametric response suggests a flattening effect for ICP measurements larger than about 1.0 mg/cm^2. Further restriction of the data to ICP measurements less than 1.0 mg/cm^2 was employed in order to obtain model estimates and to describe instrument performance at lower ICP levels.

Table 6-117 gives the results of fitting XRF measurement models, by machine. Machine 30 data produced different intercept estimates when analyzed by city: 0.947 (.089) in Denver, and 1.305 (.086) in Philadelphia. The difference is slightly less than 3 standard errors in magnitude, a marginally significant result. Since the operator in both cities was the same, the possible existence of city effects should be considered. The intercepts also bring out differences between machines that were reflected in the sign test. Machine 32 stood out as having an especially large bias. Since the slopes (b) are all within 3 standard errors of being equal to 1.0, the instrument appeared to respond proportionately to the lead level.

Table 6-118 gives a summary of the control block data. The same ordering of the machines suggested in the field sample data (Machine 32 bigger than 30 bigger than 31) was reflected in the control blocks as well. The SD estimates were generally lower from the control block data than from the field sample data.

6.4.4.7.3 XK-3 on Drywall

There were 237 observations of the XK-3 on drywall, 2 of which were designated as outliers (80332 and 80345), leaving 235

observations for analysis. Breakdown of the data by machines, cities and operators was as follows:

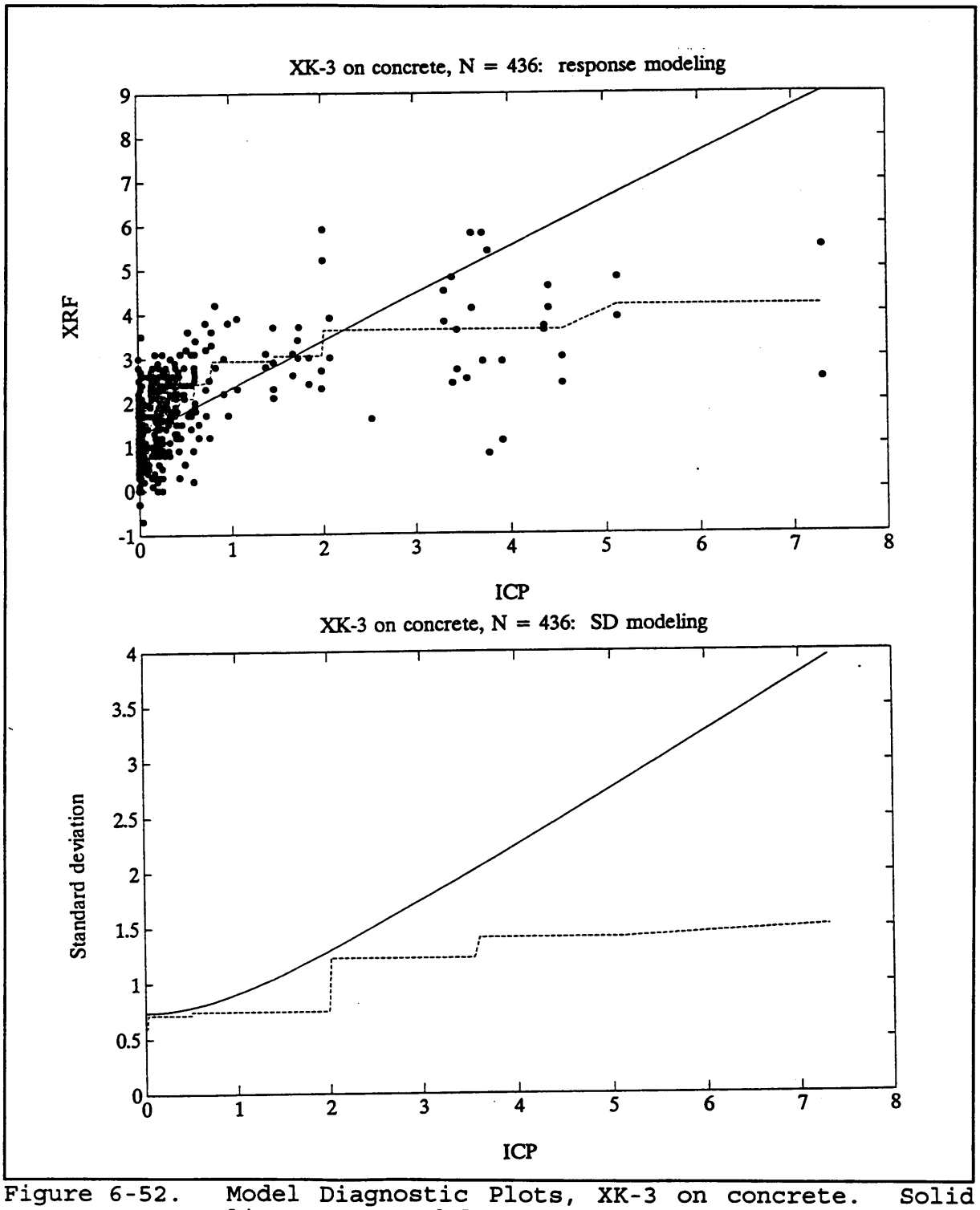


Figure 6-52. Model Diagnostic Plots, XK-3 on concrete. Solid lines are model estimates. Dashed lines are nonparametric (monotone regression) estimates.

Table 6-117. XK-3 on Concrete: Model Estimates.

DEVICE	SAMPLE SIZE	MODEL PARAMETERS				Pb=0.0 mg/cm²		Pb=1.0 mg/cm²	
		a	b	c	d	BIAS	SD	BIAS	SD
Machine 30, Denver (ICP < 1)	79	0.947 (.089)	1.137 (.622)	0.440 (.071)	-0-	0.947 (.089)	0.663	1.084 (.587)	0.663
Machine 30, Philadelphia (ICP < 1)	112	1.305 (.086)	1.337 (.246)	0.335 (.047)	-0-	1.305 (.086)	0.579	1.642 (.210)	0.579
Machine 30, Combined (ICP < 1)	191	1.083 (.063)	1.668 (.227)	0.404 (.043)	-0-	1.083 (.063)	0.636	1.751 (.200)	0.636
Machine 31 (ICP < 1)	79	0.660 (.036)	0.570 (.254)	0.718 (.017)	-0-	0.660 (.036)	0.847	0.230 (.237)	0.847
Machine 32 (ICP < 1)	97	1.837 (.080)	1.732 (.244)	0.258 (.039)	-0-	1.837 (.193)	0.508	2.569 (.193)	0.508

Table 6-118. XK-3 on Concrete: Control Block Summary.

DEVICE	SAMPLE SIZE	BARE (0.0 mg/cm²)		RED NIST SRM (1.02 mg/cm²)		YELLOW NIST SRM (3.53 mg/cm²)	
		BIAS	SD	BIAS	SD	BIAS	SD
Machine 30	70	0.804 (.056)	0.470	0.860 (.073)	0.610	0.516 (.108)	0.901
Machine 31	38	0.582 (.039)	0.238	0.638 (.043)	0.266	0.275 (.056)	0.346
Machine 32	32	1.456 (.079)	0.446	1.746 (.073)	0.416	1.286 (.102)	0.577
Machines 30, 31, and 32	140	0.893 (.035)	0.414	1.002 (.042)	0.496	0.626 (.061)	0.718

Machine 30:	Operator M	112 total (104 D, 8 P)
Machine 31:	Operator L	115 total (104 D, 11 L)
Machine 32:	Operator M	0 total
	Operator N	8 total (all P)

Upper truncation of the XRF readings at 10.0 mg/cm^2 was not observed, which was most likely due to the fact that none of the ICP measurements were above 1.0 mg/cm^2 on drywall.

Matched pairs analysis: Several sign tests were performed on matched pairs arising from the field classifications:

(1) Machine 30 (Operator M) was compared to Machine 31 (Operator L) on 103 Denver sites of common measurement. Machine 30 gave a higher reading only 8 times, with 3 ties. The sign test has a p-value of almost zero percent, which strongly suggests a significant effect, with Machine 31 giving higher readings.

(2) Machine 30 (Operator M) was compared to Machine 32 (Operator N) on 8 Philadelphia sites of common measurement. Machine 30 did not give a higher reading on any of these, with no ties. The sign test has a p-value of 0.39 percent, which suggests that Machine 30 read less than Machine 32.

Figure 6-53 shows the response and SD components of the estimated model before provision for the combined effect of spatial variation and laboratory error in ICP measurements. Both components appear to agree well with the nonparametric estimates. Table 6-119 gives the results of fitting XRF measurement models to the data by machine. The lower Machine 30 readings were reflected in a lower intercept estimate (-0.327) relative to that of Machine 31 (0.245).

Table 6-120 gives a summary of the control block data. Again, both Machines 31 and 32 read higher than Machine 30. For both Machines 30 and 31, however, higher bias estimates were obtained from the control block data than from the model. The SD estimates are comparable at 0.0 mg/cm^2, but the model estimates are larger at 1.0 mg/cm^2, where the limited ICP range may not make these estimates reliable.

6.4.4.7.4 XK-3 on Metal

There were 406 observations of the XK-3 on metal, 2 of which were designated as outliers (Machine 30: 80407; Machine 32: 81840), leaving 404 observations for analysis. Breakdown of the

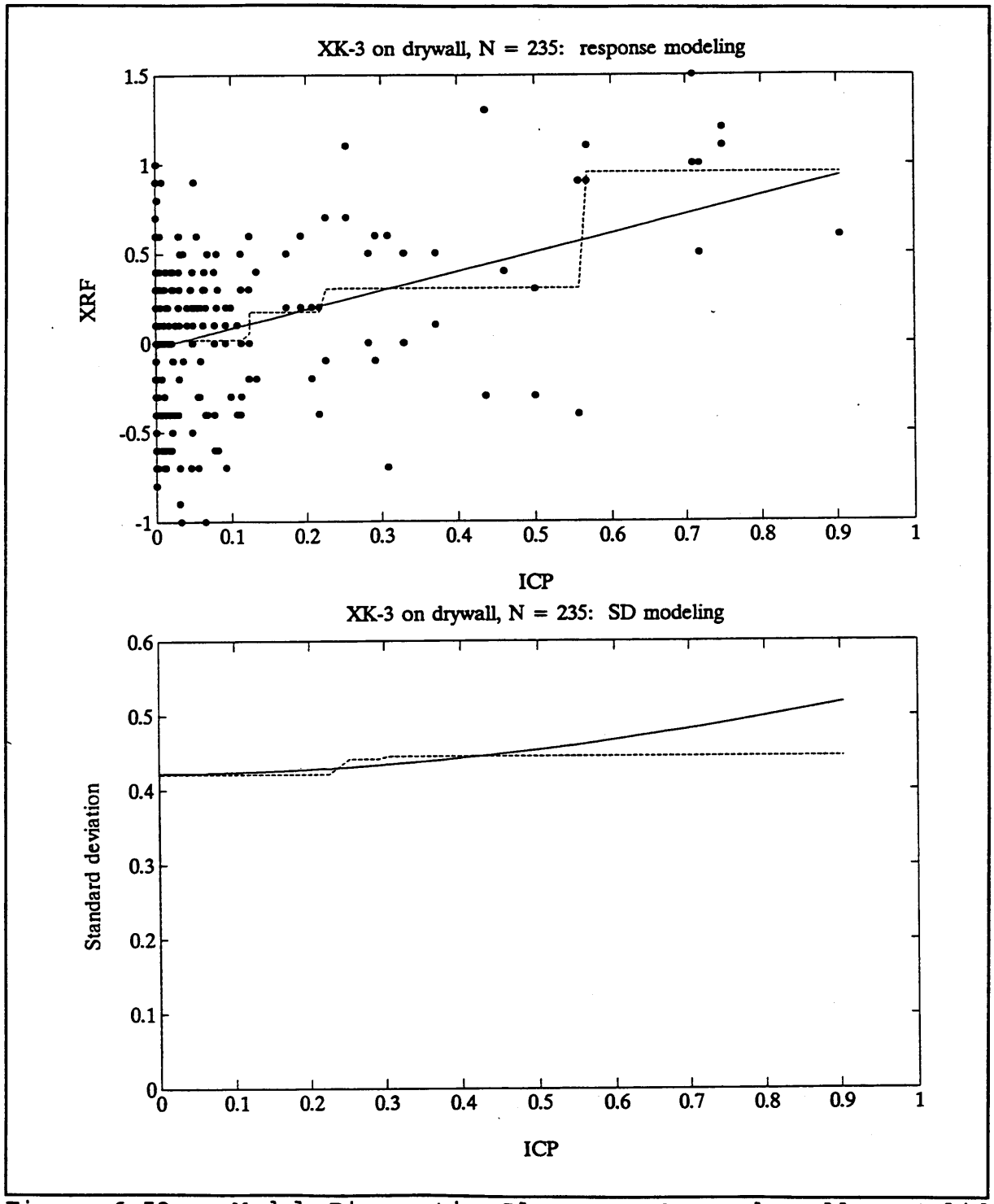


Figure 6-53. Model Diagnostic Plots, XK-3 on drywall. Solid lines are model estimates. Dashed lines are nonparametric (monotone regression) estimates.

Table 6-119. XK-3 on Drywall: Model Estimates.

DEVICE	SAMPLE SIZE	MODEL PARAMETERS				Pb=0.0 mg/cm²		Pb=1.0 mg/cm²	
		a	b	c	d	BIAS	SD	BIAS	SD
Machine 30	112	-0.327 (.040)	1.234 (.254)	0.127 (.019)	0.189 (.363)	-0.327 (.040)	0.356	-0.093 (.236)	0.562
Machine 31	104	0.245 (.025)	0.939 (.191)	0.043 (.007)	.257 (.186)	0.245 (.025)	0.206	0.184 (.180)	0.547

Table 6-120. XK-3 on Drywall: Control Block Summary.

DEVICE	SAMPLE SIZE	BARE (0.0 mg/cm²)		RED NIST SRM (1.02 mg/cm²)		YELLOW NIST SRM (3.53 mg/cm²)	
		BIAS	SD	BIAS	SD	BIAS	SD
Machine 30	58	-0.038 (.047)	0.359	0.233 (.062)	0.475	0.630 (.047)	0.360
Machine 31	35	0.397 (.031)	0.182	0.617 (.030)	0.180	0.919 (.062)	0.367
Machine 32	24	0.325 (.075)	0.367	0.880 (.129)	0.633	0.862 (.097)	0.477
Machines 30, 31, and 32	117	0.167 (.029)	0.319	0.481 (.042)	0.451	0.764 (.036)	0.389

data by machines, cities and operators was as follows:

Machine 30:	Operator M	187 total (61 D, 126 P)
Machine 31:	Operator L	90 total (62 D, 28 L)
Machine 32:	Operator M	16 total (all P)
	Operator N	111 total (all P)

Upper truncation of the XRF readings at 10.0 mg/cm^2 was observed at only 1 location, having an ICP measurement of 2.0723 mg/cm^2. Since this truncated reading was not unusual with respect to other (non-truncated) readings with similar ICP measurements, it was included in the analysis.

Matched pairs analysis: Several sign tests were performed on matched pairs arising from the field classifications:

(1) Machine 30 (Operator M) was compared to Machine 31 (Operator L) on 61 Denver sites of common measurement. Machine 30 gave a higher reading 14 times, with no ties. The sign test has a p-value of less than 0.01 percent, suggesting an effect where Machine 31 read higher than Machine 30.

(2) Machine 30 (Operator M) was compared to Machine 32 (Operator M) on 16 Philadelphia sites of common measurement. Machine 30 gave a higher reading 6 times, with no ties. This result is plausible under a hypothesis of no machine or operator effects.

(3) Machine 30 (Operator M) was compared to Machine 32 (Operator N) on 110 Philadelphia sites of common measurement. Machine 30 gave a higher reading only 12 times, with 1 tie. The sign test was highly significant, having a p-value of less than 0.01 percent, and it appears that Machine 32 read higher than Machine 30.

The possibility that the significant sign test obtained in (3) may be due to non-machine factors was explored with a Fisher's exact test. This was done by creating a contingency table with the results in (2) and (3), which refer to the same pair of machines, but different pairs of operators. The Fisher's exact test has a p-value of about 1.2 percent, which is small but not suggestive of a rare occurrence. This result does, however, give evidence that at least some of the apparent difference between Machines 30 and 32 may be due to the use of different

operators, or to differences in the painted samples themselves.

Figure 6-54 shows the response and SD components of the estimated model before provision for the combined effect of spatial variation and laboratory error in ICP measurements. There is agreement with the nonparametric response estimate for ICP measurements as high as 3.0 mg/cm^2, above which the nonparametric estimate suggests a flatter response. The SD estimates appear to be in close agreement up to that point as well.

Table 6-121 gives the results of fitting XRF measurement models to the data. The data were restricted to ICP measurements less than 5.0 mg/cm^2, which resulted in the exclusion of only 4 readings. Machine 30 was split by city (Denver and Philadelphia), and a significant difference can be seen in the estimated intercepts (0.661 Denver versus 0.264 Philadelphia). A chi-square test on the four model parameters gave a p-value that is less than 0.01 percent, mainly due to the intercepts. A similar split of Machine 31 into Denver and Louisville results likewise found a significant city effect, due once again mainly to the intercept terms. The operator was the same in both intercity comparisons. Comparing intercept estimates for Machines 30 versus 31 (Denver), and Machines 30 versus 32 (Philadelphia) corroborated the results of the matched pairs analysis in (1) and (3). The model was not fit to data from all machines combined, due to the incongruity in performance between machines.

Table 6-122 gives a summary of the control block data. Machine 30 once again was seen to read lower than Machines 31 and 32 at 0.0 mg/cm^2 and at 1.02 mg/cm^2, with the difference narrowing as the lead level increases. Machine 31 was also seen to have the lowest SD estimates on the control blocks, which was reflected in the model estimates. Machine 32 appeared to perform similarly in both measurement settings, but with a larger SD on painted samples, which was possibly due to non-instrumental sources of variability.

6.4.4.7.5 XK-3 on Plaster

There were 462 observations of the XK-3 on plaster, 1 of which was designated as an outlier (Machine 31: 80031), leaving 461 observations for analysis. Breakdown of the data by

machines, cities and operators was as follows:

Machine 30:	Operator M	221 total (100 D, 121 P)
Machine 31:	Operator L	119 total (99 D, 20 L)
Machine 32:	Operator M	18 total (all P)

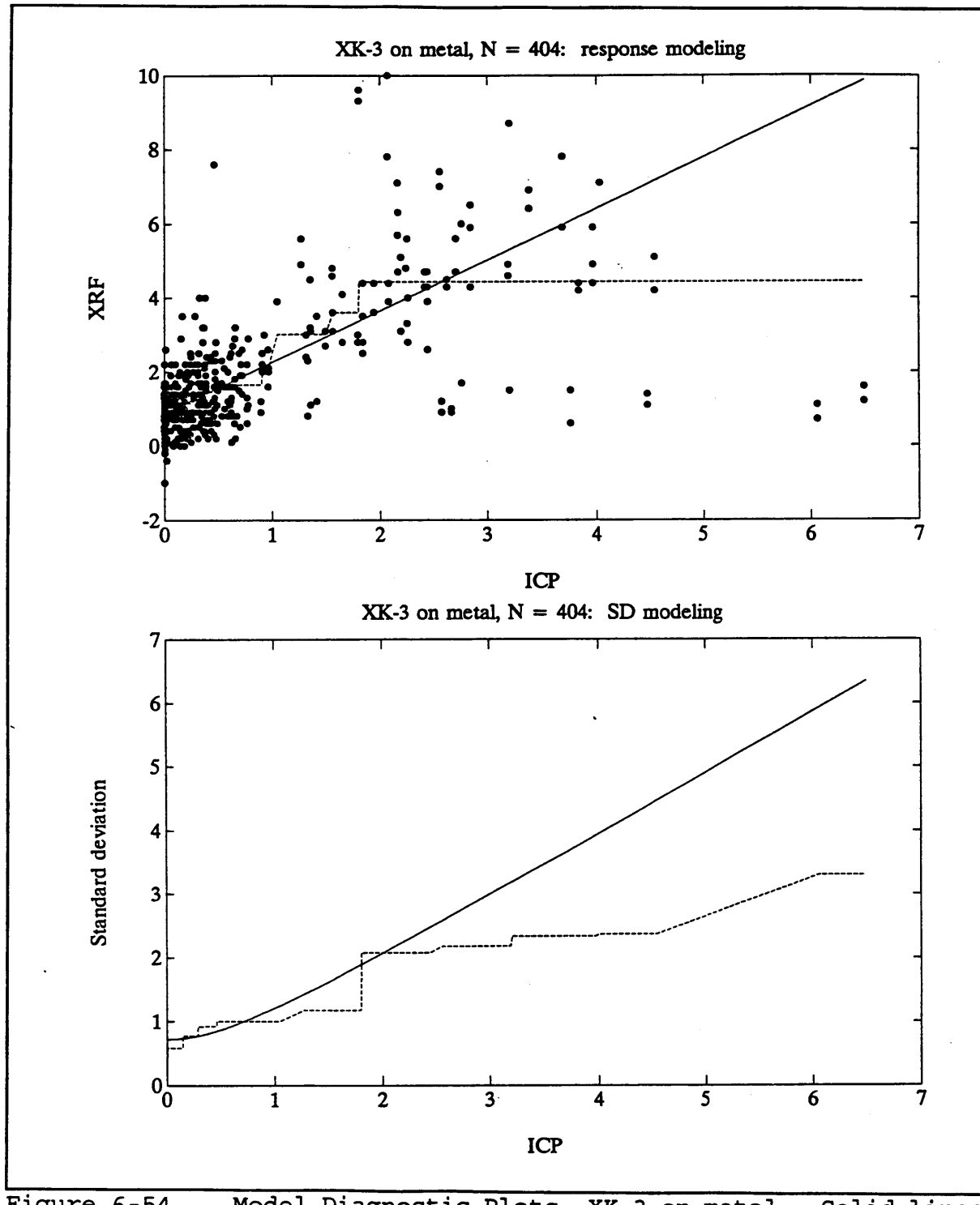


Figure 6-54. Model Diagnostic Plots, XK-3 on metal. Solid lines are model estimates. Dashed lines are nonparametric (monotone regression) estimates.

Table 6-121. XK-3 on Metal: Model Estimates.

DEVICE	SAMPLE SIZE	MODEL PARAMETERS				Pb=0.0 mg/cm²		Pb=1.0 mg/cm²	
		a	b	c	d	BIAS	SD	BIAS	SD
Machine 30, Denver (ICP < 5)	61	0.661 (.103)	1.648 (.397)	0.412 (.093)	2.234 (1.131)	0.661 (.103)	0.642	1.309 (.376)	1.627
Machine 30, Phila. (ICP < 5)	124	0.264 (.059)	1.545 (.148)	0.112 (.025)	0.750 (.173)	0.264 (.059)	0.335	0.809 (.118)	0.929
Machine 30, combined (ICP < 5)	185	0.451 (.058)	1.405 (.140)	0.267 (.037)	0.852 (.192)	0.451 (.058)	0.517	0.856 (.118)	1.058
Machine 31, Denver (ICP < 5)	62	1.090 (.057)	1.521 (.142)	0.135 (.029)	0.105 (.106)	1.090 (.057)	0.367	1.611 (.132)	0.490
Machine 31, Louisville (ICP<5)	28	0.505 (.188)	1.869 (.258)	0.484 (.148)	-0-	0.505 (.188)	0.696	1.374 (.194)	0.696
Machine 32, Phila. (ICP < 5)	125	1.480 (.089)	1.205 (.162)	0.368 (.069)	0.615 (.185)	1.480 (.089)	0.607	1.685 (.124)	0.992

Table 6-122. XK-3 on Metal: Control Block Summary.

DEVICE	SAMPLE SIZE	BARE (0.0 mg/cm²)		RED NIST SRM (1.02 mg/cm²)		YELLOW NIST SRM (3.53 mg/cm²)	
		BIAS	SD	BIAS	SD	BIAS	SD
Machine 30	73	0.792 (.053)	0.456	1.133 (.065)	0.557	1.971 (.082)	0.701
Machine 31	40	1.400 (.031)	0.196	1.523 (.037)	0.234	1.845 (.081)	0.515
Machine 32	33	1.451 (.059)	0.337	1.595 (.085)	0.487	1.876 (.114)	0.654
Machines 30, 31, and 32	146	1.099 (.031)	0.375	1.344 (.039)	0.474	1.915 (.053)	0.645

Operator N 103 total (all P)

Upper truncation of the XRF readings at 10.0 mg/cm^2 was observed at 13 locations, where the lowest ICP measurement was 8.7783 mg/cm^2.

Matched pairs analysis: Several sign tests were performed on matched pairs arising from the field classifications:

(1) Machine 30 (Operator M) was compared to Machine 31 (Operator L) on 99 Denver sites of common measurement. Machine 30 gave a higher reading 41 times, with 15 ties. The sign test does not suggest that a machine effect existed.

(2) Machine 30 (Operator M) was compared to Machine 32 (Operator M) on 18 Philadelphia sites of common measurement. Machine 30 gave a higher reading only 2 times, with no ties. The sign test has a p-value of about 0.07 percent, which suggests that an effect where Machine 32 read higher than Machine 30 may have existed.

(3) Machine 30 (Operator M) was compared to Machine 32 (Operator N) on 103 Philadelphia sites of common measurement. Machine 30 gave a higher reading only 9 times, with 2 ties. The sign test is highly significant, having a p-value of less than 0.01 percent, and it appears that Machine 32 read higher than Machine 30.

Results from (2) and (3) are very similar in spite of the different Machine 32 operators. A Fisher's exact test corroborated this, having a p-value that is close to 100 percent. Although this does not definitively rule out operator effects, it points to a machine effect as the more plausible explanation.

Figure 6-55 shows the response and SD components of the estimated model before provision for the combined effect of spatial variation and laboratory error in ICP measurements. The data were restricted to ICP measurements less than 10.0 mg/cm^2 to avoid truncated XRF measurements. Both model components appear to agree with the nonparametric estimates, except for divergence in the SD estimates above 2.0 mg/cm^2.

Table 6-123 gives the results of fitting XRF measurement models to the data. The small number of Machine 31 data from

Louisville were not used, in order to facilitate comparison of results obtained from Denver and Philadelphia data. Machine 30, split into Denver and Philadelphia, may have exhibited a city

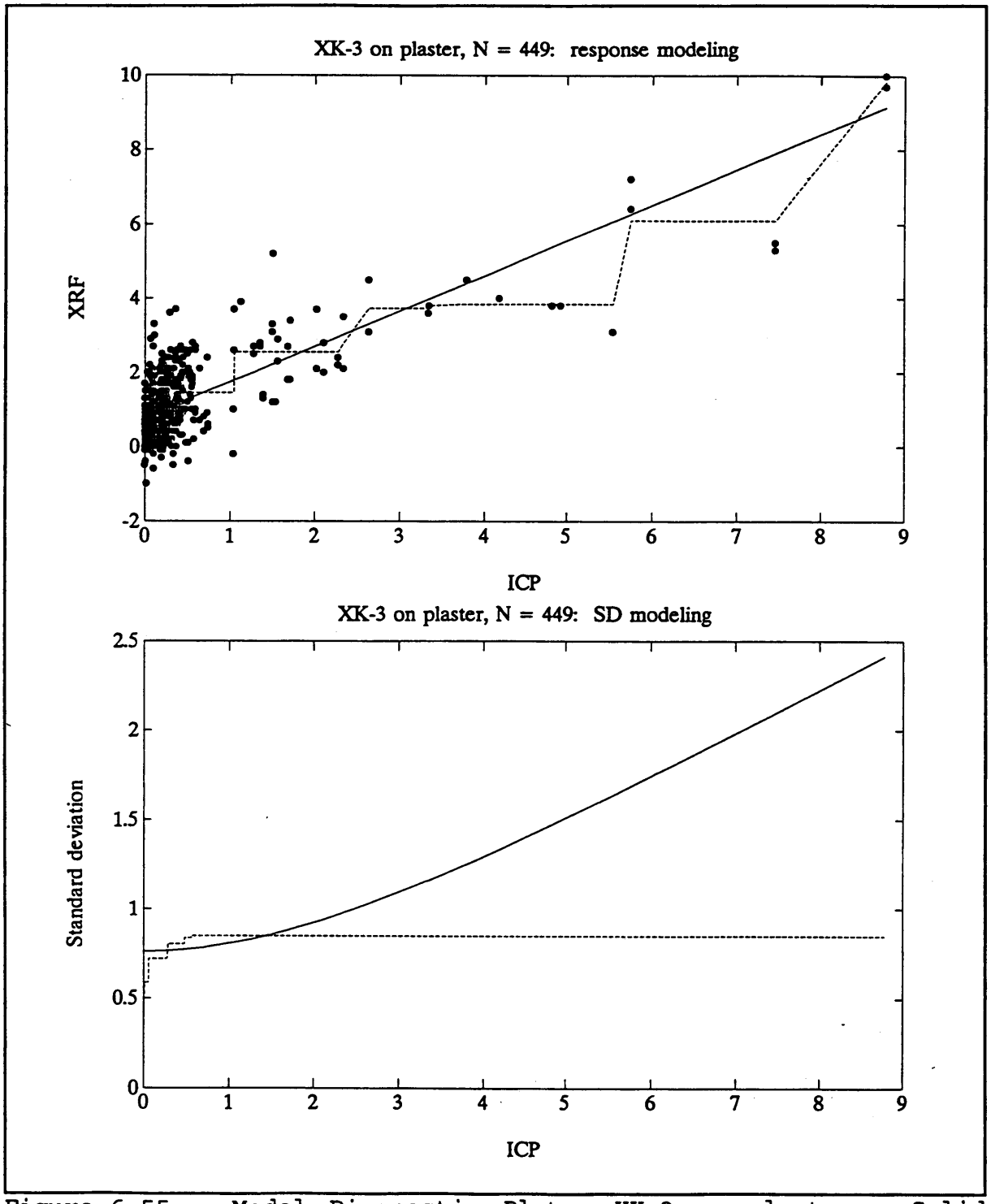


Figure 6-55. Model Diagnostic Plots, XK-3 on plaster. Solid lines are model estimates. Dashed lines are nonparametric (monotone regression) estimates.

Table 6-123. XK-3 on Plaster: Model Estimates.

DEVICE	SAMPLE SIZE	MODEL PARAMETERS				Pb=0.0 mg/cm²		Pb=1.0 mg/cm²	
		a	b	c	d	BIAS	SD	BIAS	SD
Machine 30, Denver (ICP < 10)	94	0.381 (.068)	0.797 (.239)	0.283 (.049)	0.374 (.319)	0.381 (.068)	0.532	0.179 (.212)	0.810
Machine 30, Phila. (ICP < 10)	121	0.729 (.065)	0.935 (.122)	0.244 (.037)	0.022 (.066)	0.729 (.065)	0.494	0.663 (.088)	0.516
Machine 30, combined (ICP < 10)	215	0.535 (.049)	1.035 (.112)	0.298 (.035)	0.103 (.098)	0.538 (.049)	0.546	0.571 (.091)	0.633
Machine 31, Denver (ICP < 10)	93	0.382 (.046)	0.835 (.078)	0.163 (.025)	-0-	0.382 (.046)	0.404	0.217 (.076)	0.404
Machine 32, Phila. (ICP < 10)	121	1.675 (.075)	0.952 (.164)	0.272 (.041)	0.141 (.098)	1.675 (.075)	0.521	1.627 (.120)	0.645

effect, seen mainly in the intercept estimates (0.381 Denver, 0.729 Philadelphia), for which the difference is highly statistically significant. The high intercept for Machine 32 appears to explain the results of the sign tests indicated in (2) and (3) above. The SD estimates are similar across machines at both lead levels indicated. The model was not fit to data combined across machines because of the different intercept estimates.

Table 6-124 gives a summary of the control block data. Once again, Machine 32 stands out as reading much higher than Machines 30 and 31. With the exception of Machine 30, the SD estimates are a little lower in the control block data summary than in the model estimates.

6.4.4.7.6 XK-3 on Wood

There were 743 observations of the XK-3 on wood, 1 of which was designated as an outlier (Machine 30: 80030), leaving 742 observations for analysis. Breakdown of the data by machines, cities and operators was as follows:

Machine 30:	Operator M	354 total (302 D, 52 P)
Machine 31:	Operator L	336 total (303 D, 33 L)
Machine 32:	Operator M	4 total (all P)
	Operator N	48 total (all P)

Upper truncation of the XRF readings at 10.0 mg/cm^2 was observed at 30 locations, where the lowest ICP measurement was 8.2867 mg/cm^2.

<u>Matched pairs analysis</u>: Several sign tests were performed on matched pairs arising from the field classifications:

(1) Machine 30 (Operator M) was compared to Machine 31 (Operator L) on 302 Denver sites of common measurement. Machine 30 gave a higher reading only 52 times, with 28 ties. This result is highly significant under a sign test (the p-value is nearly zero), suggesting that Machine 30 read lower than Machine 31.

(2) Machine 30 (Operator M) was compared to Machine 32 (Operator M) on 4 Philadelphia sites of common measurement. Machine 30 gave a higher reading 2 times, with no ties, which is a

very typical result in the absence of a machine effect, but with too small a sample size to make a firm conclusion.

Table 6-124. XK-3 on Plaster: Control Block Summary.

DEVICE	SAMPLE SIZE	BARE (0.0 mg/cm²)		RED NIST SRM (1.02 mg/cm²)		YELLOW NIST SRM (3.53 mg/cm²)	
		BIAS	SD	BIAS	SD	BIAS	SD
Machine 30	64	0.784 (.073)	0.584	0.685 (.110)	0.881	0.512 (.064)	0.510
Machine 31	32	0.519 (.043)	0.244	0.589 (.055)	0.309	0.495 (.054)	0.305
Machine 32	32	1.247 (.086)	0.489	1.368 (.095)	0.538	1.470 (.121)	0.683
Machines 30, 31, and 32	128	0.834 (.044)	0.496	0.832 (.062)	0.698	0.747 (.046)	0.519

(3) Machine 30 (Operator M) was compared to Machine 32 (Operator N) on 48 Philadelphia sites of common measurement. Machine 30 gave a higher reading only 5 times, with 1 tie. The sign test is highly significant, having a p-value of less than 0.01 percent, and it appears that Machine 32 read higher than Machine 30.

Figure 6-56 shows the response and SD components of the estimated model before provision for the combined effect of spatial variation and laboratory error in ICP measurements. A restriction of the data to ICP measurements less than 10.0 mg/cm^2 was enforced to avoid truncated XRF measurements. The model and nonparametric estimates show close agreement at lower ICP levels, with a flattening of the response at higher lead levels suggested by the nonparametric estimate.

Table 6-125 gives the results of fitting XRF measurement models to the data. It is noteworthy that city effects do not appear in the intercept (parameter *a*) estimates as they did on other substrates. This is true both for Machine 30 (Denver and Philadelphia) and Machine 31 (Denver and Louisville). Slope (parameter *b*) estimates greater than 1.0 are consistently indicated, but the over-responsiveness to changes in the lead level that this might suggest should be weighed in light of the flattening of the response indicated by the nonparametric estimate in Figure 6-56. The model was not fit to data combined across machines, because of the presence of substantial machine effects. The bias estimates for Machine 30 are lower than those for Machines 31 and 32, which corroborates the sign test results reported in (1) and (3).

Table 6-126 gives a summary of the control block data. Machines 31 and 32 were both found to read higher than Machine 30, but the effect was much smaller than that seen on other substrates. Still, the differences in bias estimates are significant, and consistent with conclusions drawn from both the sign tests and the modeling exercises. All three instruments showed progressively increasing bias on the control blocks as the lead level increased. The SD estimates in the control block summary also tend to increase with the lead level, which was corroborated on painted samples as well. The SD estimates obtained from the model are substantially larger, most notably for Machine 32 at the 1.0 mg/cm^2 lead level. Machines 30 and 31 exhibited agreement in performance on the control blocks and on

painted samples with respect to bias.

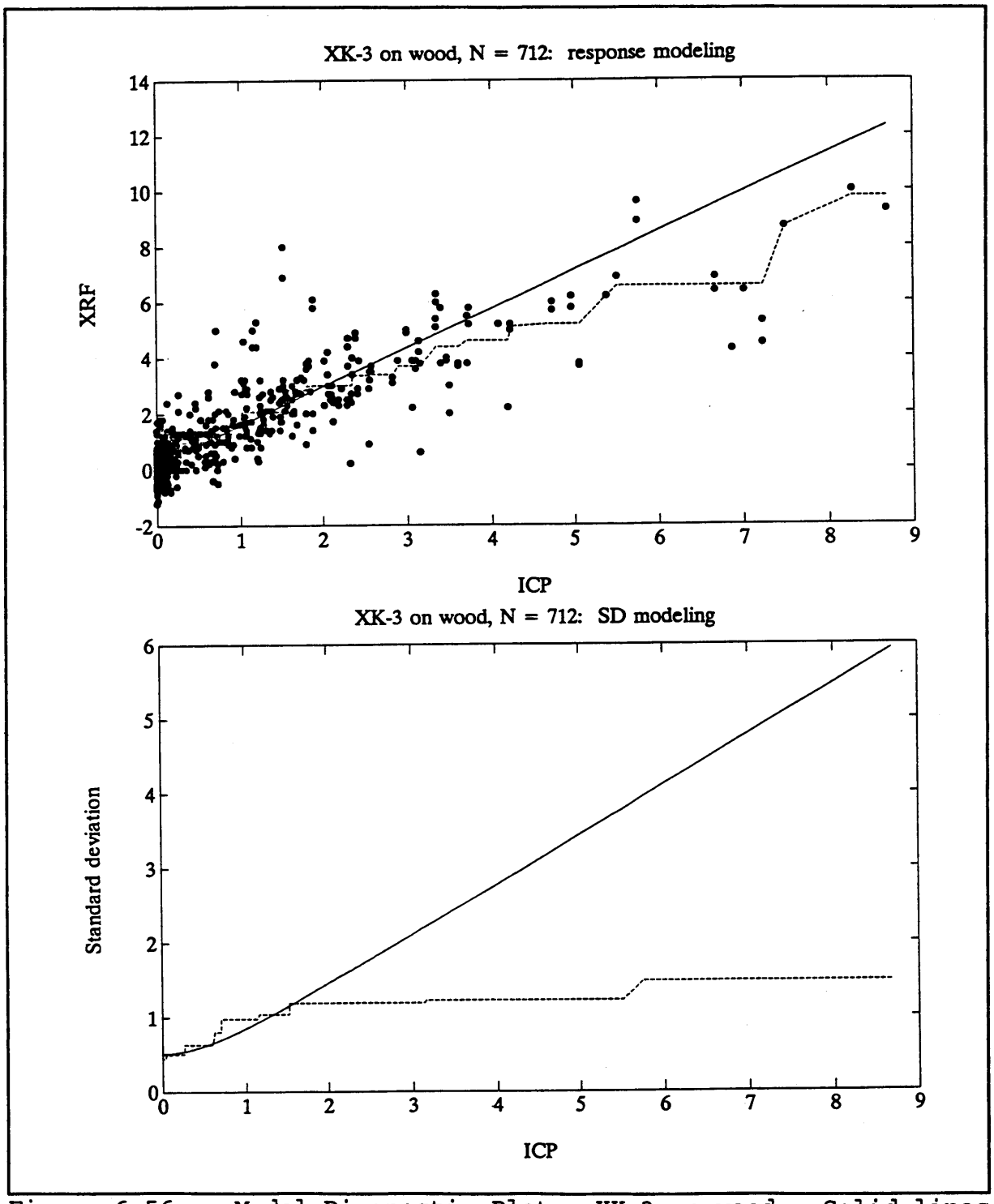


Figure 6-56. Model Diagnostic Plots, XK-3 on wood. Solid lines are model estimates. Dashed lines are nonparametric (monotone regression) estimates.

Table 6-125. XK-3 on Wood: Model Estimates.

DEVICE	SAMPLE SIZE	MODEL PARAMETERS				Pb=0.0 mg/cm²		Pb=1.0 mg/cm²	
		a	b	c	d	BIAS	SD	BIAS	SD
Machine 30, Denver (ICP < 10)	290	-0.061 (.038)	1.366 (.074)	0.264 (.028)	0.177 (.060)	-0.061 (.038)	0.513	0.305 (.067)	0.664
Machine 30, Phila. (ICP < 10)	52	0.021 (.072)	1.369 (.163)	0.062 (.025)	0.542 (.177)	0.021 (.072)	0.249	0.389 (.135)	0.777
Machine 30, combined (ICP < 10)	342	-0.065 (.035)	1.418 (.073)	0.236 (.024)	0.235 (.064)	-0.065 (.035)	0.486	0.352 (.064)	0.686
Machine 31, Denver (ICP < 10)	291	0.339 (.024)	1.426 (.069)	0.098 (.013)	0.099 (.058)	0.339 (.024)	0.313	0.765 (.062)	0.444
Machine 31, Louisville (ICP < 10)	27	0.326 (.128)	1.036 (.096)	0.193 (.083)	0.046 (.040)	0.326 (.128)	0.439	0.362 (.121)	0.489
Machine 32, Phila. (ICP < 10)	52	0.933 (.135)	1.294 (.249)	0.238 (.093)	1.090 (.377)	0.933 (.135)	0.488	1.227 (.197)	1.152

Table 6-126. XK-3 on Wood: Control Block Summary.

DEVICE	SAMPLE SIZE	BARE (0.0 mg/cm²)		RED NIST SRM (1.02 mg/cm²)		YELLOW NIST SRM (3.53 mg/cm²)	
		BIAS	SD	BIAS	SD	BIAS	SD
Machine 30	70	0.114 (.059)	0.492	0.281 (.061)	0.508	0.840 (.084)	0.704
Machine 31	38	0.426 (.031)	0.188	0.627 (.035)	0.213	1.049 (.071)	0.438
Machine 32	33	0.315 (.053)	0.307	0.759 (.072)	0.413	0.822 (.109)	0.626
Machines 30, 31, and 32	141	0.245 (.033)	0.391	0.486 (.036)	0.425	0.892 (.053)	0.625

6.4.4.7.7 XK-3: Summary of Analysis

The XK-3 resembled the other K-shell instruments used in the study, in that bias was indicated in the intercept terms of the models. Slope estimates greater than 1.0 were often obtained, which may be due, in part, to the inability of the model to capture a flattening out of the response indicated by the nonparametric estimates on several substrates. The result is that bias estimates at the 1.0 mg/cm^2 lead level obtained with the model are typically larger than at 0.0 mg/cm^2, which is a pattern that appears in the control block estimates as well. Bias estimates varied substantially between machines, and possibly between operators or cities. City effects reflect, to a certain extent, different distributions of lead levels in paint, as well as other differences that may affect XRF instrument performance. The bias was usually positive across substrates and machines, which was reflected both in the estimated models and the control block data summary.

One effect that did appear to generalize across substrates is that Machine 32 read higher than the other two machines used in the full study. Machine 30 read higher than Machine 31 on brick and concrete, but the opposite was true on drywall, metal and wood. The ordering of machines with respect to bias is consistently reflected in the sign tests, model estimates, and control block summary statistics. The XK-3 was distinguished from the other instruments in its similarity between the control block and painted sample data in this respect.

6.4.4.8 Results for XL

The XL is an L-shell instrument that was used in the full, but not the pilot study. Three different machines were used by 2 different operators, as indicated below:

Machine 40 (Operator J)	Denver
Machine 41 (Operators K and J)	Denver
Machine 42 (Operator J)	Philadelphia

The XL truncated its measurements on the lower end at 0.0 mg/cm^2, so that it did not give negative readings. It also truncated on the higher end at 5.0 mg/cm^2. Since XL readings had only one decimal place, it was not unusual for XL readings to have a predominance of zeros at lower lead levels.

The XRF measurement model did not effectively describe certain aspects of the performance of the XL. The model did not give reliable estimates of bias at 1.0 mg/cm^2, which may reflect a change in the responsiveness of the instrument near that lead level. For this reason, the estimates of bias at 1.0 mg/cm^2 presented in the tables for all substrates, except drywall, are nonparametric estimates, based on monotone regression. Nonparametric estimates for drywall are not reported, because the lack of ICP readings at or above 1.0 mg/cm^2 did not allow reliable estimates to be obtained. Although monotone regression did not account for spatial variation and laboratory error in ICP measurements, the effect of this is believed to be small on the bias estimates. The nonparametric bias estimates were therefore judged to be better indicators of instrument performance at a lead level of 1.0 mg/cm^2 than those obtained from the model. Standard error estimates reported for the bias at 1.0 mg/cm^2 were obtained using the bootstrap technique. The SD estimates reported at both 0.0 mg/cm^2 and 1.0 mg/cm^2, however, were derived from the model.

The outlier analysis of section 6.3 excluded the XL, because its truncation made it difficult to apply the same methodology for defining outliers used with the other instruments.

6.4.4.8.1 XL on Brick

There were 93 observations of the XL on brick. They were broken down by machine and operator as indicated below:

Machine 40:	Operator J	41 total (all Denver)
Machine 41:	Operator K	21 total (all Denver)
	Operator J	19 total (all Denver)
Machine 42:	Operator J	12 total (all Philadelphia)

Lower truncation at 0.0 mg/cm^2 occurred 17 times, the highest ICP measurement of which was 0.1942 mg/cm^2. Upper truncation at 5.0 mg/cm^2 occurred 3 times, the lowest ICP measurement of which was 27.206 mg/cm^2.

Figure 6-57 shows the response and SD components of the estimated model before provision for the combined effect of spatial variation and laboratory error in ICP measurements. The data were restricted to ICP measurements less than 25.0 mg/cm^2 to avoid problems with upper truncation, which reduced the sample

size to 90. While the agreement between the model and nonparametric estimates appears to be good, the nonparametric estimate suggests much less bias near ICP = 1.0 mg/cm^2 than the model. This may be near a point of transition in the response function of the instrument.

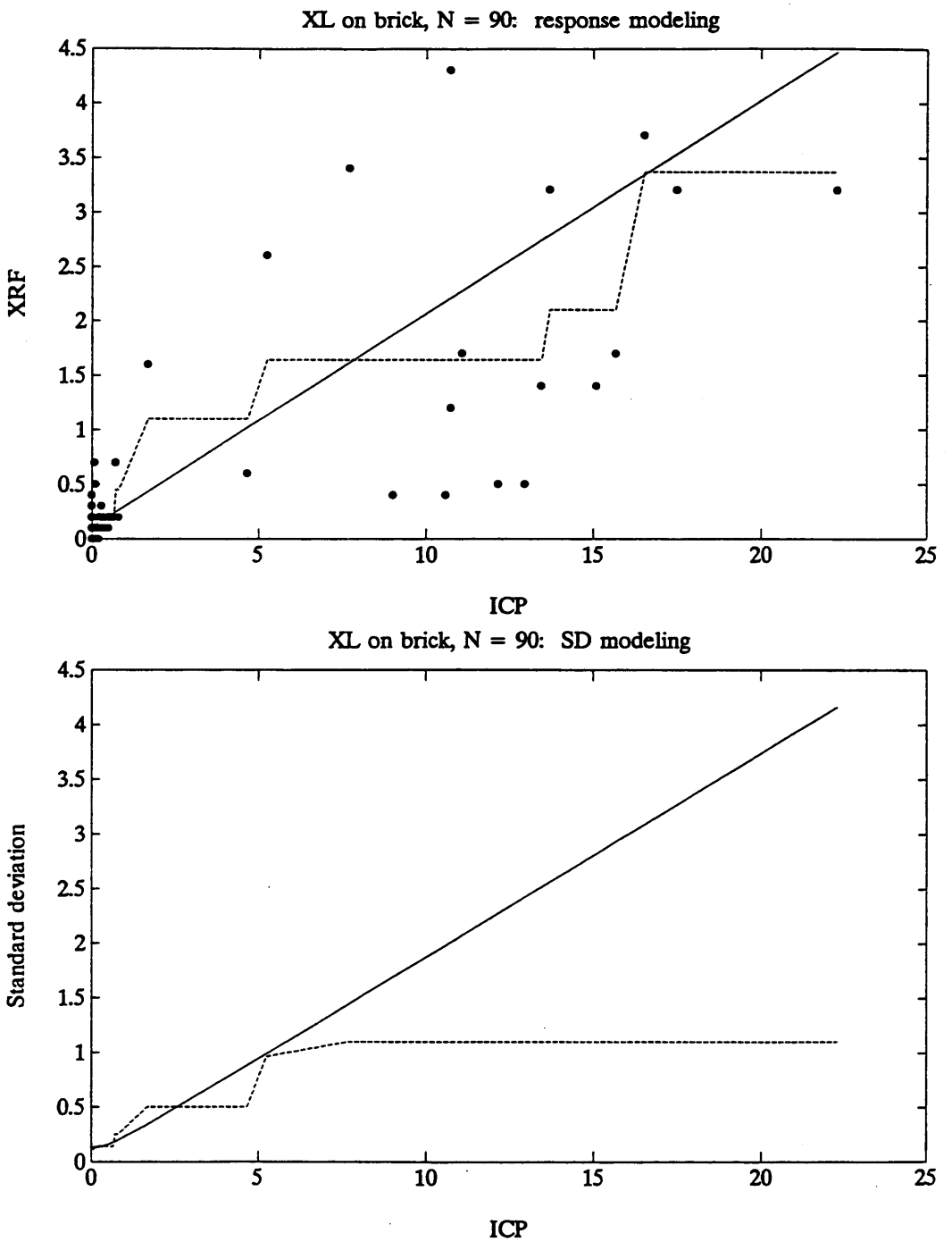


Figure 6-57. Model Diagnostic Plots, XL on brick. Solid lines are model estimates. Dashed lines are nonparametric (monotone regression) estimates.

It would not, however, appear correct to infer that unbiasedness was a characteristic of the instrument on brick, as readings below 1.0 mg/cm^2 were observed on 3 of the 16 samples where the ICP measurement exceeded 10.0 mg/cm^2. All 16 of these readings were made by Operator K, using Machine 41, at the same address in Denver.

Table 6-127 gives the results of fitting XRF measurement models to the data, and nonparametric estimates of the bias at the 1.0 mg/cm^2 lead level. The model was not fit to Machine 40 data separately, because the largest corresponding ICP measurement was only 0.8042 mg/cm^2, and the readings only assumed 5 distinct values. A separate model was not fit to Machine 42, because only 12 readings were available. The slope estimates (parameter *b*) suggest a responsiveness on the order of about one fifth to changes in the lead level, which although typical of L-shell instruments, may be misleading except at low lead levels.

The nonparametric bias estimate for Machine 41 at 1.0 mg/cm^2 was -0.337 mg/cm^2, which is smaller in magnitude than the model would imply (-.748 mg/cm^2). The standard error of the nonparametric estimate (0.188 mg/cm^2) is large relative to the magnitude of the estimate, which is due to the effect of the jump in the nonparametric response function near the 1.0 mg/cm^2 ICP level. The instrument may have been more responsive to lead at 1.0 mg/cm^2 than indicated by the model, but the responsiveness flattened out at higher lead levels.

Table 6-128 gives a summary of the control block data. Differences between machines are evident in the bias estimates. Machine 42 had very small, positive bias on the control blocks at the three lead levels, but the bias was negative and substantial at 3.53 mg/cm^2 for Machines 40 and 41. Since the same operator (J) used Machines 40 and 42 in this controlled measurement setting, the different bias estimates point to a machine effect. The nonparametric estimates suggest much greater negative bias at 1.0 mg/cm^2 using the instrument on painted samples than on the control blocks.

Biases exhibited on the control blocks at 0.0 mg/cm^2 were consistent with readings obtained on painted samples with respect to the frequency of zero readings at low lead levels. At sample locations with ICP measurements less than 0.1 mg/cm^2, only 4 out of 24 readings with Machine 40 were zeros, compared to 6 out of

10 with Machine 41, and 4 out of 8 with Machine 42. The lower frequency of zero readings with Machine 40 corresponds to the higher bias estimate obtained for this machine on the control

Table 6-127. XL on Brick: Model Estimates: Model Estimates.

DEVICE	SAMPLE SIZE	MODEL PARAMETERS				Pb=0.0 mg/cm²		Pb=1.0 mg/cm²	
		a	b	c	d	BIAS	SD	BIAS[a]	SD
Machine 41 (ICP < 25)	37	0.038 (.016)	0.214 (.041)	0.003 (.002)	.025 (.012)	0.038 (.016)	0.056	-0.337 (.188)	0.167
Machines 40, 41, 42 (ICP < 25)	90	0.109 (.016)	0.183 (.033)	0.016 (.003)	0.018 (.008)	0.109 (.016)	0.126	-0.403 (.219)	0.183

[a] Nonparametric estimates reported. Standard error estimates obtained by bootstrapping.

Table 6-128. XL on Brick: Control Block Summary.

DEVICE	SAMPLE SIZE	BARE (0.0 mg/cm²)		RED NIST SRM (1.02 mg/cm²)		YELLOW NIST SRM (3.53 mg/cm²)	
		BIAS	SD	BIAS	SD	BIAS	SD
Machine 40	14	0.293 (.041)	0.154	0.044 (.025)	0.093	-0.501 (.088)	0.329
Machine 41	17	0.100 (.024)	0.100	-0.085 (.031)	0.127	-0.401 (.143)	0.588
Machine 42	30	0.070 (.014)	0.075	0.097 (.008)	0.046	0.047 (.059)	0.321
Machines 40, 41, and 42	61	0.130 (.013)	0.104	0.034 (.011)	0.086	-0.204 (.053)	0.414

blocks at 0.0 mg/cm^2.

6.4.4.8.2 XL on Concrete

There were 217 observations of the XL on concrete. They were broken down by machine and operator as indicated below:

Machine 40:	Operator J	23 total (all Denver)
Machine 41:	Operator K	25 total (all Denver)
	Operator J	49 total (all Denver)
Machine 42:	Operator J	120 total (all Philadelphia)

Lower truncation at 0.0 mg/cm^2 occurred 50 times, the highest ICP measurement of which was 0.3572 mg/cm^2. Upper truncation at 5.0 mg/cm^2 occurred 1 time, with a corresponding ICP measurement of 13.4071 mg/cm^2.

Figure 6-58 shows the response and SD components of the estimated model before provision for the combined effect of spatial variation and laboratory error in ICP measurements. The data were restricted to ICP measurements less than 10.0 mg/cm^2 to avoid upper truncation, leaving 213 observations for analysis (there were no samples with ICP measurements between 8.0 mg/cm^2 and 10.0 mg/cm^2). The nonparametric response flattens for ICP levels above 1.0 mg/cm^2. It also suggests that the bias near a lead level of 1.0 mg/cm^2 was lower than indicated by the model.

Table 6-129 gives the results of fitting XRF measurement models, and nonparametric estimates of the bias at 1.0 mg/cm^2. Like the results for brick, it is difficult to infer from the model the bias of the instrument at 1.0 mg/cm^2, because of a possible change in the response relationship near that level of lead. The nonparametric estimates of the bias at 1.0 mg/cm^2 are lower than those from the model, and appear to give the more accurate reflection of bias at that lead level.

The reported levels of bias are more characteristic of some of the K-shell instruments than the other L-shell instruments that were evaluated in the study. Like the other L-shell instruments, however, the response of the XL flattens out at higher lead levels, with low readings frequently obtained on samples with high ICP measurements. Of the 16 samples with ICP measurements greater than 3.0 mg/cm^2, 6 had readings below 1.0 mg/cm^2 with the XL, although none of these occurred at ICP levels

greater than 5.0 mg/cm^2.

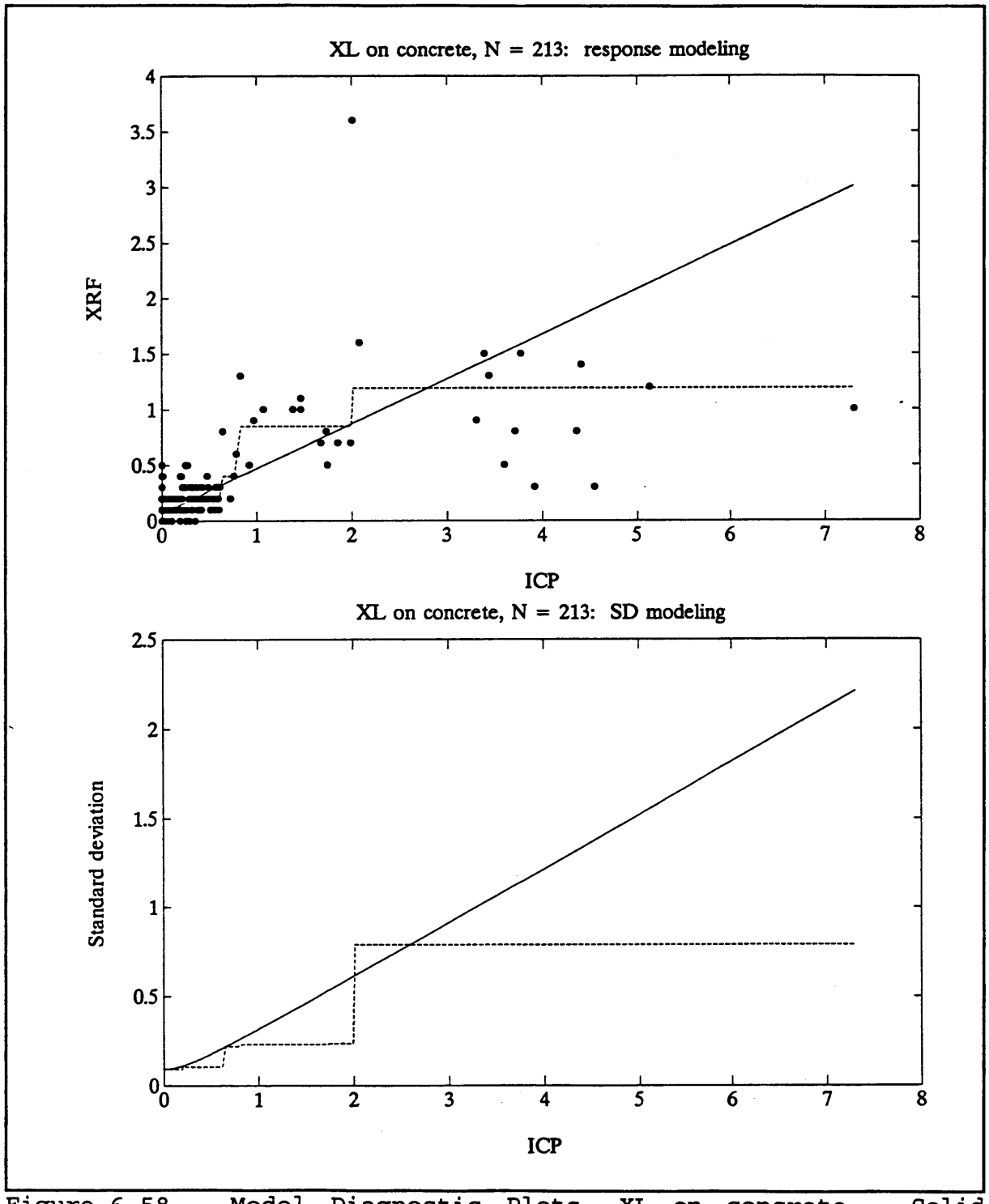


Figure 6-58. Model Diagnostic Plots, XL on concrete. Solid lines are model estimates. Dashed lines are nonparametric (monotone regression) estimates.

Table 6-129. XL on Concrete: Model Estimates.

DEVICE	SAMPLE SIZE	MODEL PARAMETERS				Pb=0.0 mg/cm²		Pb=1.0 mg/cm²	
		a	b	c	d	BIAS	SD	BIAS[a]	SD
Machine 41 (ICP < 10)	70	0.039 (.008)	0.493 (.080)	0.003 (.001)	0.095 (.042)	0.039 (.008)	0.050	-0.378 (.085)	0.312
Machine 42 (ICP < 10)	120	0.054 (.013)	0.376 (.044)	0.007 (.001)	0.038 (.011)	0.054 (.013)	0.083	-0.191 (.092)	0.211
Machines 40, 41, and 42 (ICP < 10)	213	0.066 (.009)	0.391 (.035)	0.008 (.001)	0.051 (.013)	0.066 (.009)	0.091	-0.150 (.068)	0.244

[a] Nonparametric estimates reported. Standard error estimates obtained by bootstrapping.

Table 6-130 gives a summary of the control block data. The estimated bias at 1.02 mg/cm^2 is small for all three machines, but for Machine 40 the bias is significant and positive at 0.0 mg/cm^2, and significant and negative at 3.53 mg/cm^2. Since Operator J used Machines 40 and 42 on the control blocks, the difference in bias estimates indicates a machine effect. Further evidence of this was seen on painted samples. None of the readings with Machine 40 were zeros, even on the 13 locations where the ICP measurement was less than 0.1 mg/cm^2. Machine 41 gave zero readings on 27 of 45, and Machine 42 on 18 of 32 locations where the ICP measurement was less than 0.1 mg/cm^2.

6.4.4.8.3 XL on Drywall

There were 113 observations of the XL on drywall. They were broken down by machine and operator as indicated below:

Machine 40:	Operator J	37 total (all Denver)
Machine 41:	Operator K	17 total (all Denver)
	Operator J	51 total (all Denver)
Machine 42:	Operator J	8 total (all Philadelphia)

Lower truncation at 0.0 mg/cm^2 occurred 53 times, the highest ICP measurement of which was 0.9049 mg/cm^2. Upper truncation at 5.0 mg/cm^2 was not observed, possibly due to the fact that none of the ICP measurements exceeded 1.0 mg/cm^2 on drywall.

Figure 6-59 shows the response and SD components of the estimated model before provision for the combined effect of spatial variation and laboratory error in ICP measurements. The model and nonparametric response estimates appear to be close, but given the limited ICP range of these data, it is hard to expect this agreement to persist at higher ICP measurements. Stratification of the XRF readings at values 0.1 apart is clearly evident in the scatterplot.

Table 6-131 gives the results of fitting XRF measurement models to the data. Reliable nonparametric estimates of the bias at 1.0 mg/cm^2 could not given due to the restricted ICP range. The same is true of the model, considering that on other substrates, disparity between model and nonparametric response estimates emerged at about the 1.0 mg/cm^2 lead level. Although the model estimates of the bias are given, they are mainly

indicative of the under-responsiveness of the instrument to lead levels below 1.0 mg/cm^2. Machine 40 does not have separate estimates because all but 2 of the corresponding ICP measurements

Table 6-130. XL on Concrete: Control Block Summary.

DEVICE	SAMPLE SIZE	BARE (0.0 mg/cm²)		RED NIST SRM (1.02 mg/cm²)		YELLOW NIST SRM (3.53 mg/cm²)	
		BIAS	SD	BIAS	SD	BIAS	SD
Machine 40	21	0.438 (.034)	0.156	0.018 (.020)	0.092	-0.640 (.092)	0.421
Machine 41	20	0.035 (.011)	0.049	-0.035 (.033)	0.150	-0.215 (.166)	0.742
Machine 42	35	0.040 (.011)	0.065	0.111 (.015)	0.090	0.076 (.044)	0.261
Machines 40, 41, and 42	76	0.149 (.011)	0.096	0.047 (.013)	0.109	-0.198 (.054)	0.473

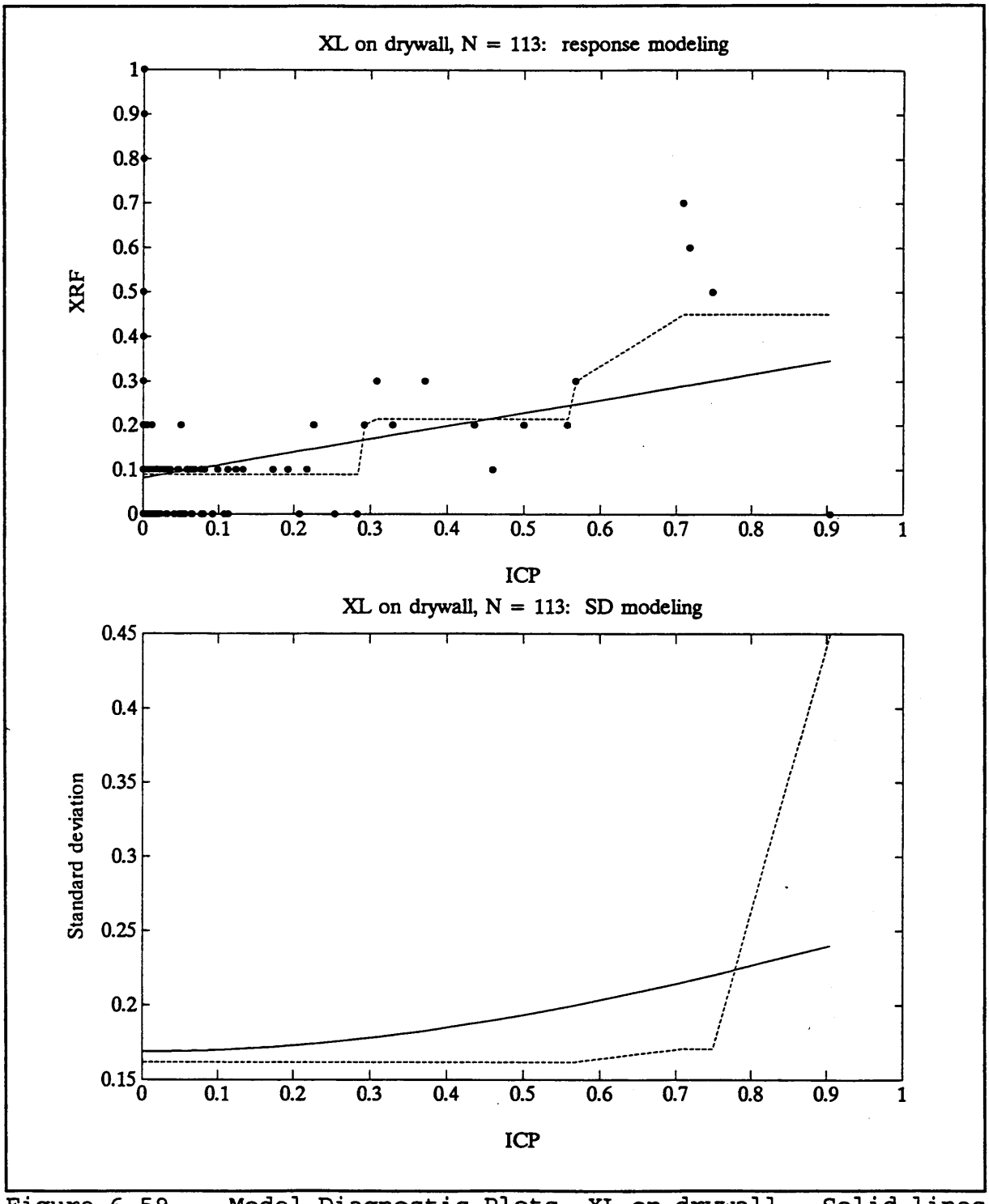


Figure 6-59. Model Diagnostic Plots, XL on drywall. Solid lines are model estimates. Dashed lines are nonparametric (monotone regression) estimates.

Table 6-131. XL on Drywall: Model Estimates.

DEVICE	SAMPLE SIZE	MODEL PARAMETERS				Pb=0.0 mg/cm²		Pb=1.0 mg/cm²	
		a	b	c	d	BIAS	SD	BIAS	SD
Machine 41	68	0.014 (.009)	0.423 (.083)	0.002 (.001)	0.068 (.031)	0.014 (.009)	0.049	-0.564 (.077)	0.266
Machines 40, 41, and 42	113	0.082 (.019)	0.289 (.109)	0.029 (.004)	0.027 (.035)	0.082 (.019)	0.169	-0.629 (.101)	0.238

were less than 0.06 mg/cm^2. There were too few data to give separate estimates for Machine 42.

Little bias is inferred for Machine 41 at 0.0 mg/cm^2 from the model, which is in contrast to the control block data summary given in Table 6-132. Machines 40 and 41 both exhibited large positive bias in the absence of lead on the control blocks. The large bias for Machine 40 at 0.0 mg/cm^2 (1.082) estimated from the control block data was not confirmed in the data from painted samples, but it was substantial nonetheless: the 21 readings made by Machine 40 on painted samples with ICP measurements less than 0.005 mg/cm^2 had an average of 0.238 mg/cm^2. The scatterplot in Figure 6-59 shows that a number of relatively high XRF readings, all made with Machine 40, were obtained at very low ICP levels. At 1.02 mg/cm^2 the control block data suggest that Machines 40 and 41 exhibited very little bias, and that the bias for both machines became substantially negative at 3.53 mg/cm^2. Machine 42, by contrast, exhibited bias of a small order that changed very little with the lead level. The different bias estimates for Machines 40 and 42 are noteworthy, because both machines were used by the same operator (J), indicating a possible machine effect.

The control block summary shows a pattern where the SD estimates are smaller at 1.02 mg/cm^2 than at 0.0 mg/cm^2. All three machines evaluated in the study exhibited this pattern. A similar pattern was also evident in the field sample data obtained with Machine 40. The 21 readings with Machine 40 on samples with ICP measurements less than 0.005 mg/cm^2 had an SD of 0.307 mg/cm^2, while the 16 readings on samples with ICP measurements between 0.005 mg/cm^2 and 0.5 mg/cm^2 had an SD of only 0.093. A similar analysis with Machines 41 and 42 was not conducted, because of the lack of data at low ICP levels. These patterns, present in both the control block and the field sample data, suggest that XL readings were more variable at very low lead levels that at somewhat higher levels.

With Machine 40, only 12 of the 35 field sample readings for ICP measurements less than 0.1 mg/cm^2 were zeros, compared to 30 of 43 with Machine 41. These results are consistent with the conclusion from the control block data that Machine 40 read systematically higher than Machine 41 at low lead levels.

6.4.4.8.4 XL on Metal

There were 189 observations of the XL on metal. To avoid problems with upper truncation, only the 187 observations with

Table 6-132. XL on Drywall: Control Block Summary.

DEVICE	SAMPLE SIZE	BARE (0.0 mg/cm²)		RED NIST SRM (1.02 mg/cm²)		YELLOW NIST SRM (3.53 mg/cm²)	
		BIAS	SD	BIAS	SD	BIAS	SD
Machine 40	17	1.082 (.131)	0.539	-0.002 (.023)	0.095	-0.642 (.101)	0.415
Machine 41	19	0.484 (.139)	0.604	-0.073 (.039)	0.171	-0.583 (.160)	0.698
Machine 42	26	0.154 (.052)	0.263	0.118 (.010)	0.050	0.124 (.050)	0.257
Machines 40, 41, and 42	62	0.510 (.059)	0.468	0.027 (.014)	0.112	-0.303 (.060)	0.473

ICP measurements less than 5.0 mg/cm^2 were used in the analysis. They were broken down by machine and operator as follows:

Machine 40:	Operator J	28	total (all Denver)
Machine 41:	Operator K	18	total (all Denver)
	Operator J	16	total (all Denver)
Machine 42:	Operator J	125	total (all Philadelphia)

Lower truncation at 0.0 mg/cm^2 occurred 32 times, the highest ICP measurement of which was 2.0836 mg/cm^2. Upper truncation at 5.0 mg/cm^2 occurred 3 times, the lowest ICP measurement of which was 4.4758 mg/cm^2.

Figure 6-60 shows the response and SD components of the estimated model before provision for the combined effect of spatial variation and laboratory error in ICP measurements. Although the model and nonparametric response estimates appear to be close, the nonparametric estimate has a sharp increase near 1.0 mg/cm^2. The instrument was possibly not as biased at that lead level as the model would indicate. The scatterplot itself suggests that the instrument more frequently gave higher readings when the lead level exceeded 1.0 mg/cm^2. Readings less than 1.0 mg/cm^2 occurred on only 2 of 16 field samples where the ICP measurement was greater than 3.0 mg/cm^2.

Table 6-133 gives the results of fitting XRF measurement models, and nonparametric bias estimates at the 1.0 mg/cm^2 lead level. The higher intercept for Machine 40 (0.163) is consistent with the finding on other substrates that this machine read higher than the others at low lead levels. Differences in the slope parameters *b* may reflect the inability of the model to capture the transition in instrument performance at higher lead levels. The nonparametric bias estimates at 1.0 mg/cm^2, however, are not substantially different from those obtained from the model for Machine 40 (-.481 mg/cm^2) and Machine 41 (-.625 mg/cm^2). Machine 42 reveals the greatest discrepancy between the model (-.331 mg/cm^2) and nonparametric (.517 mg/cm^2) estimates. The estimated standard error (.294) of the nonparametric estimate also is large, which reflects the apparent jump in the nonparametric response function near the 1.0 mg/cm^2 lead level.

Table 6-134 presents the control block data summary. The only prominent machine effect is the large negative bias at 3.53 mg/cm^2 estimated for Machine 40. Since Operator J used both

Machines 40 and 42, the difference in bias estimates at this lead level points to a machine effect. Machine 40 had the largest bias at 0.0 mg/cm^2 across machines on both the control blocks and

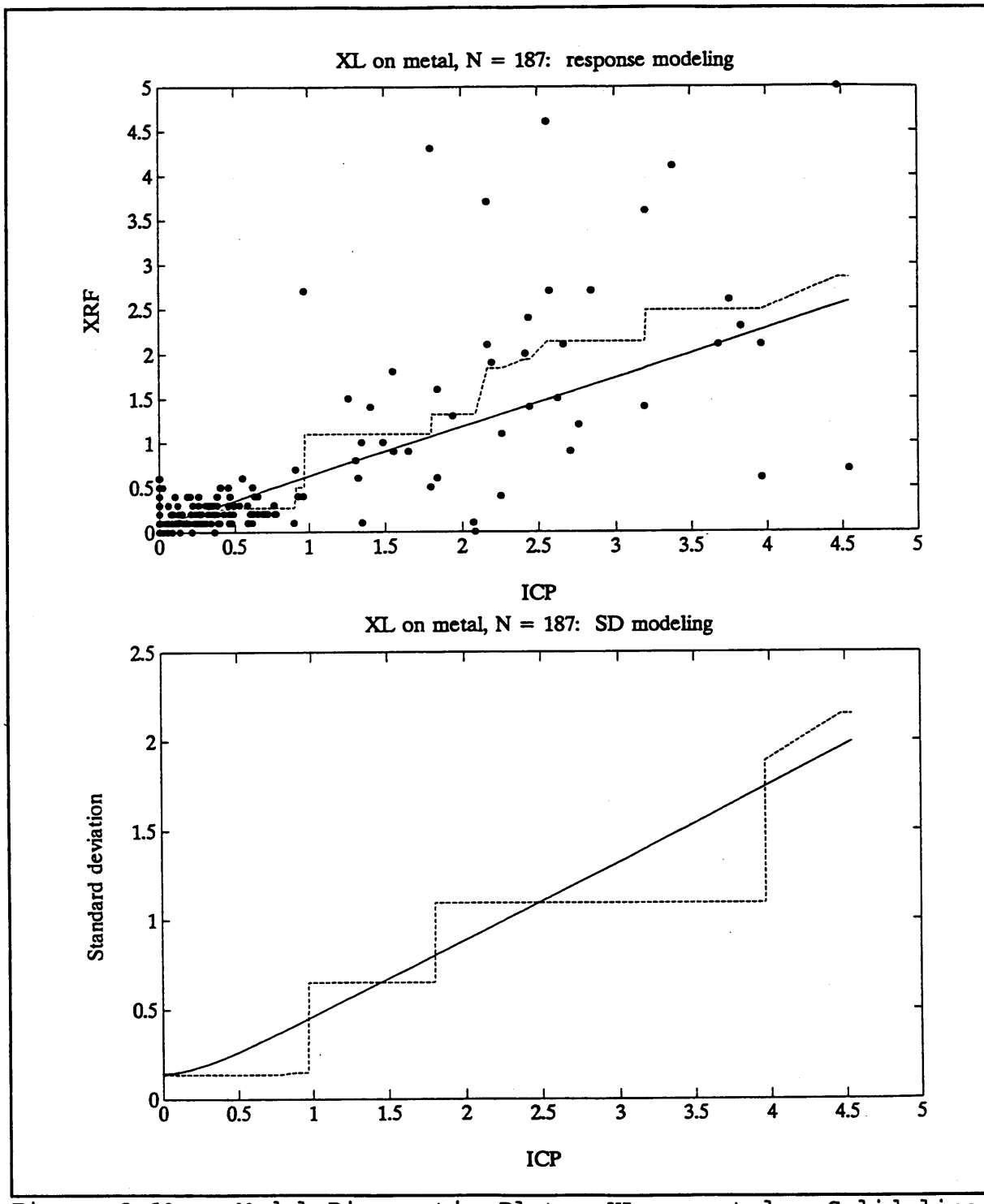


Figure 6-60. Model Diagnostic Plots, XL on metal. Solid lines are model estimates. Dashed lines are nonparametric (monotone regression) estimates.

Table 6-133. XL on Metal: Model Estimates.

DEVICE	SAMPLE SIZE	MODEL PARAMETERS				Pb=0.0 mg/cm²		Pb=1.0 mg/cm²	
		a	b	c	d	BIAS	SD	BIAS[a]	SD
Machine 40 (ICP < 5)	28	0.163 (.044)	0.357 (.104)	0.038 (.012)	0.040 (.032)	0.163 (.044)	0.196	-0.480 (.131)	0.280
Machine 41 (ICP < 5)	34	0.031 (.032)	0.345 (.048)	0.025 (.008)	-0-	0.031 (.032)	0.158	-0.623 (.070)	0.158
Machine 42 (ICP < 5)	125	0.054 (.024)	0.616 (.064)	0.015 (.003)	0.141 (.029)	0.054 (.024)	0.124	0.517 (.294)	0.396
Machines 40, 41, and 42 (ICP < 5)	187	0.074 (.017)	0.546 (.050)	0.020 (.003)	0.132 (.025)	0.074 (.017)	0.141	-0.100 (.230)	0.389

[a] Nonparametric estimates reported. Standard error estimates obtained by bootstrapping.

Table 6-134. XL on Metal: Control Block Summary.

DEVICE	SAMPLE SIZE	BARE (0.0 mg/cm²)		RED NIST SRM (1.02 mg/cm²)		YELLOW NIST SRM (3.53 mg/cm²)	
		BIAS	SD	BIAS	SD	BIAS	SD
Machine 40	20	0.055 (.011)	0.051	0.035 (.017)	0.076	-0.515 (.100)	0.449
Machine 41	25	0.004 (.004)	0.020	-0.044 (.036)	0.181	-0.166 (.137)	0.686
Machine 42	41	0.020 (.006)	0.040	0.085 (.010)	0.063	0.111 (.037)	0.238
Machines 40, 41, and 42	86	0.023 (.004)	0.039	0.036 (.012)	0.113	-0.115 (.049)	0.457

in the model estimates, although the magnitude of the bias was small in both cases.

A closer look at the field sample data would also anticipate a higher Machine 40 bias at low lead levels. Only 6 of the 18 Machine 40 readings at ICP measurements less than 0.1 mg/cm^2 were zeros, compared to 16 out of 20 for Machine 41. The average of the 18 Machine 40 readings was 0.178, which is higher than the control block estimate of the bias at 0.0 mg/cm^2, but consistent with the model estimate.

For all three machines, the control block summary shows little bias at the 0.0 mg/cm^2 and 1.02 mg/cm^2 lead levels, and SD estimates that increase with the lead level. While this pattern is typical for other instruments evaluated in the study, it is in contrast to the pattern exhibited by the XL on drywall, plaster, and less uniformly on brick and concrete, where the SD (and bias) actually was smaller at 1.02 mg/cm^2 than at 0.0 mg/cm^2. The field sample data for Machine 40, however, did give evidence of the pattern seen on other substrates: the 10 readings at samples with ICP measurements less than 0.005 mg/cm^2 had an SD of 0.245 mg/cm^2, while the 11 readings at samples with ICP measurements between 0.005 mg/cm^2 and 0.5 mg/cm^2 had an SD of only 0.083. Model estimates, which did not allow SD estimates to decrease with the lead level, could not detect this pattern.

6.4.4.8.5 XL on Plaster

There were 222 observations of the XL on plaster. They were broken down by machine and operator as indicated below:

Machine 40:	Operator J	88	total (all Denver)
Machine 41:	Operator K	13	total (all Denver)
	Operator J	0	total
Machine 42:	Operator J	121	total (all Philadelphia)

Lower truncation at 0.0 mg/cm^2 occurred 25 times, the highest ICP measurement of which was 1.0387 mg/cm^2. Upper truncation at 5.0 mg/cm^2 was not observed on plaster.

Although there were 6 locations with ICP measurements above 10.0 mg/cm^2, upper truncation of the XL at 5.0 was not observed. These locations were in the same unit, in Denver, which was built in 1890. All of the L-shell instruments, including the XL, gave

low readings at these locations. In fact, 5 of the 6 readings were less than 1.0 mg/cm^2. The two locations with the highest ICP measurements, at 20.4 mg/cm^2 and 37.3 mg/cm^2, had XL readings of 0.5 mg/cm^2. Restriction of the ICP range to under 5.0 mg/cm^2 was employed to obtain model estimates and to infer instrument performance at lower lead levels. There were no field samples with ICP measurements between 3.0 mg/cm^2 and 5.0 mg/cm^2.

Figure 6-61 shows the response and SD components of the estimated model before provision for the combined effect of spatial variation and laboratory error in ICP measurements. Agreement with the nonparametric estimates appears to be good, except that the nonparametric estimate suggests a higher response when the ICP measurement exceeded 1.0 mg/cm^2. As seen on other substrates, the responsiveness of the XL appeared to change near the 1.0 mg/cm^2 lead level.

Table 6-135 gives the results of fitting XRF measurement models. Results for Machine 41 were not given because of insufficient data. Nonparametric estimates are reported for the bias at 1.0 mg/cm^2. Unlike other substrates, Machine 40 did not stand out as having significantly greater bias than Machine 42 at 0.0 mg/cm^2. Both machines exhibited substantial negative bias as the lead level increased. The estimate of the bias at 1.0 mg/cm^2 for Machine 40 obtained from the model (-.453 mg/cm^2) is close to the nonparametric estimate (-.481 mg/cm^2). The estimates for Machine 42 (-.501 mg/cm^2 model, -.255 mg/cm^2 nonparametric) exhibit greater disparity.

Table 6-136 gives a summary of the control block data. Machine 40 was substantially biased (1.850) at 0.0 mg/cm^2, but there was no evidence of this in the field sample data. Of the 46 Machine 40 measurements at ICP measurements less than 0.1 mg/cm^2, the average XL reading was only 0.135, which contradicts the control block finding. Readings of 0.0 mg/cm^2 were made on 14 of the 46 field samples with Machine 40, compared to 4 of 10 with Machine 42, which gives little indication of a machine effect. The control block data summary indicates a generally constant bias for Machine 42 as the lead level increased, while Machines 40 and 41 exhibited substantial negative bias at 3.53 mg/cm^2. Standard deviation estimates obtained from the control block data are lower at 1.02 mg/cm^2 than at 0.0 mg/cm^2 and 3.53 mg/cm^2 with all three machines.

6.4.4.8.6 XL on Wood

There were 355 observations of the XL on wood. To avoid problems with upper truncation, the 343 observations with ICP measurements less than 10.0 mg/cm^2 were used in the analysis. They were broken down by machine and operator as follows:

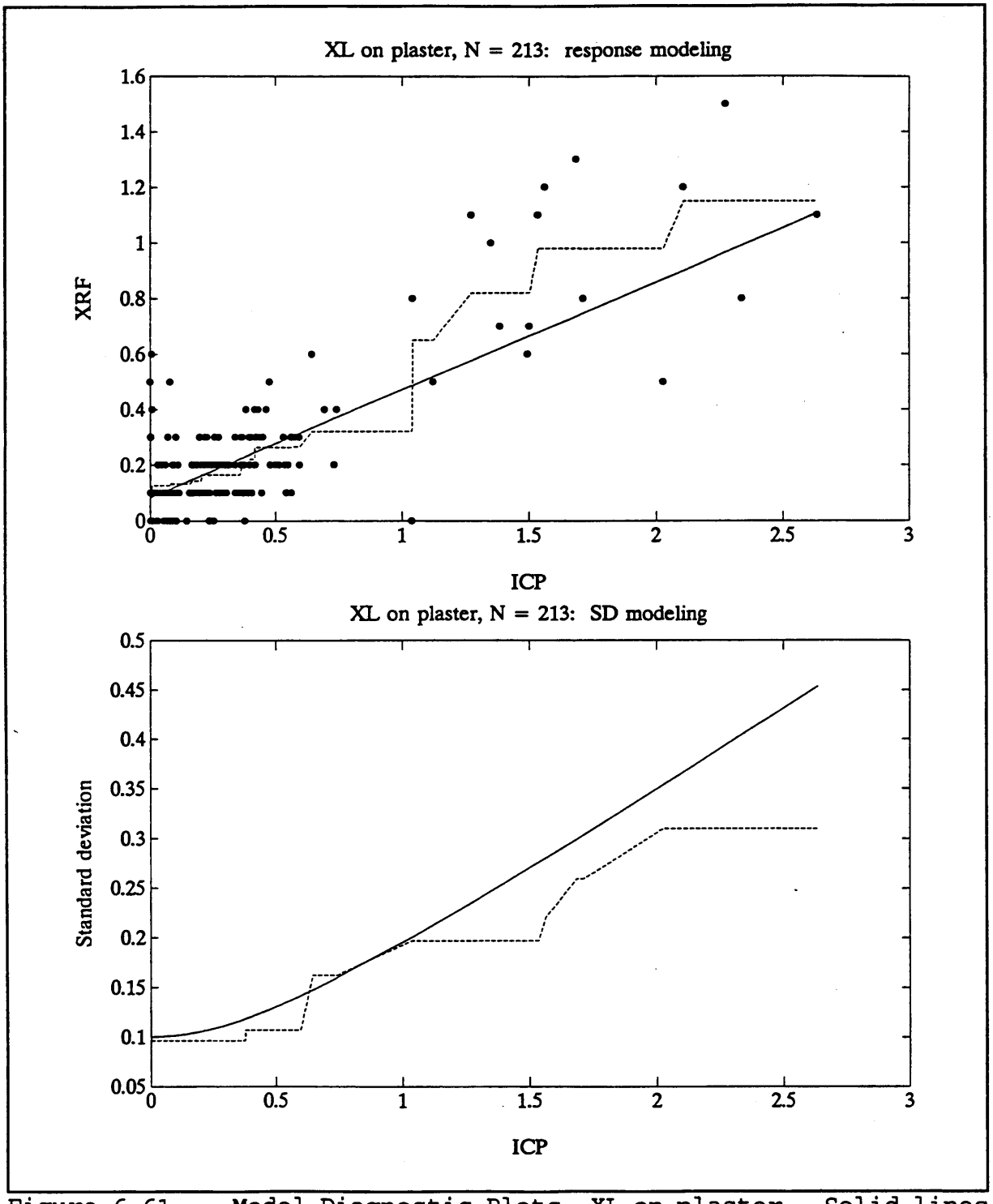


Figure 6-61. Model Diagnostic Plots, XL on plaster. Solid lines are model estimates. Dashed lines are nonparametric (monotone regression) estimates.

Table 6-135. XL on Plaster: Model Estimates.

DEVICE	SAMPLE SIZE	MODEL PARAMETERS				Pb=0.0 mg/cm²		Pb=1.0 mg/cm²	
		a	b	c	d	BIAS	SD	BIAS[a]	SD
Machine 40, ICP < 5	88	0.097 (.022)	0.450 (.127)	0.015 (.002)	-0-	0.097 (.022)	0.123	-0.481 (.101)	0.123
Machine 42, ICP < 5	121	0.048 (.011)	0.451 (.042)	0.002 (.001)	0.040 (.010)	0.048 (.011)	0.046	-0.255 (.090)	0.205
Machines 40 and 42, ICP < 5	209	0.081 (.011)	0.405 (.037)	0.010 (.001)	0.017 (.007)	0.081 (.011)	0.101	-0.257 (.072)	0.164

[a] Nonparametric estimates reported. Standard error estimates obtained by bootstrapping.

Table 6-136. XL on Plaster: Control Block Summary.

DEVICE	SAMPLE SIZE	BARE (0.0 mg/cm²)		RED NIST SRM (1.02 mg/cm²)		YELLOW NIST SRM (3.53 mg/cm²)	
		BIAS	SD	BIAS	SD	BIAS	SD
Machine 40	20	1.850 (.169)	0.756	0.035 (.021)	0.095	-0.550 (.121)	0.540
Machine 41	13	0.723 (.294)	1.062	-0.020 (.042)	0.153	-0.299 (.216)	0.778
Machine 42	40	0.258 (.050)	0.319	0.120 (.009)	0.059	0.118 (.045)	0.282
Machines 40, 41, and 42	73	0.777 (.074)	0.636	0.072 (.011)	0.092	-0.140 (.056)	0.477

Machine 40:	Operator J	151 total (all Denver)
Machine 41:	Operator K	51 total (all Denver)
	Operator J	89 total (all Denver)
Machine 42:	Operator J	52 total (all Philadelphia)

Lower truncation at 0.0 mg/cm^2 occurred 121 times, the highest ICP measurement of which was 5.0579 mg/cm^2. Upper truncation at 5.0 mg/cm^2 occurred 8 times, the lowest ICP measurement of which was 11.2525 mg/cm^2.

Figure 6-62 shows the response and SD components of the estimated model before provision for the combined effect of spatial variation and laboratory error in ICP measurements. The data were restricted to ICP measurements less than 10.0 mg/cm^2 to avoid problems with upper truncation. The model and nonparametric estimates appear to agree for ICP levels as large as 2.0 mg/cm^2, above which the nonparametric response appears flatter.

An important feature that is not apparent in Figure 6-62 is the predominance of zero readings at low lead levels. All 41 readings with Machine 41 corresponding to ICP measurements less than 0.067 mg/cm^2 were zeros. Of the 152 readings with ICP measurements less than 0.1 mg/cm^2, 51 of 55 Machine 41 readings were zeros, as were all 8 readings with Machine 42, and 49 of 89 readings with Machine 40. The predominance of zero readings at low ICP measurements, with Machine 41 especially, was observed more often on wood than on other substrates. This phenomenon posed a problem for model fitting, because the apparent standard deviation at lead levels close to 0.0 mg/cm^2 is essentially zero for Machine 41. For this reason, only data in the ICP range 0.1 mg/cm^2 to 10.0 mg/cm^2 were used to obtain model estimates.

Table 6-137 gives the results of fitting XRF measurement models to the data. Nonparametric estimates of the bias are reported for the 1.0 mg/cm^2 lead level. Bias and SD estimates at 0.0 mg/cm^2 reported for Machines 40 and 41 are sample means and standard deviations for the data restricted to ICP measurements less than 0.1 mg/cm^2. The model estimates are reported for Machine 42, because of the lack of data in the restricted range. Machine 40 exhibited low bias at 1.0 mg/cm^2 that was comparable to some of the K-shell instruments. The high slope parameter (.830) for Machine 40 is consistent with the low nonparametric bias estimate. The bias exhibited by Machines 41 and 42 was

substantial and negative. The XL gave readings less than 1.0 mg/cm^2 with Machines 41 and 42 on 5 sample locations with ICP measurements greater than 5.0 mg/cm^2.

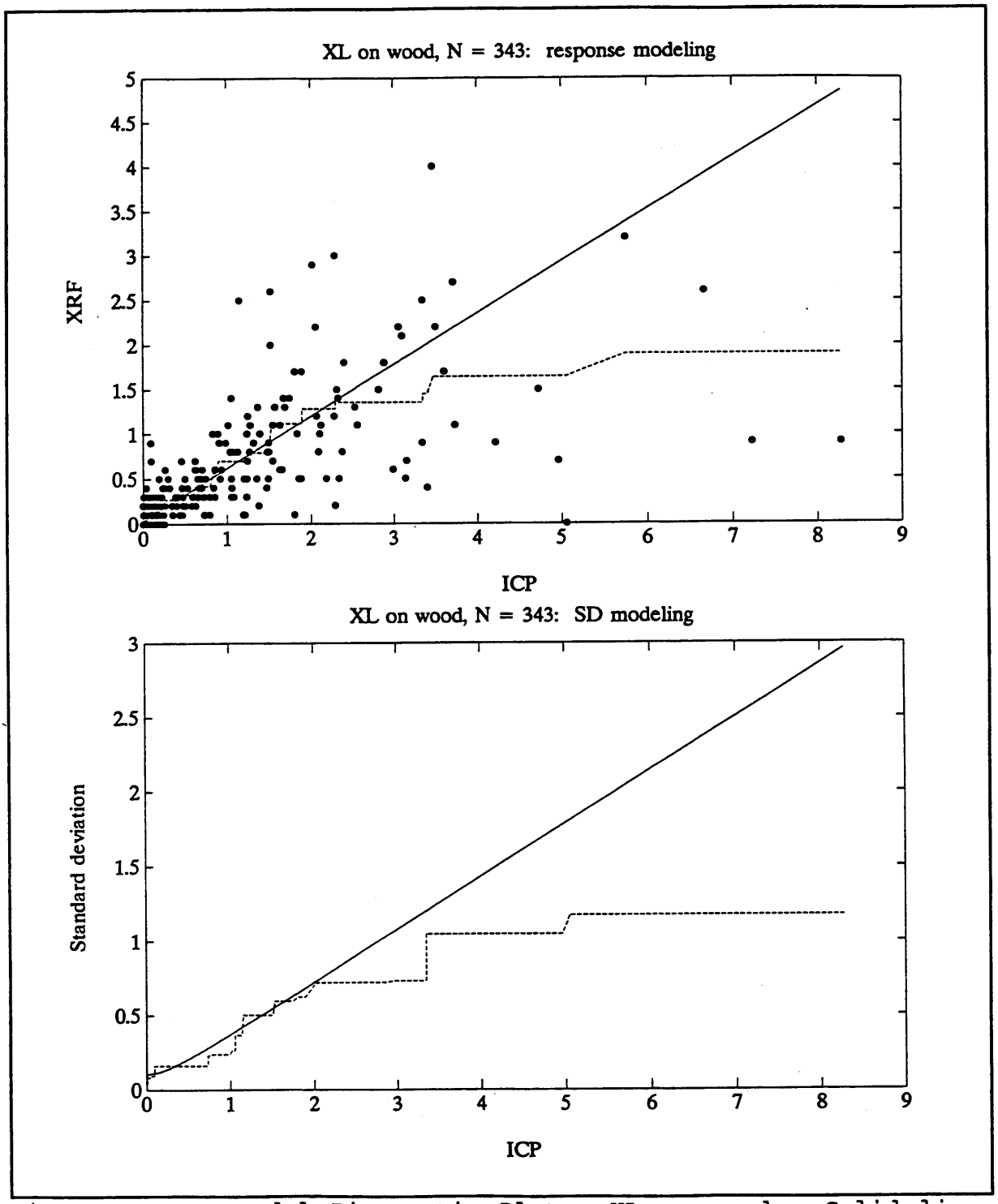


Figure 6-62. Model Diagnostic Plots, XL on wood. Solid lines are model estimates. Dashed lines are nonparametric (monotone regression) estimates.

Table 6-137. XL on Wood: Model Estimates.

DEVICE	SAMPLE SIZE	MODEL PARAMETERS				Pb=0.0 mg/cm²		Pb=1.0 mg/cm²	
		a	b	c	d	BIAS	SD	BIAS[a]	SD
Machine 40 (0.1<ICP<10)	62	0.015 (.027)	0.830 (.072)	0.007 (.004)	0.069 (.033)	0.080[b] (.013)	0.120[b]	-0.044 (.100)	0.276
Machine 41 (0.1<ICP<10)	85	0.040 (.028)	0.433 (.048)	0.007 (.004)	0.053 (.015)	0.022[b] (.017)	0.123[b]	-0.363 (.061)	0.243
Machine 42 (0.1<ICP<10)	44	0.092 (.056)	0.461 (.087)	0.014 (.008)	0.091 (.031)	0.092 (.056)	0.119	-0.483 (.080)	0.325
Machines 40, 41 and 42 (0.1<ICP<10)	191	0.049 (.017)	0.546 (.037)	0.008 (.002)	0.091 (.015)	0.055[b] (.010)	0.121[b]	-0.300 (.079)	0.315

[a] **Nonparametric estimates reported. Standard error estimates obtained by bootstrapping.**
[b] **Estimates based on sample summary statistics for ICP < 0.1 mg/cm²**

Table 6-138. XL on Wood: Control Block Summary.

DEVICE	SAMPLE SIZE	BARE (0.0 mg/cm²)		RED NIST SRM (1.02 mg/cm²)		YELLOW NIST SRM (3.53 mg/cm²)	
		BIAS	SD	BIAS	SD	BIAS	SD
Machine 40	23	0.009 (.006)	0.029	0.041 (.019)	0.089	-0.260 (.097)	0.468
Machine 41	19	0.000 (.000)	0.000	-0.104 (.032)	0.139	-0.467 (.155)	0.676
Machine 42	35	0.000 (.000)	0.000	0.097 (.009)	0.051	0.159 (.047)	0.281

Machines 40, 41, and 42	77	0.003 (.002)	0.016	0.031 (.010)	0.091	-0.121 (.053)	0.461

Table 6-138 gives a summary of the control block data. The zero SD estimates for Machines 41 and 42 at 0.0 mg/cm^2 reflect the fact that all of the readings for these two machines on bare substrate were zeros. Like the field sample data, this predominance of zero readings on the wood control blocks was not exhibited on other substrates. The fact that zero readings were less prevalent with Machine 40 on the control blocks, which was also the case on the field samples, points to a machine effect, since the same operator (J) used both Machines 40 and 42. Machine 42 exhibited increasing, but not substantial bias as the lead level increased, while the bias for Machines 40 and 41 became increasingly negative and substantial.

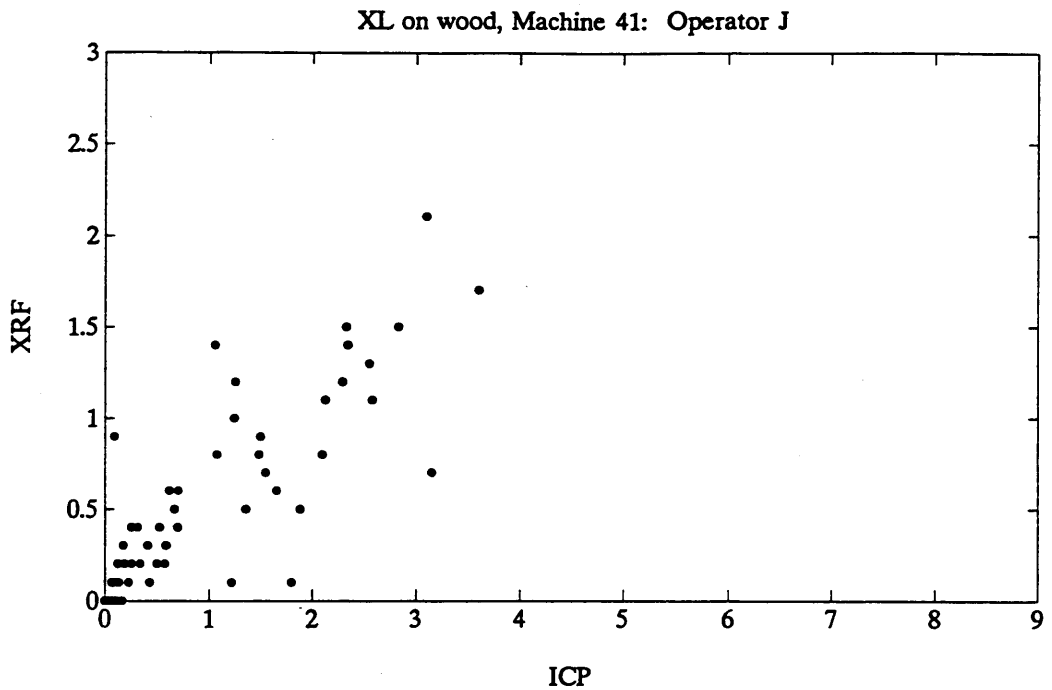
It is possible to see a mild operator effect within Machine 41, which was the only machine used by two operators. Figure 6-63 gives XRF-ICP scatterplots for the two operators of this machine. The scatterplot for Operator J suggests a greater slope (responsiveness to change in the lead level) than that for Operator K. The possibility that other factors, such as paint thickness, may explain the apparent operator difference cannot be dismissed.

6.4.4.8.7 XL: Summary of Analysis

The model did not adequately capture certain aspects of the performance of the XL. This is demonstrated graphically in Figures 6-57, 6-58, and 6-60, where the nonparametric response at the 1.0 mg/cm^2 ICP level is appreciably higher than the model response. The nonparametric estimates indicate less bias than the model at the 1.0 mg/cm^2 lead level. Although the nonparametric estimates did not account for the combined effect of spatial variation and laboratory error in ICP measurements, it is highly unlikely that provision for it would change this conclusion. On brick, concrete, metal and plaster, the response of the instrument seemed to change at or near a lead level of 1.0 mg/cm^2.

The XL produced readings on the field samples near the 1.0 mg/cm^2 ICP level that had less bias than readings from the other L-shell instruments evaluated. This finding agrees with the classification results presented in section 6.5, which show that the XL gave higher readings than the other L-shell instruments at lead levels above 1.0 mg/cm^2. But like the other L-shell instruments, the XL was also capable of giving readings less than

1.0 mg/cm^2 at ICP measurements in excess of 10.0 mg/cm^2. Of the 38 instances where the ICP measurement exceeded 10.0 mg/cm^2, 2 of the XL readings were below 0.4 mg/cm^2, and 1 was equal to 0.4



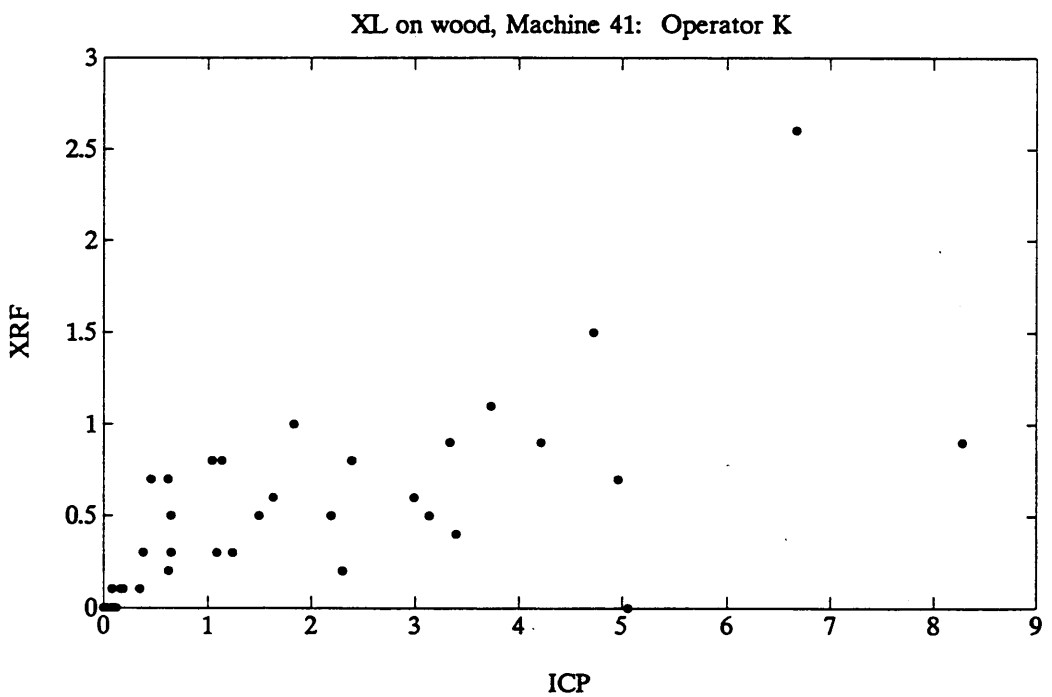


Figure 6-63. XL on Wood, Operators K versus J scatterplots on MACHINE 41.

mg/cm^2. The corresponding sample locations usually were found in older buildings, with thick layers of paint. The K-shell instruments were more consistent in giving readings above 1.0 mg/cm^2 in the presence of high lead levels.

Machine effects are an important factor to consider in evaluating the performance of the XL. These effects were apparent in (1) the model and nonparametric estimates derived from the field sample data; (2) the frequency of zero readings at very low (less than 0.1 mg/cm^2) ICP measurements; (3) the control block data summary statistics. All three sets of results point to Machine 40 as giving higher readings than either Machines 41 or 42 for lead levels less than 1.0 mg/cm^2. Machine 40 also produced fewer zero readings at low lead levels on painted samples than the other two machines. The same operator (J) used both Machines 40 and 42 in the study, including on the control blocks where lead levels and other factors that may have affected performance were controlled. It therefore is reasonable to attribute the higher Machine 40 readings to a machine effect. This does not rule out the possibility that other effects, such as those attributable to operators or related to the field samples, were present in conjunction with those attributable to machines.

On drywall and plaster, a pattern is present in the SD estimates obtained from the control block data, where the SD is substantially larger at 0.0 mg/cm^2 than at 1.02 mg/cm^2. A large positive bias at 0.0 mg/cm^2 can also be seen where this pattern is present. It was possible to reproduce the same pattern directly for the field sample data obtained with Machine 40 on several substrates, for which there were ample readings at very low ICP measurements. Standard deviations were computed for field sample readings with ICP measurements less than 0.005 mg/cm^2, and for readings with ICP measurements between 0.005 mg/cm^2 and 0.5 mg/cm^2. On all substrates except wood, Machine 40 had a higher SD estimate for the lower ICP range. A similar pattern was not seen in Machine 41 data taken on painted samples. This result suggest that, for certain XL machines or operators, readings at very low or zero lead levels were more variable than at somewhat higher lead levels.

6.4.5 Use of the First XRF Reading Versus the Average of Three Readings

In the full study, three successive, nominal 15-second readings were made with each XRF instrument, at each sampled location. In section 6.5 it is demonstrated that the use of the average of three readings did not significantly improve the classification accuracy obtained by using the first reading alone. The purpose of this section is to elaborate on this finding in the context of the XRF measurement model, and to explain why using the average of three readings was not found to substantially reduce the variability of XRF measurements on painted surfaces under field conditions.

Basic statistical reasoning suggests that the average of three readings should be more accurate than one reading, since the standard error of the average is smaller than the standard deviation of one reading. If the successive readings are statistically independent, the standard deviation of the average is approximately 0.58 (one divided by the square root of 3) times the standard deviation of one reading. In practice, however, this level of improvement was demonstrably absent. In section 6.4.5.1 it is shown that the standard deviation of the average was usually at least 0.70 times the standard deviation of one reading, and in some cases 1.00 times (no improvement) when the lead level was 1.0 mg/cm^2 or greater.

Further analyses suggest two reasons why the average of three readings fell short of the performance suggested by the 0.58 multiplier. The first is that *the assumption of independence was not valid*, with the possible exception of the MAP-3 K-shell. Estimated correlations between successive readings on the control blocks revealed, with the exception of the MAP-3, that successive readings were dependent to a substantial degree. The information obtained from three successive (but dependent) readings was less than if the readings were independent. The estimated correlations are presented in section 6.4.5.2, where this issue is discussed in more detail.

The second, and possibly more important reason why averaging three readings did not yield a large reduction in variability, is the existence of *non-instrumental sources of variability,* that replication cannot diminish. Factors associated neither with the level of lead in paint nor the instrument contributed to variation in XRF readings. In section 6.4.5.3, a nonparametric residual analysis demonstrates that the K-shell instruments were moderately intercorrelated, with above-average readings from the

MAP-3 associated with above-average readings from the Microlead I, for example. The same was also true for the L-shell instruments. These results suggest that instruments of the same shell were sensitized to factors other than lead that were associated with the sample locations.

Using the triplicate measurements from the full study and the correlations obtained in section 6.4.5.2, it was possible to partition the observed XRF variability into instrumental and non-instrumental components. This was done on the K-shell instruments for metal and wood substrates, and the results are presented in section 6.4.5.4. It is demonstrated that non-instrumental sources of variability were substantial, and exceeded instrumental variability as the lead level increased.

6.4.5.1 XRF Estimation With the Average of Three Readings

Section 6.4.2 describes the XRF measurement model that was applied to the first regular paint reading. The same model was also applied to the average of three readings. Estimated standard deviations at lead levels of 0.0 mg/cm^2 and 1.0 mg/cm^2 can be compared to similar quantities obtained using only the first reading. Tables 6-139 through 6-146 give estimated standard deviations by machine for each instrument, resulting from the model. Results for bias are not presented, because they were not noticeably affected by the change in number of readings.

The ratios reported in the tables are the standard deviations of the average divided by the standard deviations of the first XRF reading. Ratios close to, or in some cases larger than one due to sampling variability, indicate little or no accuracy gain from using the average of three readings. A ratio of 0.58 corresponds to the reduction in variability that would be obtained if the three readings were independent, and if only instrumental error contributed to the variability of XRF readings at a fixed level of lead. Table 6-147 is a summary of the information in Tables 6-139 through 6-146, obtained by pooling data across substrates and machines.

The MAP-3 K-shell (Table 6-141) exhibited the greatest improvement with the use of three, as opposed to one reading. Still, the standard deviation ratios were larger than 0.58, and some were substantially larger. The L-shell instruments (Tables 6-140, 6-142, 6-144, and 6-146) benefitted the least from using

three measurements. The ratios for both the K- and L-shell instruments usually increased as the lead level increased from 0.0 mg/cm^2 to 1.0 mg/cm^2, where they often became close to or even exceeded 1.0. Correct classification of sites having high lead levels improved only minimally with the use of three averaged readings, as demonstrated in section 6.5.

The results presented in Table 6-139 through 6-146 demonstrate that taking three *successive* readings at a *fixed*

Table 6-139. Change in Standard Deviations: One Versus Three Paint Readings for Lead Analyzer K-shell.

SUBSTRATE	XRF CODE NO.	Pb = 0.0 mg/cm²			Pb = 1.0 mg/cm²		
		ONE	THREE	RATIO	ONE	THREE	RATIO
Brick	1	0.167	0.173	1.04	0.226	0.220	0.97
Concrete	1	0.113	0.072	0.63	0.366	0.369	1.01
Concrete	2	0.127	0.080	0.63	0.328	0.182	0.56
Drywall	1	0.076	0.048	0.62	0.354	0.300	0.84
Metal	1	0.169	0.148	0.87	0.421	0.387	0.92
Metal	2	0.242	0.164	0.68	0.242	0.164	0.68
Plaster	1	0.142	0.145	1.03	0.241	0.223	0.93
Plaster	2	0.123	0.071	0.58	0.250	0.328	1.31
Wood	1	0.082	0.067	0.82	0.440	0.429	0.98

Table 6-140. Change in Standard Deviations: One Versus Three Paint Readings for Lead Analyzer L-shell.

SUBSTRATE	XRF CODE NO.	Pb = 0.0 mg/cm²			Pb = 1.0 mg/cm²		
		ONE	THREE	RATIO	ONE	THREE	RATIO
Brick	1	0.057	0.059	1.04	0.060	0.061	1.02
Concrete	1	0.011	0.012	1.09	0.109	0.104	0.95
Concrete	2	0.014	0.013	0.93	0.021	0.021	1.00
Drywall	1	0.006	0.007	1.08	0.172	0.167	0.97
Metal	1	0.014	0.012	0.87	0.232	0.245	1.06
Plaster	1	0.005	0.006	1.04	0.159	0.179	1.13
Plaster	2	0.014	0.008	0.58	0.035	0.044	1.26
Wood	1	0.012	0.012	1.00	0.188	0.205	1.09

location did not, typically, realize the gain anticipated for three *independent* readings under *variable* sampling conditions. The following three sections explain why this occurred.

6.4.5.2 Dependence of Successive XRF Measurements

One reason why the average of three successive XRF readings did not yield a large improvement is that the three successive readings were substantially correlated. Obtaining an unusually

high first reading made it more likely that the second reading would also be high, and similarly for the third reading. Three *successive* readings were therefore less informative than three

Table 6-141. Change in Standard Deviations: One Versus Three Paint Readings for MAP-3 K-shell.

SUBSTRATE	XRF CODE NO.	Pb = 0.0 mg/cm²			Pb = 1.0 mg/cm²		
		ONE	THREE	RATIO	ONE	THREE	RATIO
Brick	10	0.762	0.524	0.69	0.771	0.547	0.71
Brick	11	1.012	0.685	0.68	1.018	0.694	0.68
Concrete	10	0.751	0.469	0.62	0.890	0.714	0.80
Concrete	11	1.078	0.788	0.73	1.158	0.893	0.77
Concrete	12	0.987	0.613	0.62	0.987	0.613	0.62
Drywall	10	0.324	0.206	0.64	0.324	0.206	0.64
Drywall	11	0.380	0.283	0.74	0.380	0.283	0.74
Metal	10	0.330	0.245	0.74	0.481	0.388	0.81
Metal	11	0.445	0.343	0.77	0.445	0.349	0.78
Metal	12	0.374	0.245	0.66	0.620	0.451	0.73
Plaster	10	0.702	0.513	0.73	0.781	0.608	0.78
Plaster	11	1.048	0.658	0.63	1.070	0.774	0.72
Plaster	12	0.754	0.482	0.64	0.782	0.555	0.71
Wood	10	0.449	0.375	0.84	0.629	0.586	0.93
Wood	11	0.528	0.497	0.94	0.530	0.498	0.94
Wood	12	0.525	0.393	0.75	0.999	0.780	0.78

independent readings.

The average of the correlations between first and second, first and third, and second and third readings determines the reduction in variability gained by using the average of three successive readings, as opposed to using only the first. For three independent readings, the correlations are each equal to zero, and the standard deviation of the average is $(1/3)^{0.5} = 0.58$ times as large as the standard deviation of one reading. Generally, the standard deviation of the average of three readings is the square root of the quantity one-third plus two-thirds times the average correlation multiplied by the standard deviation of a single reading:

$$SD_{three} = [1/3 + 2 \cdot C/3]^{0.5} \cdot SD_{one}.$$

For example, suppose that the first and second readings have a correlation of 0.28, the first and third readings 0.23, and the second and third readings 0.21. The average of the three correlations is C = 0.24, and the multiplier is

$$[1/3 + 2\cdot(0.24)/3]^{0.5} = 0.70,$$

which is equivalent to the statement that the ratio of SD_{three} to

Table 6-142. Change in Standard Deviations: One Versus Three Paint Readings for MAP-3 L-shell.

SUBSTRATE	XRF CODE NO.	Pb = 0.0 mg/cm²			Pb = 1.0 mg/cm²		
		ONE	THREE	RATIO	ONE	THREE	RATIO
Brick	10	0.237	0.229	0.97	0.239	0.231	0.97
Brick	11	0.219	0.238	1.09	0.228	0.246	1.08
Concrete	10	0.087	0.066	0.76	0.161	0.170	1.06
Concrete	11	0.094	0.114	1.21	0.230	0.221	0.96
Concrete	12	0.075	0.058	0.77	0.093	0.080	0.86
Drywall	10	0.039	0.029	0.74	0.255	0.232	0.91
Drywall	11	0.041	0.059	1.44	0.247	0.379	1.53
Metal	10	0.381	0.384	1.01	0.475	0.478	1.01
Metal	11	0.437	0.428	0.98	0.540	0.533	0.99
Metal	12	0.165	0.122	0.74	0.263	0.232	0.88
Plaster	10	0.081	0.065	0.80	0.161	0.163	1.01
Plaster	11	0.100	0.086	0.86	0.185	0.194	1.05
Plaster	12	0.077	0.065	0.84	0.099	0.090	0.91
Wood	10	0.095	0.085	0.89	0.282	0.300	1.06
Wood	11	0.086	0.086	1.00	0.253	0.301	1.19
Wood	12	0.147	0.158	1.07	0.192	0.166	0.86

SD_{one} is equal to 0.70. This is more than the 0.58 multiplier that would apply if the three readings were independent. In fact, a multiplier of 0.70 suggests that three successive (but correlated) readings have approximately the same information as two independent (uncorrelated) readings with the same instrument.

Table 6-148 reports average correlations for the eight XRF instrument types estimated from the control block data. Triplicate XRF readings were used to estimate the correlations between the first and second, first and third, and second and third readings. The advantage of using the control block data instead of the field sample data is that the lead levels were fixed at known values on the control blocks, which removed a potential source of spurious correlation. SD ratios were calculated from the correlations in the same manner as the above example, and are alternatives to the estimates presented in Tables 6-139 through 6-147.

Only the MAP-3 K-shell gave nearly uncorrelated successive readings. The L-shell instruments produced more highly correlated readings than the K-shell instruments. The MAP-3 L-

shell, however, had correlations more similar to the K-shell instruments than to the other L-shell instruments. The L-shell instruments had higher correlations at 0.0 mg/cm^2 than at higher

Table 6-143. Change in Standard Deviations: One Versus Three Paint Readings for Microlead I.

SUBSTRATE	XRF CODE NO.	Pb = 0.0 mg/cm²			Pb = 1.0 mg/cm²		
		ONE	THREE	RATIO	ONE	THREE	RATIO
Concrete	20 (Den)	0.559	0.370	0.66	0.708	0.549	0.78
Concrete	20 (Phi)	0.543	0.386	0.71	0.543	0.488	0.90
Concrete	21	0.724	0.635	0.88	0.790	0.706	0.89
Concrete	22	1.244	1.306	1.05	1.309	1.306	1.00
Concrete	23	0.483	0.346	0.72	0.483	0.607	1.26
Drywall	20	0.345	0.235	0.68	0.345	0.387	1.12
Drywall	21	0.351	0.244	0.70	0.351	0.395	1.13
Drywall	22	0.534	0.538	1.01	0.534	0.538	1.01
Metal	20	0.617	0.563	0.91	0.684	0.630	0.92
Metal	21	0.732	0.685	0.94	0.790	0.744	0.94
Metal	22	0.808	0.850	1.05	0.808	0.850	1.05
Metal	23	0.366	0.223	0.61	0.545	0.467	0.86
Metal	24	0.723	0.691	0.96	0.723	0.691	0.96
Plaster	20	0.510	0.350	0.69	0.584	0.448	0.77
Plaster	21	0.660	0.484	0.73	0.807	0.670	0.83
Plaster	22	1.013	0.579	0.57	1.013	0.579	0.57
Plaster	23	0.370	0.322	0.87	0.531	0.415	0.78
Wood	20	0.624	0.499	0.80	0.915	0.867	0.95
Wood	21	0.663	0.602	0.91	0.758	0.740	0.98
Wood	22	0.831	0.800	0.96	0.851	0.835	0.98
Wood	23	0.500	0.419	0.84	0.550	0.645	1.17
Wood	24	0.578	0.568	0.98	0.578	0.568	0.98

Table 6-144. Change in Standard Deviations: One Versus Three Paint Readings for X-MET 880.

SUBSTRATE	XRF CODE NO.	Pb = 0.0 mg/cm²			Pb = 1.0 mg/cm²		
		ONE	THREE	RATIO	ONE	THREE	RATIO
Concrete	50	0.027	0.027	1.00	0.067	0.066	0.99
Drywall	50	0.013	0.013	1.00	0.133	0.134	1.01
Metal	50	0.141	0.141	1.00	0.238	0.237	1.00
Metal	51	0.278	0.260	0.94	0.278	0.260	0.94
Plaster	50	0.022	0.022	1.00	0.083	0.082	0.99
Wood	50	0.024	0.022	0.90	0.289	0.296	1.02

Table 6-145. Change in Standard Deviations: One Versus Three Paint Readings for XK-3.

SUBSTRATE	XRF CODE NO.	Pb = 0.0 mg/cm²			Pb = 1.0 mg/cm²		
		ONE	THREE	RATIO	ONE	THREE	RATIO
Brick	30	0.591	0.493	0.83	0.591	0.493	0.83
Brick	31	0.320	0.173	0.54	0.320	0.719	2.25
Concrete	30	0.636	0.506	0.80	0.636	0.506	0.80
Concrete	31	0.847	0.255	0.30	0.847	0.255	0.30
Concrete	32	0.508	0.478	0.94	0.508	0.478	0.94
Drywall	30	0.356	0.203	0.57	0.562	0.845	1.50
Drywall	31	0.206	0.182	0.88	0.547	0.361	0.66
Metal	30	0.517	0.435	0.84	1.058	0.962	0.91
Metal	32	0.607	0.465	0.77	0.992	0.912	0.92
Plaster	30	0.546	0.474	0.87	0.633	0.547	0.86
Plaster	31	0.404	0.359	0.89	0.404	0.360	0.89
Plaster	32	0.521	0.352	0.68	0.645	0.452	0.70
Wood	30	0.486	0.376	0.77	0.686	0.679	0.99
Wood	31	0.313	0.269	0.86	0.444	0.503	1.13
Wood	32	0.488	0.304	0.62	1.152	1.010	0.88

Table 6-146. Change in Standard Deviations: One Versus Three Paint Readings for XL.

SUBSTRATE	XRF CODE NO.	Pb = 0.0 mg/cm²			Pb = 1.0 mg/cm²		
		ONE	THREE	RATIO	ONE	THREE	RATIO
Brick	41	0.056	0.043	0.77	0.167	0.176	1.05
Concrete	41	0.050	0.044	0.88	0.312	0.349	1.12
Concrete	42	0.083	0.066	0.80	0.211	0.200	0.95
Drywall	41	0.049	0.058	1.18	0.266	0.225	0.85
Metal	40	0.196	0.192	0.98	0.280	0.278	0.99
Metal	41	0.158	0.107	0.68	0.158	0.107	0.68
Metal	42	0.124	0.110	0.89	0.396	0.391	0.99
Plaster	40	0.123	0.095	0.77	0.123	0.095	0.77
Plaster	42	0.046	0.039	0.85	0.205	0.182	0.89
Wood	40	0.120	0.088	0.73	0.276	0.271	0.99
Wood	41	0.123	0.052	0.42	0.243	0.217	0.89
Wood	42	0.119	0.097	0.82	0.325	0.305	0.94

Table 6-147. Standard Deviation (SD) Ratios, Pooled by Instrument.

Instrument	Pb=0.0 mg/cm^2 SD Ratio	Pb=1.0 mg/cm^2 SD Ratio
Lead Analyzer K-shell	0.84	0.95
Lead Analyzer L-shell	1.02	1.06
MAP-3 K-shell	0.71	0.78
MAP-3 L-shell	0.98	1.05
Microlead I	0.86	0.93
X-MET 880	0.98	1.01
XK-3	0.77	0.93
XL	0.79	0.96

levels of lead. The high correlation for the XL at 0.0 mg/cm^2 may be explained by the fact that it often gave three successive zero readings at lower lead levels, due to its lower truncation property.

The SD ratios reported in Table 6-148 are generally smaller than those reported in Table 6-147. Since a smaller ratio implies a greater reduction in variability using the average of three readings, XRF readings on the field samples (the data used to derive Table 6-147) were affected by sources of variability that averaging did not reduce, and that were absent from XRF readings on the control blocks. Thus, correlations between successive readings alone did not account for the small reduction in standard deviations obtained from the XRF measurement model, implying the existence of non-instrumental factors that affected XRF variability.

6.4.5.3 Correlation of XRF Readings Across Instruments

Non-instrumental factors that affected the variability of XRF readings were associated with the locations at which measurements were made. Non-instrumental factors have the potential to affect readings made with all instruments at a given location, although the impact can vary with the instrument. If several instruments are affected in a similar manner, these factors may be detectable as correlations across instrument readings.

Correlations across instruments were estimated using the

nonparametric standardized residuals defined in section 6.3. The nonparametric standardized residuals were obtained by subtracting

Table 6-148. Correlations for Successive Readings, Estimated From the Control Block Data.

INSTRUMENT	NUMBER OF READINGS	BARE 0.0 mg/cm²	RED NIST SRM 1.02 mg/cm²	YELLOW NIST SRM 3.53 mg/cm²	AVERAGE	SD RATIO
Lead Analyzer K-shell	1255	0.12	0.12	0.34	0.19	0.68
Lead Analyzer L-shell	1245	0.96	0.73	0.77	0.82	0.94
MAP-3 K-shell	1206	0.05	-0.06	0.06	0.02	0.59
MAP-3 L-shell	1202	0.46	0.13	0.14	0.24	0.70
Microlead I	636	0.32	0.41	0.31	0.34	0.75
X-MET 880	1145	0.97	0.83	0.84	0.88	0.96
XK-3	1179	0.33	0.42	0.36	0.37	0.76
XL	1305	0.96	0.74	0.83	0.85	0.95

from the XRF readings a nonparametric estimate of their mean relationship with the ICP measurements, and dividing the differences by a nonparametric estimate of the standard deviation. The resulting quantities can be viewed as the XRF readings with their dependence on the ICP-measured lead level separated out. Thus, although the nature of an XRF-ICP relationship changed substantially across instruments and substrates, the nonparametric standardized residuals exhibited comparable behavior, and their derivation did not require the use of a model.

The 8 measurement modes, obtained from the 6 instruments and 2 shells, were grouped into 12 field classifications. Each field classification represented a full set of XRF measurements on all sample locations. Since each sample location had 12 associated XRF readings, it was possible to calculate the 12 by 12 correlation matrix of nonparametric standardized residuals. This was done for 333 sample locations on wood substrates. The correlations are presented graphically in Figures 6-64 through 6-75. The K-shell instruments are shown with dark shading, and the L-shell instruments with light shading. The Lead Analyzer and two MAP-3 instrument field classes, which made measurements using both shells, are shown side by side.

The correlations exhibit an interesting pattern: the K-shell instruments were more correlated with other K-shell instruments than with the L-shell instruments, and similarly for the L-shell instruments. The Lead Analyzer K-shell, for instance, exhibited higher correlations with other K-shell instruments than with its own L-shell, as did the MAP-3. The XL and Lead Analyzer L-shell exhibited weaker correlations than the other two L-shell instruments, but both were more correlated with L-shell than with K-shell instruments.

The process that was used to derive the residuals eliminated the contribution of the ICP-measured lead levels to the correlations. Instrumental variability, which by definition is independent across different machines at a fixed level of lead, was likewise not a contributing factor to the correlations. Therefore, the substantial correlations that these residuals exhibited across instruments reflect the influence of non-instrumental factors, related to the locations where instrument readings were made, that contributed to XRF performance. These non-instrumental factors affected the performance of the K-shell

and L-shell instruments in different ways.

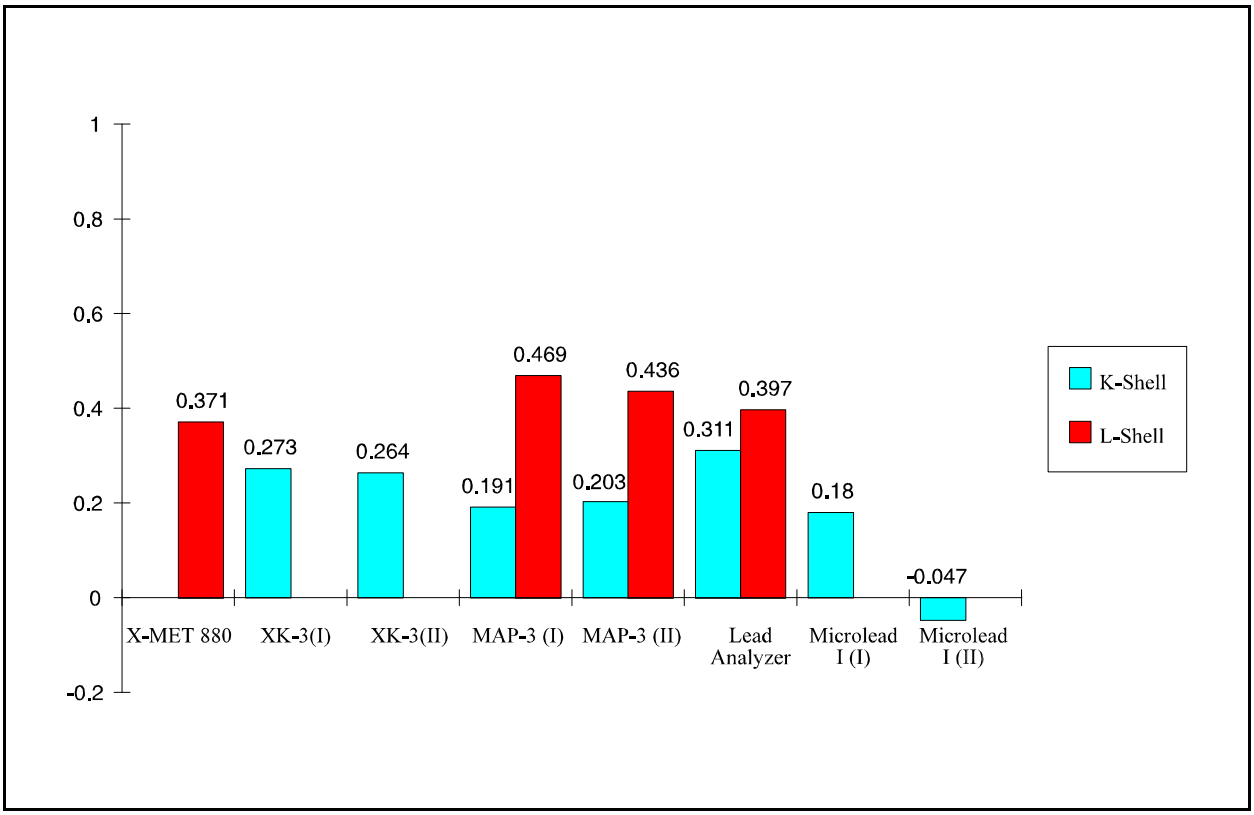


Figure 6-64. XL: Correlation of nonparametric standardized residuals w
other instruments. Substrate=WOOD

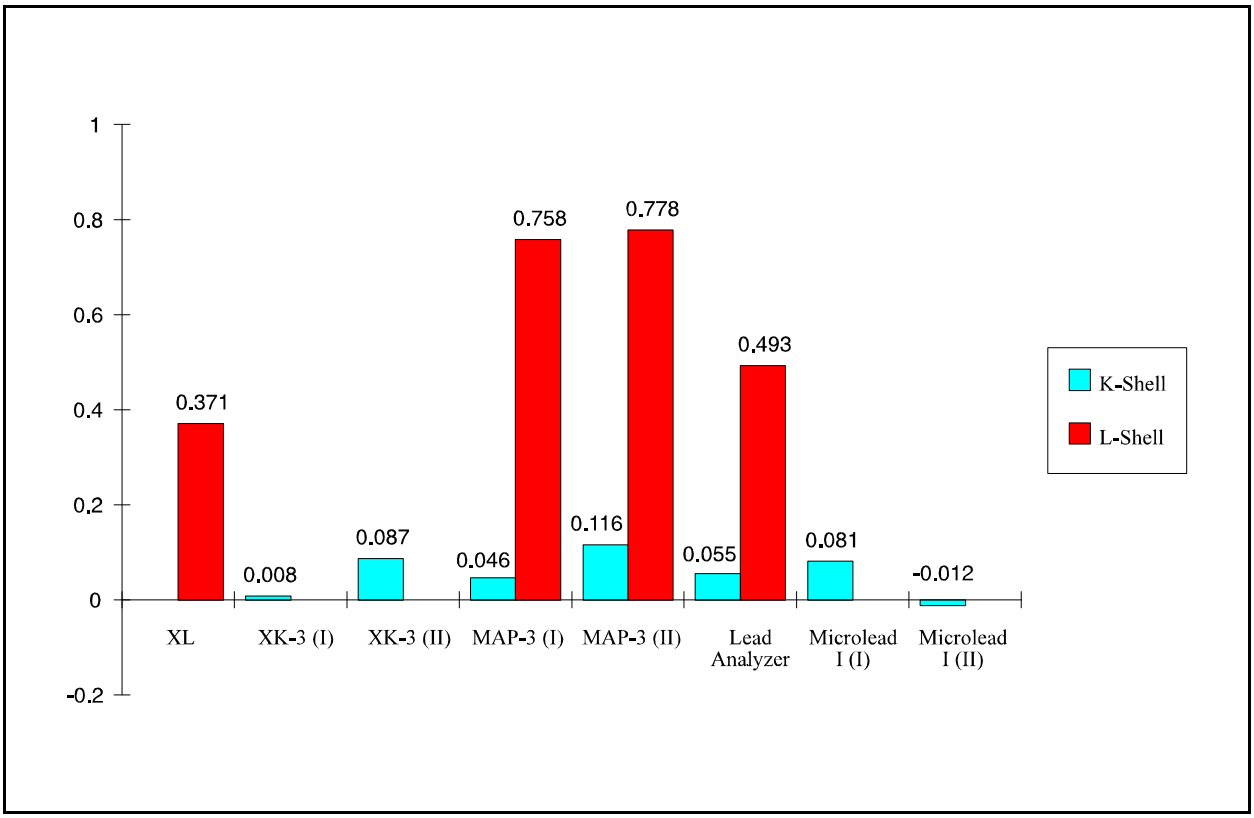


Figure 6-65. X-MET 880: Correlation of nonparametric standardized resi
with other instruments. Substrate=WOOD

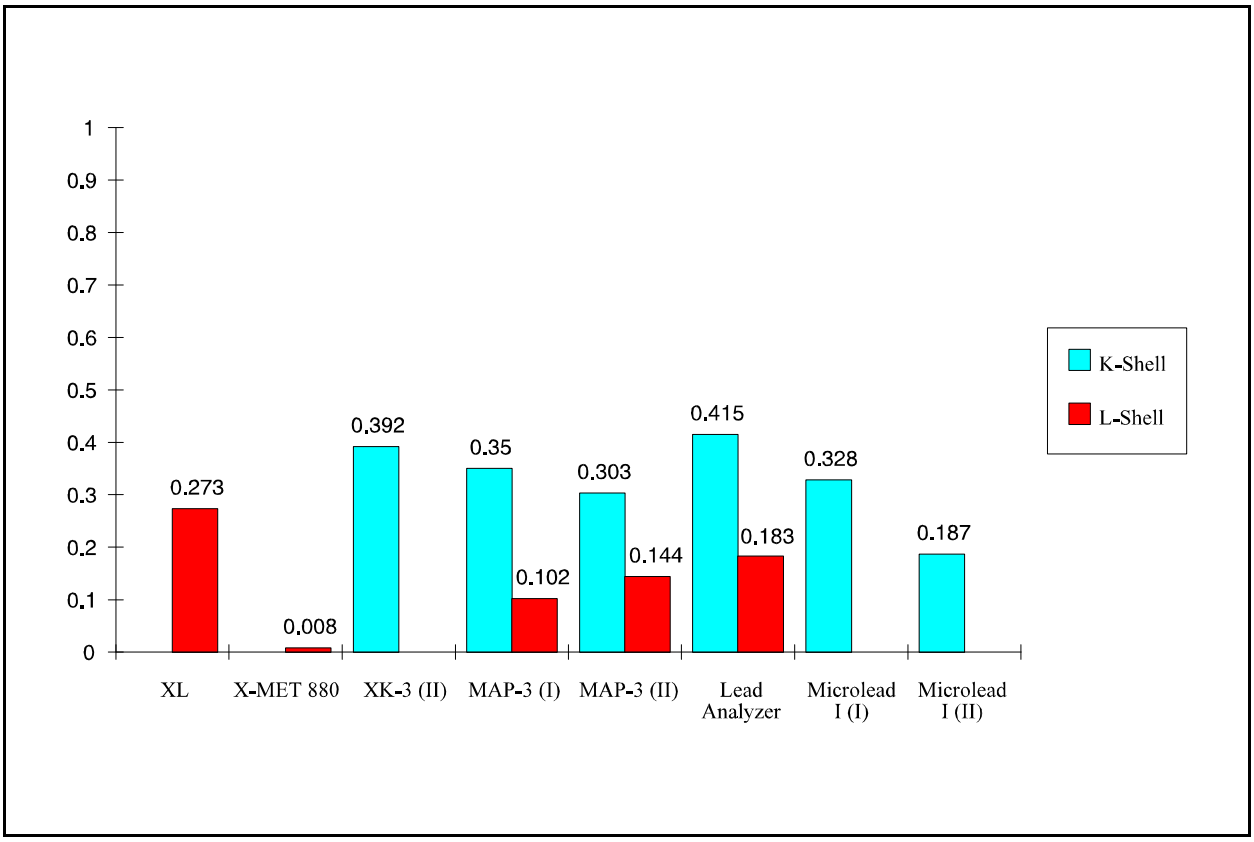


Figure 6-66. XK-3 (I): Correlation of nonparametric standardized resic with other instruments. Substrate=WOOD

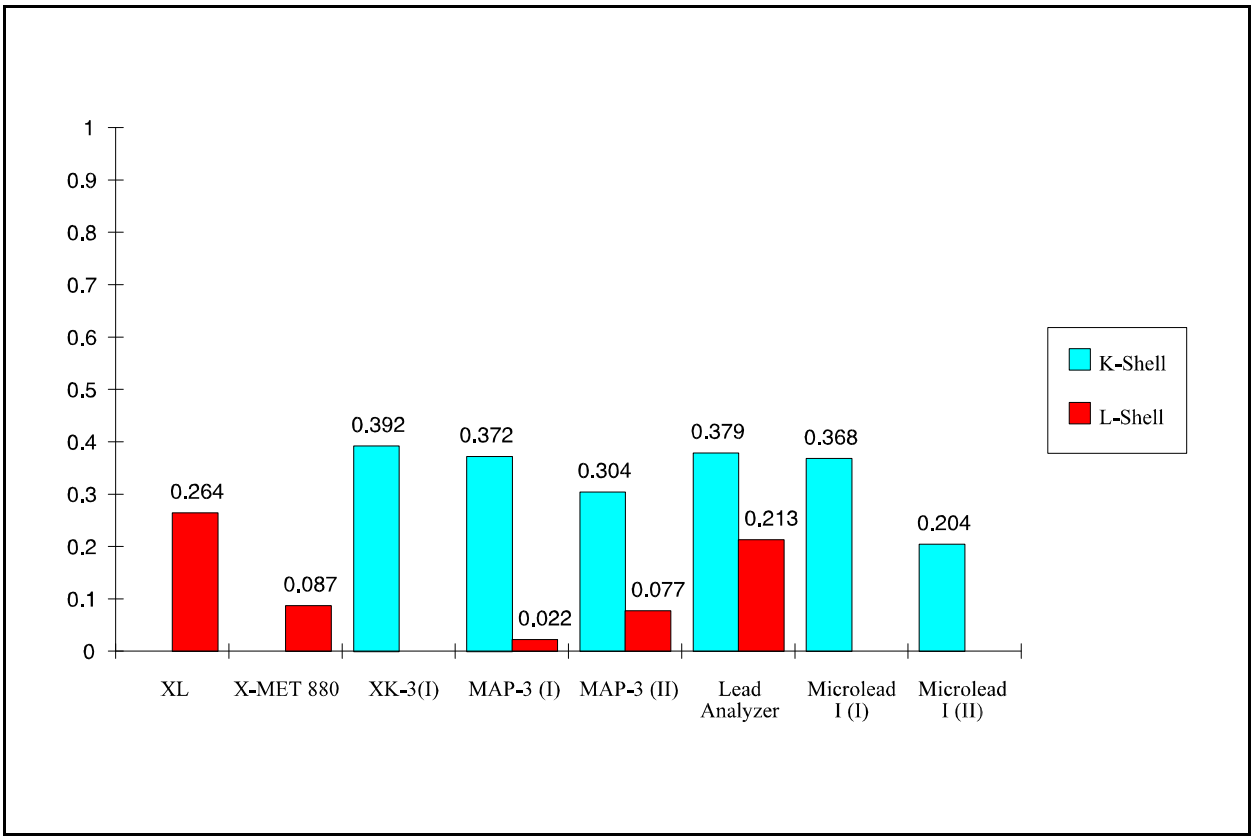


Figure 6-67. XK-3 (II): Correlation of nonparametric standardized resi
with other instruments. Substrate=WOOD

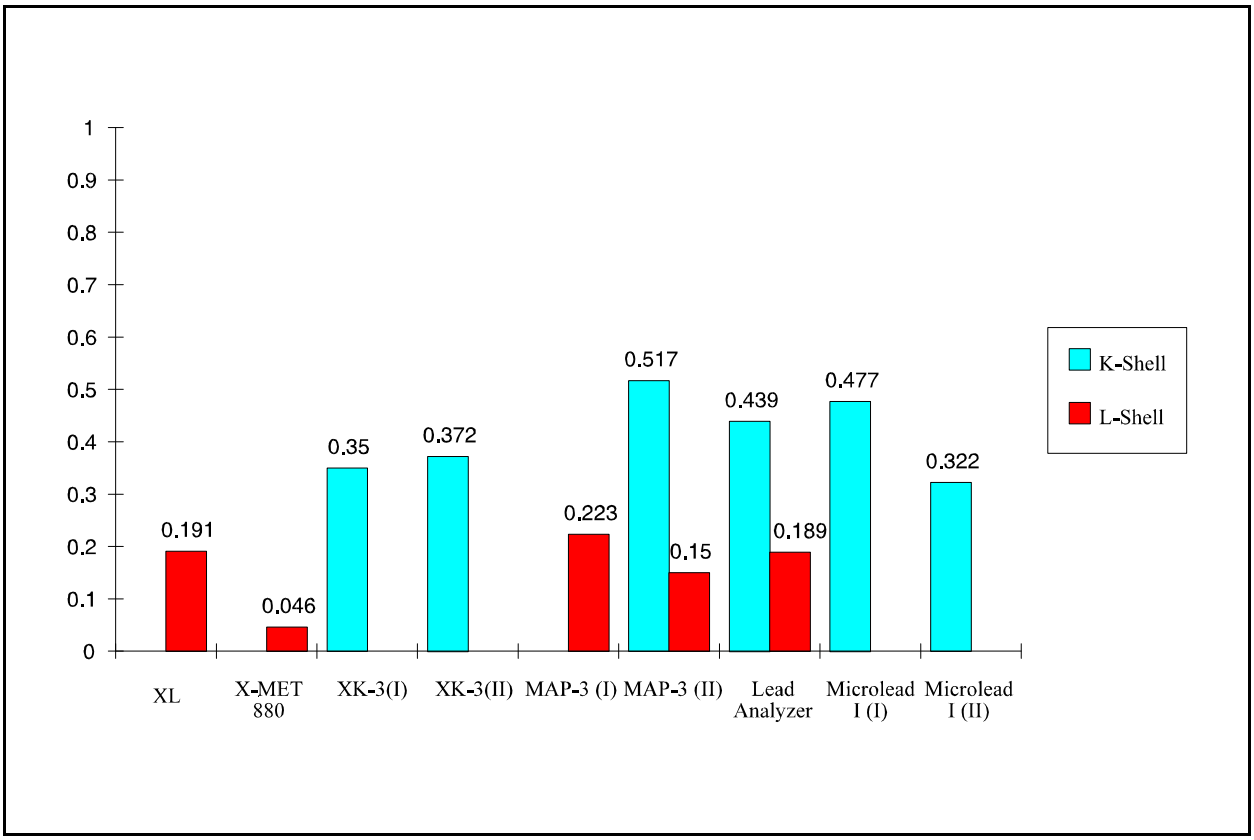


Figure 6-68. MAP-3 (I) K-shell: Correlation of nonparametric standardi residuals with other instruments. Substrate=WOOD.

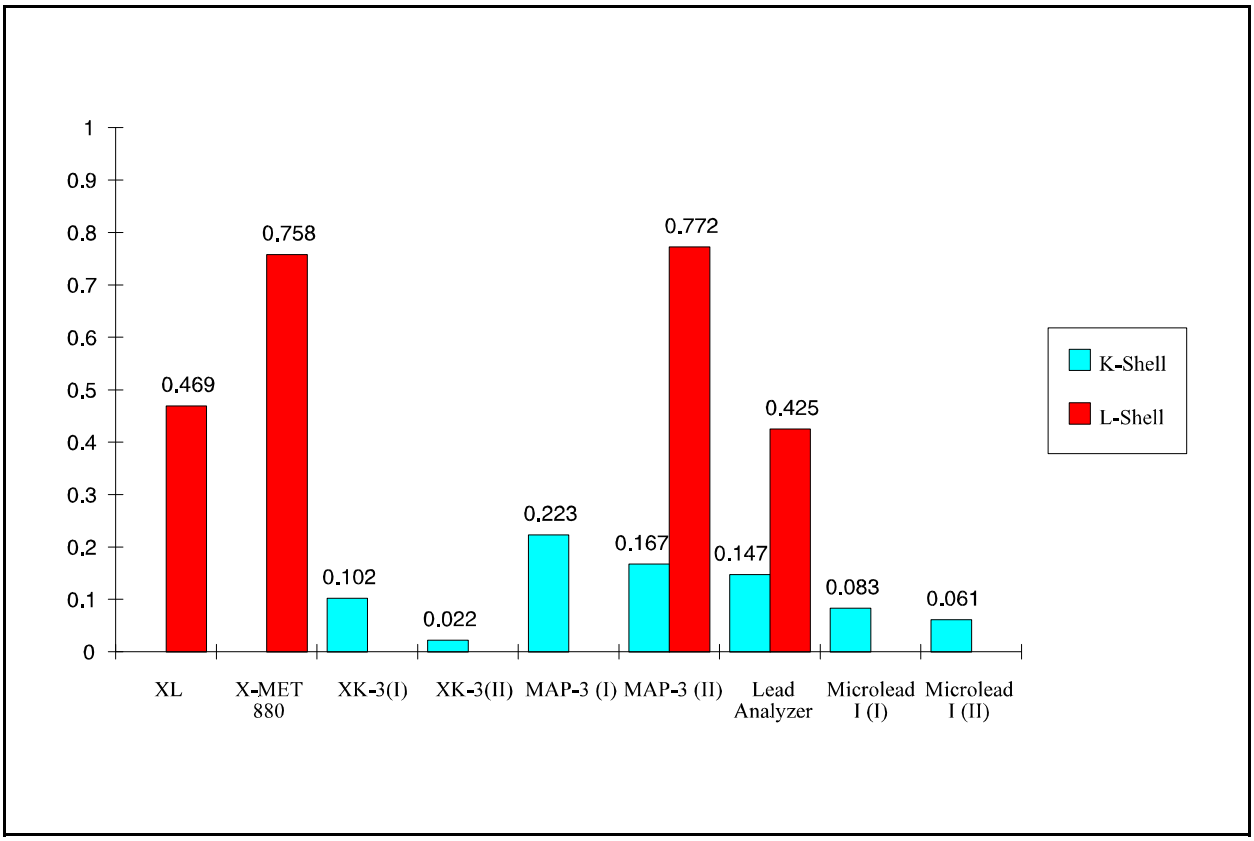


Figure 6-69. MAP-3 (I) L-shell: Correlation of nonparametric standardi residuals with other instruments. Substrate=WOOD

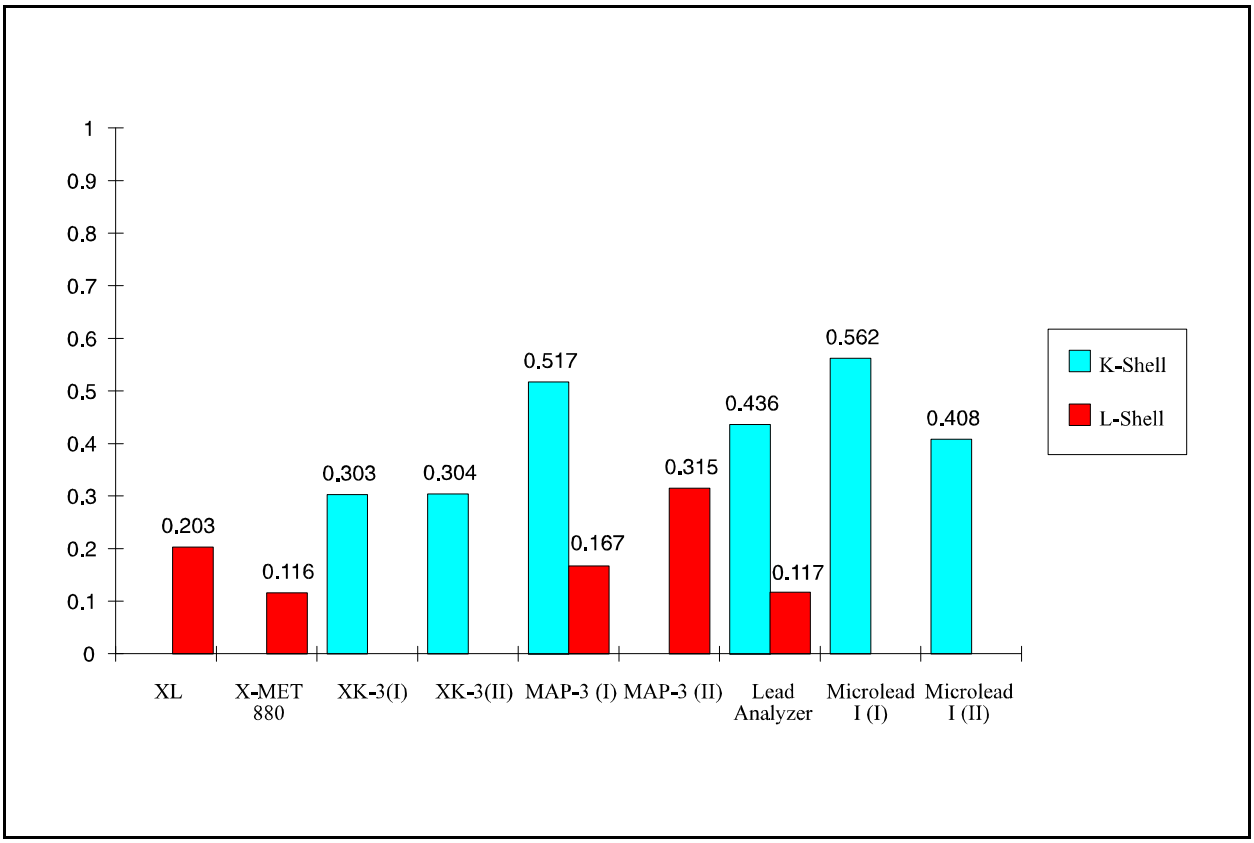


Figure 6-70. MAP-3 (II) K-shell: Correlation of nonparametric standard residuals with other instruments. Substrate=WOOD.

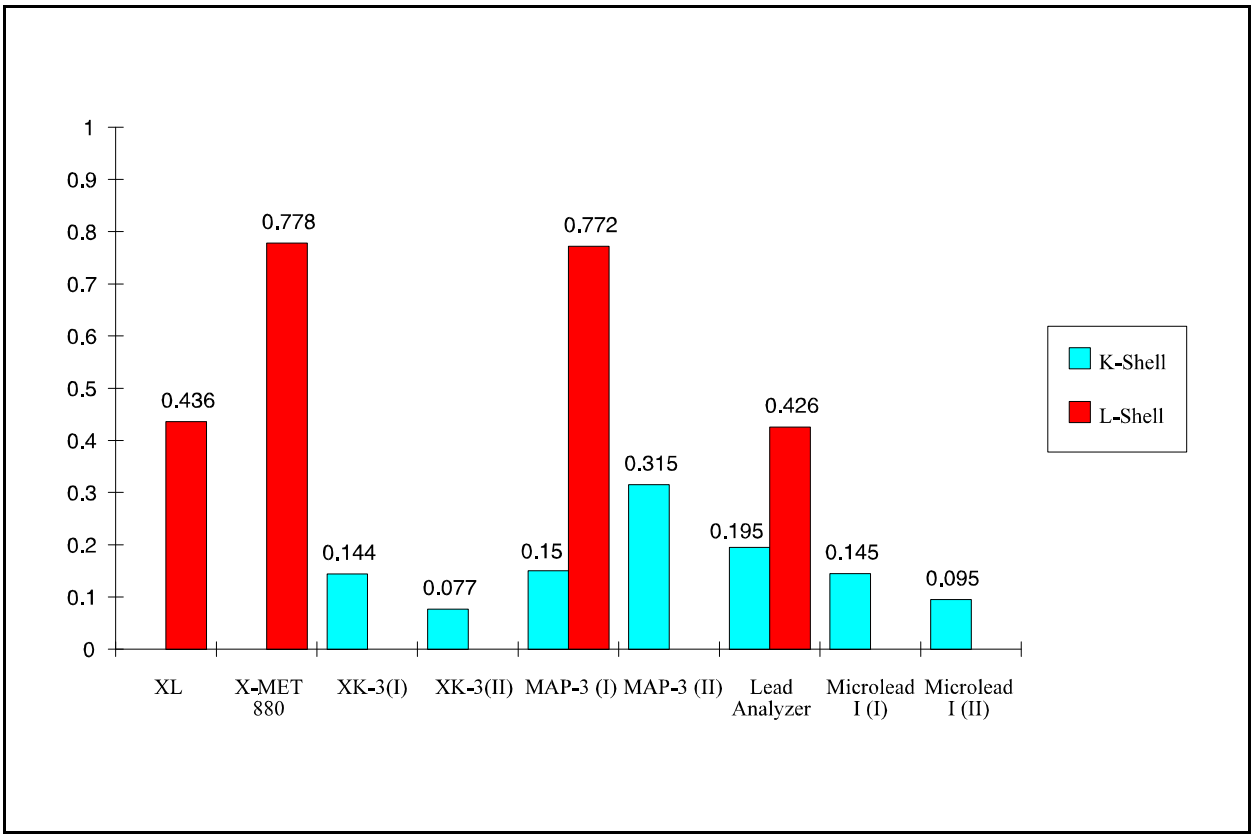


Figure 6-71. MAP-3 (II) L-shell: Correlation of nonparametric standard residuals with other instruments. Substrate=WOOD

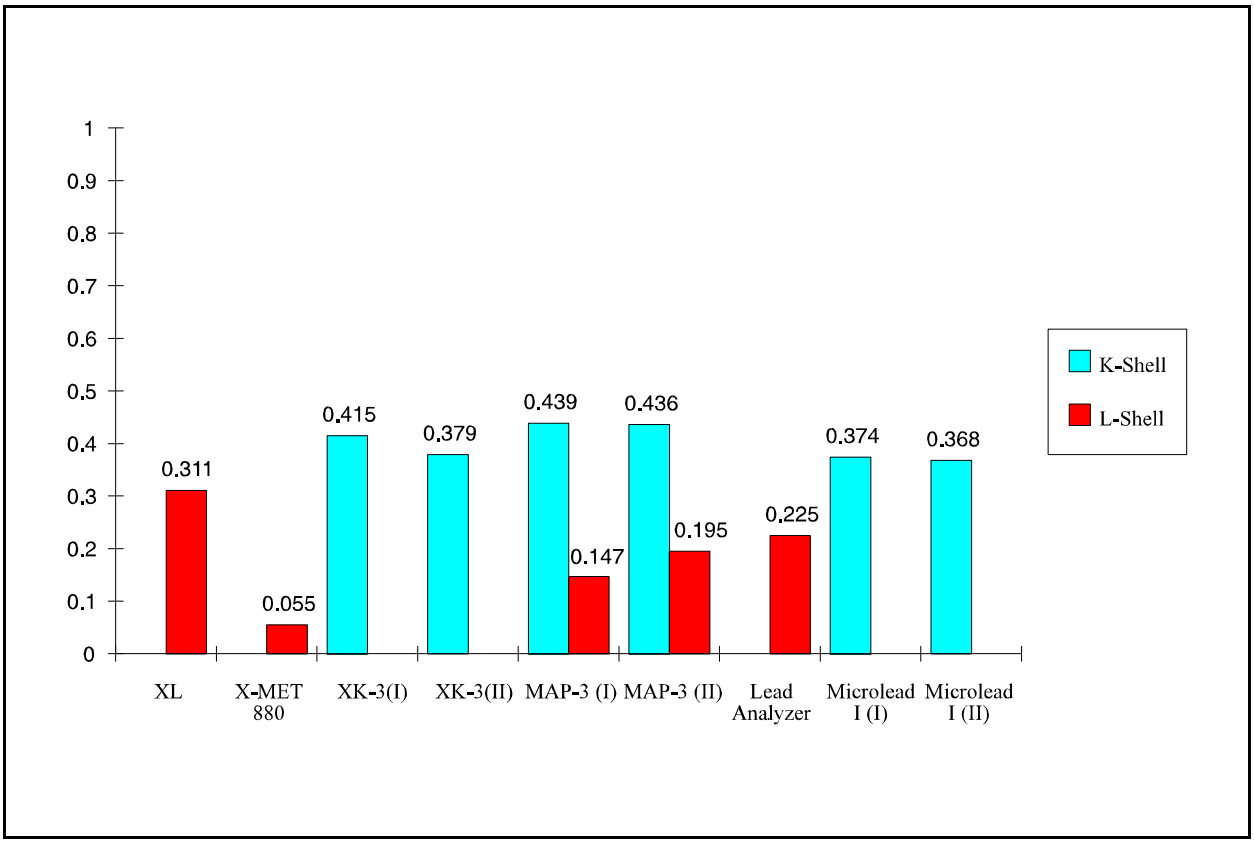


Figure 6-72. Lead Analyzer K-shell: Correlation of nonparametric standardized residuals with other instruments. Substrate=

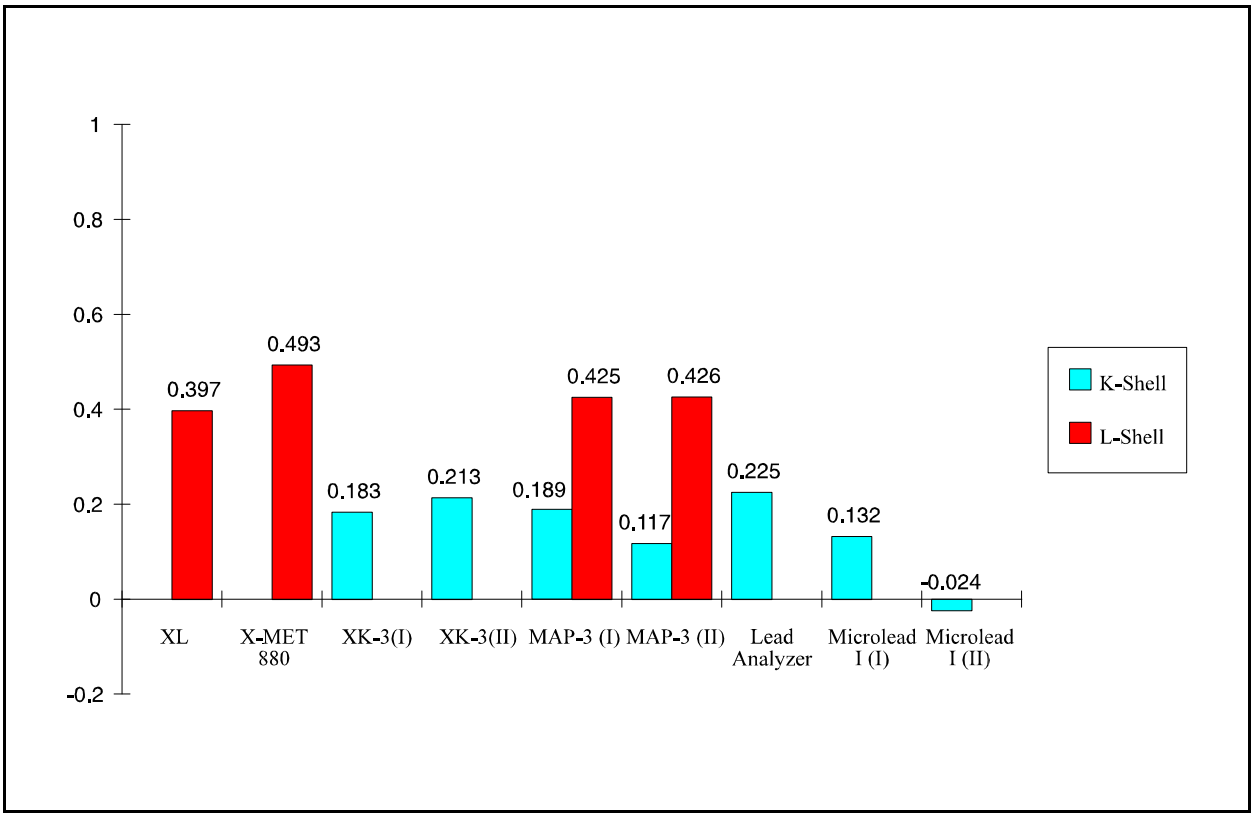


Figure 6-73. Lead Analyzer L-shell: Correlation of nonparametric standardized residuals with other instruments. Substrate=

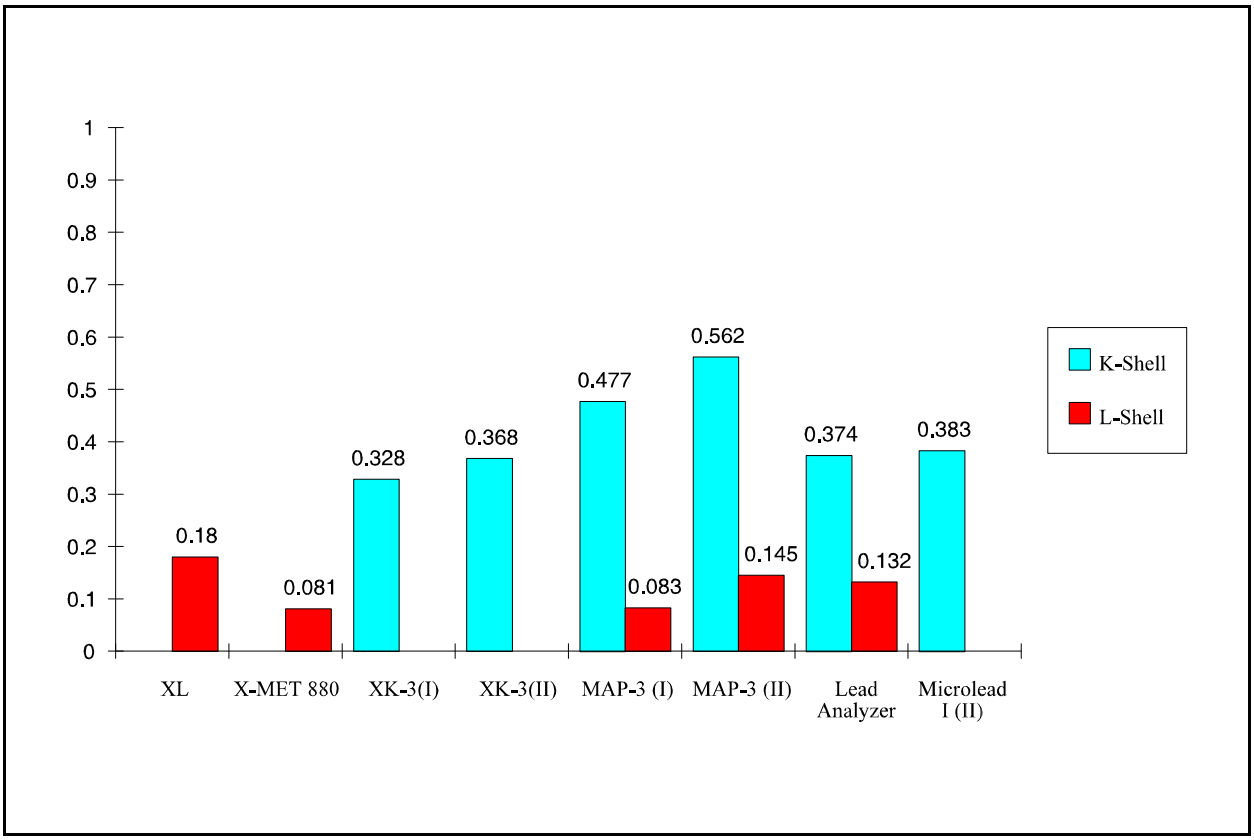


Figure 6-74. Microlead I (I): Correlation of nonparametric standardize residuals with other instruments. Substrate=WOOD.

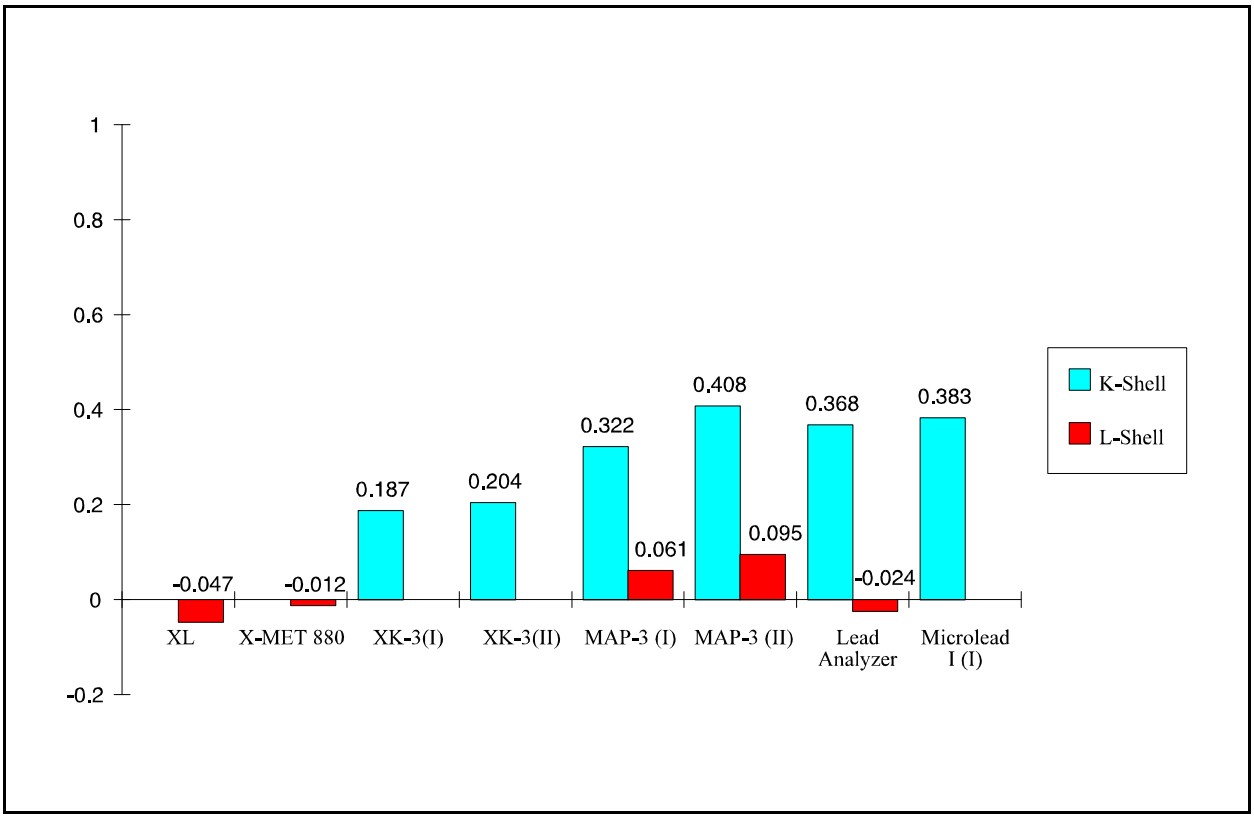


Figure 6-75. Microlead I (II): Correlation of nonparametric standardiz residuals with other instruments. Substrate=WOOD.

Repeated readings at the same location with the same instrument repeated the realization of location-specific factors that affected XRF performance. Therefore, averaging repeated readings did not reduce the variability that these factors imparted to XRF measurements. The MAP-3 K-shell, for instance, was clearly correlated with the other K-shell instruments, which suggests that non-instrumental factors contributed significantly to the variability of its readings on painted surfaces under field conditions. Although it was established in section 6.4.5.2 that its three successive readings were essentially uncorrelated, using the average of three readings instead of the first could not achieve a commensurate reduction in variability, as seen in Table 6-141.

6.4.5.4 Separating Instrumental and Non-instrumental Variability

Separation of instrumental and non-instrumental components of variability in XRF measurements illustrates the relative contribution of each component to total variability. This was done explicitly with the full study data. The triplicate measurements taken at each sample location were used to estimate the SD due to instrumental factors, taking into account the correlations presented in Table 6-148. Estimation of the XRF-ICP relationship was used to estimate the full SD due to *both* instrumental and non-instrumental factors. The SD due to non-instrumental factors was estimated by taking the square root of the full SD squared minus the instrumental SD squared. In cases where, due to small sample variability, the estimated instrumental SD exceeded the estimated full SD, the non-instrumental SD estimate was set to zero.

Figures 6-76 through 6-83 illustrate the effect of separation with the four K-shell instruments, on metal and wood substrates. The solid line in each graph shows the full SD for a single reading, as a function of the ICP level (mg/cm^2). The full SD was estimated using monotone regression of the first XRF reading against the ICP measurement. The dashed line shows the instrumental SD, which was estimated using monotone regression on the variances calculated from the three readings at each sample location, and then taking the square root. Since the variances based on three readings were underestimates of the true variances, they were adjusted upward using the correlations in Table 6-148.

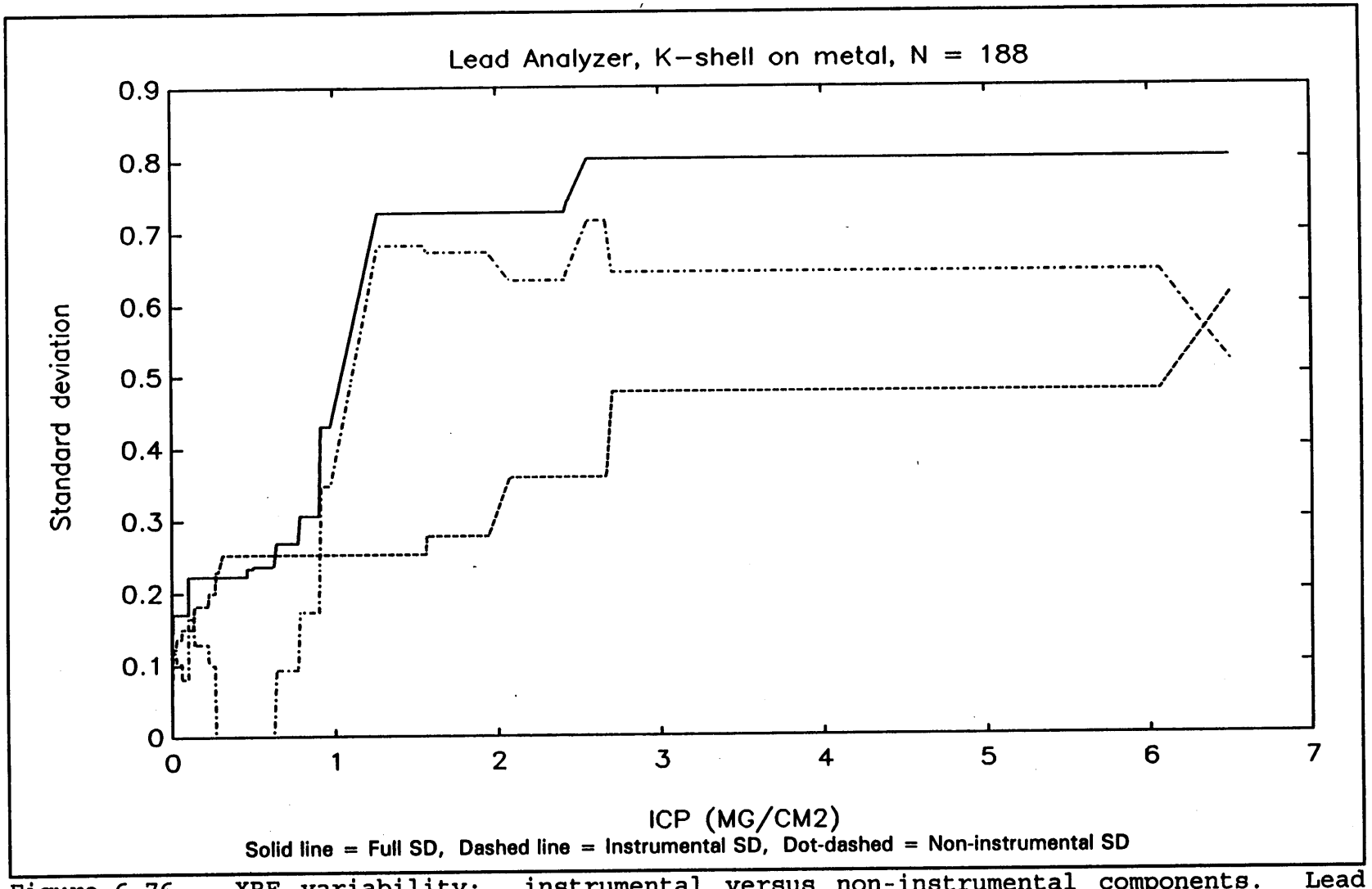


Figure 6-76. XRF variability: instrumental versus non-instrumental components. Lead Analyzer K-shell on metal.

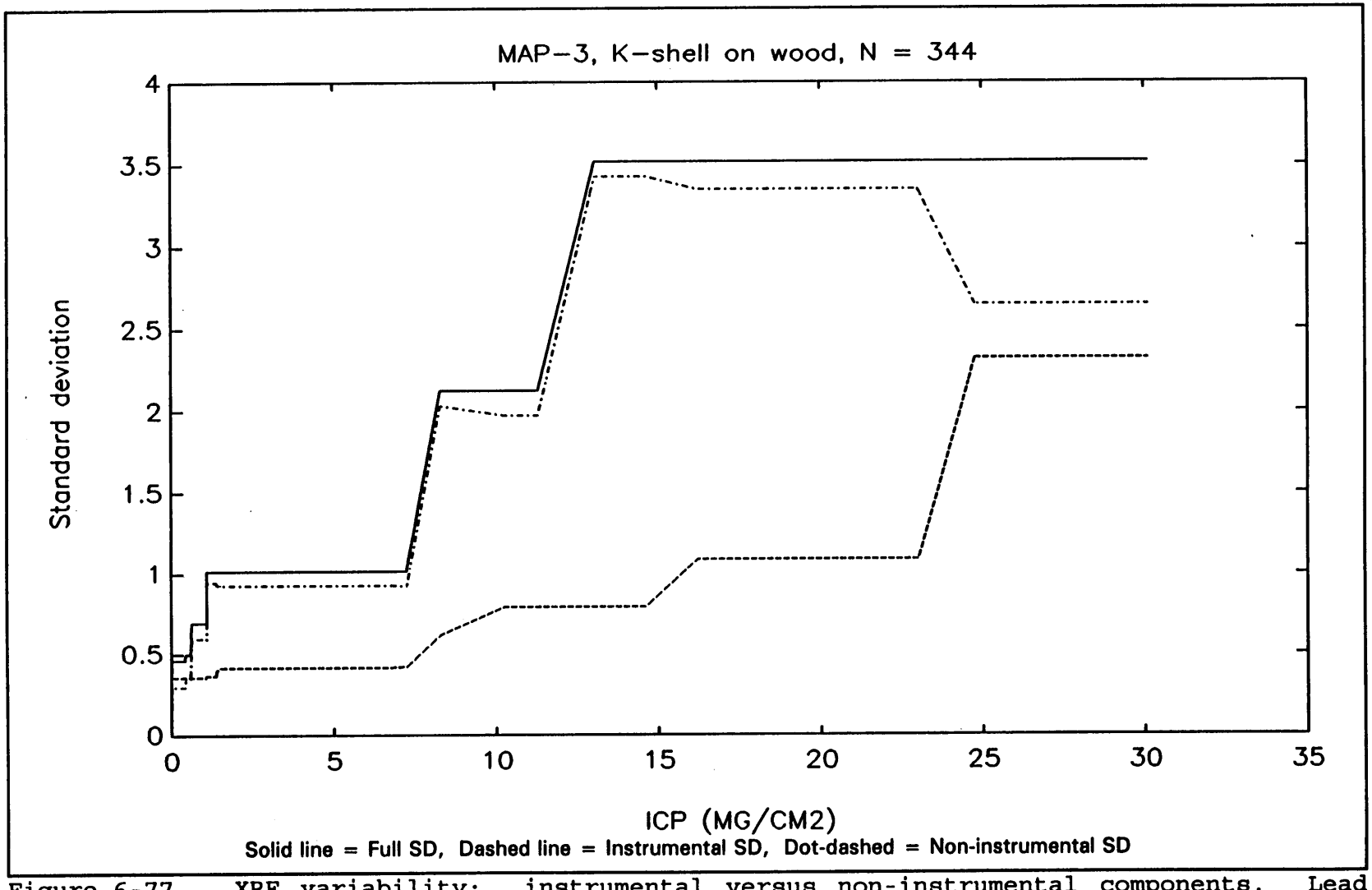


Figure 6-77. XRF variability: instrumental versus non-instrumental components. Lead Analyzer K-shell on wood.

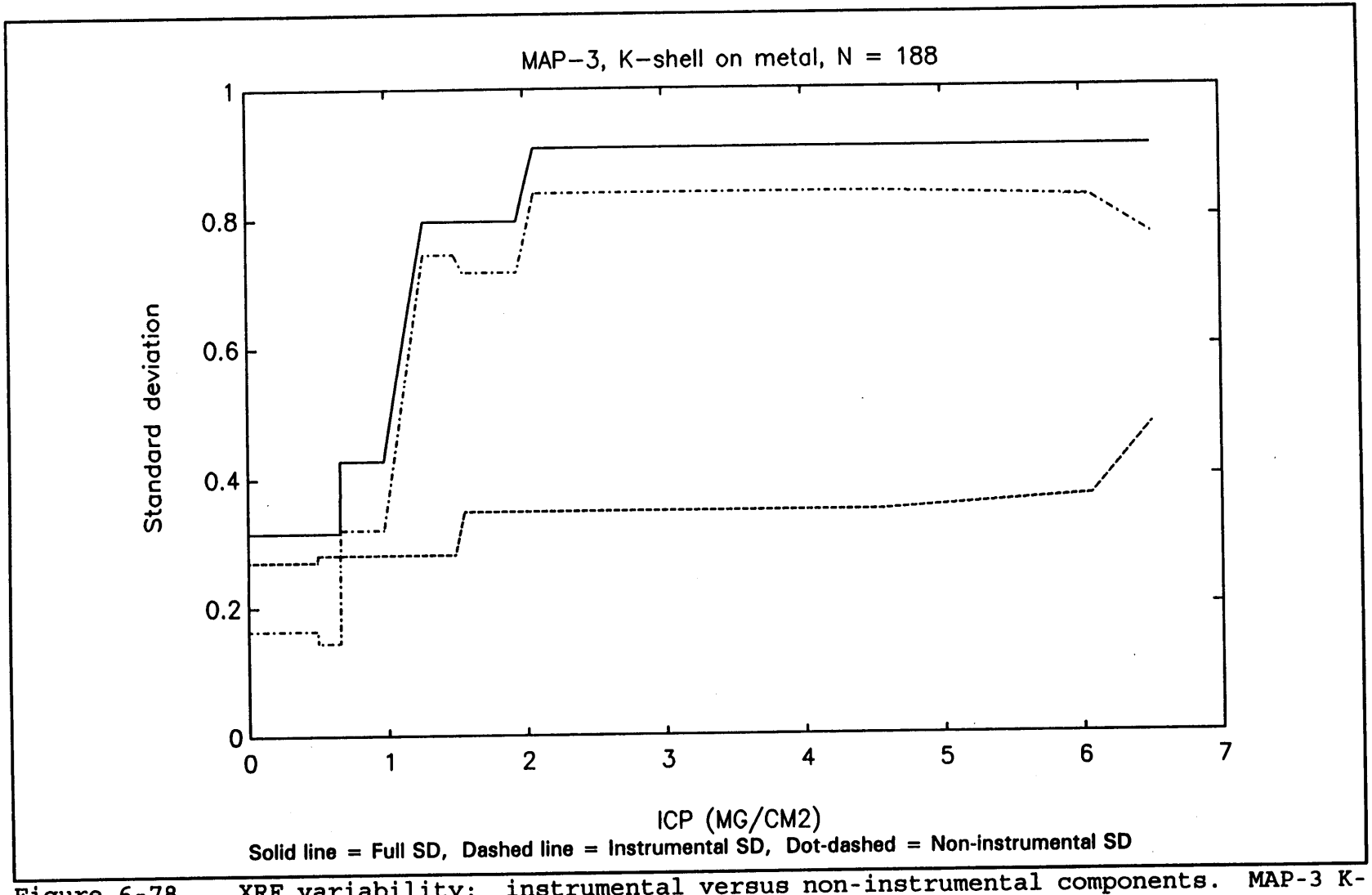


Figure 6-78. XRF variability: instrumental versus non-instrumental components. MAP-3 K-shell on metal.

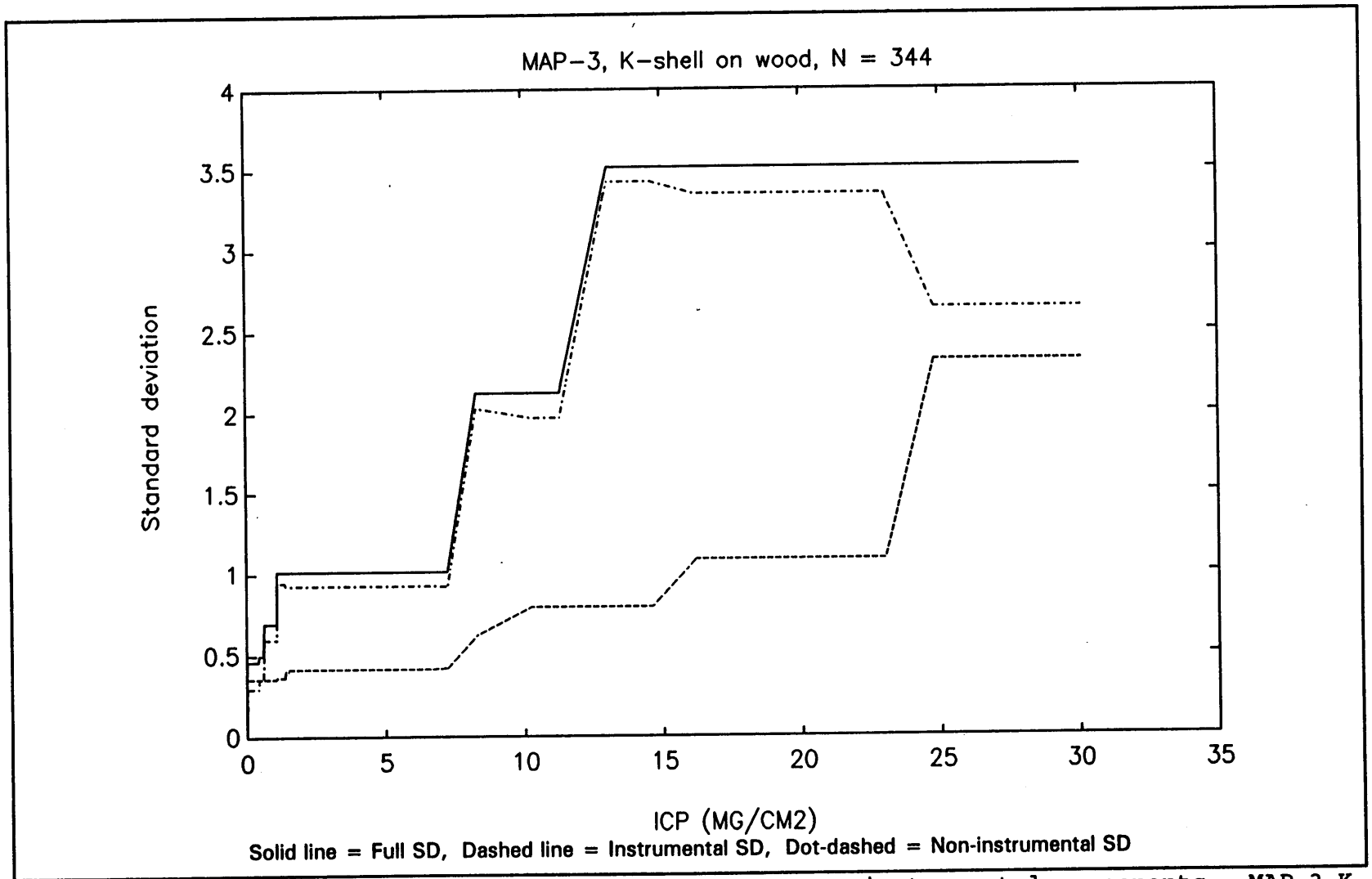


Figure 6-79. XRF variability: instrumental versus non-instrumental components. MAP-3 K-shell on wood.

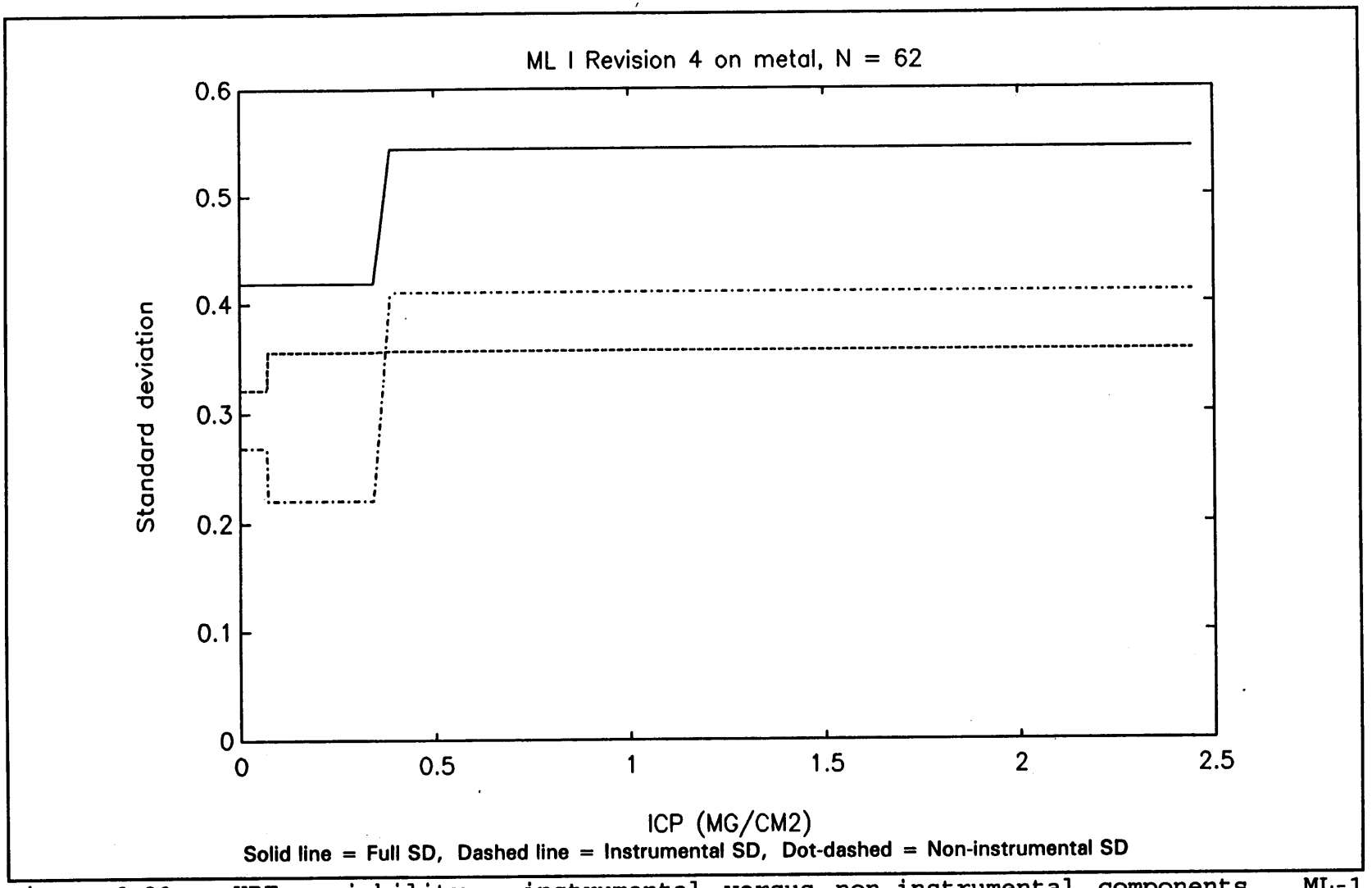


Figure 6-80. XRF variability: instrumental versus non-instrumental components. ML-1 Revision 4 on metal.

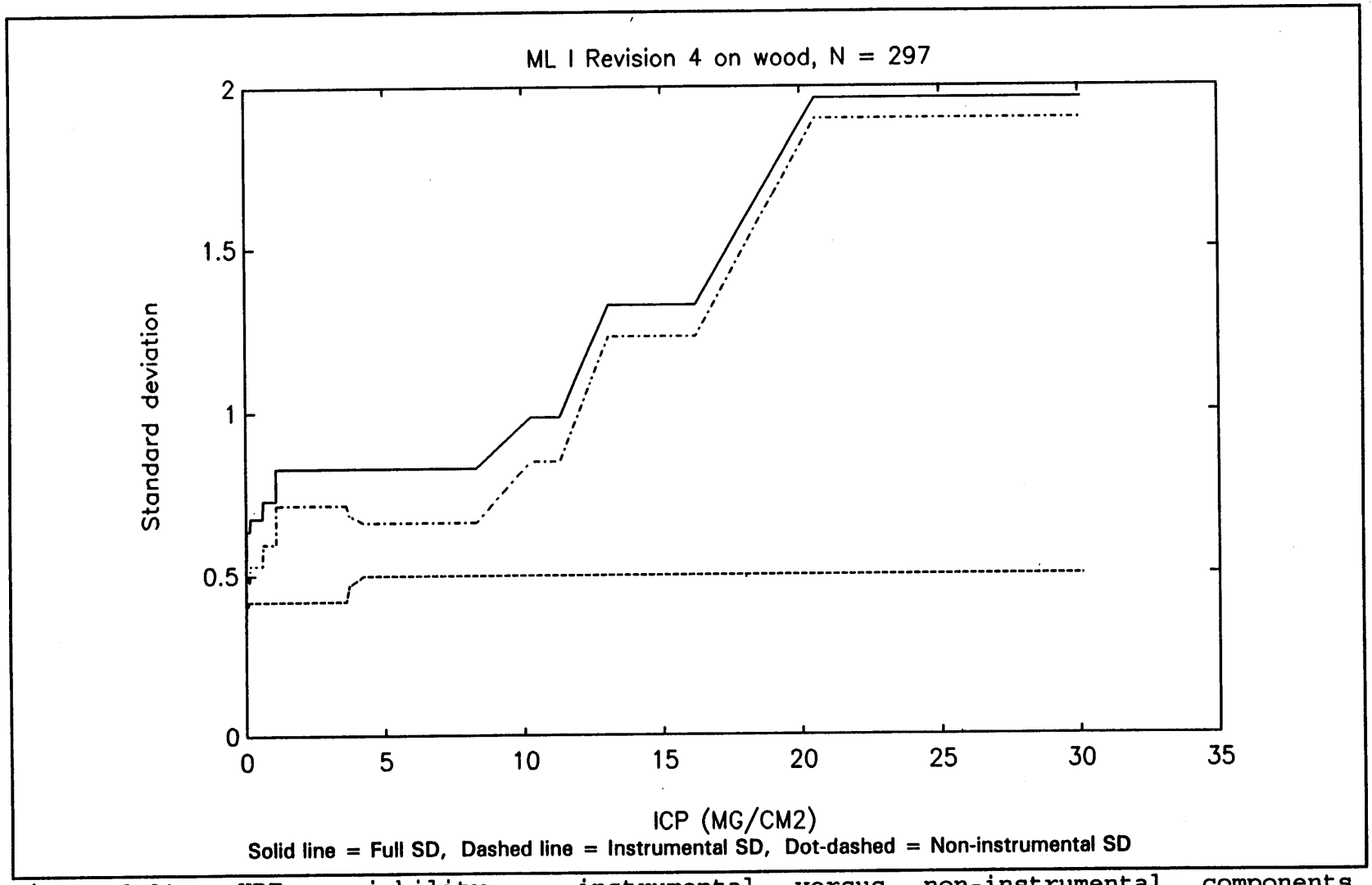


Figure 6-81. XRF variability: instrumental versus non-instrumental components. Microlead I on wood.

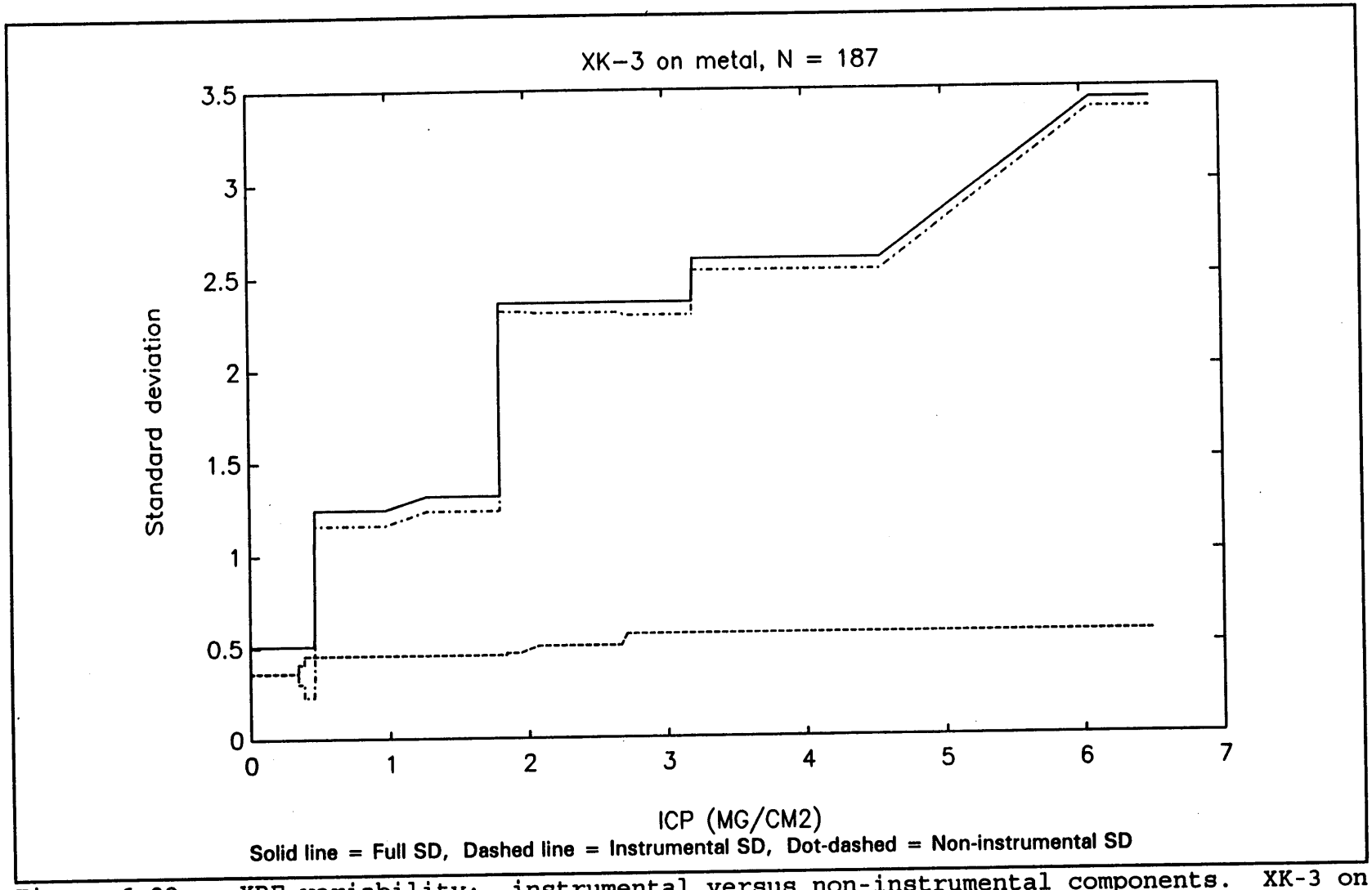


Figure 6-82. XRF variability: instrumental versus non-instrumental components. XK-3 on metal.

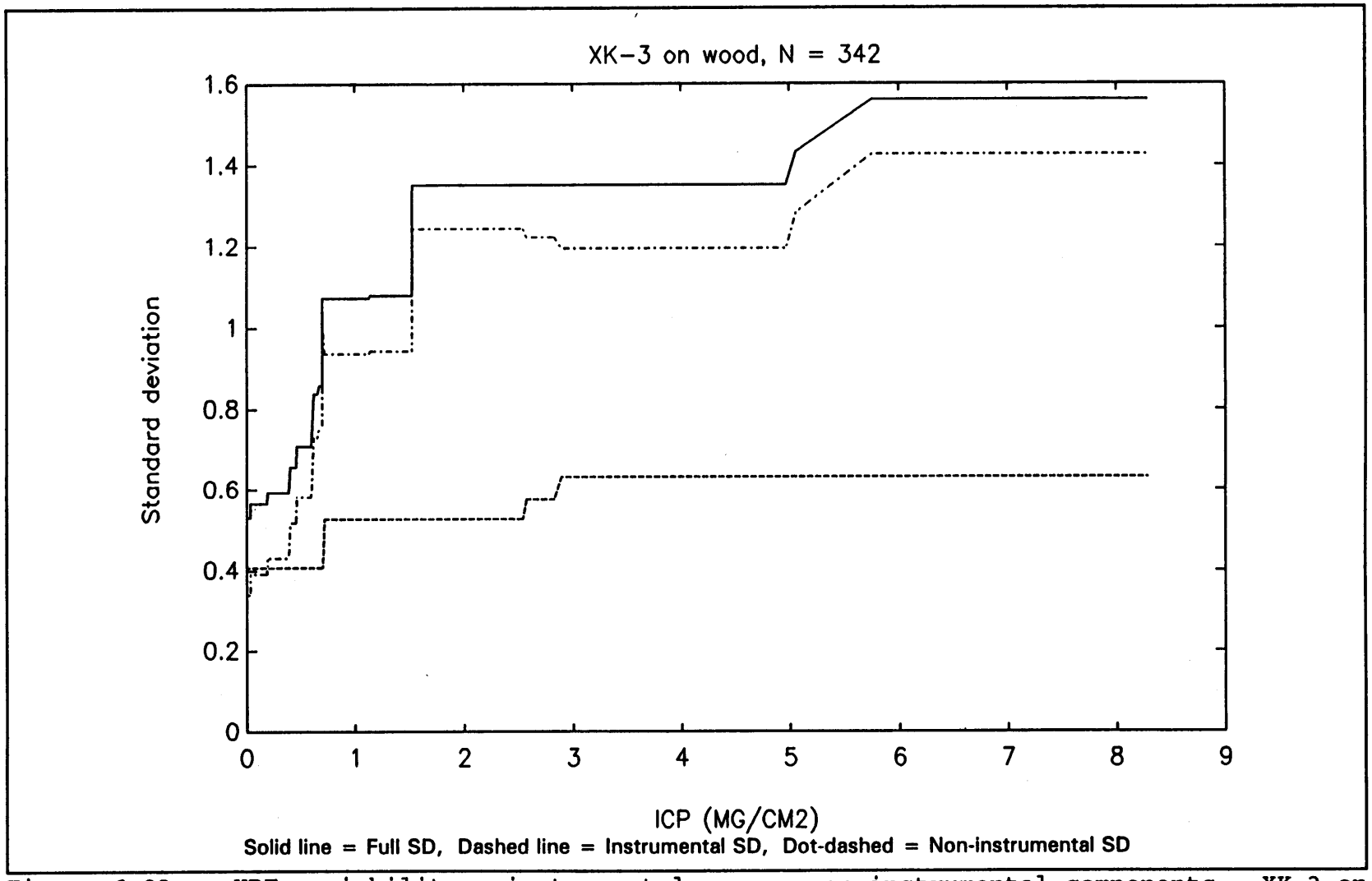


Figure 6-83. XRF variability: instrumental versus non-instrumental components. XK-3 on wood for ICP less than 10 mg/cm².

The dot-dashed line shows the non-instrumental SD. The non-instrumental SD was estimated as zero over a narrow ICP range for the Lead Analyzer (Figures 6-76 and 6-77) where the estimated instrumental SD exceeded the estimated full SD, as explained above.

The full SD estimates are not the same as those presented in section 6.4.4, because they were not derived from the XRF measurement model, which takes into account the combined effect of spatial variation and laboratory error in ICP measurements. The relationship of XRF to ICP measurements, unlike the relationship of XRF measurements to the true lead level, was directly observable, and gave an approximate basis upon which the change in response to the lead level of the different components of variability could be expressed.

Usually, the non-instrumental components of variability were larger than the instrumental components, especially at higher levels of lead. These figures demonstrate that non-instrumental sources of variability, which were not reduced by taking repeated readings at the same place, dominated the XRF measurement error process as the lead level in the paint increased. Combined with the high instrumental correlation between successive readings observed for all instruments with the exception of the MAP-3, repeated readings can be expected to result in only a modest improvement in the precision of measurement. As an example, suppose that the instrumental and non-instrumental SDs are each equal to 0.7 mg/cm^2, and that the average correlation between successive readings is 0.35. This is a fairly typical case. Then the full SD of a single reading is $(0.7^2 + 0.7^2)^{0.5} = 0.99$ mg/cm^2. The full SD of the average of three readings taken at a single location is $[0.7^2 + 0.7^2 \cdot (1/3 + 0.35/3 + 0.35/3)]^{0.5} = 0.88$ mg/cm^2, a reduction of only 11 percent.

6.4.5.5 Conclusions

Sections 6.4.5.2 through 6.4.5.4 illustrate in different, but related ways, that using the average of three successive XRF readings did not substantially reduce the variability compared to using only the first reading. This is the result seen from fitting the XRF measurement model to the average, and comparing SD estimates with those from fitting the model to the first reading. There are two distinct reasons for this: (1) successive readings were not independent, except possibly for the

MAP-3 K-shell; (2) instrumental variability, which is the only kind that can be reduced by taking repeated measurements, was not the only kind that was exhibited. Non-instrumental sources of variability, which were specific to the sample locations, were substantial and dominated instrumental variability as the level of lead in paint increased.

Most instruments exhibited little or no improvement using the average of three readings. Improvements, when realized, were typically less than expected from three independent readings taken under variable field conditions. The ability of an XRF instrument to correctly classify the lead level in paint with respect to the 1.0 mg/cm^2 federal standard would therefore not be expected to improve appreciably by taking the average of three readings.

6.4.6 Correction of XRF Measurements for Bias

The analyses presented in section 6.4.4 demonstrate that every XRF instrument, with the exception of the Lead Analyzer K-shell, was prone to exhibit bias on at least some substrates, or under certain conditions. The L-shell instruments were generally under-responsive to the level of lead in paint: an increase in the lead level by a certain amount led to a smaller increase in the expected XRF reading. This under-responsiveness is reflected in model slope estimates that were less than 1.0, leading to negative bias that became more prominent as the lead level increased. The K-shell instruments, by contrast, usually had slope estimates near 1.0, and exhibited bias mainly as "add-on" effects that are indicated by intercept estimates that differed significantly from 0.0. These effects varied markedly between different machines of the same instrument type, between substrates, and possibly between other factors such as operators, substrate or paint composition.

The use of XRF readings taken on NIST SRM films, with known lead levels, was considered as a means for correcting bias in the regular measurements. Readings on NIST SRM films over control blocks were made at the beginning and end of testing in each unit, and whenever the substrate changed, at lead levels of 1.02 mg/cm^2 (red) and 3.53 mg/cm^2 (yellow), and also on the bare control block. In the full study, paint was removed from the substrate at sampled locations, and readings were made with the red NIST SRM film placed over the bare substrate.

Three strategies for bias correction using red NIST SRM readings were considered. Each used the average of three nominal 15-second readings on the red NIST SRM film as a single measurement:

(1) Control correction used the red NIST SRM measurements for beginning, continuing, and end of day control blocks to calculate a correction factor specific to the unit and the substrate. The average of these measurements minus 1.02 mg/cm^2 was used to correct the regular XRF measurements made in the same unit on the same substrate. For example, if the average of the red NIST SRM measurements on metal control blocks in a particular unit was 1.54 mg/cm^2, the correction factor was 1.54 - 1.02 = 0.52 mg/cm^2. A regular XRF reading of 4.77 mg/cm^2 on metal substrate in the same unit would be corrected to read 4.77 - 0.52 = 4.25 mg/cm^2.

(2) Full correction used the red NIST SRM measurements at each bared sampled location to correct the regular XRF readings individually. Unlike control correction, full correction had the potential to reflect site-specific attributes. It was, however, a destructive and labor-intensive procedure, and is considered here for the sake of comparison. Full correction is not a practical field procedure.

(3) Red NIST SRM average correction was a compromise between control block and full correction. The red NIST SRM measurements at each bared sample locations were averaged by unit and substrate to calculate the correction factors.

As described, red NIST SRM average correction requires the same physical effort as full correction, and is therefore not practical for field use. It was considered because of its similarity to a more practical method that has been proposed, which consists of randomly selecting at most three locations per unit for a given substrate, removing the paint, and making red NIST SRM readings over the bare substrate. There is no appreciable difference between the two methods in their ability to reduce bias, the distinction being that the red NIST SRM average correction factor is based on a potentially larger sample, and should therefore contribute less variability to the corrected XRF measurements than the randomized procedure.

Tables 6-149 through 6-156 show the effects of bias

correction, by machine and substrate, for the eight types of XRF instruments considered in the full study. It is immediately apparent that none of the three correction methods were effective on the L-shell readings. This is not surprising, because the corrections are additive in nature, while the bias of the L-shell instruments consisted primarily in deficient responsiveness to the lead level. Only the K-shell instruments stood to benefit from the three techniques that were considered.

Table 6-149. Effect of Bias Correction Methods on Lead Analyzer, K-shell. Model Estimates of Bias: b(0) = bias at 0.0 mg/cm², b(1) = bias at 1.0 mg/cm².

SUBSTRATE	XRF CODE NUMBER	CORRECTION METHOD (results in mg/cm²)							
		NONE		CONTROL		FULL		RED NIST AVERAGE	
		b(0)	b(1)	b(0)	b(1)	b(0)	b(1)	b(0)	b(1)
Brick	1	0.080	-0.219	-0.100	-0.416	0.003	-0.325	-0.004	-0.312
Concrete	1	0.010	-0.016	-0.054	-0.032	-0.017	-0.018	-0.026	-0.040
Concrete	2	0.066	-0.069	-0.039	-0.121	0.017	-0.138	0.020	-0.123
Drywall	1	-0.018	0.178	-0.095	0.015	-0.106	0.143	-0.129	0.188
Metal	1	0.075	0.037	-0.018	0.022	-0.023	-0.001	-0.076	-0.005
Metal	2	0.096	-0.152	-0.019	-0.194	0.104	-0.136	0.085	-0.121
Plaster	1	0.022	-0.116	-0.060	-0.133	-0.041	-0.009	-0.046	-0.059
Plaster	2	0.060	-0.101	-0.128	-0.243	0.020	-0.118	0.005	-0.097
Wood	1	0.013	0.282	-0.049	0.229	-0.057	0.120	-0.090	0.141

Table 6-150. Effect of Bias Correction Methods on Lead Analyzer, L-shell. Model Estimates of Bias: b(0) = bias at 0.0 mg/cm², b(1) = bias at 1.0 mg/cm².

SUBSTRATE	XRF CODE NUMBER	CORRECTION METHOD (results in mg/cm²)							
		NONE		CONTROL		FULL		RED NIST AVERAGE	
		b(0)	b(1)	b(0)	b(1)	b(0)	b(1)	b(0)	b(1)
Brick	1	0.035	-0.928	-0.003	-0.963	0.043	-0.920	0.043	-0.919
Concrete	1	0.008	-0.818	-0.020	-0.921	0.042	-0.893	0.044	-0.898
Concrete	2	0.016	-0.935	0.011	-0.978	0.063	-0.909	0.061	-0.906
Drywall	1	-0.006	-0.704	-0.047	-0.659	-0.023	-0.595	-0.022	-0.611
Metal	1	0.013	-0.724	-0.031	-0.826	0.050	-0.825	0.050	-0.826
Metal	2	0.019	-0.878	-0.024	-0.867	0.157	-0.907	0.101	-0.797
Plaster	1	0.000	-0.756	-0.039	-0.872	0.027	-0.826	0.034	-0.846
Plaster	2	0.017	-0.918	0.007	-0.907	0.058	-0.856	0.061	-0.863
Wood	1	-0.019	-0.730	-0.062	-0.789	-0.020	-0.776	-0.017	-0.767

Table 6-151. Effect of Bias Correction Methods on MAP-3, K-shell. Model Estimates of Bias: b(0) = bias at 0.0 mg/cm², b(1) = bias at 1.0 mg/cm².

SUBSTRATE	XRF CODE NUMBER	CORRECTION METHOD (results in mg/cm²)							
		NONE		CONTROL		FULL		RED NIST AVERAGE	
		b(0)	b(1)	b(0)	b(1)	b(0)	b(1)	b(0)	b(1)
Brick	10	-0.554	-0.733	-0.515	-0.698	-0.775	-0.997	-0.787	-1.002
Brick	11	-0.616	-0.847	-0.396	-0.631	-0.879	-1.131	-0.887	-1.143
Concrete	10	-0.590	-0.325	-0.540	-0.241	-0.641	-0.463	-0.671	-0.442
Concrete	11	-0.722	-0.616	-0.359	-0.277	-0.786	-0.863	-0.854	-0.834
Concrete	12	-0.766	-0.513	-0.378	-0.015	-0.562	-0.229	-0.549	-0.247
Drywall	10	-0.058	0.209	-0.112	0.039	-0.219	0.258	-0.254	0.215
Drywall	11	0.112	-0.500	0.036	-0.563	-0.150	-0.374	-0.169	-0.416
Metal	10	0.311	0.455	0.171	0.319	0.022	0.094	-0.058	0.095
Metal	11	0.381	0.666	0.114	0.423	0.058	0.147	-0.037	0.271
Metal	12	0.292	0.316	0.178	0.223	0.074	-0.005	-0.023	0.002
Plaster	10	-0.602	-0.438	-0.291	-0.169	-0.617	-0.394	-0.597	-0.457
Plaster	11	-0.550	-0.509	-0.059	0.008	-0.602	-0.498	-0.593	-0.532
Plaster	12	-0.975	-0.709	-0.413	-0.119	-0.731	-0.452	-0.712	-0.478
Wood	10	-0.044	0.383	0.048	0.445	-0.134	0.066	-0.255	0.087
Wood	11	-0.039	0.217	0.102	0.347	-0.252	-0.098	-0.338	-0.101
Wood	12	-0.246	0.546	-0.177	0.653	-0.324	0.123	-0.610	0.168

Table 6-152. Effect of Bias Correction Methods on MAP-3, L-shell. Model Estimates of Bias: b(0) = bias at 0.0 mg/cm², b(1) = bias at 1.0 mg/cm².

SUBSTRATE	XRF CODE NUMBER	CORRECTION METHOD (results in mg/cm²)							
		NONE		CONTROL		FULL		RED NIST AVERAGE	
		b(0)	b(1)	b(0)	b(1)	b(0)	b(1)	b(0)	b(1)
Brick	10	0.034	-0.864	-0.158	-1.052	-0.227	-1.131	-0.228	-1.131
Brick	11	0.025	-0.863	-0.157	-1.046	-0.189	-1.084	-0.191	-1.083
Concrete	10	-0.117	-0.892	-0.334	-1.095	-0.339	-1.127	-0.344	-1.125
Concrete	11	-0.130	-0.812	-0.318	-1.021	-0.255	-1.074	-0.294	-0.971
Concrete	12	-0.195	-1.057	-0.333	-1.209	-0.331	-1.188	-0.328	-1.187
Drywall	10	-0.123	-0.615	-0.284	-0.786	-0.313	-0.711	-0.310	-0.724
Drywall	11	-0.097	-0.656	-0.275	-0.789	-0.277	-0.799	-0.289	-0.729
Metal	10	0.054	-0.662	0.015	-0.702	-0.090	-0.791	-0.099	-0.790
Metal	11	0.252	-0.290	0.178	-0.361	0.063	-0.632	0.006	-0.565
Metal	12	-0.109	-0.868	-0.115	-0.878	-0.156	-0.868	-0.126	-0.891
Plaster	10	-0.112	-0.911	-0.313	-1.093	-0.333	-1.174	-0.328	-1.186
Plaster	11	-0.112	-0.842	-0.273	-1.063	-0.261	-0.956	-0.257	-0.980
Plaster	12	-0.180	-1.010	-0.305	-1.156	-0.327	-1.155	-0.331	-1.150
Wood	10	-0.084	-0.630	-0.176	-0.733	-0.275	-0.864	-0.294	-0.853
Wood	11	-0.074	-0.607	-0.211	-0.751	-0.262	-0.830	-0.274	-0.819
Wood	12	-0.051	-0.886	-0.143	-0.971	-0.190	-1.037	-0.211	-1.033

Table 6-153. Effect of Bias Correction Methods on the Microlead I. Model Estimates of Bias: b(0) = bias at 0.0 mg/cm², b(1) = bias at 1.0 mg/cm².

SUBSTRATE	XRF CODE NUMBER	CORRECTION METHOD (results in mg/cm²)							
		NONE		CONTROL		FULL		RED NIST AVERAGE	
		b(0)	b(1)	b(0)	b(1)	b(0)	b(1)	b(0)	b(1)
Concrete	20 (Den)	-0.030	-0.008	-0.649	-0.671	0.248	0.085	0.174	0.101
Concrete	20 (Phi)	0.589	0.457	-0.426	-0.520	0.253	0.068	0.228	0.107
Concrete	21	0.670	0.595	0.231	0.198	-0.237	-0.366	-0.267	-0.388
Concrete	22	0.892	1.230	-0.653	-0.457	0.067	0.301	-0.009	0.438
Concrete	23	0.110	0.152	-0.369	-0.350	-0.036	0.162	0.014	0.061
Drywall	20	0.004	0.183	0.384	0.450	-0.061	-0.194	-0.145	0.083
Drywall	21	0.202	0.162	0.084	-0.161	0.035	0.189	0.053	0.102
Drywall	22	0.658	1.787	0.464	1.562	-0.229	-0.386	-0.330	-0.321
Metal	20	0.351	0.451	-1.946	-2.307	0.084	0.057	-0.133	0.040
Metal	21	-0.381	-0.174	0.399	0.664	-0.020	-0.299	-0.238	-0.119
Metal	22	1.080	1.361	0.748	1.038	-0.242	-0.178	-0.283	-0.095
Metal	23	-0.415	-0.174	0.043	0.256	-0.225	0.037	-0.266	-0.024
Plaster	20	-0.043	-0.098	-0.513	-0.625	0.110	0.121	0.120	0.050
Plaster	21	-0.035	0.177	0.001	0.092	0.046	0.040	0.017	0.051
Plaster	22	-0.081	-0.316	-1.263	-1.073	-0.173	0.199	-0.250	-0.325
Plaster	23	0.217	0.010	-0.175	-0.393	0.012	-0.097	0.036	-0.135
Wood	20	0.001	0.425	-0.490	-0.424	-0.028	0.071	-0.172	0.087
Wood	21	0.505	0.896	0.598	0.968	0.015	0.067	-0.108	0.072
Wood	22	0.601	0.740	-0.033	0.115	-0.104	-0.062	-0.159	-0.063
Wood	23	0.329	0.743	0.106	0.830	-0.047	0.115	-0.448	0.167

Table 6-154. Effect of Bias Correction Methods on the X-MET 880. Model Estimates of Bias: b(0) = bias at 0.0 mg/cm², b(1) = bias at 1.0 mg/cm².

SUBSTRATE	XRF CODE NUMBER	CORRECTION METHOD (results in mg/cm²)							
		NONE		CONTROL		FULL		RED NIST AVERAGE	
		b(0)	b(1)	b(0)	b(1)	b(0)	b(1)	b(0)	b(1)
Concrete	50	0.045	-0.890	0.002	-0.909	0.002	-0.883	0.001	-0.909
Drywall	50	0.038	-0.739	-0.007	-0.894	-0.034	-0.851	-0.024	-0.918
Metal	50	0.112	-0.769	0.066	-0.799	0.036	-0.815	0.004	-0.867
Plaster	50	0.048	-0.880	0.119	-0.838	0.004	-0.809	0.014	-0.885
Wood	50	0.042	-0.699	-0.031	-0.826	-0.031	-0.772	-0.031	-0.821

Table 6-155. Effect of Bias Correction Methods on the XK-3. Model Estimates of Bias: b(0) = bias at 0.0 mg/cm², b(1) = bias at 1.0 mg/cm².

SUBSTRATE	XRF CODE NUMBER	CORRECTION METHOD (results in mg/cm²)							
		NONE		CONTROL		FULL		RED NIST AVERAGE	
		b(0)	b(1)	b(0)	b(1)	b(0)	b(1)	b(0)	b(1)
Brick	30	1.001	1.329	0.100	0.097	0.110	0.320	0.167	0.074
Brick	31	0.472	0.653	-0.269	-0.031	-0.057	-0.307	-0.100	-0.101
Concrete	30	1.083	1.751	0.559	0.725	0.382	0.076	0.286	0.262
Concrete	31	0.660	0.230	0.049	-0.628	0.122	-0.652	0.100	-0.644
Concrete	32	1.837	2.569	0.150	0.680	0.255	0.453	0.159	0.691
Drywall	30	-0.327	-0.093	-0.392	-0.097	-0.243	-0.209	-0.259	-0.069
Drywall	31	0.245	0.184	-0.285	-0.589	-0.083	-0.219	-0.124	-0.024
Metal	30	0.451	0.856	-0.617	-0.047	-0.029	0.186	-0.193	0.247
Metal	31 (Den)	1.090	1.611	-0.467	0.081	-0.138	0.166	-0.226	0.297
Metal	32	1.480	1.685	-0.043	0.073	-0.055	0.015	-0.120	0.041
Plaster	30	0.538	0.571	-0.210	-0.128	0.030	-0.073	0.012	-0.074
Plaster	31	0.382	0.217	-0.095	-0.249	-0.080	-0.172	-0.068	-0.222
Plaster	32	1.675	1.627	0.355	0.162	0.454	0.260	0.434	0.286
Wood	30	-0.065	0.352	-0.476	0.044	-0.136	0.095	-0.250	0.146
Wood	31	0.339	0.765	-0.276	0.153	-0.093	0.227	-0.120	0.273
Wood	32	0.933	1.227	0.285	0.565	0.287	0.438	0.195	0.430

Table 6-156. Effect of Bias Correction Methods on the XL. Model Estimates of Bias: b(0) = bias at 0.0 mg/cm^2; nonparametric estimates of b(1) = bias at 1.0 mg/cm^2.

SUBSTRATE	XRF CODE NUMBER	CORRECTION METHOD (results in mg/cm^2)							
		NONE		CONTROL		FULL		RED NIST AVERAGE	
		b(0)	b(1)[a]	b(0)	b(1)[a]	b(0)	b(1)[a]	b(0)	b(1)[a]
Brick	41	0.038	-0.337	0.107	-0.276	0.119	-0.284	0.104	-0.275
Concrete	41	0.039	-0.378	0.042	-0.384	0.112	-0.349	0.105	-0.379
Concrete	42	0.054	-0.191	-0.062	-0.259	-0.033	-0.280	-0.023	-0.292
Drywall	41	0.014	-0.564	0.032	-0.657	0.160	-0.357	0.163	-0.378
Metal	40	0.163	-0.480	0.112	-0.494	0.186	-0.522	0.188	-0.516
Metal	41	0.031	-0.623	0.052	-0.599	0.060	-0.527	0.057	-0.562
Metal	42	0.054	0.517	-0.043	0.451	-0.005	0.453	-0.019	0.461
Plaster	40	0.097	-0.481	0.021	-0.614	0.166	-0.447	0.160	-0.405
Plaster	42	0.048	-0.255	-0.087	-0.408	-0.049	-0.337	-0.051	-0.354
Wood	40	0.080[b]	-0.044	0.056[b]	-0.042	0.073[b]	0.001	0.078[b]	-0.011
Wood	41	0.022[b]	-0.363	0.058[b]	-0.330	0.218[b]	-0.276	0.187[b]	-0.283
Wood	42	0.092	-0.483	0.006	-0.563	0.032	-0.577	0.013	-0.560

[a] Nonparametric estimates reported, except for drywall, for which model estimates are reported
[b] Estimates based on sample averages for ICP measurements less than 0.1 mg/cm^2

The Lead Analyzer K-shell (Table 6-149) exhibited little bias without correction, and was neither helped nor harmed appreciably with correction. The MAP-3 K-shell (Table 6-151) exhibited large negative bias on brick, concrete and plaster, and large positive bias on metal and wood, at a lead level of 1.0 mg/cm^2. Control correction was most effective on plaster, mildly beneficial on concrete and metal, and not effective on the other three substrates. Full correction was beneficial on metal and wood, especially at a lead level of 1.0 mg/cm^2. Red NIST SRM average correction mirrored the performance of full correction.

The Microlead I (Table 6-153) exhibited bias on all substrates (with the possible exception of plaster), that varied by machine. The bias was usually positive at a lead level of 1.0 mg/cm^2. Control correction did not reduce the estimated bias, but both full and red NIST SRM average corrections did reduce the bias across both machines and substrates. A reduction of positive bias should help to decrease the frequency of misclassifying paint as over a 1.0 mg/cm^2 threshold when the true paint level is less than that amount.

The XK-3 (Table 6-155) exhibited high, positive bias that varied more between machines than between substrates. Control correction was generally effective in reducing the bias, sometimes substantially. The reduction of high, positive bias should help to reduce the misclassification of paint with low lead levels. Both full and red NIST SRM average correction reduced the estimated bias to a similar extent.

Control correction appeared to benefit the XK-3, and the MAP-3 K-shell on painted metal and plaster surfaces. Full and red NIST SRM average correction performed similarly for the MAP-3 K-shell, Microlead I and XK-3, and appeared to benefit the Microlead I, XK-3, and, on metal and wood, the MAP-3 K-shell.

One aspect of the comparison that was omitted from the analysis is the effect that bias correction had on the variability of XRF measurements. The quantities used in control block and red NIST SRM average correction were sample averages that introduced both additional variability and dependence across readings on the same substrate-unit pair. Accounting for these factors in deriving valid estimates of variability was made difficult by the substitution of ICP measurements for true lead levels. Ignoring the combined effect of spatial variation and

laboratory error in ICP measurements, experience with generalized least squares regression showed that the standard deviations of the corrected XRF readings were sometimes substantially larger than those of the uncorrected readings. Cases where bias correction appeared to have minimal effect were possibly worsened by the increase in variability. Although this issue was not fully explored, it should be considered if the use of a bias correction methodology is contemplated.

6.4.7 XRF Measurement Accuracy: Conclusions

The analyses presented in section 6.4 were aimed at addressing the following two study objectives:

- To characterize the performance (precision and accuracy) of portable XRF instruments under field conditions;

- To evaluate the effect on XRF performance of interference from the material or substrate underlying the paint.

The six XRF instruments evaluated in the full study on two different shells (K and L) gave eight instrument-shell groupings. In section 6.4.4, the accuracy of XRF readings for each of the eight groupings was considered separately, by substrate. Data from the full study established that the K-shell and L-shell instruments shared important similarities within, but not between these classes.

The K-shell instruments were distinguished from the L-shell instruments primarily by their responsiveness to the lead level in paint under field conditions. Responsiveness refers to the property that changes in the lead level are reflected in changes of similar magnitude in XRF readings. Even K-shell instruments that exhibited substantial bias did not exhibit much change in the bias as the lead level changed. The L-shell instruments, by contrast, were under-responsive to the lead level, although certain qualifications apply to the XL, which are summarized below. This ensured that, typically, the L-shell instruments became progressively more biased as the lead level increased. Control block readings for L-shell instruments did not, however, exhibit under-responsive behavior, and created a very different impression of the accuracy of these instruments than what was realized under field conditions.

A factor that was not considered in the analyses is the mass of the paint samples, which affected the performance of the L-shell instruments to a significant degree. The reason for not including paint mass in the analyses is explained in section 6.4.8.1.1. The L-shell instruments were less responsive to changes in the lead level on heavier than on lighter samples, an effect that was not seen with the K-shell instruments. This factor may explain some of the discrepancy between the performance of the L-shell instruments on the field samples and on the control blocks, and it may also explain the emergence of certain other factors (e.g. city) that were confounded with paint mass to various degrees.

The following is a brief description of each of the instrument-shell groupings:

(1) Lead Analyzer K-shell: This performance of the K-shell of this instrument had a number of important distinguishing features. It exhibited the least bias across a wide range of lead levels over all of the instruments, K-shell and L-shell. The magnitude of the bias was typically less than 0.1 mg/cm^2 at the 0.0 mg/cm^2 lead level. At 1.0 mg/cm^2 the bias was a little larger, but usually less than 0.3 mg/cm^2. The variability of its readings, as measured by the standard deviation (SD), was the lowest among all K-shell instruments. Estimates of the SD were typically in the 0.1 to 0.2 mg/cm^2 range at 0.0 mg/cm^2, and 0.2 to 0.4 mg/cm^2 at the 1.0 mg/cm^2 lead level. The performance of this instrument was also the most stable across substrates of all K-shell instruments.

Only two Lead Analyzer machines were used by the same operator, which made it difficult to assess the stability of its performance with respect to machine differences. There was, however, no evidence that the machines performed differently, or that a pronounced difference existed between their use in Denver and Philadelphia.

(2) Lead Analyzer L-shell: The performance of the L-shell of this instrument was typical of the L-shell instruments evaluated in the study. It was minimally biased and exhibited low variability when lead was absent, but it was under-responsive to the lead level as the amount of lead increased. At the 1.0 mg/cm^2 lead level, bias on the order

of -0.7 mg/cm^2 to -0.9 mg/cm^2 was exhibited with this instrument on all substrates. Control block readings, however, only became noticeably biased at 3.53 mg/cm^2. Both the control block and field sample data indicated a flattening of the response at lead levels not much greater than 1.0 mg/cm^2, with increases in the lead level beyond that point reflected in minimal or even no change in the XRF readings, on average. Readings less than 1.0 mg/cm^2 were obtained on field samples with ICP measurements greater than 10.0 mg/cm^2 on all substrates for which such samples were represented in the full study.

The Lead Analyzer L-shell was moderately more responsive to the lead level in Denver than in Philadelphia, and the bias estimates at 1.0 mg/cm^2 obtained for Denver readings were consequently lower. Building or substrate characteristics that distinguish these two cities may have played a role, and may also shed light on the disparity in performance of this instrument on the field samples and on the control blocks.

(3) MAP-3 K-shell: The K-shell of this instrument exhibited prominent negative bias, both on the field samples and on the control blocks, on brick, concrete and plaster substrates. On these substrates the bias was estimated at about -0.5 from the field sample data, with somewhat higher or low bias estimates attributed to specific machines or operators. On metal, the bias was positive and increased with the lead level. At a lead level of 1.0 mg/cm^2, the bias on metal and wood was about 0.4, which again does not account for machine or operator differences. The control block data gave estimates of the bias that were negative and larger in magnitude than those obtained from the field sample data on brick, concrete, and plaster. On metal the control block data, like the field sample data, indicated positive bias. The MAP-3 K-shell had SD estimates in the 0.4 to 0.8 range at 0.0 mg/cm^2, and 10 to 20 percent larger at a lead level of 1.0 mg/cm^2.

Three different MAP-3 machines were used in the study, each by a different operator. It was therefore not possible to separate machine from operator effects. On several substrates differences between the performance of the machines or operators were discerned, which could possibly

be attributable as well to effects associated with non-lead factors in the paint samples. The control block data did not exhibit large differences between the machines. The benefit of correcting MAP-3 K-shell readings with the control block data was seen on metal and plaster, but not on the other substrates. Red NIST SRM average correction was effective on metal and wood.

(4) MAP-3 L-shell: The performance of the L-shell of the MAP-3 resembled that of the other L-shell instruments. It was minimally biased in the absence of lead, although it was somewhat more variable than the other L-shell instruments, with SD estimates in the range 0.1 to 0.4. At increasing levels of lead the instrument was under-responsive, to the effect that the bias became negative and progressively larger in magnitude. At a lead level of 1.0 mg/cm^2 the bias was about -0.6 in Denver, and -0.8 to -1.0 in Philadelphia. Variability of the measurements also increased slightly as the lead level increased.

The MAP-3 L-shell failed almost completely to indicate the presence of high levels of lead. This was true especially of plaster substrates, on which highly leaded paint was found in an old house in Denver. None of the L-shell instruments were able to accurately measure the lead levels in the samples taken. A more general city effect was also seen, with the instrument exhibiting greater responsiveness to lead in Denver than in Philadelphia across a number of substrates, the problem with plaster notwithstanding.

(5) Microlead I revision 4: The Microlead I exhibited prominent bias that was usually positive. The Microlead I, like all of the K-shell instruments, was responsive to lead, suggesting that the bias remained relatively constant over a wide range of lead levels. The Microlead I had SD estimates in the 0.4 to 0.8 range at 0.0 mg/cm^2 of lead that increased slightly as the lead level increased.

Five different Microlead I machines were used by four different operators, with some crossing between machines and operators. These factors, together with substrate and city, substantially affected the bias exhibited by the Microlead I on the field samples, to the extent that broad generalizations about bias having practical value are

difficult to make. Both field sample and control block data exhibited bias, but there was little congruity between the two measurement situations in this respect. Consistent differences between machines and/or operators were detected across substrates on the field samples. Differences between machines were also evident in the control block data, but the pattern did not match that seen in the field sample data. Consequently, there was no indication that the control block data could be effectively used to reduce bias. Full and red NIST SRM average corrections did, however, appear to be effective across machines and substrates with this instrument.

(6) X-MET 880: The performance of this L-shell instrument was similar to that of other L-shell instruments in the full study. Its most important attribute was its under-responsiveness to lead on the field samples, which ensured that readings of the X-MET 880 were more biased at higher lead levels. A significant difference was evident in the responsiveness of the instrument between Denver and Philadelphia. Although the X-MET 880 was more responsive in Denver, the bias remained large, at nearly -0.5 when the true lead level was 1.0 mg/cm^2. The Louisville pilot study evaluated the X-MET 880 with a different radioactive source, and found the instrument to be much more responsive than in the full study. On metal, the bias estimated from the Louisville data was on the order of -0.2 at 1.0 mg/cm^2, which was unusually small for the L-shell instruments that were evaluated in the study. The X-MET 880 exhibited minimal bias on the control blocks, as did the other L-shell instruments.

Only one X-MET 880 machine was used in the full study, by two different operators. Both operator and city effects were evident in the full study field sample data. These effects were exhibited in the responsiveness of the machine to the lead present in paint.

(7) XK-3: This K-shell instrument exhibited substantial positive bias on both the field samples and the control blocks, and the bias increased moderately with the lead level in both measurement situations. The bias varied markedly between both substrates and machines. On brick, concrete, metal, and plaster the XK-3 was prone to exhibit

bias as large as 1.0 or more. Bias was exhibited to a lesser extent on drywall and wood. Unlike the other bias-prone K-shell instruments, the XK-3 showed congruity in performance between the control blocks and field samples, to the extent that using the control blocks to correct for bias had demonstrable merit. Full and red NIST SRM average correction also were effective in reducing bias, with performance that was similar to control correction. The XK-3 had SD estimates in the 0.4 to 0.8 range at 0.0 mg/cm^2, that increased moderately as the lead level increased.

Three XK-3 machines were used by three different operators in the full study, with limited crossing of machines and operators. Prominent effects due to machines or operators emerged, which were consistent across substrates. The control block data reflected similar patterns when summarized by machines within substrates.

(8) XL: This L-shell instrument was different from the other L-shell instruments in several important respects. The XL truncated its readings at 0.0 and at 5.0, and many readings of 0.0 were obtained at low lead levels as a result. The XL was more responsive than other L-shell instruments at lead levels near 1.0 mg/cm^2. Like all L-shell instruments that were evaluated, however, the XL was capable of giving very low readings at high lead levels, in which respect the instrument failed to match the performance of the K-shell instruments that were evaluated.

Three XL machines were used by two operators, with limited crossing between machines and operators. Machine or possibly operator effects emerged from the field sample data, and were exhibited in the control block data as well.

Although the performance of the Lead Analyzer K-shell clearly distinguished itself, the use of its two machines by the same operator may have given it an advantage with respect to the other K-shell machines, where operator effects (or machine effects that are truly operator effects) were exhibited. It is still noteworthy that the low-bias, low-SD performance of the Lead Analyzer was consistent across substrates, and between machines.

The other K-shell instruments exhibited prominent bias. The

consequences of bias for classifying painted locations as above or below 1.0 mg/cm^2 are different depending on whether the bias is positive or negative. In the full study, the distribution of lead levels, as indicated by ICP measurements, was heavily skewed toward the lower end, with lead levels at most locations below 1.0 mg/cm^2. Levels above 1.0 mg/cm^2 were highly dispersed. A negative XRF bias on the order exhibited by the MAP-3 does not affect the correct classification of locations with low lead levels, and with a high dispersion in lead levels above 1.0 mg/cm^2, only marginally diminishes the correct classification rate of those above. From a classification point of view, only lead levels slightly higher than 1.0 mg/cm^2 are adversely affected by small to moderate negative bias. Positive bias of the kind exhibited by the Microlead I and XK-3, on the other hand, has generally worse implications for similar reasons.

The L-shell instruments were negatively biased to a substantial degree, with the possible exception of the XL, and the X-MET 880 under certain conditions. Even at high lead levels, the L-shell instruments in the full study often failed to give readings greater than 1.0 mg/cm^2.

6.4.8 Details and Statistical Methodology

The purpose of this section is to provide details on the data used in describing XRF instrument performance, and the development of the XRF measurement model, which made provision for the combined effect of spatial variation and laboratory error in ICP measurements on the assessment of XRF instrument performance.

6.4.8.1 Non-Lead Factors that Affect XRF Performance

The readings obtained from an XRF instrument may depend on factors in addition to the level of lead present at the sample locations. These non-lead factors include

- the substrate underlying the painted surface;
- pipes, ducts, wires, screening, and other materials underlying the substrate;
- the operator of the instrument;
- the machine (usually distinguished by serial number);
- battery, source age, and source type;
- location or temporal effects that vary in an aggregate way,

and are associated with a unit or a city.

Describing how these non-lead factors affected XRF performance in the full study is important for understanding how an XRF instrument can be expected to perform in practice.

Because the study was not a factorial experiment with respect to these and perhaps other factors that affect XRF performance, it was not usually possible to discretely separate each effect from the others. Moreover, they were confounded to varying degrees with the lead levels at the sampled locations. For example, two machines would be difficult to compare if one were applied mainly to painted surfaces having low lead levels, and the other to painted surfaces having high lead levels.

In spite of this, an attempt to control for factors that affect XRF performance was made. Analyses are presented separately by substrate for each instrument. Within each substrate analyses are presented by machine, and at finer levels of detail (operator within machine, city within operator within machine, etc.) where possible.

City effects, when recognized, refer to factors associated with the units that were sampled in the three cities. Age and the mass of paint samples are examples of factors that may have affected XRF instrument performance, and that were known to vary across field samples grouped by city. There were usually too few data to meaningfully ascribe effects at this low level of detail absent the confounding effects of other factors. City effects may comprise a range of circumstances that are normally encountered in practical testing, in which case combining data across cities would give a useful indication of instrument performance under varying conditions.

The opportunity for detailed analysis varied by the instrument. For example, the X-MET 880 readings were all made on the same machine, making inter-machine comparisons impossible. While the MAP-3 readings were made on three different machines, a different operator was used for each, making it impossible to tell if observed differences were due to the operator, the machine, or both. On some instruments a limited crossing of operators with machines produced too few data to draw useful inferences. The Microlead I readings, by contrast, gave insight into operator within machine, machine within operator, and city

within operator within machine effects on several substrates.

Pooling data across factors is desirable for reaching general conclusions about the performance of an instrument on a substrate type, to account for varying practical conditions, and to give sample size strength to estimates. Where pronounced effects due to operator or machine were indicated, however, the wisdom of such pooling is questionable, since the pooled results may not reflect the performance of any one machine, operated by any one person. Pooled estimates are reported, except where doing so clearly would have failed to reflect how the instrument performed in practice.

6.4.8.1.1. Paint Mass as an Explanatory Factor

It was found that the masses of paint samples affected the performance of all L-shell instruments evaluated in the study. On heavier paint samples, the L-shell instruments were significantly less responsive to the lead level than on lighter samples. When paint mass was included as an additional explanatory variable in models fit to wood substrate data, however, the L-shell instruments were still found to remain highly under-responsive to lead. By contrast, paint mass did not affect the performance of the K-shell instruments to an appreciable degree.

Paint mass, which was considered as a surrogate for thickness, was confounded with other factors, such as the city from which field samples were obtained. On wood substrates, for example, the Philadelphia samples had a significantly higher average paint mass than the Denver samples. This fact reinforces the need for caution when attempting to ascribe apparent differences to factors that were not controlled in the full study.

An objective underlying the analyses presented in this chapter was to explain how XRF instruments performed at fixed lead levels, under practical conditions. Lead level was distinguished from other factors, such as paint mass, machine and operator, by its designation as the explanatory (or independent) variable in the analyses. The other factors were regarded as covariates, representing conditions under which the relationship of XRF readings to the lead level may vary.

The inclusion of covariates in the analyses has the effect of reducing the apparent variability of XRF readings. Whether this reduction in variability is appropriate in describing the performance of an XRF instrument depends on the appropriateness of regarding the covariates as "fixed" under practical conditions. Machines and operators were treated as covariates where it was possible to do so, because the additional variability in XRF measurements arising from the use of different machines or operators in the study would not be realized in situations where one operator used one machine. Paint mass, like the level of paint itself, is not a controllable factor in nondestructive testing. Thus, the variability that paint mass imparted to XRF measurements in the study was considered an aspect of the performance of an XRF instrument.

6.4.8.2 Statistical Description of XRF Performance

This section, which contains 8 parts, describes the methodology and reasoning used to derive the XRF measurement model, which takes into account the fact that the lead levels were only approximately known in the form of laboratory ICP measurements. At the beginning of section 6.4, it was explained that ICP measurements were imperfect substitutes for the true lead levels, because of spatial variation, and laboratory error. A sharply defined relationship between a set of XRF readings and true lead levels may appear less so when the true lead levels are replaced by estimates. An objective in describing the performance of an XRF instrument was to develop a statistical methodology that provided reliable estimates with respect to true lead levels, although the ICP measurements themselves were used.

The development of a model that explains how XRF readings were related to the true levels of lead in paint is presented in three stages. First, a model is described which does not take the imperfect substitution of ICP measurements for true lead levels into account. Second, the impact of this imprecision on assessing the performance of an XRF instrument is considered. Third, and finally, a modification of the model that accounts for this imprecision is presented.

6.4.8.2.1 A Model for the Relationship of XRF to ICP Measurements

The discussion in section 6.4.2.1 suggests that, as an

approximation, a linear response model, with a standard deviation (SD) that increases with the lead level, was a reasonable choice for describing XRF readings as a function of the ICP level. The SD should increase in such a way that it remains positive even in the absence of lead, as measured by the ICP level. A simple specification that incorporates these features has the following form:

$$\mathrm{XRF} = a + b\cdot(\mathrm{ICP}) + , + J\cdot(\mathrm{ICP})$$
$$\mathrm{Var}(,) = c, \quad \mathrm{Var}(J) = d,$$

where , and J represent independent normal random variables. The mean response of XRF at a fixed ICP level is a linear function of ICP given by the expression $a + b\cdot(\mathrm{ICP})$. The variance of XRF at a fixed ICP level is a linear function of ICP squared, given by the expression $c + d\cdot(\mathrm{ICP})^2$, where c is the variance of , and d is the variance of J. The SD, which is the square root of the variance, approaches the form of a linear function of ICP as ICP increases. For ICP = 0.0 mg/cm^2, the SD of XRF readings is equal to the square root of c, while at ICP = 2.0 mg/cm^2, for instance, the SD is given by the square root of $c + 4\cdot d$.

6.4.8.2.2 Sources of XRF Variability

The terms , and J in the model allow for fluctuation of XRF measurements around a "mean response" value for a fixed level of lead, as represented by the ICP measurement. These fluctuations occurred for a number of reasons, the most obvious of which was instrumental error. Repeated XRF measurement under identical conditions did not typically produce identical readings. This was clearly seen in the control block data. Repeated measurement on the field samples at different locations having approximately the same lead levels exhibited not only instrumental error, but fluctuations due to location-specific factors that are less well understood. A detailed discussion of this issue can be found in section 6.4.5.3.

Combining data across machines, operators, or cities often increased the SD estimates for an instrument. Variability due to the pooling of nonhomogeneous data is not characteristic of instrument readings obtained by a single human operator using a single machine at a single place. This again highlights the fact that care must be exercised in combining data across factors. To

some extent, however, such combination was unavoidable.

6.4.8.2.3 Nonparametric Estimation Based on Monotone Regression

It was possible to derive nonparametric estimates of the mean XRF response to the ICP level, and the standard deviation of XRF readings as a function of the ICP level, without resorting to a strictly specified model.

The following two assumptions formed the basis for the derivation of nonparametric estimates: (1) On average, XRF readings did not decrease as the lead level, as measured by ICP, increased; (2) The SD of XRF readings also did not decrease as the ICP level increased.

These assumptions also underlie the derivation of the nonparametric standardized residuals, used both to identify XRF outliers (section 3.2.5) and to calculate correlations between XRF instrument readings (section 3.2.4.3). Monotone regression was the technique used to derive nonparametric estimates that were consistent with the assumptions. Like regular linear regression, monotone regression sought to minimize the sum of squared errors between the actual XRF readings and the estimated mean XRF reading at the observed ICP measurements. But rather than enforcing a constraint that the mean XRF reading be a linear function of the ICP level, the only requirement was that larger ICP measurements could not result in smaller estimates.

Monotone regression is the solution to a quadratic programming problem, and is obtained with the "pool adjacent violators" (PAV) algorithm. The solution takes the form of a step function, formed by averaging data over subgroups in a way that the averages do not decrease. Although a monotone regression cannot be "smooth" in appearance, it will approximate the true mean response if the sample is large, and if the true mean response is itself a nondecreasing function. A full treatment of monotone regression can be found in Barlow, Bartholomew, Bremner, Brunk [8].

Nondecreasing (as a function of the ICP level) SD estimates for XRF readings were also obtained using monotone regression. First, the monotone regression estimates described above were subtracted from the XRF readings, and the differences squared.

Then, monotone regression was applied to the squared differences. The square root of the monotone regression using squared differences was the nonparametric estimate.

The SD estimate, like the estimated mean response, becomes unbiased as the sample size increases, provided that the two assumptions stated above are valid.

6.4.8.2.4 The Effect of Substituting ICP Measurements for the True Lead Levels

The XRF-ICP relationship was not the same as the relationship between XRF measurements and the true levels of lead. This is because the ICP measurements only estimated the true level of lead in paint. Estimates of bias and variability obtained from the observable XRF-ICP relationship give an imperfect picture of how XRF measurements responded to the lead level in the study.

Deriving estimates of bias and variability was difficult for two reasons. The first reason is that the variability exhibited by XRF instruments with respect to the lead level was nonconstant. The second is that the combined spatial variation and laboratory error in ICP measurements had approximately a *log-normal* distribution, while XRF deviations from the mean response appeared to be *normal*, or at least symmetric. Standard techniques developed for the errors-in-variables problem in regression are not applicable to phenomena of this kind. One generalization that does appear to hold is that SD estimates obtained from an XRF-ICP relationship overestimated the true variability present in the corresponding XRF-true lead relationship.

6.4.8.2.5 The Magnitude of Spatial Variation and Laboratory Error in ICP Measurements

Estimates of the magnitude of laboratory error, expressed as standard deviations of the natural logarithm of the ICP level, are presented for ICP laboratory duplicates in section 4.3.1. Estimates for field duplicates, presented in section 4.3.2, reflect laboratory error and spatial variation combined. Estimates are produced separately by city, and by the six different substrates encountered. The distance between field

duplicates was approximately 9 inches in Denver and Philadelphia, and 2 inches in Louisville. For Denver and Philadelphia, field duplicate standard deviations were larger than those for laboratory duplicates.

The full study, however, maintained a distance averaging about 4 inches between the locations of XRF measurement and ICP paint sample removal. For Philadelphia and Denver, interpolation was used to impute standard deviations at 4 inches. For Louisville, where interpolation was not possible, the standard deviation was extrapolated to a distance of 4 inches in a manner similar to the change observed in Denver and Philadelphia. Using the results presented in Tables 4-16 and 4-23, plausible SD values on the logarithmic scale are approximately 0.3 in Denver, and 0.2 in Philadelphia and Louisville.

6.4.8.2.6 The Impact of Substituting ICP Measurements for True Lead Levels: Simulations

A simulation experiment was conducted to assess the consequences of not accounting for imprecision caused by substituting ICP measurements for the true lead levels. The "true" model linking XRF to the lead level used the following specification, which is described in section 6.4.8.2.1:

$$\mathrm{XRF} = a + b\cdot(\mathrm{Pb}) + , + J\cdot(\mathrm{Pb})$$
$$\mathrm{Var}(,) = c, \quad \mathrm{Var}(J) = d,$$

with $a = 0$, $b = 1.2$, $c = 0.01$, and $d = 0.30$. This model is based on behavior exhibited by several of the K-shell instruments on wood substrates. Nonconstant XRF variability is a notable feature of the model, because d is large relative to c. Since Pb (the true lead level) was not observable, a model component linking ICP to Pb is also part of the simulation model. It takes the form

$$log(\mathrm{ICP}) = log(\mathrm{Pb}) + *,$$

where *log* refers to the natural logarithm. The term * is a normally distributed error having mean equal to 0.0 and SD taken at the five values 0.1 through 0.5 in the experiment. Both normal and uniform random variates were generated to simulate *log*(Pb), with mean equal to -2.16 and SD equal to 2.72, and a

sample size of N = 300. These values again were typical of wood substrate analyses. Estimation of the model parameters was based on the 300 pairs of (ICP, XRF) measurements randomly generated according to the model. The method of estimation was normal maximum likelihood, which treated the ICP measurements as if they were the true lead levels.

Table 6-157 gives the results of the simulation experiment, based on 100 replications at each of five error SD levels. In addition, 100 replications were conducted with no random error relating ICP to Pb. This was done to determine the comparable normal maximum likelihood estimates of the parameters for the case where the ICP measurements are regarded as the true lead levels.

As the error SD increased with both normal and uniform distributions of *log*(Pb), bias increased in the estimated model parameters *b* (the slope), and to a greater extent in *d* (the nonconstant variance component). The intercept term *a* and the baseline variance *c* were, however, affected very little. The bias tended to overstate the slope in the XRF-Pb relationship to a small extent, but overstated the variability in a way that became more severe as the lead level increased. Little difference is seen between results obtained for the normal and uniform cases.

Since error SD imputations were in the 0.2 to 0.3 range, the results of this experiment suggest that a failure to account for such error could make an XRF instrument appear to perform worse than it does. With log-normally distributed lead levels for instance, and an error SD of 0.2, the XRF SD at a lead level of 1.0 mg/cm^2 should be close to the square root of 0.010 + 0.373 or .619 mg/cm^2, compared to a true SD of 0.557 mg/cm^2 (the square root of 0.01 + 0.30). With an error SD of 0.3, the XRF SD diverged even more, centering near 0.7 mg/cm^2.

In this experiment, the main consequence of failing to account for the imprecise substitution of ICP measurements for true lead levels was an overstatement of the SD of XRF measurements, especially at higher lead levels, with bias in the mean response a less prominent phenomenon. Bias in the slope parameter *b* is a well-known consequence of regression with errors in the independent variables, and there is an extensive statistical literature that deals with this problem. Results

from the literature, however, assume that variability of the dependent variable remains constant as a function of the independent variable, which was not true in the example presented above.

Table 6-157. Simulation Results (Based on 100 Replications), to Assess the Effect of Spatial Variability and Laboratory Error in ICP Measurements on Model Estimates.

log(Pb) DISTRIBUTION	TRUE VALUE	ERROR SD					
		0.0	**0.1**	**0.2**	**0.3**	**0.4**	**0.5**
Normal	*a*=0.00	-.0003	-.0007	-.0001	.0016	.0023	.0066
	b=1.20	1.1948	1.2046	1.2060	1.2194	1.2374	1.2412
	c=0.01	.0097	.0097	.0100	.0103	.0102	.0104
	d=0.30	.3034	.3197	.3772	.4702	.6288	.8179
Uniform	*a*=0.00	-.0011	-.0003	.0006	.0012	.0033	.0021
	b=1.20	1.1992	1.2044	1.2150	1.2257	1.2485	1.2925
	c=0.01	.0099	.0100	.0101	.0101	.0100	.0101
	d=0.30	.2978	.3163	.3731	.4778	.6522	.8946

An objective of the study was to obtain accurate descriptions of XRF instrument performance, with respect to fixed levels of lead in painted surfaces. In order to meet this objective, it was necessary to develop a methodology that recognized both the imprecise substitution of ICP measurements for true lead levels, and the nature of the relationship between XRF measurements, ICP measurements, and the true levels of lead in paint.

6.4.8.2.7 The XRF Measurement Model

The following model fully describes the XRF-true lead relationship in the presence of spatial variation and laboratory error in ICP measurements:

$$\mathrm{XRF} = a + b\cdot(\mathrm{Pb}) + , + J\cdot(\mathrm{Pb})$$
$$log(\mathrm{ICP}) = log(\mathrm{Pb}) + *,$$
$$\mathrm{Var}(,) = c, \quad \mathrm{Var}(J) = d, \quad \mathrm{Var}(*) = \mathrm{F}_*^2.$$

The terms ,, J and * are normally distributed random variables having zero means and variances as indicated. The true lead Pb is unobservable, and is assumed to have a log-normal distribution with unknown mean and variance. Since ICP is observable, the mean and variance of Pb is estimable, given

knowledge of F_*.

Estimates of the model parameters *a*, *b*, *c* and *d* based on XRF and ICP can be derived using maximum likelihood. This requires expression of the joint density of XRF and ICP, which is an integral that does not have a closed form. To implement maximum likelihood requires the use of numerical integration. It was found that using Riemann sums with 200 equally spaced subintervals achieved a reasonable compromise between accuracy and computational speed to make maximum likelihood practicable. Using 1000 subintervals increased numerical accuracy very little but did increase the computational time substantially in a limited number of cases where it was tried. Newton-Raphson iteration normally provided convergence in less than ten iterations depending on the starting values supplied. The maximum likelihood estimates have approximately normal distributions in large samples. The matrix of second derivatives used in the Newton-Raphson iterations allows standard error estimates of the model parameters to be derived.

A small simulation exercise was conducted to determine how well maximum likelihood can estimate the model of the previous section.

Ten simulations with log-normal Pb and $F_* = 0.2$ produced average estimates $a = .006$, $b = 1.090$, $c = .010$ and $d = .278$. The slope *b* produced the greatest divergence between the estimator and the true value of the parameter (here, equal to 1.2), but variation in the simulated estimates may explain the divergence. The XRF variability parameter estimate *d* appears to have overcome the effect of error caused by substituting ICP for Pb, shown in Table 6-157.

Although the maximum likelihood method as developed is not designed to work with uniformly distributed *log*(Pb), ten simulations show that it not only seems to work well but that it might even work a little better than in the normal case. The average estimates were $a = -.003$, $b = 1.251$, $c = .010$ and $d = .297$. This result is noteworthy, because it indicates that maximum likelihood is not highly sensitive to misspecification of the *log*(Pb) distribution, which is important because departures from normality can be expected.

6.4.8.2.8 Model Limitations

The purpose of the XRF measurement model was to describe, in an approximate way, the behavior of XRF readings in the presence of varying lead levels in paint. The eight instrument classes did not all exhibit similar performance, and performance varied markedly with the substrate. The model did a good job describing XRF behavior on certain substrates, but not on others. When a poor model fit was obtained, it could have been for one of several observable reasons:

1. A small group of data stood out as different from the rest.
2. The XRF-true lead relationship either was not linear, or was linear over a restricted range of lead levels. This was usually true for the L-shell instruments, especially on substrates where high ICP measurements were present.
3. XRF readings were *truncated*, or constrained not to read above or below certain values. Two of the instruments evaluated in the full study produced truncated readings. The XK-3 did not read above 10 mg/cm^2. The XL did not read below 0 mg/cm^2 or above 5 mg/cm^2.

Data anomalies, aside from outliers that were formally identified and removed from the analyses, consisted of near outliers, or isolated groups of data for which it was not possible to tell if the data were unusual, or if the relationship itself may have changed. In the former case, discretion was used in deciding whether or not the anomalies should be removed. In the latter case, and where the global validity of the model was doubtful, analyses on restricted ICP ranges were conducted.

Truncation of XRF readings, especially at the upper end, can make an otherwise linear XRF-true lead relationship take on a nonlinear character. Upper end truncation of the XK-3 and XL instruments was usually seen at ICP measurements much larger than 1.0 mg/cm^2. A model that accounted both for truncation, and the combined effect of spatial variation and laboratory error in ICP measurements, would be complex, and reap very little benefit in describing performance at lower lead levels where interest was primarily focused. Instead, restriction of the data to an ICP range where upper end truncation was infrequent was used to fit the XRF measurement model. Truncation of the XL at 0.0 mg/cm^2 appeared to have little effect on the linearity of the relationship in the low ICP range, except on wood substrates,

where truncated zero readings predominated at ICP levels below 0.1 mg/cm^2.

6.4.8.3 The Analysis of Field Classified Data

Two sets of readings, with different machines and operators, were made at each sampled location with the MAP-3, Microlead I, and XK-3. For these instruments it was possible to assign machines and operators to two field classifications. Direct comparisons between certain machines, operators, and cities were made without the need for fitting models, or accounting for the substitution of ICP measurements for true lead levels. On the other hand, pooling data across field classifications needed to take into account the fact that location-specific sources of variability in XRF readings introduced dependence between the two sets of measurements.

6.4.8.3.1 Analyses Based on Matched Pairs

If two machines of the same instrument model produce XRF readings at the same location, there is a 50-50 chance that one machine will read higher than the other if the machines are indistinguishable in their performance. A simple technique for testing this hypothesis is the *sign test*, which depends only on the sign (positive or negative) of the difference in readings. To illustrate, suppose that in 20 readings on common locations, Machine 1 gave higher readings than Machine 2 on 19 occasions. The *p-value*, or probability that one machine will produce a higher reader than the other on at least 19 occasions assuming that the 50-50 chance hypothesis is correct, is calculated to be $2\cdot(19 + 1)\cdot(0.5)^{20}$, which is less than 40 in one million. This is not a very likely occurrence, suggesting that Machine 1 systematically produced higher readings than Machine 2.

For large sample sizes a normal approximation was used to estimate the p-value. Tied readings (zero differences) were handled with a conditional sign test, using the remaining cases. Correlations of the differences between field classified readings and ICP measurements were calculated to determine if the performance of the two machines relative to each other changed with the lead level.

Comparisons between machines, operators or cities that are not directly matched were sometimes made using *Fisher's exact*

test for 2 by 2 contingency tables. This test required the use of a third machine-operator as a point of common reference. This can be useful for finding effects of one factor within another, and is illustrated in the following example. Operator E of the Microlead I used two different machines (21 and 22) in Denver, matched against Operator G using a third machine (20). On metal substrates Machine 20 was matched against Machine 21 35 times, of which 3 had a higher Machine 20 reading. Machine 20 was matched against Machine 22 24 times (removing ties), and 18 had a higher Machine 20 reading. These results can be presented in a 2 by 2 contingency table as follows:

	Machine 20 smaller	Machine 20 larger
Machines 20 vs 21	32	3
Machines 20 vs 22	6	18

The resulting chi-square statistic for the 2 by 2 table is 27.4, which has a p-value of less than one in ten-thousand, suggesting either that Machine 21 read systematically higher than Machine 22, or that some other factor came into play. Since the sample locations within Denver are non-overlapping in the two comparisons described above, differences in paint samples between units in Denver, for example, may be the reason for the significant chi-square statistic.

6.4.8.3.2 Combining Across Field Classifications

When it is appropriate to do so, pooling data across instruments, operators, and cities is desirable. Pooling within field classifications, which avoids the combination of paired measurements, is straightforward. But, combining paired measurements across field classifications introduces the problem of dependence. The effect of this is difficult to determine on estimates obtained with the XRF measurement model, which assumes that observations are independent.

The problem was avoided by first pooling within field classifications, estimating model parameters, and then averaging the estimates across field classifications. Conservative standard error estimates were obtained using the "triangle inequality", which states that the standard error of the sum of

two estimators is no greater than the sum of the individual standard errors.

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6.5 Comparison of Different Types of XRF Measurements Using Classification Results

This section compares classifications of XRF measurements to classifications of the ICP measurements measured in mg/cm^2 lead. The purpose of this analysis is to examine the accuracy of XRF instruments relative to the ICP measurement and to compare the different types of XRF measurements and addresses the following study objectives:

- to characterize the performance (precision and accuracy) or portable XRF instruments under field conditions
- to evaluate the effect on XRF performance of interference from material (the substrate) underlying the paint
- to evaluate field quality assurance and control methods.

Both the ICP measurement and the XRF measurement were compared by classifying them against the 1.0 mg/cm^2 lead federal standard. Note that "XRF measurement" is a term used for general discussion purposes. In each subsection where a specific classification analysis is discussed, the XRF measurement will be defined as either a single reading, a single reading corrected for substrate bias, an average of three readings, or an average of three readings corrected for substrate bias. Due to the large number of tables presented in this section, tables showing results are not intermingled with text, but instead, tables referenced in a given subsection appear after the text for that subsection.

The previous section of this chapter provided a detailed model-based examination of XRF instrument behavior. Among its findings were that 1) a single reading taken at a sampling location provided almost as much information as an average of three readings taken at that same location, 2) XRF instruments' behavior is influenced by substrate, 3) substrate correction is beneficial in selected cases, 4) the K-shell instruments behave differently from the L-shell instruments, and, 5) XRF instruments may be positively or negatively biased depending on the substrate. The classification results presented in this section provide empirical evidence in support of these findings. However, these results apply only to the set of sampling locations tested in this study. Another set of locations with significantly different lead levels than the tested locations might provide different results, even if the same instruments

were used. Other paint characteristics, such as thicker paint, could also provide different results.

Outliers were not omitted from this analysis.

6.5.1 XRF and ICP Measurement Classification Rules

Both the primary sample ICP measurement and the XRF measurement were classified at each sampling location. For all results provided here, the ICP measurements were always classified using the 1.0 mg/cm^2 lead federal standard. That is, a ICP measurement was classified:

POSITIVE if the ICP measurement was 1.0 mg/cm^2 lead or greater;

NEGATIVE if the ICP measurement was less than 1.0 mg/cm^2 lead.

For a given analysis, the XRF measurements were classified using one of two methods. The first method classified an XRF measurement either positive or negative and the second method added an inconclusive classification. The first method classified the XRF measurements the same way that the ICP measurement was classified as shown above. That is, an XRF measurement was classified using the following rules:

POSITIVE if the XRF measurement was 1.0 mg/cm^2 lead or greater;

NEGATIVE if the XRF measurement was less than 1.0 mg/cm^2 lead.

The second method added an inconclusive range in the classification. An XRF measurement could be classified inconclusive if it fell within a range bounded above and below by pre-specified values. A measurement above the upper bound was classified positive, and one below the lower bound, negative. For this analysis, two sets of bounds were applied. One set of bounds had an upper bound equal to 1.6 mg/cm^2 and a lower bound equal to 0.4 mg/cm^2. The other set had 1.3 mg/cm^2 and 0.7 mg/cm^2 as upper and lower bounds. Specifically, an XRF measurement was classified negative, positive, or inconclusive using the

following rules:

POSITIVE if the XRF measurement was 1.6 (or 1.3) mg/cm^2 or greater,

NEGATIVE if the XRF measurement was 0.4 (or 0.7) mg/cm^2 or less, and

INCONCLUSIVE if the XRF measurements were greater than 0.4 (or 0.7) mg/cm^2 and less than 1.6 (or 1.3) mg/cm^2.

Once the ICP and XRF measurements were classified at each sampling location, the classifications were compared. Several outcomes are possible. Three outcomes, the false negative, false positive, and inconclusive outcomes are presented in detail in this section to describe the behavior of the XRF instruments. For this analysis, rates or percentages were computed for these outcomes. A false negative for an XRF measurement is defined as an XRF measurement classified negative that was taken from a sampling location that had a corresponding ICP measurement classified as positive. A false positive is, conversely, a sampling location with an XRF measurement classified positive and an ICP measurement classified as negative.

Other data presented in the tables in this section are the XRF measurement sample sizes that were classified and compared to the ICP measurement. The sample sizes depend on the number of instruments collecting data, variations in the data collection protocol, and missing data, all of which were described in section 6.1. Three of the XRF instruments represented in this study, the MAP-3, the Microlead I revision 4 (ML I), and the XK-3, each had two different instruments operating at the same time in Denver and Philadelphia. As a result, these three instruments had two results for each sampling location in Denver and Philadelphia. For this analysis, results were obtained for these three XRF instruments by combining all measurements from each pair of instruments prior to computing the misclassification and inconclusive rates. The other XRF instruments were represented in this study by a single instrument at a time, and thus, only one measurement per sampling location was available. The sample size for those instruments that had two different instruments operating at the same time was approximately double the sample size for the other instruments.

Sampling locations from the XRF instruments that had both K-shell and L-shell measurements were further classified into a K-shell and an L-shell instrument, for purposes of analysis. Applying this methodology resulted in eight XRF categories, four K-shell instruments and four L-shell instruments which are presented in the tables in this section.

6.5.2 Classification Results Without an Inconclusive Range

6.5.2.1 Standard XRF Measurements

The first set of tables presented are results for the first standard paint reading as defined in the first section of this chapter. Table 6-158 shows the overall false positive and false negative percentages for the eight XRF instruments based on the first standard paint measurements. In Table 6-158, overall the Lead Analyzer K-shell had the lowest misclassification rates. Among all instruments, only the MAP-3 and Lead Analyzer had both misclassification rates less than 10%, but both rates associated with the Lead Analyzer were less than those associated with the MAP-3. Also, among the K-shell instruments, the Lead Analyzer had the lowest false positive rate. Two other K-shell instruments, the Microlead I and the XK-3, have a lower false negative rate, but the false positive rates for these two instruments were 20.3% and 39.7%, respectively. Excluding the Lead Analyzer and the MAP-3, the other XRF instruments had at least one misclassification rate greater than 20%, ranging from 20.3% to 89.1%.

Tables 6-159 and 6-160 show the same information for four categories of ICP measurements. These two tables indicate where the misclassification errors are occurring relative to the ICP measurement. For all sampling locations with ICP measurements less than 0.1964 mg/cm^2 (the median of the 1,290 ICP measurements), Table 6-159 shows a difference between the L-shell and K-shell instruments. Overall, the false positive rates for the L-shell instruments range from 0.0% to 0.8% and the for the K-shell instruments the false positive rates range from 1.0% to 64.2%. Comparisons between L-shell and K-shell instruments from sampling locations with results in the median ICP measurement to 1.0 mg/cm^2 range show greater differences. In this ICP measurement range, the false positive rates for the L-shell instruments range from 0.0% to 1.0% and the for the K-shell

instruments the false positive rates range from 6.6% to 64.2%.

In contrast, a different relationship between the L-shell and K-shell instruments is shown in Table 6-160. For all sampling locations with ICP measurements greater than or equal to 1.0 mg/cm^2 lead but less than 0.24891 (the 90th percentile of the 1,290 ICP measurements), Table 6-160 shows that false negative rates for the L-shell instruments range from 50.8% to 96.6% and the for the K-shell instruments the false negative rates range from 4.1% to 12.0%. From sampling locations with results greater than the 90th percentile, the table shows that false negative rates for the L-shell instruments range from 31.4% to 82.2% and the for the K-shell instruments the false negative rates range from 0.0% to 4.0%. Thus, comparisons of these two tables illustrates differences between the K-shell and L-shell instruments. The higher misclassification errors for the K-shell instruments occur at lower ICP measurements shown in Table 6-159. This is in contrast to the L-shell instruments which have higher misclassification errors from locations with higher ICP measurements shown in Table 6-160.

The information in Tables 6-158 and 6-159 is presented graphically in Figures 6-84 through 6-91, for each XRF instrument classification. In these figures, each horizontal bar in the graphs corresponds to one of the four ICP measurement categories shown in Tables 6-159 and 6-160. The top bar, labeled "high neg", represents XRF data collected at sampling locations with corresponding ICP measurement less than the ICP measurement median (0.1964 mg/cm^2). The next bar down ("low neg") represents XRF data collected at sampling locations with corresponding ICP measurement equal to or greater than the ICP measurement median (0.1964 mg/cm^2) but less than 1.0 mg/cm^2 lead standard. The third bar from the top ("low pos") represents XRF data from sampling locations equal to or greater than 1.0 mg/cm^2 lead but less than the ICP measurement 90th percentile, 2.4891 mg/cm^2. Finally, the bottom bar ("high pos") represents XRF data from sampling locations equal to and greater than the 90th percentile. Overall frequency and percent of sampling locations by ICP measurement category is given in the figures as "FREQ." and "PCT.", respectively.

Each bar is divided into "AGREE" (no shading) categories and "DISAGREE" (black shading) categories. A sampling location is categorized as *agree* if the XRF measurement provides the same

classification of the sampling location relative to the 1.0 mg/cm^2 lead standard as does the ICP measurement. In other words, the classification provided when the XRF measurement and ICP measurement agree. A sampling location is categorized as *disagree* if the XRF measurement, or first standard paint reading in this case, provides a different classification of the sampling location relative to the 1.0 mg/cm^2 lead standard than does the classification provided by the ICP measurement. That is, a sampling location is categorized as disagree if the XRF measurement is greater than 1.0 mg/cm^2 lead and the ICP measurement is less than 1.0 mg/cm^2 lead or vice versa.

Figures 6-84 through 6-91 clearly illustrate the differences between K-shell and L-shell instruments. Most of the time the K-shell instruments' first standard paint readings where able to correctly classify the high levels of lead whereas those from the L-shell instruments had high false negative rates for high levels of lead. This can be observed in Figures 6-84 through 6-91 by comparing the *disagree* categories (black shading) that occurred on the bottom two bars. The L-shell instruments show a greater frequency of false negative results (a greater amount of black shading) than do the K-shell instruments.

To further illustrate differences between K-shell and L-shell instrument results, a nonparametric statistic was computed to measure the amount of *agreement* between two XRF instruments. First, the first standard paint readings were classified negative or positive relative to the 1.0 mg/cm^2 lead standard as described above. Next, the results from one instrument were cross-tabulated against the results of another instrument and computed from each cross-tabulation result was an agreement statistic, *K* [13], which was used to compare the one XRF instrument to another. The agreement statistic was computed for all pairs of XRF instruments with first standard paint readings from sampling locations with corresponding ICP measurement greater or equal to its 90th percentile (2.4891 mg/cm^2) and the results are given in Table 6-161. The Microlead I did not have any negative classifications for its first standard paint readings from sampling locations used in this analysis.

Interpreting K depends on its sign and magnitude. The sign measures agreement or disagreement. For example, a +1.0 indicates total agreement and a -0.17 indicates disagreement but

less than total disagreement. Herein lies the limitation of the agreement statistic, *K*. The interpretation of K does not allow quantitative measures of the relative amounts of agreement or disagreement. However, differences can be observed by comparing the K-shell instruments to L-shell instruments using the K statistics provided in Table 6-161. The K statistics computed between one K-shell instrument and another K-shell instrument were an order of magnitude greater than the K statistics computed from a K-shell and L-shell instrument pairing. Similar results can be observed by comparing results from the pairing of two L-shell instruments to the results from a K-shell and L-shell instrument pairing. Therefore, Table 6-161 provides additional evidence that the first standard paint readings were similar among K-shell instruments and dissimilar from L-shell instruments and vice versa. However, one K-shell instrument, the XK-3, showed differences from the other K-shell instruments. The XK-3 had negative agreement statistics computed for the other K-shell instruments whereas all of the agreement statistics for the other K-shell instruments among each other, except for the XK-3, were positive.

Tables 6-162 through 6-169 provide the misclassification rates for each XRF classification by substrate. Results were fairly consistent across all substrates for the Lead Analyzer. The results for the other instruments were more variable by substrate.

6.5.2.2 First Standard Paint Reading Versus Average of Three Readings

The averages of the three standard paint readings at a sampling location were classified. Table 6-170 is analogous to Table 6-158 except that it provides results for the average of the three paint readings. Likewise, Tables 6-171 through 6-178 provide the same information for the eight XRF classifications by substrate.

For the K-shell instruments the error percentages (false positive and false negative) are the same in one case, slightly larger for the average in one case, and slightly smaller for the average in six cases. For the L-shell instruments the error percentages are the same in three cases, while the average is slightly superior in the remaining cases. In no case, either for K- or L-shell instruments, does the average represent a

significant improvement over the first standard paint reading. In particular, cases where the error rate was high, (the false negative rates for the L-shell instruments and false positive rates for the Microlead I and XK-3), the error rates were only very minimally improved by use of the average of three readings.

Comparisons by substrate were made by comparing Tables 6-162 through 6-169 to Tables 6-171 through 6-178. For the four K-shell instruments, there are a total of 44 error percentages when broken down by substrate. In 26 cases, the error percentages for the average are smaller, in seven cases they are larger, and in 11 cases no change occurred between the average and the error percentages for the first standard paint readings. For the L-shell instruments, 32 cases are the same, 10 show the average as better, and two show the first standard paint reading as better. In no case was any improvement of the average over the first standard paint reading significant. For example, there are 31 error rates exceeding 10%; of these, only two were reduced below 10% by use of the average, and both improvements were small (11.1 % versus 10.2% false negative rates for the Lead Analyzer K-shell on concrete and 7.4% versus 6.8% false negative rates for the Microlead I on plaster).

The conclusion from examining these classification results is that, although use of the average of three 15-second readings may result in more accurate classification of paint than the use of only a single reading, the likelihood of improvement is small. In any case, improvement was always too small to be of practical significance. Thus, it appears that the additional effort involved in taking three 15-second readings at a sampling location versus only one is not justified by an increase in the accuracy of classification of paint. Experience in the field in this study suggests that approximately 50% of on-site time is spent taking XRF readings. Thus, reducing the number of readings from three to one would reduce inspection time in the field on the order of 33%.

The conclusion that there is very little difference in the accuracy of paint classification between a single reading and the average of three readings is somewhat paradoxical. The expectation that the average will perform much better than a single reading is based on the statistical fact that the variance of the average of three <u>independent</u> readings is one-third the variance of a single reading, so that the average is much more

precise than a single reading. This expectation is not borne out by the XRF data for two reasons. First, for most instruments, successive readings taken at the same point are positively correlated, so that the independence assumption is violated. Thus, the gain in precision from taking repeated readings is generally much less than if the readings were independent. Second, taking repeated readings and averaging them reduces only the component of variability due solely to the performance of the instrument ("instrumental variability"). As shown in section 6.4, the study data demonstrates clearly that there are additional sources of variability in XRF readings that are generally at least as large as the instrumental component. Taking repeated readings cannot reduce the impact of these additional sources of variation. The additional variation is due to location-specific factors such as paint and substrate composition. Much greater detail on this issue can be found in section 6.4 of this chapter. However, another discussion comparing a single reading to an average of three readings with the addition of the inconclusive range is found in section 6.5.3.3.

6.5.2.3 Impact of Correcting for Substrate Bias

The tables given in the last two sections that break down results by substrate (Tables 6-162 through 6-169 and 6-171 through 6-178) illustrate the effect that the underlying substrate can have on classifying XRF measurments. The next set of tables provide error percentages for XRF readings after they have been *corrected for substrate bias*. A single XRF reading was "corrected" by subtracting a known offset value. For this analysis, the first standard paint reading at a sampling location was corrected. There are three types of corrections as defined in section 6.1:

- control correction
- full correction
- red NIST SRM average correction.

Discussions comparing a single reading to corrected readings are found in this section. Discussions comparing a single reading to corrected readings with the addition of the inconclusive classification are found in section 6.5.3.3.

6.5.2.3.1 Impact of Control Correction

The first standard paint readings were "control corrected" by subtracting the average of all the initial and end red NIST SRM control block measurements in the dwelling, minus 1.02 mg/cm^2. Table 6-179 shows the overall false positive and false negative percentages for the eight XRF instruments based on the first standard paint reading control corrected for all locations. Tables 6-180 and 6-181 show the same information for four categories of ICP measurements. Tables 6-182 through 6-189 break down the results for the first standard paint measurement by substrate.

Table 6-179 shows that for the L-shell instruments, control correction is ineffective since false negative rates remained high. For the Lead Analyzer K-shell, there was little impact; the false positive rate decreased slightly and the false negative rate increased slightly. Similarly for the MAP-3 K-shell; the false positive rate increased slightly and the false negative rate decreased slightly. However, a high false positive rate on metal and high false negative rates on concrete and plaster were all reduced by control correction as shown in Tables 6-164 and 6-184. For the Microlead I, control correction was ineffective; the false negative rate increased five fold to 18.3% and the false positive rate decreased to 12.5%. The increase in the false negative rate was due to the high false negative rates on concrete, metal, and plaster shown in Table 6-186. For the XK-3, high false positive rates were reduced, but at the expense of a substantial increases in the false negative rates for metal, plaster, and wood as shown in Table 6-188.

The results shown in Tables 6-180 and 6-181 are analogous to the results shown in Tables 6-159 and 6-160. Comparisons of Table 6-159 to Table 6-180 show that the control correction greatly improved the XK-3 performance in the ICP measurement range 0.0 to 0.1964 mg/cm^2 (the ICP measurement median) and showed substantial improvement in the ICP measurement range 0.1964 to 1.0 mg/cm^2 lead. The Microlead I showed some improvement in the 0.0 to 0.1964 mg/cm^2 range. However, comparisons of the results in Table 6-160 with those in Table 6-181 show that the false negative rate for the Microlead I increased for ICP measurements equal to or greater than 1.0 mg/cm^2 lead.

6.5.2.3.2 Impact of Full Correction

The first standard paint reading was "fully corrected" by subtracting the average of the three standard red NIST SRM readings taken at the same location, minus 1.02 mg/cm^2. Table 6-190 shows overall error rates by instrument for the first standard paint reading fully corrected. Tables 6-191 through 6-198 break down the information by substrate.

For the L-shell instruments, full correction was ineffective since false negative rates still remain high. Similarly, for the Lead Analyzer K-shell, there was little impact; error rates were low before correction and decrease slightly after correction. For the MAP-3 K-shell, full correction was effective on some substrates. High false negative rates on concrete and plaster were not reduced by full correction but false positive rates on metal and wood were substantially reduced as shown in Tables 6-164 and 6-193. For the XK-3, full correction was effective; false positive rates were substantially reduced without an unacceptable increase in false negative rates. It must, of course, be remembered that full correction is never a practical field procedure.

6.5.2.3.3 Impact of Red NIST SRM Average Correction

The first standard paint readings were corrected using red NIST SRM average correction. This was done by subtracting from the first standard paint reading, the corresponding substrate average of all red NIST SRM readings taken at the sample locations in the dwelling, minus 1.02 mg/cm^2. Table 6-199 shows overall error rates by instrument for the first standard paint reading red NIST SRM average corrected. Tables 6-200 through 6-207 break down the information by substrate. The impact was very similar to full correction.

Table 6-158. First Standard Paint Reading Without an Inconclusive Range.

XRF	Sample Size	% False Positive	% False Negative
Lead Analyzer K-shell	1,190	3.1	5.9

Lead Analyzer L-shell	1,190	0.0	89.1
MAP-3 K-shell	2,367	8.0	8.3
MAP-3 L-shell	2,367	0.9	69.7
Microlead I K-shell	2,475	20.3	3.8
X-Met 880 L-shell	1,174	0.0	87.1
XK-3 K-shell	2,478	39.7	3.6
XL L-shell	1,189	0.5	41.8

Table 6-159. False Positive Results for First Standard Paint Readings Without an Inconclusive Range, Categorized by Their Corresponding ICP Measurement Above and Below the 0.1964 mg/cm^2 Median of the 1,290 ICP Measurements.

XRF	Sample Size	ICP Measurement Range (mg/cm^2)	% False Positive
Lead Analyzer K-shell	608	[0 - median)	1.0
	362	[median - 1.0)	6.6
Lead Analyzer L-shell	608	[0 - median)	0.0
	362	[median - 1.0)	0.0
MAP-3 K-shell	1,209	[0 - median)	4.6
	723	[median - 1.0)	13.6
MAP-3 L-shell	1,209	[0 - median)	0.8
	723	[median - 1.0)	1.0
Microlead I K-shell	1,252	[0 - median)	15.8
	754	[median - 1.0)	27.7
X-Met 880 L-shell	596	[0 - median)	0.0
	361	[median - 1.0)	0.0
XK-3 K-shell	1,251	[0 - median)	24.9
	754	[median - 1.0)	64.2
XL L-shell	607	[0 - median)	0.2
	362	[median - 1.0)	1.1

Table 6-160. False Negative Results for First Standard Paint Readings Without an Inconclusive Range Categorized by Their Corresponding ICP Measurement Above and Below the 2.4891 mg/cm^2 90th Percentile of the 1,290 ICP Measurements.

XRF	Sample Size	ICP Measurement Range (mg/cm^2)	% False Negative
Lead Analyzer K-shell	118	[1.0 - 90th %tile)	9.3
	102	[90th %tile - ∞)	2.0
Lead Analyzer L-shell	118	[1.0 - 90th %tile)	96.6
	102	[90th %tile - ∞)	80.4
MAP-3 K-shell	233	[1.0 - 90th %tile)	12.0
	202	[90th %tile - ∞)	4.0
MAP-3 L-shell	233	[1.0 - 90th %tile)	77.7
	202	[90th %tile - ∞)	60.4
Microlead I K-shell	240	[1.0 - 90th %tile)	7.5
	229	[90th %tile - ∞)	0.0
X-Met 880 L-shell	116	[1.0 - 90th %tile)	91.4
	101	[90th %tile - ∞)	82.2
XK-3 K-shell	242	[1.0 - 90th %tile)	4.1
	231	[90th %tile - ∞)	3.0
XL L-shell	118	[1.0 - 90th %tile)	50.8
	102	[90th %tile - ∞)	31.4

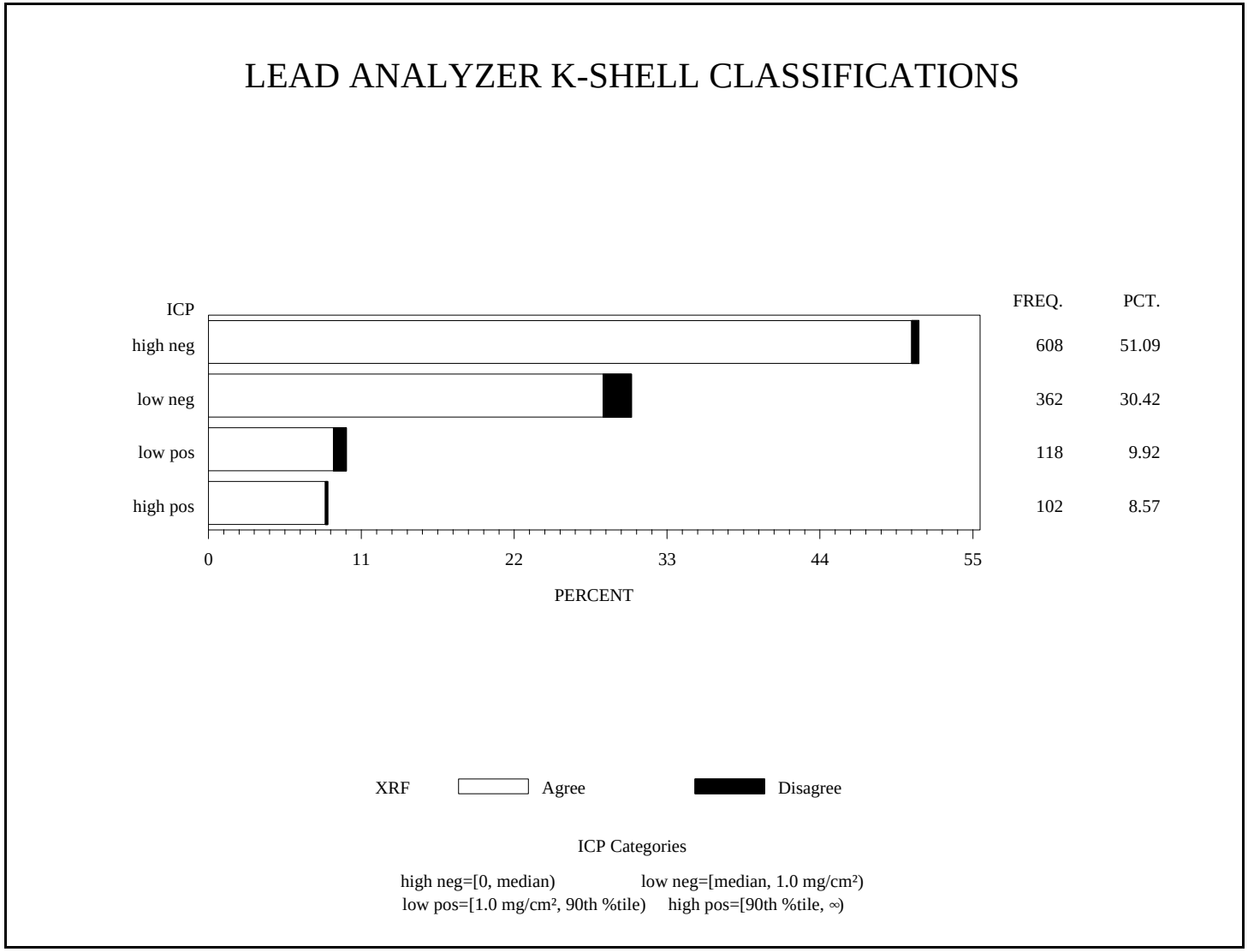

Figure 6-84. Bar chart of classifications by laboratory ICP categories for Lead Analyzer K-shell, no inconclusive range.

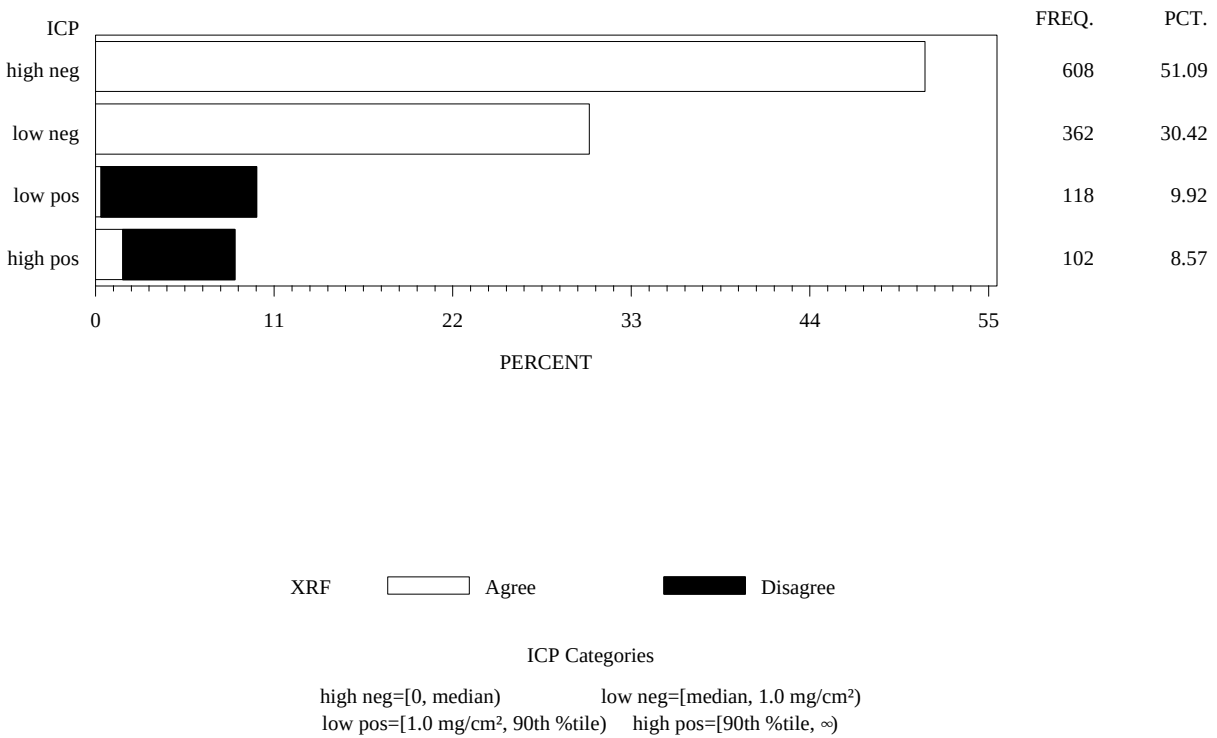

Figure 6-85. Bar chart of classifications by laboratory ICP categories for Lead Analyzer L-shell, no inconclusive range.

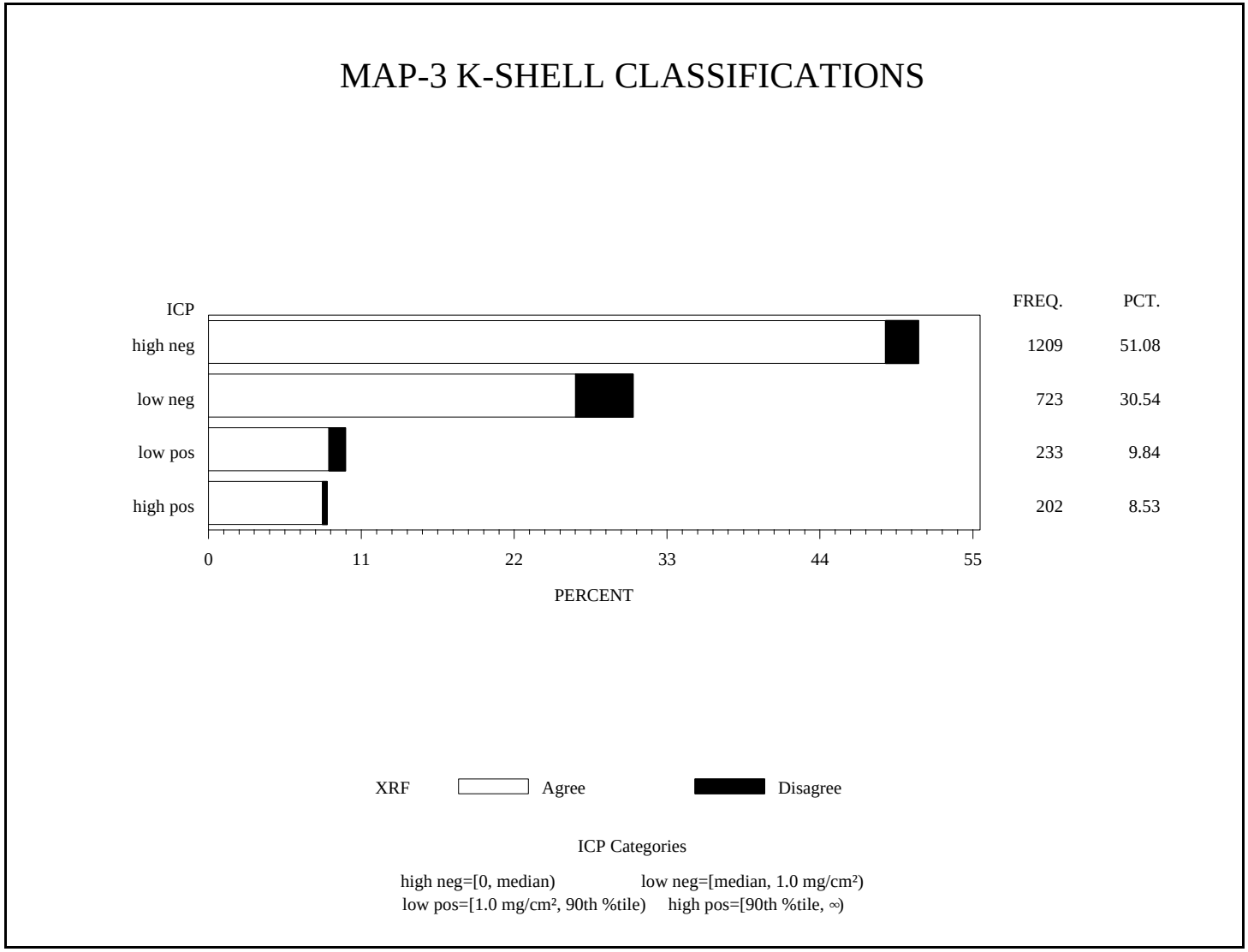

Figure 6-86. Bar chart of classifications by laboratory ICP categories for MAP-3 K-shell, no inconclusive range.

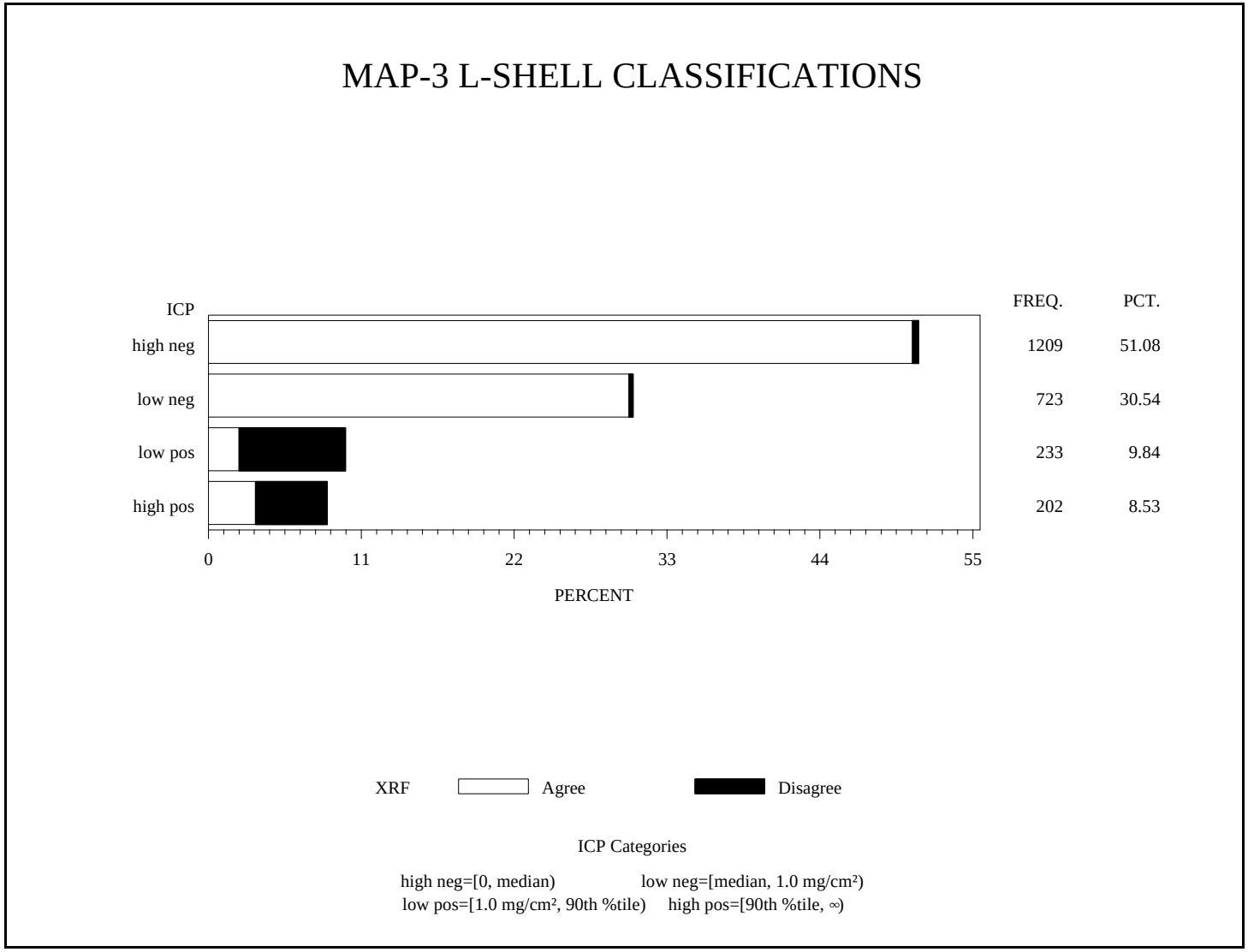

Figure 6-87. Bar chart of classifications by laboratory ICP categories for MAP-3 L-shell, no inconclusive range.

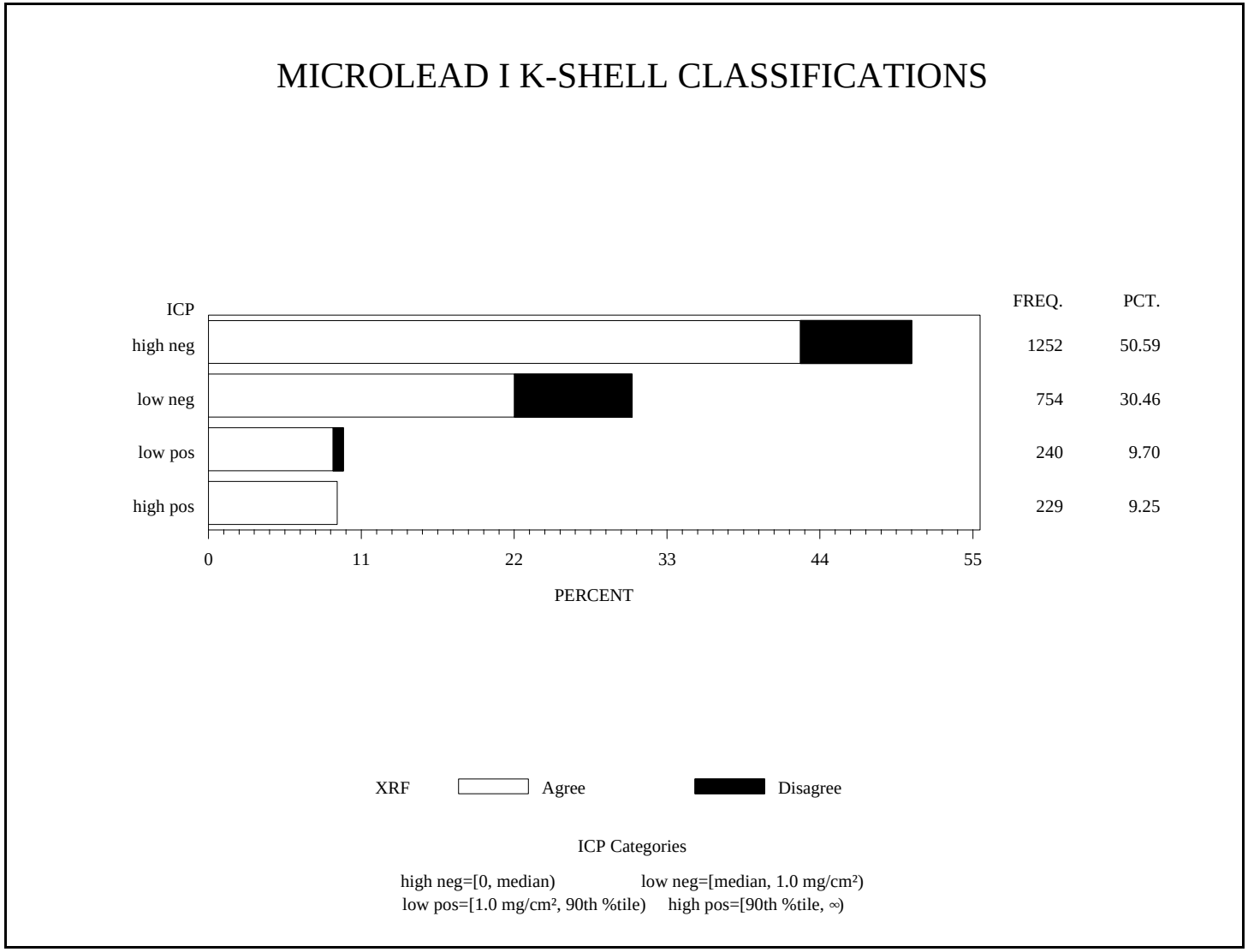

Figure 6-88. Bar chart of classifications by laboratory ICP categories for Microlead I, no inconclusive range.

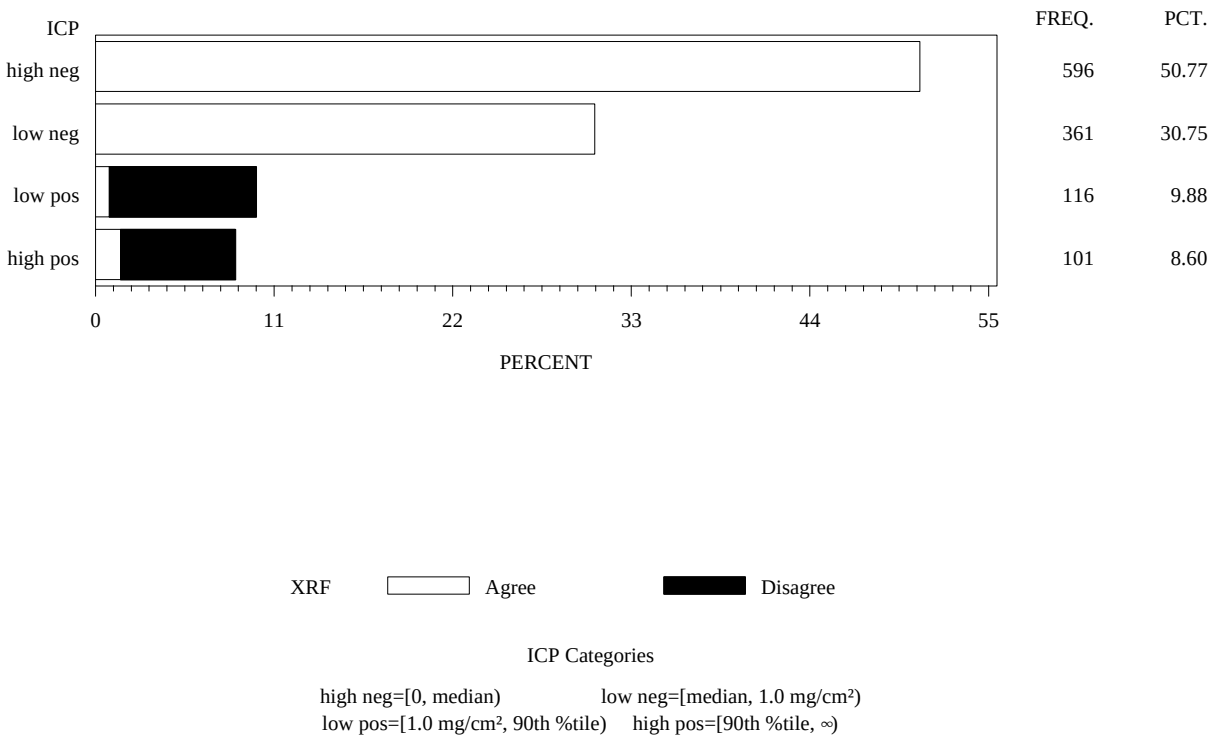

Figure 6-89. Bar chart of classifications by laboratory ICP categories for X-MET 880, no inconclusive range.

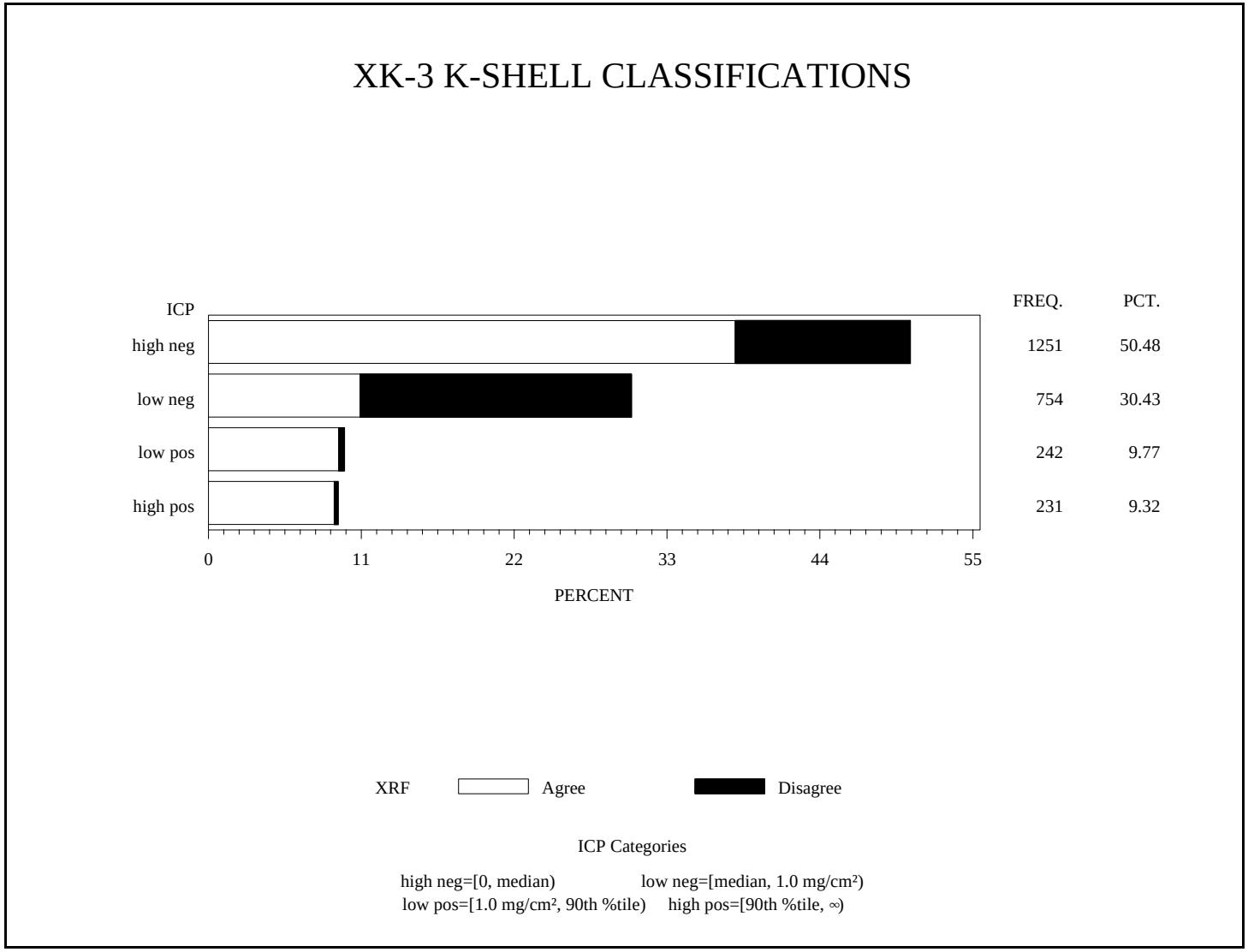

Figure 6-90. Bar chart of classifications by laboratory ICP categories for XK-3, no inconclusive range.

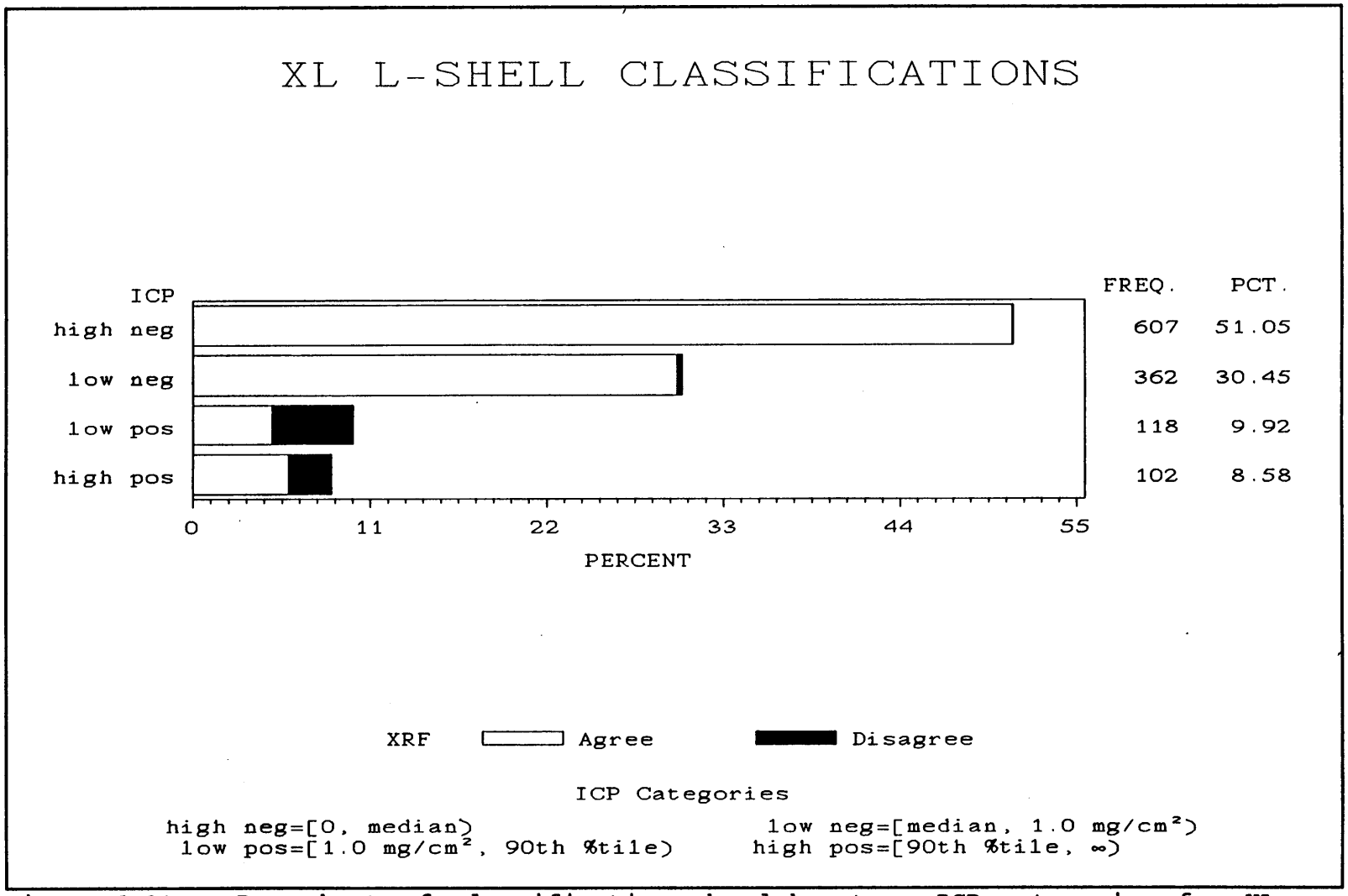

Figure 6-91. Bar chart of classifications by laboratory ICP categories for XL, no inconclusive range.

Table 6-161. Agreement Statistic, K, For All Pairs of XRF Readings Taken At Testing Locations From Which the ICP Measurement in mg/cm² Units Was Greater To or Equal to the 90th percentile of all 1,290 Testing Locations.

	Lead Anal K	Lead Anal L	MAP-3 (I) K	MAP-3 (II) K	MAP-3 (I) L	MAP-3 (II) L	ML I[a] (I)	ML I[a] (II)	X-MET 880	XK-3 (I)	XK-3 (II)	XL
Lead Analyzer K	1.00	0.01	0.66	0.27	0.01	0.01	-	-	0.00	-0.03	-0.02	0.02
Lead Analyzer L		1.00	0.01	0.03	0.53	0.56	-	-	0.94	0.02	-0.06	0.20
MAP-3 (I) K			1.00	0.48	0.03	0.03	-	-	0.01	-0.03	-0.02	0.08
MAP-3 (II) K				1.00	0.05	0.08	-	-	0.03	0.16	-0.04	0.06
MAP-3 (I) L					1.00	0.92	-	-	0.48	-0.01	-0.06	0.48
MAP-3 (II) L						1.00	-	-	0.51	0.05	-0.06	0.45
ML I (I)							-	-	-	-	-	-
ML I (II)								-	-	-	-	-
X-MET 880									1.00	0.02	-0.06	0.18
XK-3 (I)										1.00	-0.03	-0.02
XK-3 (II)											1.00	-0.06
XL												1.00

[a] The Microlead I did not have any negative classifications for the sampling locations used in this analysis.

Table 6-162. Lead Analyzer K-shell by Substrate for the First Standard Paint Reading Without an Inconclusive Range.

Substrate	Sample Size	% False Positive	% False Negative
Brick	93	2.8	0.0
Concrete	218	1.6	11.1
Drywall	113	1.8	na[a]
Metal	189	3.4	6.8
Plaster	222	1.0	3.8
Wood	355	6.3	5.9
Overall	1,190	3.1	5.9

[a] Not available since drywall ICP measurements were all less than 1.0 mg/cm^2 lead.

Table 6-163. Lead Analyzer L-shell by Substrate for the First Standard Paint Reading Without an Inconclusive Range.

Substrate	Sample Size	% False Positive	% False Negative
Brick	93	0.0	90.5
Concrete	218	0.0	96.3
Drywall	113	0.0	na[a]
Metal	189	0.0	81.8
Plaster	222	0.0	100.0
Wood	355	0.0	87.3
Overall	1,190	0.0	89.1

[a] Not available since drywall ICP measurements were all less than 1.0 mg/cm^2 lead.

Table 6-164. MAP-3 K-shell by Substrate for the First Standard Paint Reading Without an Inconclusive Range.

Substrate	Sample Size	% False Positive	% False Negative
Brick	185	4.2	0.0
Concrete	436	5.8	24.1
Drywall	226	3.5	na[a]
Metal	378	19.3	1.1
Plaster	444	2.3	21.2
Wood	698	10.6	5.5
Overall	2,367	8.0	8.3

[a] Not available since drywall ICP measurements were all less than 1.0 mg/cm^2 lead.

Table 6-165. MAP-3 L-shell by Substrate for the First Standard Paint Reading Without an Inconclusive Range.

Substrate	Sample Size	% False Positive	% False Negative
Brick	185	0.0	28.6
Concrete	436	0.0	85.2
Drywall	226	0.4	na[a]
Metal	378	5.2	60.2
Plaster	444	0.0	100.0
Wood	698	0.2	70.4
Overall	2,367	0.9	69.7

[a] Not available since drywall ICP measurements were all less than 1.0 mg/cm^2 lead.

Table 6-166. Microlead I by Substrate for the First Standard Paint Reading Without an Inconclusive Range.

Substrate	Sample Size	% False Positive	% False Negative
Brick	186	22.2	2.4
Concrete	444	25.5	1.8
Drywall	237	17.9	na[a]
Metal	406	18.8	2.2
Plaster	463	9.7	10.2
Wood	739	26.2	3.6
Overall	2,475	20.3	3.8

[a] Not available since drywall ICP measurements were all less than 1.0 mg/cm^2 lead.

Table 6-167. X-MET 880 by Substrate for the First Standard Paint Reading Without an Inconclusive Range.

Substrate	Sample Size	% False Positive	% False Negative
Brick	93	0.0	85.7
Concrete	218	0.0	96.3
Drywall	113	0.0	na[a]
Metal	175	0.0	69.8
Plaster	222	0.0	100.0
Wood	353	0.0	89.0
Overall	1,174	0.0	87.1

[a] Not available since drywall ICP measurements were all less than 1.0 mg/cm^2 lead.

Table 6-168. XK-3 by Substrate for the First Standard Paint Reading Without an Inconclusive Range.

Substrate	Sample Size	% False Positive	% False Negative
Brick	186	52.8	2.4
Concrete	444	66.2	1.8
Drywall	237	5.1	na[a]
Metal	406	57.3	5.4
Plaster	462	45.9	1.7
Wood	743	16.5	4.0
Overall	2,478	39.7	3.6

[a] Not available since drywall ICP measurements were all less than 1.0 mg/cm^2 lead.

Table 6-169. XL by Substrate for the First Standard Paint Reading Without an Inconclusive Range.

Substrate	Sample Size	% False Positive	% False Negative
Brick	93	0.0	23.8
Concrete	217	0.5	40.7
Drywall	113	0.9	na[a]
Metal	189	0.7	29.6
Plaster	222	0.0	57.7
Wood	355	0.8	47.1
Overall	1,189	0.5	41.8

[a] Not available since drywall ICP measurements were all less than 1.0 mg/cm^2 lead.

Table 6-170. Standard Paint Average Without an Inconclusive Range.

XRF	Sample Size	% False Positive	% False Negative
Lead Analyzer K-shell	1,190	2.5	5.9
Lead Analyzer L-shell	1,190	0.0	89.1
MAP-3 K-shell	2,367	6.0	7.4
MAP-3 L-shell	2,367	0.8	68.7
Microlead I K-shell	2,475	18.2	2.3
X-Met 880 L-shell	1,174	0.0	86.6
XK-3 K-shell	2,478	40.1	3.0
XL L-shell	1,189	0.4	43.2

Table 6-171. Lead Analyzer K-shell by Substrate for the Standard Paint Average Without an Inconclusive Range.

Substrate	Sample Size	% False Positive	% False Negative
Brick	93	1.4	0.0
Concrete	218	1.6	7.4
Drywall	113	1.8	na[a]
Metal	189	2.1	6.8
Plaster	222	1.0	7.7
Wood	355	5.1	5.9
Overall	1,190	2.5	5.9

[a] Not available since drywall ICP measurements were all less than 1.0 mg/cm^2 lead.

Table 6-172. Lead Analyzer L-shell by Substrate for the Standard Paint Average Without an Inconclusive Range.

Substrate	Sample Size	% False Positive	% False Negative
Brick	93	0.0	90.5
Concrete	218	0.0	96.3
Drywall	113	0.0	na[a]
Metal	189	0.0	81.8
Plaster	222	0.0	100.0
Wood	355	0.0	87.3
Overall	1,190	0.0	89.1

[a] Not available since drywall ICP measurements were all less than 1.0 mg/cm^2 lead.

Table 6-173. MAP-3 K-shell by Substrate for the Standard Paint Average Without an Inconclusive Range.

Substrate	Sample Size	% False Positive	% False Negative
Brick	185	2.8	0.0
Concrete	436	1.8	22.2
Drywall	226	3.1	na[a]
Metal	378	13.8	1.1
Plaster	444	1.5	26.9
Wood	698	10.2	2.5
Overall	2,367	6.0	7.4

[a] Not available since drywall ICP measurements were all less than 1.0 mg/cm^2 lead.

Table 6-174. MAP-3 L-shell by Substrate for the Standard Paint Average Without an Inconclusive Range.

Substrate	Sample Size	% False Positive	% False Negative
Brick	185	0.0	26.2
Concrete	436	0.0	83.3
Drywall	226	0.0	na[a]
Metal	378	5.2	59.1
Plaster	444	0.0	100.0
Wood	698	0.0	69.8
Overall	2,367	0.8	68.7

[a] Not available since drywall ICP measurements were all less than 1.0 mg/cm^2 lead.

Table 6-175. Microlead I by Substrate for the Standard Paint Average Without an Inconclusive Range.

Substrate	Sample Size	% False Positive	% False Negative
Brick	186	18.1	0.0
Concrete	444	19.6	1.8
Drywall	237	20.3	na[a]
Metal	406	18.2	1.1
Plaster	463	5.7	6.8
Wood	739	26.2	2.3
Overall	2,475	18.2	2.3

[a] Not available since drywall ICP measurements were all less than 1.0 mg/cm^2 lead.

Table 6-176. X-MET 880 by Substrate for the Standard Paint Average Without an Inconclusive Range.

Substrate	Sample Size	% False Positive	% False Negative
Brick	93	0.0	85.7
Concrete	218	0.0	96.3
Drywall	113	0.0	na[a]
Metal	175	0.0	69.8
Plaster	222	0.0	100.0
Wood	353	0.0	88.0
Overall	1,174	0.0	86.6

[a] Not available since drywall ICP measurements were all less than 1.0 mg/cm^2 lead.

Table 6-177. XK-3 by Substrate for the Standard Paint Average Without an Inconclusive Range.

Substrate	Sample Size	% False Positive	% False Negative
Brick	186	53.5	2.4
Concrete	444	69.6	0.0
Drywall	237	4.2	na[a]
Metal	406	57.0	4.3
Plaster	462	48.6	3.4
Wood	743	13.9	3.1
Overall	2,478	40.1	3.0

[a] Not available since drywall ICP measurements were all less than 1.0 mg/cm^2 lead.

Table 6-178. XL by Substrate for the Standard Paint Average Without an Inconclusive Range.

Substrate	Sample Size	% False Positive	% False Negative
Brick	93	0.0	23.8
Concrete	217	0.5	59.3
Drywall	113	0.0	na[a]
Metal	189	0.7	25.0
Plaster	222	0.0	65.4
Wood	355	0.8	45.1
Overall	1,189	0.4	43.2

[a] Not available since drywall ICP measurements were all less than 1.0 mg/cm^2 lead.

Table 6-179. First Standard Paint Reading Control Corrected Without an Inconclusive Range.

XRF	Sample Size	% False Positive	% False Negative
Lead Analyzer K-shell	1,190	2.3	7.3
Lead Analyzer L-shell	1,190	0.0	90.0
MAP-3 K-shell	2,367	10.2	5.7
MAP-3 L-shell	2,367	0.8	73.8
Microlead I K-shell	2,475	12.5	18.3
X-Met 880 L-shell	1,174	0.0	88.5
XK-3 K-shell	2,478	11.3	10.6
XL L-shell	1,189	0.5	45.5

Table 6-180. False Positive Results for First Standard Paint Readings Control Corrected Without an Inconclusive Range, Categorized by Their Corresponding ICP Measurement Above and Below the 0.1964 mg/cm^2 Median of the 1,290 ICP Measurements.

XRF	Sample Size	ICP Measurement Range (mg/cm^2)	% False Positive
Lead Analyzer K-shell	608	[0 - median)	0.5
	362	[median - 1.0)	5.2
Lead Analyzer L-shell	608	[0 - median)	0.0
	362	[median - 1.0)	0.0
MAP-3 K-shell	1,209	[0 - median)	6.2
	723	[median - 1.0)	17.0
MAP-3 L-shell	1,209	[0 - median)	0.7
	723	[median - 1.0)	0.8
Microlead I K-shell	1,252	[0 - median)	9.5
	754	[median - 1.0)	17.4
X-Met 880 L-shell	596	[0 - median)	0.0
	361	[median - 1.0)	0.0
XK-3 K-shell	1,251	[0 - median)	5.1
	754	[median - 1.0)	21.5
XL L-shell	607	[0 - median)	0.2
	362	[median - 1.0)	1.1

Table 6-181. False Negative Results for First Standard Paint Readings Control Corrected Without an Inconclusive Range Categorized by Their Corresponding ICP Measurement Above and Below the 2.4891 mg/cm^2 90th Percentile of the 1,290 ICP Measurements.

XRF	Sample Size	ICP Measurement Range (mg/cm^2)	% False Negative
Lead Analyzer K-shell	118	[1.0 - 90th %tile)	11.9
	102	[90th %tile - ∞)	2.0
Lead Analyzer L-shell	118	[1.0 - 90th %tile)	96.6
	102	[90th %tile - ∞)	82.4
MAP-3 K-shell	233	[1.0 - 90th %tile)	8.2
	202	[90th %tile - ∞)	3.0
MAP-3 L-shell	233	[1.0 - 90th %tile)	81.5
	202	[90th %tile - ∞)	64.9
Microlead I K-shell	240	[1.0 - 90th %tile)	27.1
	229	[90th %tile - ∞)	9.2
X-Met 880 L-shell	116	[1.0 - 90th %tile)	93.1
	101	[90th %tile - ∞)	83.2
XK-3 K-shell	242	[1.0 - 90th %tile)	12.8
	231	[90th %tile - ∞)	8.2
XL L-shell	118	[1.0 - 90th %tile)	56.8
	102	[90th %tile - ∞)	32.4

Table 6-182. Lead Analyzer K-shell by Substrate for the First Standard Paint Reading Control Corrected Without an Inconclusive Range.

Substrate	Sample Size	% False Positive	% False Negative
Brick	93	1.4	0.0
Concrete	218	1.6	14.8
Drywall	113	1.8	na[a]
Metal	189	2.1	6.8
Plaster	222	0.5	7.7
Wood	355	4.7	6.9
Overall	1,190	2.3	7.3

[a] Not available since drywall ICP measurements were all less than 1.0 mg/cm^2 lead.

Table 6-183. Lead Analyzer L-shell by Substrate for the First Standard Paint Reading Control Corrected Without an Inconclusive Range.

Substrate	Sample Size	% False Positive	% False Negative
Brick	93	0.0	90.5
Concrete	218	0.0	96.3
Drywall	113	0.0	na[a]
Metal	189	0.0	84.1
Plaster	222	0.0	100.0
Wood	355	0.0	88.2
Overall	1,190	0.0	90.0

[a] Not available since drywall ICP measurements were all less than 1.0 mg/cm^2 lead.

Table 6-184. MAP-3 K-shell by Substrate for the First Standard Paint Reading Control Corrected Without an Inconclusive Range.

Substrate	Sample Size	% False Positive	% False Negative
Brick	185	7.7	0.0
Concrete	436	9.7	16.7
Drywall	226	3.1	na[a]
Metal	378	12.1	1.1
Plaster	444	12.2	11.5
Wood	698	12.0	4.5
Overall	2,367	10.2	5.7

[a] Not available since drywall ICP measurements were all less than 1.0 mg/cm^2 lead.

Table 6-185. MAP-3 L-shell by Substrate for the First Standard Paint Reading Control Corrected Without an Inconclusive Range.

Substrate	Sample Size	% False Positive	% False Negative
Brick	185	0.0	33.3
Concrete	436	0.0	85.2
Drywall	226	0.0	na[a]
Metal	378	5.2	62.5
Plaster	444	0.0	100.0
Wood	698	0.0	77.4
Overall	2,367	0.8	73.8

[a] Not available since drywall ICP measurements were all less than 1.0 mg/cm^2 lead.

Table 6-186. Microlead I by Substrate for the First Standard Paint Reading Control Corrected Without an Inconclusive Range.

Substrate	Sample Size	% False Positive	% False Negative
Brick	186	16.0	2.4
Concrete	444	6.7	28.6
Drywall	237	16.5	na[a]
Metal	406	13.7	31.5
Plaster	463	6.2	27.1
Wood	739	18.0	10.9
Overall	2,475	12.5	18.3

[a] Not available since drywall ICP measurements were all less than 1.0 mg/cm^2 lead.

Table 6-187. X-MET 880 by Substrate for the First Standard Paint Reading Control Corrected Without an Inconclusive Range.

Substrate	Sample Size	% False Positive	% False Negative
Brick	93	0.0	90.5
Concrete	218	0.0	96.3
Drywall	113	0.0	na[a]
Metal	175	0.0	72.1
Plaster	222	0.0	100.0
Wood	353	0.0	90.0
Overall	1,174	0.0	88.5

[a] Not available since drywall ICP measurements were all less than 1.0 mg/cm^2 lead.

Table 6-188. XK-3 by Substrate for the First Standard Paint Reading Control Corrected Without an Inconclusive Range.

Substrate	Sample Size	% False Positive	% False Negative
Brick	186	9.0	2.4
Concrete	444	24.2	3.6
Drywall	237	0.8	na[a]
Metal	406	9.2	20.7
Plaster	462	13.9	13.6
Wood	743	6.2	8.9
Overall	2,478	11.3	10.6

[a] Not available since drywall ICP measurements were all less than 1.0 mg/cm^2 lead.

Table 6-189. XL by Substrate for the First Standard Paint Reading Control Corrected Without an Inconclusive Range.

Substrate	Sample Size	% False Positive	% False Negative
Brick	93	0.0	23.8
Concrete	217	0.5	48.1
Drywall	113	0.9	na[a]
Metal	189	0.7	31.8
Plaster	222	0.0	69.2
Wood	355	0.8	49.0
Overall	1,189	0.5	45.5

[a] Not available since drywall ICP measurements were all less than 1.0 mg/cm^2 lead.

Table 6-190. First Standard Paint Fully Corrected Reading Without an Inconclusive Range.

XRF	Sample Size	% False Positive	% False Negative
Lead Analyzer K-shell	1,190	1.9	6.8
Lead Analyzer L-shell	1,190	0.0	86.8
MAP-3 K-shell	2,366	4.8	10.8
MAP-3 L-shell	2,366	0.4	78.4
Microlead I K-shell	2,475	9.4	10.0
X-Met 880 L-shell	1,174	0.0	88.9
XK-3 K-shell	2,478	10.1	9.9
XL L-shell	1,187	0.5	43.4

Table 6-191. Lead Analyzer K-shell by Substrate for the First Standard Paint Fully Corrected Reading Without an Inconclusive Range.

Substrate	Sample Size	% False Positive	% False Negative
Brick	93	2.8	0.0
Concrete	218	1.6	7.4
Drywall	113	0.9	na[a]
Metal	189	2.8	6.8
Plaster	222	0.0	11.5
Wood	355	3.2	6.9
Overall	1,190	1.9	6.8

[a] Not available since drywall ICP measurements were all less than 1.0 mg/cm^2 lead.

Table 6-192. Lead Analyzer L-shell by Substrate for the First Standard Paint Fully Corrected Reading Without an Inconclusive Range.

Substrate	Sample Size	% False Positive	% False Negative
Brick	93	0.0	85.7
Concrete	218	0.0	96.3
Drywall	113	0.0	na[a]
Metal	189	0.0	72.7
Plaster	222	0.0	100.0
Wood	355	0.0	87.3
Overall	1,190	0.0	86.8

[a] Not available since drywall ICP measurements were all less than 1.0 mg/cm^2 lead.

Table 6-193. MAP-3 K-shell by Substrate for the First Standard Paint Fully Corrected Reading Without an Inconclusive Range.

Substrate	Sample Size	% False Positive	% False Negative
Brick	185	2.8	2.4
Concrete	435	8.4	24.1
Drywall	226	1.3	na[a]
Metal	378	5.5	2.3
Plaster	444	4.1	23.1
Wood	698	4.2	9.5
Overall	2,366	4.8	10.8

[a] Not available since drywall ICP measurements were all less than 1.0 mg/cm^2 lead.

Table 6-194. MAP-3 L-shell by Substrate for the First Standard Paint Fully Corrected Reading Without an Inconclusive Range.

Substrate	Sample Size	% False Positive	% False Negative
Brick	185	0.0	50.0
Concrete	435	0.5	87.0
Drywall	226	0.0	na[a]
Metal	378	1.0	64.8
Plaster	444	0.5	100.0
Wood	698	0.2	82.4
Overall	2,366	0.4	78.4

[a] Not available since drywall ICP measurements were all less than 1.0 mg/cm^2 lead.

Table 6-195. Microlead I by Substrate for the First Standard Paint Fully Corrected Reading Without an Inconclusive Range.

Substrate	Sample Size	% False Positive	% False Negative
Brick	186	11.8	0.0
Concrete	444	15.2	14.3
Drywall	237	1.7	na[a]
Metal	406	10.5	8.7
Plaster	463	8.9	8.5
Wood	739	7.7	11.8
Overall	2,475	9.4	10.0

[a] Not available since drywall ICP measurements were all less than 1.0 mg/cm^2 lead.

Table 6-196. X-MET 880 by Substrate for the First Standard Paint Fully Corrected Reading Without an Inconclusive Range.

Substrate	Sample Size	% False Positive	% False Negative
Brick	93	0.0	85.7
Concrete	218	0.0	100.0
Drywall	113	0.0	na[a]
Metal	175	0.0	76.7
Plaster	222	0.0	100.0
Wood	353	0.0	89.0
Overall	1,174	0.0	88.9

[a] Not available since drywall ICP measurements were all less than 1.0 mg/cm^2 lead.

Table 6-197. XK-3 by Substrate for the First Standard Paint Fully Corrected Reading Without an Inconclusive Range.

Substrate	Sample Size	% False Positive	% False Negative
Brick	186	10.4	2.4
Concrete	444	16.2	10.7
Drywall	237	2.1	na[a]
Metal	406	11.1	16.3
Plaster	462	12.4	8.5
Wood	743	6.6	8.9
Overall	2,478	10.1	9.9

[a] Not available since drywall ICP measurements were all less than 1.0 mg/cm^2 lead.

Table 6-198. XL by Substrate for the First Standard Paint Fully Corrected Reading Without an Inconclusive Range.

Substrate	Sample Size	% False Positive	% False Negative
Brick	93	0.0	23.8
Concrete	216	0.5	51.9
Drywall	113	0.9	na[a]
Metal	188	0.7	30.2
Plaster	222	0.0	61.5
Wood	355	0.8	46.1
Overall	1,187	0.5	43.4

[a] Not available since drywall ICP measurements were all less than 1.0 mg/cm^2 lead.

Table 6-199. First Standard Paint Reading Red NIST Average Corrected Without an Inconclusive Range.

XRF	Sample Size	% False Positive	% False Negative
Lead Analyzer K-shell	1,190	1.9	7.7
Lead Analyzer L-shell	1,190	0.0	88.2
MAP-3 K-shell	2,367	4.6	9.7
MAP-3 L-shell	2,367	0.3	77.9
Microlead I K-shell	2,475	9.1	9.0
X-Met 880 L-shell	1,174	0.0	89.4
XK-3 K-shell	2,478	10.6	9.9
XL L-shell	1,189	0.5	42.7

Table 6-200. Lead Analyzer K-shell by Substrate for the First Standard Paint Reading Red NIST SRM Average Corrected Without an Inconclusive Range.

Substrate	Sample Size	% False Positive	% False Negative
Brick	93	2.8	0.0
Concrete	218	1.0	11.1
Drywall	113	1.8	na[a]
Metal	189	1.4	9.1
Plaster	222	0.5	3.8
Wood	355	3.6	8.8
Overall	1,190	1.9	7.7

[a] Not available since drywall ICP measurements were all less than 1.0 mg/cm^2 lead.

Table 6-201. Lead Analyzer L-shell by Substrate for the First Standard Paint Reading Red NIST SRM Average Corrected Without an Inconclusive Range.

Substrate	Sample Size	% False Positive	% False Negative
Brick	93	0.0	85.7
Concrete	218	0.0	96.3
Drywall	113	0.0	na[a]
Metal	189	0.0	79.5
Plaster	222	0.0	100.0
Wood	355	0.0	87.3
Overall	1,190	0.0	88.2

[a] Not available since drywall ICP measurements were all less than 1.0 mg/cm^2 lead.

Table 6-202. MAP-3 K-shell by Substrate for the First Standard Paint Reading Red NIST SRM Average Corrected Without an Inconclusive Range.

Substrate	Sample Size	% False Positive	% False Negative
Brick	185	2.1	2.4
Concrete	436	6.5	20.4
Drywall	226	2.2	na[a]
Metal	378	4.8	1.1
Plaster	444	3.1	21.2
Wood	698	6.0	9.0
Overall	2,367	4.6	9.7

[a] Not available since drywall ICP measurements were all less than 1.0 mg/cm^2 lead.

Table 6-203. MAP-3 L-shell by Substrate for the First Standard Paint Reading Red NIST SRM Average Corrected Without an Inconclusive Range.

Substrate	Sample Size	% False Positive	% False Negative
Brick	185	0.0	50.0
Concrete	436	0.0	87.0
Drywall	226	0.0	na[a]
Metal	378	2.1	63.6
Plaster	444	0.0	100.0
Wood	698	0.0	81.9
Overall	2,367	0.3	77.9

[a] Not available since drywall ICP measurements were all less than 1.0 mg/cm^2 lead.

Table 6-204. Microlead I by Substrate for the First Standard Paint Reading Red NIST SRM Average Corrected Without an Inconclusive Range.

Substrate	Sample Size	% False Positive	% False Negative
Brick	186	12.5	0.0
Concrete	444	12.1	16.1
Drywall	237	2.5	na[a]
Metal	406	8.9	5.4
Plaster	463	8.7	8.5
Wood	739	9.2	10.5
Overall	2,475	9.1	9.0

[a] Not available since drywall ICP measurements were all less than 1.0 mg/cm^2 lead.

Table 6-205. X-MET 880 by Substrate for the First Standard Paint Reading Red NIST SRM Average Corrected Without an Inconclusive Range.

Substrate	Sample Size	% False Positive	% False Negative
Brick	93	0.0	85.7
Concrete	218	0.0	96.3
Drywall	113	0.0	na[a]
Metal	175	0.0	79.1
Plaster	222	0.0	100.0
Wood	353	0.0	90.0
Overall	1,174	0.0	89.4

[a] Not available since drywall ICP measurements were all less than 1.0 mg/cm^2 lead.

Table 6-206. XK-3 by Substrate for the First Standard Paint Reading Red NIST SRM Average Corrected Without an Inconclusive Range.

Substrate	Sample Size	% False Positive	% False Negative
Brick	186	11.1	2.4
Concrete	444	18.0	7.1
Drywall	237	2.1	na[a]
Metal	406	11.1	18.5
Plaster	462	11.9	13.6
Wood	743	7.3	7.6
Overall	2,478	10.6	9.9

[a] Not available since drywall ICP measurements were all less than 1.0 mg/cm^2 lead.

Table 6-207. XL by Substrate for the First Standard Paint Reading Red NIST SRM Average Corrected Without an Inconclusive Range.

Substrate	Sample Size	% False Positive	% False Negative
Brick	93	0.0	23.8
Concrete	217	0.5	55.6
Drywall	113	0.9	na[a]
Metal	189	0.7	27.3
Plaster	222	0.0	69.2
Wood	355	0.8	43.1
Overall	1,189	0.5	42.7

[a] Not available since drywall ICP measurements were all less than 1.0 mg/cm^2 lead.

6.5.3 Impact of An Inconclusive Range With a 1.6 mg/cm^2 Upper Bound and a 0.4 mg/cm^2 Lower Bound

All of the error percentage tables given above were results from classifying XRF measurements as negative or positive. This section and the next section present results from classifying XRF measurements as negative, positive, or inconclusive. In this section, an XRF measurement was classified positive if the XRF measurement was 1.6 mg/cm^2 or greater, negative if the XRF measurement was 0.4 mg/cm^2 or less, and in the inconclusive range if the XRF measurement was between 0.4 and 1.6 mg/cm^2. As noted above, an "XRF measurement" is a term used for general discussion purposes. In each subsection below where a specific classification analysis is discussed, the XRF measurement will be defined as either a single reading, a single reading corrected for substrate bias, an average of three readings, or an average of three readings corrected for substrate bias.

6.5.3.1 First Standard Paint Readings With an (0.4 - 1.6 mg/cm^2) Inconclusive Range

Table 6-208 shows overall error rates by instrument for the first standard paint reading using the (0.4 - 1.6 mg/cm^2) inconclusive range. Tables 6-209 and 6-210 provide the same information for four ICP measurement categories and Tables 6-211 through 6-218 provide the same information by substrate.

In Table 6-208, the Lead Analyzer K-shell had the lowest false positive and false negative rates (0.5% and 1.4%, respectively) for those instruments with both misclassification rates (the false positive and false negative percentages) less than 10%. The MAP-3 K-shell had both misclassification rates under 4%. The Microlead I also had low misclassification rates with a 7.5% false positive rate and a 1.1% false negative rate. The XL had a somewhat higher false negative rate (11.4%) and a very low false positive rate (0.1%). All other instruments had at least one misclassification rate greater than 20% ranging from the 21.8% false positive rate for the XK-3 to the 66.4% false negative rate from the X-MET 880.

Comparisons of Table 6-208 to Table 6-158 shows that the addition of the inconclusive range substantially reduces the error percentages in all cases. However, the Lead Analyzer L-shell, the MAP-3 L-shell, the X-MET 880, and the XK-3

instruments still had at least one error rate greater than 20%. Two XRF instruments had inconclusive percentages less than 10%: the Lead Analyzer L-shell and the X-MET 880, but, both of these instruments had false negative rates greater than 65%. Furthermore, Table 6-217 shows that XK-3 instrument's results on concrete, metal, and plaster substrates had false positive rates greater than 26%.

Tables 6-209 and 6-210 show the same information as Table 6-208 but for four categories of ICP measurements and Figures 6-92 through 6-99 graphically illustrates the same information. These two tables indicate where the misclassification errors are occurring relative to the ICP measurement. Tables 6-209 and 6-210 are analogous to Tables 6-159 and 6-160 with the addition of the inconclusive range. Tables 6-209 and 6-210 show differences between K-shell instruments and L-shell instruments by comparing the percent in the inconclusive range. In Table 6-209, the inconclusive percentages for the L-shell instruments were all five percent or less except for the XL which had 10.5% of its first standard paint readings in the higher ICP range classified as inconclusive. This is in contrast to the K-shell instruments that had inconclusive rates greater than 35% in the ICP measurement category bounded by the ICP measurement median and 1.0 mg/cm^2 lead. Figures 6-92 through 6-99 clearly illustrate this difference. For example, in the bottom two bars, the L-shell instruments a greater percentage of inconclusive and *disagree* results than did the K-shell instruments.

6.5.3.2 Average of Three Standard XRF Readings With an (0.4 - 1.6 mg/cm^2) Inconclusive Range

Table 6-219 shows overall error rates by instrument for the average of three first standard paint readings using the (0.4 - 1.6 mg/cm^2) inconclusive range. Comparisons of Table 6-219 and Table 6-208 show that for the K-shell instruments the error percentages (false positive and false negative) are the same in two cases and slightly smaller for the average in six cases. The error percentages for the L-shell instruments are the same in six cases, while the average is slightly smaller the remaining two cases. In no case, either for K- or L-shell instruments, does the average represent a significant improvement over the first standard paint reading. In particular, in cases where the error rate was high, the false negative for the L-shell instruments and false positive for the XK-3, only very minimal improvement

occurred by use of the average of three readings.

Tables 6-211 through 6-218 break down the results for the first standard paint reading by substrate. Tables 6-220 through 6-227 are the companion tables for the average of three readings. For the four K-shell instruments, there are a total of 44 error percentages when broken down by substrate. In 22 cases, the error percentages for the average are smaller, in 20 cases there is no difference between the average and the first standard paint reading error percentages, and in two cases the error percentages are higher for the average. For the L-shell instruments, 35 cases are the same, four cases show the average as lower, and five cases show the average as higher. In no case was any improvement of the average over the first standard paint reading very great. For example, there are 24 error rates exceeding 10%; of these, only four were reduced below 10% by use of the average, and all of the improvements were small. For the MAP-3, 11.1 % and 11.5% false negative rates on concrete and plaster for the first standard paint reading were reduced to 9.3% and 9.6%. On wood, an XL false negative rate of 13.7% was reduced to 7.8% and a Microlead I false positive rate of 12.3 was reduced to 10.4%.

The conclusion from examining these classification results is the same as given above. That is, although use of the average of three 15-second readings may result in a more accurate classification of paint than use of only a single reading, the improvement is usually minimal and not of practical significance. Thus, it appears that the additional effort involved in taking three 15-second readings at a sampling location versus only one is not justified by an increase the accuracy of classification of paint.

6.5.3.3 Standard XRF Readings Control Corrected With an (0.4 - 1.6 mg/cm^2) Inconclusive Range

The first standard paint reading was "control corrected" by subtracting the average of the initial and ending red NIST SRM control block readings in the dwelling, minus 1.02 mg/cm^2. Table 6-228 shows overall error rates by instrument for the first standard paint control corrected readings using the (0.4 - 1.6 mg/cm^2) inconclusive range. This table should be compared to Table 6-208, which shows the same information for the first standard paint [uncorrected] reading. For the MAP-3 K-shell and the Lead Analyzer K-shell, both error rates were low before and

after correction, so the procedure again had little impact. For the XK-3, control correction reduces the false positive rate from 21.8% to 3.5% with only a small increase in the false negative rate, from 1.1% to 4.0%. For the Microlead I, the false positive rate was decreased from 7.5% to 4.9%, but at the expense of an increase in the false negative rate from 1.1% to 12.4%. The performance of the XK-3 was improved by control correction, while that of the Microlead I was worsened by control correction. The L-shell instruments showed no improvement. Overall, then, control correction did not improve the performance of L-shell instruments. Thus, the impact of control correction appears to be instrument-specific, so that no general recommendation on its use can be made.

Tables 6-229 and 6-230 provide the same information by ICP measurement category. Tables 6-209 and 6-210 are the companion tables for the first standard [uncorrected] reading with a (0.4 - 1.6 mg/cm^2) inconclusive range.

Tables 6-231 through 6-238 break down the control corrected error rates by substrate for the eight instruments, and are to be compared to Tables 6-211 through 6-218 for the first standard paint [uncorrected] reading. For the L-shell instruments, the same picture emerges as from the overall data. False negative rates by substrate remain high after correction. In the case of the XL, correction has a substantial negative impact on concrete. For the Lead Analyzer K-shell, error rates by substrate were low without correction and remain so after correction, confirming that the procedure has little impact. For the MAP-3 K-shell, false positive rates by substrate were generally increased slightly with a corresponding decrease in false negative rate. However, the false negative rates for concrete and plaster shown in Table 6-213 were above 10% before correction (11.1% and 11.5%), and were reduced by correction (to 7.4% and 5.8%) as shown in Table 6-233. Thus, on an individual substrate basis, control correction has merit for the MAP-3 K-shell. For the XK-3, four of the substrates have high false positive rates which were dramatically reduced by control correction. However, the false negative rate on metal was increased sharply, from 0 to 15.2%. On a substrate-specific basis, control correction usually improves accuracy for the XK-3, but not always. For the Microlead I, metal and plaster have very high false negative rates after control correction, which outweighs the modest reductions in false positive rates. Thus, substrate-specific

analyses generally confirm the overall results, except that some positive impact of control correction for the MAP-3 K-shell was indicated on concrete, metal, and plaster, while the approach appears somewhat less effective for the XK-3 than indicated by the overall data.

6.5.3.4 Standard XRF Readings Fully Corrected With an (0.4 - 1.6 mg/cm^2) Inconclusive Range

For this analysis, the first standard paint reading was "fully corrected" by subtracting the average of the three standard red NIST SRM readings taken from the same sampling location, minus 1.02 mg/cm^2. Table 6-239 shows overall error rates by instrument for the first standard paint fully corrected readings using the (0.4 - 1.6 mg/cm^2) inconclusive range. This table should be compared to Table 6-208, which shows the same information for the first standard paint [uncorrected] reading. Tables 6-240 through 6-247 provide the same information by substrate category. Tables 6-211 through 6-218 are the companion tables for the first standard [uncorrected] reading with a (0.4 - 1.6 mg/cm^2) inconclusive range. Full correction was effective in reducing error rates the Microlead I and the XK-3 and on wood and metal substrates for the MAP-3.

6.5.3.5 Standard XRF Readings Red NIST SRM Average Corrected With an (0.4 - 1.6 mg/cm^2) Inconclusive Range

The first standard paint reading was "red NIST SRM average corrected" by subtracting the corresponding substrate average of all red NIST SRM reading taken at each sampling location in the dwelling, minus 1.02 mg/cm^2. Table 6-248 shows overall error rates by instrument. Tables 6-249 through 6-256 break down the information by substrate. This method of correction provided results similar to full correction.

6.5.4 Impact of An Inconclusive Range With a 1.3 mg/cm^2 Upper Bound and a 0.7 mg/cm^2 Lower Bound

In this section, an alternative inconclusive range was used to classify the XRF readings as negative, positive, or inconclusive. The previous section defined the inconclusive range as having a 1.6 mg/cm^2 upper bound and a 0.4 mg/cm^2 lower

bound. This section defines the inconclusive range as having a 1.3 mg/cm^2 upper bound and a 0.7 mg/cm^2 lower bound, and will be referred to as the alternate inconclusive range. The impact of the alternate inconclusive range can be assessed by comparing the results in this section to the results presented in the previous section.

Table 6-208. First Standard Paint Reading With an Inconclusive Range Bounded by 0.4 mg/cm^2 and 1.6 mg/cm^2.

XRF	Sample Size	% False Positive	% False Negative	% Inconclusive
Lead Analyzer K-shell	1,190	0.5	1.4	18.1
Lead Analyzer L-shell	1,190	0.0	65.5	6.1
MAP-3 K-shell	2,367	2.3	3.7	23.4
MAP-3 L-shell	2,367	0.0	36.8	12.2
Microlead I K-shell	2,475	7.5	1.1	30.3
X-Met 880 L-shell	1,174	0.0	66.4	6.8
XK-3 K-shell	2,478	21.8	1.1	35.1
XL L-shell	1,189	0.1	11.4	15.3

Table 6-209. False Positive Results for First Standard Paint Readings With an Inconclusive Range Bounded by 0.4 mg/cm² and 1.6 mg/cm², Categorized by Their Corresponding ICP Measurement Above and Below the 0.1964 mg/cm² Median of the 1,290 ICP Measurements.

XRF	Sample Size	ICP Measurement Range (mg/cm²)	% False Positive	% Inconclusive
Lead Analyzer K-shell	608	[0 - median)	0.2	4.6
	362	[median - 1.0)	1.1	40.3
Lead Analyzer L-shell	608	[0 - median)	0.0	0.3
	362	[median - 1.0)	0.0	1.4
MAP-3 K-shell	1,209	[0 - median)	1.6	19.3
	783	[median - 1.0)	3.5	37.1
MAP-3 L-shell	1,209	[0 - median)	0.0	4.1
	723	[median - 1.0)	0.0	4.4
Microlead I K-shell	1,252	[0 - median)	5.6	28.4
	754	[median - 1.0)	10.7	45.0
X-Met 880 L-shell	596	[0 - median)	0.0	1.5
	361	[median - 1.0)	0.0	3.0
XK-3 K-shell	1,251	[0 - median)	9.9	39.9
	754	[median - 1.0)	41.5	42.7
XL L-shell	607	[0 - median)	0.0	3.0
	362	[median - 1.0)	0.3	10.5

Table 6-210. False Negative Results for First Standard Paint Readings With an Inconclusive Range Bounded by 0.4 mg/cm^2 and 1.6 mg/cm^2 Categorized by Their Corresponding ICP Measurement Above and Below the 2.4891 mg/cm^2 90th Percentile of the 1,290 ICP Measurements.

XRF	Sample Size	ICP Measurement Range (mg/cm^2)	% False Negative	% Inconclusive
Lead Analyzer K-shell	118	[1.0 - 90th %tile)	1.7	33.9
	102	[90th %tile - ∞)	1.0	1.0
Lead Analyzer L-shell	118	[1.0 - 90th %tile)	69.5	30.5
	102	[90th %tile - ∞)	60.8	28.4
MAP-3 K-shell	233	[1.0 - 90th %tile)	4.7	21.5
	202	[90th %tile - ∞)	2.5	2.0
MAP-3 L-shell	233	[1.0 - 90th %tile)	44.6	47.2
	202	[90th %tile - ∞)	27.7	48.5
Microlead I K-shell	240	[1.0 - 90th %tile)	2.1	22.1
	229	[90th %tile - ∞)	0.0	1.3
X-Met 880 L-shell	116	[1.0 - 90th %tile)	68.1	31.0
	101	[90th %tile - ∞)	64.4	23.8
XK-3 K-shell	242	[1.0 - 90th %tile)	2.1	13.6
	231	[90th %tile - ∞)	0.0	6.9
XL L-shell	118	[1.0 - 90th %tile)	13.6	69.5
	102	[90th %tile - ∞)	8.8	43.1

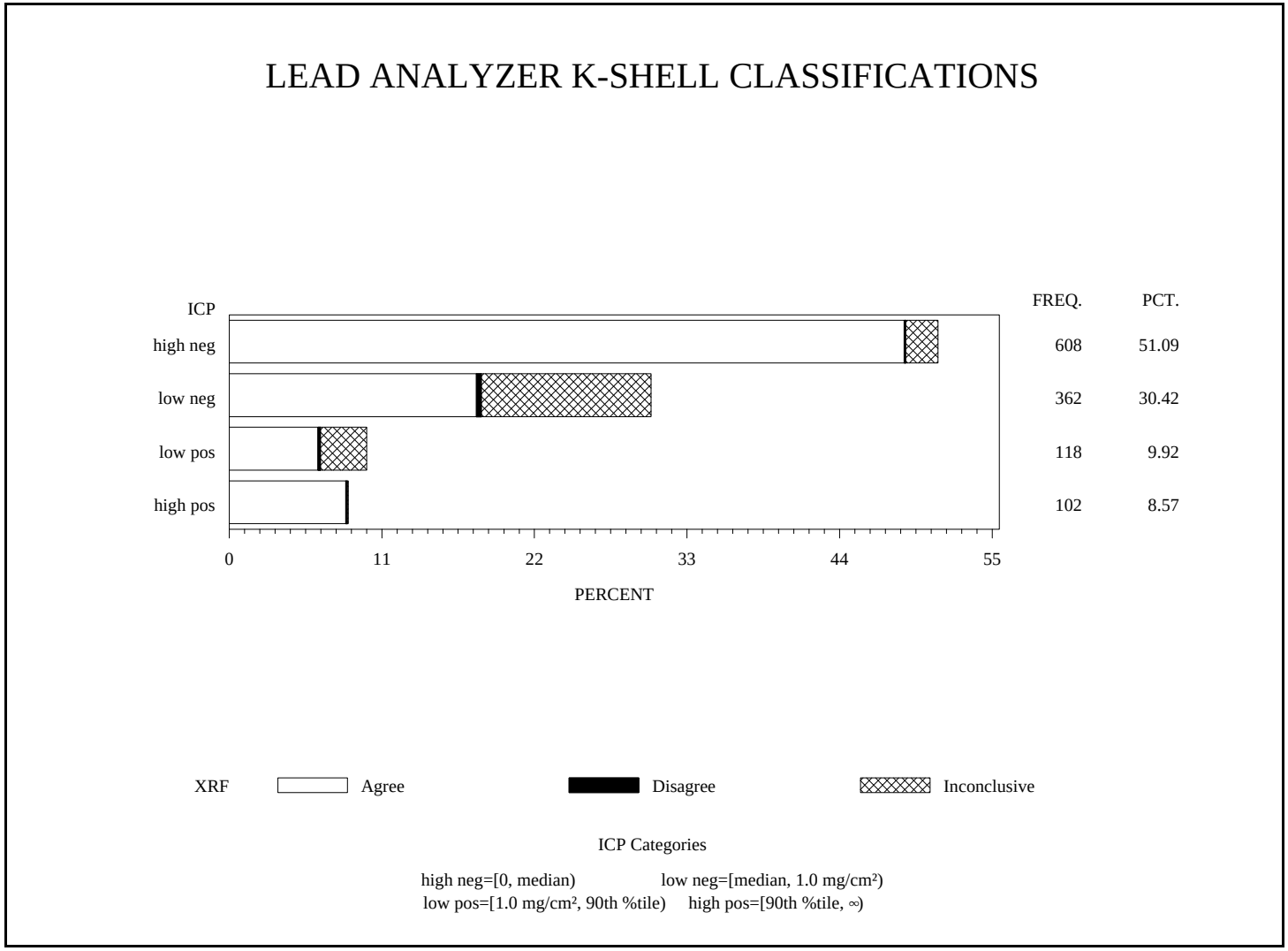

Figure 6-92. Bar chart of classifications by laboratory ICP categories for Lead Analyzer K-shell, with an inconclusive range bounded by 0.4 mg/cm^2 and 1.6 mg/cm^2.

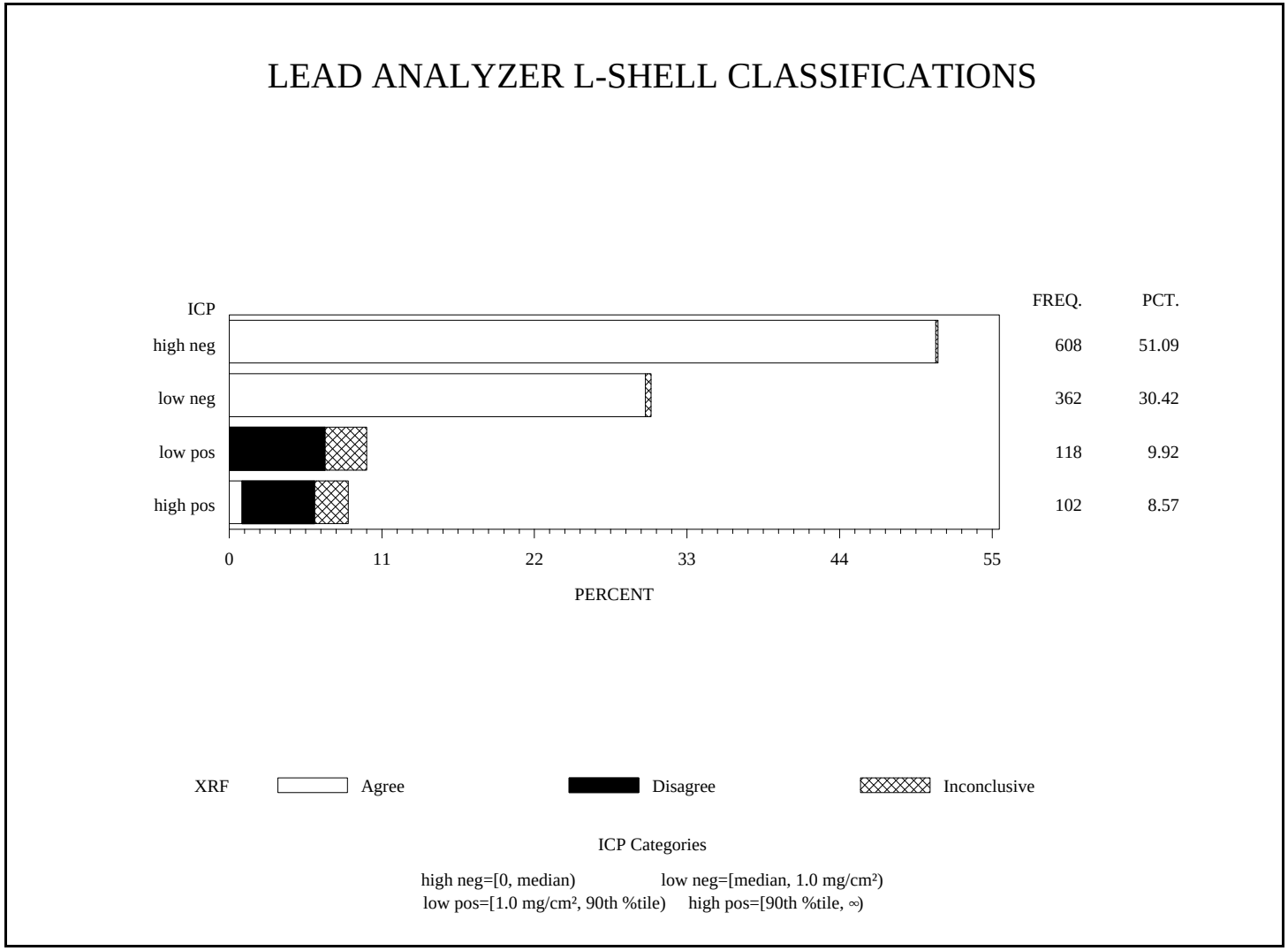

Figure 6-93. Bar chart of classifications by laboratory ICP categories for Lead Analyzer L-shell, with an inconclusive range bounded by 0.4 mg/cm^2 and 1.6 mg/cm^2.

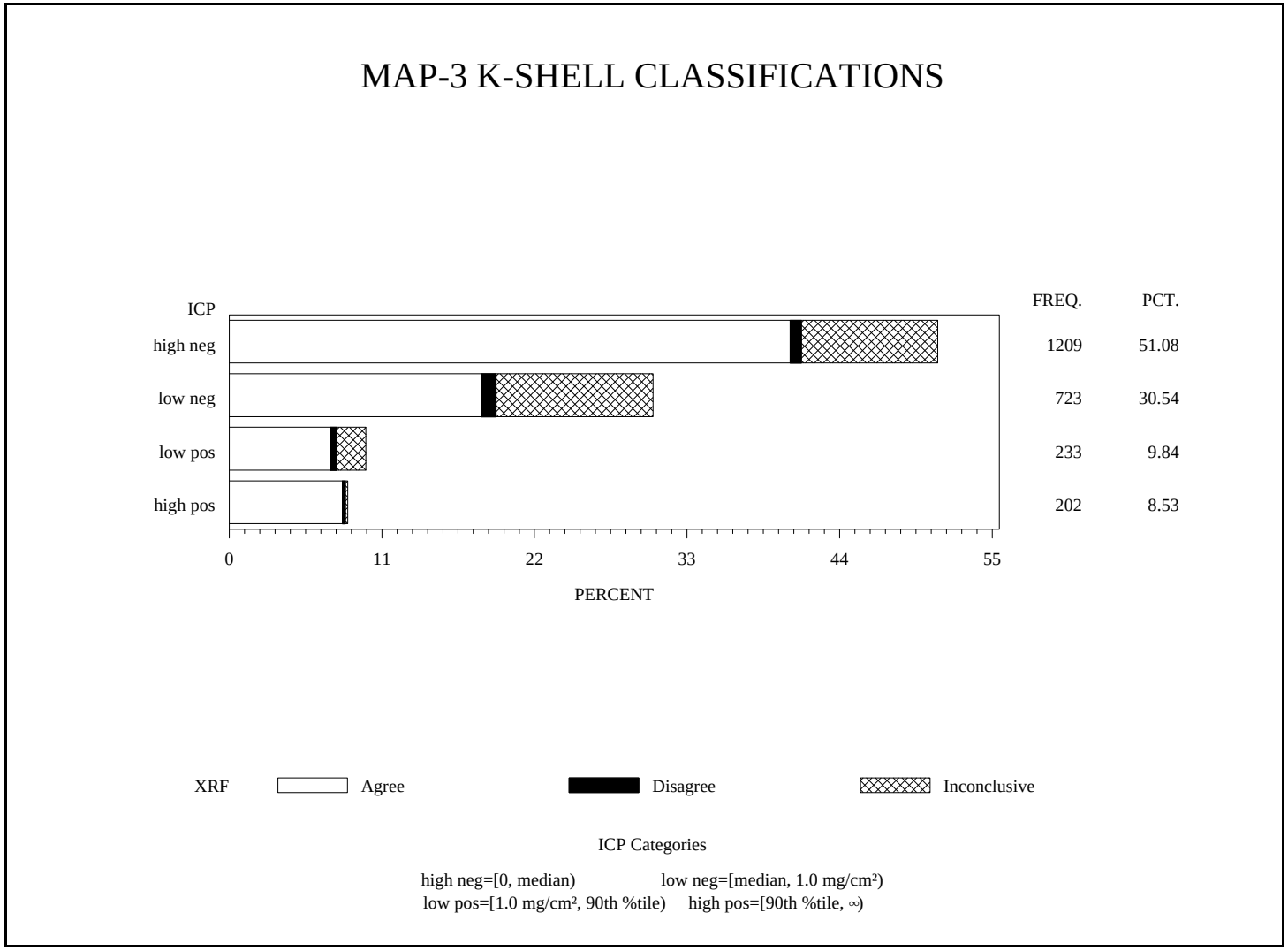

Figure 6-94. Bar chart of classifications by laboratory ICP categories for MAP-3 K-shell, with an inconclusive range bounded by 0.4 mg/cm^2 and 1.6 mg/cm^2.

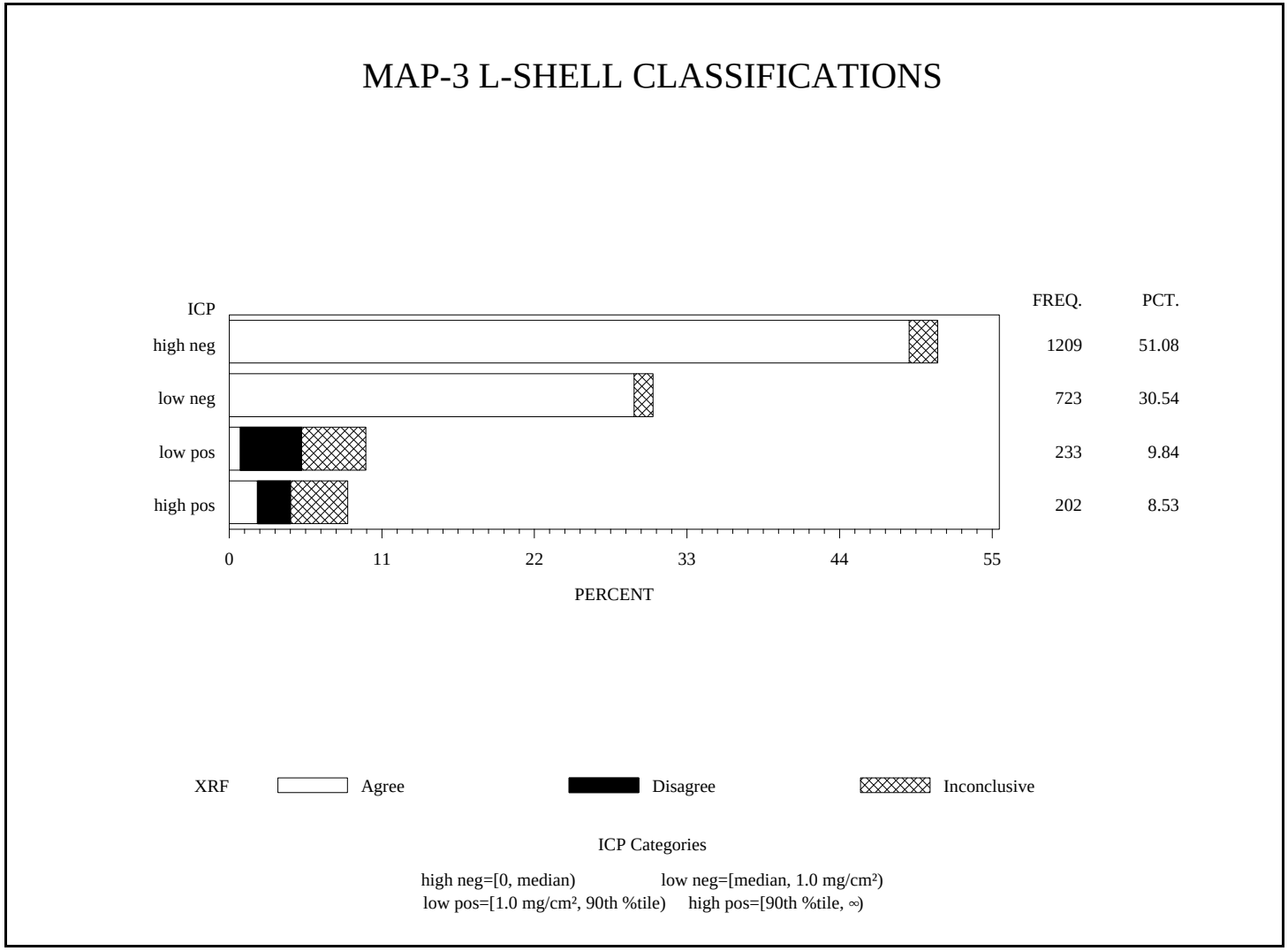

Figure 6-95. Bar chart of classifications by laboratory ICP categories for MAP-3 L-shell, with an inconclusive range bounded by 0.4 mg/cm^2 and 1.6 mg/cm^2.

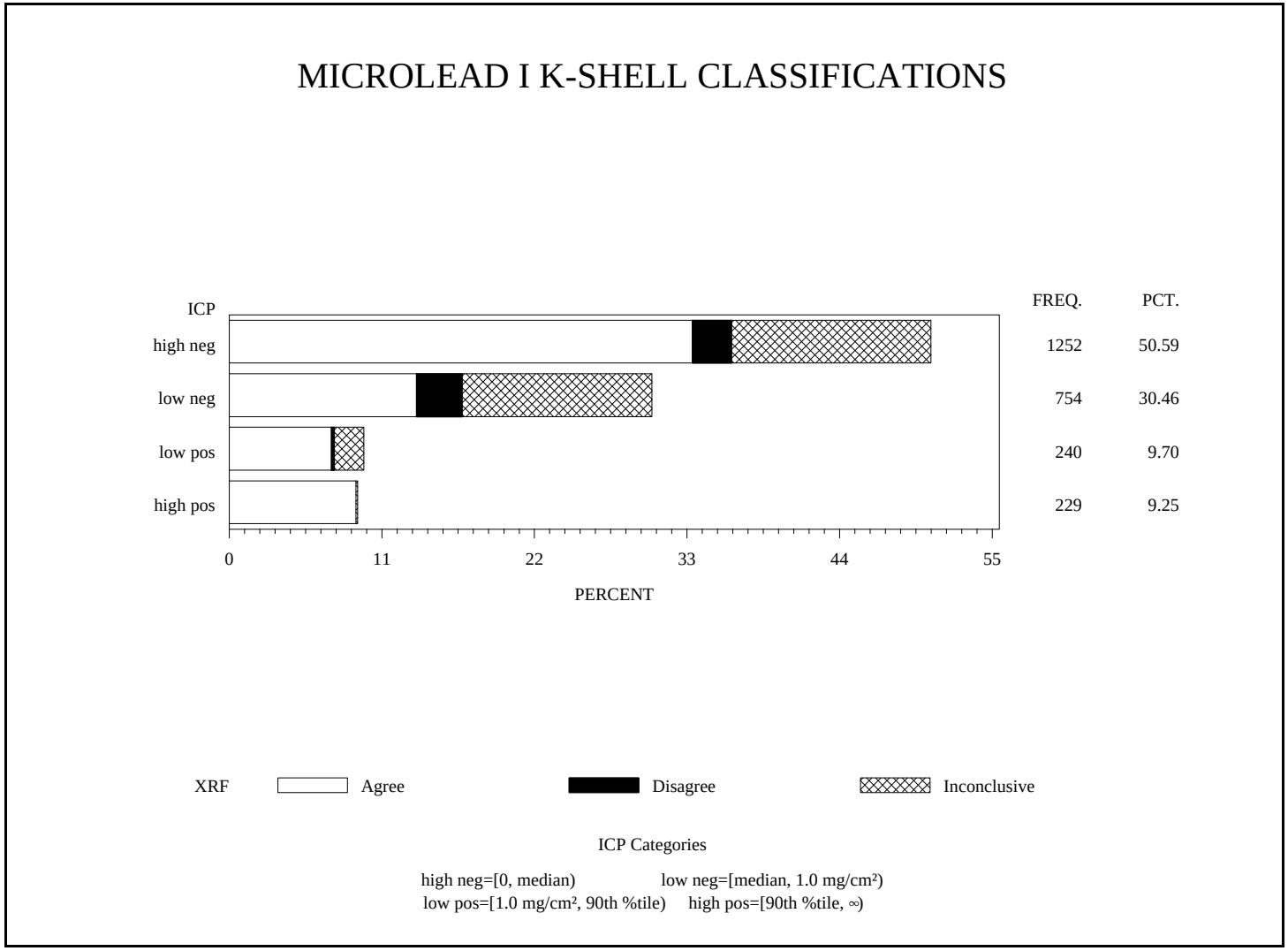

Figure 6-96. Bar chart of classifications by laboratory ICP categories for Microlead I, with an inconclusive range bounded by 0.4 mg/cm^2 and 1.6 mg/cm^2.

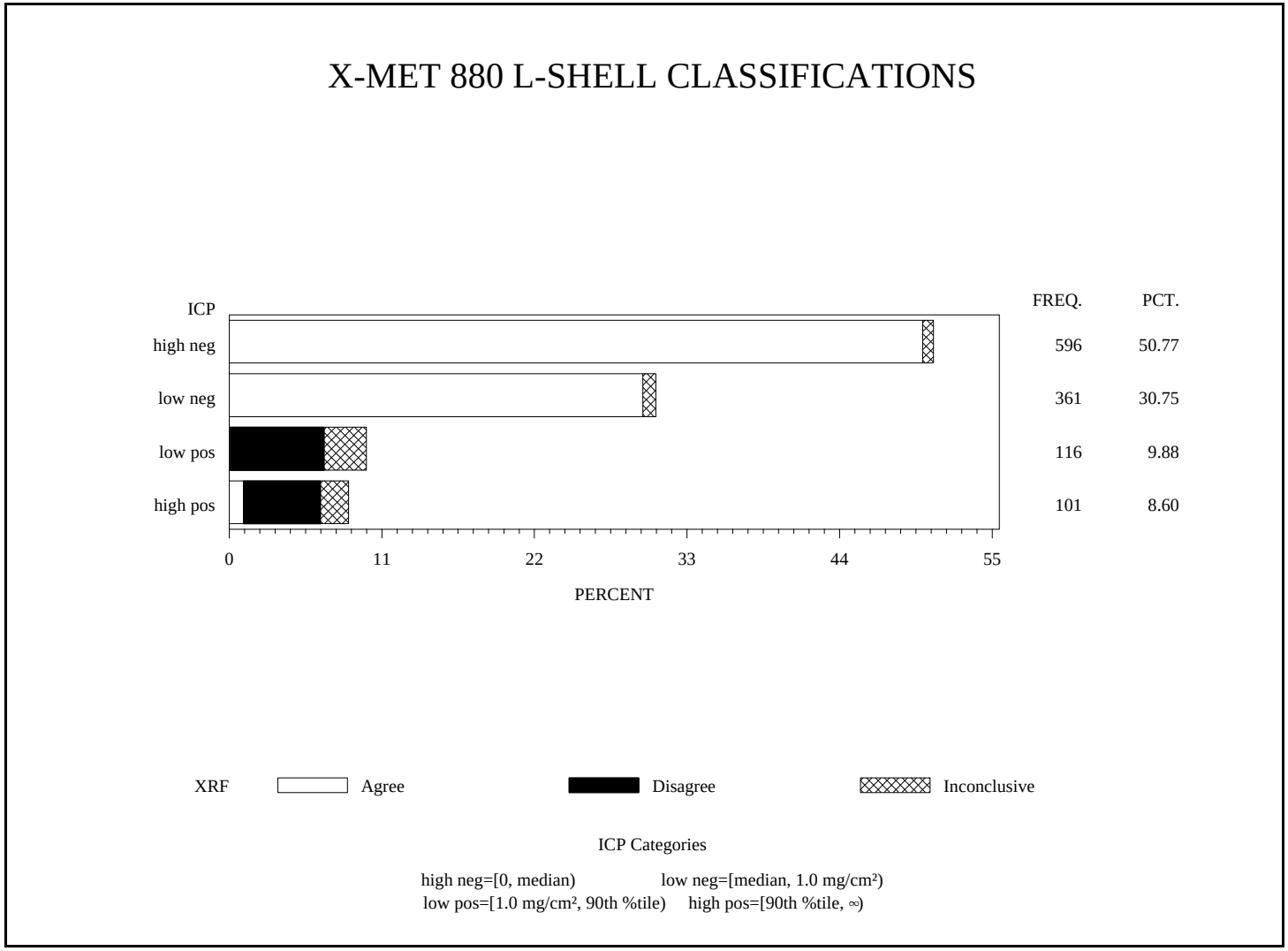

Figure 6-97. Bar chart of classifications by laboratory ICP categories for X-MET 880, with an inconclusive range bounded by 0.4 mg/cm^2 and 1.6 mg/cm^2.

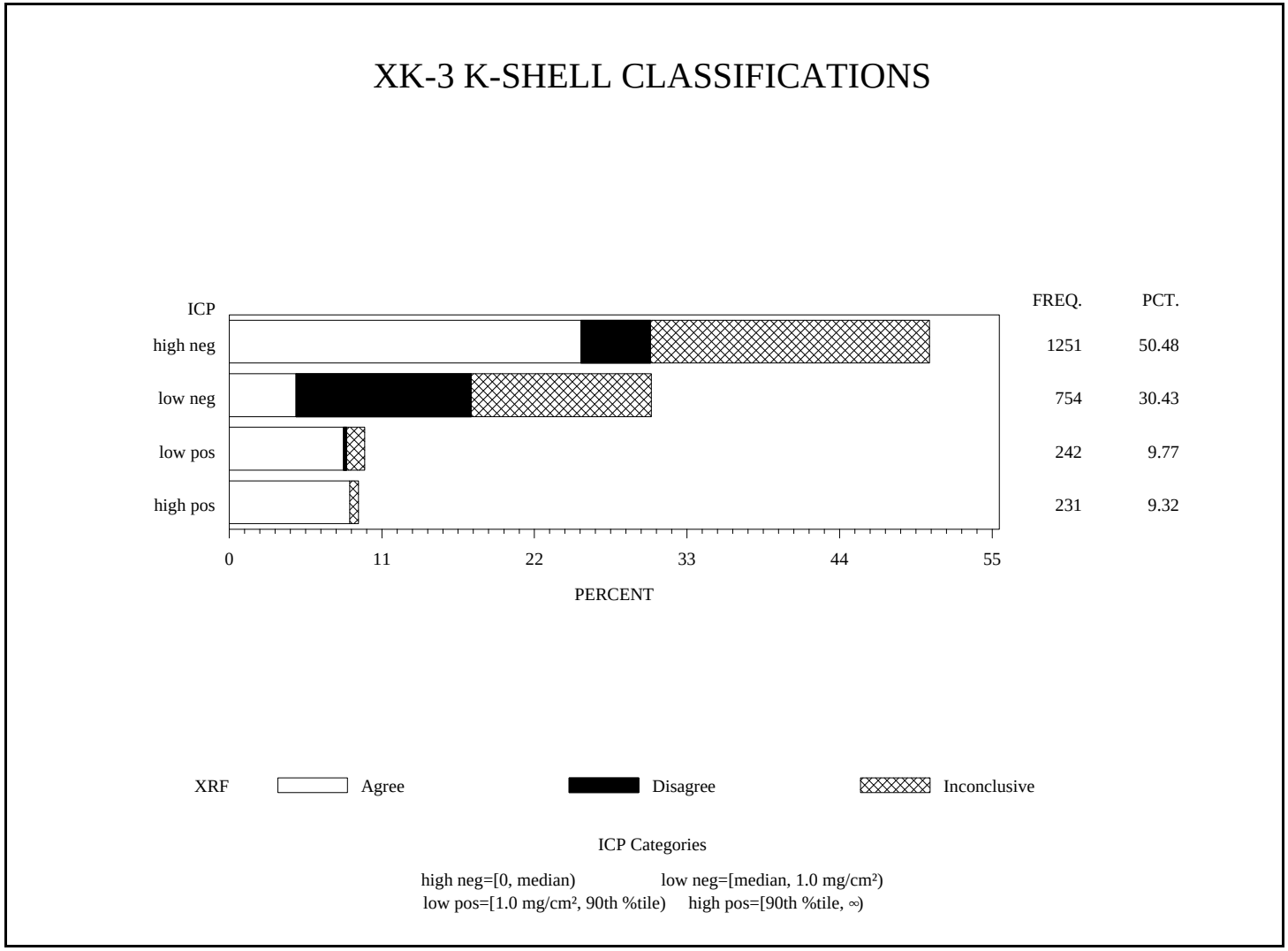

Figure 6-98. Bar chart of classifications by laboratory ICP categories for XK-3, with an inconclusive range bounded by 0.4 mg/cm^2 and 1.6 mg/cm^2.

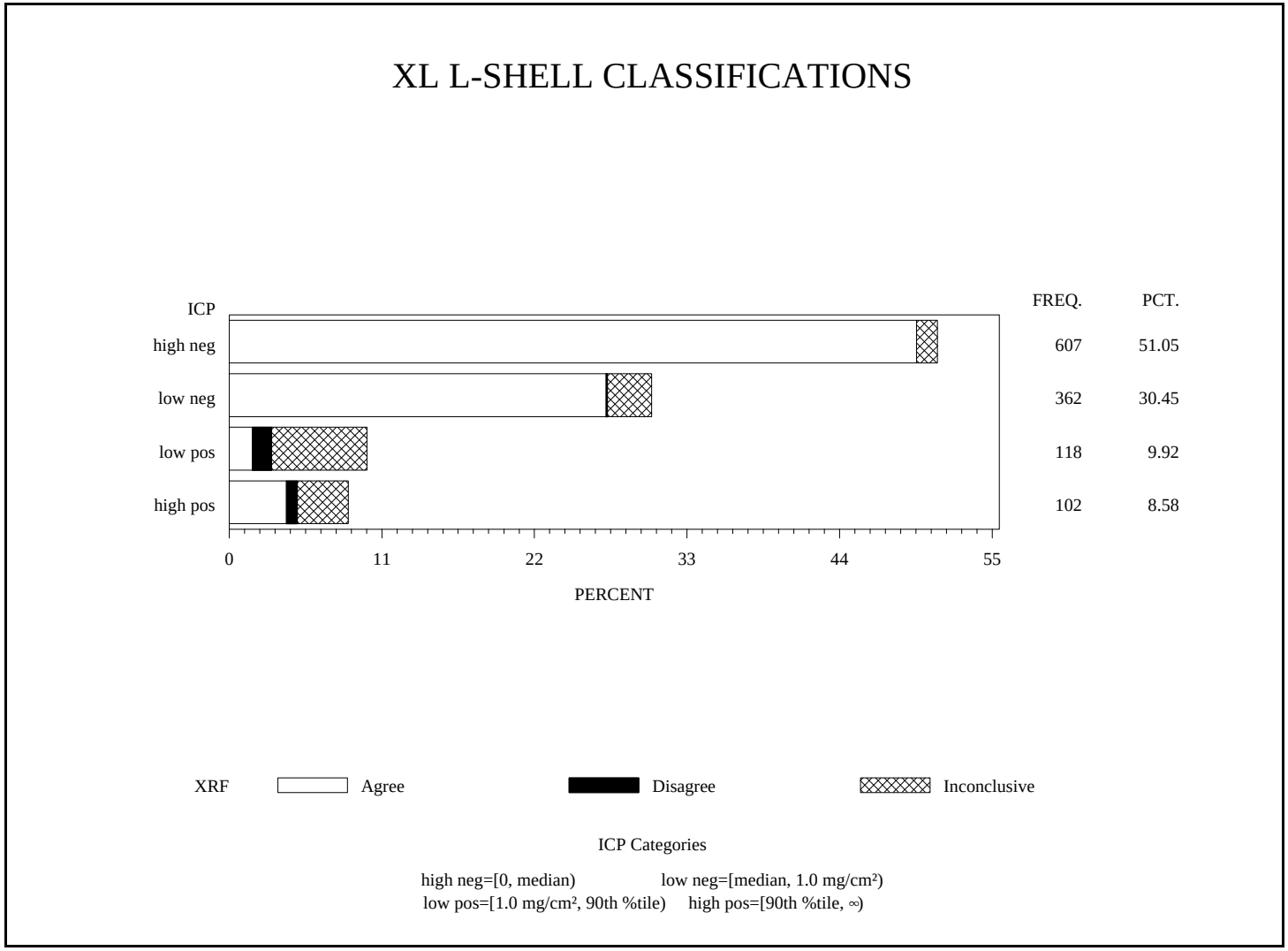

Figure 6-99. Bar chart of classifications by laboratory ICP categories for XL, with an inconclusive range bounded by 0.4 mg/cm^2 and 1.6 mg/cm^2.

Table 6-211. Lead Analyzer K-shell by Substrate for the First Standard Paint Reading With an Inconclusive Range Bounded by 0.4 mg/cm^2 and 1.6 mg/cm^2.

Substrate	Sample Size	% False Positive	% False Negative	% Inconclusive
Brick	93	0.0	0.0	16.1
Concrete	218	1.1	0.0	17.4
Drywall	113	0.0	na[a]	9.8
Metal	189	0.0	0.0	25.4
Plaster	222	0.5	0.0	14.4
Wood	355	0.8	2.9	20.0
Overall	1,190	0.5	1.4	18.1

[a] Not available since drywall ICP measurements were all less than 1.0 mg/cm^2 lead.

Table 6-212. Lead Analyzer L-shell by Substrate for the First Standard Paint Reading With an Inconclusive Range Bounded by 0.4 mg/cm^2 and 1.6 mg/cm^2.

Substrate	Sample Size	% False Positive	% False Negative	% Inconclusive
Brick	93	0.0	38.1	11.8
Concrete	218	0.0	88.9	1.4
Drywall	113	0.0	na[a]	0.0
Metal	189	0.0	54.5	10.1
Plaster	222	0.0	88.5	1.4
Wood	355	0.0	63.7	10.1
Overall	1,190	0.0	65.5	6.1

[a] Not available since drywall ICP measurements were all less than 1.0 mg/cm^2 lead.

Table 6-213. MAP-3 K-shell by Substrate for the First Standard Paint Reading With an Inconclusive Range Bounded by 0.4 mg/cm^2 and 1.6 mg/cm^2.

Substrate	Sample Size	% False Positive	% False Negative	% Inconclusive
Brick	185	0.7	0.0	14.6
Concrete	436	2.6	11.1	15.1
Drywall	226	1.3	na[a]	21.7
Metal	378	1.7	1.1	48.4
Plaster	444	1.3	11.5	14.2
Wood	698	4.0	1.5	23.9
Overall	2,367	2.3	3.7	23.4

[a] Not available since drywall ICP measurements were all less than 1.0 mg/cm^2 lead.

Table 6-214. MAP-3 L-shell by Substrate for the First Standard Paint Reading With an Inconclusive Range Bounded by 0.4 mg/cm^2 and 1.6 mg/cm^2.

Substrate	Sample Size	% False Positive	% False Negative	% Inconclusive
Brick	185	0.0	19.0	16.8
Concrete	436	0.0	38.9	6.4
Drywall	226	0.0	na[a]	1.8
Metal	378	0.0	38.6	21.4
Plaster	444	0.0	71.2	3.6
Wood	698	0.0	30.2	18.5
Overall	2,367	0.0	36.8	12.2

[a] Not available since drywall ICP measurements were all less than 1.0 mg/cm^2 lead.

Table 6-215. Microlead I by Substrate for the First Standard Paint Reading With an Inconclusive Range Bounded by 0.4 mg/cm^2 and 1.6 mg/cm^2.

Substrate	Sample Size	% False Positive	% False Negative	% Inconclusive
Brick	186	3.5	0.0	32.3
Concrete	444	9.8	1.8	38.7
Drywall	237	5.1	na[a]	32.5
Metal	406	8.3	1.1	25.9
Plaster	463	1.5	1.7	30.7
Wood	739	12.3	0.9	26.4
Overall	2,475	7.5	1.1	30.3

[a] Not available since drywall ICP measurements were all less than 1.0 mg/cm^2 lead.

Table 6-216. X-Met 880 by Substrate for the First Standard Paint Reading With an Inconclusive Range Bounded by 0.4 mg/cm^2 and 1.6 mg/cm^2.

Substrate	Sample Size	% False Positive	% False Negative	% Inconclusive
Brick	93	0.0	61.9	8.6
Concrete	218	0.0	77.8	2.8
Drywall	113	0.0	na[a]	0.0
Metal	175	0.0	55.8	15.4
Plaster	222	0.0	92.3	0.9
Wood	353	0.0	62.0	10.5
Overall	1,174	0.0	66.4	6.8

[a] Not available since drywall ICP measurements were all less than 1.0 mg/cm^2 lead.

Table 6-217. XK-3 by Substrate for the First Standard Paint Reading With an Inconclusive Range Bounded by 0.4 mg/cm^2 and 1.6 mg/cm^2.

Substrate	Sample Size	% False Positive	% False Negative	% Inconclusive
Brick	186	19.4	2.4	49.5
Concrete	444	44.8	0.0	38.7
Drywall	237	0.4	na[a]	18.6
Metal	406	31.2	0.0	44.8
Plaster	462	26.6	1.7	40.0
Wood	743	5.6	1.3	26.2
Overall	2,478	21.8	1.1	35.1

[a] Not available since drywall ICP measurements were all less than 1.0 mg/cm^2 lead.

Table 6-218. XL by Substrate for the First Standard Paint Reading With an Inconclusive Range Bounded by 0.4 mg/cm^2 and 1.6 mg/cm^2.

Substrate	Sample Size	% False Positive	% False Negative	% Inconclusive
Brick	93	0.0	9.5	9.7
Concrete	217	0.0	7.4	13.4
Drywall	113	0.0	na[a]	6.2
Metal	189	0.7	9.1	15.3
Plaster	222	0.0	11.5	11.3
Wood	355	0.0	13.7	23.4
Overall	1,189	0.1	11.4	15.3

[a] Not available since drywall ICP measurements were all less than 1.0 mg/cm^2 lead.

Table 6-219. Standard Paint Average With an Inconclusive Range Bounded by 0.4 mg/cm^2 and 1.6 mg/cm^2.

XRF	Sample Size	% False Positive	% False Negative	% Inconclusive
Lead Analyzer K-shell	1,190	0.5	0.9	19.2
Lead Analyzer L-shell	1,190	0.0	65.5	6.2
MAP-3 K-shell	2,367	1.5	3.0	20.0
MAP-3 L-shell	2,367	0.0	37.7	12.0
Microlead I K-shell	2,475	6.2	0.2	36.0
X-Met 880 L-shell	1,174	0.0	66.4	6.6
XK-3 K-shell	2,478	21.1	1.1	37.4
XL L-shell	1,189	0.1	9.1	16.6

Table 6-220. Lead Analyzer K-shell by Substrate for the Standard Paint Average With an Inconclusive Range Bounded by 0.4 mg/cm^2 and 1.6 mg/cm^2.

Substrate	Sample Size	% False Positive	% False Negative	% Inconclusive
Brick	93	0.0	0.0	14.0
Concrete	218	1.0	0.0	19.3
Drywall	113	0.0	na[a]	9.7
Metal	189	0.0	0.0	29.1
Plaster	222	0.5	0.0	16.7
Wood	355	0.8	2.0	19.7
Overall	1,190	0.5	0.9	19.2

[a] Not available since drywall ICP measurements were all less than 1.0 mg/cm^2 lead.

Table 6-221. Lead Analyzer L-shell by Substrate for the Standard Paint Average With an Inconclusive Range Bounded by 0.4 mg/cm^2 and 1.6 mg/cm^2.

Substrate	Sample Size	% False Positive	% False Negative	% Inconclusive
Brick	93	0.0	38.1	11.8
Concrete	218	0.0	85.2	1.8
Drywall	113	0.0	na[a]	0.0
Metal	189	0.0	54.5	10.1
Plaster	222	0.0	88.5	1.8
Wood	355	0.0	64.7	10.1
Overall	1,190	0.0	65.5	6.2

[a] Not available since drywall ICP measurements were all less than 1.0 mg/cm^2 lead.

Table 6-222. MAP-3 K-shell by Substrate for the Standard Paint Average With an Inconclusive Range Bounded by 0.4 mg/cm^2 and 1.6 mg/cm^2.

Substrate	Sample Size	% False Positive	% False Negative	% Inconclusive
Brick	185	0.7	0.0	7.6
Concrete	436	1.1	9.3	9.4
Drywall	226	0.9	na[a]	15.9
Metal	378	1.4	1.1	52.9
Plaster	444	0.5	9.6	11.0
Wood	698	3.2	1.0	19.1
Overall	2,367	1.5	3.0	20.0

[a] Not available since drywall ICP measurements were all less than 1.0 mg/cm^2 lead.

Table 6-223. MAP-3 L-shell by Substrate for the Standard Paint Average With an Inconclusive Range Bounded by 0.4 mg/cm^2 and 1.6 mg/cm^2.

Substrate	Sample Size	% False Positive	% False Negative	% Inconclusive
Brick	185	0.0	19.1	16.8
Concrete	436	0.0	46.3	5.7
Drywall	226	0.0	na[a]	1.3
Metal	378	0.0	38.6	21.2
Plaster	444	0.0	73.1	3.7
Wood	698	0.0	29.7	18.9
Overall	2,367	0.0	37.7	12.0

[a] Not available since drywall ICP measurements were all less than 1.0 mg/cm^2 lead.

Table 6-224. Microlead I by Substrate for the Standard Paint Average With an Inconclusive Range Bounded by 0.4 mg/cm² and 1.6 mg/cm².

Substrate	Sample Size	% False Positive	% False Negative	% Inconclusive
Brick	186	3.5	0.0	36.6
Concrete	444	5.7	0.0	48.9
Drywall	237	6.8	na[a]	34.2
Metal	406	6.7	0.0	30.8
Plaster	463	1.5	0.0	35.9
Wood	739	10.4	0.5	31.8
Overall	2,475	6.2	0.2	36.0

[a] Not available since drywall ICP measurements were all less than 1.0 mg/cm² lead.

Table 6-225. X-Met 880 by Substrate for the Standard Paint Average With an Inconclusive Range Bounded by 0.4 mg/cm² and 1.6 mg/cm².

Substrate	Sample Size	% False Positive	% False Negative	% Inconclusive
Brick	93	0.0	61.9	8.6
Concrete	218	0.0	77.8	2.8
Drywall	113	0.0	na[a]	0.0
Metal	175	0.0	55.8	14.9
Plaster	222	0.0	92.3	0.9
Wood	353	0.0	62.0	9.9
Overall	1,174	0.0	66.4	6.6

[a] Not available since drywall ICP measurements were all less than 1.0 mg/cm² lead.

Table 6-226. XK-3 by Substrate for the Standard Paint Average With an Inconclusive Range Bounded by 0.4 mg/cm^2 and 1.6 mg/cm^2.

Substrate	Sample Size	% False Positive	% False Negative	% Inconclusive
Brick	186	18.8	2.4	57.0
Concrete	444	46.7	0.0	41.2
Drywall	237	0.0	na[a]	17.7
Metal	406	27.7	0.0	47.5
Plaster	462	25.3	1.7	44.2
Wood	745	5.0	1.3	26.9
Overall	2,478	21.1	1.1	37.4

[a] Not available since drywall ICP measurements were all less than 1.0 mg/cm^2 lead.

Table 6-227. XL by Substrate for the Standard Paint Average With an Inconclusive Range Bounded by 0.4 mg/cm^2 and 1.6 mg/cm^2.

Substrate	Sample Size	% False Positive	% False Negative	% Inconclusive
Brick	93	0.0	4.8	14.0
Concrete	217	0.0	7.4	12.4
Drywall	113	0.0	na[a]	6.2
Metal	189	0.7	9.1	18.0
Plaster	222	0.0	19.2	10.4
Wood	355	0.0	7.8	26.2
Overall	1,189	0.1	9.1	16.6

[a] Not available since drywall ICP measurements were all less than 1.0 mg/cm^2 lead.

Table 6-228. First Standard Paint Control Corrected With an Inconclusive Range Bounded by 0.4 mg/cm^2 and 1.6 mg/cm^2.

XRF	Sample Size	% False Positive	% False Negative	% Inconclusive
Lead Analyzer K-shell	1,190	0.4	1.8	18.1
Lead Analyzer L-shell	1,190	0.0	68.6	5.1
MAP-3 K-shell	2,367	2.8	2.3	27.3
MAP-3 L-shell	2,367	0.0	45.5	9.7
Microlead I K-shell	2,475	4.9	12.4	24.0
X-Met 880 L-shell	1,174	0.0	71.4	5.0
XK-3 K-shell	2,478	3.5	4.0	25.1
XL L-shell	1,189	0.1	11.8	15.7

Table 6-229. False Positive Results for First Standard Paint Readings Control Corrected With an Inconclusive Range Bounded by 0.4 mg/cm² and 1.6 mg/cm², Categorized by Their Corresponding ICP Measurement Above and Below the 0.1964 mg/cm² Median of the 1,290 ICP Measurements.

XRF	Sample Size	ICP Measurement Range (mg/cm²)	% False Positive	% Inconclusive
Lead Analyzer K-shell	608	[0 - median)	0.0	4.1
	362	[median - 1.0)	1.1	40.3
Lead Analyzer L-shell	608	[0 - median)	0.0	0.0
	362	[median - 1.0)	0.0	0.8
MAP-3 K-shell	1,209	[0 - median)	2.1	22.7
	723	[median - 1.0)	4.1	44.0
MAP-3 L-shell	1,209	[0 - median)	0.0	3.1
	723	[median - 1.0)	0.0	2.1
Microlead I K-shell	1,252	[0 - median)	3.8	22.9
	753	[median - 1.0)	6.8	31.9
X-Met 880 L-shell	596	[0 - median)	0.0	0.5
	361	[median - 1.0)	0.0	1.9
XK-3 K-shell	1,253	[0 - median)	1.6	15.2
	754	[median - 1.0)	6.8	46.2
XL L-shell	608	[0 - median)	0.0	2.8
	362	[median - 1.0)	0.3	11.9

Table 6-230. False Negative Results for First Standard Paint Readings Control Corrected With an Inconclusive Range Bounded by 0.4 mg/cm^2 and 1.6 mg/cm^2 Categorized by Their Corresponding ICP Measurement Above and Below the 2.4891 mg/cm^2 90th Percentile of the 1,290 ICP Measurements.

XRF	Sample Size	ICP Measurement Range (mg/cm^2)	% False Negative	% Inconclusive
Lead Analyzer K-shell	118	[1.0 - 90th %tile)	1.7	36.4
	102	[90th %tile - ∞)	2.0	1.0
Lead Analyzer L-shell	118	[1.0 - 90th %tile)	72.9	27.1
	102	[90th %tile - ∞)	63.7	25.5
MAP-3 K-shell	233	[1.0 - 90th %tile)	2.6	20.6
	202	[90th %tile - ∞)	2.0	3.0
MAP-3 L-shell	233	[1.0 - 90th %tile)	52.4	41.2
	202	[90th %tile - ∞)	37.6	40.1
Microlead I K-shell	240	[1.0 - 90th %tile)	17.5	25.4
	229	[90th %tile - ∞)	7.0	3.1
X-Met 880 L-shell	116	[1.0 - 90th %tile)	73.3	25.9
	101	[90th %tile - ∞)	69.3	18.8
XK-3 K-shell	242	[1.0 - 90th %tile)	2.9	31.8
	231	[90th %tile - ∞)	5.2	3.5
XL L-shell	118	[1.0 - 90th %tile)	15.3	69.5
	102	[90th %tile - ∞)	7.8	44.1

Table 6-231. Lead Analyzer K-shell by Substrate for the First Standard Paint Control Corrected Reading With an Inconclusive Range Bounded by 0.4 mg/cm² and 1.6 mg/cm².

Substrate	Sample Size	% False Positive	% False Negative	% Inconclusive
Brick	93	0.0	0.0	5.4
Concrete	218	1.0	3.7	18.8
Drywall	113	0.0	na[a]	8.0
Metal	189	0.0	0.0	27.0
Plaster	222	0.0	0.0	17.6
Wood	355	0.8	2.9	19.7
Overall	1,190	0.4	1.8	18.1

[a] Not available since drywall ICP measurements were all less than 1.0 mg/cm² lead.

Table 6-232. Lead Analyzer L-shell by Substrate for the First Standard Paint Control Corrected Reading With an Inconclusive Range Bounded by 0.4 mg/cm² and 1.6 mg/cm².

Substrate	Sample Size	% False Positive	% False Negative	% Inconclusive
Brick	93	0.0	33.3	12.9
Concrete	218	0.0	88.9	1.4
Drywall	113	0.0	na[a]	0.0
Metal	189	0.0	56.8	9.0
Plaster	222	0.0	92.3	0.9
Wood	355	0.0	69.6	7.6
Overall	1,190	0.0	68.6	5.1

[a] Not available since drywall ICP measurements were all less than 1.0 mg/cm² lead.

Table 6-233. MAP-3 K-shell by Substrate for the First Standard Paint Control Corrected Reading With an Inconclusive Range Bounded by 0.4 mg/cm^2 and 1.6 mg/cm^2.

Substrate	Sample Size	% False Positive	% False Negative	% Inconclusive
Brick	185	1.4	0.0	18.4
Concrete	436	3.7	7.4	21.1
Drywall	226	1.3	na[a]	20.4
Metal	378	1.4	1.1	40.5
Plaster	444	1.8	5.8	27.3
Wood	698	5.0	1.0	28.8
Overall	2,367	2.8	2.3	27.3

[a] Not available since drywall ICP measurements were all less than 1.0 mg/cm^2 lead.

Table 6-234. MAP-3 L-shell by Substrate for the First Standard Paint Control Corrected Reading With an Inconclusive Range Bounded by 0.4 mg/cm^2 and 1.6 mg/cm^2.

Substrate	Sample Size	% False Positive	% False Negative	% Inconclusive
Brick	185	0.0	19.0	14.1
Concrete	436	0.0	57.4	4.4
Drywall	226	0.0	na[a]	0.4
Metal	378	0.0	40.9	19.3
Plaster	444	0.0	96.2	0.5
Wood	698	0.0	36.7	15.6
Overall	2,367	0.0	45.5	9.7

[a] Not available since drywall ICP measurements were all less than 1.0 mg/cm^2 lead.

Table 6-235. Microlead I by Substrate for the First Standard Paint Control Corrected Reading With an Inconclusive Range Bounded by 0.4 mg/cm^2 and 1.6 mg/cm^2.

Substrate	Sample Size	% False Positive	% False Negative	% Inconclusive
Brick	186	2.1	0.0	27.4
Concrete	444	2.1	8.9	17.1
Drywall	237	6.0	na[a]	43.0
Metal	406	6.4	29.3	27.1
Plaster	463	2.5	16.9	18.4
Wood	739	8.3	7.3	23.1
Overall	2,475	4.9	12.4	24.0

[a] Not available since drywall ICP measurements were all less than 1.0 mg/cm^2 lead.

Table 6-236. X-Met 880 by Substrate for the First Standard Paint Control Corrected Reading With an Inconclusive Range Bounded by 0.4 mg/cm^2 and 1.6 mg/cm^2.

Substrate	Sample Size	% False Positive	% False Negative	% Inconclusive
Brick	93	0.0	61.9	6.5
Concrete	218	0.0	88.9	1.4
Drywall	113	0.0	na[a]	0.0
Metal	175	0.0	55.8	12.6
Plaster	222	0.0	96.2	0.5
Wood	353	0.0	69.0	7.6
Overall	1,174	0.0	71.4	5.0

[a] Not available since drywall ICP measurements were all less than 1.0 mg/cm^2 lead.

Table 6-237. XK-3 by Substrate for the First Standard Paint Control Corrected Reading With an Inconclusive Range Bounded by 0.4 mg/cm^2 and 1.6 mg/cm^2.

Substrate	Sample Size	% False Positive	% False Negative	% Inconclusive
Brick	186	1.4	2.4	19.4
Concrete	444	9.0	0.0	43.7
Drywall	237	0.0	na[a]	8.0
Metal	406	3.2	15.2	19.2
Plaster	462	3.5	1.7	33.1
Wood	743	1.9	1.3	19.2
Overall	2,478	3.5	4.0	25.1

[a] Not available since drywall ICP measurements were all less than 1.0 mg/cm^2 lead.

Table 6-238. XL by Substrate for the First Standard Paint Control Corrected Reading With an Inconclusive Range Bounded by 0.4 mg/cm^2 and 1.6 mg/cm^2.

Substrate	Sample Size	% False Positive	% False Negative	% Inconclusive
Brick	93	0.0	9.5	9.7
Concrete	217	0.0	14.8	12.9
Drywall	113	0.0	na[a]	7.1
Metal	189	0.7	9.1	15.3
Plaster	222	0.0	11.5	10.8
Wood	355	0.0	12.7	25.1
Overall	1,189	0.1	11.8	15.7

[a] Not available since drywall ICP measurements were all less than 1.0 mg/cm^2 lead.

Table 6-239. First Standard Paint Fully Corrected With an Inconclusive Range Bounded by 0.4 mg/cm^2 and 1.6 mg/cm^2.

XRF	Sample Size	% False Positive	% False Negative	% Inconclusive
Lead Analyzer K-shell	1,190	0.2	2.3	19.3
Lead Analyzer L-shell	1,190	0.0	61.4	6.8
MAP-3 K-shell	2,366	1.6	4.6	19.7
MAP-3 L-shell	2,366	0.0	54.0	8.2
Microlead I K-shell	2,475	2.1	1.9	30.3
X-Met 880 L-shell	1,174	0.0	71.9	4.9
XK-3 K-shell	2,478	2.2	4.7	27.6
XL L-shell	1,187	0.1	10.0	18.7

Table 6-240. Lead Analyzer K-shell by Substrate for First Standard Paint Fully Corrected Reading With an Inconclusive Range Bounded by 0.4 mg/cm^2 and 1.6 mg/cm^2.

Substrate	Sample Size	% False Positive	% False Negative	% Inconclusive
Brick	93	0.0	0.0	12.9
Concrete	218	1.0	3.7	17.9
Drywall	113	0.0	na[a]	8.0
Metal	189	0.0	4.5	29.1
Plaster	222	0.0	0.0	20.7
Wood	355	0.0	2.0	19.4
Overall	1,190	0.2	2.3	19.3

[a] Not available since drywall ICP measurements were all less than 1.0 mg/cm^2 lead.

Table 6-241. Lead Analyzer L-shell by Substrate for First Standard Paint Fully Corrected Reading With an Inconclusive Range Bounded by 0.4 mg/cm^2 and 1.6 mg/cm^2.

Substrate	Sample Size	% False Positive	% False Negative	% Inconclusive
Brick	93	0.0	38.1	11.8
Concrete	218	0.0	81.5	2.3
Drywall	113	0.0	na[a]	0.0
Metal	189	0.0	40.9	13.8
Plaster	222	0.0	88.5	1.4
Wood	355	0.0	62.8	10.1
Overall	1,190	0.0	61.4	6.8

[a] Not available since drywall ICP measurements were all less than 1.0 mg/cm^2 lead.

Table 6-242. MAP-3 K-shell by Substrate for First Standard Paint Fully Corrected Reading With an Inconclusive Range Bounded by 0.4 mg/cm^2 and 1.6 mg/cm^2.

Substrate	Sample Size	% False Positive	% False Negative	% Inconclusive
Brick	185	1.4	0.0	7.0
Concrete	435	4.5	13.0	16.3
Drywall	226	0.9	na[a]	15.0
Metal	378	0.7	1.1	31.2
Plaster	444	1.3	11.5	17.3
Wood	698	0.6	3.0	22.1
Overall	2,366	1.6	4.6	19.7

[a] Not available since drywall ICP measurements were all less than 1.0 mg/cm^2 lead.

Table 6-243. MAP-3 L-shell by Substrate for First Standard Paint Fully Corrected Reading With an Inconclusive Range Bounded by 0.4 mg/cm^2 and 1.6 mg/cm^2.

Substrate	Sample Size	% False Positive	% False Negative	% Inconclusive
Brick	185	0.0	28.8	13.0
Concrete	435	0.0	63.0	4.6
Drywall	226	0.0	na[a]	0.0
Metal	378	0.0	44.3	17.2
Plaster	444	0.0	90.4	1.8
Wood	698	0.0	51.8	11.0
Overall	2,366	0.0	54.0	8.2

[a] Not available since drywall ICP measurements were all less than 1.0 mg/cm^2 lead.

Table 6-244. Microlead I by Substrate for First Standard Paint Fully Corrected Reading With an Inconclusive Range Bounded by 0.4 mg/cm^2 and 1.6 mg/cm^2.

Substrate	Sample Size	% False Positive	% False Negative	% Inconclusive
Brick	186	2.8	0.0	27.4
Concrete	444	4.4	1.8	36.7
Drywall	237	0.0	na[a]	18.1
Metal	406	3.2	2.2	28.8
Plaster	463	0.5	3.4	38.0
Wood	739	1.9	1.8	27.1
Overall	2,475	2.1	1.9	30.3

[a] Not available since drywall ICP measurements were all less than 1.0 mg/cm^2 lead.

Table 6-245. X-Met 880 by Substrate for First Standard Paint Fully Corrected Reading With an Inconclusive Range Bounded by 0.4 mg/cm^2 and 1.6 mg/cm^2.

Substrate	Sample Size	% False Positive	% False Negative	% Inconclusive
Brick	93	0.0	61.9	6.5
Concrete	218	0.0	85.2	1.8
Drywall	113	0.0	na[a]	0.0
Metal	175	0.0	65.1	9.1
Plaster	222	0.0	96.2	0.5
Wood	353	0.0	67.0	8.5
Overall	1,174	0.0	71.9	4.9

[a] Not available since drywall ICP measurements were all less than 1.0 mg/cm^2 lead.

Table 6-246. XK-3 by Substrate for First Standard Paint Fully Corrected Reading With an Inconclusive Range Bounded by 0.4 mg/cm^2 and 1.6 mg/cm^2.

Substrate	Sample Size	% False Positive	% False Negative	% Inconclusive
Brick	186	1.4	2.4	24.7
Concrete	444	3.9	3.6	39.4
Drywall	237	0.0	na[a]	8.9
Metal	406	2.5	13.0	29.1
Plaster	462	2.5	3.4	36.1
Wood	743	1.8	2.2	21.3
Overall	2,478	2.2	4.7	27.6

[a] Not available since drywall ICP measurements were all less than 1.0 mg/cm^2 lead.

Table 6-247. XL by Substrate for First Standard Paint Fully Corrected Reading With an Inconclusive Range Bounded by 0.4 mg/cm^2 and 1.6 mg/cm^2.

Substrate	Sample Size	% False Positive	% False Negative	% Inconclusive
Brick	93	0.0	0.0	11.8
Concrete	216	0.0	7.4	14.1
Drywall	113	0.0	na[a]	15.9
Metal	188	0.7	9.3	16.0
Plaster	222	0.0	11.5	15.3
Wood	355	0.0	12.8	27.9
Overall	1,187	0.1	10.0	18.7

[a] Not available since drywall ICP measurements were all less than 1.0 mg/cm^2 lead.

Table 6-248. First Standard Paint Reading Red NIST SRM Average Corrected With an Inconclusive Range Bounded by 0.4 and 1.6 mg/cm^2.

XRF	Sample Size	% False Positive	% False Negative	% Inconclusive
Lead Analyzer K-shell	1,190	0.3	3.2	18.5
Lead Analyzer L-shell	1,190	0.0	62.7	6.5
MAP-3 K-shell	2,367	1.1	3.9	17.8
MAP-3 L-shell	2,367	0.0	51.3	8.3
Microlead I K-shell	2,475	3.1	2.1	26.9
X-Met 880 L-shell	1,174	0.0	71.4	5.0
XK-3 K-shell	2,478	2.3	4.2	25.4
XL L-shell	1,189	0.1	10.5	17.5

Table 6-249. Lead Analyzer K-shell by Substrate for the First Standard Paint Reading Red NIST SRM Average Corrected With an Inconclusive Range Bounded by 0.4 and 1.6 mg/cm^2.

Substrate	Sample Size	% False Positive	% False Negative	% Inconclusive
Brick	93	0.0	0.0	8.6
Concrete	218	1.0	3.7	18.8
Drywall	113	0.0	na[a]	9.7
Metal	189	0.0	6.8	24.9
Plaster	222	0.0	0.0	21.2
Wood	355	0.4	2.9	18.6
Overall	1,190	0.3	3.2	18.5

[a] Not available since drywall ICP measurements were all less than 1.0 mg/cm^2 lead.

Table 6-250. Lead Analyzer L-shell by Substrate for the First Standard Paint Reading Red NIST SRM Average Corrected With an Inconclusive Range Bounded by 0.4 and 1.6 mg/cm^2.

Substrate	Sample Size	% False Positive	% False Negative	% Inconclusive
Brick	93	0.0	23.8	15.1
Concrete	218	0.0	88.9	1.4
Drywall	113	0.0	na[a]	0.0
Metal	189	0.0	40.9	13.2
Plaster	222	0.0	92.3	0.9
Wood	355	0.0	65.7	9.3
Overall	1,190	0.0	62.7	6.5

[a] Not available since drywall ICP measurements were all less than 1.0 mg/cm^2 lead.

Table 6-251. MAP-3 K-shell by Substrate for the First Standard Paint Reading Red NIST SRM Average Corrected With an Inconclusive Range Bounded by 0.4 and 1.6 mg/cm^2.

Substrate	Sample Size	% False Positive	% False Negative	% Inconclusive
Brick	185	1.4	0.0	7.6
Concrete	436	1.8	13.0	16.5
Drywall	226	0.9	na[a]	12.4
Metal	378	0.0	1.1	30.2
Plaster	444	1.0	9.6	15.5
Wood	698	1.4	2.0	17.8
Overall	2,367	1.1	3.9	17.8

[a] Not available since drywall ICP measurements were all less than 1.0 mg/cm^2 lead.

Table 6-252. MAP-3 L-shell by Substrate for the First Standard Paint Reading Red NIST SRM Average Corrected With an Inconclusive Range Bounded by 0.4 and 1.6 mg/cm^2.

Substrate	Sample Size	% False Positive	% False Negative	% Inconclusive
Brick	185	0.0	26.2	13.5
Concrete	436	0.0	61.1	3.7
Drywall	226	0.0	na[a]	0.4
Metal	378	0.0	46.6	15.9
Plaster	444	0.0	92.3	0.9
Wood	698	0.0	45.2	12.9
Overall	2,367	0.0	51.3	8.3

[a] Not available since drywall ICP measurements were all less than 1.0 mg/cm^2 lead.

Table 6-253. Microlead I by Substrate for the First Standard Paint Reading Red NIST SRM Average Corrected With an Inconclusive Range Bounded by 0.4 and 1.6 mg/cm^2.

Substrate	Sample Size	% False Positive	% False Negative	% Inconclusive
Brick	186	2.8	0.0	23.7
Concrete	444	2.8	3.6	36.3
Drywall	237	1.3	na[a]	14.3
Metal	406	4.5	1.1	22.7
Plaster	463	1.5	3.4	36.7
Wood	739	4.6	2.3	22.2
Overall	2,475	3.1	2.1	26.9

[a] Not available since drywall ICP measurements were all less than 1.0 mg/cm^2 lead.

Table 6-254. X-MET 880 by Substrate for the First Standard Paint Reading Red NIST SRM Average Corrected With an Inconclusive Range Bounded by 0.4 and 1.6 mg/cm^2.

Substrate	Sample Size	% False Positive	% False Negative	% Inconclusive
Brick	93	0.0	61.9	6.5
Concrete	218	0.0	85.2	1.8
Drywall	113	0.0	na[a]	0.0
Metal	175	0.0	58.1	10.9
Plaster	222	0.0	96.2	0.5
Wood	353	0.0	69.0	8.2
Overall	1,174	0.0	71.4	5.0

[a] Not available since drywall ICP measurements were all less than 1.0 mg/cm^2 lead.

Table 6-255. XK-3 by Substrate for the First Standard Paint Reading Red NIST SRM Average Corrected With an Inconclusive Range Bounded by 0.4 and 1.6 mg/cm^2.

Substrate	Sample Size	% False Positive	% False Negative	% Inconclusive
Brick	186	2.8	2.4	21.0
Concrete	444	3.6	3.6	37.6
Drywall	237	0.0	na[a]	8.0
Metal	406	2.9	12.0	24.6
Plaster	462	1.7	1.7	35.9
Wood	743	2.5	2.2	18.6
Overall	2,478	2.3	4.2	25.4

[a] Not available since drywall ICP measurements were all less than 1.0 mg/cm^2 lead.

Table 6-256. XL by Substrate for the First Standard Paint Reading Red NIST SRM Average Corrected With an Inconclusive Range Bounded by 0.4 and 1.6 mg/cm^2.

Substrate	Sample Size	% False Positive	% False Negative	% Inconclusive
Brick	93	0.0	0.0	12.9
Concrete	217	0.0	7.4	13.4
Drywall	113	0.0	na[a]	12.4
Metal	189	0.7	9.1	15.9
Plaster	222	0.0	11.5	12.6
Wood	355	0.0	13.7	26.8
Overall	1,189	0.1	10.5	17.5

[a] Not available since drywall ICP measurements were all less than 1.0 mg/cm^2 lead.

6.5.4.1 First Standard XRF Readings With an Alternate (0.7 - 1.3 mg/cm^2) Inconclusive Range

Table 6-257 shows overall error rates by instrument for the first standard paint reading using the (0.7 - 1.3 mg/cm^2) inconclusive range. Comparisons to Table 6-208, which shows overall error rates by instrument for the first standard paint reading using the (0.4 - 1.6 mg/cm^2) inconclusive range, were made to examine differences between the two methods for classifying results using two different inconclusive ranges.

All but two of the sixteen error percentages in Table 6-257 are larger than those in Table 6-208. The exceptions that remained unchanged were the zero percent false positive results for the Lead Analyzer L-shell and X-MET 880. All of the false negative rate increases for the L-shell instruments were substantial including an increase from 11.4% to 29.5% for the XL.

All inconclusive rates in Table 6-258 decreased from those in Table 6-208. The largest of the inconclusive percentages for the L-shell instruments was attributable to the XL which had 5.9% of its results classified as inconclusive. For the K-shell instruments, inconclusive percentages ranged from 6.0% for the Lead Analyzer to 17.0% for the XK-3.

Similar comparative results are shown in Tables 6-258 through 6-265 which provide results for the first standard paint reading by substrate. Tables 6-211 and 6-218 are the companion tables for classifying the first standard paint reading with a (0.4 - 1.6 mg/cm^2) inconclusive range.

Table 6-158 is useful for making comparisons to Table 6-257. Table 6-158 displays results for the first standard paint reading without an inconclusive range. Comparing Table 6-257 to Table 6-158 shows that the results described for XRF classification without an inconclusive range in Table 6-158 are reflected in Table 6-257. That is, the false negative rates for the L-shell instruments are high in both tables and the error percentages for the K-shell instruments are similar in the two tables, although lower in Table 6-257. Using the alternate inconclusive range, Table 6-258 shows that the Lead Analyzer K-shell instrument has all error rates below five percent and inconclusive rates ranging from 3.5% on drywall to 9.6% on wood with an overall rate of 6.0%. Table 6-260 shows that the MAP-3 K-shell instrument has

all error rates below ten percent except for two false positive rates: 13.0% on concrete and 15.4% on plaster. The inconclusive percentages for the MAP-3 range from 6.0% on brick to 25.4% on metal with an overall inconclusive rate of 11.1%. Table 6-262 shows that all of the false negative rates for the Microlead I were low but its false positive rates range from 3.0% on plaster to 19.3% on wood and its inconclusive rates range from 10.8% on wood to 19.4% on concrete. Similarly, the XK-3 has low false negative rates on all substrates but its false positive and inconclusive rates were high. Table 6-264 shows that the overall false positive rate for the XK-3 was 29.4% and over the individual substrates it ranged from 1.3% on drywall to 55.7% on concrete. The inconclusive rates for the XK-3 range from 6.8% on drywall to 25.4% on metal.

6.5.4.2 Average of Three Standard XRF Readings With an Alternate (0.7 - 1.3 mg/cm^2) Inconclusive Range

The average of the three standard paint readings at a sampling location were classified using the (0.7 - 1.3 mg/cm^2) alternate inconclusive range. Table 6-266 shows overall error rates by instrument. Comparisons to Table 6-219, which shows overall error rates by instrument for the standard paint average using the (0.4 - 1.6 mg/cm^2) inconclusive range, were made to examine differences between the two methods for classifying the average of three readings using different inconclusive ranges.

All inconclusive rates shown in Table 6-266 decreased from those shown in Table 6-219. The Lead Analyzer K-shell and the MAP-3 K-shell instruments have all overall rates (error rates and inconclusive rates) less than 10%. The other two K-shell instruments, the Microlead I and XK-3, have 13.6 and 18.8% inconclusive rates and 10.4% and 29.0% false positive rates, respectively.

Similar comparative results are shown in Tables 6-267 through 6-274 which provide results by substrate. Tables 6-220 and 6-226 are the companion tables for the (0.4 - 1.6 mg/cm^2) inconclusive range.

6.5.4.3 Standard XRF Readings Control Corrected With an Alternate (0.7- 1.3 mg/cm^2) Inconclusive Range

The first standard paint reading was "control corrected" by subtracting the average of all the initial and ending red NIST SRM control block readings in the dwelling, minus 1.02 mg/cm^2. Table 6-275 shows overall error rates by instrument for the first standard paint control corrected readings using the (0.7 - 1.3 mg/cm^2) inconclusive range. This table should be compared to Table 6-228, which shows the same information for the first standard paint control corrected reading using the (0.4 - 1.6 mg/cm^2) inconclusive range.

All but two of the sixteen error percentages shown in Table 6-275 are larger than those in Table 6-228. The exceptions that remained unchanged were the zero percent false positive results for the Lead Analyzer L-shell and X-MET 880. Again, all of the false negative rate increases for the L-shell instruments were substantial including an increase from 11.8% to 28.6% for the XL. The false positive rates show relatively small differences between the two tables.

Tables 6-276 through 6-283 display the control corrected error rates by substrate for the eight instruments, and are to be compared to Tables 6-231 through 6-238 which applied the (0.4 - 1.6 mg/cm^2) inconclusive range.

6.5.4.4 Standard XRF Readings Fully Corrected With an (0.7 - 1.3 mg/cm^2) Inconclusive Range

Table 6-284 shows overall error rates by instrument for the first standard paint fully corrected readings using the (0.7 - 1.3 mg/cm^2) inconclusive range. This table should be compared to Table 6-239, which shows the same information for the results classified using the (0.4 - 1.6 mg/cm^2) inconclusive range. Tables 6-285 through 6-292 provide the results by substrate categories for the (0.7 - 1.3 mg/cm^2) inconclusive range and Tables 6-240 through 6-247 are the companion tables for the (0.4 - 1.6 mg/cm^2) inconclusive range.

Again, similarities and differences noted in the last two sections apply here when comparing the results in Table 6-284 to Table 6-239. However, the results in Table 6-284 show that K-shell instruments have error rates less than ten percent for

either false positive or false negative while maintaining inconclusive percentages near ten percent. The largest inconclusive percentage was for the Microlead I which had 13.8% of its results classified as inconclusive.

6.5.4.5 Standard XRF Readings Red NIST SRM Average Corrected With an (0.7 - 1.3 mg/cm^2) Inconclusive Range

For this analysis, the first standard paint reading was "red NIST SRM average corrected". Table 6-293 shows overall error rates by instrument using the (0.7 - 1.3 mg/cm^2) inconclusive range. Tables 6-294 through 6-301 display the information by substrate. Table 6-248 provides similar information for results classified using the (0.4 - 1.6 mg/cm^2) inconclusive range and the comparative tables by substrate are Tables 6-249 through 6-256.

All but three of the sixteen error percentages shown in Table 6-293 are larger than those in Table 6-248. The exceptions that remained unchanged were the zero percent false positive results for the Lead Analyzer L-shell and X-MET 880 and the 0.1% false positive rate for the XL. Again, all of the false negative rate increases for the L-shell instruments were substantial including an increase from 10.5% to 25.5% for the XL. Similarly, the false positive rate increases were small.

The results in Table 6-293 show that K-shell instruments have error rates less than ten percent while maintaining inconclusive percentages near ten percent. The largest inconclusive percentage was for the XK-3 which had 11.9% of its results classified as inconclusive. The results shown in Table 6-293 show that, for the K-shell instruments, the inconclusive rates decreased noticeably compared to those in Table 6-248 and that there were relatively small increases in the error rates.

Tables 6-285, 6-287, 6-289, and 6-291 show results by substrate for the K-shell instruments. Misclassification rates for the K-shell instruments were relatively consistent across substrates with three exceptions. The exceptions were the false negative rates for the MAP-3 K-shell on concrete and plaster and the false negative rate for the XK-3 on metal.

Table 6-257. First Standard Paint Reading With an Alternative Inconclusive Range Between 0.7 and 1.3 mg/cm^2.

XRF	Sample Size	% False Positive	% False Negative	% Inconclusive
Lead Analyzer K-shell	1,190	1.2	2.7	6.0
Lead Analyzer L-shell	1,190	0.0	83.6	1.5
MAP-3 K-shell	2,367	4.1	4.6	11.1
MAP-3 L-shell	2,367	0.3	58.6	5.1
Microlead I K-shell	2,475	12.3	2.1	14.5
X-Met 880 L-shell	1,174	0.0	82.5	2.0
XK-3 K-shell	2,478	29.6	1.7	17.0
XL L-shell	1,189	0.2	29.5	5.9

Table 6-258. Lead Analyzer K-shell by Substrate for the First Standard Paint Reading With an Alternative Inconclusive Range Between 0.7 and 1.3 mg/cm^2.

Substrate	Sample Size	% False Positive	% False Negative	% Inconclusive
Brick	93	0.0	0.0	4.3
Concrete	218	1.0	3.7	3.7
Drywall	113	1.8	na[a]	3.5
Metal	189	0.7	4.5	6.3
Plaster	222	0.5	3.8	4.1
Wood	355	2.4	3.9	9.6
Overall	1,190	1.2	2.7	6.0

[a] Not available since drywall ICP measurements were all less than 1.0 mg/cm^2 lead.

Table 6-259. Lead Analyzer L-shell by Substrate for the First Standard Paint Reading With an Alternative Inconclusive Range Between 0.7 and 1.3 mg/cm^2.

Substrate	Sample Size	% False Positive	% False Negative	% Inconclusive
Brick	93	0.0	85.7	1.1
Concrete	218	0.0	92.6	0.9
Drywall	113	0.0	na[a]	0.0
Metal	189	0.0	65.9	4.8
Plaster	222	0.0	100.0	0.0
Wood	355	0.0	84.3	1.7
Overall	1,190	0.0	83.6	1.5

[a] Not available since drywall ICP measurements were all less than 1.0 mg/cm^2 lead.

Table 6-260. MAP-3 K-shell by Substrate for the First Standard Paint Reading With an Alternative Inconclusive Range Between 0.7 and 1.3 mg/cm^2.

Substrate	Sample Size	% False Positive	% False Negative	% Inconclusive
Brick	185	2.1	0.0	6.0
Concrete	436	3.9	13.0	8.0
Drywall	226	1.8	na[a]	8.8
Metal	378	6.2	1.1	25.4
Plaster	444	2.0	15.4	6.5
Wood	698	6.2	2.0	10.3
Overall	2,367	4.1	4.6	11.1

[a] Not available since drywall ICP measurements were all less than 1.0 mg/cm^2 lead.

Table 6-261. MAP-3 L-shell by Substrate for the First Standard Paint Reading With an Alternative Inconclusive Range Between 0.7 and 1.3 mg/cm^2.

Substrate	Sample Size	% False Positive	% False Negative	% Inconclusive
Brick	185	0.0	26.2	4.9
Concrete	436	0.0	66.7	2.5
Drywall	226	0.0	na[a]	0.4
Metal	378	1.7	53.4	9.5
Plaster	444	0.0	98.1	0.2
Wood	698	0.0	55.3	8.9
Overall	2,367	0.3	58.6	5.1

[a] Not available since drywall ICP measurements were all less than 1.0 mg/cm^2 lead.

Table 6-262. Microlead I by Substrate for the First Standard Paint Reading With an Alternative Inconclusive Range Between 0.7 and 1.3 mg/cm^2.

Substrate	Sample Size	% False Positive	% False Negative	% Inconclusive
Brick	186	11.1	0.0	15.1
Concrete	444	15.5	1.8	19.4
Drywall	237	9.7	na[a]	14.8
Metal	406	11.1	2.2	15.0
Plaster	463	3.0	5.1	14.9
Wood	739	19.3	1.8	10.8
Overall	2,475	12.3	2.1	14.5

[a] Not available since drywall ICP measurements were all less than 1.0 mg/cm^2 lead.

Table 6-263. X-MET 880 by Substrate for the First Standard Paint Reading With an Alternative Inconclusive Range Between 0.7 and 1.3 mg/cm^2.

Substrate	Sample Size	% False Positive	% False Negative	% Inconclusive
Brick	93	0.0	85.7	1.1
Concrete	218	0.0	92.6	0.9
Drywall	113	0.0	na[a]	0.0
Metal	175	0.0	65.1	6.9
Plaster	222	0.0	100.0	0.0
Wood	353	0.0	82.0	2.5
Overall	1,174	0.0	82.5	2.0

[a] Not available since drywall ICP measurements were all less than 1.0 mg/cm^2 lead.

Table 6-264. XK-3 by Substrate for the First Standard Paint Reading With an Alternative Inconclusive Range Between 0.7 and 1.3 mg/cm^2.

Substrate	Sample Size	% False Positive	% False Negative	% Inconclusive
Brick	186	32.6	2.4	24.7
Concrete	444	55.7	0.0	20.7
Drywall	237	1.3	na[a]	6.8
Metal	406	41.4	2.2	25.4
Plaster	462	35.2	1.7	19.9
Wood	743	10.6	1.8	9.8
Overall	2,478	29.6	1.7	17.0

[a] Not available since drywall ICP measurements were all less than 1.0 mg/cm^2 lead.

Table 6-265. XL by Substrate for the First Standard Paint Reading With an Alternative Inconclusive Range Between 0.7 and 1.3 mg/cm^2.

Substrate	Sample Size	% False Positive	% False Negative	% Inconclusive
Brick	93	0.0	23.8	1.1
Concrete	217	0.5	25.9	6.0
Drywall	113	0.0	na[a]	2.7
Metal	189	0.7	20.5	4.2
Plaster	222	0.0	46.2	4.1
Wood	355	0.0	31.4	10.1
Overall	1,189	0.2	29.5	5.9

[a] Not available since drywall ICP measurements were all less than 1.0 mg/cm^2 lead.

Table 6-266. Standard Paint Average With an Alternative Inconclusive Range Between 0.7 mg/cm^2 and 1.3 mg/cm^2.

XRF	Sample Size	% False Positive	% False Negative	% Inconclusive
Lead Analyzer K-shell	1,190	0.8	3.2	7.0
Lead Analyzer L-shell	1,190	0.0	83.6	1.6
MAP-3 K-shell	2,367	2.6	3.7	8.7
MAP-3 L-shell	2,367	0.3	57.9	5.0
Microlead I K-shell	2,475	10.4	1.1	13.6
X-Met 880 L-shell	1,174	0.0	82.5	2.0
XK-3 K-shell	2,478	29.0	1.7	18.8
XL L-shell	1,189	0.3	26.4	6.8

Table 6-267. Lead Analyzer K-shell by Substrate for the Standard Paint Average With an Alternative Inconclusive Range Between 0.7 mg/cm^2 and 1.3 mg/cm^2.

Substrate	Sample Size	% False Positive	% False Negative	% Inconclusive
Brick	93	0.0	0.0	5.4
Concrete	218	1.1	3.7	3.2
Drywall	113	0.0	na[a]	6.2
Metal	189	0.0	2.3	9.5
Plaster	222	0.5	3.9	5.0
Wood	355	2.0	3.9	9.9
Overall	1,190	0.8	3.2	7.0

[a] Not available since drywall ICP measurements were all less than 1.0 mg/cm^2 lead.

Table 6-268. Lead Analyzer L-shell by Substrate for the Standard Paint Average With an Alternative Inconclusive Range Between 0.7 mg/cm^2 and 1.3 mg/cm^2.

Substrate	Sample Size	% False Positive	% False Negative	% Inconclusive
Brick	93	0.0	85.7	1.1
Concrete	218	0.0	92.6	0.9
Drywall	113	0.0	na[a]	0.0
Metal	189	0.0	65.9	4.8
Plaster	222	0.0	100.0	0.5
Wood	355	0.0	84.3	1.7
Overall	1,190	0.0	83.6	1.6

[a] Not available since drywall ICP measurements were all less than 1.0 mg/cm^2 lead.

Table 6-269. MAP-3 K-shell by Substrate for the Standard Paint Average With an Alternative Inconclusive Range Between 0.7 mg/cm^2 and 1.3 mg/cm^2.

Substrate	Sample Size	% False Positive	% False Negative	% Inconclusive
Brick	185	1.4	0.0	3.8
Concrete	436	1.3	13.0	3.4
Drywall	226	0.9	na[a]	3.5
Metal	378	3.8	1.1	24.6
Plaster	444	0.8	11.5	5.0
Wood	698	5.6	1.0	8.6
Overall	2,367	2.6	3.7	8.7

[a] Not available since drywall ICP measurements were all less than 1.0 mg/cm^2 lead.

Table 6-270. MAP-3 L-shell by Substrate for the Standard Paint Average With an Alternative Inconclusive Range Between 0.7 mg/cm^2 and 1.3 mg/cm^2.

Substrate	Sample Size	% False Positive	% False Negative	% Inconclusive
Brick	185	0.0	26.2	5.4
Concrete	436	0.0	70.4	2.1
Drywall	226	0.0	na[a]	0.0
Metal	378	2.1	53.4	8.5
Plaster	444	0.0	94.2	0.7
Wood	698	0.0	53.8	9.2
Overall	2,367	0.3	57.9	5.0

[a] Not available since drywall ICP measurements were all less than 1.0 mg/cm^2 lead.

Table 6-271. Microlead I by Substrate for the Standard Paint Average With an Alternative Inconclusive Range Between 0.7 mg/cm^2 and 1.3 mg/cm^2.

Substrate	Sample Size	% False Positive	% False Negative	% Inconclusive
Brick	186	8.3	0.0	14.5
Concrete	444	10.3	0.0	19.4
Drywall	237	11.4	na[a]	14.3
Metal	406	10.8	0.0	11.1
Plaster	463	1.7	3.4	11.7
Wood	739	17.0	1.4	12.3
Overall	2,475	10.4	1.1	13.6

[a] Not available since drywall ICP measurements were all less than 1.0 mg/cm^2 lead.

Table 6-272. X-MET 880 by Substrate for the Standard Paint Average With an Alternative Inconclusive Range Between 0.7 mg/cm^2 and 1.3 mg/cm^2.

Substrate	Sample Size	% False Positive	% False Negative	% Inconclusive
Brick	93	0.0	85.7	1.1
Concrete	218	0.0	92.6	0.9
Drywall	113	0.0	na[a]	0.0
Metal	175	0.0	65.1	6.9
Plaster	222	0.0	100.0	0.0
Wood	353	0.0	82.0	2.5
Overall	1,174	0.0	82.5	2.0

[a] Not available since drywall ICP measurements were all less than 1.0 mg/cm^2 lead.

Table 6-273. XK-3 by Substrate for the Standard Paint Average With an Alternative Inconclusive Range Between 0.7 mg/cm^2 and 1.3 mg/cm^2.

Substrate	Sample Size	% False Positive	% False Negative	% Inconclusive
Brick	186	33.3	2.4	28.0
Concrete	444	57.0	0.0	23.0
Drywall	237	0.9	na[a]	7.6
Metal	406	40.4	2.2	26.1
Plaster	462	34.2	1.7	24.0
Wood	743	8.9	1.8	10.4
Overall	2,478	29.0	1.7	18.8

[a] Not available since drywall ICP measurements were all less than 1.0 mg/cm^2 lead.

Table 6-274. XL by Substrate for the Standard Paint Average With an Alternative Inconclusive Range Between 0.7 mg/cm^2 and 1.3 mg/cm^2.

Substrate	Sample Size	% False Positive	% False Negative	% Inconclusive
Brick	93	0.0	23.8	0.0
Concrete	217	0.5	25.9	6.0
Drywall	113	0.0	na[a]	2.7
Metal	189	0.7	18.2	5.3
Plaster	222	0.0	34.6	5.4
Wood	355	0.4	28.4	12.1
Overall	1,189	0.3	26.4	6.8

[a] Not available since drywall ICP measurements were all less than 1.0 mg/cm^2 lead.

Table 6-275. First Standard Paint Reading Control Corrected With an Alternative Inconclusive Range Bounded by 0.7 mg/cm^2 and 1.3 mg/cm^2.

XRF	Sample Size	% False Positive	% False Negative	% Inconclusive
Lead Analyzer K-shell	1,190	0.9	4.5	6.2
Lead Analyzer L-shell	1,190	0.0	84.5	1.3
MAP-3 K-shell	2,367	5.2	3.7	11.8
MAP-3 L-shell	2,367	0.2	64.1	4.4
Microlead I K-shell	2,475	8.4	14.5	11.1
X-Met 880 L-shell	1,174	0.0	85.3	1.4
XK-3 K-shell	2,478	6.5	6.8	12.4
XL L-shell	1,189	0.2	28.6	6.7

Table 6-276. Lead Analyzer K-shell by Substrate for the First Standard Paint Reading Control Corrected With an Alternative Inconclusive Range Bounded by 0.7 mg/cm^2 and 1.3 mg/cm^2.

Substrate	Sample Size	% False Positive	% False Negative	% Inconclusive
Brick	93	0.0	0.0	3.2
Concrete	218	1.0	7.4	2.8
Drywall	113	0.9	na[a]	2.7
Metal	189	0.7	6.8	6.9
Plaster	222	0.5	3.8	5.4
Wood	355	1.6	3.9	10.4
Overall	1,190	0.9	4.5	6.2

[a] Not available since drywall ICP measurements were all less than 1.0 mg/cm^2 lead.

Table 6-277. Lead Analyzer L-shell by Substrate for the First Standard Paint Reading Control Corrected With an Alternative Inconclusive Range Bounded by 0.7 mg/cm^2 and 1.3 mg/cm^2.

Substrate	Sample Size	% False Positive	% False Negative	% Inconclusive
Brick	93	0.0	85.7	1.1
Concrete	218	0.0	92.6	0.9
Drywall	113	0.0	na[a]	0.0
Metal	189	0.0	65.9	4.8
Plaster	222	0.0	100.0	0.0
Wood	355	0.0	86.3	1.1
Overall	1,190	0.0	84.5	1.3

[a] Not available since drywall ICP measurements were all less than 1.0 mg/cm^2 lead.

Table 6-278. MAP-3 K-shell by Substrate for the First Standard Paint Reading Control Corrected With an Alternative Inconclusive Range Bounded by 0.7 mg/cm^2 and 1.3 mg/cm^2.

Substrate	Sample Size	% False Positive	% False Negative	% Inconclusive
Brick	185	5.6	0.0	7.6
Concrete	436	6.0	13.0	8.5
Drywall	226	1.3	na[a]	8.0
Metal	378	3.8	1.1	18.8
Plaster	444	4.1	7.7	14.9
Wood	698	8.0	2.0	10.5
Overall	2,367	5.2	3.7	11.8

[a] Not available since drywall ICP measurements were all less than 1.0 mg/cm^2 lead.

Table 6-279. MAP-3 L-shell by Substrate for the First Standard Paint Reading Control Corrected With an Alternative Inconclusive Range Bounded by 0.7 mg/cm^2 and 1.3 mg/cm^2.

Substrate	Sample Size	% False Positive	% False Negative	% Inconclusive
Brick	185	0.0	26.2	8.1
Concrete	436	0.0	83.3	0.7
Drywall	226	0.0	na[a]	0.4
Metal	378	1.0	52.3	10.1
Plaster	444	0.0	100.0	0.0
Wood	698	0.0	62.8	6.9
Overall	2,367	0.2	64.1	4.4

[a] Not available since drywall ICP measurements were all less than 1.0 mg/cm^2 lead.

Table 6-280. Microlead I by Substrate for the First Standard Paint Reading Control Corrected With an Alternative Inconclusive Range Bounded by 0.7 mg/cm^2 and 1.3 mg/cm^2.

Substrate	Sample Size	% False Positive	% False Negative	% Inconclusive
Brick	186	9.7	0.0	11.8
Concrete	444	4.4	17.9	8.3
Drywall	237	9.3	na[a]	21.5
Metal	406	7.6	30.4	13.1
Plaster	463	4.7	23.7	7.6
Wood	739	13.9	7.3	10.3
Overall	2,475	8.4	14.5	11.1

[a] Not available since drywall ICP measurements were all less than 1.0 mg/cm^2 lead.

Table 6-281. X-MET 880 by Substrate for the First Standard Paint Reading Control Corrected With an Alternative Inconclusive Range Bounded by 0.7 mg/cm^2 and 1.3 mg/cm^2.

Substrate	Sample Size	% False Positive	% False Negative	% Inconclusive
Brick	93	0.0	85.7	1.1
Concrete	218	0.0	92.6	0.9
Drywall	113	0.0	na[a]	0.0
Metal	175	0.0	67.4	5.1
Plaster	222	0.0	100.0	0.0
Wood	353	0.0	87.0	1.1
Overall	1,174	0.0	85.3	1.4

[a] Not available since drywall ICP measurements were all less than 1.0 mg/cm^2 lead.

Table 6-282. XK-3 by Substrate for the First Standard Paint Reading Control Corrected With an Alternative Inconclusive Range Bounded by 0.7 mg/cm² and 1.3 mg/cm².

Substrate	Sample Size	% False Positive	% False Negative	% Inconclusive
Brick	186	4.9	2.4	9.7
Concrete	444	15.7	3.6	20.0
Drywall	237	0.0	na[a]	4.2
Metal	406	5.4	17.4	9.9
Plaster	462	7.4	10.2	15.2
Wood	743	2.9	3.1	10.8
Overall	2,478	6.5	6.8	12.4

[a] Not available since drywall ICP measurements were all less than 1.0 mg/cm² lead.

Table 6-283. XL by Substrate for the First Standard Paint Reading Control Corrected With an Alternative Inconclusive Range Bounded by 0.7 mg/cm² and 1.3 mg/cm².

Substrate	Sample Size	% False Positive	% False Negative	% Inconclusive
Brick	93	0.0	23.8	1.1
Concrete	217	0.5	25.9	6.5
Drywall	113	0.0	na[a]	3.5
Metal	189	0.7	20.5	4.2
Plaster	222	0.0	50.0	4.1
Wood	355	0.0	28.4	12.4
Overall	1,189	0.2	28.6	6.7

[a] Not available since drywall ICP measurements were all less than 1.0 mg/cm² lead.

Table 6-284. First Standard Paint Reading Fully Corrected With an Alternative Inconclusive Range Bounded by 0.7 mg/cm^2 and 1.3 mg/cm^2.

XRF	Sample Size	% False Positive	% False Negative	% Inconclusive
Lead Analyzer K-shell	1,190	0.5	3.6	6.2
Lead Analyzer L-shell	1,190	0.0	82.3	1.8
MAP-3 K-shell	2,366	2.5	7.8	8.2
MAP-3 L-shell	2,366	0.1	67.6	3.9
Microlead I K-shell	2,475	4.2	4.7	13.8
X-Met 880 L-shell	1,174	0.0	85.7	1.3
XK-3 K-shell	2,478	5.0	6.3	12.3
XL L-shell	1,187	0.1	26.0	7.2

Table 6-285. Lead Analyzer K-shell by Substrate for the First Standard Paint Reading Fully Corrected With an Alternative Inconclusive Range Bounded by 0.7 mg/cm^2 and 1.3 mg/cm^2.

Substrate	Sample Size	% False Positive	% False Negative	% Inconclusive
Brick	93	0.0	0.0	4.3
Concrete	218	1.0	3.7	5.0
Drywall	113	0.0	na[a]	2.7
Metal	189	0.0	6.8	6.3
Plaster	222	0.0	3.8	5.9
Wood	355	1.2	2.9	8.7
Overall	1,190	0.5	3.6	6.2

[a] Not available since drywall ICP measurements were all less than 1.0 mg/cm^2 lead.

Table 6-286. Lead Analyzer L-shell by Substrate for the First Standard Paint Reading Fully Corrected With an Alternative Inconclusive Range Bounded by 0.7 mg/cm^2 and 1.3 mg/cm^2.

Substrate	Sample Size	% False Positive	% False Negative	% Inconclusive
Brick	93	0.0	76.2	3.2
Concrete	218	0.0	92.6	0.9
Drywall	113	0.0	na[a]	0.0
Metal	189	0.0	61.4	6.3
Plaster	222	0.0	100.0	0.0
Wood	355	0.0	85.3	1.4
Overall	1,190	0.0	82.3	1.8

[a] Not available since drywall ICP measurements were all less than 1.0 mg/cm^2 lead.

Table 6-287. MAP-3 K-shell by Substrate for the First Standard Paint Reading Fully Corrected With an Alternative Inconclusive Range Bounded by 0.7 mg/cm^2 and 1.3 mg/cm^2.

Substrate	Sample Size	% False Positive	% False Negative	% Inconclusive
Brick	185	2.1	2.4	1.6
Concrete	435	5.2	20.4	9.2
Drywall	226	0.9	na[a]	3.1
Metal	378	1.7	1.1	13.5
Plaster	444	2.0	19.2	6.8
Wood	698	2.0	5.5	9.2
Overall	2,366	2.5	7.8	8.2

[a] Not available since drywall ICP measurements were all less than 1.0 mg/cm^2 lead.

Table 6-288. MAP-3 L-shell by Substrate for the First Standard Paint Reading Fully Corrected With an Alternative Inconclusive Range Bounded by 0.7 mg/cm^2 and 1.3 mg/cm^2.

Substrate	Sample Size	% False Positive	% False Negative	% Inconclusive
Brick	185	0.0	31.0	9.2
Concrete	435	0.0	85.2	0.9
Drywall	226	0.0	na[a]	0.0
Metal	378	0.0	53.4	8.2
Plaster	444	0.3	98.1	0.5
Wood	698	0.0	68.8	5.6
Overall	2,366	0.1	67.6	3.9

[a] Not available since drywall ICP measurements were all less than 1.0 mg/cm^2 lead.

Table 6-289. Microlead I by Substrate for the First Standard Paint Reading Fully Corrected With an Alternative Inconclusive Range Bounded by 0.7 mg/cm^2 and 1.3 mg/cm^2.

Substrate	Sample Size	% False Positive	% False Negative	% Inconclusive
Brick	186	4.9	0.0	16.1
Concrete	444	8.8	3.6	17.3
Drywall	237	0.8	na[a]	6.8
Metal	406	4.8	4.3	12.8
Plaster	463	2.2	3.4	17.9
Wood	739	3.5	6.4	11.2
Overall	2,475	4.2	4.7	13.8

[a] Not available since drywall ICP measurements were all less than 1.0 mg/cm^2 lead.

Table 6-290. X-MET 880 by Substrate for the First Standard Paint Reading Fully Corrected With an Alternative Inconclusive Range Bounded by 0.7 mg/cm^2 and 1.3 mg/cm^2.

Substrate	Sample Size	% False Positive	% False Negative	% Inconclusive
Brick	93	0.0	85.7	1.1
Concrete	218	0.0	92.6	0.9
Drywall	113	0.0	na[a]	0.0
Metal	175	0.0	69.8	5.1
Plaster	222	0.0	100.0	0.0
Wood	353	0.0	87.0	0.8
Overall	1,174	0.0	85.7	1.3

[a] Not available since drywall ICP measurements were all less than 1.0 mg/cm^2 lead.

Table 6-291. XK-3 by Substrate for the First Standard Paint Reading Fully Corrected With an Alternative Inconclusive Range Bounded by 0.7 mg/cm^2 and 1.3 mg/cm^2.

Substrate	Sample Size	% False Positive	% False Negative	% Inconclusive
Brick	186	6.3	2.4	9.7
Concrete	444	8.5	5.4	19.1
Drywall	237	0.0	na[a]	3.8
Metal	406	5.4	14.1	11.8
Plaster	462	5.5	5.1	17.1
Wood	743	3.9	4.5	9.0
Overall	2,478	5.0	6.3	12.3

[a] Not available since drywall ICP measurements were all less than 1.0 mg/cm^2 lead.

Table 6-292. XL by Substrate for the First Standard Paint Reading Fully Corrected With an Alternative Inconclusive Range Bounded by 0.7 mg/cm^2 and 1.3 mg/cm^2.

Substrate	Sample Size	% False Positive	% False Negative	% Inconclusive
Brick	93	0.0	19.0	1.1
Concrete	216	0.0	33.3	6.5
Drywall	113	0.0	na[a]	3.5
Metal	188	0.7	18.6	6.4
Plaster	222	0.0	42.3	5.4
Wood	355	0.0	24.5	11.8
Overall	1,187	0.1	26.0	7.2

[a] Not available since drywall ICP measurements were all less than 1.0 mg/cm^2 lead.

Table 6-293. First Standard Paint Red NIST SRM Average Corrected Reading With an Alternative Inconclusive Range Bounded by 0.7 mg/cm^2 and 1.3 mg/cm^2.

XRF	Sample Size	% False Positive	% False Negative	% Inconclusive
Lead Analyzer K-shell	1,190	0.7	4.1	5.7
Lead Analyzer L-shell	1,190	0.0	82.3	1.7
MAP-3 K-shell	2,367	2.6	7.4	6.8
MAP-3 L-shell	2,367	0.1	66.0	4.2
Microlead I K-shell	2,475	4.9	5.3	11.7
X-Met 880 L-shell	1,174	0.0	84.8	1.5
XK-3 K-shell	2,478	5.5	6.6	11.9
XL L-shell	1,189	0.1	25.5	7.4

Table 6-294. Lead Analyzer K-shell by Substrate for the First Standard Paint Reading Red NIST SRM Average Corrected With an Alternative Inconclusive Range Bounded by 0.7 mg/cm^2 and 1.3 mg/cm^2.

Substrate	Sample Size	% False Positive	% False Negative	% Inconclusive
Brick	93	0.0	0.0	3.2
Concrete	218	1.0	3.7	4.1
Drywall	113	0.0	na[a]	3.5
Metal	189	0.7	6.8	5.8
Plaster	222	0.5	3.8	4.5
Wood	355	1.2	3.9	8.7
Overall	1,190	0.7	4.1	5.7

[a] Not available since drywall ICP measurements were all less than 1.0 mg/cm^2 lead.

Table 6-295. Lead Analyzer L-shell by Substrate for the First Standard Paint Reading Red NIST SRM Average Corrected With an Alternative Inconclusive Range Bounded by 0.7 mg/cm^2 and 1.3 mg/cm^2.

Substrate	Sample Size	% False Positive	% False Negative	% Inconclusive
Brick	93	0.0	85.7	1.1
Concrete	218	0.0	92.6	0.9
Drywall	113	0.0	na[a]	0.0
Metal	189	0.0	61.4	5.3
Plaster	222	0.0	100.0	0.0
Wood	355	0.0	83.3	2.0
Overall	1,190	0.0	82.3	1.7

[a] Not available since drywall ICP measurements were all less than 1.0 mg/cm^2 lead.

Table 6-296. MAP-3 K-shell by Substrate for the First Standard Paint Reading Red NIST SRM Average Corrected With an Alternative Inconclusive Range Bounded by 0.7 mg/cm^2 and 1.3 mg/cm^2.

Substrate	Sample Size	% False Positive	% False Negative	% Inconclusive
Brick	185	1.4	0.0	3.2
Concrete	436	3.9	18.5	7.1
Drywall	226	1.8	na[a]	2.7
Metal	378	1.7	1.1	11.6
Plaster	444	2.0	19.2	6.1
Wood	698	3.2	5.5	6.6
Overall	2,367	2.6	7.4	6.8

[a] Not available since drywall ICP measurements were all less than 1.0 mg/cm^2 lead.

Table 6-297. MAP-3 L-shell by Substrate for the First Standard Paint Reading Red NIST SRM Average Corrected With an Alternative Inconclusive Range Bounded by 0.7 mg/cm^2 and 1.3 mg/cm^2.

Substrate	Sample Size	% False Positive	% False Negative	% Inconclusive
Brick	185	0.0	28.6	9.7
Concrete	436	0.0	83.3	0.7
Drywall	226	0.0	na[a]	0.4
Metal	378	0.3	53.4	9.0
Plaster	444	0.0	98.1	0.2
Wood	698	0.0	66.3	6.2
Overall	2,367	0.1	66.0	4.2

[a] Not available since drywall ICP measurements were all less than 1.0 mg/cm^2 lead.

Table 6-298. Microlead I by Substrate for the First Standard Paint Reading Red NIST SRM Average Corrected With an Alternative Inconclusive Range Bounded by 0.7 mg/cm^2 and 1.3 mg/cm^2.

Substrate	Sample Size	% False Positive	% False Negative	% Inconclusive
Brick	186	4.2	0.0	14.0
Concrete	444	5.2	10.7	16.0
Drywall	237	1.3	na[a]	5.5
Metal	406	5.7	1.1	9.4
Plaster	463	3.7	5.1	16.0
Wood	739	6.9	6.8	9.1
Overall	2,475	4.9	5.3	11.7

[a] Not available since drywall ICP measurements were all less than 1.0 mg/cm^2 lead.

Table 6-299. X-MET 880 by Substrate for the First Standard Paint Reading Red NIST SRM Average Corrected With an Alternative Inconclusive Range Bounded by 0.7 mg/cm^2 and 1.3 mg/cm^2.

Substrate	Sample Size	% False Positive	% False Negative	% Inconclusive
Brick	93	0.0	85.7	1.1
Concrete	218	0.0	92.6	0.9
Drywall	113	0.0	na[a]	0.0
Metal	175	0.0	67.4	5.7
Plaster	222	0.0	100.0	0.0
Wood	353	0.0	86.0	1.4
Overall	1,174	0.0	84.8	1.5

[a] Not available since drywall ICP measurements were all less than 1.0 mg/cm^2 lead.

Table 6-300. XK-3 by Substrate for the First Standard Paint Reading Red NIST SRM Average Corrected With an Alternative Inconclusive Range Bounded by 0.7 mg/cm^2 and 1.3 mg/cm^2.

Substrate	Sample Size	% False Positive	% False Negative	% Inconclusive
Brick	186	6.3	2.4	8.6
Concrete	444	10.1	3.6	16.9
Drywall	237	0.0	na[a]	4.6
Metal	406	5.1	15.2	11.8
Plaster	462	5.5	6.8	18.0
Wood	743	4.8	4.5	8.2
Overall	2,478	5.5	6.6	11.9

[a] Not available since drywall ICP measurements were all less than 1.0 mg/cm^2 lead.

Table 6-301. XL by Substrate for the First Standard Paint Reading Red NIST SRM Average Corrected With an Alternative Inconclusive Range Bounded by 0.7 mg/cm^2 and 1.3 mg/cm^2.

Substrate	Sample Size	% False Positive	% False Negative	% Inconclusive
Brick	93	0.0	23.8	1.1
Concrete	217	0.0	33.3	6.5
Drywall	113	0.0	na[a]	3.5
Metal	189	0.7	18.2	6.9
Plaster	222	0.0	46.2	4.5
Wood	355	0.0	21.6	13.0
Overall	1,189	0.1	25.5	7.4

[a] Not available since drywall ICP measurements were all less than 1.0 mg/cm^2 lead.

6.5.5 The Effect of Spatial Variation and Laboratory Error in ICP Measurements on XRF Classification Rates

The false positive and false negative rates presented in Tables 6-158 through 6-301 did not account for the fact that ICP measurement was not a perfect substitute for the true lead level. At the beginning of section 6.4, it was explained that the substitution was subject to both spatial variation and laboratory error. Chapter 4 elaborates more fully on both types of imprecision.

A simulation experiment was conducted to assess the effect that the substitution might have had on the reported classification rates. For each of the 48 combinations of XRF instrument type with substrate, random errors were introduced to the ICP measurements obtained in the study. This created sets of "new" ICP measurements, treating the original ICP measurements as if they were true lead levels. A new ICP measurement was generated by adding a normally distributed random error with mean zero and SD = 0.3 to the logarithm of an original ICP measurement, and exponentiating. The choice of 0.3 for the SD reasonably reflects the size of the combined effect of spatial variation and laboratory error in ICP measurements, as demonstrated in section 6.4.8.2.5. A total of 1000 new ICP measurements for each instrument-substrate combination were generated in this manner. False positive and false negative rates for each of these samples were computed, based on the first uncorrected nominal 15-second XRF readings observed in the study. This experiment was similar to one conducted for classifications using lead test kit data, reported in section 5.1.1.

Table 6-302 summarizes the results of the experiment. The sample false positive (FP) and false negative rates (FN), the means of the 1000 simulated values, and ranges consisting of the 2.5^{th} and 97.5^{th} percentiles of the simulated values are presented. It is apparent that the introduction of random errors did not markedly affect the classification rates. False negative rates exhibited greater variability than did false positive rates, which was due at least in part to the fact that the sample sizes for ICP measurements greater than 1.0 mg/cm^2 lead were much smaller than for ICP measurements below this value. In no case would a substantially different conclusion about the classification ability of an XRF instrument be reached based on the simulation results. A similar conclusion may be reached

concerning the use of the FP and FN rates reported in Tables 6-211 through 6-301 as substitutes for rates based on the unobservable true lead levels.

Table 6-302. Simulation Study Percentage Results of the Effect of Spatial Variation and Laboratory Error in ICP Measurements on Reported False Positive and False Negative Rates for XRF Instruments.

XRF INSTRUMENT	SUBSTRATE	FALSE POSITIVE RESULTS			FALSE NEGATIVE RESULTS		
		FP%[a]	MEAN%[b]	95% INTERVAL[c]	FN%[d]	MEAN%[e]	95% INTERVAL[c]
Lead Analyzer K-shell	Brick	3	3	1.4, 4.1	0	1	0.0, 4.8
	Concrete	2	1	0.5, 2.6	11	15	7.7, 21.9
	Drywall	2	2	0.9, 1.8	na[f]	na[f]	na[f]
	Metal	3	4	2.1, 5.4	7	12	6.7, 18.0
	Plaster	1	2	1.0, 3.0	4	6	0.0, 13.8
	Wood	6	8	6.3, 10.3	6	8	4.4, 10.7
Lead Analyzer L-shell	Brick	0	0	0.0, 0.0	91	91	90.0, 91.3
	Concrete	0	0	0.0, 0.0	96	97	96.2, 96.9
	Drywall	0	0	0.0, 0.0	na[f]	na[f]	na[f]
	Metal	0	0	0.0, 0.0	82	83	81.0, 84.3
	Plaster	0	0	0.0, 0.0	100	100	100, 100
	Wood	0	0	0.0, 0.0	87	87	85.9, 87.5
MAP-3 K-shell	Brick	4	4	3.5, 5.5	0	2	0.0, 8.7
	Concrete	6	6	5.0, 6.5	24	27	21.2, 33.3
	Drywall	4	3	2.7, 3.6	na[f]	na[f]	na[f]
	Metal	19	19	17.1, 20.8	1	4	1.1, 8.3
	Plaster	2	3	2.3, 3.8	21	22	15.2, 29.3
	Wood	11	12	10.6, 14.3	6	7	4.7, 9.9
MAP-3 L-shell	Brick	0	0	0.0, 1.4	29	30	28.6, 34.8
	Concrete	0	0	0.0, 0.0	85	86	84.6, 87.5
	Drywall	0	0	0.4, 0.5	na[f]	na[f]	na[f]
	Metal	5	5	3.8, 5.9	60	61	57.3, 64.3
	Plaster	0	0	0.0, 0.0	100	100	100, 100
	Wood	0	1	0.2, 1.2	70	70	67.9, 72.0

[a] Rounded false positive percent reported in Tables 6-162 through 6-165.
[b] Simulation false positive percent.
[c] Simulation 95% coverage interval.
[d] Rounded false negative percent reported in Tables 6-162 through 6-165.
[e] Simulation false negative percent.
[f] Not available since drywall ICP measurements were all less than 1.0 mg/cm^2 lead.

Table 6-302 (cont). Simulation Study Percentage Results of the Effect of Spatial Variation and Laboratory Error in ICP Measurements on Reported False Positive and False Negative Rates for XRF Instruments.

XRF INSTRUMENT	SUBSTRATE	FALSE POSITIVE RESULTS			FALSE NEGATIVE RESULTS		
		FP%[a]	MEAN%[b]	95% INTERVAL[c]	FN%[d]	MEAN%[e]	95% INTERVAL[c]
Microlead I	Brick	22	22	21.1, 22.9	2	4	0.0, 10.4
	Concrete	26	25	24.3, 26.3	2	4	0.0, 8.3
	Drywall	18	18	17.2, 18.0	na[f]	na[f]	na[f]
	Metal	19	19	17.3, 20.3	2	6	2.3, 10.8
	Plaster	10	10	9.5, 11.3	10	11	5.7, 16.9
	Wood	26	28	26.0, 29.1	4	5	2.8, 6.5
X-MET 880	Brick	0	0	0.0, 1.4	86	86	85.7, 90.0
	Concrete	0	0	0.0, 0.0	96	97	96.2, 97.0
	Drywall	0	0	0.0, 0.0	na[f]	na[f]	na[f]
	Metal	0	0	0.0, 1.5	70	72	69.0, 75.0
	Plaster	0	0	0.0, 0.0	100	100	100, 100
	Wood	0	0	0.0, 0.0	89	89	87.8, 89.3
XK-3	Brick	53	53	51.4, 52.9	2	2	0.0, 6.5
	Concrete	66	66	65.3, 66.6	2	2	1.5, 4.8
	Drywall	5	5	3.5, 5.1	na[f]	na[f]	na[f]
	Metal	57	57	55.9, 57.8	5	6	4.3, 8.2
	Plaster	46	46	45.8, 47.1	2	3	0.0, 7.3
	Wood	17	18	16.6, 19.9	4	5	3.3, 7.3
XL	Brick	0	0	0.0, 1.4	24	26	23.8, 30.4
	Concrete	1	1	0.0, 1.6	41	46	40.0, 51.7
	Drywall	1	1	0.9, 0.9	na[f]	na[f]	na[f]
	Metal	1	1	0.0, 2.1	30	34	27.9, 38.3
	Plaster	0	0	0.0, 1.0	58	58	52.2, 63.0
	Wood	1	2	0.4, 2.7	47	47	43.5, 50.5

[a] Rounded false positive percent reported in Tables 6-166 through 6-169.
[b] Simulation false positive percent.
[c] Simulation 95% coverage interval.
[d] Rounded false negative percent reported in Tables 6-166 through 6-169.
[e] Simulation false negative percent.
[f] Not available since drywall ICP measurements were all less than 1.0 mg/cm^2 lead.

6.5.6 Summary of Classification Results

Presented in this section were classification results for the set of sampling locations tested in this study. Another set of locations with significantly different lead levels than the tested locations might provide different results, even if the same instruments were used. The classification results provided empirical evidence that classifying the K-shell XRF instrument results against the federal standard of 1.0 mg/cm^2 lead without an inconclusive range, and with substrate correction if needed, produced low classification error rates, no greater than 11% overall, that is, averaged for all substrates. However, error rates on particular substrates could be substantially higher than the overall rates. These results provided further evidence of differences between the K- and L-shell instruments. All L-shell instruments had high false negative rates and low false positive rates when classifying against the 1.0 mg/cm^2 lead standard without an inconclusive range. The overall false negative rate for the XL was 41.8%, and the other L-shell instruments had higher false negative rates. The XL had a low false positive rate of 0.5%, which was typical for an L-shell instrument. Substrate correction did not significantly improve these results for the L-shell instruments.

The Lead Analyzer and MAP-3 had overall misclassification rates less than 10% compared to the 1.0 mg/cm^2 lead federal standard without substrate correction. For some K-shell instruments, error rates were reduced when readings were corrected for substrate bias. For the Microlead I and XK-3, overall classification error rates were reduced to 10% or less by full and red NIST SRM average correction methods. Control correction produced mixed results: for the XK-3, overall error rates were about 11% or less, but, the false positive rate on concrete was greater than 24% and the false negative rate on metal was greater than 20%. Control correction did not improve the error rates for the Microlead I. The MAP-3 K-shell had its high false positive rate on metal and high false negative rates on concrete and plaster reduced by control correction.

The classification results showed that using an inconclusive range provided satisfactory results for K-shell instruments, but, false negative rates remained high for the L-shell instruments. Results showed that the K-shell XRF instruments provided satisfactory classification results using an inconclusive range

between 0.4 and 1.6 mg/cm^2 and correcting for substrate biases in XRF readings if needed. With the exception of the XK-3 false positive rates, all error rates for the K-shell XRF instruments were below 10%. On individual substrates, most error rates were still below 10%. The exceptions were: false negative rates for the MAP-3 on concrete and plaster; the Microlead I false positive rate on wood; and, false positive rates for the XK-3 on brick, concrete, metal, and plaster. These false positive rates for the XK-3 were dramatically reduced by substrate correction.

When the inconclusive range was narrowed to 0.7 to 1.3 mg/cm^2, percentages in the inconclusive range were reduced by at least 50% for all instruments compared to the 0.4 - 1.6 mg/cm^2 inconclusive range. Observed changes in the error rates coupled with this dramatic decrease in percentages in the inconclusive range indicates that a balance needs to be struck between error rates and the inconclusive range, which determines the number of paint-chip samples requiring laboratory confirmation.

The K-shell instruments continued to provide error rates near ten percent using the 0.7 - 1.3 mg/cm^2 range. The Microlead I and the XK-3 both needed substrate correction to achieve false positive rates near ten percent. On individual substrates, error rates were generally below 10%. The exceptions were the false negative rate for the MAP-3 on concrete; and the false negative rate for the XK-3 on metal. The results for the Microlead I were substantially improved by full correction.

With the exception of the XL, classifying L-shell instrument results using either inconclusive range provided very high false negative rates, reflecting the large negative biases exhibited by these instruments. False positive rates were very low for all L-shell instruments. Using the 0.4 to 1.6 mg/cm^2 inconclusive range, the XL had a false negative rate of approximately 11% and a false positive rate of 0.1%. However, the instrument still provided readings below 0.4 mg/cm^2 on a number of samples with lead levels in excess of 10.0 mg/cm^2, which were classified as false negative. With the narrower inconclusive range of 0.7 to 1.3 mg/cm^2, the XL had an overall false negative rate of 28.6% and a 0.2% false positive rate.

Classification results in this section show that a single XRF reading at a point provided almost as much information as an average of three XRF readings at the same point. When paint

samples were classified with or without the use of an inconclusive range, there was very little difference in the error rates (false positive and false negative) for the average of three 15-second readings versus a single 15-second reading. For example, when classifying results using the 0.4 to 1.6 mg/cm^2 inconclusive range, the overall error percentages for the K-shell XRF instruments were the same in two cases and slightly lower for the average in six cases. For L-shell instruments, the error percentages were the same in six cases and slightly lower for the average in two cases. Thus, the small improvement in classification accuracy did not justify the additional time and expense of taking three repeat readings at the same point. This remained true when substrate corrected readings and different inconclusive ranges were considered.

6.6 EFFECTS RELATED TO CHANGING FROM ONE SUBSTRATE TO ANOTHER 6-460

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Table 6-304. Counts of the Number of Dwellings an XRF Instrument Tested on **CONCRETE**, the Number of Dwelling Maximum and Minimum Values, and the Total Number of Extreme Values that Occurred for the First Paint Reading Minus Laboratory ICP Differences 6-462
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6.6 EFFECTS RELATED TO CHANGING FROM ONE SUBSTRATE TO ANOTHER

It has been hypothesized that an XRF device may operate erratically when a change has been made from one substrate to another. To study the effect of changing from one substrate to another, the laboratory ICP results measured in mg/cm^2 were paired with the standard first paint readings made on each sample location over the painted surface. This analysis addresses the following study objectives:

- to characterize the performance (precision and accuracy) or portable XRF instruments under field conditions
- to evaluate the effect on XRF performance of interference from material (the substrate) underlying the paint
- to investigate XRF measurements that were very different than their corresponding laboratory results.

Differences between the XRF reading on paint and the laboratory ICP result (measured in mg/cm^2) were computed for each sample location. These differences were used to examine the hypothesis that the XRF instruments behaved erratically after changing from one substrate to another. A description of the analysis and results follows.

If the XRF instruments behaved erratically when changing substrate, one would expect differences (that is, XRF reading minus ICP result differences) computed from the first sampling location on the new substrate to differ systematically from the subsequent differences on the same substrate. Erratic behavior would be detected if the observed incidence of extreme differences (maxima or minima) on the first reading of a new substrate were higher than the expected incidence of extremes. Section 6.1 and Tables 6-5 and 6-6 describe the substrates for each dwelling and the order in which testing on substrates was done. In Tables 6-5 and 6-6, the "Number of Substrate Changes" values are the number of opportunities from which extremes per substrate per dwelling were tabulated.

Tables 6-303 through 6-308 provide a count of the number of maxima and minima per dwelling that occurred on the first standard paint reading on a substrate. Also found in the tables are the number of days an XRF instrument tested in Denver, Philadelphia, and Louisville, and the total number of daily

extreme values that occurred for the first regular paint reading minus laboratory ICP differences. Standard data as defined in section 6.1 were used for this analysis.

Table 6-303. Counts of the Number of Dwellings an XRF Instrument Tested on **BRICK**, the Number of Dwelling Maximum and Minimum Values, and the Total Number of Extreme Values that Occurred for the First Paint Reading Minus Laboratory ICP Differences.

XRF INSTRUMENT	FIELD CODE NO.	THREE CITIES?	NO. OF DWELLINGS	NO. OF MINIMUM VALUES	NO. OF MAXIMUM VALUES	TOTAL NO. EXTREME
Lead Analyzer K-shell	na	no	11	1	0	1
Lead Analyzer L-shell	na	no	11	0	1	1
MAP-3 K-shell	I	no	11	0	1	1
MAP-3 K-shell	II	no	11	1	0	1
MAP-3 L-shell	I	no	11	1	0	1
MAP-3 L-shell	II	no	11	0	0	0
Microlead I K-shell	I	yes	11	0	0	0
Microlead I K-shell	II	no	11	1	0	1
X-MET 880 L-shell	na	no	11	0	0	0
XK-3 K-shell	I	yes	11	2	0	2
XK-3 K-shell	II	no	11	0	0	0
XL L-shell	na	no	11	0	0	0

The total number of daily extreme values observed for each instrument and substrate in these tables was evaluated using a statistical model. The field classification of the XRF instruments was used to insure independence of readings between sampling locations. Assume that the number of testing locations for a substrate in a housing unit is N_j. If extreme values are equally likely to occur at every location, the probability that an extreme (maximum or minimum) occurs on the first reading is $2/N_j$. The expected number of extreme values, M, for an instrument on a substrate is then found by summing the quantities $2/N_j$ over all units tested by the instrument. Further, under the

independence assumption, the variance of the number of extremes is found by summing the quantity $(2/N_j)*(1 - 2/N_j)$ over all units tested. The square root of this quantity, S, is the standard

Table 6-304. Counts of the Number of Dwellings an XRF Instrument Tested on **CONCRETE**, the Number of Dwelling Maximum and Minimum Values, and the Total Number of Extreme Values that Occurred for the First Paint Reading Minus Laboratory ICP Differences.

XRF INSTRUMENT	FIELD CODE NO.	THREE CITIES?	NO. OF DWELLINGS	NO. OF MINIMUM VALUES	NO. OF MAXIMUM VALUES	TOTAL NO. EXTREME
Lead Analyzer K-shell	na	no	17	1	1	2
Lead Analyzer L-shell	na	no	17	2	3	5
MAP-3 K-shell	I	no	17	2	1	3
MAP-3 K-shell	II	no	17	2	2	4
MAP-3 L-shell	I	no	17	2	2	4
MAP-3 L-shell	II	no	17	2	2	4
Microlead I K-shell	I	yes	19	0	2	2
Microlead I K-shell	II	no	17	2	1	3
X-MET 880 L-shell	na	no	17	2	4	6
XK-3 K-shell	I	yes	19	2	2	4
XK-3 K-shell	II	no	17	1	1	2
XL L-shell	na	no	17	1	1	2

deviation of the number of extremes.

Approximate statistical tests of significance for the observed number of extremes can be constructed using the quantities M and S. For example, under the asymptotic normality assumption, the 95th percentile of the number of extremes is given by M + 1.645*S. If the observed number of extremes exceeds this, then there is significant evidence, at the 0.05 level, that the number of extremes for the first reading is elevated above what would be expected by chance. There are 72 combinations of

instrument and substrate represented in Tables 6-303 through 6-308. Thus, to achieve an overall significance level of 0.05, a significance level of 0.05 ÷ 72 = 0.0007 should be used for each

Table 6-305. Counts of the Number of Dwellings an XRF Instrument Tested on **DRYWALL**, the Number of Dwelling Maximum and Minimum Values, and the Total Number of Extreme Values that Occurred for the First Paint Reading Minus Laboratory ICP Differences.

XRF INSTRUMENT	FIELD CODE NO.	THREE CITIES?	NO. OF DWELLINGS	NO. OF MINIMUM VALUES	NO. OF MAXIMUM VALUES	TOTAL NO. EXTREME
Lead Analyzer K-shell	na	no	11	0	1	1
Lead Analyzer L-shell	na	no	11	0	0	0
MAP-3 K-shell	I	no	11	1	0	1
MAP-3 K-shell	II	no	11	0	1	1
MAP-3 L-shell	I	no	11	0	2	2
MAP-3 L-shell	II	no	11	0	1	1
Microlead I K-shell	I	yes	13	2	1	3
Microlead I K-shell	II	no	11	0	1	1
X-MET 880 L-shell	na	no	11	0	2	2
XK-3 K-shell	I	yes	13	1	2	3
XK-3 K-shell	II	no	11	0	1	1
XL L-shell	na	no	11	0	1	1

individual instrument and substrate combination. The calculated limit for the number of extremes is then M + 3.2*S. Table 6-309 shows the critical limits for obtaining an overall significance level of 0.05 for two cases. The first is for XRF instruments which tested in all three cities; the second is for Denver and Philadelphia combined, that is, excluding Louisville. Note that in the tables, the XRF instruments are classified according to the field classification. This classification was necessary in order to maintain the assumption that extreme values are equally likely to occur on any reading within a group of readings. If we

had used the classifications of eight XRF instruments defined previously in this report, this assumption would have been violated.

Table 6-306. Counts of the Number of Dwellings an XRF Instrument Tested on **METAL**, the Number of Dwelling Maximum and Minimum Values, and the Total Number of Extreme Values that Occurred for the First Paint Reading Minus Laboratory ICP Differences.

XRF INSTRUMENT	FIELD CODE NO.	THREE CITIES?	NO. OF DWELLINGS	NO. OF MINIMUM VALUES	NO. OF MAXIMUM VALUES	TOTAL NO. EXTREME
Lead Analyzer K-shell	na	no	18	2	2	4
Lead Analyzer L-shell	na	no	18	4	2	6
MAP-3 K-shell	I	no	18	3	3	6
MAP-3 K-shell	II	no	18	3	4	7
MAP-3 L-shell	I	no	18	4	3	7
MAP-3 L-shell	II	no	18	4	2	6
Microlead I K-shell	I	yes	20	3	3	6
Microlead I K-shell	II	no	18	2	2	4
X-MET 880 L-shell	na	no	18	3	1	4
XK-3 K-shell	I	yes	20	3	2	5
XK-3 K-shell	II	no	18	3	4	7
XL L-shell	na	no	18	2	3	5

The total number of extreme values from all three cities for the Microlead I revision 4 and XK-3 instruments was compared to the three city critical limits provided in Table 6-309. As described in section 6.1, the Microlead I and XK-3 were used in all three cities. These instruments were given the field designation "I" and are so designated in the tables in this section. Extreme values for all other XRF instruments were compared to the two city (Denver and Philadelphia) critical limits provided in Table 6-309. Comparisons of Tables 6-303 through 6-308 to Table 6-309 shows that none of the total number of extreme values exceeded the critical limits. Hence, since

there is no statistically significant evidence, at the overall 0.05 level, the incidence of extreme values recorded on the first reading of a substrate is not unusually high. Thus, there is no

Table 6-307. Counts of the Number of Dwellings an XRF Instrument Tested on **PLASTER**, the Number of Dwelling Maximum and Minimum Values, and the Total Number of Extreme Values that Occurred for the First Paint Reading Minus Laboratory ICP Differences.

XRF INSTRUMENT	FIELD CODE NO.	THREE CITIES?	NO. OF DWELLINGS	NO. OF MINIMUM VALUES	NO. OF MAXIMUM VALUES	TOTAL NO. EXTREME
Lead Analyzer K-shell	na	no	14	2	0	2
Lead Analyzer L-shell	na	no	14	3	1	4
MAP-3 K-shell	I	no	14	2	1	3
MAP-3 K-shell	II	no	14	1	1	2
MAP-3 L-shell	I	no	14	1	0	1
MAP-3 L-shell	II	no	14	2	0	2
Microlead I K-shell	I	yes	16	1	1	2
Microlead I K-shell	II	no	14	2	0	2
X-MET 880 L-shell	na	no	14	3	0	3
XK-3 K-shell	I	yes	16	2	1	3
XK-3 K-shell	II	no	14	1	1	2
XL L-shell	na	no	14	1	3	4

statistically significant evidence of erratic behavior of the first reading on a substrate.

However, observations of Tables 6-303 through 6-308 indicate that a higher incidence of extremes occurred more often on metal and wood substrates. Although this suggests the possibility of increased erratic behavior on the first reading for metal and wood, the evidence is weak. First, as previously explained, the results are not significant using an overall significance level of 0.05. Second, the statistical model is approximate, in that

it assumes that extremes are equally likely to occur on any reading. The testing order could invalidate this assumption in some cases. For example, if the higher lead levels were tested

Table 6-308. Counts of the Number of Dwellings an XRF Instrument Tested on **WOOD**, the Number of Dwelling Maximum and Minimum Values, and the Total Number of Extreme Values that Occurred for the First Paint Reading Minus Laboratory ICP Differences.

XRF INSTRUMENT	FIELD CODE NO.	THREE CITIES?	NO. OF DWELLINGS	NO. OF MINIMUM VALUES	NO. OF MAXIMUM VALUES	TOTAL NO. EXTREME
Lead Analyzer K-shell	na	no	17	3	4	7
Lead Analyzer L-shell	na	no	17	4	1	5
MAP-3 K-shell	I	no	17	0	4	4
MAP-3 K-shell	II	no	17	2	4	6
MAP-3 L-shell	I	no	17	2	1	3
MAP-3 L-shell	II	no	17	2	0	2
Microlead I K-shell	I	yes	19	2	5	7
Microlead I K-shell	II	no	17	0	5	5
X-MET 880 L-shell	na	no	17	3	1	4
XK-3 K-shell	I	yes	19	0	4	4
XK-3 K-shell	II	no	17	1	5	6
XL L-shell	na	no	17	3	1	4

Table 6-309. Critical Values for the Observed Number of Extreme Absolute XRF minus ICP Differences for XRF Readings Taken at the First Sampling Location Tested for a Given Substrate.

OVERALL 0.05 SIGNIFICANCE LEVEL	SUBSTRATE					
	Brick	Concrete	Drywall	Metal	Plaster	Wood
Three Cities	3.5	8.3	5.9	9.3	6.7	8.8
Denver & Philadelphia	3.5	6.7	4.4	8.7	5.9	8.3

earlier, then extreme differences between XRF and ICP would be more likely early in the testing.

6.7 DESCRIPTIVE STATISTICS FOR "SPECIAL" AND NON-STANDARD DATA 6-466
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Table 6-310. Summary Statistics of Lead Measured in mg/cm^2 Units of the First Bare Substrate Reading ("Special" Data) For All XRF Instrument Types Except the MAP-3 6-469
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6.7 DESCRIPTIVE STATISTICS FOR "SPECIAL" AND NON-STANDARD DATA

The first section of this chapter described XRF data and categorized the data as standard, control, special, and non-standard data. This section provides summary statistics for "special" and non-standard data that include the number of readings, mean, median, maximum, minimum, 25th percentile, and 75th percentile and addresses the following study objectives:

- to characterize the performance (precision and accuracy) or portable XRF instruments under field conditions
- to evaluate the effect on XRF performance of interference from material (the substrate) underlying the paint
- to investigation XRF measurements that were very different than their corresponding laboratory results
- to evaluate field quality assurance and control methods.

Due to the large number of tables presented in this section, the organization of this section is a departure from normal. For this section only, most tables are not intermingled with text, but instead, all tables referenced in a given subsection that provide summary statistics were placed after the text for that subsection.

For this analysis, eight distinct XRF classifications were analyzed as if they were a separate XRF instrument:

- Lead Analyzer K-shell
- Lead Analyzer L-shell
- MAP-3 K-shell
- MAP-3 L-shell
- Microlead I (K-shell)
- X-MET 880 (L-shell)
- XK-3 (K-shell)
- XL (L-Shell)

6.7.1 Summary Statistics for "Special" Data

This section provides summary statistics for the "special" data for the eight classifications of XRF instruments. For an in-depth discussion of "special" data refer to section 6.1. There are two types of "special" data: "special" readings and "special-special" readings. "Special-special" locations were

used in Denver and Philadelphia by the MAP-3 instruments only. The data collection protocol at "special" locations depended upon the XRF instrument type and whether data were being collected for the pilot study or the full study and affected the number of readings and nominal reading times. These differences provide a method for examining instrument readings relative to the number of readings and nominal reading times. The information to make these comparisons is provided in the tables below.

The next seven tables contain summary statistics for "special" data in the full study and standard data collected at "special" locations in the full study. Tables 6-310 through 6-313 provide summary statistics for the "special" readings taken in Denver and Philadelphia. Tables 6-310, 6-311, and 6-612 provide results for all instruments except the MAP-3. The "special" data results for the MAP-3 are provided separately from the other XRF results in Table 6-313 since the "special" data collection protocol used by the MAP-3 was unique. Tables 6-314, 6-315, and 6-316 are for making comparisons to the tables of "special" data and show results of standard data that was collected only at "special" locations. Table 6-314 provides summary statistics for the MAP-3 of standard data collected at "special" and "special-special" locations and will be useful for making comparisons to Table 6-313. Table 6-315 provides summary statistics for the first red NIST SRM reading taken from "special" locations in the full study minus 1.02 mg/cm^2. Table 6-316 provides summary statistics for the first standard paint fully corrected reading at "special" locations in the full study. Tables 6-315 and 6-316 will be useful for making comparisons to Tables 6-310, 6-311, and 6-312.

Comparisons of Table 6-313 to 6-314 show that the extended nominal reading times did not greatly affect the result as shown by comparing the "PAINT" or "NIST" between the two tables or by comparing the "Bare" results in Table 6-313 to "Paint-ICP" or "NIST-1.02" results in Table 6-314. Further evidence is given by the results of paired Student's *t* tests which were performed to determine if the longer nominal reading times significantly affected the outcome. The "special" data were paired with nominal 15-second readings and also nominal 60-second readings were paired with nominal 240-second readings. Twenty-four *t* statistics were computed for each instrument by shell classification for all possible pairs. Using an overall significance level of 0.002 (0.05 ÷ 24 = 0.002), no statistically

significant results were found for readings made on paint or for readings made on paint compared to readings made on the bare substrate. Similar results were found comparing the readings made on the bare substrate to those made on the bare substrates covered with the red (1.02 mg/cm^2) NIST SRM minus 1.02 mg/cm^2 and for comparing the "special" readings taken on the bare substrates covered with the red (1.02 mg/cm^2) NIST SRM to the (standard) readings taken on the bare substrates covered with the red (1.02 mg/cm^2) NIST SRM at "special" locations. Therefore, the longer 60-second and 240-second nominal reading times made by the MAP-3 at "special" locations appeared to have little effect on the outcome compared to the nominal 15-second reading.

The bare substrate "special" readings in Tables 6-310 to 6-312 were consistent with the bare results given in Table 6-315. However, results in Tables 6-310 to 6-312 were not as consistent as the results shown in Table 6-316, which are the results for the first standard paint readings minus the appropriate ICP measurement in mg/cm^2 taken from "special" locations. This could be due in part to the lead levels found in the paint.

The next five tables provide summary statistics for "special" data collected in Louisville. Tables 6-317 through 6-321 provide summary statistics for the "special" readings taken in Louisville by the MAP-3 K-shell, MAP-3 L-shell, Microlead I, X-MET 880, and XK-3, respectively. The results given in these tables are very similar to readings taken at "special" locations using a different data collection protocol. That is, the non-special means were not significantly different from the "special" means computed for readings taken at the same locations. This was observed in Tables 6-322 and 6-323. These tables provide results for the (pilot) standard data for the first paint reading and the first red NIST SRM reading for all instruments.

Table 6-310. Summary Statistics of Lead Measured in mg/cm^2 Units of the First Bare Substrate Reading ("Special" Data) For All XRF Instrument Types Except the MAP-3.

XRF TYPE	NUMBER OF READINGS	ARITHMETIC MEAN	MAXIMUM	MINIMUM	25th PERCENTILE	MEDIAN	75TH PERCENTILE
Lead Analyzer K-shell	299	0.073	1.700	-0.300	-0.020	0.030	0.100
Lead Analyzer L-shell	299	0.013	0.970	-0.051	-0.008	0.000	0.016
Microlead I	601[a]	0.395	4.500	-1.600	-0.200	0.200	0.700
X-MET 880	255[b]	0.048	1.444	0.022	0.028	0.033	0.039
XK-3	596	0.636	4.000	-1.000	0.200	0.500	1.000
XL	301	0.101	1.200	0.000	0.000	0.000	0.100

[a] One Microlead I reading was omitted from this analysis due to known instrument problems and two additional readings were made.
[b] Forty seven sampling locations composed of metal substrates are missing.

Table 6-311. Summary Statistics of Lead Measured in mg/cm^2 Units of the Second Bare Substrate Reading ("Special" Data) For All XRF Instrument Types Except the MAP-3.

XRF TYPE	NUMBER OF READINGS	ARITHMETIC MEAN	MAXIMUM	MINIMUM	25th PERCENTILE	MEDIAN	75TH PERCENTILE
Lead Analyzer K-shell	299	0.066	2.100	-0.300	-0.030	0.020	0.090
Lead Analyzer L-shell	299	0.013	0.970	-0.053	-0.009	0.000	0.016
Microlead I	600[a]	0.413	4.600	-2.300	-0.100	0.200	0.700
X-MET 880	255[b]	0.047	1.355	0.022	0.028	0.033	0.039
XK-3	596	0.653	3.400	-1.200	0.100	0.500	1.100
XL	301	0.108	1.600	0.000	0.000	0.000	0.100

[a] One Microlead I reading was omitted from this analysis due to known instrument problems and one additional reading was made.
[b] Forty seven sampling locations composed of metal substrates are missing.

Table 6-312. Summary Statistics of Lead Measured in mg/cm^2 Units of the Third Bare Substrate Reading ("Special" Data) For All XRF Instrument Types Except the MAP-3.

XRF TYPE	NUMBER OF READINGS	ARITHMETIC MEAN	MAXIMUM	MINIMUM	25th PERCENTILE	MEDIAN	75TH PERCENTILE
Lead Analyzer K-shell	299	0.067	2.500	-0.300	-0.030	0.020	0.090
Lead Analyzer L-shell	299	0.013	0.970	-0.055	-0.008	0.000	0.016
Microlead I	599[a]	0.402	4.600	-2.800	-0.300	0.200	0.900
X-MET 880	255[b]	0.046	1.092	0.022	0.028	0.033	0.039
XK-3	596	0.628	3.600	-1.000	0.100	0.500	1.000
XL	301	0.113	1.300	0.000	0.000	0.100	0.100

[a] One Microlead I reading was omitted from this analysis due to known instrument problems.
[b] Forty seven sampling locations composed of metal substrates are missing.

Table 6-313. Summary Statistics of Lead Measured in mg/cm^2 Units of the "Special" Readings for the MAP-3 for the Full Study on the Painted Surface, the Bare Substrates Covered With the Red (1.02 mg/cm^2) NIST SRM Film, and the Bare Substrates.

SHELL	TYPE OF SPECIAL[a]	READING SURFACE	NUMBER OF READINGS	ARITHMETIC MEAN	MAXIMUM	MINIMUM	25th PERCENTILE	MEDIAN	75TH PERCENTILE
K-shell	Special (60-sec.)	Paint	601[b]	0.843	23.760	-2.929	-0.486	0.008	0.840
		NIST	601[b]	1.128	4.041	-0.381	0.849	1.112	1.347
		Bare	600	-0.448	2.950	-2.834	-1.129	-0.295	0.187
	"Special-special" (240-sec.)	Paint	162	0.691	14.222	-2.601	-0.640	-0.042	0.804
		NIST	162	1.151	2.911	0.193	0.910	1.095	1.312
		Bare	0	na	na	na	na	na	na
L-shell	Special (60-sec.)	Paint	601[b]	0.139	5.098	-1.129	-0.135	-0.060	0.132
		NIST	601[b]	1.207	1.776	-0.401	1.149	1.207	1.271
		Bare	600	-0.031	2.414	-1.234	-0.175	-0.138	-0.085
	"Special-special" (240-sec.)	Paint	162	0.081	2.445	-0.265	-0.138	-0.080	0.089
		NIST	162	1.201	1.691	0.391	1.149	1.205	1.263
		Bare	0	na	na	na	na	na	na

[a] Nominal reading times are shown in parenthesis.
[b] One additional reading was taken at a "special" location.

Table 6-314. Summary Statistics of Lead Measured in mg/cm² Units For Standard Readings for the MAP-3 at the "Special" and "Special-Special" Full Study Locations Taken on the Painted Surface, the Bare Substrates Covered With the Red (1.02 mg/cm²) NIST SRM Film, the Painted Surface Minus Its Corresponding Laboratory Result in mg/cm² From Each Sampling Location, and the Red NIST SRM Film Minus 1.02 mg/cm² For Nominal 15-Second Readings.

SHELL	TYPE OF LOCATION	READING	NUMBER OF READINGS	ARITHMETIC MEAN	MAXIMUM	MINIMUM	25th PERCENTILE	MEDIAN	75TH PERCENTILE
K	Special	Paint	600	0.8997	25.369	-4.439	-0.385	0.107	0.888
		NIST	600	1.223	3.983	-0.855	0.858	1.160	1.559
		Paint-ICP	600	-0.214	12.371	-13.231	-0.702	-0.163	0.338
		NIST-1.02	600	0.203	2.963	-1.875	-0.163	0.140	0.539
	"Special -special"	Paint	162	0.660	12.884	-4.439	-0.612	0.019	0.830
		NIST	162	1.283	3.695	-0.292	0.989	1.299	1.614
		Paint-ICP	162	-0.232	2.641	-4.453	-0.816	-0.166	0.401
		NIST-1.02	162	0.263	2.675	-1.312	-0.031	0.279	0.594
L	Special	Paint	600	0.138	5.129	-1.275	-0.127	-0.050	-0.150
		NIST	600	1.189	1.694	-0.983	1.126	1.207	1.300
		Paint-ICP	600	-0.973	1.523	-26.257	-0.527	-0.232	-0.130
		NIST-1.02	600	0.169	0.674	-2.003	0.106	0.187	0.279
	"Special -special"	Paint	162	0.076	2.534	-1.109	-0.132	-0.058	0.107
		NIST	162	1.189	1.694	-0.404	1.113	1.201	1.299
		Paint-ICP	162	-0.816	0.746	-13.870	-0.492	-0.226	-0.130
		NIST-1.02	162	0.169	0.674	-1.424	0.093	0.181	0.279

Table 6-315. Summary Statistics of Lead Measured in mg/cm^2 Units for First Standard Red NIST SRM Reading Minus 1.02 mg/cm^2 for All XRF Instrument Types Except the MAP-3 at Full Study "Special" and "Special-special" Locations Only.

XRF TYPE	NUMBER OF READINGS	ARITHMETIC MEAN	MAXIMUM	MINIMUM	25th PERCENTILE	MEDIAN	75TH PERCENTILE
Lead Analyzer K-shell	302	0.077	2.280	-1.020	-0.120	0.080	0.180
Lead Analyzer L-shell	302	-0.026	0.270	-1.010	-0.040	-0.005	0.030
Microlead I	601	0.427	26.480	-1.620	-0.220	0.280	0.680
X-MET 880	301	0.076	0.460	-0.073	0.029	0.071	0.109
XK-3	602	0.699	3.280	-1.020	0.180	0.580	1.080
XL	301	-0.001	0.780	-0.420	-0.120	-0.020	0.080

Table 6-316. Summary Statistics of Lead Measured in mg/cm² Units for First Standard Paint Reading Minus the Laboratory Result in mg/cm² For All XRF Instrument Types Except the MAP-3 at Full Study "Special" and "Special-Special" Locations Only.

XRF TYPE	NUMBER OF READINGS	ARITHMETIC MEAN	MAXIMUM	MINIMUM	25th PERCENTILE	MEDIAN	75TH PERCENTILE
Lead Analyzer K-shell	302	-0.127	5.002	-15.783	-0.093	-0.003	0.089
Lead Analyzer L-shell	302	-0.999	0.176	-28.353	-0.470	-0.142	-0.019
Microlead I	601	0.206	7.858	-12.383	-0.218	0.174	0.727
X-MET 880	295	-0.966	0.579	-27.577	-0.403	-0.101	0.024
XK-3	602	0.512	7.928	-20.583	0.098	0.598	1.258
XL	301	-0.711	2.040	-25.583	-0.271	-0.038	0.019

Table 6-317. Summary Statistics of Lead Measured in mg/cm^2 Units of the Paint and Red (1.02 mg/cm^2) NIST SRM Readings ("Special" Data) For MAP-3 K-shell in Louisville Only.

READING	NUMBER OF READINGS	ARITHMETIC MEAN	MAXIMUM	MINIMUM	25th PERCENTILE	MEDIAN	75TH PERCENTILE
First Paint	26	1.865	5.873	0.000	0.441	1.468	2.935
Second Paint	26	2.096	8.515	0.000	0.308	1.357	3.407
Third Paint	26	1.713	6.455	0.000	0.000	1.009	2.704
First Red NIST SRM	26	1.504	3.210	0.434	0.904	1.481	1.946
Second Red NIST SRM	26	1.592	2.777	0.000	1.349	1.680	1.982
Third Red NIST SRM	26	1.661	5.784	0.258	1.065	1.449	1.954

Table 6-318. Summary Statistics of Lead Measured in mg/cm^2 Units of the Paint and Red (1.02 mg/cm^2) NIST SRM Readings ("Special" Data) For MAP-3 L-shell in Louisville Only.

READING	NUMBER OF READINGS	ARITHMETIC MEAN	MAXIMUM	MINIMUM	25th PERCENTILE	MEDIAN	75TH PERCENTILE
First Paint	26	0.384	2.977	0.000	0.000	0.000	0.189
Second Paint	26	0.417	3.210	0.000	0.000	0.000	0.286
Third Paint	26	0.409	3.072	0.000	0.000	0.000	0.225
First Red NIST SRM	26	1.366	2.026	0.892	1.156	1.380	1.522
Second Red NIST SRM	26	1.360	2.012	0.804	1.165	1.354	1.608
Third Red NIST SRM	26	1.296	1.870	0.576	1.108	1.328	1.461

Table 6-319. Summary Statistics of Lead Measured in mg/cm^2 Units of the Paint and Red (1.02 mg/cm^2) NIST SRM Readings ("Special" Data) For Microlead I in Louisville Only.

READING	NUMBER OF READINGS	ARITHMETIC MEAN	MAXIMUM	MINIMUM	25th PERCENTILE	MEDIAN	75TH PERCENTILE
First Paint	26	1.608	5.600	-0.600	0.000	0.900	3.500
Second Paint	26	1.654	6.000	-0.600	0.100	1.050	3.600
Third Paint	26	1.654	5.500	-0.600	0.000	1.000	3.600
Fourth Paint	26	1.700	5.700	-1.000	0.000	1.100	3.900
First Red NIST SRM	26	1.381	3.100	0.100	0.700	1.400	1.700
Second Red NIST SRM	26	1.335	3.100	0.300	0.800	1.200	1.800
Third Red NIST SRM	26	1.438	3.100	0.400	0.900	1.300	2.000
Fourth Red NIST SRM	26	1.554	3.500	0.700	1.000	1.450	1.900

Table 6-320. Summary Statistics of Lead Measured in mg/cm^2 Units of the Paint and Red (1.02 mg/cm^2) NIST SRM Readings ("Special" Data) For X-MET 880 in Louisville Only.

READING	NUMBER OF READINGS	ARITHMETIC MEAN	MAXIMUM	MINIMUM	25th PERCENTILE	MEDIAN	75TH PERCENTILE
First Paint	26	0.995	4.973	0.000	0.143	0.337	1.286
Second Paint	0	na	na	na	na	na	na
Third Paint	0	na	na	na	na	na	na
Fourth Paint	0	na	na	na	na	na	na
First Red NIST SRM	26	1.143	2.478	0.130	1.084	1.124	1.171
Second Red NIST SRM	0	na	na	na	na	na	na

Third Red NIST SRM	0	na	na	na	na	na	na
Fourth Red NIST SRM	0	na	na	na	na	na	na

Table 6-321. Summary Statistics of Lead Measured in mg/cm^2 Units of the Paint and Red (1.02 mg/cm^2) NIST SRM Readings ("Special" Data) For XK-3 in Louisville Only.

READING	NUMBER OF READINGS	ARITHMETIC MEAN	MAXIMUM	MINIMUM	25th PERCENTILE	MEDIAN	75TH PERCENTILE
First Paint	26	1.738	6.000	-0.300	0.200	0.850	3.000
Second Paint	26	1.754	6.000	-0.600	0.300	1.000	2.500
Third Paint	26	1.677	5.800	-0.600	0.300	0.950	2.400
Fourth Paint	26	1.573	5.600	-0.500	0.100	0.750	2.900
First Red NIST SRM	26	1.427	3.700	0.400	0.900	1.300	1.800
Second Red NIST SRM	26	1.438	3.700	0.400	1.000	1.350	1.900
Third Red NIST SRM	26	1.346	3.100	0.400	0.800	1.350	1.700
Fourth Red NIST SRM	26	1.435	3.200	0.400	0.900	1.400	1.700

Table 6-322. Summary Statistics of Lead Measured in mg/cm² Units For First Paint Reading (Standard) data for All XRF Instrument Types at Pilot Study "Special" Locations Only.

XRF TYPE	NUMBER OF READINGS	ARITHMETIC MEAN	MAXIMUM	MINIMUM	25th PERCENTILE	MEDIAN	75TH PERCENTILE
MAP-3 K-shell	26	1.842	6.571	0.000	0.015	1.146	3.629
MAP-3 L-shell	26	0.411	3.109	0.000	0.000	0.000	0.303
Microlead I	26	1.619	5.500	-0.400	0.300	0.850	3.400
X-MET 880	26	0.968	4.947	0.000	0.113	0.280	1.248
XK-3	26	1.692	6.500	-0.200	0.200	0.950	2.500

Table 6-323. Summary Statistics of Lead Measured in mg/cm² Units For First Red NIST SRM Reading (Standard) data for All XRF Instrument Types at Pilot Study "Special" Locations Only.

XRF TYPE	NUMBER OF READINGS	ARITHMETIC MEAN	MAXIMUM	MINIMUM	25th PERCENTILE	MEDIAN	75TH PERCENTILE
MAP-3 K-shell	26	1.452	3.001	0.824	1.184	1.250	1.647
MAP-3 L-shell	26	1.298	1.910	0.000	1.109	1.345	1.485
Microlead I	26	1.438	3.500	0.100	1.000	1.400	1.800
X-MET 880	26	1.130	2.548	0.879	0.997	1.072	1.127
XK-3	26	1.458	3.800	0.400	1.100	1.400	1.600

6.7.2 Summary Statistics for Non-Standard Data

This section provides summary statistics for the non-standard data that were collected by the eight classifications of XRF instruments. For an in depth discussion of non-standard data refer to section 6.1. Non-standard data are XRF readings that were taken for the pilot study that are not directly comparable with data collected in the full study and are composed of too few data to make parameter estimations using model based procedures. However, comparisons based on summary statistics can be made. Non-standard data are:

- XRF readings made by the MAP-3 in Louisville.
- XRF readings made by the X-MET 880 in Louisville.
- Variability XRF readings taken on the sampling locations that followed a change in substrate in Louisville. (See chapter 3, section 5.2.4 for a detailed explanation of variability XRF readings).
- XRF readings taken on the bare concrete substrates covered by the yellow (3.53 mg/cm^2) NIST SRM film in Louisville.

The summary statistics shown in Tables 6-324 and 6-325 are for XRF readings taken by the MAP-3 and X-MET 880 in Louisville, respectively. These data were collected during the pilot study. The data obtained by the X-MET 880 in the pilot study consisted of only 100 sampling locations. Sufficient data, however, was collected from locations composed of metal and wood by the X-MET 880 in Louisville to allow limited analyses to be performed. These data were analyzed separately from the Denver and Philadelphia data and are presented in section 6.4.

The next six tables, 6-326 through 6-331, provide summary statistics of the variability readings. In Louisville, variability readings were taken on the first sampling location after a change in substrate had occurred. These reading were taken to examine if the XRF instruments behaved erratically when changing from one substrate to another. Variability readings were an additional five repetitions of readings using the same data collection protocol as was used when the readings were first taken at that same sampling location. Thus, at each sampling location after a change in substrate had occurred, a total of six repetitions of readings were taken using the same data collection protocol. The latter five were designated as variability

readings.

All four participating XRF instruments were used to take the five repetitions of variability readings. Since, in Louisville, a change in substrate occurred eight times, there were forty (eight sampling locations times five repetitions) applications of variability readings taken per XRF instrument. However, the Microlead I only took variability readings at seven of the sampling locations after a change in substrate had occurred. Tables 6-326 through 6-328 show the results for all variability readings taken on the painted surface of the sampling location. Tables 6-329 through 6-331 show the results for all variability readings taken on the red NIST SRM film that had been placed on the bare substrate area of the sampling location.

To make comparisons, results from standard readings taken at variability locations in the full study are shown in the next two tables. Tables 6-332 and 6-333 provide summary statistics for the first standard paint and the first standard red NIST SRM readings taken at variability locations, respectively.

The summary statistics for all XRF instrument types show that the standard paint readings taken prior to the variability readings at the same locations were very consistent with the mean of the five variability (first-paint) readings shown in Table 6-326. Similarly, the same is true for the readings taken on the bare substrate covered with the red NIST SRM film. The Microlead I, the X-MET 880, and the XK-3 instruments also show very consistent results when comparing the second and third paint readings to the variability reading means. (The MAP-3 made only one paint reading per sampling location except for "special" readings.) Specifically, the results shown in Table 6-332 are very consistent with results shown in Tables 6-326, 6-327, and 6-328 as are the results shown in Table 6-333 compared to Tables 6-329, 6-330, and 6-331. This implies that the instruments remained in control after a change of substrate occurred and that significant variability did not occur, at least when testing was performed on like substrate components grouped together as was done in this study.

Table 6-334 provides results from collecting readings on concrete substrate in Louisville. According to the data collection protocol used in Louisville, several additional readings were made if the substrate was concrete. These

additional readings were made on yellow (3.53 mg/cm^2) NIST SRM film over bare concrete substrate. Table 6-334 provides summary statistics for each type of reading minus 3.53 mg/cm^2. For the MAP-3 K-shell, the "special" results are consistently greater than the 60-second or variability results. This same relationship was not present for the MAP-3 L-shell. The results for the Microlead I and X-MET 880 were consistent across data types. The XK-3 displayed differences across data types.

To make comparisons to standard data results, results from standard readings taken at locations composed of concrete in Louisville are shown in the next four tables. Tables 6-335 and 6-336 provide summary statistics for the first paint and red NIST SRM reading taken at concrete locations in Louisville, respectively. Tables 6-337 provides summary statistics for the first standard paint reading minus the corresponding laboratory result in mg/cm^2 at concrete substrate locations in Louisville. Table 6-338 provides summary statistics for the first red NIST SRM reading taken from "special" locations in the pilot study minus 1.02 mg/cm^2. Of these four tables, the first standard red NIST SRM results in Table 6-338 provide the most informative comparison with the results for readings taken using the yellow NIST SRM in Table 6-334.

Table 6-324. Summary Statistics of Lead Measured in mg/cm² Units of the MAP-3 Paint and Red (1.02 mg/cm²) NIST SRM Readings (Non-Standard) in Louisville Only.

DATA SOURCE	READING	NUMBER OF READINGS	ARITHMETIC MEAN	MAXIMUM	MINIMUM	25th PERCENTILE	MEDIAN	75TH PERCENTILE
K-shell	Paint	100	2.728	21.277	0.000	0.019	0.872	3.598
	NIST	100	1.560	3.958	0.200	1.182	1.500	1.836
L-shell	Paint	100	0.744	6.204	0.000	0.000	0.000	0.685
	NIST	100	1.3679	2.508	0.000	1.134	1.402	1.563
Laboratory	Paint	100	1.9324	14.047	0.0001	0.128	0.388	2.555

Table 6-325. Summary Statistics of Lead Measured in mg/cm² Units of the X-MET 880 Non-Standard Readings, Louisville Only.

READING	NUMBER OF READINGS	ARITHMETIC MEAN	MAXIMUM	MINIMUM	25th PERCENTILE	MEDIAN	75TH PERCENTILE
First Paint	100	1.303	8.065	0.000	0.095	0.301	2.035
Second Paint	100	1.299	8.177	0.000	0.121	0.306	2.003
Third Paint	100	1.292	8.200	0.000	0.096	0.275	2.097
First NIST SRM	100	1.213	3.093	0.879	1.045	1.134	1.228
Second NIST SRM	100	1.196	2.758	0.902	1.036	1.112	1.202
Third NIST SRM	100	1.184	2.651	0.800	1.047	1.130	1.203
Laboratory	100	1.9324	14.047	0.0001	0.128	0.388	2.555

Table 6-326. Summary Statistics of Lead Measured in mg/cm² Units of the First Variability Paint Reading (Non-Standard Data) For All XRF Instrument Types.

XRF TYPE	NUMBER OF READINGS	ARITHMETIC MEAN	MAXIMUM	MINIMUM	25th PERCENTILE	MEDIAN	75TH PERCENTILE
MAP-3 K-shell	40	1.685	5.933	0.000	0.000	0.622	3.134
MAP-3 L-shell	40	0.209	0.944	0.000	0.000	0.000	0.372
Microlead I	35	1.683	5.900	-0.700	0.200	0.700	4.200
X-MET 880	40	0.687	2.636	0.000	0.036	0.210	1.226
XK-3	40	1.220	4.900	-0.700	0.000	0.400	2.350

Table 6-327. Summary Statistics of Lead Measured in mg/cm² Units of the Second Variability Paint Reading (Non-Standard Data) For All XRF Instrument Types.

XRF TYPE	NUMBER OF READINGS	ARITHMETIC MEAN	MAXIMUM	MINIMUM	25th PERCENTILE	MEDIAN	75TH PERCENTILE
MAP-3 K-shell	0	na	na	na	na	na	na
MAP-3 L-shell	0	na	na	na	na	na	na
Microlead I	35	1.654	5.600	-0.500	0.000	0.600	4.700
X-MET 880	40	0.682	2.522	0.000	0.024	0.171	1.268
XK-3	40	1.330	4.700	-0.800	0.100	0.550	2.650

Table 6-328. Summary Statistics of Lead Measured in mg/cm^2 Units of the Third Variability Paint Reading (Non-Standard Data) For All XRF Instrument Types.

XRF TYPE	NUMBER OF READINGS	ARITHMETIC MEAN	MAXIMUM	MINIMUM	25th PERCENTILE	MEDIAN	75TH PERCENTILE
MAP-3 K-shell	0	na	na	na	na	na	na
MAP-3 L-shell	0	na	na	na	na	na	na
Microlead I	35	1.737	5.600	-0.800	0.200	0.900	4.800
X-MET 880	40	0.684	2.444	0.000	0.042	0.223	1.269
XK-3	40	1.268	4.600	-0.400	0.100	0.450	2.700

Table 6-329. Summary Statistics of Lead Measured in mg/cm^2 Units of the First Variability Red (1.02 mg/cm^2) NIST SRM Reading (Non-Standard) For All XRF Instrument Types.

XRF TYPE	NUMBER OF READINGS	ARITHMETIC MEAN	MAXIMUM	MINIMUM	25th PERCENTILE	MEDIAN	75TH PERCENTILE
MAP-3 K-shell	40	1.320	2.326	0.301	0.959	1.217	1.715
MAP-3 L-shell	40	1.332	1.687	1.055	1.213	1.342	1.429
Microlead I	35	1.289	1.900	0.400	1.000	1.300	1.600
X-MET 880	40	1.132	1.392	0.909	1.058	1.145	1.213
XK-3	40	1.283	3.800	0.600	1.000	1.200	1.450

Table 6-330. Summary Statistics of Lead Measured in mg/cm² Units of the Second Variability Red (1.02 mg/cm²) NIST SRM Reading (Non-Standard) For All XRF Instrument Types.

XRF TYPE	NUMBER OF READINGS	ARITHMETIC MEAN	MAXIMUM	MINIMUM	25th PERCENTILE	MEDIAN	75TH PERCENTILE
MAP-3 K-shell	0	na	na	na	na	na	na
MAP-3 L-shell	0	na	na	na	na	na	na
Microlead I	35	1.180	2.300	0.000	0.900	1.200	1.500
Microlead I	40	1.122	1.466	0.915	1.025	1.136	1.194
XK-3	40	1.243	2.000	0.200	0.900	1.200	1.650

Table 6-331. Summary Statistics of Lead Measured in mg/cm² Units of the Third Variability Red (1.02 mg/cm²) NIST SRM Reading (Non-Standard) For All XRF Instrument Types.

XRF TYPE	NUMBER OF READINGS	ARITHMETIC MEAN	MAXIMUM	MINIMUM	25th PERCENTILE	MEDIAN	75TH PERCENTILE
MAP-3 K-shell	0	na	na	na	na	na	na
MAP-3 L-shell	0	na	na	na	na	na	na
Microlead I	35	1.140	2.200	-0.400	0.600	1.200	1.500
X-MET 880	40	1.145	1.342	0.819	1.063	1.156	1.216
XK-3	40	1.143	2.200	0.400	0.900	1.000	1.300

Table 6-332. Summary Statistics of Lead Measured in mg/cm^2 Units of the First Standard Paint Reading at Variability Locations For All XRF Instrument Types.

XRF TYPE	NUMBER OF READINGS	ARITHMETIC MEAN	MAXIMUM	MINIMUM	25TH PERCENTILE	MEDIAN	75TH PERCENTILE
MAP-3 K-shell	8	1.653	5.369	0.000	0.010	0.386	3.532
MAP-3 L-shell	8	0.208	0.861	0.000	0.000	0.003	0.400
Microlead I	7	1.786	5.600	-0.400	0.100	0.700	5.100
X-MET 880	8	0.678	2.457	0.000	0.022	0.188	1.276
XK-3	8	1.300	4.500	-0.100	0.150	0.550	2.300

Table 6-333. Summary Statistics of Lead Measured in mg/cm^2 Units of the First Standard Red NIST SRM Reading at Variability Locations For All XRF Instrument Types.

XRF TYPE	NUMBER OF READINGS	ARITHMETIC MEAN	MAXIMUM	MINIMUM	25TH PERCENTILE	MEDIAN	75TH PERCENTILE
MAP-3 K-shell	8	1.411	2.345	0.824	1.018	1.185	1.856
MAP-3 L-shell	8	1.340	1.594	1.121	1.296	1.316	1.392
Microlead I	7	1.400	2.400	0.700	0.900	1.500	1.800
X-MET 880	8	1.112	1.247	0.943	1.051	1.133	1.168
XK-3	8	1.288	1.900	0.800	1.000	1.200	1.600

Table 6-334. Summary Statistics of Lead Measured in mg/cm² Units of the Yellow (3.53 mg/cm²) NIST SRM Readings minus 3.53 mg/cm² (Non-Standard) on Concrete in Louisville Only.

XRF TYPE	DATA TYPE	NUMBER OF MEASUREMENTS	ARITHMETIC MEAN	MAXIMUM	MINIMUM	25th PERCENTILE	MEDIAN	75TH PERCENTILE
MAP-3 K-shell	60-second[a]	8	0.066	0.639	-2.017	-0.002	0.191	0.541
	Special[b]	6	0.578	1.100	0.132	0.163	0.545	0.982
	Variability[a]	10	0.165	0.739	-0.498	0.086	0.151	0.372
MAP-3 L-shell	60-second[a]	8	-0.101	0.544	-2.254	-0.351	0.354	0.450
	Special[b]	6	0.189	0.665	-0.069	0.007	0.069	0.396
	Variability[a]	10	0.346	0.985	-0.158	-0.072	0.248	0.777
Microlead I	15-second[c]	24	-0.167	0.570	-1.030	-0.430	-0.130	0.170
	Special[d]	8	-0.030	0.370	-0.830	-0.230	0.120	0.220
	Variability[b]	30	-0.150	0.570	-1.130	-0.430	-0.030	0.170
X-MET 880	15-second	24	0.483	1.035	0.077	0.357	0.443	0.658
	Special[b]	2	0.565	0.575	0.555	0.555	0.565	0.575
	Variability[a]	30	0.580	0.904	0.220	0.423	0.583	0.692
XK-3	15-second[c]	24	-0.730	0.070	-1.230	-1.030	-0.730	-0.430
	Special[d]	8	-0.292	0.170	-0.730	-0.630	-0.180	-0.080
	Variability[b]	30	-0.403	0.470	-1.230	-0.630	-0.430	-0.130

[a] One 60-second reading.
[b] Average of three 15-second readings.
[c] One 15-second reading.
[d] Average of four 15-second readings.

Table 6-335. Summary Statistics of Lead Measured in mg/cm^2 Units For First Paint Reading (Standard) Data for All XRF Instrument Types at Pilot Study Concrete Locations Only.

XRF TYPE	NUMBER OF READINGS	ARITHMETIC MEAN	MAXIMUM	MINIMUM	25th PERCENTILE	MEDIAN	75TH PERCENTILE
MAP-3 K-shell	8	0.754	3.480	0.000	0.000	0.025	1.252
MAP-3 L-shell	8	0.540	3.915	0.000	0.000	0.005	0.196
Microlead I	8	1.188	4.000	0.000	0.500	0.700	1.500
X-MET 880	8	0.542	2.027	0.079	0.111	0.289	0.715
XK-3	8	0.563	2.500	-0.700	0.100	0.300	0.950

Table 6-336. Summary Statistics of Lead Measured in mg/cm^2 Units For First Red (1.02 mg/cm^2) NIST SRM Reading (Standard) Data For All XRF Instrument Types at Pilot Study Concrete Locations Only.

XRF TYPE	NUMBER OF READINGS	ARITHMETIC MEAN	MAXIMUM	MINIMUM	25th PERCENTILE	MEDIAN	75TH PERCENTILE
MAP-3 K-shell	8	1.583	3.958	0.554	0.953	1.370	1.754
MAP-3 L-shell	8	1.242	1.578	0.686	1.146	1.301	1.390
Microlead I	8	1.525	2.100	0.900	1.200	1.600	1.800
X-MET 880	8	1.092	1.224	0.971	1.047	1.072	1.153
XK-3	8	0.988	1.600	0.700	0.700	0.950	1.150

Table 6-337. Summary Statistics of Lead Measured in mg/cm² Units For First Paint Reading (Standard) Data Corrected by ICP For All XRF Instrument Types at Pilot Study Concrete Locations Only.

XRF TYPE	NUMBER OF READINGS	ARITHMETIC MEAN	MAXIMUM	MINIMUM	25th PERCENTILE	MEDIAN	75TH PERCENTILE
MAP-3 K-shell	8	-0.164	-0.036	-0.433	-0.222	-0.138	-0.061
MAP-3 L-shell	8	-0.378	0.371	-2.324	-0.251	-0.239	-0.047
Microlead I	8	0.270	0.764	-0.334	-0.082	0.397	0.550
X-MET 880	8	-0.376	0.094	-1.589	-0.804	0.001	0.047
XK-3	8	-0.355	0.143	-1.044	-0.835	-0.192	0.057

Table 6-338. Summary Statistics of Lead Measured in mg/cm² Units For First Red NIST SRM Reading (Standard) Data Minus 1.02 mg/cm² For All XRF Instrument Types at Pilot Study Concrete Locations Only.

XRF TYPE	NUMBER OF READINGS	ARITHMETIC MEAN	MAXIMUM	MINIMUM	25th PERCENTILE	MEDIAN	75TH PERCENTILE
MAP-3 K-shell	8	0.563	2.938	-0.466	-0.067	0.350	0.734
MAP-3 L-shell	8	0.222	0.558	-0.334	0.126	0.281	0.370
Microlead I	8	0.505	1.080	-0.120	0.180	0.580	0.780
X-MET 880	8	0.072	0.204	-0.049	0.027	0.052	0.133
XK-3	8	-0.033	0.580	-0.320	-0.320	-0.070	0.130

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Chapter 7 Summary: Data Quality Assurance and Quality Control

- **Four types of errors were investigated.**

  -- Monitor/Operator errors,
  -- Data Entry errors,
  -- Programming errors, and
  -- "Other" errors.

- **Several quality control methods and systems were employed to assure the quality of the field data. These were:**

  -- Data Entry Systems,
  -- Exploratory Data Analysis,
  -- Captured Data Comparisons,
  -- Double Data Entry, and
  -- 100 Percent Verification.

- **Three error rates were computed. The first is the error rate found through the compare procedure using captured data, the second is the error rate found through double data entry, and the third is the residual error rate remaining in the data sets after double data entry and captured data comparison procedures were completed, that is, after completion of all QC steps.**

  -- The overall compare procedure error rate is:
  XRF Standard and Non-Standard data: 1.96%,
  XRF Control data: 2.00%.

  -- The overall double data entry error rate is:
  All XRF data: 0.41%.

  -- The overall residual data entry error rate is:
  Sample definition data: 0.07%,
  Test kit data: 0.10%,
  XRF Standard and Non-Standard data: 0.04%,
  XRF Control data: 0.011%.

- **Laboratory system audits, performance audits, and data audits were performed by the Senior Quality Assurance**

7 DATA QUALITY ASSURANCE AND QUALITY CONTROL

7.1 QUALITY ASSURANCE AND QUALITY CONTROL PROCEDURES

Quality control (QC) procedures were employed throughout the data management process to assure the quality of the field data. Five data sets were created. The common variable shared by all five data sets and used to map data between data sets is the sample identification number. Ideally, each analysis data set contains at least one record for each sample identification number. The names of the analysis data sets that were created, a brief description of each, and the numbers of records and variables per record included in them are given in Table 7-1.

7.2 ERROR IDENTIFICATION

Although many measures were taken during field work and data management to minimize the occurrence of errors in the study data, it is impossible to completely eliminate all errors. Several types of errors were possible. These error types are identified below.

Monitor/Operator errors: These are errors that occurred during the actual testing process when incorrect data was recorded. For QC discussions, an operator either performs test kit testing or operates an XRF. A monitor is the XRF monitor. Examples are: transposition of numbers by operator or monitor, adding extra decimal places to the reading of a single decimal place XRF, failure to record data, or reversal of the XRF reading sign. These errors occur in the Test Kit, XRF, and XRF Control data sets.

Data Entry errors: These are errors made by the data entry personnel when entering the data. Examples include number transposition, entering the wrong data point, misplacement of the decimal point, and failure to enter data which results in missing data. Such errors occur more often in the XRF and XRF Control data sets due to the more complicated data (i.e., multiple decimal XRF readings) and larger amounts of data.

Programming errors: These are errors which occur during or are the direct result of errors in data transfer or analysis. Examples may be XRF or test kit record mis-identification.

Table 7-1. Data Set Descriptions.

DATA SET	CONTENTS	NUMBER OF:	
		RECORDS	VARIABLES PER RECORD
SAMPLE DEFINITION	Sample location and substrate information	1,314	13
TEST KIT	Test kit data	7,185	7
XRF	Standard and "special" XRF data excluding control data	15,836	21
XRF CONTROL	Initial, continuing, and ending XRF control data	5,594	18
LABORATORY	Laboratory ICP paint chip analysis results	1,314	18

"Other" errors: "Other" errors are errors that cannot be attributed to a specific cause. An example of this type of error would be if a device reacted to the heat of direct sunlight and recorded extreme measurements. This error is only present in the XRF and XRF Control data sets.

7.3 QUALITY CONTROL METHODS AND SYSTEMS

7.3.1 Data Entry Systems

Quality control began with data entry. The data entry personnel used a menu-driven data entry software that provides the ability to develop specific data entry regimes. Screens resembling the data forms were designed to facilitate the data entry process and reduce errors. The data entry software also performs simple data entry error checks, such as limiting numeric variables to an assigned range, thus identifying data entry errors before the information is written to a data file. The user can sort and list records to identify inconsistent data, and has easy access to individual records for updating or editing in the event that an error is identified. Output from this data entry software were flat files storing data from Philadelphia, Denver, and Louisville separately.

Output from data entry along with the disk files provided by the laboratory were input into statistical analysis software programs that created the analysis data sets.

7.3.2 Exploratory Data Analysis

Exploratory data analysis methods, derived from the pilot study, were applied to the Sample Definition, Test Kit, XRF, and XRF Control data sets. These techniques were used to identify data errors of large magnitude within these four data sets. Data errors of smaller magnitude were identified through other means discussed later in this chapter. Data errors found included data entry errors, monitor errors, operator errors, programming errors, and "other" errors. The techniques applied included data sorts and tabulations, frequency tables, and outlier analysis using summary statistics and graphics.

Data sorts and tabulations help to point out possible data entry, operator/monitor, and "other" errors. Data sorts were performed on the data sets before any other QC was performed. Data sorts rearrange the observations according to the values of a specific variable. After the sort, observations out of order or which exceeded specified ranges were flagged as possible data errors. For example, a sort of the difference between the test kit start time and end time was used to identify errors present in the start and end times. Sorts were performed on the following variables:

- Time (both start and end times)
- Testing date
- The difference between test kit start time and end time
- Test kit sampler or XRF operator
- Test kit identification codes
- XRF device identification codes.

Frequency tables were a useful means for providing counts of categorized data and were applied to the data sets. Also, frequency counts by values of a variable show error. Count discrepancies indicated that the type of error could be an operator/monitor errors or data entry errors. For example, the number of results categorized by substrate should be equal for all test kits. Other examples of frequency counts performed for test kit categorizations and, in similar fashion, for the XRF results are listed below.

- Substrates by test kit
- Identification number by unit
- Substrate by address
- XRF device by operator

- Test kit by tester
- XRF readings per device

Once frequency counts were completed and verified, outlier data point identification was performed on the Test Kit and XRF data sets. A description of the methodology follows. Specific groups of XRF and test kit data were created. The arithmetic means and sample standard deviations were computed for each grouping. Next, any data point greater than three standard deviations from its corresponding group mean was flagged as a possible error. These data points were then verified against copies of the original data sheets for accuracy. This methodology was applied to variables stored in the Test Kit, XRF and XRF Control data sets and was especially useful in identifying monitor/operator and "other" errors. Most of the errors identified at this stage were large in magnitude. Smaller errors were identified through other means, discussed later in this chapter.

Graphical analyses provided a visual method for detecting data errors. Scatter plots applied summary statistics and were designed to detect large errors by comparing XRF readings taken at the same sampling location. These plots were created for specific groups of XRF readings from both the XRF and XRF Control data sets. For each city by XRF device by operator combination, scatter plots were generated which compared readings performed at common sampling locations. At each sampling location, several XRF readings were made. Typically, three readings were performed on the painted surface (designated here as paint 1, paint 2, and paint 3, respectively). Likewise, three XRF readings taken on the red NIST standard and on the bare substrate are designated red 1, red 2, red 3, bare 1, bare 2, and bare 3, respectively. Each axis on the scatter plots measures a reading taken at a sampling location. Scatter plots were created by matching pairs of readings. For example, the vertical axis measured the paint 1 readings and the horizontal axis measured the paint 2 readings. The regression of the two variables where the vertical axis measures the dependent variable was drawn with the 99% confidence limits on the individual predicted values. Any data point outside of the 99% confidence limit was researched. Scatter plots were created for each city by XRF by operator combination for the pairs of readings given below.

- paint 1 vs paint 2

- paint 1 vs paint 3
- red 1 vs red 2
- red 1 vs red 3
- bare 1 vs bare 2
- bare 1 vs bare 3

Scatter plots of the average initial control readings versus the average end control readings were developed using the same city by XRF device by operator combinations as described above.

When researching data flagged as possible errors, all data was compared to copies of the original data sheets and information provided by the operators. Corrections were made where needed.

7.3.3 Captured Data Comparisons

Once the aforementioned exploratory data analysis had been completed, comparisons of the already entered data versus the captured data files provided by the XRF operators were performed using programs written in statistical analysis software. Comparisons were performed using XRF and XRF Control data sets. Captured data comparisons are a unique method for identifying and obtaining missing data and identifying data entry errors, monitor errors, and operator errors. In a few instances, the captured data gave indication that a machine error had occurred. All types of discrepancies found during captured data comparison are discussed in the Error Rates section. The captured data comparison methodology is described below.

Data disks storing Denver and Philadelphia data were received from operators of the two MAP-3 instruments (MAP-3 (I) and MAP-3 (II)), the X-MET 880, and the Lead Analyzer. No data disk were available from any of the Louisville XRF operators. Complete data were present from Philadelphia. However, several of the XRF operators did not provide data for all of the addresses in Denver. The X-MET 880 data logger was inoperative during testing of buildings B, I, J, and portions of building H. Also, the second MAP-3 operator did not provide data from all of buildings C and D, and portions of building B data. Some data was also missing because the MAP-3 devices in Denver stored all negative readings as 0.000 mg/cm^2 rather than the actual negative value as displayed to the operator. This problem did not occur during testing in Philadelphia because both MAP-3 devices had been modified to store negative values, however, many negative readings were incorrectly stored as 0.000 mg/cm^2 in the Denver captured data files. The number of Denver readings incorrectly stored as a result of this phenomenon is shown in Table 7-2.

Since each XRF device has its own method for data storage, a more detailed description is given below.

- The MAP-3 devices stored the data in the form of a tabular listing which included ID number, component structure, sample number (1-3 for the three paint readings, 4-6 for the three red NIST readings), K-shell reading, L-shell reading, soil reading, reading length in seconds, and date (Figure 7-1).

Table 7-2. Number of Negative Readings Incorrectly Stored in the MAP-3 Denver Captured Data Files.

DEVICE	DATA TYPE	
	Standard and Special	Control
MAP-3 (I) K-shell	1167	751
MAP-3 (I) L-shell	1531	969
MAP-3 (II) K-shell	710	541
MAP-3 (II) L-shell	847	619

- The Lead Analyzer stored data in the form of text. The captured data files included the date, time of reading, operator entered substrate sequence number, L-shell reading, and K-shell reading. Figure 7-2 provides an example. The substrate sequence number denotes the sample's placement within the current substrate. For example, the first wood sample within a house would be labeled 1W. Each wood sample after that would be sequentially labeled 2W, 3W, etc. If the next substrate present in that house was drywall, the first drywall sample would be labeled 1D, with the next samples sequentially labeled 2D, 3D, etc. The substrate sequence number was used in place of the identification number.

```
Application:Pb-IN-PAINT            Q015 4-JULY-1993
Meas Time:  12-OCT-1993  14:17:38
ID: <43M>
(    ) (      )
                 Value        Std. dev.
       PbL        0.580052       0.0146903 mg/cm^2
       PbK         2.24696        0.257504 mg/cm^2

Application:Pb-IN-PAINT            Q015 4-JULY-1993
Meas Time:  12-OCT-1993  14:18:32
ID: <44M>
(    ) (      )
                 Value        Std. dev.
       PbL         1.01137       0.0191814 mg/cm^2
       PbK         1.45181        0.214201 mg/cm^2

Application:Pb-IN-PAINT            Q015 4-JULY-1993
Meas Time:  12-OCT-1993  14:19:13
ID: <45M>
(    ) (      )
                 Value        Std. dev.
       PbL         1.01945       0.0192311 mg/cm^2
       PbK        0.568510        0.174674 mg/cm^2

Application:Pb-IN-PAINT            Q015 4-JULY-1993
Meas Time:  12-OCT-1993  14:19:50
ID: <46M>
(    ) (      )
                 Value        Std. dev.
       PbL         1.02456       0.0192766 mg/cm^2
       PbK        0.790094        0.188462 mg/cm^2

Application:Pb-IN-PAINT            Q015 4-JULY-1993
Meas Time:  12-OCT-1993  14:20:55
ID: <47M>
(    ) (      )
                 Value        Std. dev.
       PbL        0.119683      0.00678722 mg/cm^2
       PbK         2.83148        0.288397 mg/cm^2
```

Figure 7-2. Example of the Lead Analyzer data storage method.

```
>
MODEL 3 ?
MODEL 3 ? 2
OLD ASSAY MODEL PBDRYPLAST

>
MEASURING TIME 15 ? 16

> 41
WHAT?

>
(MODEL 2: PBDRYPLAST)  DATE: 05.08.93  TIME: 10-05-10
MEASURING: PROBE 6 TYPE DOPS (A)
               16 SECONDS
ASSAYS:PB 3.694

>
(MODEL 2: PBDRYPLAST)  DATE: 05.08.93  TIME: 10-05-38
MEASURING: PROBE 6 TYPE DOPS (A)
               16 SECONDS
ASSAYS:PB 3.766

>
(MODEL 2: PBDRYPLAST)  DATE: 05.08.93  TIME: 10-06-06
MEASURING: PROBE 6 TYPE DOPS (A)
               16 SECONDS
ASSAYS:PB 3.712
```

Figure 7-3. Example of the X-MET 880 data storage method.

- The X-MET 880 also stored the data in the form of text, similar to the Lead Analyzer. The captured data from the Denver X-MET 880 included the sampling location identification number, operator input substrate mode, date, time of reading, length of reading, and reading result

ID#	Comp Struct	Sample#	K-Shell mg/cm^2	L-Shell mg/cm^2	Pb-Soil ppm	Time seconds	Date
355		1	0.000	0.000	0	16	0
355		2	0.000	0.003	0	16	0
355		3	0.000	0.000	0	16	0
355		4	1.129	1.224	0	16	0
355		5	1.642	1.354	0	16	0
355		6	0.891	1.306	0	16	0
356		1	0.000	0.050	0	16	0
356		2	0.000	0.000	0	16	0
356		3	0.000	0.029	0	16	0
356		4	0.959	1.286	0	16	0
356		5	1.065	1.281	0	16	0
356		6	0.982	1.207	0	16	0
357		1	0.170	0.000	0	16	0
357		2	0.000	0.000	0	16	0
357		3	0.000	0.000	0	16	0
357		4	0.158	1.215	0	16	0
357		5	1.221	1.331	0	16	0
357		6	0.873	1.221	0	16	0
358		1	0.000	0.040	0	16	0
358		2	0.000	0.000	0	16	0
358		3	0.000	0.000	0	16	0
358		4	0.822	1.189	0	16	0
358		5	1.364	1.373	0	16	0
358		6	0.814	1.219	0	16	0
359		1	0.000	0.450	0	16	0
359		2	1.006	0.397	0	16	0
359		3	0.000	0.400	0	16	0
359		4	0.368	0.367	0	16	0
359		5	0.384	1.401	0	16	0
359		6	0.633	1.454	0	16	0
359		7	1.216	1.351	0	16	0

Figure 7-1. Example of the MAP-3 data storage method.

measured in mg/cm^2. The operator entered one of four substrate modes depending upon the underlying substrate of the sample. Figure 7-3 provides an example. The captured data collected in Philadelphia contained the same information, but <u>without an identification number</u>.

To complete the comparison procedure, the captured data files passed through three stages of processing. Because of the differing storage formats of the files, the files were modified from their original form. This is the first stage of captured data processing. This was done by modifying the captured data files with a FORTRAN program that would arrange the data into one common file format.

Output from the FORTRAN program consisted of eight files, one for each instrument by Denver and Philadelphia combination. These files entered the second processing stage. The purpose of the second stage is to properly order records in the captured data files so that the individual data items can be compared to those of the data set during stage three. A description of the second processing stage follows.

Along with the expected information, the captured data files also contained extra data such as additional readings, incorrect readings (for example, taken with the wrong NIST standard, taken on the wrong surface, etc.), and the operator's calibration readings. In a very few cases, unexplained text appeared in place of readings. Before the captured data and the previously entered data could be compared, it was necessary to manually edit and remove extra data from each of the eight files. To accomplish this, each file was compared with the data set. After each pass of the comparison procedure, records were identified for output. These records were output because they belonged to a group of mismatched records. Records became mismatched when, for example, missing or extra data was present in the captured data files. Those records output were visually compared with the project data sheets and adjustments made where necessary. In most cases, the reason for the mismatch was that an extra reading had been performed (for example, one or two additional readings per identification number) or readings were not saved to the captured data file. Frequently there were comments from the operator on the field data sheet explaining the presence of extra readings. Additional readings were only used when operator comments indicated the original reading was incorrectly tested. For each mismatch, the problem was corrected so that the order of records would be properly matched.

Next, the captured data files entered the third and final stage of processing. In this stage, the comparison procedure was again performed, but instead of comparing the order in which readings occur, individual data items were compared. A description of the final processing stage follows.

For each XRF device, XRF readings were compared for observations with matching identification data (eg., identification number, sample number, etc.), and to be considered a match the data set had to <u>exactly</u> match the captured data. Any readings which did not match the captured data were researched against copies of the original field data sheets, documented and corrected if needed. However, it was found that in most instances the Lead Analyzer captured data and both Denver MAP-3 captured data files did not exactly match the data set.

As a result of their storage of negative values as 0.000 mg/cm^2, no negative MAP-3 data set readings from Denver matched their captured data counterparts. To contend with this, the compare code converted all negative data set readings to 0.000 mg/cm^2, eliminating the mismatch during the compare procedure, but not guaranteeing error free data. For example: if a negative reading was incorrectly recorded or entered as another negative reading, it would still exactly match the captured data reading because they would both be compared as 0.000 mg/cm^2. Only if the negative reading was incorrectly recorded as a positive reading, or incorrectly entered as a positive reading would it be identified as an error through the compare procedure. Again, data from the MAP-3 devices used in Philadelphia did not present this problem.

In addition to the MAP-3 data storage problem, the Lead Analyzer also presented a data storage problem. In many cases throughout Denver and Philadelphia testing, the Lead Analyzer had rounded the actual value of the reading when it was displayed to the operator, but stored the complete reading in the captured data. As a result, the compare procedure identified a majority of readings as mismatched data. To prevent this, the comparison procedure for the Lead Analyzer was changed so that the data set value had to be at least ± 0.05 mg/cm^2 different from the captured data value to be considered a mismatch. This method was useful for identifying significant discrepancies between the captured data and data sets, however, small discrepancies of magnitude less than 0.05 mg/cm^2 were not identified through this process.

All types of discrepancies found during captured data comparison are discussed in the Error Rates section.

7.3.4 Double Data Entry

Double data entry comparisons were performed on the XRF data set for the XL, XK-3, and Microlead I XRF devices, because captured data were not available for comparison. Double data entry comparisons were also performed on the XRF data from those buildings missing data in the MAP-3 and X-MET 880 captured data files from Denver.

Double data entry was performed at the same time, but independent of the captured data comparison process. Data entry personnel entered the XRF instrument, sample identification number, and XRF readings into a data base. The resulting file was input into a statistical analysis software program and compared with the original XRF data set. Discrepancies between

the two data sets were researched. Data entry errors were the only discrepancy types identifiable through double data entry comparisons. All types of discrepancies found during double data entry comparison are discussed in the Error Rates section.

7.3.5 100 Percent Verification

For some study data, every item of every record was compared to copies of the original data sheets. This procedure will be referred to as 100% verification and was performed for the following:

- Sample Definition data set
- Louisville test kit and XRF data
- XRF Control data set

100% verification was done for the Sample Definition data set, XRF Control data set, and Louisville test kit and XRF data because it reduced data errors while remaining time efficient due to the size of these data sets. 100% verification can identify only data entry errors.

7.4 ERROR RATES

Three different error rates were computed. The first is the error rate found through the compare procedure using the captured data. The second is the error rate found through double data entry. The third is the residual error rate remaining in the data sets even after double data entry and captured data comparison procedures were completed, that is, after completion of all QC steps. Whenever possible, error rates have been broken down to individual test kits or XRF instruments. These three error rates are summarized below.

It is important to note that all data error rates were computed on the basis of data items, not records. By convenience, all samples were randomly selected using records as the sampling unit. However, each record contains between 8 and 21 data items, depending upon the data set from which it was retrieved. The formula for computing the error rate is the number of data item errors divided by the number of data items in the sample. Data items were used instead of records as the basis for computing error rates because an error rate computed in this manner is more indicative of the true error rate than an error rate computed on the basis of records. This is supported by the random occurrence of the errors by item and that multiple errors in a single record are rare. For example, one datum in error on a record does not cause the remaining record data items to be in

error.

7.4.1 Comparison Discrepancies

After comparisons had been completed using captured data and double data entry, there were three types of discrepancies identified. All of the identified discrepancies described below were corrected.

1) Monitor/Operator errors: These are errors that occur during the actual testing process. Such errors encompass transposition of numbers by operator or monitor, adding extra decimal places to the reading of a single decimal place XRF, failure to record the necessary data, etc.

2) Data Entry errors: These are errors made by the data entry personnel when entering the data. They include number transposition, entering the wrong data point, misplacement of the decimal point, etc. Data entry errors are the only discrepancy type identified by double data entry comparisons.

3) Undetermined Discrepancies: Some discrepancies which were identified by the captured data comparison could not be attributed to a specific cause. The cause of the discrepancy cannot be determined but it is known that they are not data entry errors. The cause could be an "other" error, or a monitor/operator error. Due to the nature of this discrepancy, it was not possible to determine whether the captured data was correct or whether the field data sheet was correct. In the event of a undetermined discrepancy, the value on the field data sheet was not changed.

Error rates for data within the XRF data set (standard and "special" XRF measurements) determined through the captured data compare process are shown in Table 7-3. The error rates are categorized by discrepancy type for each XRF. Table 7-4 contains error rates computed through captured data comparisons for data from the XRF control data set. Data entry error rates for data from the XRF data set from those devices without captured data were determined through the double data entry process and are shown in Table 7-5. Note that the data entry error rates are much lower than the monitor/operator error rates computed from captured data comparisons. Total error rates for the XRF data set are displayed in Table 7-6 and are categorized by discrepancy type. Table 7-7 contains the error rates categorized by discrepancy type for the XRF control data set.

The magnitude of the three types of errors/discrepancies

identified by comparing the captured data files to the data sets is given in Tables 7-8 through 7-10. Computed for each identified error/discrepancy was the difference and absolute difference between the value found in the data set and the value found in the captured data file. Table 7-8 provides the sample size, arithmetic mean, sample standard deviation, minimum, 25th percentile, median, 75th percentile and maximum of XRF standard and "special" measurements for each type of discrepancy. Tables 7-9 and 7-10 display the same summary statistics as Table 7-8, but for the XRF control data set and the two data sets combined, respectively. A frequency plot of the absolute difference categorized by magnitude of the difference is provided in Figure 7-4. Each category represents the number of discrepancies with absolute differences within a 0.2 mg/cm^2 range. In Figure 7-4, the categories are labeled by their corresponding range midpoint except for the category labeled "2.0+". The range for this category are all absolute differences greater than 1.8 mg/cm^2. The range for the smallest category includes all values less than 0.2 mg/cm^2.

7.4.2 Residual Error Rates

After data entry, exploratory data analysis, captured data comparisons, double data entry comparisons, and 100% verification were completed, random samples of each analysis data set were selected for visual verification against copies of the field data sheets. A stratified sampling scheme was used to randomly select

Table 7-3. Error Rates from Denver and Philadelphia Captured Data Comparison Procedure of XRF Standard and "Special" Readings. Errors are Listed for each Discrepancy Type.

DEVICE	SAMPLE SIZE	DISCREPANCY TYPE	NO. OF ERRORS	% ERROR RATE
Lead Analyzer K-shell	7,986	Mon./Oper Error	35	0.44
		Data Entry Error	45	0.56
		Undetermined Discrepancy	20	0.25
Lead Analyzer L-shell	7,986	Mon./Oper. Error	22	0.28
		Data Entry Error	15	0.19
		Undetermined Discrepancy	3	0.04
MAP-3 (I) K-shell	8,038	Mon./Oper. Error	66	0.82
		Data Entry Error	66	0.82
		Undetermined Discrepancy	16	0.20
MAP-3 (I) L-shell	8,043	Mon./Oper. Error	72	0.90
		Data Entry Error	38	0.47
		Undetermined Discrepancy	36	0.45
MAP-3 (II) K-shell	6,363	Mon./Oper. Error	155	2.44
		Data Entry Error	20	0.31
		Undetermined Discrepancy	71	1.12
MAP-3 (II) L-shell	6,363	Mon./Oper. Error	213	3.35
		Data Entry Error	14	0.22
		Undetermined Discrepancy	33	0.52
X-Met 880	6,163	Mon./Oper. Error	35	0.57
		Data Entry Error	43	0.70
		Undetermined Discrepancy	1	0.02
TOTAL	50,942		1,019	2.00

10% of the total number of records from each data set. The data were stratified by city and XRF device or test kit. This process resulted in samples that will be referred to as the 10% random sample. Code was written in statistical analysis software to perform the random selection process. **Estimates of the residual data entry error rates were made from the 10% random sample.**

Only the residual data entry error rate can be estimated. However, we would expect that monitor/operator errors in the

Table 7-4. Error Rates from Denver and Philadelphia Captured Data Comparison Procedure of XRF Control Readings. Error Rates are Listed for each Discrepancy Type.

DEVICE	SAMPLE SIZE	DISCREPANCY TYPE	NO. OF ERRORS	% ERROR RATE
Lead Analyzer K-shell	3,807	Mon.Oper. Error	22	0.58
		Data Entry Error	4	0.11
		Undetermined Discrepancy	35	0.92
Lead Analyzer L-shell	3,807	Mon./Oper. Error	4	0.11
		Data Entry Error	0	0.00
		Undetermined Discrepancy	5	0.13
MAP-3 (I) K-shell	5,037	Mon./Oper. Error	30	0.60
		Data Entry Error	13	0.26
		Undetermined Discrepancy	7	0.14
MAP-3 (I) L-shell	5,032	Mon./Oper. Error	36	0.72
		Data Entry Error	11	0.22
		Undetermined Discrepancy	24	0.48
MAP-3 (II) K-shell	3,881	Mon./Oper. Error	107	2.76
		Data Entry Error	20	0.52
		Undetermined Discrepancy	38	0.98
MAP-3 (II) L-shell	3,881	Mon./Oper. Error	141	3.63
		Data Entry Error	14	0.36
		Undetermined Discrepancy	28	0.72
X-MET 880	2,590	Mon./Oper. Error	6	0.23
		Data Entry Error	4	0.15
		Undetermined Discrepancy	0	0.00
TOTAL	28,035		549	1.96

instruments without captured data (XL, X-MET 880, and Microlead I) to occur less frequently than those instruments with captured data. This is due to the fact that the captured data instruments have multiple decimal place readings while those instruments without captured data have single decimal place readings. Of the monitor/operator errors identified through captured data comparisons, 50.53 percent occurred in the decimal numbers to

the right of the first decimal place. Also, five of the seven errors found through 10% random verification occurred in the

Table 7-5. Data Entry Error Rates from Denver and Philadelphia Double Data Entry Comparison Procedure Categorized by Device.

DEVICE	SAMPLE SIZE	NO. OF ERRORS	% ERROR RATE
MAP-3 (II) K-shell	1,547	9	0.58
MAP-3 (II) L-shell	1,547	13	0.84
Microlead I (I)	9,252	44	0.48
Microlead I (II)	9,266	24	0.26
X-MET 880	9,273	25	0.27
XK-3 (I)	9,239	52	0.56
XK-3 (II)	9,266	39	0.42
XL	9,273	25	0.27
TOTAL	51,684	212	0.41

Table 7-6. Error Rates from Denver and Philadelphia Captured Data Comparison Procedure for 50,942 XRF Standard and "Special" Readings Listed by Discrepancy Type.

DISCREPANCY TYPE	**CAPTURED DATA**	
	NO. OF ERRORS	**% ERROR RATE**
Mon./Oper. Error	598	1.17
Data Entry Error	241	0.47
Undetermined Discrepancy	180	0.35
All Types Combined	1,019	2.00

Table 7-7. Error Rates from Denver and Philadelphia Captured Data Comparison Procedure for 28,035 XRF Control Readings Listed by Discrepancy Type.

DISCREPANCY TYPE	**CAPTURED DATA**	
	NO. OF ERRORS	**% ERROR RATE**
Mon./Oper. Error	346	1.23
Data Entry Error	66	0.24
Undetermined Discrepancy	137	0.49
All Types Combined	549	1.96

Table 7-8. Summary Statistics from Denver and Philadelphia Captured Data Comparison Procedure of XRF Standard and "Special" Measurements Listed by Discrepancy Type.

STATISTIC		MON./OPER. ERROR	DATA ENTRY ERROR	UNDETERMINED DISCREPANCY
Difference mg/cm^2	Sample Size	598	226	180
	Mean	0.026	-0.004	0.307
	STD Deviation	0.773	0.605	0.866
	Minimum	-3.796	-3.000	-3.374
	25th Percentile	-0.072	-0.104	0.020
	Median	0.001	-0.005	0.133
	75th Percentile	0.133	0.089	0.866
	Maximum	6.108	3.322	2.622
Absolute Difference mg/cm^2	Sample Size	598	226	180
	Mean	0.381	0.322	0.615
	STD Deviation	0.673	0.512	0.682
	Minimum	0.001	0.000	0.001
	25th Percentile	0.008	0.029	0.098
	Median	0.100	0.090	0.344
	75th Percentile	0.500	0.375	1.076
	Maximum	6.108	3.322	3.374

readings of XRFs with multiple decimal numbers. The other two errors occurred in data fields other than XRF reading results. No errors were found in readings from 10% random samples of XRF instruments that output only a single decimal number.

7.4.2.1 Residual Data Entry Error Rates

The residual data entry error rates for the four data sets (Laboratory data set QC is discussed in section 7.5) are summarized below.

- One error was found out of 1,424 data items in the 10% random sample of the Sample Definition data set equalling a data entry error rate of 0.07 percent.

- The data entry error rate computed from the 10% sample of the Test kit data set is five errors in 5,020 data items equaling 0.10 percent.

Table 7-9. Summary Statistics from Denver and Philadelphia Captured Data Comparison Procedure of XRF Control Readings Listed by Discrepancy Type.

STATISTIC		MON./OPER. ERROR	DATA ENTRY ERROR	UNDETERMINED DISCREPANCY
Difference mg/cm^2	Sample Size	346	66	137
	Mean	0.021	-0.038	-0.499
	STD Deviation	0.897	0.881	7.655
	Minimum	-3.000	-6.174	-73.664
	25th Percentile	-0.100	-0.050	-0.041
	Median	0.001	0.000	0.100
	75th Percentile	0.180	0.060	0.642
	Maximum	5.984	1.412	3.409
Absolute Difference mg/cm^2	Sample Size	346	66	137
	Mean	0.457	0.348	1.481
	STD Deviation	0.773	0.809	7.526
	Minimum	0.000	0.000	0.001
	25th Percentile	0.009	0.020	0.100
	Median	0.153	0.052	0.241
	75th Percentile	0.600	0.481	0.918
	Maximum	5.984	6.174	73.664

- Data entry error rates for the XRF data set are shown in Table 7-11. In this sample, 75 records from Denver and 44 records from Philadelphia were randomly selected independently for each XRF device. Note that the third and fourth columns of Table 7-11 describe errors concerning the actual reading values, whereas the fifth and sixth columns comprise errors in other variables such as date, time, operator, etc. The percent error rates for each error type were computed using that portion of the 'Total Number of Items' composed only of data items from the variables associated with that error type. The '% Overall Rate' is computed using the 'No. of XRF Errors' over the 'Total Number of Items'. Notice that four out of five of the XRF reading errors occur in the Lead Analyzer readings. These four were not observed during the captured data comparison

process because they fell within the ± 0.05 mg/cm^2 allowance for rounding.

Table 7-10. Summary Statistics from Denver and Philadelphia Captured Data Comparison Procedure of XRF Standard, "Special" and Control Readings Listed by Discrepancy Type.

STATISTIC		MON./OPER. ERROR	DATA ENTRY ERROR	UNDETERMINED DISCREPANCY
Difference mg/cm^2	Sample Size	944	292	317
	Mean	0.024	-0.011	-0.041
	STD Deviation	0.820	0.676	5.080
	Minimum	-3.796	-6.174	-73.664
	25th Percentile	-0.080	-0.081	-0.005
	Median	0.001	-0.005	0.105
	75th Percentile	0.151	0.081	0.690
	Maximum	6.108	3.322	3.409
Absolute Difference mg/cm^2	Sample Size	944	292	317
	Mean	0.409	0.328	0.989
	STD Deviation	0.712	0.591	4.982
	Minimum	0.000	0.000	0.001
	25th Percentile	0.008	0.027	0.100
	Median	0.100	0.081	0.271
	75th Percentile	0.529	0.400	1.000
	Maximum	6.108	6.174	73.664

- Even though the XRF Control data set had 100% verification performed, a 10% random sample was still selected for residual error rate determination. One error was found in the XRF Control data set. With 9,443 items in the sample the resulting error rate was 0.011% for the XRF Control data set.

7.5 RESULTS OF LABORATORY AUDITS

An evaluation of the laboratory analysis activity was performed by the Senior Quality Assurance Officer (QAO) for the program who is independent from the laboratory operation with respect to line management of the laboratory. The evaluation procedures and results, including system audits, performance audits, and data audits are discussed in sections 7.5.1, 7.5.2,

and 7.5.3. An audit is a systematic evaluation to determine the quality of the operational function.

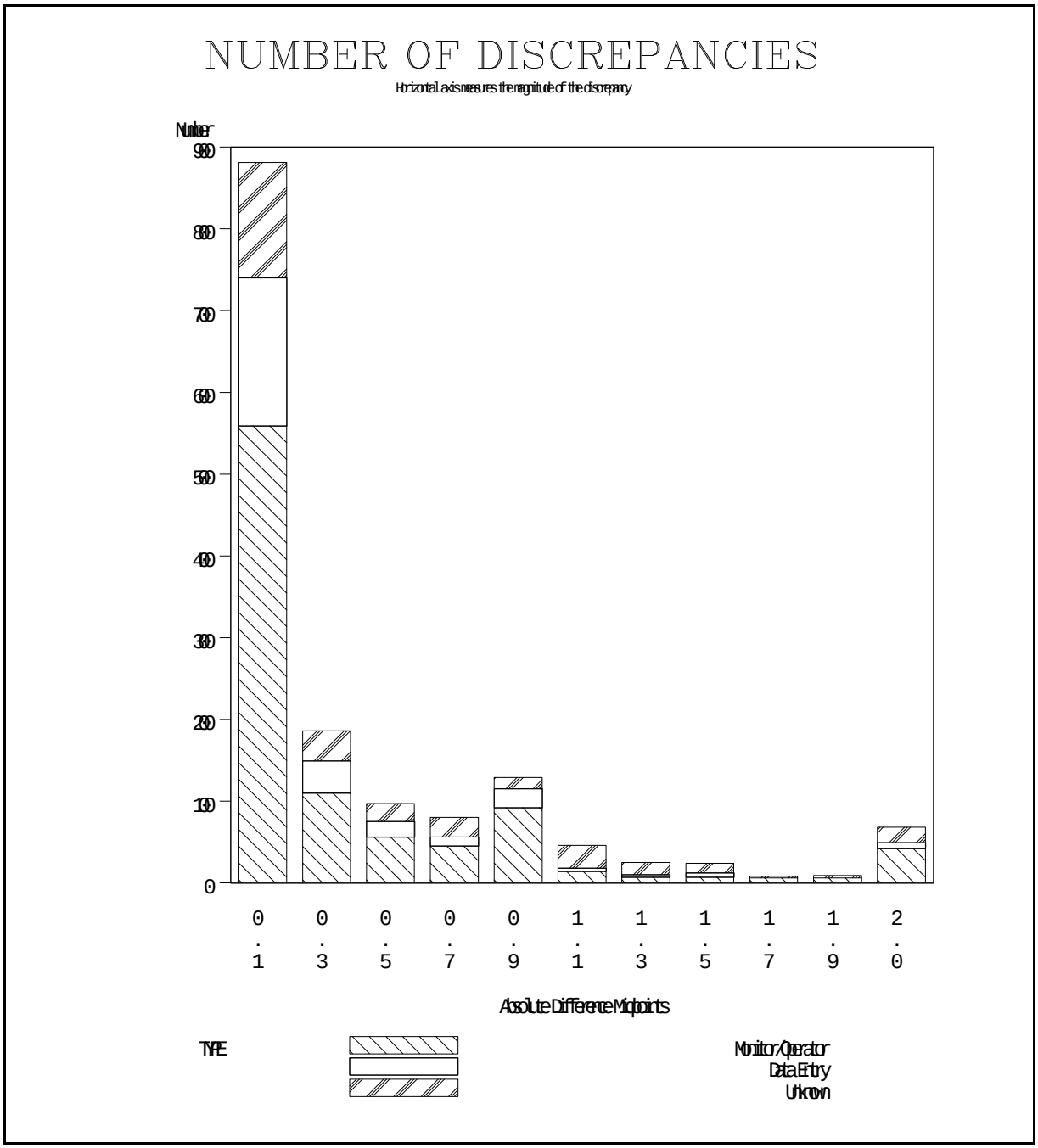

Figure 7-4. Frequency plot of the absolute differences of the errors found through the Denver and Philadelphia captured data comparison process.

Table 7-11. Residual Data Entry Error Rates and Counts in the XRF_NORM Data Set.

DEVICE	TOTAL NUMBER OF ITEMS	No. XRF ERRORS	% XRF ERROR RATE	No. OTHER ERRORS	% OTHER ERROR RATE	TOTAL NO. ERRORS	% OVERALL RATE
Lead Analyzer K-shell	1,613	1	0.13	0	0.00	1	0.06
Lead Analyzer L-shell	1,616	3	0.38	0	0.00	3	0.19
MAP-3 K-shell (I)	1,547	1	0.14	0	0.00	1	0.06
MAP-3 L-shell (I)	1,541	0	0.00	0	0.00	0	0.00
MAP-3 K-shell (II)	1,536	0	0.00	0	0.00	0	0.00
MAP-3 L-shell (II)	1,540	0	0.00	0	0.00	0	0.00
Microlead (I)	1,644	0	0.00	0	0.00	0	0.00
Microlead (II)	1,681	0	0.00	0	0.00	0	0.00
X-MET 880	1,613	0	0.00	0	0.00	0	0.00
XK-3 (I)	1,628	0	0.00	0	0.00	0	0.00
XK-3 (II)	1,634	0	0.00	1	0.12	1	0.06
XL	1,646	0	0.00	1	0.12	1	0.06
TOTAL	19,239	5	0.05	2	0.02	7	0.04

An additional evaluation of the analytical activity was performed separately by EPA. Results of these audits are presented in section 7.5.4.

7.5.1 System Audit

The system audit for this work was a qualitative examination of the facility and the conduct of the analytical task which is all the work required to analyze samples. Requirements for the facility and the conduct of the analytical task are different and therefore, a separate inspection was performed for each. The results of the system audits are given below.

7.5.1.1 Facility Inspection

Facility inspections at the laboratory are performed on a quarterly basis. The items covered in the facility inspection were the equipment, sample and standards storage, and documentation. The facility was found to be adequately maintained. The equipment necessary for the operation of the facility was available and in operational condition. Calibration and maintenance of the equipment were documented in instrumental log books and were found to be current. No systematic problems were seen with the facility or with the equipment and the associated documentation for the equipment.

7.5.1.2 Analytical Task

The system audit of the analytical task was conducted in August, 1993. The areas inspected during the audit were personnel qualifications, sample control, sample preparation techniques (on samples similar to those to be analyzed for this work), and Standard Operating Procedures. A detailed listing of items checked during a system audit is presented in Table 7-12. A separate inspection of the homogenization technique used for the paint chips was conducted during the initial testing in April 1993. No systematic problems were observed during this audit.

7.5.2 Performance Audit

Three Performance Evaluation Samples (PESs) were prepared for each sample preparation batch. The PESs were prepared by the project sample custodian using a National Institute of Standards and Technology (NIST) Standard Reference Material (SRM) and two

American Industrial Hygiene Association (AIHA) materials as discussed in section 3.2.2. The lead levels were determined through round robin testing performed under the Environmental

Table 7-12. Table of Items Checked During the Laboratory System Audit.

Category	Items Checked
Personnel	Qualifications Safety—Chemical Hygiene Plan Training
Facilities	Adequacy Housekeeping Maintenance Safety Security
Equipment	Adequacy Maintenance Safety Security Standard Operating Procedures
Sample Control	Personnel Equipment Facilities Standard Operating Procedures
Sample Preparation	Personnel Equipment Facilities Standard Operating Procedures
Instrumental Measurement	Personnel Equipment Facilities Standard Operating Procedures
Data Collection—Validation and Verification	Personnel Equipment Facilities Standard Operating Procedures
Standard Operating Procedures	Corporate Department Section Project Specific
Documentation	Personnel—Qualifications and Training Facilities SOPs Equipment SOPs Data—Samples, Standards, and Quality Control Collection Validation and Verification Archival
Sample Handling as Defined by the Quality Assurance Project and Work Plans	Collection Preparation Analysis Storage Disposal

Lead Proficiency Analytical Testing (ELPAT) Program. The NIST SRM No. 1579a containing 11.995% lead was used as a high, lead-level reference material. The same ELPAT material was used for the two PESs in each batch. Four different ELPAT lead concentrations used during the study were 0.2007%, 0.3809%, 3.218%, and 9.5536%.

The results of the blind samples are reported in section 4.4.2.2 Figures 4-30, 4-31, and 4-32. Results, in terms of the data quality objectives, are discussed in section 2.2.4. One control situation with the NIST SRM, which did not meet the accuracy objectives at the start of the Philadelphia sample analyses, was found during a data audit and is discussed in section 7.5.3.

7.5.3 Data Audit

The data audit is a qualitative and a quantitative evaluation of the documentation and procedures associated with the measurements to verify that the resulting data are of known and acceptable quality. The analytical data were audited using the criteria given in Table 2-18 for the instrumental quality control and Table 2-16 for the method performance. Both tables can be found in the design elaboration section of the report, section 3.3.2. Selected data in 100% of the instrumental measurement batches were audited. The instrumental measurement batches consisted of one or more sample preparation batches. Analyses within each instrumental measurement batch were randomly selected following the *American National Standard Sampling Procedures by Attributes, MIL-STD-105-D*. These sampling procedures specify the number of analyses in each instrumental measurement batch that must be sampled to guarantee that the fraction of unacceptable analyses in the batch is less than 5% at the 95% confidence level. Acceptability of an analysis is defined as per data and measurement quality objectives defined in the study plans. Randomly selected analytical results from each instrumental measurement batch were followed through the analytical process to evaluate the data generation and reporting system for systematic problems and to follow the audit trail. This procedure starts with the reported data and back calculates this number to the original raw data output (instrument response). Then the sample is tracked through each step of the analytical process, as documented in the notebook and other analytical records, to the instrumental measurement. The raw data and documentation for the

analysis process includes but is not limited to the weighing records, the laboratory notebook entries, instrumental output, and summary tables containing final calculated data.

Of the 3,765 ICP measurements in this study, approximately 18% of the analytical results in 26 instrumental measurement batches were audited. The audits found that the data folder format provided a systematic means for ensuring a complete audit trail. The audit of the analytical results found no systematic problems in sample preparation, instrumental measurement, or generation of the analytical data.

The data audit for the full study data included a 100% check of data for 270 randomly selected paint sample results from the full study. From this check, a total of 9 random errors were detected and corrected prior to releasing the data for further statistical analysis testing.

Only one systematic error was found during the data auditing process, as noted in section 7.5.2, where the low recoveries resulted in a control situation in specific sample preparation batches for the NIST blind PESs that indicated a systemic problem. The change in control posture for these sample preparation batches was investigated, resulting in no explanation. To evaluate if the low recoveries had an effect on the field sample results in these sample preparation batches, experiments were performed as discussed in sections 4.4.2.2.1 and 4.4.2.2.2. The statistical evaluation of the results from these experiments indicated that the results from the questionable sample preparation batches are consistent with those of acceptable sample preparation batches, therefore, a systematic problem is unlikely.

7.5.4 Results of EPA Audits

Several audits were conducted on study activities both in the field and the laboratory by the EPA work assignment manager. These audits included the evaluation of the field training conducted for the test kit operators and the performance of field test kit supervisors overseeing the operators. A system audit on laboratory operations was conducted during the initial analysis of sample preparation and instrumental measurement batches associated with the full study. Data audits conducted included the evaluation of laboratory data values associated with 75 final

sample results. These audits included the review of over 700 values tracing the raw sample data and the associated quality control sample values affiliated with the final results through the system to the final reported data. No errors were revealed in the data audits.

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9. GLOSSARY

The terms presented in this glossary are defined specifically for this report and are not intended as universal definitions for these terms across other projects or work. Defined terms that appear within definitions for other terms are presented in italics.

area units	Expression of the lead level as the mass of lead in the specimen divided by the area of the specimen. The units were reported in milligrams per centimeter squared (mg/cm^2).
bare substrate	A building component targeted for testing that had been scraped clean of paint.
baseline probability (rate) of positives, negatives	For a lead testing method, the ***baseline probability (rate) of positives*** is the probability of a ***positive result*** in the absence of lead in paint. The ***baseline probability (rate) of negatives*** is the probability of a ***negative result*** in the presence of maximal lead content.
batching	See ***sample batching***.
beginning control block reading	A quality control ***XRF reading*** performed on a ***control block*** at the beginning of a testing day.
bias	The systematic tendency of a measurement process to either underestimate or overestimate the quantity of interest. ***Bias*** and ***variability*** together describe the accuracy and precision of the process, respectively.
bias correction	A method for removing the ***bias*** present in an ***XRF measurement***. The three methods considered in the study were ***control correction***, ***full correction***, and ***red NIST SRM averaged correction***.
blind reference	

material

A paint sample originating from the National Institute of Standards and Technology (NIST) that has a precisely known level of lead. These samples were submitted for analysis along with other ***field samples*** in a manner blind to the laboratory personnel performing the analysis.

blind performance material

A paint sample originating from the American Industrial Hygiene Association (AIHA) that has a level of lead determined from round robin analysis. These samples were submitted for analysis along with other ***field samples*** in a manner blind to the laboratory personnel performing the analysis.

bootstrap

A statistical technique for estimating the ***bias***, ***standard error***, or other attributes of an estimator, which entails randomly recycling the sample data through computer simulation.

classification

Designation of a specimen as having a high or low lead level relative to a fixed criterion, such as the 1.0 mg/cm^2 federal standard; or, in the case of ***test kits***, the visual observation of a colored chemical reaction indicating the presence of lead. The ***classification*** is ***negative*** (***positive***) if a low (high) lead level is indicated.

chemical test kit

see test kit

clock time

The total time that elapsed during the observation of a single ***XRF reading***.

confidence interval

An estimated range of values in which the quantity of interest lies. A 95% ***confidence interval*** covers the quantity of interest 95 percent of the time.

continuing control block readings	A quality ***control XRF reading*** made on a ***control block*** covered by a ***NIST SRM film*** during the testing day whenever a substrate change occurred.
control block readings	Any of the quality control ***XRF readings*** made on a ***control block*** either covered or not covered by ***NIST SRM films***. Readings may be taken 1) at the beginning of the day, 2) at the end of the day, or 3) whenever a substrate change occurred.
control average reading	The average of a set of ***control block readings***, made by a specific ***XRF instrument*** on a specific ***substrate type*** within a ***dwelling***.
control block	A sample designed for the purpose of XRF quality control, constructed from one of the following commonly encountered ***substrate*** materials: brick, concrete, drywall, metal, plaster, and wood.
control correction	***Bias correction*** of an ***XRF measurement***, obtained by subtracting from it the ***control average reading*** computed for its common ***substrate*** and ***dwelling*** minus the lead level of the ***red NIST SRM film*** (1.02 mg/cm^2).
control XRF data	***XRF readings*** made on ***control blocks*** for the purpose of quality control.
dead time	The time that elapsed in making a single ***XRF reading*** when the detector was not actively accumulating X-rays for producing a lead result.
detection limit	A modification of the ***instrumental detection limit (IDL)***, specific to a paint-chip sample, that accounts for sample preparation parameters involving dilution, mass, and

	collected sample area.
digestion	See ***hot-plate digestion***.
digestion blank	See ***method blank***.
DL	See ***detection limit***.
dwelling	A ***unit*** or pair of ***units*** targeted for testing.
ending control block reading	A quality control ***XRF reading*** performed on a ***control block*** at the end of a testing day.
endpoint effect	In ***nonparametric*** estimation of a ***response function***, the phenomenon whereby the function exhibits greater ***variability*** and/or ***bias*** near an upper or lower endpoint of its domain.
enhanced logistic regression model	The ***model*** that was used to estimate the ***operating characteristic (OC) curves*** of the ***test kits***.
false negative	An erroneous ***negative classification*** obtained by a lead testing method such as a ***test kit*** or an ***XRF instrument***; i.e., for which a ***positive classification*** is indicated by the ***true lead level***.
false positive	An erroneous ***positive classification*** obtained by a lead testing method such as a ***test kit*** or an ***XRF instrument***; i.e., for which a ***negative classification*** is indicated by the ***true lead level***.

field blank	A paint collection container that was taken to the field, not used to hold a paint sample, but designated to be analyzed for lead as an assessment of potential lead contamination resulting from field collection and sample transport activities.
field classification	One of two designations made for measurements made with three of the ***XRF instruments*** evaluated in the ***full study*** (MAP-3, Microlead 1, and XK-3), where two machines made readings at each ***sampling location***. The two ***field classifications*** were denoted I and II, and were used for scheduling and data identification purposes.
field duplicate	A second paint-chip sample taken at a ***sampling location*** within the same proximity as the ***primary sample***.
field sample	A paint-chip sample collected in the field. Also used as a synonym for ***sampling location***.
fifty percent point	For a lead testing method, the level of lead at which there is a 50% probability that a ***positive result*** is obtained.
first paint reading	The first of a possible series of ***paint readings*** comprising an ***XRF measurement*** at a ***standard location***.
full study	The portion of the activity described in this report that took place from July through October 1993 in Denver and Philadelphia.
full correction	***Bias correction*** of an ***XRF measurement***, obtained by subtracting from it the ***red NIST SRM average reading*** at the same ***sampling location*** minus the lead level of the ***red NIST SRM film*** (1.02 mg/cm^2).

	See ***sample homogenization***.
homogenization	A sample preparation method for paint-chip samples that used heat from hot-plates to facilitate dissolution of the sample. The batch size varied from one to approximately 40 paint-chip samples.
hot-plate digestion	
ICP or ICP-AES	A laboratory instrument, inductively coupled plasma atomic emission spectrometer, used to make lead measurements on prepared samples.
ICP instrumental error	Error in measuring the ***true lead level*** of a specimen that is attributable to the ***ICP*** instrument used for that purpose.
IDL	See ***instrumental detection limit***.
instrumental analysis QC sample	A laboratory quality control sample that contains known levels of lead and other analytes. These samples were processed during the same time period as the prepared ***field samples*** for the purpose of evaluating adequate laboratory instrument operation.
instrumental detection limit	Three (3) times the ***standard deviation*** of a minimum of 5 replicate ***ICP*** measurements obtained from a 0.1 µg/mL lead standard measured during the processing of a given ***instrumental analysis batch***. All ***ICP*** results below this limit were identified as ***non-detectable***.
instrumental analysis batch	A group of ***digested*** paint-chip samples that were analyzed together in a sequential manner following calibration of the ***ICP*** instrument. The ***batch*** size varied from one to approximately 200 ***digested*** paint-chip samples.

K-shell reading	An ***XRF reading*** that originates from emission lines that correspond to the X-ray fluorescence transitions from the K electron orbital of the lead atom.
L-shell reading	An ***XRF reading*** that originates from emission lines that correspond to the X-ray fluorescence transitions from the L electron orbital of the lead atom.
laboratory duplicate	A second portion of a single ***field sample*** prepared and analyzed for lead.
laboratory error	A source of ***measurement error*** **in ICP analysis**, arising from preparation of a sample in the laboratory prior to lead measurement by an ***ICP*** instrument and/or by the ***ICP*** instrument itself. Possible sources of ***laboratory error*** include variation in ***subsampling***, incomplete ***homogenization***, incomplete ***digestion***, handling of the digestate prior to ***ICP*** analysis, and instrumental measurement.
laboratory QC sample	See ***instrumental analysis QC sample***.
LBP	Lead-based paint or leaded paint.
live time	The time that elapsed in making a single ***XRF reading*** while the detector was actively accumulating X-rays for producing a lead result.
measurement	

error	For an instrument, laboratory or other procedure, ***measurement error*** is the measurement obtained minus the true value of the quantity of interest. An unbiased procedure is considered more accurate (precise) than another if its ***measurement errors*** have a smaller ***standard deviation (SD)***.
method blank	A ***sample preparation quality control sample*** that is processed in the same manner as field sample except that no sample is placed into the ***digestion*** vessel. These samples were placed into ***batches*** of ***field samples*** at the beginning of the sample preparation process to determine the extent of the potential lead contamination originating from laboratory handling processes.
microwave digestion	A sample preparation method for paint-chip samples that uses microwave energy to facilitate dissolution of the sample.
model	A mathematical (functional) relationship that relates a response to a measurable independent variable or set of variables.
model fit	Refers to the ability of a ***model*** to correctly predict a response from a known independent variable or set of variables.
model parameters	Mathematical elements which together comprise a ***model***.
monotone regression	A ***nonparametric*** method for estimating the ***response function*** of one variable with respect to another, that minimizes the sum of squared errors under the constraint that the estimated ***response*** be a ***non-decreasing function***.
	See ***classification***.

negative classification (result)

One of the paint film samples, SRM 2579 lead-based paint films, originating from the National Institute of Standards and Technology (NIST) that have precisely known levels of lead. The films are layers of paint with known lead content sandwiched between two layers of plastic. Two of the five films within SRM 2579 were used in the study. This included the ***red NIST SRM*** film containing lead at 1.02 mg/cm^2 and the ***yellow NIST SRM*** film containing lead at 3.53 mg/cm^2.

NIST SRM film

An ***XRF instrument*** surface exposure and x-ray data collection time that is based on a new, non-decayed, radiation source.

nominal reading time

In mathematics, a function of one variable that has the property that larger values of the variable do not result in smaller values of the function.

non-decreasing function

Refers to a lead level, as measured by ***ICP***, that is below the ***instrumental detection limit (IDL)***.

non-detectable

XRF data that were collected using measurement protocols, or under conditions, that differed from those typically used.

non-standard XRF data

Refers to statistical procedures that do not depend on the formulation of a ***model***, and that have validity over a wide range of conditions.

nonparametric

The ***monotone regression*** of a set of ***XRF readings*** with respect to ICP measurements, used as an approximation to the mean ***XRF readings*** expressed as a function of the ***true lead level***.

nonparametric response

The square root of the ***monotone regression***, with respect to ICP measurements, of the squared differences between a set of ***XRF readings*** and the ***nonparametric response***, used as an approximation to the ***standard deviation (SD)*** of ***XRF readings*** expressed as a function of the ***true lead level***.

nonparametric SD

A set of ***XRF readings*** minus the estimated ***nonparametric response***, divided by the ***nonparametric SD***. These "transformed" ***XRF readings*** exhibit little or no dependence on the lead level.

nonparametric standardized residuals

The probability of an event (e.g., a ***positive result*** with a ***test kit***) expressed as a function of the lead level.

operating characteristic (OC) curve

A data value that is unusual with respect to other data observed under apparently similar conditions. An ***outlier*** may represent erroneous data, or measurement conditions that are actually dissimilar.

outlier

A mathematical rule or procedure that is used to identify ***outliers***.

outlier criterion

See ***responsive***.

over-responsive

The average of three ***paint readings*** comprising an ***XRF measurement*** at a ***standard sampling location***.

paint average reading

Any of the ***XRF readings*** taken on the painted surface during an ***XRF measurement*** at a ***standard location***.

paint reading	Expression of the lead level as the ratio of the mass of lead in the specimen (grams) to the total mass of the specimen (grams), reported as a percentage.
percent by weight units	The portion of the activity described in this report that took place in Louisville, Kentucky in March and April 1990.
pilot study	See ***classification***.
positive classification (result)	The first paint-chip sample collected from a ***sampling location***.
primary sample	A single open-shutter XRF instrument event, including exposure of the painted surface with energy from the ***XRF instrument*** radiation source; emission of X-rays from fluorescence transitions within lead atoms residing in the painted surface; counting of the X-rays received at the detector; electronic processing of the detector signals; and displaying a lead-area value result in mg/cm^2. One lead result was produced from each ***XRF reading***.
QC	An abbreviation for quality control.
reading time	The total time that elapsed in making a single ***XRF reading***. Synonymous with ***clock time***.
real time	One of the NIST SRM 2579 lead-based paint films that contains 1.02 mg/cm^2 of lead. See ***NIST SRM film***.

red NIST SRM

The average of the ***bare substrate red NIST SRM*** film covered ***XRF readings*** taken during an ***XRF measurement*** at a specific ***standard location***. Consists of the average of the first, second, and third ***bare substrate red NIST SRM film*** covered ***XRF readings*** for a specific ***XRF instrument***.

red NIST SRM average reading

Bias correction of an ***XRF measurement***, obtained by subtracting from it the average of ***red NIST SRM average readings*** obtained at ***sampling locations*** in the same unit and of the same substrate type, minus the lead level of the ***red NIST SRM film*** (1.02 mg/cm^2).

red NIST SRM averaged correction

The average (mean) ***XRF reading*** expressed as a function of the lead level.

response, response function

An ***XRF instrument*** is ***responsive*** if a unit change in the lead level results in a unit change in the average ***XRF reading***. It is ***under-responsive*** if less, and ***over-responsive*** if more than a unit change in average ***XRF reading*** results.

responsive

An estimate of the relationship between two variables, obtained by averaging the values of one variable corresponding to the other variable taking on nearly constant values.

See ***standard deviation***.

See ***standard error***.

running mean

An abbreviation for standard reference material. See ***NIST SRM film***.

The grouping of paint-chip samples for

	simultaneous processing in a laboratory procedure.
SD	
SE	The act of grinding samples into a powder-like form to permit subsampling in a uniform and representative manner.
SRM	A quality control sample placed into a ***batch*** of samples at the beginning of the sample preparation process.
sample batching	A specific location on a painted ***substrate*** within a ***unit*** where lead testing was performed. The ***sampling location*** covered the entire ***template***.
sample homogenization	A statistical test based on the number of times an event was observed in a sample, under the hypothesis that the event occurred with a 50 percent probability each time. Observing the event many more, or many fewer times than one-half the sample size constitutes evidence against the hypothesis.
sample preparation QC sample	
sampling location	The difference in ***true lead levels*** between painted areas within the same ***template***: (1) for ***field duplicates***, the difference is between the two paint-chip samples; (2) for ***test kit*** and ***XRF instrument*** analyses, the difference is between the ***primary sample*** and where measurements were made on the painted surface.
sign test	
	XRF data that were collected using ***special readings***.
	A specified set of ***special readings*** taken at a ***special sampling location***.
spatial variation	

	An ***XRF reading*** taken with a MAP-3 ***XRF instrument*** using a ***nominal reading time*** of 60 seconds at a ***special sampling location***.
special data	A ***sampling location*** that was specifically designated to receive additional ***XRF measurements*** with a ***nominal reading time*** of 60 seconds.
special measurement	XRF data that were collected using ***special-special readings***.
special reading	A specified set of ***special-special readings*** taken at a ***special-special sampling location***.
special sampling location	An ***XRF reading*** taken with a MAP-3 ***XRF instrument*** using a ***nominal reading time*** of 240 seconds at a ***special-special sampling location***.
special-special data	A ***sampling location*** that was specifically designated to receive additional ***XRF measurements*** with a ***nominal reading time*** of 240 seconds.
special-special measurement	XRF data that were collected using ***standard readings***.
special-special reading	A measure of ***variability*** in a population (or process) from which data are obtained, quantified by the square root of the expected squared difference between the the value obtained from the population and the population mean. An ***SD*** is equal to zero if and only if the population (or process) generates the same value every time, i.e., if it does not vary.
special-special sampling location	

standard data	A measure of ***variability*** in a sample of data, quantified by the square root of the average squared difference between the sample values and the sample mean. A ***sample SD*** is equal to zero if and only if all sample data have the same value. If the sample was obtained in a manner that is representative of a population or process, the ***sample SD*** can serve as an estimator of the ***population SD***.
standard deviation (SD), of a population	A measure of ***variability*** applied to an estimator, which is the analog of the ***standard deviation (SD)*** applied to a population.
standard deviation (SD), of a sample	A specified set of ***standard readings*** taken at a ***standard sampling location***.
standard error (SE)	An ***XRF reading*** taken with a ***nominal reading time*** of 15 seconds at a ***standard sampling location***.
standard measurement	A ***sampling location*** that was designated to receive ***XRF measurements*** with a ***nominal reading time*** of 15-seconds.
standard reading	That portion of a ***homogenized*** sample that is used in the laboratory procedure for analysis with an ***ICP*** instrument.
standard sampling location	The building material that lies under the paint.
	The type of building material that lies under the paint. All ***substrate types*** in the study were classified as one of the following: brick, concrete, drywall, metal, plaster, or wood.

subsampling	A marking design used to physically mark the ***sampling locations*** within each ***dwelling***.
substrate	A set of chemicals and other supplies that are packaged together with instructions for use in making lead measurements on painted surfaces.
substrate type	A specific location on a painted ***substrate*** within a ***unit*** where lead testing is performed. The ***testing location*** covers the entire ***template***. Synonymous with ***sampling location***.
template	The probability of a ***positive result*** when the ***true lead level*** in paint is 1.0 mg/cm^2.
test kit	The actual lead level in a paint specimem or sample.
testing location	See ***responsive***.
threshold probability	An unoccupied structure that is used to house a person or family. May be all or a portion of a single structure.
true lead level	Fluctuation in data generated under similar conditions. ***Bias*** and ***variability*** together describe the accuracy and precision of a measurement process, respectively. A commonly used measure of ***variability*** is the ***standard deviation (SD)***.
under-responsive	Louisville sampling locations which were selected to take additional ***XRF readings***. Variability locations occurred immediately following a substrate change.
unit	

variability	Of a random variable or a collection of data, the ***standard deviation*** squared. Of an estimator, the ***standard error*** squared.
variability location	X-ray fluoresence A portable lead detection instrument that detects fluoresced x-rays from lead. These instruments contain a radioactive source to induce lead to emit x-rays for detection.
variance	A specified set of ***XRF readings*** taken at a sampling location.
XRF	The ***model*** that was used to describe the relationship between ***XRF readings*** and the ***true lead level*** in paint. Estimation was performed using the observable ***ICP*** measurements, taking into account the combined effect of ***spatial variation*** and ***laboratory error***.
XRF instrument	A lead measurement collected from a surface using an ***XRF instrument*** operating under a specified ***nominal reading time***.
XRF measurement	One of the NIST SRM 2579 lead-based paint films that contains 3.53 mg/cm^2 of lead. See ***NIST SRM film***.
XRF measurement model	
XRF reading	

yellow NIST SRM

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INTRODUCTION AND CLARIFICATIONS

The appendices presented in this volume contain the following three types of information:

- Protocols and procedures used for the performance of the full study, represented using single letter designations A through J.
- Protocols and procedures used for the performance of the pilot study, represented using double letter designations AA through II.
- Laboratory sample preparation experiments represented using the triple letter designation of AAA.

Some modifications to planned protocols were made during the performance of the studies. Modifications to the full study affected three appendices: Appendix B, Appendix C, and Appendix D. Modifications to full study protocols are presented immediately following each of these corresponding Appendices and are further identified by addition of an "m" to the appropriate Appendix letter designations. Modifications to pilot study protocols have been incorporated into the pilot study appendices and are differentiated from planned protocols through the use of footnotes.

The term "dried paint sample" has been converted to "paint chip sample" throughout these appendices. These two terms are synonymous and were interspersed throughout the original planned protocols used for these studies. The conversion of the two terms to a single term has been made to improve the readability of this document.

Portions of the protocols or entire protocols have not been reproduced in these Appendices because copyright and proprietary information considerations. Protocols not present in these Appendices are summarized Table A-1 below.

Table A-1. Summary of Appendix Protocol Information not present	
INFORMATION NOT PRESENT	REASON FOR DELETION
Appendix C and Appendix CC: Manufacturer printed test procedures only	Copyright considerations
Appendix I and Appendix HH	Proprietary information
Appendix J and Appendix II	Proprietary information

SUMMARY OF PROTOCOL DIFFERENCES BETWEEN THE PILOT AND FULL STUDIES

Some protocol changes were made between the pilot and full studies. These changes are discussed in detail in the design section of Volume II. A summary of these changes are presented in Table A-2 below.

TABLE A-2. SUMMARY OF DIFFERENCES BETWEEN PROTOCOLS FOR PILOT AND FULL STUDIES.

PROTOCOL	APPENDIX LETTER		DESCRIPTION OF PRIMARY DIFFERENCES
	FULL STUDY	PILOT STUDY	
Selection of Sampling Locations	A	AA	**!** Some changes in the format of the sampling template were made between the full and pilot studies. See Figures 1-1 and 1-2 in Volume II of this report. **!** For the full study, the substrate order for XRF testing was metal, wood, brick, drywall, concrete, and plaster. For the pilot, the substrate order for XRF testing was wood, drywall, plaster, concrete, and metal. **!** For the full study, the starting substrate during XRF testing was varied among units and fixed for a specific unit for all instruments. For the pilot, the starting substrate during XRF testing was the same among all units and for all instruments. **!** For the full study, relative positions on a given sampling location tested by a given test kit operator was randomized by assignment of specific test kit to specific positions. For the pilot, relative testing positions were indirectly determined by staggering the starting sequence. **!** For full study, prefix "S" barcode labels were used to identify field duplicate samples. For the pilot, a "DUP" suffix was added to the sample ID to identify field duplicate samples. **!** For full study, a "Sample Locations Data Form" was used to identify testing areas. For the pilot, a marked up drawing of a floor-plan was used to identify testing areas.

TABLE A-2. SUMMARY OF DIFFERENCES BETWEEN PROTOCOLS FOR PILOT AND FULL STUDIES.

PROTOCOL	APPENDIX LETTER		DESCRIPTION OF PRIMARY DIFFERENCES
	FULL STUDY	PILOT STUDY	
XRF Testing	B	BB	**!** Some changes in reading times and replicates were made between the full and pilot studies. See Tables 4-5 and 4-6 in Volume II of this report. **!** For full study, control block measurements were made using both the red (3.53 mg/cm²) and yellow (1.02 mg/cm²) NIST standard films. For the pilot, only the concrete control block was measured using both red and yellow NIST standard films. **!** For full study, NIST standard film covered substrate measurements were made at all sampling locations using the yellow (1.02 mg/cm²) film. For the pilot, additional NIST standard film covered substrate measurements were made at the concrete sampling locations using the red (3.53 mg/cm²) film. **!** For the full study, measurements at special sample locations and on control blocks included bare substrate testing (no NIST films). For the pilot, bare substrate testing was not performed. **!** For the full study, QC variability checks were not performed. For the pilot, these checks were performed. **!** For the full study, special measurements were performed on days separate from standard measurements. For the pilot, special measurements were performed during standard measurement days. **!** For the full study using the ML-1, performance of replicate measurements using a single trigger pull was formalized into the protocol. For the pilot, the same procedure for the ML-1 was used. However, it was not formalized within the written protocols. **!** For the full study, "coverage" and "density" values were collected for the XL and ML-1 instruments respectively as presented in Note 2. For the pilot, specific instructions for collection of this information was not provided. However, some density values were collected for the ML-1s. **!** Field data recording forms were changed to reflect changes in protocols between the full and pilot studies.

TABLE A-2. SUMMARY OF DIFFERENCES BETWEEN PROTOCOLS FOR PILOT AND FULL STUDIES.

PROTOCOL SUMMARY	APPENDIX LETTER		DESCRIPTION OF PRIMARY DIFFERENCES
	FULL STUDY	PILOT STUDY	
Test Kits Testing	C	CC	**!** For the full study using the Lead Detective kit, a cotton swab was used to deliver reagent to the testing surface. For the pilot, a plastic stirring rod or toothpick was used for delivery of reagent to the testing surface. **!** For the full study using the Lead Detective kit, the paint chip removed during notching of the test surface was retained an used for further testing if testing results were negative or doubtful. For the pilot, this procedure was not included in the protocols. **!** For the full study using the Lead Alert kit (labeled "F"), paint was exposed using a sanding method and testing was performed on the resulting dust. For the pilot, paint was exposed using a notch method and testing was performed on the exposed paint layers. **!** For the full study using the Lead Alert kit (labeled "F") and the Lead Alert All-in-One kit (labeled "B"), the indicator mixing time was reduced from that used in the pilot to reflect changes in kit instruction sets supplied from the manufacturer. **!** For the full study using the Lead Alert kit (labeled "F") and the Lead Alert All-in-One kit (labeled "B"), one drop of each reagent was used during QC checks during the full study. For the pilot two drops of each reagent were used during QC checks. **!** For the full study using the Lead Alert All-in-One kit (labeled "B"), the importance of cleaning the coring tool between samples was formalized into the protocols. For the pilot, importance of cleaning the coring tool was emphasized during training. **!** Field data recording forms were changed between the full and pilot studies. Full study forms were simplified by removing information blocks that were not needed. In addition, shaded blocks were added to full study forms for use in reporting shading from gray to black for kits that used sodium sulfide.

TABLE A-2. SUMMARY OF DIFFERENCES BETWEEN PROTOCOLS FOR PILOT AND FULL STUDIES.

PROTOCOL SUMMARY	APPENDIX LETTER		DESCRIPTION OF PRIMARY DIFFERENCES
	FULL STUDY	PILOT STUDY	
Collection of Paint Chip Samples	D	DD	! Full study protocols contained more detailed descriptions on paint removal methods than that contained in the pilot protocols. ! Field data recording forms were changed between the full and pilot studies. Full study forms were simplified by removing information blocks that were not needed.
Weighing, Homogenization and Preparation of Paint Chip Samples	E	EE and FF	! For full study, determination of total collected sample weight preceded sample homogenization. For the pilot, sample homogenization preceded determination of total collected sample weight.
ICP-AES Analysis of Prepared Paint Chip Samples	F	GG	! There were no differences between full and pilot studies.
Packaging and Shipping of Paint Chip Samples	G	HH	! There were no differences between full and pilot studies.
Glassware Cleaning	H	II	! There were no differences between full and pilot studies.
Acid Bath Cleaning	I	JJ	! There were no differences between full and pilot studies.

APPENDIX A

FULL STUDY PROTOCOLS:
SELECTION OF MEASUREMENT AND SAMPLING LOCATIONS

SELECTION OF MEASUREMENT AND SAMPLING LOCATIONS

1.0 SUMMARY

Selection of interior and exterior sampling sites will be made from as many painted substrate types as can be found in the test structure (metal, wood, brick, drywall, concrete, and plaster).

The Field Team Leader (field statistician, provided by David C. Cox & Associates) will be responsible for all selection and marking of measurement and sampling locations. The DCC&A Field Team Leader will also assist the MRI supervisor during the course of the field sampling efforts.

The Field Team Leader will be responsible for attaching the correct bar-code sets to each location. The bar codes will be removed by the various samplers and applied to the individual's test results data form at the time the test is performed.

The Team Leader will numerically order the sampling locations so that all locations with the same substrate material will be tested sequentially by the XRF instruments. The order in which the substrates are tested will be: metal, wood, brick, drywall, concrete, and plaster. This ensures the maximum number of transitions between light and dense materials in order to best simulate transitions that are likely to be encountered under testing more commonly to be encountered during routine LBP investigations. For each unit, the starting substrate for XRF testing will be fixed. This starting point will be determined on a random or judgmental basis. The order of XRF testing for beginning and end-of-day control block measurements will be the same for all units (metal, wood, brick, drywall, concrete, and plaster).

Test kit operators will not follow the same testing order as the XRFs and will be instructed to test all locations within a room. The relative position on a given location tested by a given test kit operator will be randomized by assignment of specific test kits to specific positions.

2.0 DETAILED MARKING PROCEDURE

1. Obtain or create a rough floor plan of the targeted

structure.

2. Perform a review of available Lead testing data and summarize to aid in selection of sampling locations. This is anticipated to be performed prior to on-site selection and marking activities.

3. Perform a walk through of each unit as an aid to selecting locations. Make notes on a copy of the floor plan as needed for later marking of locations.

4. Perform location selections. For each location selected, mark the sampling locations using an indelible marking pen and attach bar code labels as follows:

   a. Draw an outline of the testing location, which includes separate boxes for XRF testing, paint chip sample collection, and test kit measurements. A typical testing location will be a rectangle approximately 4 in high by 14 in long. The rectangle will include two squares approximately 4 in by 4 in the left and center portions of the rectangle, and six smaller rectangles 4 in high by 1 in wide in the right portion of the rectangle. In addition, for those locations targeted for a side-by-side samples, an approximately 2 in x 2 in square will be drawn at one end of the rectangle. For components where a 4 in x 14 in rectangle cannot be obtained, the field statistician will exercise judgment in defining a comparable sampling area. There will be six smaller rectangles for test kits and five test kits in the study. The sixth rectangle will be used for contingencies.

   b. Divide the middle large squares into four individual 2 in x 2 in squares using the marking pen. Indicate with an arrow pointing to one of the small squares that portion to be sampled by the paint chip collectors for a regular paint chip sample. For those locations targeted for a side-by-side sample, the independent approximately 2 in x 2 in square drawn at one end of the rectangle will be used for collection of this extra paint

chip sample.

c. Mark the six rectangles approximately 4 in high by 1 in wide with the codes A-E to designate test kit position assignments (the supervisor will assign each test kit with a letter to follow for determining testing position).

d. Attach a resealable plastic bag containing a minimum of 30 bar code labels matching the location ID number in the vicinity of the marked area. Use duct tape and a staple gun (if needed) to properly secure the bag. If the location is an exterior location, then attach the bag to inside the nearest inside area and write a note describing the placement of the bag next to the marked testing location.

   For locations that are targeted for side-by-side paint samples, attach a second resealable plastic bag containing a minimum of 12 bar code labels matching the location ID number combined with a preceding "S" in the vicinity of the marked area. Attach these in the vicinity of the first bag of bar code labels containing location ID numbers.

e. Mark, in large print, the location number in two places around the location area.

f. Mark the location with any other needed indicator, such as "SPECIAL," "SPECIAL-SPECIAL," or "ARCHIVE" to indicate additional testing requirements to this location.

g. On the floor plan, indicate the position of the sampling location (and its number).

5. After completing the location marking activity, review each location and compile a comprehensive list of all locations within the unit.

6. Make copies of floor plans with identified locations and the comprehensive listing for all supervisors and testers.

3.0 NUMBERING SYSTEM

Two sets of bar-code numbers will be used. The first type is for use on XRF data forms, test kit data forms, and regular paint chip sample containers and data forms. The second type is for use on side-by-side paint chip sample containers and data forms. Both sets will contain the same five digit numbers starting with 80001. The second set will differ from the first by inclusion of a preceding "S." Each unit will be assigned a range of 100 numbers for marking locations.
Bar code labels with identical ID numbers will be pre-loaded into plastic bags and sorted into folders prior to shipment to the field. Sorting bar codes into separate folders and placing them in consecutive order will ease marking activities.

Sample Locations Data Form

APPENDIX B

FULL STUDY PROTOCOLS:
MEASUREMENT PROTOCOLS FOR XRF TESTING

MEASUREMENT PROTOCOLS FOR XRF TESTING

1.0 SUMMARY

NOTE: READ ENTIRE APPENDIX B BEFORE DOING ANY WORK!!

This document describes the standard protocol for collecting XRF measurement data on painted surfaces and corresponding substrate surfaces. This document also includes instructions for recording the measurements and making QC checks for XRF instruments participating in this study.

In general, XRF operators will be requested to make measurements according to their manufacturers' general operating procedures. In situations where this study protocol (contained in this Appendix) differs dramatically from the manufacturers' protocol, or when this study protocol cannot be followed because of operational limitations, the XRF operator is required to discuss the situation with the acting MRI field supervisor to resolve the problems. It is the responsibility of the XRF monitor to record as much information as possible about the operation of a given XRF instrument during this full field study.

Any deviations from this protocol must be agreed to by the acting MRI field supervisor and fully documented **before** implementing the deviation. In any case, each XRF must be operated in a consistent manner throughout this study.

2.0 MATERIALS AND EQUIPMENT

- Portable field XRF instrument with any extra required supporting equipment. (To be provided by XRF contractor.)

- One set of NIST paint films for each XRF instrument (SRM 2579); contains five films of different Lead levels. (To be provided by XRF contractor.)

- Dosimeter badges; one for each XRF operator and one for each individual working within the same unit where XRF testing takes place. (Operator badges will be provided by XRF contractor, badges for monitors and supervisors hired by DCC&A will be provided by DCC&A, and badges

for MRI personnel will be provided by MRI).

- Reporting forms; see exemplars in this protocol (to be provided by MRI.)

- Adhesive labels or bar-code labels for identifying samples. (To be provided by MRI; will be available at each sampling location.)

- Waterproof (indelible) permanent marking pen. (To be provided by MRI; will be available at site.)

- Watch, clock, or other equivalent timepiece. (Each team member in the field will be required to have a timepiece for reporting the sampling times on the data forms.)

- Device(s) to measure temperature and relative humidity. (To be provided by MRI; will be available at site and operated by the acting MRI field supervisor or designee at a frequency deemed necessary to gather supplemental information during testing activities).

- Pre-moistened wipes for cleaning of tools, hands, etc. (To be provided by MRI; will be available at site.)

- QC test blocks, each approximately 4 in x 4 in. The thicknesses given are approximate: $^3/_4$ in wood (pine), 2 in concrete (with aggregate), $^1/_2$ in sheet rock, 20 to 25 gauge metal, and 1 in plaster. A full set of labelled QC test blocks will be prepared by MRI and placed in unit.

- One 12-in thick Styrofoam block for supporting QC test blocks under measurement. (To be provided and labelled by MRI; will be available at site.)

3.0 MEASUREMENT PROCEDURES

AN ORDERED LIST OF MEASUREMENTS SPECIFIC FOR EACH UNIT WILL BE PROVIDED BY THE SUPERVISOR FOR EACH UNIT. TESTERS MUST FOLLOW THAT ORDER EXACTLY.

The starting point for each unit will be based on a specific

substrate type (metal, wood, brick, sheetrock, concrete, or plaster). Units will be assigned different substrates for initiation of testing, but testing will always follow the same substrate order. The substrate starting point will be fixed for a given unit and will be indicated on the testing order instructions provided by the supervisor.

The general order of testing is metal → wood → brick → sheetrock → concrete → plaster. An example of the general work plan, testing order, and measurements for a unit targeted to beginning with brick is given below:

1. Receive beginning-of-day instructions from MRI field supervisor

2. Perform initial manufacturer's calibration checks

3. Perform additional manufacturer's calibration checks at intervals as required by the manufacturer's specifications

4. Perform beginning-of-day control block measurements (all six blocks).

5a. Perform continuing drift check on <u>brick</u> control block.

5b. Perform measurements on all <u>brick</u> substrates, including standard measurements and any required special location measurements.

5c. Perform continuing drift check on <u>brick</u> control block.

6a. Perform continuing drift check on <u>sheetrock</u> control block.

6b. Perform measurements on all <u>sheetrock</u> substrates, including standard measurements and any required special location measurements.

6c. Perform continuing drift check on <u>sheetrock</u> control block.

7a. Perform continuing drift check on <u>concrete</u> control block.

7b. Perform measurements on all <u>concrete</u> substrates, including standard measurements and any required special location measurements.

7c. Perform continuing drift check on concrete control block.

8a. Perform continuing drift check on plaster control block.

8b. Perform measurements on all plaster substrates, including standard measurements and any required special location measurements.

8c. Perform continuing drift check on plaster control block.

9a. Perform continuing drift check on metal control block.

9b. Perform measurements on all metal substrates, including standard measurements and any required special location measurements.

9c. Perform continuing drift check on metal control block.

10a. Perform continuing drift check on wood control block.

10b. Perform measurements on all wood substrates, including standard measurements and any required special location measurements.

10c. Perform continuing drift check on wood control block.

11. Perform end-of-day control block measurements (all six blocks).

12. Review data forms for completeness and transfer all data forms to the MRI field supervisor. Receive end-of-day instructions from MRI field supervisor.

3.1 BEGINNING OF ALL XRF TESTING AT A SITE PROCEDURE

XRF operators and data monitors will receive detailed overview instructions from the acting MRI field supervisor on the first XRF testing day that will include the following topics:

- General safety instructions
- Definitions: housing units, testing locations, measurements, sampling time

- Specific site issues and description of marked locations and what markings signify

- Use of testing location listings and order of performing measurements

- Use of each data form and placement of bar codes and other data on forms

- Responsibilities of XRF operators to call out all readings real-time

- Responsibilities of monitors to record **all** data real-time and use verbal feedback to verify data value. (No reading is to be discarded; however more data can be taken if insisted on by the XRF operator.)

- Completion of the "XRF Instrument Information" form (See exemplar p. B-13)

- Responsibilities of monitors to periodically observe the actual instrument readout (particularly for recording both K and L shell data).

- Definition of 15-sec reading: 15 sec is based on a **new** radiation source. XRF operators will be instructed to compensate for source age as needed to give radiation flux equivalent to a 15-sec exposure with a new source.

3.2 BEGINNING OF EACH DAY ON-SITE PROCEDURES

The XRF operator and data monitor will receive initial instructions from the acting MRI field supervisor at the beginning of each testing day. Items will generally include a brief overview of those listed under Section 3.1. plus any additional items that are dictated by variable field conditions.

Two types of XRF testing days will be performed: a "standard" measurement day and a "special" measurement day. All XRF instruments will perform the "standard" measurements day of testing. Only the Scitec instruments will perform the "special" measurements day (in addition to the "standard" measurements day).

At the beginning of each type of measurements day at a given unit, the XRF operator will perform tests and instrument checks that are required by the manufacturer of the XRF to prepare the instrument for taking Lead measurements. The XRF operator must inform the data monitor that a manufacturer-recommended procedure is being performed, and the name and nature of the procedure. The data monitor will record the time and nature of all such manufacturer-recommended procedures in the "Comments" column of the "Control Blocks" form.

The XRF operator will perform XRF "standard" measurements as follows:

- BEGINNING-OF-DAY control block measurements, as described in Section 3.3.

- STANDARD location measurements in the order listed on unit list received from acting MRI supervisor. STANDARD measurements are described in Section 3.4.

- CONTINUING DRIFT CHECK substrate transition measurements each time the substrate changes, as described in Section 3.6.

- END-OF-DAY control block measurements as described in Section 3.3.

The Scitec XRF operators will perform XRF "special" measurements as follows:

- BEGINNING-OF-DAY control block measurements as described in Section 3.3.

- SPECIAL location measurements in the order listed on unit list received from the acting MRI supervisor. SPECIAL measurements are described in Section 3.5.

- END-OF-DAY control block measurements as described in Section 3.3.

NOTE: No continuing drift checks are preformed during "special" day testing.

3.3 CONTROL BLOCK MEASUREMENTS—BEGINNING- AND END-OF-DAY

At the beginning and end of each day, each XRF operator will perform a set of measurements on six control blocks with two of the NIST SRM 2579 standards (red, 1.02 mg/cm^2; and yellow, 3.53 mg/cm^2) and with no NIST standard. These calibration checks will be carried out for the XRF instruments using sets of six substrate blocks (metal, wood, brick, drywall, concrete, and plaster). One set of these blocks will be placed in each unit for use by the testers making measurements in that unit. Before the start of testing in each unit, one measurement will be taken on each block with each of the three NIST films designated above. Data from these beginning and end drift check measurements will be recorded on the "XRF QC DATA: CONTROL BLOCKS" form. A step-by-step description is provided below:

At the beginning of each testing day, before starting testing in any unit perform the following procedures:

1. For each new "XRF QC DATA: CONTROL BLOCKS" form needed, complete the header of the form (see exemplar p. B-14).

2. Fill in the columns for each control block measurement taken and "time of measurement," "test block type," and "NIST std film used."

3. Perform whatever normal instrument checks are required by the XRF manufacturer to prepare the instrument for taking Lead measurements. Inform the data monitor what the procedure is and why it is being done. The data monitor will write this information in the "Comments" column on the "XRF QC DATA: CONTROL BLOCKS" form.

4. Perform NIST/test block measurements for two NIST films and the base control block. Place the test blocks, one at a time, in the center of the Styrofoam support block. For the two measurements requiring the NIST standard films, center the appropriate NIST film on the test block and place the XRF probe to take readings through the NIST film into the center of the control block. Perform the measurements in the following order.

   a. <u>Metal</u>—Center the metal test block on the Styrofoam support. Center the yellow NIST film on the metal test block and perform one measurement (three nominal 15-sec read cycles). Then perform one measurement using the red NIST film, followed by the bare control block.

Call out the value after each reading. The monitor will write each read cycle value on the "XRF QC DATA—CONTROL BLOCKS" form, verbally verifying the value written. The monitor will record other information in the "Comments" column.

b. Wood—Center the wood test block on the Styrofoam support. Center the yellow NIST film on the wood test block and perform one measurement (three nominal 15-sec read cycles). Then perform one measurement using the red NIST film, followed by the bare control block. Call out the value after each reading. The monitor will write each read cycle value on the "XRF QC DATA—CONTROL BLOCKS" form, verbally verifying the value written. The monitor will record other information in the "Comments" column.

c. Brick—Center the brick test block on the Styrofoam support. Center the yellow NIST film on the brick test block and perform one measurement (three nominal 15-sec read cycles). Then perform one measurement using the red NIST film, followed by the bare control block. Call out the value after each reading. The monitor will write each read cycle value on the "XRF QC DATA—CONTROL BLOCKS" form, verbally verifying the value written. The monitor will record other information in the "Comments" column.

d. Sheetrock—Center the sheetrock test block on the Styrofoam support. Center the yellow NIST film on the sheetrock test block and perform one measurement (three nominal 15-sec read cycles). Then perform one measurement using the red NIST film, followed by the bare control block. Call out the value after each reading. The monitor will write each read cycle value on the "XRF QC DATA—CONTROL BLOCKS" form, verbally verifying the value written. The monitor will record other information in the "Comments" column.

e. Concrete—Center the concrete test block on the Styrofoam support. Center the yellow NIST film on the concrete test block and perform one measurement (three nominal 15-sec read cycles). Then perform one measurement using the red NIST film, followed by the bare control block. Call out the value after each

reading. The monitor will write each read cycle value on the "XRF QC DATA - CONTROL BLOCKS" form, verbally verifying the value written. The monitor will record other information in the "Comments" column.

f. Plaster—Center the plaster test block on the Styrofoam support. Center the yellow NIST film on the plaster test block and perform one measurement (three nominal 15-sec read cycles). Then perform one measurement using the red NIST film, followed by the bare control block. Call out the value after each reading. The monitor will write each read cycle value on the "XRF QC DATA—CONTROL BLOCKS" form, verbally verifying the value written. The monitor will record other information in the "Comments" column.

At the end of the testing day (regardless of whether all locations in a given unit were completed) perform all of the above control block measurements exactly as they were performed at the beginning of the day.

3.4 PROCEDURE FOR STANDARD MEASUREMENTS AT EACH SAMPLING LOCATION

For each instrument, one standard measurement will consist of three consecutive nominal 15-sec readings on the paint followed by three consecutive nominal 15-sec readings on the bare substrate covered with the red NIST standard (1.02 mg/cm^2). At each sampling location perform the following steps:

1. For each new "XRF TEST DATA - STANDARD MEASUREMENTS" form needed, complete the header of the form (see exemplar p. B-15).

2. Affix the sampling location/identification bar code in the correct box on the "XRF TEST DATA - STANDARD MEASUREMENTS." These bar code labels should be present in close proximity to the sampling location marked by the field team leader (see Note 1). If a bar code label is not available, write in the sampling location number written at the location.

   NOTE 1: The sampling location will be marked in advance by the field team leader using a dark colored marking pen. The marking will be in the form of squares

and rectangles with letters. The painted surface location to be used for XRF measurements will be the largest painted square, approximately 4 in x 4 in. The exposed substrate surface location to be used for XRF measurements will be the largest exposed area present at the sampling location.

3. Perform the normal instrument checks required by the manufacturer of the XRF to prepare the instrument for taking Lead measurements. Inform the data monitor what the procedure is and why it is being done. The data monitor will write this information in the "Comments" column.
4. Perform measurements on the painted and exposed surfaces as follows (See Note 2):

   a. Perform a "measurement" (three nominal 15-sec readings) on the painted surface at the sampling location. Call out the value after each reading. The monitor will write each read cycle value on the "XRF TEST DATA - STANDARD MEASUREMENTS," verbally verifying the value written. The monitor will record other information in the "Comments" column.

   b. Perform a "measurement" (three nominal 15-sec read cycles) on the exposed substrate surface covered with the 1.02 mg/cm^2 NIST standard film (red) at the sampling location (see Note 3).

   c. If the location is market as a "special" location, then perform a "measurement" (three nominal 15-sec read cycles) on the exposed substrate surface (bare, with NO NIST film). Call out the value after each reading. The monitor will write each read cycle value on the "XRF TEST DATA - STANDARD MEASUREMENTS," verbally verifying the value written. The monitor will record other information in the "Comments" column. DO NOT DO THIS STEP FOR THE **Scitec**.

NOTE 2: For the ML-1, the three readings will be obtained with a single pull of the instrument's trigger. The readout corresponding to each "beep" of the instrument will be recorded. For other XRF instruments that can take multiple read cycles using a single trigger pull event, perform replicate read cycles in this manner recording

each transient read cycle value. In addition, any special operations performed during measurement (such as use of a reset button for PGT) must be noted in the "Comments" column of data forms. For the NITON XRF, record the "coverage" value in the "Comments" column. The "density" value for the ML-1 will be recorded in the "Comments" column.

NOTE 3: If difficulties are encountered holding the NIST film against the substrate surface, try using a small piece of masking tape to hold it in place. Be sure the tape is placed such that it adheres only to areas outside the marked location.

3.5 PROCEDURE FOR SPECIAL MEASUREMENTS

Two additional sets of measurements, called "Special" and "Special-Special" measurements, will be carried out at selected sampling locations for each substrate. The special measurements will be used to test alternative protocols for the instruments on a case-by-case basis. "Special" measurements are in addition to standard measurements and are only being performed by the Scitec XRF instruments on a separate testing day.

Procedure for performing a "Special" measurement is as follows:

1. For each new "XRF TEST DATA - SPECIAL MEASUREMENTS" form needed, complete the header of the form. (See exemplar p. B-16)

2. Affix the sampling location/identification bar code in the correct box on the "XRF TEST DATA - SPECIAL MEASUREMENTS." These bar code labels should be present in close proximity to the sampling location marked by the field team leader (see Note 1). If a bar code label is not available, write in the sampling location number written at the location.

NOTE 1: The "SPECIAL" AND "SPECIAL-SPECIAL" sampling location will be marked in advance by the field team leader using a dark colored marking pen. The words "SPECIAL" AND "SPECIAL-SPECIAL" will be marked on these locations to signify the testing required.

3. Perform the normal instrument checks required by the manufacturer of the XRF to prepare the instrument for taking Lead measurements. Inform the data monitor what the procedure is and why it is being done. The data monitor will write this information in the "Comments" column of the form.

4. Perform SPECIAL measurements on the painted and exposed surfaces as follows (See Note 2):

   a. The Scitec MAP will perform one nominal 60-sec reading (Test Mode) on the painted surface (this corresponds to the TEST mode of the Scitec). Call out the value after reading. The monitor will write the read cycle value on the "XRF SPECIAL LOCATIONS DATA FORM," verbally verifying the value written. The monitor will record other information in the "Comments" column. The XRF Monitor will record the nominal time of 60 sec in the "Approx. Sampling Time (Sec.)" column.

   b. Perform the same measurement as described in the previous step (a) except on the bare substrate covered by the red NIST film (1.02 mg/cm^2) as opposed to the painted surface (see Note 3).

   c. Perform the same measurement as described in the previous step (a) except on the bare substrate (with <u>NO</u> NIST film) as opposed to the painted surface.

IF the location is marked as a "SPECIAL-SPECIAL" location, first perform the "SPECIAL" measurement (described above). Then perform the "SPECIAL-SPECIAL" measurements listed below, recording the results of the "SPECIAL-SPECIAL" readings using a new row of the same "XRF TEST DATA - SPECIAL MEASUREMENTS" form.

Procedure for performing a "Special-Special" measurement is as follows:

1. For each new "XRF TEST DATA - SPECIAL MEASUREMENTS" form needed, complete the header of the form (see exemplar p. B-16).

2. Affix the sampling location/identification bar code in the correct box on the "XRF TEST DATA - SPECIAL MEASUREMENTS." These bar code labels should be present in close proximity

to the sampling location marked by the field team leader (see Note 1). If a bar code label is not available, write in the sampling location number written at the location.

3. Perform whatever normal instrument checks are required by the manufacturer of the XRF to prepare the instrument for taking Lead measurements. Inform the data monitor what the procedure is and why it is being done. The data monitor will write this information in the "Comments" column of the form.

4. Perform SPECIAL-SPECIAL measurements on the painted and exposed surfaces as follows (See Notes 2 and 4):

   a. The Scitec MAP will perform one nominal 240-second reading (confirm mode) on the painted surface (this corresponds to the CONFIRM mode of the Scitec). Call out the value after readings. The monitor will write the read cycle value on the "XRF SPECIAL LOCATIONS DATA FORM," verbally verifying the value written. The monitor will record other information in the "Comments" column. The XRF Monitor will record the nominal time of 240 sec. in the "Approx. Sampling Time (Sec.)" column.

   b. Perform the same measurement as described in the previous step (a) except on the bare substrate covered by the red NIST film (1.02 mg/cm^2) as opposed to the painted surface (see Note 3).

   NOTE 4: No measurements using the nominal 240-sec reading (CONFIRM mode) will be taken on the bare substrate.

3.5 PROCEDURES FOR CONTINUING DRIFT CHECKS

Continuing drift checks are performed when the location substrate changes from one type to another. The first metal-continuing drift check measurement is performed immediately following the beginning-of-day control block measurements and before the first painted metal location is measured. **<u>NO</u> continuing drift checks are to be performed on the "special" measurement day by the Scitec instruments.**

If the surface substrate is of a different type than the previous location then perform the following measurements:

1. For each new "XRF TEST DATA - CONTINUING DRIFT CHECKS" form needed, complete the header of the form (see exemplar, p. B-17)

2. Perform two measurements each using two NIST standard films and one measurement on the bare control block (three nominal 15-sec readings with the yellow NIST film, red NIST, and no NIST film in that order) on the test block corresponding to the substrate just completed. Call out the value after reading. The monitor will write the read cycle value on the "XRF TEST DATA—CONTINUING DRIFT CHECKS" form, verbally verifying the value written. The monitor will record other information in the "Comments" column.

3. Perform two measurements each using two NIST standard films and one measurement on the bare control block (three nominal 15-sec readings with the yellow NIST film, red NIST, and no NIST film in that order) on the test block corresponding to the NEXT substrate to be tested. Call out the value after reading. The monitor will write the read cycle value on the "XRF DATA - CONTINUING DRIFT CHECKS" form, verbally verifying the value written. The monitor will record other information in the "Comments" column.

   For example, after completing the beginning-of-day test block readings, perform the continuing drift check measurements on the metal test block, then proceed to test all metal substrates in the unit as listed. After completion of all painted metal substrate locations, repeat the continuing drift check measurements on the metal test block, then perform the continuing drift check measurements on the wood test block. Next, repeat for all wood locations, etc. Consult the test-order list received from the supervisor AND FOLLOW THAT ORDER EXACTLY.

3.6 END-OF-DAY ACTIVITIES

XRF operator and monitor will ascertain that all form headers are completed, including the appropriate pagination. Paginate the forms of the same type in chronological order for that day of testing only starting with page 1. XRF operator and monitor will verify that all required locations and required

measurements at each location have been made. Verification will be performed by reviewing the data forms and adding a check mark to each location on the test-order list provided by the supervisor for each data entry found on the data forms. Transfer of XRF data forms to the acting MRI field supervisor will be made at the end of each day. The acting MRI field supervisor will check the data forms for completeness and conduct other end-of-day activities before releasing workers for the day.

XRF information form

XRF Test Data - Standard Measurements form

XRF Test Data - Special Measurements form

XRF QC Data: Control Blocks form

XRF Test Data - Continuing Drift Checks form

APPENDIX Bm

MODIFICATIONS TO FULL STUDY PROTOCOLS: MEASUREMENT PROTOCOLS FOR XRF TESTING

MODIFICATION SUMMARY	
Appendix no.	B
Modification no.	1 of 4
Effective date	August 2, 1993
Modification type	Addition to appendix
Portion of work affected	Denver and Philadelphia
Description	A summary of XRF testing was generated by EPA for both Denver and Philadelphia immediately preceding initiation of XRF testing in Denver. The summary was used as a tool to aid in training of field personnel for XRF measurements.

XRF SUMMARY

1. On "regular sampling" days, all instruments will be operated so that a measurement is defined as the average of three readings of approximately 15 sec with the shutter open with a fresh source. One slight exception to this is the Warrington ML-1 instrument, which will be operated so that a single trigger pull will result in three beeps, signifying that three readings have been taken. All instruments except type TN Lead Analyzer and the Outokumpu X-MET 880 can automatically adjust for source age. The TN Lead Analyzer and the Outokumpu X-MET 880 will be adjusted for source age by setting the time the shutter is open to somewhat more than 15 sec.

2. On regular sampling days, all instruments will perform the beginning and end of day drift checks, the continuing drift checks, and measurements on all the sampling areas. At the sampling areas not marked SPECIAL or SPECIAL-SPECIAL, measurements will be taken on the painted area designated for XRF and the scraped area covered by the NIST 1.02 standard. For all instruments except the MAP-3s, at the SPECIAL and SPECIAL-SPECIAL locations, measurements will be

taken on the painted area designated for the XRF, the scraped area covered by the NIST 1.02 standard, and the scraped area without any standard. For the MAP-3s, at the SPECIAL and SPECIAL-SPECIAL locations, measurements will be taken on the painted area designated for the XRF and the scraped area covered by the NIST 1.02 standard. The order of measurements at the sample locations will be: painted area, NIST standard over painted area, and (if applicable), bare substrate.

3. The MAP-3S (and only the MAP-3S) have been designated for "special" sampling days. On special sampling days, measurements will be taken for the beginning and end of day drift checks using three SCREEN mode readings to define a measurement, measurements will be taken at the SPECIAL and SPECIAL-SPECIAL locations using the TEST mode of the MAP-3, and measurements will be taken at SPECIAL-SPECIAL locations with THE CONFIRM mode of the MAP-3. At the SPECIAL and SPECIAL-SPECIAL locations, measurements will be made with the TEST mode on the painted area, the scraped area covered by the NIST 1.02 standards, and the scraped substrate area without any standard, in that order. At the SPECIAL-SPECIAL locations, measurements in the CONFIRM mode will be made on the painted area and the scraped substrate area covered by the NIST 1.02 standard, in that order. At SPECIAL-SPECIAL locations, TEST measurements will be done before CONFIRM measurements.

4. For each house, a starting substrate will be selected. An order of substrates will be designated for the study, and sampling at that house will follow the order established by the starting substrate and the study order of substrates.

5. The beginning and end of day drift checks will follow the order of substrates designated for the study. This will be a constant that does not change from house to house. Within each substrate, the order of standards will be: 3.52, 1.02, bare.

6. For continuing drift checks, the order of standards will be 3.52, 1.02, bare.

MODIFICATION SUMMARY	
Appendix no.	B
Modification no.	2 of 4
Effective date	August 2, 1993
Modification type	Addition and changes to Appendix
Portion of work affected	Denver and Philadelphia

Description	Note 4 added to appendix to clarify performance control block measurements during special testing days. All other following note references changed as a result of addition of note 4 as shown below: **!** Add "(See note 4)" after "separate testing day." located at the end of the first paragraph under subsection 3.5, page B-12: **!** Insert the following after the above addition located at the end of the first paragraph under subsection 3.5, page B-12: **NOTE 4:** Perform control block measurements in the same manner as that described in Section 3.3 (i.e., use 3 nominal 15-s read cycles, not the 60-s or 240-s read cycles). **!** Change "(see Note 1)" to "(see Note 5)" located under item 2 on page B-12. **!** Change "**NOTE 1:**" to "**NOTE 5:**" located under item 2 on page B-12. **!** Change "(See Notes 2 and 4)" to "(see Notes 5 and 6)" located under item 4 on page B-13. **!** Change "**NOTE 4:**" to "**NOTE 6:**" located under item 4 on page B-14.

MODIFICATION SUMMARY	
Appendix no.	B
Modification no.	3 of 4
Effective date	September 7, 1993
Modification type	Replacement of pages in Appendix
Portion of work affected	Philadelphia only
Description	Replacement 3 of the original XRF data forms with 4 forms as follows: ! Original:"XRF Instrument Information" form. Replace with 2 forms:"Initial—XRF Instrument Information" form and "Daily—XRF Instrument Information" form as attached ! Original:"XRF QC Data: Control Blocks" form. Replace with:"XRF QC Data: Control Blocks" form as attached ! Original:"XRF Test Data: Continuing Drift Checks" form. Replace with:"XRF Test Data: Continuing Drift Checks" form as attached

Initial XRF Instrument Information Form

Daily XRF Instrument Information Form

XRF Test Data Continuing Drift Checks Form

XRF QC Data: Control Blocks

MODIFICATION SUMMARY	
Appendix no.	B
Modification no.	4 of 4
Effective date	October 1, 1993
Modification type	Addition to Appendix
Portion of work affected	Philadelphia only
Description	A XRF testing handout was generated for field testing in Denver and Philadelphia. This handout consisted of a testing schedule and selected pages from the QAPjP (Chapters 9, 10, and Appendix B). For Philadelphia, an additional summary of Appendix B titled "XRF TESTING REMINDERS" was generated and incorporated into the handout. The 1 page summary is attached and is hereby presented as an addition to Appendix B.

XRF TESTING REMINDERS

! Measure and record in the Daily Information Data form Daily the actual reading cycle times for each XRF instrument. Check the reading time against that expected for a source age. If the readings are other than expected contact the general supervisor for a decision on corrective action.

! FOR THE WARRINGTON: Record the density data in the comments column of the data forms. It is desirable to record this for all locations <u>during the painted surface only</u> readings. However, at a minimum, record the coverage index data for all the substrate transition points (i.e., record the density value for the last and first location of a given substrate).

! FOR THE NITON: Record the coverage index data in the comments column of the data forms. It is desirable to record this for all locations <u>during the painted surface only</u> readings. However, at a minimum, record the coverage index data for all the substrate transition points (i.e., record the density value for the last and first location of a given substrate).

! For SPECIAL measurements (Scitec only):

- Note that all modes of the Scitec are used during the SPECIAL measurement days (Screen, Test, and Confirm).
- Perform Control Block Measurements using 3 nominal 15-sec read cycles.
- Perform Control Block Measurements only at the beginning and end of day regardless of whether than more than one unit is tested in that day (Philadelphia only).
  Be sure to perform End-of-Day (EOD) measurements on the same control blocks as those used for Beginning-of-Day (BOD) measurements (i.e., go back to the BOD control blocks to perform the EOD measurements). Do not move the control blocks from the unit and general location established for control block testing.

APPENDIX C

FULL STUDY PROTOCOLS:
MEASUREMENT PROTOCOLS FOR SPOT TEST KITS

MEASUREMENT PROTOCOLS FOR SPOT TEST KITS

1.0 SUMMARY

This appendix describes the field protocols for using commercial test kits for testing *in situ* painted surfaces for Lead content. The chemistry and instructions vary from kit to kit but basic steps common to all kits are:

- Select the area or item to be tested;
- Prepare the test kit reagents;
- Perform the quality control test included in the package;
- Clean the surface to be tested;
- Expose all layers of the paint by sanding or cutting; and
- Test the paint.

The actual test methods involve reaction of Lead in the paint with the active reagent(s) in the test kit to produce a color change, a precipitate, or both. Methods of reacting the Lead with the reagents vary and include:

- Swabbing *in situ* with a reagent-soaked applicator;
- Pressing a reagent-soaked pad to the *in situ* surface for a specified length of time;
- Adding drops of one or more solutions to the *in situ* paint;
- Removal of a paint chip or dust to a vial to which reagents are added to produce the precipitate or color change; and
- Removal of a paint chip and applying test reagents to all surfaces and edges of the paint chip.

2.0 MATERIALS AND EQUIPMENT

Materials and equipment needs vary from kit-to-kit. Equipment and supplies are listed under the individual kit

protocols.

3.0 TEST KITS SELECTED FOR THE STUDY

The four commercially available Lead test kits selected for inclusion in this study are listed in Table C-1. In addition to the kits listed in Table C-1, a licensed Lead inspector will be contracted to perform Lead testing with the Massachusetts state-approved sulfide reagents and procedures. The protocol for the Lead inspector will be the state-approved protocol included in this Appendix, Section 4.5.

Table C-1. LEAD TEST KITS TARGETED FOR USE IN THE FULL STUDY

MANUFACTURER	KIT NAME	CODE LETTER	TEST	KIT METHOD CHOSEN
ENZONE	Lead Zone	A	Proprietary	Reagent-impregnated pad
Frandon/Pace	Lead Alert (All-in-One)	B	Rhodizonate	Core sample paint chip
Innovative Synthesis	Lead Detective	C	Sodium Sulfide	Apply reagent to notch or paint chip
HybriVet Systems	Lead Check	D	Rhodizonate	Reagent-impregnated swabs
MA State Protocol	NA	E	Sodium Sulfide	Apply reagent to notch or paint chip

4.0 TESTING PAINTED SURFACES FOR LEAD

In order to provide a reasonably uniform comparison of methods for this study, differences among the kit instructions preclude use of only the package-insert instructions for training and testing. For purposes of this study, instructions supplied by the manufacturers were edited to conform to the six steps listed above in the Appendix C Summary (Section 1.0).

NOTE FOR ALL TEST KITS: If a new test kit is opened

for use, properly discard any chemicals or reagents from previously used test kits and make fresh from the new kit.

4.1 ENZONE "Lead Zone" (PROPRIETARY CHEMICAL COMPOSITION)

This kit is designated with the code letter A.

4.1.1 List of Supplies Needed for Testing One Housing Unit Containing up to 75 Locations

1	Plastic tote to carry supplies
1	Clipboard
1	Map of dwelling and/or instructions from supervisor
1	"Completed Testing" Checklist for the unit being tested
1	"Lead Zone" WA57 field testing protocol
1 pad	Test Kit Results Recording Form (will be several pages)
2	Ball point pens
2 boxes	Baby wipes
1 bag	Disposable plastic gloves (100 pr per bag)
1	Flashlight
1	50-mL dropping bottle full of ASTM Type I water
1 pair	Scissors
1 box	Resealable plastic bags, 1 qt. (20/box)
2	Trash bags
1 roll	Duct tape
17 kits	"Lead Zone" test kits—enough to perform 100 tests
1	Stopwatch
1	Watch or other time piece

4.1.2 Performing the Lead Zone Tests

Perform Lead testing in a safe manner as instructed in the training class.

1. Obtain the "Lead Zone" test kits, data recording forms, and other supplies in the above list from the field supervisor.

2. Obtain instructions (starting point, other) from the field supervisor.

3. Fill out the header information on the test form.

4. Find the location to be tested according to instructions received from the field supervisor. The location map will be provided by the supervisor, or alternately, may be posted in the dwelling.

5. Remove one bar code label corresponding to the sampling location from the strips held inside the plastic bag attached to the test location and affix it in the bar code column on the results recording form.

6. Open one Lead Zone Kit and prepare the test kit pads. Be careful not to contaminate the test pads or painted surfaces with Lead from the test spots on the verification card enclosed in the package. Open additional kits as needed.

   a. Use scissors to cut each of the two Lead Zone Test Pads into three equal sized pieces, creating six smaller Lead Zone test pads.

   b. Store the cut test pad pieces in a resealable plastic bag. Remove one at a time as needed.

7. Perform the quality control (QC) test <u>before the first location</u> is tested and <u>after each negative result</u> to verify that the test reagents are working as listed below:

   a. Remove one test pad piece from the resealable plastic bag.

   b. Moisten the test pad with a few drops of ASTM Type I water (an orange color may develop when the pad is moistened. The orange color is due to the reagents and is not a positive test for Lead).

   c. Press the moistened pad against one of the test dots on the verification card. Hold the pad against the surface for up to 2 min.

   d. If a pink to purple color develops on the test dot or pad (or both), the reagents are working correctly. If no color develops on the test dot within the 2 min, consult the supervisor.

   e. Dispose of the used pad in the trash bag.

8. Clean the surface to be tested by wiping with a baby wipe.

9. Expose all layers of the paint by cutting through all paint layers down to the substrate. Use the bevelled V-cut (as taught in the training class.) Do not cut into the substrates. If the substrate is cut, then make a new V-notch for testing. (Be sure to make the V-notch such that the paint layers are highly exposed. Use of a shallow V is preferable to a deep V.)

10. Test the exposed paint layers as listed below:

    a. Remove one cut test pad piece from the plastic bag.

    b. Moisten the test pad with a few drops of ASTM Type I water.

    c. Press the moistened pad against the exposed paint layers. Hold for up to 2 min.

    d. If a pink to purple color develops in any of the paint layers or on the test pad within the 2 min, the test is positive for Lead.

    e. Dispose of the used test pad in the trash bag.

11. Record the test results as positive or negative on the data form—a positive result is an observed change in color on pad, or on any of the exposed paint layers from the original color to a pink or purple color. Use a flashlight if needed for observation. Record any comments on the test form in the appropriate columns.

12. Cover the tested spot with a small piece of duct tape to conceal the results from the next tester.

13. Test the remaining locations in the structure as instructed by the supervisor. Follow steps 5 through 13 until all locations in the structure have been tested. Six tests may be performed with each Lead Zone kit. Use the verification cards prior to the first test in the structure <u>and after any negative tests to verify that the moistened pad is working correctly</u>. As long as positive tests are being obtained, it is not necessary to use the verification card for each kit

opened. If a moistened pad does not produce a pink color on the test dot, consult the supervisor.

14. At the end of the testing day, perform the following:

a. Check all test results recording forms for completeness.

b. Use the "Completed Testing" Checklist to verify testing of all locations within the housing unit. If one is found to be missing, return and perform testing on it.

c. Return the completed checklist, data forms, all supplies, and remaining test kits to the supervisor.

A photocopy of the Lead Zone Lead Test Kit instructions provided with the test kit is shown in Figure C-1.

Lead Zone Test Kit—package insert copy removed because of copyright considerations.

Figure C-1 was presented on 1 page.

(Insert from packages obtained in June 1993 from Enzone Corporation, College Point, NY 11356)

Figure C-1. Photocopy of Lead Zone Test Kit instructions.

4.2 FRANDON/PACE LEAD ALERT ALL-IN-ONE (RHODIZONATE)

This kit is designated with the code letter B.

4.2.1 List of Supplies Needed for Testing One Housing Unit Containing up to 75 Locations

1	Plastic tote to carry supplies
1	Clipboard
1	Map of dwelling and/or instructions from supervisor
1	"Completed Testing" Checklist for the unit being tested
1	"Lead-Alert" All-in-One WA57 field testing protocol
1 pad	Test Kit Results Recording Form (will be several pages)
2	Ball point pens
2 boxes	Baby wipes
1 bag	Disposable plastic gloves (100 pr per bag)
1	Flashlight
1	50-mL dropping bottle full of ASTM Type I water
1 pair	Scissors
1 box	Resealable plastic bags, 1 qt. (20/box)
2	Trash bags
1 roll	Duct tape
1 kit	"Lead-Alert" All-In-One test kits—enough to perform 100 tests
1	Circular boring tool and cleaning brush
1	Stopwatch
1	Watch or other time piece
2 boxes	Kimwipes

4.2.2 Performing the "Lead-Alert" All-In-One Test

The Frandon Lead Alert All-in-One kit offers the user three different methods of sampling for Lead, two of which are also offered in the "Homeowners" kit. For purposes of this study, we are only interested in total Lead content of a given sample. Therefore, only one of the three—removal of a paint sample using the "coring technique"—will be used.

1. Obtain the "Lead-Alert" All-in-One test kits, data recording forms, and other supplies listed above from the field supervisor.

2. Obtain instructions (starting point, other) from the field supervisor.

3. Prepare a new batch of indicating solution at the beginning of each day of testing as listed below:

   a. Remove red cap and clear dropper insert from the bottle labelled "Indicating Solution." Be careful not to spill the contents.

   b. Take the tablet from the foil wrapper and drop it into the indicating solution bottle. Replace dropper insert.

   c. Shake the bottle for 60 to 70 sec. Allow to stand for an additional minute. Shake again for 30 sec. Reagent is ready for use. When testing has been interrupted for 15 min, shake the indicating solution bottle vigorously for 5 to 10 sec before resuming testing (shaking the solution bottle should be performed periodically during the testing day).

4. Perform a Quality Control (QC) Test on the freshly made indicating solution as listed below:

   a. Remove the QC test sheet from its bag and apply 1 drop of Leaching Solution to the center of an unused test circle. Let it sit for 10 sec.

   b. Add one drop of Indicating Solution to the same circle (do not touch the dropper to any surface).

   c. A pink to rose/red color is a positive test, indicating that the reagents are performing correctly. Record the QC test results in the "Comments" column of data form for the first sample location to be tested for the day. If the test is negative, replace cap on red top bottle and shake for an additional 60 sec. Repeat the QC test. If test is still negative, mark the reagent bottles as bad with a marking pen and consult the supervisor. Under these conditions, the supervisor will generally request that you go back to step 3 using a new reagent from a new test kit and test kit.

5. Fill out the header information on the data recording form.

6. Find the first location to be tested according to instructions received from the field supervisor. The

location map will be provided by the supervisor, or alternately, may be posted in the dwelling.

7. Remove one bar code label corresponding to the sampling location from the strips held inside the plastic bag attached to the test location and affix it in the bar code column on the results recording form.

8. Clean the test area with a pre-moistened wipe.

9. Perform the Coring Test for Total Lead.

   a. Remove one of the adhesive-backed collection papers and fold it in half. Apply the paper directly underneath the area to be tested as shown in the package instructions.

   b. Using the circular coring tool, cut down into the surface (use a drilling type motion to aid in cutting through all layers of paint). Scrape the paint inside the circle onto the paper. Be sure to remove all layers of paint. Do not cut into the substrate. If the substrate is cut, start over.

   c. Transfer the paint from the paper to a plastic vial. Grind up the paint for about 10 sec using a new plastic rod for each sample (Lead paint grinds easily whereas Latex-based paint will be harder to grind).

   d. Add three drops of Leaching Solution to the vial (do **not** touch the dropper to the vial or contents) and grind the contents for another 10 sec. Let the vial sit for 20 sec.

   e. Add three drops of Indicating Solution to the tip of a fresh applicator (always use a fresh applicator tip for each sample and do not touch the applicator or any other surface with the dropper), then touch the surface of the liquid in the plastic vial with the tip of the applicator.

   f. Observe for color changes on the applicator. A pink to rose/red color indicates a positive test.

10. Record the results on the data recording form and enter

any comments in the appropriate columns.

11. Cover the completed test with duct tape to conceal the results from the next tester.

12. Clean the coring tool with a dry paper tissue followed by the brush before proceeding to the next location. EXTREMELY IMPORTANT: THE CORING TOOL **MUST** BE CLEANED AFTER COLLECTING EACH SAMPLE. If the coring tool becomes dull, see the supervisor to have it sharpened.

13. Test the remaining locations in the structure as instructed by the supervisor. Follow steps 5 through 12 until all locations in the structure have been tested.

14. At the end of the testing day, perform the following:

    a. Check all test results recording forms for completeness.

    b. Use the "Completed Testing" Checklist to verify testing of all locations within the housing unit. If one is found to be missing, return and perform testing on it.

    c. Return the completed checklist, data forms, all supplies, and remaining test kits to the supervisor.

A photocopy of the package instructions is shown in Figure C-2.

Frandon Lead Alert All In One Kit—test kit package

insert copy removed because of copyright considerations.

Figure C-2 was presented on 4 pages.

(Insert from packages obtained in June 1993 from Pace Environs, 207 Rutherglen Drive, Cary, NC 27511)

Figure C-2. Photocopy of Frandon Lead-Alert Kit instructions.

4.3 LEAD DETECTIVE (Sodium Sulfide)

This kit is designated with the code letter C.

4.3.1 List of Supplies Needed for Testing One Housing Unit Containing up to 75 Locations

1	Plastic tote to carry supplies
1	Clipboard
1	Map of dwelling and/or instructions from supervisor
1	"Completed Testing" Checklist for the unit being tested
1	"Lead Detective" WA57 field testing protocol
1 pad	Test Kit Results Recording Form (several pages)
2	Ball point pens
2 boxes	Baby wipes
1 bag	Disposable plastic gloves (100 pr per bag)
1	Flashlight
1	50-mL dropping bottle full of ASTM Type I water
1 pair	Scissors
1 box	Resealable plastic bags, 1 qt. (20/box)
2	Trash bags
1 roll	Duct tape
1 kit	"Lead-Detective" test kit—approximately 100 tests
1	Magnifying glass
1 roll	Waxed paper
1	Stopwatch
1	Watch or other time piece
1 box	round toothpicks

4.3.2 Performing the Lead Detective Tests

The "Lead Detective" kit detects Lead (and other heavy metals) by reacting with the Lead to form a black insoluble precipitate of Lead sulfide. Perform Lead testing in a safe manner as instructed in the training class including wearing of safety glasses at all times and wearing of leather gloves during cutting or scraping activities. Wear disposable gloves when using this and any other sodium sulfide test kit. The package instructions included with the Lead Detective are contained in a 33-page instruction booklet. A photocopy of this booklet is included in this Appendix C as an attachment.

1. Obtain the "Lead Detective" test kits, data recording forms, and supplies from the field supervisor.

2. Obtain sample location instructions (starting point, other) from the field supervisor.

3. Fill out the header information on the data recording form.

4. Prepare a new batch of reagents at the beginning of each day of testing as listed below:

   a. Carefully add the contents of the kit water bottle to the bottle containing the sodium sulfide crystals.

   b. Screw on the dropper cap and shake vigorously for 5 min or until the crystals are dissolved. Do not use the reagent until the crystals are totally dissolved.

5. Perform the quality control check on the freshly prepared sodium sulfide solution.

   a. Remove a quality control strip (or the paint chip) from the plastic bag.

   b. While holding the strip in the forceps, add a drop of the sodium sulfide solution to the strip.

   d. If black coloring appears, the QC test is positive, indicating the reagents are working. Record the results in the "Comments" column of the data recording form. If a black color does not appear, mark the reagent bottles as bad with a marking pen and consult the supervisor. Under these conditions, the supervisor will generally request that you go back to step 4 using a new reagent from a new test kit and repeat the test.

6. Find the location to be tested according to instructions received from the field supervisor. The location map will be provided by the supervisor, or alternately, may be posted in the dwelling.

7. Remove one bar code label corresponding to the sampling location from the strips held inside the plastic bag attached to the test location and affix it in the bar code column on the results recording form.

8. Clean the surface of the test location with a pre-moistened wipe.

9. Cut through all layers of the paint down to the substrate with a bevelled V-notch. Save the paint chip removed from the notch on a clean, waxed paper square.

10. Add a drop of the sodium sulfide solution to the notch, being careful not to drip the reagent on the surfaces below or adjacent to the test notch. Use a toothpick as needed to direct the solution into the notch. Use a flashlight and/ or magnifying glass if needed to observe the paint for changes in color. A black or gray color is a positive test for Lead. Circle the box in the Comments column that comes closest to matching the color observed.

11. If the test is negative or doubtful, apply a drop of the test reagent to the front, back, and edges of the retained paint chip from the notch. Use a flashlight if needed to observe the paint for changes in color. A black or gray color is a positive test for Lead. Circle the box in the Comments column that comes closest to matching the color observed. Indicate use of the retained chip by writing "chip" in the Comments column.

12. Record the results on the data form and any comments in the appropriate columns. Be sure to designate whether the recorded results are for the notched surface or the removed paint chip.

13. Cover the completed test spot with a small piece of duct tape to conceal the results from the next tester.

14. Test the remaining locations in the structure as instructed by the supervisor. Follow steps 6 through 14 until all locations in the structure have been tested.

15. At the end of the testing day, perform the following:

    a. Check all test results recording forms for completeness.

    b. Use the "Completed Testing" Checklist to verify testing of all locations within the housing unit. If one is found to be missing, return and perform testing on it.

c. Return the completed checklist, data forms, all supplies, and remaining test kits to the supervisor.

The "Lead Detective" instructions in the kit consists of a 33-page booklet. A photocopy of the test kit operating instructions portion of this booklet is shown in Figure C-3.

Lead Detective Lead Paint Detection Kit—package insert

copy removed because of copyright considerations.

Figure C-3 was presented on 7 pages.

(Insert from packages obtained in June 1993 from Innovative Synthesis Corporation, 1425 Beacon Street, Newton, MA 02168)

Figure C-3. Photocopy of "Lead Detective" instructions

4.4 LEAD CHECK SWABS

This kit is designated with the code letter D.

4.4.1 List of Supplies Needed for Testing One Housing Unit Containing up to 75 Locations

1	Plastic tote to carry supplies
1	Clipboard
1	Map of dwelling and/or instructions from supervisor
1	"Completed Testing" Checklist for the unit being tested
1	"Lead Check Swabs" WA57 field testing protocol
1 pad	Test Kit Results Recording Form (several pages)
2	Ball point pens
2 boxes	Baby wipes
1 bag	Disposable plastic gloves (100 pr per bag)
1	Flashlight
2	Trash bags
1 roll	Duct tape
100	"Lead Check" swabs and several control cards
100	Disposable 10-mL beakers
1	Pliers
1	Stopwatch
1	Watch or other time piece
1	Razor knife holder
75-100	Disposable razor blades
75-100	Cotton-tipped swabs
1 bottl	Vinegar

4.4.2 Performing the Lead Check Test

The "Lead Check" swabs contain rhodizonate, which reacts with Lead to form a pink to red color. Perform Lead testing in a safe manner as instructed in the training class.

1. Obtain the "Lead Check" rhodizonate test swabs, data recording forms, and supplies from the field supervisor.

2. Obtain instructions (starting point, other) from the field supervisor.

3. Fill out the header information on the data recording form. Measure and record temperature, relative humidity, and other required information.

4. Find the location to be tested according to instructions received from the field supervisor. The location map will be provided by the supervisor, or alternately, may be posted in the dwelling.

5. Remove one bar code label corresponding to the sampling location from the strips held inside the plastic bag attached to the test location and affix it in the bar code column on the results recording form.

6. Clean the test surface with a pre-moistened wipe.

7. Cut a beveled V notch through all paint layers down to the substrate.

8. Check for leachable pink or red paint. Moisten a clean, unused cotton-tipped swab with vinegar and rub the swab in the notch. If the tip turns pink or red from vinegar only, make a comment in the Comments column and continue on with test.

9. Remove one "Lead Check" swab and reseal the package.

10. With the swab pointing up, squeeze points A and B to crush the internal glass ampules (use pliers to perform this task if needed).

11. With the swab pointing down, shake the swab twice, then gently squeeze it until the yellow liquid appears on the swab tip.

12. While gently squeezing, rub the swab tip on the test area for 30 sec.

13. Observe <u>swab tip for coloration</u>. Use a flashlight to read the results. Pink to red indicates positive test for Lead. Orange plus pink is also positive for Lead.

IF a positive result is obtained, THEN

a. Tape a plastic disposable beaker, using duct tape, over the tested notch.

b. Record the results in the appropriate box on the form.

IF no color change is observed within 2 min, THEN

a. Touch the swab to one of the dots on the Lead confirmation card.

If no color develops on the QC dot, discard the swab and retest the paint layers with a new swab (steps 8 through 12).

If color develops on the QC dot, tape a plastic disposable beaker, using duct tape, over the tested notch and proceed to the next spot. Record the time and return to re-observe this spot in 30 min. If no color change has occurred, cover and return to check the paint after another 30 min. If, after 1 hr, no color has developed, the spot tested negative for Lead. Record all observations, subsequent examinations, and other comments on the data form. Pink to red is positive for Lead. If an <u>orange</u> color develops, orange is positive for barium, <u>not</u> positive for Lead.

14. Test the remaining locations in the structure as instructed by the supervisor. Follow steps 4 through 13 until all locations in the structure have been tested. Do not reuse any of the swabs, even if no color change was observed. As long as positive tests are being obtained on the painted surfaces and underlying layers, there is no need to perform the Lead confirmation test on the test confirmation card.

15. At the end of the testing day, perform the following:

a. Check all test results recording forms for completeness.

b. Use the "Completed Testing" Checklist to verify testing of all locations within the housing unit. If one is found to be missing, return and perform testing on it.

c. Return the completed checklist, data forms, all supplies, and remaining test kits to the supervisor.

A photocopy of the "Lead Check" Swabs Test Kit package instructions is shown in Figure C-4.

Lead Check Swabs—test kit package insert copy removed

because of copyright considerations.

Figure C-4 was presented on 2 pages.

(Insert from packages obtained in June 1993 from Hybrivet Systems, Inc., P.O. Box 1210, Framingham, MA 01701)

Figure C-4. Photocopy of Lead Check Swabs Test Kit instructions.

4.5 MASSACHUSETTS SODIUM SULFIDE TEST

This test is designated with the code letter E.

A licensed lead inspector, qualified by the state of Massachusetts, will perform this test according to the protocols in Attachment 2 of this Appendix. The Massachusetts professional will provide all of their supplies and equipment with the exception of the data recording forms and masking tape, according to the following protocol:

Perform Lead testing in a safe manner as instructed in the training class, including wearing safety glasses at all times and wearing leather gloves and respirator during cutting or scraping activities. Wear disposable gloves when using this and any other sodium sulfide test kit.

1. Obtain the data recording forms, masking tape, pre-moistened wipes, and other supplies from the field supervisor.

2. Obtain instructions (starting point, other) from the field supervisor.

3. Fill out the header information on the data recording form.

4. Find the location to be tested according to instructions received from the field supervisor. The location map will be provided by the supervisor, or alternately, may be posted in the dwelling.

5. Remove one bar code label corresponding to the sampling location from the strips held inside the plastic bag attached to the test location and affix it in the bar code column on the results recording form.

6. Clean the test surface with a pre-moistened wipe.

7. Perform the test according to the Massachusetts protocol.

8. Record the results in the appropriate box on the data recording form. Record all observations, subsequent examinations, and other comments in the data form.

9. Cover the completed test with a small piece of duct tape to conceal the results from the next tester.

10. Test the remaining locations in the structure as instructed by the supervisor. Follow steps 4 through 9 until all locations in the structure have been tested.

11. At the end of the testing day perform the following:

    a. Check all test results recording forms for completeness.

    b. Use the "Completed Testing" Checklist to verify testing of all locations within the housing unit. If one is found to be missing, return and perform testing on it.

    c. Return the completed checklist, data forms, all supplies, and remaining test kits to the supervisor.

A photocopy of the general Massachusetts protocol is shown in Figure C-5.

General Massachusetts Protocol removed because of

copyright considerations.

Figure C-5 was presented on 5 pages.

(Obtained in March 1993 from the State of Massachusetts.)

Figure C-5. Photocopy of the General Massachusetts Protocol.

APPENDIX Cm

MODIFICATIONS TO FULL STUDY PROTOCOLS: MEASUREMENT PROTOCOLS FOR SPOT TEST KITS

MODIFICATION SUMMARY	
Appendix no.	C
Modification no.	1 of 2
Effective date	July 14, 1993
Modification type	Addition to Appendix
Portion of work affected	Denver and Philadelphia
Description	Addition of protocols for performance of a test kit assigned to the letter "F". This addition is shown on pages Cm-4 through Cm-9.

The Lead Alert "Homeowner" Kit (Product 1040) will be included in the study. The sanding technique described in the kit's instructions will be used to test layers of paint until either (1) a positive result is obtained, or (2) the bottom layer of paint is tested. This test kit will be assigned letter "F" for identification purposes in the study. A detailed protocol following the instructions in the kit follows.

Each location marked off for sampling will include 6 4-in. by 1-in. rectangles for test kit applications. A letter representing each test kit will be randomly assigned to each of the 6 rectangles at each location. Rectangles marked with letter F will be designated for testing by the Lead Alert Homeowner Kit (Product 1040).

Testing with the Lead Alert Homeowner Kit using the sanding technique is expected to take approximately four times longer than the other kits. Therefore, for each house, the test kit operator assigned to the Lead Alert sanding technique for that house will be asked to apply the kit only at the locations marked "Special." (One-fourth of the locations in each house will be marked "Special.") It is expected that the operator of the Lead Alert Homeowner Kit will be able to complete testing at the "Special" locations in the day and one-half allocated for testing at each house.

If the test kit operator for the Lead Alert Homeowner Kit is able to complete the "Special" locations ahead of schedule, the operator will alternate between the substrates in the house as follows: (1) first regular location for each of metal, wood, brick, drywall, concrete, and plaster; (2) second regular location for each of metal, wood, brick, drywall, concrete, and plaster; (3) third regular location for each substrate, and so on until available time for the unit is exhausted or until all regular substrates are tested. The starting substrate for the regular locations will change for each unit. The supervisor will issue instructions for each unit to the tester applying the Lead Alert sanding technique test.

Because the Lead Alert Homeowner Kit will be used with the sanding technique, contamination avoidance is especially important. Contamination avoidance techniques will include the following:

1. During the marking phase, attempts will be made to avoid

placing one location directly over another.

2. Operators of the Lead Alert Homeowner Kit will attempt to sand so as to minimize the spreading of paint dust. Where possible, paper or collection receptacles will be used to catch the dust.

3. Where possible, operators of the Lead Alert Homeowner Kit will apply the kit within the bottom most 2-in. of the rectangle assigned to the kit (on a horizontal template) and all other operators will use the top most 2-in. On vertical templates, the Lead Alert operator will use the left most 2-in. and all other operators will use the right most 2-in. On unusual locations which do not fit a standard template, the immediate supervisor for the house will give directions to the operators.

4. All operators will be told of the importance of wiping rectangles before applying the kits. Wipes should be firm enough to remove surface dust, but not so firm as to remove paint.

5. Operators of the Lead Alert Homeowner Kit will be instructed to dispose of all sand paper properly and to clean their hands (preferably with soap and water, but baby-wipes may be used if running water and soap are not available) before applying the test at the next location.

6. Paint chip collectors will wipe the area for paint chip collection before collecting paint chip samples. Paint chip collectors will wipe the area for XRF testing before moving to the next location.

7. To the extent possible, the Lead Check tester and the Lead Alert Sanding Technique tester will be on different teams.

<u>List of Supplies Needed for Testing One Housing Unit Containing up to 75 Locations (one-fourth designated as "Special.")</u>

1	Plastic tote to carry supplies
1	Clipboard
1	Map of dwelling and/or instructions from
1	supervisor
1	"Completed Testing" Checklist for the unit being
1 pad	tested
2	Lead Alert (Product 1040) WA57 field testing
2 boxes	protocol

1 bag	Test Kit Results Recording Form (will be several
1	pages)
1 pair	Ball point pens
1 box	Baby wipes
2	Disposable plastic gloves (100 pr per bag)
1 roll	Flashlight
1 kit	Scissors
1 box	Resealable plastic bags, 1 qt. (20/box)
	Trash bags
1 box	Duct tape
	"Lead Alert" test kit (Product 1040) and extra sandpaper
	Kimwipes

Performing the "Lead Alert" Sanding Technique

1. Obtain the Lead Alert Product 1040 test kit, data recording forms, and other supplies listed above from the field supervisor.

2. Obtain instructions (starting point, check list of locations, other) from the field supervisor.

3. Prepare a new batch of indicating solution at the beginning of each day of testing as instructed in the package instructions:

a. Remove red cap and clear dropper insert from the bottle labelled "Indicating Solution." Be careful not to spill the contents.

b. Take the tablet from the foil wrapper and drop it into the indicating solution bottle. Replace the dropper insert.

c. Shake the bottle for 60 to 70 sec. Allow to stand for an additional minute. Shake again for 30 sec. Reagent is ready for use. When testing has been interrupted for 15 min, shake the indicating solution bottle vigorously for 5 to 10 sec before resuming testing (shaking the solution bottle should be performed periodically during the testing day).

4. Perform a Quality Control (QC) Test on the freshly made indicating solution as listed below:

a. Remove the QC test sheet from its bag and apply 1 drop of Leaching solution to the center of an unused test circle. Let it sit for 10 sec.

b. Add one drop of Indicating Solution to the same circle (do not touch the dropper to any surface).

c. A pink to rose/red color is a positive test, indicating that the reagents are performing correctly. Record the QC test results in the "Comments" column of the data form for the first location to be tested for the day. If the test is negative, replace cap on red top bottle and shake for an additional 60 sec. Repeat the AC test. If test is still negative, mark the reagent bottles as bad with a marking pen and consult the supervisor. Under these conditions, the supervisor will generally request that you go back to step 3 using a new reagent from a new test kit.

5. Fill out the header information on the data recording form.

6. Find the first location to be tested according to instructions received from the field supervisor. The location map will be provided by the supervisor, or alternately, may be posted in the dwelling.

7. Remove one bar code label corresponding to the sampling location from the strips held inside the plastic bag attached to the test location and affix it in the bar code column on the results recording form.

8. Clean the test area with a pre-moistened wipe.

9. Perform the sanding test according to the following instructions:

10. Take a clean paper square and tape it to the wall directly underneath the test rectangle. This paper will catch the paint particles loosened by the sand paper.

11. Proceed with testing, following instructions given below:

<u>"Underlying layers of paint</u>: If the surface layer of paint is not positive for lead then all other layers should be tested until either a positive is obtained for the underlying surface (substrate) which has been painted (wood, brick, etc.) is

reached. Layers of paint may be tested individually or several at a time. After sanding, follow specific instructions as listed under Particles of paint, metal, dust, etc. below.

NOTE: Sulfates present in plaster, dust, or stucco may interfere with the color development in test procedures. Care should be taken not to sand through into these substrates during testing. Drywall contains gypsum. Care should be taken not to penetrate the fiber layer (paper) of drywall. If, however, plaster; gypsum; or stucco is exposed during testing and that test is positive, it is a valid test. Lead in paint for residential use was banned in the USA in 1978. If the test result is negative and your home was built prior to 1978-80 we recommend that a sample of all layers of paint from that test site be taken and sent to a qualified laboratory for further analysis.

Particles of paint, metal, dust, etc.:

a. Apply two drops of leaching solution to applicator tip.

b. Pick up a very small amount of fine particles of the material to be tested (such as sanded paint, ground paint chips, paint dust, house dust, or dust from vacuum cleaner bag) on the moistened applicator tip.

c. Apply one more drop of leaching solution over the particles on the applicator tip. Wait 30 seconds.

d. Apply two drops of indicating solution to the applicator tip and watch for color change. Interpret the results as follows:

(1) Positive result - The appearance of a pinkish to rose/red color. Leachable lead has been detected.

(2) Negative result - The appearance of a yellow stain that fades away within a few minutes. No leachable lead has been detected.

NOTE: The appearance of an orange color that doesn't turn pinkish, or a yellow color that does not fade after a few minutes may indicate the presence of barium that is often used as an extender in paint. This is also to be

interpreted as a negative for lead."

12. Interpret the results. A pink to rose/red color is positive for lead. A yellow stain is negative for lead.

13. Record the results on the test kit results form. Some locations will have sufficient layers of paint to require the sanding test to be performed in several steps; other locations can be completed in only one or two sanding steps.

i. If the test was positive, STOP testing at this location, and proceed to the next location.

ii. If the test was negative, proceed with the sanding technique. Continue testing until a positive test is obtained, or until the bottom most layer of paint has been tested. Remember that some locations will have sufficient layers of paint to require the sanding test to be performed in several steps; other locations can be completed in only one or two sanding steps.

NOTE: Whenever a pink or red paint color is encountered, look at the applicator tip after rubbing the paint with leaching solution but before adding the indicating solution. The leaching solution may leach the natural pink or red color from some paints thus leading to a false positive for lead. If the leaching solution leaches pink or red from the paint, record this information in the "Comments" column for that location, and consult the supervisor.

14. Cover the completed test with duct tape to conceal the results from the next tester.

15. Test the remaining locations in the structure as instructed by the supervisor. Follow steps 6 through 14 until all locations have been tested.

16. At the end of the testing day, perform the following:

a. Check all test results recording forms for completeness.

b. Use the "Completed Testing" Checklist to verify testing of all locations within the housing unit. If any are found to be missing, return and perform testing.

c. Return the completed checklist, data forms, all supplies, and remaining test kits to the supervisor.

MODIFICATION SUMMARY	
Appendix no.	C
Modification no.	2 of 2
Effective date	July 14, 1993
Modification type	Change to Appendix
Portion of work affected	Denver and Philadelphia
Description	Change of a step to include use of cotton swab for delivering sodium sulfide reagent to test surface when using the Lead Detective Test Kit. **!** Replace step 10 located on page B-15 with the following: 10. Add a drop or two of the sodium sulfide solution to a cotton swab, being careful not to touch the swab to the reagent container. Rub the swab tip on the test area for 30 sec. Observe test surface for coloration. Use a flashlight and/ or magnifying glass if needed to observe the paint for changes in color. A black or gray color is a positive test for Lead. Circle the box in the Comments column that comes closest to matching the color observed.

APPENDIX D

FULL STUDY PROTOCOLS
COLLECTION OF PAINT CHIP SAMPLES IN AND AROUND BUILDINGS AND RELATED STRUCTURES

COLLECTION OF PAINT CHIP SAMPLES IN AND AROUND BUILDINGS AND RELATED STRUCTURES

SUMMARY

This document describes the standard protocol for obtaining a single paint chip sample from a painted substrate. This standard also includes instructions for sample storage and transport requirements.

MATERIALS AND EQUIPMENT

ITEM	No. per pair of collectors
Safety goggles	2 + 1 extra
Leather gloves	2 pair
Disposable gloves	1 bag 100 pair
Respirator with organic vapor filters	2 - one fitted to each collector
Razor blade holder	2 + 1 extra
Razor blades	25
Wood chisel	2
Hammer	1
White paper, 8.5 x 11	300 sheets
Masking tape	20 rolls, 1-inch
Duct tape	8 rolls, 2-inch
Marking pens	6
Clip board with timepiece	2 + 1 extra
"Paint Chip Collection" data forms	Enough for 300 samples
Sample containers (plastic centrifuge tubes, plastic resealable bags)	a minimum of 300 tubes

ITEM	No. per pair of collectors
Resealable plastic bags	a minimum of 300 1 qt bags
Extra shipping container for paint chip samples	4
Trouble lights and spare bulbs or equivalent lighting	2
Extension cords	200 ft.
Power generator	1 at site
Pocket knife	2 + 1 extra
Metal marking template	2
Heat gun	2
Replacement heat gun element	2
Tool pouch with belt	1 per tester
Fire extinguisher	2 at site, (1 for each team, in each unit during paint chip collection)

Note: Other items as needed.

COLLECTION PROCEDURE

At each sampling location, perform the following steps (See Note 1):

NOTE 1: A regular sample will be collected at all locations. Some locations will require collection of an additional sample called a side-by-side sample. Locations that require a side-by-side sample are identified by the presence of an individual 2 in x 2 in square placed at one end of the marked location. For locations having a side-by-side sample, follow steps 1 through 9 below for collection of the regular sample first. After completing this sample collection, collect the side-by-side sample using the same procedure using different bar code number as described in step 2.

1. For each new "Paint Chip Collection Reporting" form needed (see attached), complete the header of the form.

2. Record the sampling location/identification (ID) on an open line of the " Paint Chip Collection Reporting" data form as follows:

   FOR REGULAR SAMPLES: Use the bar code labels that correspond to the specific sampling location (NO PRECEDING LETTERS). Collection of regular samples is done from inside the middle, large square that is divided into four individual 2 in x 2 in squares. The sample to be collected is indicated by the arrow to a specific 2 in x 2 in square. The bar code labels should be present in close proximity to the sampling location marked by the field team leader.

   FOR SIDE-BY-SIDE SAMPLES: Use the bar code labels that correspond to the specific sampling location preceded by an "S." Collection of side-by-side samples is done from the individual 2 in x 2 in square placed at one end of the marked location. The bar code labels should be present in close proximity to the sampling location marked by the field team leader.

3. Affix an ID label to the outside of the container into which the sample is to be placed, and ensure that the label adheres well. Place 11 extra identical labels into a 1-qt resealable plastic bag which will hold the paint collection container when sampling is complete.

4. Place the 5 cm x 5 cm template over the sampling site and hold firmly; tape can be used to hold template in position. Using a cutting tool and the template as a guide, score the perimeter of the area to be removed. If it is impractical to use the template, the score can be made using the outside edge of the template as a guide. The area scored using the alternative method should be approximately equivalent to the area scored when using the inside of the template. Avoid using pencil or pen to mark the sample outline.

5. Affix a closed bottom paper funnel (or other appropriate collection shape) made from a clean white sheet of paper or equivalent collection device directly below the sampling location. The collection device should be located as close as possible to the sampling site but should not interfere

with the removal procedure.

6. **PRIMARY PAINT REMOVAL METHOD:** Using a heat gun, heat the sample area. Extreme caution should be exercised when using the heat gun. Be sure to have a fire extinguisher nearby during heat gun use. Do not overheat the sample area, heat only until the paint becomes soft and supple. If working in teams of two persons, have one collector heat the area while the other removes the sample with a paint scrapper. Remove all paint down to the bare substrate. If the paint does not become soft and supple in a minute or two, discontinue the use of heat and try the alternative paint removal method. If the paint is accidentally burned by the heat gun, then contact the supervisor for selection of a new sample to collect.

   Avoid the inclusion of the substrate in the collection device. If substrate does fall into the collection device, remove only that substrate which can be easily removed without losing any of the paint sample. Do not remove any substrate which cannot be separated from the paint sample. The laboratory will remove extraneous substrate if possible, under laboratory conditions.

   ALTERNATIVE PAINT REMOVAL METHOD: Using the appropriate cutting tool for a particular substrate or condition of the sample site, begin removing the paint from the substrate. If possible peel the paint off of the substrate by sliding the blade along the score and underneath the paint. Remove all paint down to the bare substrate.

   In areas where extreme difficulty is experienced in removing the paint sample, consult with the field supervisor for advice.

7. Transfer the collected paint sample to the sample container and seal. Exercise care to ensure that <u>all</u> paint taken from the recorded area is placed into the sample container. Use the Styrofoam holder that comes with the sample containers to aid in holding the container during transfer.

8. Carefully and accurately measure the sampling area dimensions. Do not attempt to calculate areas in the field. Record the data and dimensions including units used (e.g., 5.1 cm x 5.0 cm) on the "Paint Chip Collection Reporting"

data form using a permanent marker. Try to use only centimeters for recording data. Avoid making measurement in inches. Any irregularities or problems which arise in the process, should be noted in the Comments column of the form.

9. Seal the container and place it into the plastic bag containing the 11 extra bar code labels identical to the one on the paint collection container. Store the samples in a safe place during sampling until shipment can be made back to the laboratory. Return all completed "Paint Chip Collection Reporting" forms and samples by the end of each sampling day to the field supervisor.

SUBSTRATE CLEARING PROCEDURE

At each sampling location, after collection of all paint chip samples, clear an area down to the substrate for later XRF testing as follows:

Enlarge the exposed substrate area made during paint chip collection of regular samples to a minimum of 4 in x 4 in using the same general cutting and scraping methods followed for paint chip collection (See Note 2). Avoid pitting or significantly damaging the substrate surface. This area will be used by XRF testers for taking substrate measurements.

NOTE 2: For some locations, a full 4 in x 4 in area may not be possible. For these locations, make the largest exposed area possible up to the desired 4 in x 4 in exposed surface.

Paint Chip Collection Report Form (graphics)

APPENDIX Dm

MODIFICATIONS TO FULL STUDY PROTOCOLS: COLLECTION OF PAINT CHIP SAMPLES IN AND AROUND BUILDINGS AND RELATED STRUCTURES

MODIFICATION SUMMARY	
Appendix no.	D
Modification no.	1 of 2
Effective date	September 18, 1993
Modification type	Addition to Appendix
Portion of work affected	Philadelphia
Description	A Paint Collection handout was generated for both Denver and Philadelphia field work to aid in training of field personnel. This handout consisted of a testing schedule and selected pages from the QAPjP (Chapters 9, 10, and Appendix D). For Philadelphia, an additional summary of Appendix D titled "PAINT COLLECTION REMINDERS" was generated and incorporated into the handout. The 1 page summary is attached and is hereby presented as an addition to Appendix D.

PAINT COLLECTION REMINDERS

! BE AWARE OF THE FIRE EXTINGUISHER LOCATION AT ALL TIMES.

! BE SURE TO COLLECT FIELD BLANKS (ONE PER UNIT):

1) Pull one empty centrifuge tube from a package of tubes being used for each unit sampled.

2) Label the empty centrifuge tube using a permanent marking pen as follows:

For RUBY TERRACE building use

RUBY BLK # where # is equal to the unit number (1A, 1B, 3A, or 3B).

For 54TH DRIVE building use

54TH BLK # where # is equal to the unit number (1A, 1B, 3A, or 3B).

3) Fill in a line on the data form that corresponds to taking the field blank.

4) Package and ship along with other paint samples.

! BE SURE TO MEASURE AND RECORD THE AREA SAMPLED IMMEDIATELY AFTER COLLECTING EACH SAMPLE.

! ON WOOD SUBSTRATES, SCRAPE WITH GRAIN NOT ACROSS GRAIN.

! STRESS THE USE OF LESS STRENGTH AND MORE CAREFUL PAINT COLLECTION TO AVOID SUBSTRATE INCLUSION AND PRODUCTION OF SMOOTH SCRAPED SURFACES.

! BE SURE TO SCRAPE THE ENLARGED EXPOSED SUBSTRATE AREAS TO THE SAME DEGREE AS THE AREA WHERE PAINT COLLECTION WAS PERFORMED (DON'T LEAVE THE LOCATION UNTIL THE AREA SCRAPED FOR LATER XRF MEASUREMENTS LOOKS THE SAME AS THE PAINT SAMPLED AREA.)

MODIFICATION SUMMARY	
Appendix no.	D
Modification no.	2 of 2
Effective date	September 20, 1993
Modification type	Change to previous modification to Appendix
Portion of work affected	Philadelphia
Description	Labeling of field blanks was changed. **!** Replace step 2, under the second bullet of the "PAINT COLLECTION REMINDERS" with the following: 2) Label the empty centrifuge tube using a permanent marking pen as a field blank and place the marked tube along with others collected from the unit undergoing active sampling. The sample receiving personnel at the laboratory will assign a unique barcode number to field blank samples for laboratory processing. The sample receiving personnel at the laboratory will also document ID assignments used for the field blanks for tracking and reporting purposes.

APPENDIX E

FULL STUDY PROTOCOLS:
GENERATION OF TOTAL FIELD SAMPLE WEIGHTS
AND HOMOGENIZATION OF PAINT CHIP SAMPLES

GENERATION OF TOTAL FIELD SAMPLE WEIGHTS AND HOMOGENIZATION OF PAINT CHIP SAMPLES

1.0 SUMMARY

Paint chip samples (chips, powder, etc.) are weighed and homogenized to prepare them for digestion using a subsample of the original collected sample. The total weight data is used to determine the correction factors needed to convert a lead result obtained from a homogenized subsample to the lead result of the entire sample collected in the field. This permits calculation and reporting of lead data on a mg/cm^2 basis under conditions when the entire sample collected in the field is not digested for analysis.

2.0 APPARATUS

2.1 Instrumentation

! Analytical balance; suitable for weighing samples to ±0.0001 grams.

2.2 Glassware, and Supplies

! Resealable plastic centrifuge tubes, 50-mL
! Plastic rods with flat or round faces for breaking up paint chip samples
! Dry ice

2.3 Reagents

! ASTM Type I water (D 1193)

3.1 WEIGHING PROCEDURE

1. Don a new, clean pair of vinyl gloves.

2. Label a new, clean centrifuge tube with lid with the sample ID number.

3. Label the lid of the original sample container with the

sample ID number using an indelible marking pen. Allow the ink to dry.

4. Wipe off the outside of the paint sample container with a clean laboratory paper wipe to remove any foreign material or oils. Using an analytical balance shown to be operating within normal calibration specifications, weigh the sample container with lid containing the entire paint sample. Record the total paint sample plus container weight (and if provided, the area sampled) in a laboratory data form, notebook, or equivalent recording device.

5. Transfer the remaining paint sample into a new, clean, labeled centrifuge tube by carefully pouring the contents of the original sample container into the new tube. Use a clean glass rod to assist in the transfer as needed. Seal the new tube and store for archival use.

6. Remove any remaining sample powder from the original sample container and lid (received from the field) by rinsing with ASTM Type I water. Set the container aside and allow it to dry at room temperature.

7. After the original sample container has completely dried, reweigh the container with lid and record the empty container weight.

8. Determine the total field sample weight by subtracting the empty container weight from the total paint sample plus container weight generated in step 3.

3.2 HOMOGENIZATION PROCEDURE

1. Don a new, clean pair of vinyl gloves to perform sample handling.

2. Remove any large amounts of substrate that may be present in the sample. Exercise care when removing substrate to avoid any paint losses. Leaving substrate in the sample is preferred over paint chip loss. If required, use a clean safety razor blade or equivalent tool to aid in substrate removal.

3. Immerse the bottom portion of sample container into a

container containing dry ice. The depth of the container should be sufficient to cover all paint present within the sample container.

4. Allow the paint chip sample to freeze for a minimum of 10 min. Add more dry ice as needed to freeze the paint chip sample.

5. Using a clean plastic rod or other appropriate clean tool, breakup the frozen paint chip sample inside the sample container into a fine powder. Samples or sample portions that resist homogenization should be noted in laboratory records.

6. After completing breakup of the sample, tap off any powder remaining on the tool used for breaking up the paint chips back into the sample container.

7. Seal the container and roll for about a minute or two to mix the samples. Rolling can be done by hand or by using automated equipment.

APPENDIX F

FULL STUDY PROTOCOLS:
PREPARATION OF PAINT CHIP SAMPLES FOR SUBSEQUENT ATOMIC SPECTROMETRY LEAD ANALYSIS

PREPARATION OF PAINT CHIP SAMPLES FOR SUBSEQUENT ATOMIC SPECTROMETRY LEAD ANALYSIS

1.0 SUMMARY

Lead in paint chip samples (chips, powder, etc.) is solubilized by extraction with nitric acid and hydrogen peroxide facilitated by heat after sample homogenization. The lead content of the digested sample is then in a form ready for measurement by Atomic Spectrometry. This procedure is similar to NIOSH Method 7082. Modifications have been made to convert this air particulate method to a method appropriate for processing paint chip samples.

2.0 APPARATUS

2.1 Instrumentation

! Electric hot plate; suitable for operation at temperatures up to at least 100°C as measured by a thermometer inside a solution-filled container placed on the surface of the hot plate.

2.2 Glassware and Supplies

! 150-mL or 250-mL beakers (borosilicate glass) equipped with watch glass covers
! Class A borosilicate 250-mL volumetric flasks
! Class A borosilicate volumetric pipets; volume as needed
! 50-mL or 100-mL linear polyethylene tubes or bottles with caps
! Borosilicate or plastic funnels
! Glass rods and appropriate devices for breaking up paint chip samples

2.3 Reagents

! Concentrated nitric acid (16.0 M $HN0_3$); spectrographic grade or equivalent
! Nitric acid, 10% (v/v): Add 100-mL concentrated HNO_3 to 500 mL ASTM Type I water and dilute to 1 L
! Hydrogen peroxide, 30% H_2O_2 (w/w); ACS reagent grade

! ASTM Type I water (D 1193)

3.0 PROCEDURE

3.1 WEIGHING OF SUBSAMPLES

For each homogenized sample, weigh into beakers for sample digestion as described below:

1. Weigh a sub-sample of homogenized paint from the contents of the sample container into a tared beaker labeled with the sample ID. Weigh approximately 0.5 grams to 0.0001 grams.

2. Record the sub-sample weight (and if provided, the area sampled) in a laboratory data form, notebook, or equivalent recording device.

3.2 SAMPLE DIGESTION

For each sample weighed into beakers, plus any QC samples, perform digestion as described below:

1. Wet the sample with about 2 to 3 mL of water from a squirt bottle filled with ASTM Type I water.

2. Add 7.5 mL of concentrated HNO_3 and 2.5 mL 30% H_2O_2, and cover with a watch glass.

3. Gently reflux on a hot plate for about 15 min (See Note 1).

4. Remove the watch glass and evaporate gently on a hot plate until the sample volume is reduced to about 1 to 2 mL (See Note 2).

5. Replace the watch glass and remove the beaker containing sample from the hot plate and allow it to cool (See Note 3).

NOTE 1: The original NIOSH method called for temperatures of 140°C as based on the use of digitally programmable hot-plates, which measure the temperature on the inside of the hot plate head. Temperature drops of 40° to 50°C are not unusual between the inside of the hot plate head and the

temperature actually experienced by the sample solution. The temperatures of sample solution should be between 85° to 100°C to prevent spattering of the solution. Monitor solution temperature on the hot plate by placing a thermometer in a flask or beaker filled with water during digestion activities.

NOTE 2: The original NIOSH method calls for evaporation until most of the acid has been evaporated. However, in order to avoid potential losses caused by sample splattering at low volumes, the method has been modified to specifically leave some solution remaining in the digestion vessels. Reduction volumes given are approximate and can be dependent on the sample size and beaker size used for preparation. Volumes should be reduced to as low a level as comfortably possible without causing sampling splattering or complete drying out of the sample.

NOTE 3: Cooling the sample is performed to avoid potential splattering losses and resulting safety hazards caused by addition of reagents to a partially digested hot sample during subsequent processing steps. Samples do not have to be cooled completely to room temperature for safe further processing of paint chip samples. However, the operator must be aware that the potential for splattering losses and resulting safety hazards increases with increasing temperature of the sample digest.

6. Add 5 mL of concentrated HNO_3 and 2.5 mL 30% H_2O_2, and re-cover with a watch glass.

7. Gently reflux on a hot plate for about 15 min (see Note 1).

8. Remove the watch glass and evaporate gently on a hot plate until the sample volume is reduced to about 1 to 2 mL (see Note 2).

9. Replace the watch glass and remove the beaker containing sample from the hot plate and allow it to cool (see Note 3).

10. Add 5 mL of concentrated HNO_3 and 2.5 mL 30% H_2O_2, and recover with a watch glass.

11. Gently reflux on a hot plate for about 15 min (see Note 1).

12. Remove the watch glass and evaporate gently on a hot plate until the sample volume is reduced to about 1 to 2 mL (see Note 2).

13. Replace the watch glass and remove the beaker containing sample from the hot plate and allow it to cool (see Note 3).

14. Rinse the watch glass and beaker walls with 3 to 5 mL of 10% HNO_3 into the beaker.

15. Remove the watch glass and evaporate gently on a hot plate until the sample volume is reduced to about 1 to 2 mL (see Note 2).

16. Replace the watch glass and cool to room temperature.

17. Add 1 mL concentrate HNO_3 to the residue; swirl to dissolve soluble species.

18. Use a wash bottle filled with ASTM Type I water, rinse the beaker walls and underside of the watch glass with Type I water into the beaker.

19. Quantitatively transfer the digested sample into a 250-mL volumetric flask using several rinses with ASTM Type I water (see Note 4). A plastic or glass funnel should be used to avoid spillage during transfer from the beaker to the volumetric flask.

20. Dilute to volume with ASTM Type I water and mix thoroughly. The sample digest contains approximately 1% (v/v) HNO_3.

21. Portions used for analysis must be filtered or centrifuged prior to instrumental measurement to remove undissolved material. Instrumental measurement should be performed using calibration standards that are matched to the same approximate acid levels as those in sample digest aliquot analyzed for analyte content.

NOTE 4: Due to potential losses during filtration, it is

recommended to filter samples after dilution to final volume. Additional volume consumed by undissolved material will not cause any significant bias.

APPENDIX G

FULL STUDY PROTOCOLS:
STANDARD TEST PROTOCOL FOR THE ANALYSIS OF DIGESTED SAMPLES FOR LEAD BY INDUCTIVELY COUPLED PLASMA-ATOMIC EMISSION SPECTROSCOPY (ICP-AES), FLAME ATOMIC ABSORPTION (FAAS), OR GRAPHITE FURNACE ATOMIC ABSORPTION (GFAAS) TECHNIQUES

STANDARD TEST PROTOCOL FOR THE ANALYSIS OF DIGESTED SAMPLES FOR LEAD BY INDUCTIVELY COUPLED PLASMA-ATOMIC EMISSION SPECTROSCOPY (ICP-AES), FLAME ATOMIC ABSORPTION (FAAS), OR GRAPHITE FURNACE ATOMIC ABSORPTION (GFAAS) TECHNIQUES

1.0 SUMMARY

A sample digestate is analyzed for Lead content using ICP-AES, Flame-AAS, or Graphite Furnace-AAS techniques. Instrumental Quality Control samples are analyzed along with sample digestates to assure adequate instrumental performance. This procedure is similar to SW-846 Method 6010. It is equivalent to the draft procedure currently under consideration in ASTM Subcommittee E06.23.

2.0 DEFINITIONS

2.1 *Digestion* - The sample preparation process which will solubilize targeted analytes present in the sample and results in an acidified aqueous solution called the digestate.

2.2 *Digestate* - An acidified aqueous solution which results from performing sample preparation (digestion) activities. Lead measurements are made using this solution.

2.3 *Batch* - A group of field with QC samples which are processed together using the same reagents and equipment.

2.4 *Serial Dilution* - A method of producing a less concentrated solution through one or more consecutive dilution steps. Dilution step for a standard or sample is performed by volumetrically placing a small aliquot of a higher concentrated solution into a volumetric flask and diluting to volume with water containing the same acid levels as found in original sample digestates.

2.5 *Method Blank* - A digestate which reflects the maximum treatment given any one sample within a sample batch except that it has no sample initially placed into the digestion vessel (the same reagents and processing conditions which are applied to field samples within a batch are also applied to the method blank). Analysis results from method blanks provide information on the level of potential contamination experienced by samples processed within the batch.

2.6 *No-Spiked Sample* - A portion of a homogenized sample which was targeted for addition of analyte but which is not fortified with all the target analytes before sample preparation. A method blank serves as a no-spike sample in cases where samples cannot be uniformly split as described in Section 2.7. Analysis results for this sample is used to correct for native analyte levels in the spiked and spiked duplicate samples.

2.7 *Spiked Sample and Spiked Duplicate Sample* - Two portions of a homogenized sample which were targeted for addition of analyte and are fortified with all the target analytes before preparation. In cases where samples cannot be uniformly split (such as paint chip samples taken for Lead per area determinations, a method blank can be used in place of the homogenized sample split. Use of a method blank for a spiked sample should be referred to as a "spiked method blank" or "spiked method blank duplicate." Analysis results for these samples are used to provide information on accuracy and precision of the overall analysis process.

2.8 *Analysis Run* - A period of measurement time on a given instrument during which data is calculated from a single calibration curve (or single set of curves). Re-calibration of a given instrument produces a new analysis run.

2.9 *Instrumental QC Standards* - Solutions analyzed during an instrumental analysis run which provide information on measurement performance during the instrumental analysis portion of the overall Lead measurement process.

2.10 *Semi-quantitative Screen* - An analysis run which is performed on highly diluted sample digestates for the purpose of determining the approximate analyte level in the digest. This analysis run is generally performed without inserting Instrumental QC standards except for calibration standards. Data from this run are used for determining serial dilution requirements for sample digestates to keep them within the linear range of the instrument.

2.11 *Quantitative Analysis* - An analysis run on sample digestates (or serial dilutions of sample digestates) which includes Instrumental QC standards. Data from this run are used to calculate and report final Lead analysis results.

2.12 *Initial Calibration Blank (ICB)* - A standard solution which contains no analyte and is used for initial calibration and zeroing instrument response. The ICB must be matrix matched to acid content present in sample digestates. The ICB must be measured during calibration and after calibration. The measured value is to be less than five times the instrumental detection limit.

2.13 *Calibration Standards* - Standard solutions used to calibrate instrument. Calibration Standards must be matrix matched to acid content present in sample digestates and must be measured prior to measuring any sample digestates.

2.14 *Initial Calibration Verification (ICV)* - A standard solution (or set of solutions) used to verify calibration standard levels. Concentration of analyte to be near mid-range of linear curve, which is made from a stock solution having a different manufacturer or manufacturer lot identification than the calibration standards. The ICV must be matrix matched to acid content present in sample digestates. The ICV must be measured after calibration and before measuring any sample digestates. The measured value to fall within $\pm$10% of known value.

2.15 *Interference Check Standard (ICS)* - A standard solution (or set of solutions) used for ICP-AES to verify

accurate analyte response in the presence of possible spectral interferences from other analytes present in samples. The concentration of analyte to be less than 25% of the highest calibration standard, concentrations of interferant will be 200 µg/Ml of Al, Ca, Fe, and Mg. The ICS must be matrix matched to acid content present in sample digestates. The ICS must be analyzed at least twice, once before, and once after all sample digestates. The measured analyte value is expected to be within ±20% of known value.

2.16 *Continuing Calibration Verification (CCV)* - A standard solution (or set of solutions) used to verify freedom from excessive instrumental drift. The concentration to be near mid-range of linear curve. The CCV must be matrix matched to acid content present in sample digestates. The CCV must be analyzed before and after all sample digestates and at a frequency not less than every ten sample digestates. The measured value to fall within ±10% of known value for ICP-AES or FAAS (±20% for GFAA), run once for every 10 samples.

2.17 *Continuing Calibration Blank (CCB)* - A standard solution which has no analyte and is used to verify blank response and freedom from carryover. The CCB must be analyzed after the CCV and after the ICS. The measured value is to be less than five times the instrumental detection limit.

3.0 APPARATUS AND MATERIALS

3.1 Analytical Instrumentation

3.1.1 Inductively Coupled Plasma-Atomic Emission Spectrometer (ICP-AES) - Either sequential or simultaneous, capable of measuring at least one of the primary ICP Lead emission lines. Emission line used must be demonstrated to have freedom from common major interferants such as Al, Ca, Fe, and Mg or the ability to correct for these interferants.

3.1.2 Flame Atomic Absorption Spectrometer (FAAS) - Equipped with an air-acetylene burner head, Lead hollow cathode lamp or equivalent, and capable of making Lead absorption measurements at the 283.3-nm absorption line.

NOTE: The 283.3-nm line is preferred over the 217-nm line because of the increased noise levels commonly observed at the 217-nm line for FAAS and GFAAS.

3.1.3 Graphite Furnace Atomic Absorption Spectrometer (GFAAS) - Equipped with background correction, Lead hollow cathode lamp or equivalent and capable of making Lead absorption measurements at the 283.3-nm absorption line.

3.2 Gases

Grades specified by manufacturer of the instrument employed.

3.2.1 Compressed air and acetylene for FAAS.

3.2.2 Compressed or liquid argon for ICP-AES and GFAAS.

3.2.3 Minimum of two-stage regulation of all gases.

3.3 Glassware and Miscellaneous Supplies

3.3.1 Vinyl gloves, powderless.

3.3.2 Micro-pipettors with disposable plastic tips, sizes needed to make reagent additions, and spiking standards. In general, the following sizes should be readily available: 1- to 5-mL adjustable, 1,000 µL, 500 µL, 250 µL, and 100 µL.

3.3.3 Volumetric flasks, sizes needed to make, calibration standards, serial dilutions, and Instrumental QC standards.

4.0 Reagents

4.1 *Nitric acid*, concentrated; reagent grade

4.2 *Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water as defined by Type 1 of Specification D1193 (ASTM Type I Water: Minimum resistance of 16.67 megohm-cm, or equivalent).

4.3 *Calibration stock solution*, 100 µg/mL of Pb in dilute nitric acid or equivalent (such as a multi-element stock

containing Pb).

4.4 *Check standard stock solution* (for ICV), 100 µg/mL of Pb in dilute nitric acid or equivalent. Must be sourced from a different lot number (or manufacturer) than the Calibration stock solution (7.3).

4.5 *Interferant stock solution* (for ICS; ICP-AES only), 10,000 µg/mL of Al, Ca, Fe, and Mg in dilute nitric acid or equivalent.

5.0 PROCEDURE

5.1 *Laboratory Records*—Record all reagent sources (lot numbers) used for sample preparation in a laboratory notebook. Record any inadvertent deviations, unusual happenings, or observations on a real-time basis as samples are processed. Use these records to add supplement Lead data when reporting results.

5.2 *Instrumental Setup*

5.2.1 *FAAS/GFAAS* - Set the FAAS or GFAAS spectrometer up for the analysis of Lead at 283.3 nm, according to the instructions given by the manufacturer. Be sure to allow at least a 30-min warmup of the hollow cathode lamp prior to starting calibration and analysis.

5.2.2 *ICP-AES* - Set the ICP spectrometer up for the analysis of Lead at a primary Lead emission line (such as 220.2 nm), according to the instructions given by the manufacturer. Be sure to allow at least a 30-min warmup of the system prior to starting calibration and analysis.

5.3 *Preparation of Calibration and Instrumental QC Standards*

5.3.1 *Calibration Standards* - Prepare a series of calibration standards covering the linear range of the instrumentation. Prepare these standards using serial dilution from the calibration stock solutions. Prepare these standards using the same final nitric acid concentration present in the sample digestates. Also prepare an Initial Calibration Blank (ICB) as defined in Section 3 and Table F-1.

NOTE: For FAAS/GFAAS prepare a minimum of three calibration standards plus the ICB for performing calibration of the instrument. ICP-AES can be performed using one high calibration standard and an ICB. However, more are generally preferred.

5.3.2 *Instrumental QC Standards* - Prepare Instrumental QC standards as summarized in Table F-1 using serial dilution from the required stock solutions. Prepare these standards using the same final nitric acid concentration present in the sample digestates.

NOTE: The ICV is used to assess the accuracy of the calibration standards. Therefore, it must be made from a different original source of stock solution than the stock used to make the calibration standards. Use of a different serial dilution of the same original stock is not acceptable.

5.4 *Calibration and Instrumental Measurement* - Perform calibration and quantitative Lead measurement of sample digestates and instrumental QC samples in the sequential order outlined in Table F-2.

NOTE: Performance of a semi-quantitative screen prior to quantitative analysis for sample digests containing unknown levels of Lead generally recommended. The purpose of this screen is to determine serial dilution requirements of each digestate needed to keep the instrumental response within the calibration curve. During a semi-quantitative screen all digestates are diluted to a constant large value (1-to-100 for ICP/FAAS and 1-to-1000 for GFAAS). The instrument is calibrated and diluted digestates are analyzed without inserting the instrumental QC used for a Quantitative analysis run. Data from this screen are reviewed to calculate the optimum serial dilution needed for each digestate. <u>No sample data can be reported for any analyte value not falling within the calibration range</u>. Therefore, the optimum dilution is one which achieves the maximum Lead response which is still within the calibration curve. For ICP-AES, levels of possible interferants (Al, Ca, Fe, and Mg) also may have to be considered in order to make interference corrections. For ICP-AES, digestates must be sufficiently diluted to

assure that levels of possible interferants such as Al, Ca, Fe, and Mg are at or below the levels present in the ICS.

5.5 *Instrumental QC Evaluation and Corrective Action* - Examine the data generated from the analysis of calibration standards and Instrumental QC standards. Evaluate the analysis run using the criteria shown in Table F-1. Failure to achieve the specifications shown in Table F-1 will require corrective action to be performed as described below:

5.5.1 *ICB, Calibration Standards, or ICV* - Failure to meet specifications for these Instrumental QC standards requires complete re-calibration. Sample digestates cannot be measured under these conditions. It is recommended that standards be re-prepared prior to re-calibration.

5.5.2 *High Calibration Standard Re-run* - Failure to meet specifications for this Instrumental QC standard requires complete re-calibration. Sample digestates cannot be measured under these conditions. It is recommended that standard range be reduced prior to re-calibration.

5.5.3 *ICS* - Failure to meet specifications for these Instrumental QC standards requires reanalysis of the standard until specifications are met. Sample digestates cannot be measured under these conditions. Re-preparation of the standard prior to reanalysis is recommended under these conditions. Continued failure of the ICS may require interference correction investigation or changing of instrument parameters. Consult the manufacturer's recommendations under these conditions. Any change in instrument parameters must be accompanied by re-calibration. If measured aliquots of sample digestates can be shown not to contain interferants as high as those recommended for the ICS making, then the interference levels in the ICS can be lowered. Such changes must be documented in laboratory records with data supporting the justification for the change. All measurements on sample digests must be bracketed by an ICS which meets specifications (called a "passing" ICS). Failure to meet specifications on the ICS run after the sample digestates requires rerunning of all sample digestates since the last passing ICS was measured. Since the ICS only is required to be analyzed twice, much data could be lost if the analytical run were long and the second ICS failed specifications. This is good reason for including periodic analysis of the ICS as shown in

Table F-2.

5.5.4 *CCV* - Failure to meet specifications for these Instrumental QC standards indicates excessive instrumental drift. Sample digestates cannot be measured under these conditions and any sample digestates measured since the last passing CCV must be reanalyzed. This situation requires either reanalysis of the standard until specifications are met or re-calibration. All measurements on sample digests must be bracketed by an CCV which meets specifications.

5.5.5 *CCB* - Failure to meet specifications for these Instrumental QC standards indicates the presence of possible instrumental carryover or baseline shift. Such a failure will have the most impact on sample digestates at the lower end of the calibration curve. The first corrective action is to reanalyze the CCB. If the CCB passes, then the rinse time between the samples should be increased and the analysis continued. If the instrument response is still elevated and has not significantly changed, then the instrument can be re-zeroed followed by a CCV-CCB and reanalysis of all samples since the last passing CCB which are within 5 times the response of the failed CCB.

6.0 CALCULATIONS

For FAAS/GFAAS : Prepare a calibration curve to convert instrument response (absorbance) to concentration (µg/mL) using a linear regression fit. Convert all instrumental measurements on instrumental QC standards and sample digests to Lead concentration (µg/mL) using the calibration curve.

NOTE: Some instruments will automatically prepare a calibration curve based on a linear regression fit.

For ICP-AES: All modern ICPs automatically prepare a calibration curve to convert instrumental responses (emission intensity) to concentration (µg/mL).

TABLE G-1. SUMMARY OF LABORATORY INSTRUMENTAL MEASUREMENT QC STANDARDS

Name	Use	Specification
ICB - Initial Calibration Blank	Used for initial calibration and zeroing instrument response.	Calibration Standard which contains no lead. Must be measured during calibration and after calibration. Measured value to be less than 5 times the IDL.
Calibration Standards	Used to Calibrate instrument. The high standard re-run is used to check for response linearity.	Acid content must be approximately the same as that in the sample digests. Must be measured prior to measuring any sample digests. Correlation Coefficient of $\geq$0.995, as measured using linear regression on instrument response(y) versus concentration(x). The highest level Calibration standard must be measured after calibration. The measured value to fall within $\pm$10% of known value.
ICV - Initial Calibration Verification	Used to verify calibration standard levels.	Concentration of lead to be near the middle of calibration curve. It is made from a stock solution having a different manufacturer or manufacturer lot identification than the calibration standards. Must be measured after calibration and before measuring any sample digests. Measured value to fall within $\pm$10% of known value.
ICS - Interference Check Standard	Used to verify accurate lead response in the presence of possible spectral interferences from other analytes present in samples.	Concentration of lead to be less than 25% of the highest calibration standard, concentrations of interferant are 200 µg/mL of Al, Ca, Fe, and Mg. Must be analyzed at least twice, once before and once after all sample digestates. Measured lead value to fall within ±20% of known value.
CCV - Continuing Calibration Verification	Used to verify freedom from excessive instrumental drift.	Concentration to be near the middle of the calibration curve. Must be analyzed before and after all sample digestates and at a frequency not less than once every ten samples. Measured value to fall within ±10% of known value.
CCB - Continuing Calibration Blank	Used to verify blank response and freedom from carryover.	Calibration Standard which contains no lead. Must be analyzed after each CCV and each ICS. Measured value to be less than 5 times the instrumental detection limit.

TABLE G-2. EXAMPLE OF A TYPICAL ANALYSIS ORDER FOR MEASUREMENT			
Run Order No. (relative)	**Sample ID**	**Comments**	
1	ICB	Calibration Blank	Instrument Calibration
2-4	low, med, high	Calibration Standards	
5	ICB	Calibration Blank	Calibration Verification
6	ICV	made from different stock, level is near mid-point of curve	
7	high standard	Calibration Standard	Linearity Check
8	CCB	Same as Calibration Blank	
9	ICS	Interference Check Standard	Interferant check for ICP only
10	CCB	Carryover Check	
11	CCV	Drift Check, same as near midpoint calibration standard	Continuing Calibration Verification
12	CCB	Carryover check	
*** start repeating cycle of samples-Instrumental QC here ***			
13-22	Sample IDs	Sample digestates	Max. of 10 samples
23-24	CCV CCB	Drift Check + Carryover Check	See run # 11-12
25-34	Sample IDs	Sample digestates	Max. of 10 samples
35-36	ICS CCB	Interferant Check + Carryover Check	See run # 9-10
37-38	CCV CCB	Drift Check + Carryover Check	See run # 11-12
*** end repeating cycle of samples-QC standards here ***			

APPENDIX H

FULL STUDY PROTOCOLS:
PROTOCOL FOR PACKAGING AND SHIPPING OF SAMPLES FROM THE FIELD

PROTOCOL FOR PACKAGING AND SHIPPING OF SAMPLES

1.0 INTRODUCTION

Collection and analysis of paint chip samples as specified by the QAPjP will require packaging and shipping of samples from sampling sites. The field team will be responsible for packaging and shipping the samples from each sampling site to the Sample Custodian at MRI. The following are protocols for packaging and shipping samples from the field.

2.0 SAMPLE PACKAGING PROTOCOL

The field team is responsible for preparing the samples for shipment back to MRI. Samples that are collected will be shipped on a routine basis by the acting field supervisor. The same shipping container that was used to ship sample collection containers to the field will generally be used to ship them back to MRI. All sampling materials will be packaged in accordance with Department of Transportation (DOT) regulations. The field team will include copies of the field sampling forms with the samples to identify the contents of the shipping containers. The original field sampling forms will be held by the field supervisor and, ultimately, hand carried to MRI. <u>Do not send original copies of sample data forms or other important records with the samples.</u>

3.0 SAMPLE SHIPPING METHODS

All samples will be shipped to MRI via Federal Express Economy Distribution Service in accordance with DOT shipping regulations. The MRI field team will be responsible for making the shipping arrangement with the local Federal Express office. Pre-printed Federal Express Air Bills can be obtained from the MRI Shipping and Receiving Department. All Federal Express shipments will use the standard Federal Express Air Bill. For further details, consult with MRI's S & R Department.

APPENDIX I

FULL STUDY PROTOCOLS:
GLASSWARE/PLASTICWARE CLEANING PROCEDURE

INFORMATION NOT PRESENT : PROPRIETARY INFORMATION

APPENDIX J

FULL STUDY PROTOCOLS:
ACID BATH CLEANING PROCEDURES

INFORMATION NOT PRESENT : PROPRIETARY INFORMATION

APPENDIX AA

PILOT STUDY PROTOCOLS:
SELECTION OF MEASUREMENT AND SAMPLING LOCATIONS

SELECTION OF MEASUREMENT AND SAMPLING LOCATIONS

1.0 INITIAL SURVEY

Selection of interior and exterior sampling sites will be made from as many painted substrate types as can be found in the test structure (wood, plaster, drywall, brick, steel, masonry).

The field team Leader (field statistician, provided by David C. Cox & Associates) will be responsible for drawing a rough floor plan of the targeted structure, selecting and marking the sampling locations within the structure, indicating the sampling locations on the floor plan, making a backup copy of the floor plan, and posting the floor plan for use by other field crew personnel.

The field team Leader will be responsible for drawing a rough plan of the exterior of the structure, selecting and marking exterior sampling locations, indicating the exterior sampling locations on the drawings, making a backup copy of the drawings, and posting the exterior drawings for use by other field crew personnel. The David C. Cox & Associates field team Leader will also assist the MRI supervisor during the course of the field sampling efforts.

Sampling locations corresponding to those portrayed on the drawings will be outlined on the sampling location with marking pen. A typical testing location will be a rectangle approximately 4 inches high by 14 inches long as shown in Figure A-1. The rectangle will be divided into 2 squares approximately 4 inches by 4 inches, and 6 rectangles approximately 4 inches high by 1 inch wide. The field team Leader will mark one of the 4-inch squares with an "X" for use in XRF paint surface measurements, and the other 4-inch square with an "L" for use in side by side paint chip collection. In addition, the "L" square will be subdivided into 4 smaller squares. Two of these squares will be marked with arrows to denote sub-squares to be sampled. The 6 smaller rectangles will be marked with an "S" to designate use for test-kit sampling. For components where a 4" x 14" rectangle cannot be obtained, the field statistician will exercise judgement in defining a comparable sampling area. Wood trim/baseboard/mantles, brickwork, metal trim and beams, and other such narrow, or otherwise irregular surfaces must be marked for sampling on a case-by-case basis.

Figure A-1. Testing Location Template.

The field team Leader will be responsible for attaching the correct bar-code set corresponding to each location. The bar codes will be removed by the various samplers and applied to the individual's test results data form at the time the test is performed. The team Leader will attempt to numerically order the sampling locations so that all locations with the same substrate material will be tested sequentially by the XRF instruments. The order in which the substrates are tested in the pilot will be: wood, drywall, plaster, concrete, and metal. This ensures that denser substrates will be tested towards the end of the day in order to minimize operational problems with the XRF instruments. Test kit operators will follow a staggered starting sequence so that the 6 kits will not always be tested in the same order.

APPENDIX BB

PILOT STUDY PROTOCOLS:
MEASUREMENT PROTOCOLS FOR XRF TESTING

MEASUREMENT PROTOCOLS FOR XRF TESTING

1.0 SUMMARY

This document describes the standard protocol for collecting XRF measurement data on painted surfaces and corresponding substrate surfaces. This standard also includes instructions for recording the measurements, making QC checks and data release requirements for two different classes of instruments: Direct Readers and Spectrum Analyzers.

2.0 MATERIALS AND EQUIPMENT

- Portable field XRF instrument with any extra required supporting equipment. (To be provided by XRF contractor.)
- One set of NIST paint films (SRM 2579); contains 5 films of different Lead levels. (To be provided by XRF contractor.)
- Reporting forms; see attached. (To be provided by MRI.)
- Dosimeter Badges; one for each XRF operator and one for each individual working within the same unit where XRF testing takes place. (Operator badge to be provided by XRF contractor, MRI will provide any other needed badges).
- Adhesive labels or barcode labels for identifying samples. (To be provided by MRI, will be available at each sampling location.)
- Waterproof (indelible) permanent marking pen. (To be provided by MRI, will be available at site.)
- Watch, clock or other equivalent timepiece. (Each member in the field will be required to have a timepiece for reporting the sampling times on the data forms.)
- Sling Psychrometer; or equivalent for room temperature and relative humidity measurements. (To be provided by MRI, will be available at site.)
- Pre-moistened wipes for cleaning of tools or hands. (To be provided by MRI, will be available at site.)
- QC test blocks, each approximately 4"x4", loaded on wheeled type carrier; thicknesses are approximate: 3/4" wood (pine), 2" concrete (with aggregate), 1/2" sheet rock, 20-25 gauge metal, 1" plaster and 12" thick styrofoam block. (To be provided and numbered by MRI,

will be available at site.)

- ! One 12" thick styrofoam block for holding QC test blocks under measurement. (To be provided and numbered by MRI, will be available at site.)

3.0 MEASUREMENT PROCEDURES FOR DIRECT READERS

3.1 BEGINNING OF EACH DAY ON SITE PROCEDURES

At the beginning of the sampling day at a given site, perform whatever tests and instrument checks are required by the manufacturer of the XRF to prepare the instrument for taking Lead measurements. In addition, perform the initial drift check determinations procedure as described in Section 3.4.1.

3.2 PROCEDURE FOR NORMAL MEASUREMENTS AT EACH SAMPLING LOCATION

At each sampling location perform the following steps:

1. For each new "XRF DATA-DIRECT READERS" form needed (see attached), complete the header of the form.

2. Record the sampling location/identification (ID) on an open line of the form. Use barcode labels corresponding to the specific sampling location whenever possible. These barcode labels should be present in close proximity to the sampling location marked by the field team leader (see Note 1).

3. Perform whatever normal instrument checks are required by the manufacturer of the XRF to prepare the instrument for taking Lead measurements.

4. Perform 3 read cycles each on two surfaces as follows:

   - ! Perform 3 read cycles on the painted surface at the sampling location. Record each read cycle on the "XRF DATA-DIRECT READERS" form along with the other information requested on the form.

   - ! Perform 3 read cycles on the exposed substrate surface covered with the <u>1.02</u> mg/cm² NIST standard film (red) at the sampling location. Record each read cycle on the "XRF DATA-DIRECT READERS" form along with the other information requested on the form.

IF the exposed substrate is concrete,

THEN perform 3 additional read cycles on the exposed substrate as follows:

! Perform 3 read cycles on the exposed substrate surface covered with the 3.53 mg/cm² NIST standard film at the sampling location. Record each read cycle on the "XRF DATA-DIRECT READERS" form along with the other information requested on the form.

NOTE: The sampling location will be marked in advance by the field team leader using a dark colored marking pen. The marking will be in the form of squares and rectangles with letters. The painted surface location to be used for XRF measurements will be indicated with an "X" placed adjacent to a large square or rectangle. The exposed substrate surface location to be used for XRF measurements will be the largest exposed area present at the sampling location.

5. **Special location requirements:**

IF, during testing activities, the following conditions exist:

! The location is marked as a "SPECIAL" location;

THEN perform the **SPECIAL MEASUREMENTS** as described in the Section 3.3

6. **Continuing QC drift check requirements:**

IF, during testing activities, the following conditions exist:

! The surface substrate is of a different type than the previous location.

THEN perform the **CONTINUING DRIFT CHECKS** as described in the Section 3.4.2

7. **End of day QC drift check requirements:**

IF, during testing activities, the following conditions exist:

- **!** All surfaces to be measured in a given day have been completed (end of sampling day);

THEN perform the **END OF DAY DRIFT CHECKS** as described in the Section 3.4.1

8. **QC variability check requirements:**

IF, during testing activities, the following conditions exist:

- **!** The surface substrate is of a different type than the previous location;

THEN perform a **QC VARIABILITY CHECK** as described in the Section 3.5

3.3 PROCEDURE FOR SPECIAL MEASUREMENTS AT SPECIFIC SAMPLING LOCATIONS

At specially marked sampling locations perform the following steps:

1. For each new "XRF DATA, SPECIAL LOCATIONS-DIRECT READERS" form needed (see attached), complete the header of the form.

2. Record the sampling location/identification (ID) on an open line of the form. Use barcode labels corresponding to the specific sampling location when ever possible. These barcode labels should be present in close proximity to the sampling location marked by the field team leader.

3. Perform what ever normal instrument checks are required by the manufacturer of the XRF to prepare the instrument for taking Lead measurements.

4. Perform 4 read cycles each on two surfaces (total of 8 readings) as follows:

- **!** Perform 4 read cycles on the painted surface at the sampling location. Record each read cycle on the "XRF DATA SPECIAL LOCATIONS-DIRECT READERS" form along with the other information requested on the form.

- **!** Perform 4 read cycles on the exposed substrate surface covered with the 1.02 mg/cm² NIST standard film (red) at the sampling location. Record each read cycle on the "XRF DATA, SPECIAL LOCATIONS-DIRECT READERS" form along with the other information requested on the form.

IF the exposed substrate is concrete,

THEN perform 4 additional read cycles on the exposed substrate as follows:

- **!** Perform 4 read cycles on the exposed substrate surface covered with the 3.53 mg/cm² NIST standard film at the sampling location. Record each read cycle on the "XRF DATA, SPECIAL LOCATIONS-DIRECT READERS" form along with the other information requested on the form.

NOTE: The special sampling locations will be marked in advance by the field team leader using a dark colored marking pen. The word "SPECIAL" in addition to squares and rectangles will be present at the location.

3.4 QC DRIFT CHECK PROCEDURES

3.4.1 INITIAL AND END OF DAY DRIFT CHECK DETERMINATIONS

At the beginning and end of sampling day, the instrument response on several test surfaces must be determined for use as a reference point to monitor instrumental drift. Make this determination as described below:

1. Complete the header of a new "XRF QC DATA: INITIAL\END DRIFT CHECK-DIRECT READERS" form.

2. Perform and record room temperature and humidity measurements in the same general vicinity as intended for the first sampling location.

3. Determine the reading of the XRF instrument for a NIST standard film placed on each of six test blocks as described below:

   Perform 9 read cycles on <u>each</u> test block (total of 54 readings) as follows:

   ! Place the QC test block on a 12" thick (nominal thickness) styrofoam block. Place the <u>1.02</u> mg/cm² NIST standard film (red) on the block. Perform 3 sets of 3 read cycles each down through the film and record each reading on the "XRF QC DATA: INITIAL\END DRIFT CHECK-DIRECT READERS" form.

   Perform 9 additional read cycles on the <u>concrete</u> test block as follows:

   ! Place the QC test block on a 12" thick (nominal thickness) styrofoam block. Place the <u>3.53</u> mg/cm² NIST standard film on the block. Perform 3 sets of 3 read cycles each down through the film and record each reading on the "XRF QC DATA: INITIAL\END DRIFT CHECK-DIRECT READERS" form.

3.4.2 CONTINUING DRIFT CHECKS PROCEDURES

Perform continuing drift checks as described below:

1. For each new "XRF QC DATA: CONTINUING DRIFT CHECKS-DIRECT READERS" form needed, complete the header of the form.

2. Perform and record room temperature and humidity measurements in the same general vicinity as intended for the first sampling location.

3. Determine the reading of the XRF instrument for a NIST standard film placed on <u>one</u> test block as described below:

   Use the test block that represents the closest match to the new sample location. Perform 3 read cycles on the selected test block as follows:

   ! Place the QC test block on a 12" thick (nominal thickness) styrofoam block. Place the <u>1.02</u> mg/cm² NIST standard film (red) on the block. Perform 3 read cycles

down through the film and record each reading on the "XRF QC DATA: CONTINUING DRIFT CHECKS-DIRECT READERS" form.

IF the selected test block is concrete,

THEN perform 3 additional read cycles on the test block as follows:

! Place the QC test block on a 12" thick (nominal thickness) styrofoam block. Place the 3.53 mg/cm² NIST standard film on the block. Perform 3 read cycles down through the film and record each reading on the "XRF QC DATA: CONTINUING DRIFT CHECKS-DIRECT READERS" form.

3.5 QC VARIABILITY CHECK PROCEDURES

Perform a **QC VARIABILITY CHECK** as follows:

1. Repeat the normal testing measurements at each location as described in section 3.2 step 4. five more times for a total of 6 separate Lead tests on both the painted and substrate surfaces at that location.

   Record the data on the "XRF DATA-DIRECT READERS" form by using the open lines directly below (in sequence) the original test data for that sampling location. Fill in the "Location ID" using a pen (use arrow or ") to indicate the same ID number as above whenever possible (avoid using up barcode labels for the QC variability checks).

   Mark the "Comments" column with a "QC-VC" to indicate that the data on that line is a QC variability check for the location.

4.0 MEASUREMENT PROCEDURES FOR SPECTRUM ANALYZERS

4.1 BEGINNING OF EACH DAY ON SITE PROCEDURES

At the beginning of the sampling day at a given site, perform whatever tests and instrument checks are required by the manufacturer of the XRF to prepare the instrument for taking Lead measurements. In addition, perform the initial drift check determinations procedure as described in Section 4.4.1.

4.2 PROCEDURE FOR NORMAL MEASUREMENTS AT EACH SAMPLING LOCATION

At each sampling location perform the following steps:

1. For each new "XRF DATA-SPECTRUM ANALYZERS" form needed (see attached), complete the header of the form.

2. Record the sampling location/identification (ID) on an open line of the form. Use barcode labels corresponding to the specific sampling location whenever possible. These barcode labels should be present in close proximity to the sampling location marked by the field team leader.

3. Perform whatever normal instrument checks are required by the manufacturer of the XRF to prepare the instrument for taking Lead measurements.

4. Perform 1 measurement each on two surfaces (total of 2 measurements) as follows:

   ! Perform 1 test mode reading on the painted surface at the sampling location. Record the data on the "XRF DATA-SPECTRUM ANALYZERS" form along with the other information requested on the form.

   ! Perform 1 test mode reading on the exposed substrate surface covered with the 1.02 mg/cm² NIST standard film (red) at the sampling location. Record the data on the "XRF DATA-SPECTRUM ANALYZERS" form along with the other information requested on the form.

   IF the exposed substrate is concrete,

   THEN perform 1 additional reading on the exposed substrate as follows:

   ! Perform 1 test mode reading on the exposed substrate surface covered with the 3.53 mg/cm² NIST standard film at the sampling location. Record the data on the "XRF DATA-SPECTRUM ANALYZERS" form along with the other information requested on the form.

   NOTE: The sampling location will be marked in advance by the field team leader using a dark colored marking pen. The marking will be in the form of squares and

rectangles with letters. The painted surface location to be used for XRF measurements will be indicated with an "X" placed adjacent to a large square or rectangle. The exposed substrate surface location to be used for XRF measurements will be the largest exposed area present at the sampling location.

5. **Special Location requirements:**

IF, during testing activities, the following conditions exist:

! The location is marked as a "SPECIAL" location;

THEN perform the **SPECIAL MEASUREMENTS** as described in the Section 4.3

6. **Continuing QC Drift Check requirements:**

IF, during testing activities, the following conditions exist:

! The surface substrate is of a different type than the previous location.

THEN perform the **CONTINUING DRIFT CHECKS** as described in the Section 4.4.2

7. **End of Day QC Drift Check requirements:**

IF, during testing activities, the following conditions exist:

! All surfaces to be measured in a given day have been completed (end of sampling day);

THEN perform the **END OF DAY DRIFT CHECKS** as described in the Section 4.4.1

8. **QC Variability Check requirements:**

IF, during testing activities, the following conditions exist:

! The surface substrate is of a different type than the previous location;

THEN perform a **QC VARIABILITY CHECK** as described in the Section 4.5

4.3 PROCEDURE FOR SPECIAL MEASUREMENTS AT SPECIFIC SAMPLING LOCATIONS

At each sampling location perform the following steps:

1. For each new "XRF DATA, SPECIAL LOCATIONS-SPECTRUM ANALYZERS" form needed (see attached), complete the header of the form.

2. Record the sampling location/identification (ID) on an open line of the form. Use barcode labels corresponding to the specific sampling location whenever possible. These barcode labels should be present in close proximity to the sampling location marked by the field team leader.

3. Perform whatever normal instrument checks are required by the manufacturer of the XRF to prepare the instrument for taking Lead measurements.

4. Perform 3 measurements each on two surfaces (total of 6 measurements) as follows:

   ! Perform 3 test mode readings on the painted surface at the sampling location. Record each reading on the "XRF DATA, SPECIAL LOCATIONS-SPECTRUM ANALYZERS" form along with the other information requested on the form.

   ! Perform 3 test mode readings on the exposed substrate surface covered with the <u>1.02</u> mg/cm² NIST standard film (red) at the sampling location. Record each reading on the "XRF DATA, SPECIAL LOCATIONS-SPECTRUM ANALYZERS" form along with the other information requested on the form.

   IF the exposed substrate is <u>concrete</u>,

   THEN perform 3 additional measurements on the exposed substrate as follows:

   ! Perform 3 test mode readings on the exposed substrate

surface covered with the 3.53 mg/cm² NIST standard film at the sampling location. Record each reading on the "XRF DATA, SPECIAL LOCATIONS-SPECTRUM ANALYZERS" form along with the other information requested on the form.

NOTE: The special sampling locations will be marked in advance by the field team leader using a dark colored marking pen. The word "SPECIAL" in addition to squares and rectangles will be present at the location.

4.4 QC DRIFT CHECK PROCEDURES

4.4.1 INITIAL AND END OF DAY DRIFT CHECK DETERMINATIONS

At the beginning and end of sampling day, the instrument response on several test surfaces must be determined for use as a reference point to monitor instrumental drift. Make this determination as described below:

1. Complete the header of a new "XRF QC DATA: INITIAL\END DRIFT CHECK-SPECTRUM ANALYZERS" form.

2. Perform and record room temperature and humidity measurements in the same general vicinity as intended for the first sampling location.

3. Determine the reading of the XRF instrument for a NIST standard film placed on each of five test blocks as described below:

   Perform 3 measurements on each test block (total of 15 readings) as follows:

   ! Place the QC test block on a 12" thick (nominal thickness) styrofoam block. Place the 1.02 mg/cm² NIST standard film (red) on the block. Perform 3 test mode readings down through the film and record each reading on the "XRF QC DATA: INITIAL\END DRIFT CHECK-SPECTRUM ANALYZERS" form.

   Perform 3 additional measurements on the concrete test block as follows:

- **!** Place the QC test block on a 12" thick (nominal thickness) styrofoam block. Place the 3.53 mg/cm² NIST standard film on the block. Perform 3 test mode reads down through the film and record each reading on the "XRF QC DATA: INITIAL\END DRIFT CHECK-SPECTRUM ANALYZERS" form.

4.4.2 CONTINUING DRIFT CHECKS PROCEDURES

Perform continuing drift checks as described below:

1. For each new "XRF QC DATA: CONTINUING DRIFT CHECKS-SPECTRUM ANALYZERS" form needed, complete the header of the form.

2. Perform and record room temperature and humidity measurements in the same general vicinity as intended for the first sampling location.

3. Determine the reading of the XRF instrument for a NIST standard film placed on one test block as described below:

   Use the test block that represents the closest match to the new sample location. Perform 1 measurements on the selected test block as follows:

   - **!** Place the QC test block on a 12" thick (nominal thickness) styrofoam block. Place the 1.02 mg/cm² NIST standard film (red) on the block. Perform 1 test mode reading down through the film and record the data on the "XRF QC DATA: CONTINUING DRIFT CHECKS-SPECTRUM ANALYZERS" form.

   IF the selected test block is concrete,

   THEN perform 1 additional measurement on the test block as follows:

   - **!** Place the QC test block on a 12" thick (nominal thickness) styrofoam block. Place the 3.53 mg/cm² NIST standard film on the block. Perform 1 test mode reading down through the film and record the data on the "XRF QC DATA: CONTINUING DRIFT CHECKS-SPECTRUM ANALYZERS" form.

4.5 QC VARIABILITY CHECK PROCEDURES

Perform a **QC VARIABILITY CHECK** as follows:

1. Repeat the normal testing measurements at each location as described in section 4.2 step 4. five more times for a total of 6 separate Lead tests on both the painted and substrate surfaces at that location.

   Record the data on the "XRF DATA-SPECTRUM ANALYZERS" form by using the open lines directly below (in sequence) the original test data for that sampling location. Fill in the "Location ID" using a pen (use arrow or ") to indicate the same ID number as above whenever possible (avoid using up barcode labels for the QC variability checks).

   Mark the "Comments" column with a "QC-VC" to indicate that the data on that line is a QC variability check for the location.

XRF Data - Spectrum Analyzers form

XRF QC Data: Initial/End Drift Check - Spectrum Analyzers form

XRF QC Data: Continuing Drift Check - Spectrum Analyzers form

XRF Data - Direct Readers form

XRF QC Data: Initial/End Drift Check - Direct Readers form

XRF QC Data: Continuing Drift Check - Direct Readers form

APPENDIX CC

PILOT STUDY PROTOCOLS:
MEASUREMENT PROTOCOLS FOR SPOT TEST KITS

MEASUREMENT PROTOCOLS FOR SPOT TEST KITS

1.0 SUMMARY

This appendix describes the pilot protocols which will be used with commercial test kits for testing in situ painted surfaces for Lead content. The chemistry and instructions vary from kit to kit but basic steps common to all kits are:

1. Select the area or item to be tested,
2. Prepare the test kit reagents,
3. Perform the quality control test included in the package,
4. Clean the surface to be tested,
5. Expose all layers of the paint by sanding or cutting,
6. Test the paint,
7. Record results of test, and
8. Hide tested surface from next tester.

The actual test methods involve reaction of Lead in the paint with the active reagent(s) in the test kit to produce a color change, a precipitate, or both. Methods of reacting the Lead with the reagents vary and include:

- Swabbing in situ with a reagent soaked applicator
- Pressing a reagent-impregnated pad to the in situ surface for a specified length of time
- Adding drops of one or more solutions to the in situ paint
- Removal of paint chip or dust to a vial to which reagents are added to produce the precipitate or color change

1.1 MATERIALS AND EQUIPMENT

Materials and equipment needs vary from kit-to-kit. Equipment and supplies are listed under the individual kit protocols.

1.2 TEST KITS SELECTED FOR THE STUDY

Five Lead test kits have been selected for inclusion in the Pilot Study. Table CC-1 lists the kits by manufacturer along with summary information. In addition to the kits listed in Table CC-1, a Massachusetts licensed Lead inspector will be contracted to perform Lead testing with the Massachusetts state-approved

sulfide reagents and procedures. The protocol for the Lead inspector will be the state-approved protocol. Although the Massachusetts protocol is not physically incorporated in this QAPjP, it is incorporated by reference.

TABLE CC-1. LEAD TEST KITS TARGETED FOR USE IN THE PILOT AND FULL STUDY

MANUFACTURER	KIT NAME	TEST	KIT METHOD CHOSEN
ENZONE	Lead-Zone	Proprietary	Reagent-impregnated pad
Frandon/Pace	Lead Alert	Rhodizonate	Home-owner in-situ notched paint layers
Frandon/Pace	Lead Alert	Rhodizonate	Professional-core sample paint chip
HybriVet Systems	Lead Check	Rhodizonate	Reagent-impregnated swabs
Innovative Synthesis	Lead Detective	Sodium Sulfide	Drop reagent into notched paint layers

2.0 TESTING PAINTED SURFACES FOR LEAD

In order to provide a reasonably uniform comparison of methods for this study, differences among the kit instructions preclude use of only the package-insert instructions for training and testing. For purposes of this Pilot Study, instructions supplied by the manufacturers were edited to conform to the eight steps listed above in the Appendix CC Summary.

2.1 ENZONE "LEAD-ZONE" (PROPRIETARY CHEMICAL COMPOSITION)

2.1.1 List of Supplies

Clipboard,
Map of dwelling and/or instructions from supervisor,
"Lead-Zone" WA57 field testing protocol,
Test Kit Results Recording Form (will be several pages),
Ball point pens (2),
Box of wet-wipes (200),

Disposable gloves (100 pr),
Lighted magnifying glass or other light source with magnifying glass,
Extension cord,
50 mL ASTM Type I water in dropping bottle,
Scissors,
Resealable plastic bags,
Trash bag,
Duct tape,
17 "Lead-Zone" test kits -- enough to perform 100 tests,
Stopwatch, and
Watch or other time piece.

2.1.2 Performing the Lead Zone Tests

Perform Lead testing in a safe manner as instructed in the training class including wearing of plastic gloves and safety glasses at all time, and leather gloves with respirator during cutting or scraping activities.

1. Obtain the "Lead-Zone" test kits, data recording forms, and supplies in the above list of supplies from the field supervisor.

2. Obtain sampling location instructions (starting point, other) from the field supervisor.

3. Fill out the header information on the data recording form.

4. Find the first location to be tested according to instructions received from the field supervisor. The location map will be provided by the supervisor, or alternately, may be posted in the dwelling.

5. Remove one barcode from the strip attached to the test location and affix it in the barcode column on the data recording form.

6. Prepare the test kit reagents. Take care not to contaminate the test pads or painted surfaces with Lead from the test spots on the verification card enclosed in the package!

a. Use scissors to cut each of the 2 Lead Zone Test Pads into 3 equal sized pieces, creating 6 smaller Lead zone test pads.
b. Store the test pad pieces in a resealable plastic bag until needed.

7. Perform the quality control (QC) test contained in the kit before the first location is tested and after each negative result to verify that the test reagents are working.

   a. Remove one test pad piece from the plastic bag
   b. Moisten the test pad with a few drops of ASTM Type I water
   c. Press the moistened pad against one of the test dots on the verification card. Hold the pad against the surface for two minutes. **IF** a pink to purple color develops on the test dot or pad (or both) the reagents are working correctly. If not, consult supervisor.

8. Clean the surface to be tested by wiping with a pre-moistened wipe.

9. Expose all layers of the paint by cutting through all paint layers down to the substrate. Use the bevelled V-cut as taught in the training class.

   a. Record observations of native colors of paint layers before testing.
   b. Record substrate type (e.g. wallboard, plaster, wood, metal, brick, masonry)

10. Test the exposed paint layers:

    a. Remove one test pad piece from the plastic bag
    b. Moisten the test pad with a few drops of ASTM Type I water
    c. Press the moistened pad against the exposed paint layers. Hold for two minutes. **IF** a pink to purple color develops in any of the paint layers, or on the test pad, the test is positive for Lead. If not, then the test is negative for Lead.

11. Record the test results -- color observed on pad, color

observed in any of the paint layers and any comments on the test form in the appropriate columns (use the lighted magnifying glass or equivalent to inspect for native and reagent-developed colors).

12. Cover the tested spot with duct tape to conceal the results from the next tester.

13. Test the remaining locations in the structure as instructed by the supervisor. Follow steps 5 through 13 until all locations in the structure have been tested. Record the temperature and relative humidity within the building at the beginning, end, and during the middle of the day. Use the comments column of the most current test kit data recording form to record this information. Six tests may be performed with each Lead-Zone kit. Use the verification cards prior to the first test in the structure and after any negative tests to verify that the reagents are working correctly. As long as positive tests are being obtained, it is not necessary to use the verification card for each kit opened. **IF** the test does not work on the painted surface (sampling location), consult the supervisor.

14. At the end of the testing day, check the test results recording form for completeness. Return the completed form and all supplies and remaining test kits to the supervisor.

Figure CC-1 is a photocopy of the Lead-Zone Lead Test Kit instructions provided with the test kit.

Lead Zone Test Kit—package insert copy removed because of

copyright considerations.

Figure CC-1 was presented on 1 page.

(Insert from packages obtained in March 1993 from Enzone Corporation, College Point, NY 11356)

Figure CC-1. Photocopy of Lead Zone Test Kit instructions.

2.2 FRANDON/PACE LEAD ALERT (RHODIZONATE)

2.2.1 List of Supplies

Clipboard,
Map of dwelling and/or instructions from supervisor,
"Lead-Alert" WA57 field testing protocol,
Test Kit Results Recording Form (will be several pages),
Ball point pens (2),
Box of wet-wipes (200),
Disposable gloves (100 pr),
Lighted magnifying glass or other light source with magnifying glass,
Extension cord,
Resealable plastic bags,
Trash bag,
Duct tape,
Scissors,
"Lead-Alert" homeowner test kits -- enough to perform 100 tests,
Stopwatch, and
Watch or other time piece.

2.2.2 Performing the "Lead-Alert" Test

Perform Lead testing in a safe manner as instructed in the training class. Although two types of tests can be performed with the Frandon Lead Alert Kit including wearing of plastic gloves and safety glasses at all times, and leather gloves with respirator during cutting or scraping activities-- a Surface test and an Underlying Layers test—only the Underlying Layers Test will be performed in this study.

1. Obtain the "Lead-Alert" test kits, data recording forms, and supplies from the field supervisor.

2. Obtain sample location instructions (starting point, other) from the field supervisor.

3. Fill out the header information on the data recording form.

4. Find the first location to be tested according to instructions received from the field supervisor. The location map will be provided by the supervisor, or

alternately, may be posted in the dwelling.

5. Remove one barcode from the strip attached to the test location and affix it in the barcode column on the data recording form.

6. Prepare the indicating solution:

   a. Remove red cap from plastic bottle labelled "Indicating Solution."
   b. Carefully remove the dropper insert by rolling/twisting it to the side.
   c. Open the tablet container and place only one tablet into the solution.
   d. Replace the dropper insert and the red cap and shake the bottle for one minute. Allow the bottle to stand for approximately five minutes and then shake it again until the solution turns yellow. The tablet will not be completely dissolved. This is normal.

7. Clean the test area with a pre-moistened wipe.

8. Perform a Positive Control Test (before the first test of the day and after each negative test on painted test areas in the structure)

   a. Apply two drops of Leaching Solution and two drops of Indicating solution to a cotton tipped applicator or test paper (avoid touching the dropper to any surface).
   b. Press the cotton tip or test paper on an unused test circle for 10-15 seconds
   c. Add two drops of Indicating Solution to the applicator or test paper. Do <u>not</u> touch the dropper to the applicator.
   d. Interpret the results. Use the lighted magnifying glass or equivalent to observe the color change. A pinkish to rose/red color is a positive test, indicating that the reagents are performing correctly. **IF** reagents are not performing correctly consult the supervisor. Record the results in the comments column of the data reporting form.

9. Perform the underlying layers test:

a. Cut through all layers of the paint down to the substrate with a bevelled V-notch[a]. Record native paint layer colors, substrate type (wallboard, plaster, wood, brick, metal, masonry) in appropriate blocks.
b. Apply two drops of Leaching Solution to a cotton tipped applicator or test paper
c. Rub the cotton tip or test paper on the exposed paint layers for 10-15 seconds.
d. Add two drops of Indicating Solution to the applicator or test paper. Do not touch the dropper to the applicator.
e. Interpret the results. Use the lighted magnifying glass or equivalent to observe the color change. Pinkish to rose/red color on the paint layers and/or the cotton tip (or test paper) is a positive test for Lead.

10. Record the results on the data form with any comments in the appropriate columns.

11. Cover the completed test spot with duct tape to conceal the results from the next tester.

12. Test the remaining locations in the structure as instructed by the supervisor. Follow steps 5 through 12 until all locations in the structure have been tested. Record the temperature and relative humidity within the building at the beginning, end, and during the middle of the day. Use the comments column of the most current test kit data results form to record this information. Use the Positive Control Strips only prior to the first test in the structure and after any negative tests to verify that the reagents are working correctly. After the initial Positive Control Strip test, it is not necessary to use the control strips with each kit opened as long as positive tests are being obtained on the painted surfaces and underlying layers. **IF** the test does not work on the painted surface (sampling location), consult the supervisor.

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13. At the end of the testing day, check the test results recording form for completeness. Return the completed form and all supplies and remaining test kit to the supervisor.

Figure CC-2 is a photocopy of the Frandon Lead-Alert kit instructions.

Frandon Lead Alert Kit package insert copy removed

because of copyright considerations.

Figure CC-2 was presented on 4 pages.

(Insert from packages obtained in March 1993 from Pace Environs, 207 Rutherglen Drive, Cary, NC 27511)

Figure CC-2. Photocopy of Frandon Lead-Alert Kit instructions.

2.3 FRANDON/PACE LEAD ALERT ALL-IN-ONE (RHODIZONATE)

2.3.1 List of Supplies

Clipboard,
Map of dwelling and/or instructions from supervisor,
"Lead-Alert" All-in-One WA57 field testing protocol,
Test Kit Results Recording Form (will be several pages),
Ball point pens (2),
Box of wet-wipes (200),
Disposable gloves (100 pr),
Lighted magnifying glass or other light source with magnifying glass,
Extension cord,
Resealable plastic bags,
Trash bag,
Duct tape,
Scissors,
"Lead-Alert" All-In-One test kits -- enough to perform 100 tests, and
Stopwatch.

2.3.2 Performing the "Lead-Alert" All-In-One Test

The Lead-Alert All-In-One kit contains three tests. Only one of the three -- removal of a paint chip and testing in the supplied vials -- will be performed in this study.

1. Obtain the "Lead-Alert" All-in-One test kits, data recording forms, and supplies from the field supervisor.

2. Obtain instructions (starting point, other) from the field supervisor.

3. Fill out the header information on the data recording form.

4. Find the first location to be tested according to instructions received from the field supervisor. The location map will be provided by the supervisor, or alternately, may be posted in the dwelling.

5. Remove one barcode from the strip attached to the test location and affix it in the barcode column on the data

recording form.

6. Prepare the indicating solution:

    a. Remove red cap from plastic bottle labelled "Indicating Solution."
    b. Carefully remove the dropper insert by rolling/twisting it to the side.
    c. Open the tablet container and place only one tablet into the solution.
    d. Replace the dropper insert and the red cap and shake the bottle for one minute. Allow the bottle to stand for five minutes and then shake it again until the solution turns yellow. The tablet will not be completely dissolved. This is normal.

7. Clean the test area with a pre-moistened wipe.

8. Perform a Positive Control Test (before the first test of the day and after each negative test on painted test areas in the structure).

    a. Apply two drops of Leaching Solution and two drops of Indicating solution to a cotton tipped applicator or test paper (do not touch the dropper to any surface).
    b. Press the cotton tip or test paper on an unused test circle for 10-15 seconds
    c. Add two drops of Indicating Solution to the applicator or test paper (do not touch the dropper to any surface).
    d. Interpret the results. Use the lighted magnifying glass or equivalent to observe the color change. A pinkish to rose/red color is a positive test, indicating that the reagents are performing correctly. **IF** the reagents are not performing correctly consult the supervisor. Record the results in the comments column of the data form.

9. Perform the Paint Chip Test

    a. Remove one of the adhesive backed collection papers and fold it in half. Apply the paper close to the area to be tested as shown in the package instructions.

b. Using the circular boring tool, cut down into the surface. Scrape the paint inside the circle onto the paper. Be sure to remove all layers of paint.
c. Transfer the paint from the paper to a plastic vial. Grind up the paint with a plastic rod for about 10 seconds (Lead paint grinds easily whereas Latex based paint will be harder to grind).
d. Add 3 drops of Leaching Solution to the vial (do **not** touch the dropper to the vial or contents) and grind the contents for another 10 seconds. Let the vial sit for 20 seconds.
e. Add 3 drops of Indicating Solution to the tip of an applicator (do not touch the applicator or any other surface with the dropper), then touch the surface of the liquid in the plastic vial with the tip of the applicator.
f. Interpret the results. Use the lighted magnifying glass or equivalent to observe the color changes. Pinkish to rose red color on the applicator tip is a positive test for Lead.

10. Record the results on the data form with any comments in the appropriate columns.

11. Cover the completed test spot with duct tape to conceal the results from the next tester.

12. Test the remaining locations in the structure as instructed by the supervisor. Follow steps 5 through 12 until all locations in the structure have been tested. Record the temperature and relative humidity within the building at the beginning, end, and during the middle of the day. Use the Positive Control Strips only prior to the first test in the structure and after any negative tests to verify that the reagents are working correctly. After the initial Positive Control Strip test, it is not necessary to use the control strips with each kit opened as long as positive tests are being obtained on the painted surfaces and underlying layers. **IF** the test does not work on the painted surface (sampling location), consult the supervisor.

13. At the end of the testing day, check the data recording form for completeness. Return the completed form and all supplies and remaining test kits to the supervisor.

A photocopy of the package instructions is shown in Figure CC-3.

Frandon Lead Alert Kit package insert copy removed

because of copyright considerations.

Figure CC-3 was presented on 4 pages.

(Insert from packages obtained in March 1993 from Pace Environs, 207 Rutherglen Drive, Cary, NC 27511)

Photocopy of Frandon Lead-Alert All-in-One Kit

Figure CC-3.

instructions.

2.4 LEAD DETECTIVE (Sodium Sulfide)

2.4.1 List of Supplies

Clipboard,
Map of dwelling and/or instructions from supervisor,
"Lead-Detective" WA57 field testing protocol,
Test Kit Results Recording Form (will be several pages),
Ball point pens (2),
Box of wet-wipes (200),
Disposable gloves (100 pr),
Disposable beakers, 10 mL,
Lighted magnifying glass or other light source with magnifying glass,
Extension cord,
Resealable plastic bags,
Trash bag,
Duct tape,
Scissors,
One "Lead-Detective" test kit,
Stopwatch, and
Watch or other time piece.

2.4.2 Performing the Lead Detective Tests

The Lead Detective kit detects Lead (and other heavy metals) by reacting with the Lead to form a black insoluble precipitate of Lead sulfide. Perform Lead testing in a safe manner as instructed in the training class including wearing of plastic gloves and safety glasses at all times, and leather gloves with respirator during cutting or scraping activities. The package instructions included with the Lead Detective are contained in a 33-page instruction booklet. A photocopy of this booklet is included in this Appendix CC as an attachment.

1. Obtain the "Lead-Detective" test kits, data recording forms, and supplies from the field supervisor.

2. Obtain sample location instructions (starting point, other) from the field supervisor.

3. Fill out the header information on the test form.

4. Find the first location to be tested according to instructions received from the field supervisor. The

location map will be provided by the supervisor, or alternately, may be posted in the dwelling.

5. Remove one barcode from the strip attached to the test location and affix it in the barcode column on the data recording form.

6. Carefully add the contents of the kit water bottle to the bottle containing the sodium sulfide crystals. Screw on the dropper cap and shake vigorously for 5 minutes or until the crystals are dissolved.

7. Perform the quality control check.

   a. Remove a quality control strip (or the paint chip) from the plastic bag

   b. While holding the strip in the forceps, Add a few drops of the sodium sulfide solution to the strip.

   c. **IF** black coloring appears, the QC test is positive and the reagents are working. Record the results in the comments column of the data report form. **IF** a black color does not appear, do not use the kit for testing. Consult the supervisor.

8. Clean the surface of the test location with a pre-moistened wipe.

9. Cut through all layers of the paint down to the substrate with a bevelled V-notch.

10. Add a few drops of the sodium sulfide solution to the notch, being very careful not to drip the reagent on the surfaces below or adjacent to the test spot. Use a plastic stirring rod or toothpick as needed to direct the solution into the notch[a].

11. Use the lighted magnifying glass or equivalent to observe the paint for changes. A black or gray color is a positive test for Lead.

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12. Record the results on the data form with any comments in the appropriate columns.

13. Cover the completed test spot with duct tape to conceal the results from the next tester.

14. Test the remaining locations in the structure as instructed by the supervisor. Follow steps 5 through 14 until all locations in the structure have been tested. Record the temperature and relative humidity within the building at the beginning, end, and during the middle of the day. Use the comments column of the most current testing data results form to record this information. One Lead Detective kit should be sufficient to test one structure (approximately 100 tests) Test the QC strip in the kit only prior to the first test in the structure and after any negative tests to verify that the reagents are working correctly. After the initial Positive Control Strip test, it is not necessary to use the control strips with each kit opened as long as positive tests are being obtained on the painted surfaces and underlying layers. **IF** the test does not work on the paint surface (sampling location), consult the supervisor.

15. At the end of the testing day, check the test results recording form for completeness. Return the completed form and all supplies and remaining test kits to the supervisor.

Lead Detective Lead Paint Detection Kit—booklet: package

insert copy removed because of copyright considerations.

Attachment to Appendix CC was presented on 20 pages.

(Insert from packages obtained in March 1993 from Innovative Synthesis Corporation, 1425 Beacon Street, Newton, MA 02168)

The Lead Detective, Lead Paint Detection

Appendix CC Attachment.

Kit (booklet)

2.5 LEAD CHECK SWABS

2.5.1 List of Supplies

Clipboard
Map of dwelling and/or instructions from supervisor,
"Lead-Check" swabs WA57 field testing protocol,
Test Kit Results Recording Form (will be several pages),
Ball point pens (2),
Box of wet-wipes (200),
Disposable gloves (100 pr),
Lighted magnifying glass or other light source with magnifying glass,
Extension cord,
Resealable plastic bags,
Trash bag,
Duct tape,
Scissors,
100 "Lead Check" swabs and several control cards,
Stopwatch, and
Watch or other time piece.

2.5.1 Performing the Lead Check Test

The Lead Check Swabs contain rhodizonate which reacts with Lead to form a pink to red color. Perform Lead testing in a safe manner as instructed in the training class including wearing of plastic gloves and safety glasses at all time, and leather gloves with respirator during cutting or scraping activities.

1. Obtain the "Lead-Check" rhodizonate test swabs, data recording forms, and supplies from the field supervisor.

2. Obtain sample location instructions (starting point, other) from the field supervisor.

3. Fill out the header information on the data recording form.

4. Find the first location to be tested according to instructions received from the field supervisor. The location map will be provided by the supervisor, or alternately, may be posted in the dwelling.

5. Remove one barcode from the strip attached to the test location and affix it in the barcode column on the data recording form.

6. Clean the test surface with a wet-wipe.

7. Cut a beveled V notch through all paint layers down to the substrate. Use the lighted magnifying glass to examine the paint layers revealed in the notch and record native paint colors and substrate information.

8. Remove one Lead Check Swab and reseal the package.

9. With the swab pointing up, squeeze points A and B to crush the internal glass ampoules .

10. With the swab pointing down, shake the swab twice, then gently squeeze it until the yellow liquid appears on the Swab tip.

11. While gently squeezing, rub the Swab tip on the test area for 30 seconds.

12. Observe Swab tip for coloration. Use the lighted magnifying glass or equivalent to read the results. Pink to red indicates positive test for Lead.

13. Record the results in the appropriate box on the data form.

    IF the test is positive for Lead, tape a plastic disposable beaker over the tested notch to conceal the test result from the next tester. **IF** no color change is observed within 2 minutes, touch the swab to one of the dots on the Lead confirmation card. **IF** no color develops on the QC dot, discard the swab and retest the paint layers (steps 8 through 13). **IF** color develops on the QC dot, tape a plastic disposable beaker over the tested notch and go on to the next spot. Record the time and return to re-observe this spot in approximately 30 minutes and if still no color change, cover and return to check the paint after approximately another 30 minute time period. **IF**, after 1 hour, no color has developed, the spot tested negative for Lead. Record all observations, subsequent examinations, and other comments

in the data form.

14. Test the remaining locations in the structure as instructed by the supervisor. Follow steps 5 through 14 until all locations in the structure have been tested. Record the temperature and relative humidity within the building at the beginning, end, and during the middle of the day. Use the comments column of the most current test kit data results form to record this information. Do not reuse any of the Swabs, even if no color change was observed. As long as positive tests are being obtained on the painted surfaces and underlying layers, there is no need to perform the Lead confirmation test on the test confirmation card. **IF** the test does not work on the painted surface (sampling location), consult the supervisor.

A photocopy of the Lead Check Swabs Test Kit package instructions is shown in Figure CC-4.

Lead Check Swabs—test kit package insert copy removed

because of copyright considerations.

Figure CC-4 was presented on 2 pages.

(Insert from packages obtained in March 1993 from Hybrivet Systems, Inc., P.O. Box 1210, Framingham, MA 01701)

Spot Test Kit Reporting Results Form

APPENDIX DD

PILOT STUDY PROTOCOLS:
COLLECTION OF PAINT CHIP SAMPLES IN AND AROUND BUILDINGS AND RELATED STRUCTURES

COLLECTION OF PAINT CHIP SAMPLES IN AND AROUND BUILDINGS AND RELATED STRUCTURES

SUMMARY

This document describes the standard protocol for obtaining a single paint chip sample from a painted substrate. This standard also includes instructions for sample storage and transport requirements.

MATERIALS AND EQUIPMENT

TABLE DD. EQUIPMENT-SUPPLIES LIST FOR COLLECTION OF PAINT CHIP SAMPLES

ITEM	NUMBER
Safety goggles	1/tester + 1 extra
Disposable gloves	150 pair/tester
Respirator with HEPA filters	1/tester
Single-edged razor blades	150/tester
Razor blade holder	1/tester + 1 extra
Cold chisels	several, various blade widths in duplicate
Hammer	1/tester
Wax Paper OR clean white paper, 8.5 x 11	150/tester
Masking tape	5 rolls, 1-inch
Duct tape	5 rolls, 2-inch
Marking pens	3/tester
Pencils	3/tester
Pencil sharpener	1 at site
Clip board	1/tester + 1 extra
Recording forms	Enough for 250 samples

TABLE DD. EQUIPMENT-SUPPLIES LIST FOR COLLECTION OF PAINT CHIP SAMPLES	
ITEM	**NUMBER**
Sample containers (plastic centrifuge tubes, plastic resealable bags)	250
Resealable bags for sample containers	250
Extra shipping container for paint chip samples	3
Trouble lights and spare bulbs	3
Extension cords	200 ft.
Magnifying glass with light source or lighted magnifying glass	1/tester
Power generator	1 at site
Small plane	1/tester
Pocket knife	2/tester + 1 extra
Wire brush	1/tester
Coarse soft bristle brush	2/tester
Heat gun	1/tester
Replacement heat gun element	1/tester
Metal paint chip collection tray	1/tester
Tool pouch with belt	1/tester
Face shield	1 at site
Fire extinguisher	2 at site
Note: Other items as needed.	

PROCEDURES

At each sampling location perform the following steps:

1. For each new "Paint Sampling Record" form needed (see

attached), complete the header of the form.

2. Record the sampling location/identification (ID) on an open line of the form. Use barcode labels corresponding to the specific sampling location when ever possible. These barcode labels should be present in close proximity to the sampling location marked by the field team leader.

4. Complete "Surface Type","Paint Surface Color" and "Is Surface Flat" sections of the Paint Chip Collection Reporting form. Any irregularities should be noted in the Comments column. The "Area Sampled with Units" column should be completed after the sample has been taken and can be accurately measured.

5. Affix an ID label to the outside of the container into which the sample is to be placed, and ensure that the label adheres well. If barcode labels are present at the sampling site, then affix 2 extra identical labels to the outside of the container for later use by the laboratory.

6. Don a pair of new vinyl gloves for removal of each paint sample for the laboratory.

7. Place the template (nominally 5 cm x 5 cm inside dimensions) over the sampling site and hold firmly, tape can be used to hold template in position. Do not place tape over adjacent areas marked for sampling. Using a cutting tool and the template as a guide, score the perimeter of the area to be removed. If it is impractical to use the template, the score can be made using a metal ruler as a guide. The area scored using the alternative method should be approximately equivalent to the area scored when using the template. Avoid using pencil or pen to mark the sample outline.

8. Affix a tray, paper funnel or equivalent collection device directly below the sampling location. The collection device should be located as close as possible to the sampling site but should not interfere with the removal procedure. If a paper funnel is used, either fold and tape closed the bottom of the funnel or affix a labeled open sampling container to the bottom of the funnel in a manner that will result in collection of the paint directly into the container. The collection device should be firmly secured to avoid being upset.

9. Using the appropriate cutting tool for a particular substrate or condition of the sample site, begin removing the paint from the substrate. If possible peel the paint off of the substrate by sliding the blade along the score and underneath the paint. Remove all paint down to the bare substrate.

   Avoid the inclusion of the substrate in the collection device. If substrate does fall into the collection device, remove only that substrate which can be easily removed without losing any of the paint sample. Do not remove any substrate which cannot be separated from the paint sample. The laboratory will remove extraneous substrate if possible, under laboratory conditions.

   If problems are encountered in removing the paint sample, other tools may be used. The use of a heat gun may facilitate the removal process. Extreme caution should be exercised when using the heat gun. Do not overheat the sample area, heat only until the paint becomes soft and supple. If the paint does not become soft and supple in a minute or two, discontinue the use of heat and try another means to remove the sample.

   In areas where extreme difficulty is experienced in removing the paint sample, consult with the field supervisor for advice.

10. If sample is not directly collected in the sample container using the funnel approach, transfer the collected paint sample to the sample container and seal. Exercise care to insure that <u>all</u> paint taken from the recorded area is placed into the sample container.

    If the funnel procedure was used, make sure all of the sample is in the collection container. Seal the container.

11. Carefully and accurately measure the sampling area dimensions. Do not attempt to calculate areas in the field. Record the dimensions including units used (e.g., 2" x 2" or 5 cm x 5 cm) on the sampling container using a permanent marker. Try and use only centimeters for recording data. Avoid making measurement in inches. Also, enter the dimensions (including the units used) on the "Paint Chip Collection" Reporting form in the column "Area Sampled with Units." Any irregularities or problems which arise in the

process, should be noted in the Comments column.

12. Seal the container.

13. Wrap tape around the container lid rim to ensure that the container remains sealed. Place sample container into a resealable plastic bag and seal. Place the sample container and bag into another resealable plastic bag and seal.

14. Generate a duplicate paint chip sample immediately adjacent to the first sample site, using the same procedure used to obtain the first sample. The preparations of the sample container remains unchanged except for the addition of the duplicate designation **"DUP"** to the sampling container and use of the "**DUP**" row on the reporting form. Irregularities and problems should be noted in the Comments column.

15. Enlarge the exposed substrate area made during paint chip collection to a minimum of 4" by 4" using the same general cutting and scraping methods followed for paint chip collection. Avoid pitting or significantly damaging the substrate surface. This area will be used by XRF testers for taking substrate measurements.

    NOTE: For some locations, a full 4"x 4" area may not be possible. For these locations, make the largest exposed area possible up to the desired 4"x 4" exposed surface.

16. Remove and dispose of the vinyl gloves, paper funnels, tape or other used disposable equipment prior to moving to the next sampling location. Avoid cross-contamination of samples by carefully cleaning all sampling and collection tools between each sample taken. Use pre-moistened wipes for this purpose.

17. Store the samples in a safe place during sampling until shipment can be made back to the laboratory. Turn over all completed "Paint Chip Collection Reporting" forms by the end of each sampling day to the field supervisor. Ensure that a copy of the form is made and placed into the box used for shipment back to the laboratory.

Paint Chip Collection Reporting Form

APPENDIX EE[a]

PILOT STUDY PROTOCOLS:
PREPARATION OF PAINT CHIP SAMPLES FOR SUBSEQUENT ATOMIC SPECTROMETRY LEAD ANALYSIS

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PREPARATION OF PAINT CHIP SAMPLES FOR SUBSEQUENT ATOMIC SPECTROMETRY LEAD ANALYSIS

1.0 SUMMARY

Lead in paint chip samples (chips, powder, etc.) is solubilized by extraction with nitric acid and hydrogen peroxide facilitated by heat after sample homogenization. The lead content of the digested sample is then in a form ready for measurement by Atomic Spectrometry. This procedure is similar to NIOSH Method 7082. Modifications have been made to convert this air particulate method to a method appropriate for processing paint chip samples.

2.0 APPARATUS

2.1 Instrumentation

- Electric hot plate; suitable for operation at temperatures up to at least 100°C as measured by a thermometer inside a solution filled container placed on the surface of the hot plate.

2.2 Glassware, and Supplies

- 150 mL or 250 mL beakers (borosilicate glass) equipped with watch glass covers.
- Class A borosilicate 250 mL volumetric flasks.
- Class A borosilicate volumetric pipets; volume as needed.
- 50 mL or 100 mL linear polyethylene tubes or bottles with caps.
- Borosilicate or plastic funnels.
- Glass rods and appropriate devices for breaking up paint chip samples.

2.3 Reagents

- Concentrated nitric acid (16.0 M $HN0_3$); spectrographic grade or equivalent.
- Nitric acid, 10% (v/v): Add 100 mL concentrated HNO_3 to 500 mL ASTM Type I water and dilute to 1 L.
- Hydrogen peroxide, 30% H_2O_2 (w/w); ACS reagent grade.
- ASTM Type I water (D 1193).

3.0 SAMPLE PREPARATION

3.1 SAMPLE HOMOGENIZATION

For each field sample, homogenize the paint chips inside the original sample container as described below.

1. Don a new clean pair of vinyl gloves to perform sample handling.

2. Remove any large amounts of substrate which may be present in the sample. Exercise care when removing substrate to avoid any paint losses. Leaving substrate in the sample is preferred over paint chip loss. If required, use a clean safety razor blade or equivalent tool to aid in substrate removal.

3. Immerse the bottom portion of sample container into a container containing dry ice. The depth of the container should be sufficient to cover all paint present within the sample container.

4. Allow the paint chip sample to freeze for a minimum of 10 minutes. Add more dry ice as needed to freeze the paint chip sample.

5. Using a clean glass rod or other appropriate clean tool, breakup the frozen paint chip sample inside the sample container into a fine powder. Samples or sample portions that resist homogenization should be noted in laboratory records.

6. After completing breakup of the sample, tap off any powder remaining on the tool used for breaking up the paint chips back into the sample container.

7. Seal the container and roll for about a minute or two to mix the samples. Rolling can be done by hand or using automated equipment.

3.2 WEIGHING PROCEDURE

For each sample, determine the total field sample weight, and weigh out a subsample for digestion as described below:

1. Don a new clean pair of vinyl gloves.

2. Label a clean beaker for use in digesting the sample and a new clean centrifuge tube with lid.

3. Wipe off the outside of the paint sample container with a clean laboratory paper wipe to remove any foreign material or oils. Using an analytical balance shown to be operating within normal calibration specifications, weigh the sample container containing the entire homogenized paint sample. Record the total paint sample plus container weight (and if provided, the area sampled) in a laboratory data form, notebook or equivalent recording device.

4. Weigh a sub-sample of homogenized paint from the contents of the sample container into a tared beaker labeled with the sample ID. Weigh approximately 0.5 grams to 0.0001 grams. Record the sub-sample weight (and if provided, the area sampled) in a laboratory data form, notebook or equivalent recording device.

5. Transfer the remaining homogenized paint sample into a new clean labeled centrifuge tube by carefully pouring the contents of the original sample container into the new tube. Use a clean glass rod to assist in the transfer as needed. Seal the new tube an store for archival use.

6. Remove any remaining sample powder from the original sample container (from the field) by rinsing with ASTM Type I water. Set the container aside and allow it to dry at room temperature.

7. After the original sample container has completely dried, re-weigh the container and record the empty container weight.

8. Determine the total field sample weight by subtracting the empty container weight from the total paint sample plus container weight generated in step 3.

3.3 SAMPLE DIGESTION

For each sample weighed into beakers, plus any QC samples, perform digestion as described below:

1. Wet the sample with about 2-3 mL of water from a squirt bottle filled with ASTM Type I water.

2. Add 7.5 mL of concentrated HNO_3 and 2.5 mL 30% H_2O_2, and cover with a watch glass.

3. Gently reflux on a hot plate for about 15 minutes (See Note 1).

4. Remove the watch glass and evaporate gently on a hot plate until the sample volume is reduced to about 1-2 mL (See Note 2).

5. Replace the watch glass and remove the beaker containing sample from the hot plate and allow it to cool (See Note 3).

NOTE 1: The original NIOSH method called for temperatures of 140°C as based on the use of digitally programmable hot-plates which measure the temperature on the inside of the hot plate head. A temperature drops of 40-50°C are not unusual between the inside of the hot plate head and the temperature actually experienced by the sample solution. The temperatures of sample solution should be between 85-100°C to prevent spattering of the solution. Monitor solution temperature on the hot plate by placing a thermometer in a flask or beaker filled with water during digestion activities.

NOTE 2: The original NIOSH method calls for evaporation until most of the acid has been evaporated. However, in order to avoid potential losses caused by sample splattering at low volumes, the method has been modified to specifically leave some solution remaining in the digestion vessels. Reduction volumes given are approximate and can be dependent on the sample size and beaker size used for preparation. Volumes should be reduced to as low a level as comfortably possible without causing sampling splattering or complete drying out of the sample.

NOTE 3: Cooling the sample is performed to avoid potential splattering losses and resulting safety hazards caused by addition of reagents to a partially digested hot sample during subsequent processing

steps. Samples do not have to be cooled completely to room temperature for safe further processing of paint chip samples. However, the operator must be aware that the potential for splattering losses and resulting safety hazards increases with increasing temperature of the sample digest.

6. Add 5 mL of concentrated HNO_3 and 2.5 mL 30% H_2O_2, and re-cover with a watch glass.

7. Gently reflux on a hot plate for about 15 minutes (See Note 1).

8. Remove the watch glass and evaporate gently on a hot plate until the sample volume is reduced to about 1-2 mL (See Note 2).

9. Replace the watch glass and remove the beaker containing sample from the hot plate and allow it to cool (See Note 3).

10. Add 5 mL of concentrated HNO_3 and 2.5 mL 30% H_2O_2, and re-cover with a watch glass.

11. Gently reflux on a hot plate for about 15 minutes (See Note 1).

12. Remove the watch glass and evaporate gently on a hot plate until the sample volume is reduced to about 1-2 mL (See Note 2).

13. Replace the watch glass and remove the beaker containing sample from the hot plate and allow it to cool (See Note 3).

14. Rinse the watch glass and beaker walls with 3 to 5 mL of 10% HNO_3 into the beaker.

15. Remove the watch glass and evaporate gently on a hot plate until the sample volume is reduced to about 1-2 mL (See Note 2).

16. Replace the watch glass and cool to room temperature.

17. Add 1 mL concentrate HNO_3 to the residue; swirl to dissolve soluble species.

18. Use a wash bottle filled with ASTM Type I water, rinse the beaker walls and underside of the watch glass with Type I water into the beaker.

19. Quantitatively transfer the digested sample into a 250-mL volumetric flask using several rinses with ASTM Type I water (See Note 4). A plastic or glass funnel should be used to avoid spillage during transfer from the beaker to the volumetric flask.

20. Dilute to volume with ASTM Type I water and mix thoroughly. The sample digest contains approximately 1 % (v/v) HNO_3.

21. Portions used for analysis must be filtered or centrifuged prior to instrumental measurement to remove undissolved material. Instrumental measurement should be performed using calibration standards that are matched to the same approximate acid levels as those in sample digest aliquot analyzed for analyte content.

NOTE 4: Due to potential losses during filtration, it is recommended to filter samples after dilution to final volume. Additional volume consumed by undissolved material will not cause any significant bias.

APPENDIX FF

PILOT STUDY PROTOCOLS:
STANDARD TEST PROTOCOL FOR THE ANALYSIS OF DIGESTED SAMPLES FOR LEAD BY INDUCTIVELY COUPLED PLASMA (ICP-AES), FLAME ATOMIC ABSORPTION (FAAS), OR GRAPHITE FURNACE ATOMIC ABSORPTION (GFAAS) TECHNIQUES

STANDARD TEST PROTOCOL FOR THE ANALYSIS OF DIGESTED SAMPLES FOR LEAD BY INDUCTIVELY COUPLED PLASMA (ICP-AES), FLAME ATOMIC ABSORPTION (FAAS), OR GRAPHITE FURNACE ATOMIC ABSORPTION (GFAAS) TECHNIQUES

1.0 SUMMARY

A sample digestate is analyzed for Lead content using ICP-AES, Flame-AAS, or Graphite Furnace-AAS techniques. Instrumental Quality Control samples are analyzed along with sample digestates to assure adequate instrumental performance. This procedure is similar to SW-846 Method 6010. It is equivalent to the draft procedure currently under consideration in ASTM Subcommittee E06.23.

2.0 DEFINITIONS

2.1 *Digestion* - The sample preparation process which will solubilize targeted analytes present in the sample and results in an acidified aqueous solution called the digestate.

2.2 *Digestate* - An acidified aqueous solution which results from performing sample preparation (digestion) activities. Lead measurements are made using this solution.

2.3 *Batch* - A group of field or QC samples which are processed together using the same reagents and equipment.

2.4 *Serial Dilution* - A method of producing a less concentrated solution through one or more consecutive dilution steps. Dilution step for a standard or sample is performed by volumetrically placing a small aliquot of a higher concentrated solution into a volumetric flask and diluting to volume with water containing the same acid levels as found in original sample digestates.

2.5 *Method Blank* - A digestate which reflects the maximum

treatment given any one sample within a sample batch except that it has no sample initially placed into the digestion vessel. (The same reagents and processing conditions which are applied to field samples within a batch are also applied to the method blank.) Analysis results from method blanks provide information on the level of potential contamination experienced by samples processed within the batch.

2.6 *No-Spiked Sample* - A portion of a homogenized sample which was targeted for addition of analyte but which is not fortified with all the target analytes before sample preparation. A method blank serves as a no-spike sample in cases where samples cannot be uniformly split as described in section 2.7. Analysis results for this sample is used to correct for native analyte levels in the spiked and spiked duplicate samples.

2.7 *Spiked Sample and Spiked Duplicate Sample* - Two portions of a homogenized sample which were targeted for addition of analyte and are fortified with all the target analytes before preparation. In cases where samples cannot be uniformly split (such as paint chip samples taken for Lead per area determinations, a method blank can be used in place of the homogenized sample split. Use of a method blank for a spiked sample should be referred to as a "spiked method blank" or "spiked method blank duplicate". Analysis results for these samples are used to provide information on accuracy and precision of the overall analysis process.

2.8 *Analysis Run* - A period of measurement time on a given instrument during which data is calculated from a single calibration curve (or single set of curves). Re-calibration of a given instrument produces a new analysis run.

2.9 *Instrumental QC Standards* - Solutions analyzed during an instrumental analysis run which provide information on measurement performance during the instrumental analysis portion of the overall Lead measurement process.

2.10 *Semi-quantitative Screen* - An analysis run which is performed on highly diluted sample digestates for the

purpose of determining the approximate analyte level in the digest. This analysis run is generally performed without inserting Instrumental QC standards except for calibration standards. Data from this run are used for determining serial dilution requirements for sample digestates to keep them within the linear range of the instrument.

2.11 *Quantitative Analysis* - An analysis run on sample digestates (or serial dilutions of sample digestates) which includes Instrumental QC standards. Data from this run are used to calculate and report final Lead analysis results.

2.12 *Initial Calibration Blank (ICB)* - A Standard solution which contains no analyte and is used for initial calibration and zeroing instrument response. The ICB must be matrix matched to acid content present in sample digestates. The ICB must be measured during calibration and after calibration. The measured value is to be less than 5 times the instrumental detection limit.

2.13 *Calibration Standards* - Standard solutions used to Calibrate instrument. Calibration Standards must be matrix matched to acid content present in sample digestates and must be measured prior to measuring any sample digestates.

2.14 *Initial Calibration Verification (ICV)* - A Standard solution (or set of solutions) used to verify calibration standard levels. Concentration of analyte to be near mid-range of linear curve which is made from a stock solution having a different manufacturer or manufacturer lot identification than the calibration standards. The ICV must be matrix matched to acid content present in sample digestates. The ICV must be measured after calibration and before measuring any sample digestates. The measured value to fall within $\pm$10% of known value.

2.15 *Interference Check Standard (ICS)* - A standard solution (or set of solutions) used for ICP-AES to verify accurate analyte response in the presence of possible spectral interferences from other analytes present in

samples. The concentration of analyte to be less than 25% of the highest calibration standard, concentrations of interferant will be 200 µg/Ml of Al, Ca, Fe, and Mg. The ICS must be matrix matched to acid content present in sample digestates. The ICS must be analyzed at least twice, once before and once after all sample digestates. The measured analyte value is expected to be within ±20% of known value.

2.16 *Continuing Calibration Verification (CCV)* - A standard solution (or set of solutions) used to verify freedom from excessive instrumental drift. The concentration to be near mid-range of linear curve. The CCV must be matrix matched to acid content present in sample digestates. The CCV must be analyzed before and after all sample digestates and at a frequency not less than every ten sample digestates. The measured value to fall within ±10% of known value for ICP-AES or FAAS (±20% for GFAA), run once for every 10 samples.

2.17 *Continuing Calibration Blank (CCB)* - A standard solution which has no analyte and is used to verify blank response and freedom from carryover. The CCB must be analyzed after the CCV and after the ICS. The measured value is to be less than 5 times the instrumental detection limit.

3.0 APPARATUS AND MATERIALS

3.1 Analytical Instrumentation

3.1.1 Inductively Coupled Plasma Atomic Emission Spectrometer (ICP-AES) - Either sequential or simultaneous capable of measuring at least one of the primary ICP Lead emission lines. Emission line used must be demonstrated to have freedom from common major interferants such as Al, Ca, Fe and Mg or the ability to correct for these interferants.

3.1.2 Flame Atomic Absorption Spectrometer (FAAS) - Equipped with an air-acetylene burner head, Lead hollow cathode lamp or equivalent and capable of making Lead absorption measurements at the 283.3nm absorption line.

NOTE: The 283.3nm line is preferred over the 217nm line

because of the increased noise levels commonly observed at the 217nm line for FAAS and GFAAS.

3.1.3 Graphite Furnace Atomic Absorption Spectrometer (GFAAS) - Equipped with background correction, Lead hollow cathode lamp or equivalent and capable of making Lead absorption measurements at the 283.3nm absorption line.

3.2 Gases

Grades specified by manufacturer of the instrument employed.

3.2.1 Compressed air and acetylene for FAAS.

3.2.2 Compressed or liquid argon for ICP-AES and GFAAS.

3.2.3 Minimum of two stage regulation of all gases.

3.3 Glassware and Miscellaneous Supplies

3.3.1 Vinyl Gloves, Powderless.

3.3.2 Micro-pipettors with Disposable Plastic Tips, sizes needed to make reagent additions, and spiking standards. In general, the following sizes should be readily available: 1-5mL adjustable, 1000µL, 500µL, 250µL, and 100µL.

3.3.3 Volumetric Flasks, sizes needed to make, calibration standards, serial dilutions and Instrumental QC standards.

4.0 Reagents

4.1 *Nitric acid*, concentrated; reagent grade

4.2 *Water*--Unless otherwise indicated, references to water shall be understood to mean reagent water as defined by Type 1 of Specification D1193. (ASTM Type I Water: Minimum resistance of 16.67 megohm-cm, or equivalent.)

4.3 *Calibration stock solution*, 100µg/mL of Pb in dilute nitric acid or equivalent (such as a multi-element stock containing Pb).

4.4 *Check standard stock solution* (for ICV), 100µg/mL of Pb in dilute nitric acid or equivalent. Must be sourced from a different lot number (or manufacturer) than the Calibration stock solution (7.3).

4.5 *Interferant stock solution* (for ICS; ICP-AES only), 10000µg/mL of Al, Ca, Fe, and Mg in dilute nitric acid or equivalent.

5.0 Procedure

5.1 *Laboratory Records*--Record all reagent sources (lot numbers) used for sample preparation in a laboratory notebook. Record any inadvertent deviations, unusual happenings or observations on a real-time basis as samples are processed. Use these records to add supplement Lead data when reporting results.

5.2 *Instrumental Setup*

5.2.1 *FAAS/GFAAS* - Set the FAAS or GFAAS spectrometer up for the analysis of Lead at 283.3nm according to the instructions given by the manufacturer. Be sure to allow at least a 30 minute warmup of the hollow cathode lamp prior to starting calibration and analysis.

5.2.2 *ICP-AES* - Set the ICP spectrometer up for the analysis of Lead at a primary Lead emission line (such as 220.2nm) according to the instructions given by the manufacturer. Be sure to allow at least a 30 minute warmup of the system prior to starting calibration and analysis.

5.3 *Preparation of Calibration and Instrumental QC Standards*

5.3.1 *Calibration Standards* - Prepare a series of calibration standards covering the linear range of the instrumentation. Prepare these standards using serial dilution from the calibration stock solutions. Prepare these standards using the same final nitric acid concentration present in the sample digestates. Also prepare an Initial Calibration Blank (ICB) as defined in section 3 and Table FF-1.

> NOTE: For FAAS/GFAAS prepare a minimum of 3 calibration standards plus the ICB for performing calibration of the instrument. ICP-AES can be performed using one high

calibration standard and an ICB. However, more are generally preferred.

5.3.2 *Instrumental QC Standards* - Prepare Instrumental QC standards as summarized in Table FF-1 using serial dilution from the required stock solutions. Prepare these standards using the same final nitric acid concentration present in the sample digestates.

NOTE: The ICV is used to assess the accuracy of the calibration standards. Therefore, it must be made from a different original source of stock solution than the stock used to make the calibration standards. Use of a different serial dilution of the same original stock is not acceptable.

5.4 *Calibration and Instrumental Measurement* - Perform calibration and quantitative Lead measurement of sample digestates and instrumental QC samples in the sequential order outlined in Table FF-2.

NOTE: Performance of a Semi-quantitative screen prior to quantitative analysis for sample digests containing unknown levels of Lead generally recommended. The purpose of this screen is to determine serial dilution requirements of each digestate needed to keep the instrumental response within the calibration curve. During a semi-quantitative screen all digestates are diluted to a constant large value (1-to-100 for ICP/FAAS and 1-to-1000 for GFAAS). The instrument is calibrated and diluted digestates are analyzed without inserting the instrumental QC used for a Quantitative analysis run. Data from this screen are reviewed to calculate the optimum serial dilution needed for each digestate. <u>No sample data can be reported for any analyte value not falling within the calibration range</u>. Therefore, the optimum dilution is one which achieves the maximum Lead response which is still within the calibration curve. For ICP-AES, levels of possible interferants (Al, Ca, Fe and Mg) may have to also be considered in order to make interference corrections. For ICP-AES, digestates must be sufficiently diluted to assure that levels of possible interferants such as Al, Ca, Fe and Mg are at or below the levels present in the ICS.

5.5 *Instrumental QC Evaluation and Corrective Action* - Examine the data generated from the analysis of calibration standards and Instrumental QC standards. Evaluate the analysis run using the criteria shown in Table FF-1. Failure to achieve the specifications shown in Table FF-1 will require corrective action to be performed as described below:

5.5.1 *ICB, Calibration standards, or ICV* - Failure to meet specifications for these Instrumental QC standards requires complete re-calibration. Sample digestates cannot be measured under these conditions. It is recommended that standards be re-prepared prior to re-calibration.

5.5.2 *High Calibration Standard Re-run* - Failure to meet specifications for this Instrumental QC standard requires complete re-calibration. Sample digestates cannot be measured under these conditions. It is recommended that standard range be reduced prior to re-calibration.

5.5.3 *ICS* - Failure to meet specifications for these Instrumental QC standards requires re-analysis of the standard until specifications are met. Sample digestates cannot be measured under these conditions. Re-preparation of the standard prior to re-analysis is recommended under these conditions. Continued failure of the ICS may require interference correction investigation or changing of instrument parameters. Consult the manufacturers recommendations under these conditions. Any change in instrument parameters must be accompanied by re-calibration. If measured aliquots of sample digestates can be shown not to contain interferants as high as those recommended for the ICS making, then the interference levels in the ICS can be lowered. Such changes must be documented in laboratory records with data supporting the justification for the change. All measurements on sample digests must be bracketed by an ICS which meets specifications (called a "passing" ICS). Failure to meet specifications on the ICS run after the sample digestates requires re-running of all sample digestates since the last passing ICS was measured. Since the ICS only is required to be analyzed twice, much data could be lost if the analytical run were long and the second ICS failed specifications. This is good reason for including periodic analysis of the ICS as shown in Table FF-2.

5.5.4 *CCV* - Failure to meet specifications for these Instrumental QC standards indicates excessive instrumental drift.

Sample digestates cannot be measured under these conditions and any sample digestates measured since the last passing CCV must be reanalyzed. This situation requires either re-analysis of the standard until specifications are met or re-calibration. All measurements on sample digests must be bracketed by an CCV which meets specifications.

5.5.5 *CCB* - Failure to meet specifications for these Instrumental QC standards indicates the presence of possible instrumental carryover or baseline shift. Such a failure will have the most impact on sample digestates at the lower end of the calibration curve. The first corrective action is to re-analyze the CCB. If the CCB passes, then the rinse time between the samples should be increased and the analysis continued. If the instrument response is still elevated and has not significantly changed, then the instrument can be re-zeroed followed by a CCV-CCB and re-analysis of all samples since the last passing CCB which are within 5 times the response of the failed CCB.

6.0 Calculations

For FAAS/GFAAS : Prepare a calibration curve to convert instrument response (absorbance) to concentration (µg/mL) using a linear regression fit. Convert all instrumental measurements on instrumental QC standards and sample digests to Lead concentration (µg/mL) using the calibration curve.

NOTE: Some instruments will automatically prepare a calibration curve based on a linear regression fit.

For ICP-AES: All modern ICPs automatically prepare a calibration curve to convert instrumental responses (emission intensity) to concentration (µg/g).

TABLE FF-1. SUMMARY OF LABORATORY INSTRUMENTAL MEASUREMENT QC STANDARDS

Name	Use	Specification
ICB - Initial Calibration Blank	Used for initial calibration and zeroing instrument response.	Calibration Standard which contains no lead. Must be measured during calibration and after calibration. Measured value to be less than 5 times the IDL.
Calibration Standards	Used to Calibrate instrument. The high standard re-run is used to check for response linearity.	Acid content must be approximately the same as that in the sample digests. Must be measured prior to measuring any sample digests. Correlation Coefficient of $\geq$0.995, as measured using linear regression on instrument response(y) versus concentration(x). The highest level Calibration standard must be measured after calibration. The measured value to fall within $\pm$10% of known value.
ICV - Initial Calibration Verification	Used to verify calibration standard levels.	Concentration of lead to be near the middle of calibration curve. It is made from a stock solution having a different manufacturer or manufacturer lot identification than the calibration standards. Must be measured after calibration and before measuring any sample digests. Measured value to fall within $\pm$10% of known value.
ICS - Interference Check Standard	Used to verify accurate lead response in the presence of possible spectral interferences from other analytes present in samples.	Concentration of lead to be less than 25% of the highest calibration standard, concentrations of interferant are 200 µg/mL of Al, Ca, Fe, and Mg. Must be analyzed at least twice, once before and once after all sample digestates. Measured lead value to fall within ±20% of known value.
CCV - Continuing Calibration Verification	Used to verify freedom from excessive instrumental drift.	Concentration to be near the middle of the calibration curve. Must be analyzed before and after all sample digestates and at a frequency not less than once every ten samples. Measured value to fall within ±10% of known value.
CCB - Continuing Calibration Blank	Used to verify blank response and freedom from carryover.	Calibration Standard which contains no lead. Must be analyzed after each CCV and each ICS. Measured value to be less than 5 times the instrumental detection limit.

TABLE FF-2. EXAMPLE OF A TYPICAL ANALYSIS ORDER FOR MEASUREMENT

Run Order No. (relative)	Sample ID	Comments	
1	ICB	Calibration Blank	Instrument Calibration
2-4	low, med, high	Calibration Standards	
5	ICB	Calibration Blank	Calibration Verification
6	ICV	made from different stock, level is near mid-point of curve	
7	high standard	Calibration Standard	Linearity Check
8	CCB	Same as Calibration Blank	
9	ICS	Interference Check Standard	Interferant check for ICP only
10	CCB	Carryover Check	
11	CCV	Drift Check, same as near midpoint calibration standard	Continuing Calibration Verification
12	CCB	Carryover check	
*** start repeating cycle of samples-Instrumental QC here ***			
13-22	Sample IDs	Sample digestates	Max. of 10 samples
23-24	CCV CCB	Drift Check + Carryover Check	See run # 11-12
25-34	Sample IDs	Sample digestates	Max. of 10 samples
35-36	ICS CCB	Interferant Check + Carryover Check	See run # 9-10
37-38	CCV CCB	Drift Check + Carryover Check	See run # 11-12
*** end repeating cycle of samples-QC standards here ***			

APPENDIX GG

PILOT STUDY PROTOCOLS:
PROTOCOL FOR PACKAGING AND SHIPPING OF SAMPLES

PROTOCOL FOR PACKAGING AND SHIPPING OF SAMPLES

1.0 INTRODUCTION

Collection and analysis of paint chip samples as specified by the QAPjP will require packaging and shipping of samples from sampling sites. The field team will be responsible for packaging and shipping the samples from each sampling site to the Sample Custodian at MRI. The following are protocols for packaging and shipping samples from the field.

2.0 SAMPLE PACKAGING PROTOCOL

The field team is responsible for preparing the samples for shipment back to MRI. Samples that are collected will be shipped at the end of each sampling day. The same shipping container that was used to ship sample collection containers to the field will be used to ship them back to MRI. All sampling materials will be packaged in accordance with Department of Transportation (DOT) regulations. The field team will include copies of the field sampling forms with the samples to identify the contents of the shipping containers. The original field sampling forms will be held by the field supervisor and ultimately hand carried back to MRI. <u>Do not send original copies of sample data forms or other important records with the samples.</u>

3.0 SAMPLE SHIPPING METHODS

All samples will be shipped to MRI via Federal Express Economy Distribution Service in accordance with DOT shipping regulations. The MRI field team will be responsible making the shipping arrangement with the local Federal Express Office. Preprinted Federal Express Air Bills can be obtained from the MRI Shipping and Receiving Department. All Federal Express shipments will use the standard Federal Express Air Bill. For further details consult with MRI's S & R Department.

APPENDIX HH

PILOT STUDY PROTOCOLS:
GLASSWARE/PLASTICWARE CLEANING PROCEDURE

INFORMATION NOT PRESENT : PROPRIETARY INFORMATION

APPENDIX II

PILOT STUDY PROTOCOLS:
ACID BATH CLEANING PROCEDURES

INFORMATION NOT PRESENT : PROPRIETARY INFORMATION

APPENDIX AAA

LABORATORY SAMPLE PREPARATION EXPERIMENTS

LABORATORY SAMPLE PREPARATION EXPERIMENTS

1.0 Introduction

At the initiation of this study, a draft EPA report [3], indicated that a NIOSH method 7082 would be an acceptable sample preparation method for the study since it was shown to produce high lead recoveries from paint samples. NIOSH method 7082 is designed to prepare and analyze air filter samples for analysis of a wide variety of inorganic components that also included lead. Because it is specifically written for air filter samples, modifications to NIOSH 7082 are required to make it applicable to processing paint chip samples. Based on the EPA report, this method with appropriate modifications, was selected to digest paint samples for this study.

Prior to initiation of laboratory analysis on collected field samples, a set of four experiments were conducted for the following three reasons:

1. To familiarize the laboratory with the modified NIOSH method 7082.
2. To assure that the modifications to the method were appropriate.
3. To determine the appropriate sample mass that could be processed using the modified NIOSH method 7082.

A discussion of the four experiments, referred to as Tests 1, 2, 3 and 4, performed on account of the three reasons listed above, are presented in this appendix. Since the laboratory targeted for the paint analysis activities in this study had a great deal of experience using EPA SW846 method 3050, a commonly used sample preparation procedure for the analysis of metals in solid samples, this method was used in these experiments to provide a basis of comparison to the selected modified NIOSH method 7082.

These experiments were not intended to be an exhaustive comparison study for determining the optimal sample preparation of paint chips for lead analysis. Rather, the experiments were used to familiarize the laboratory with the modified NIOSH method 7082 and to identify any obvious factors that could affect lead recoveries from paint samples.

1.1 General Experimental Approach

Two general design elements were included into each of the four experiments: (1) Use of paint sample materials well characterized for lead concentrations, and (2) Use of sample aliquots of variable mass. Each of these general design elements are discussed in the following subsections 1.1.1 and 1.1.2.

1.1.1 Use of Paint Sample Materials with Known Lead Concentrations

Paint sample materials well characterized for lead concentrations were included in the experiments to evaluate the sample preparation procedures by measuring lead recoveries. National Institute of Standards and Technology (NIST), standard reference material (SRM) No. 1579a lead-based paint, 11.995 percent lead by weight, was included in all four experiments for two reasons. First, NIST SRM No. 1579a was the only lead based paint material available that had a certified lead concentration. Second, difficulties in obtaining lead recoveries from this material had been reported by a few persons[1] which made it a good material to differentiate between rigorous and marginal methods, i.e., sample preparation methods that could obtain good recoveries from this material would provide increased confidence that high lead recoveries would be obtained from collected paint chip samples.

In addition to NIST SRM No. 1579a, paint performance evaluation samples, from rounds 02 and 03 prepared for the American Industrial Hygiene Association (AIHA) Environmental Lead Proficiency Analytical Testing (ELPAT) program, were included in two of the experiments. The ELPAT samples were included to provide additional lead recovery data on which to differentiate between the methods being examined during the experimentation.

1.1.2 Use of Sample Aliquots of Variable Mass

Sample preparation methods are sample size limited because procedures include fixed amounts of acidic reagents and extraction volumes. For a given matrix using a specific method, it is expected that, above a given sample mass, analyte recoveries would be poor. Collection of a large surface area, approximately 25 cm^2, was incorporated as a study design element to aid in reducing variation caused by potential spatial lead

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[1]Personal communications between Midwest Research Institute in Kansas City, MO and NIOSH in Cincinnati, OH and EPA/ORD in Research Triangle Park, NC

variations as discussed in section 3.2.2.1 and collection error. Therefore, average total collected sample mass was expected to be high which would require sample homogenization and subsampling to obtain a sample mass that could be effectively prepared for lead analysis in the laboratory. As a consequence, the effect of sample mass was investigated during all experiments to determine the mass limits of the tested procedures.

2.0 Discussion of Experimental Results

The first two experiments included investigations using two different hot-plate type extraction sample preparation methods, EPA SW846 method 3050 and a modified NIOSH method 7082, while the later two focused on refining procedures only for the selected method, modified NIOSH method 7082. Instrumental lead measurements for all four experiments were conducted using ICP-AES.

2.1 Discussion of Test 1

The purpose of Test 1 was to compare the two selected hot-plate digestion (extraction) methods by examining the lead recoveries from NIST SRM No. 1579a and ELPAT samples. A summary of the two extraction methods used in Test 1 are shown in Table AAA-1. The modifications to NIOSH 7082, as shown in the table, were made to convert this air filter sample method to a method that is applicable for processing paint chip samples.

Test 1 included a set of triplicate extractions for three (3) different nominal sample masses ranging from 0.5 to 5 grams for NIST SRM No. 1579a and 0.5 to 1 gram for ELPAT samples. This set of samples was prepared by a single technician using SW846 method 3050 within a single sample preparation batch to minimize any potential between batch effects. This entire set was duplicated by a second technician using the modified NIOSH method 7082 as summarized in Table AAA-2.

The following conclusions are suggested from the Test 1 results presented in Tables AAA-3 and AAA-4:

(1) Results from ELPAT samples, shown in Table AAA-3, are erratic with mean lead recoveries ranging from 77.3% to 100.8% and relative standard deviations ranging from 0.6% to 29.2% across both hot-plate extraction methods over the 0.5 to 1 gram mass range.

Table AAA-1. Summary of Modifications made to Methods for Test 1.

Sample Extraction Method	Modification to Method	Reason for Modification
EPA SW846 method 3050 with HCl option	None.	
NIOSH method 7082	References to use 140°C hot-plates were replaced with temperatures of 85-100°C.	To avoid potential losses caused by spattering
	References for evaporation to dryness were replaced by evaporation to near dryness.	
	For nominal sample mass <1 gram: increase total concentrated nitric acid volume from 6 to 9 mL and increase final dilution volume from 10 to 100 mL.	To allow for increased sample mass
	For nominal sample mass ≥1 gram: increase total concentrated nitric acid levels from 6 to 18 mL and increase final dilution volume from 10 to 200 mL.	

Table AAA-2. Summary of Design Parameters for Test 1.

Method[b]	Sample Type[a]	Nominal Sample Mass (grams)	No. of Replicates at Each Mass
SW846-B	NIST	0.5, 1, 5	3
	ELPAT	0.5, 1	3
NIOSH-C	NIST	0.5, 1, 5	3
	ELPAT	0.5, 1	3

[a] SW846-B = method SW846 method 3050 performed by technician B
NIOSH-C = modified NIOSH method 7082 performed by technician C
[b] NIST = SRM No. 1579a, lead level of 11.995%
ELPAT = performance samples from round 1, samples 3 and 4, reference values given as 0.7026% and 5.4744% respectively, triplicate samples were from either sample 3 or sample 4.

(2) Results from NIST SRM 1579a, shown in Table AAA-4, are also erratic with mean lead recoveries ranging from 54.6% to 82.8% and relative standard deviations ranging from 9.0% to 49.0% across both hot-plate extraction methods over the 0.5 to 1 gram mass range.

(3) Results for the nominal 5 gram mass showed very low lead recoveries ranging from 8.7% to 21.1%, strongly suggesting

that neither extraction procedure was capable of extracting lead from a sample mass of 5 grams.

Table AAA-3. Summary Results for Test 1: The Effect of Lead Recovery from ELPAT Samples at Variable Sample Mass using SW846 method 3050 and modified NIOSH method 7082.

Nominal Sample Mass[a] (grams)	Lead Recovery Results for ELPAT Samples					
	SW846 method 3050			modified NIOSH method 7082		
	Person Code[b]	Mean Recovery[c]	Relative Standard Deviation[d]	Person Code[b]	Mean Recovery[c]	Relative Standard Deviation[d]
0.5	B	82.2%	13.5%	C	100.8%	12.8%
1	B	99.0%	0.6%	C	77.3%	29.2%

[a] Actual sample mass was within ±25% of the nominal sample mass.
[b] Codes represent preparation of samples by specific technicians.
[c] Mean of three replicates.
[d] [(standard deviation of three replicates)/(mean recovery)](100)

Table AAA-4. Summary Results for Test 1: The Effect of Lead Recovery from NIST SRM 1579a at Variable Sample Mass using SW846 method 3050 and modified NIOSH method 7082.

Nominal Sample Mass[a] (grams)	Lead Recovery Results for NIST SRM 1579a					
	SW846 method 3050			modified NIOSH method 7082		
	Person Code[b]	Mean Recovery[c]	Relative Standard Deviation[d]	Person Code[b]	Mean Recovery[c]	Relative Standard Deviation[d]
0.5	B	64.4%	49.0%	C	82.8%	36.0%
1	B	80.9%	9.0%	C	54.6%	33.5%
5	B	21.1%	18.0%	C	8.7%	23.0%

[a] Actual sample mass was within ±10% of the nominal sample mass.
[b] Codes represent preparation of samples by specific technicians.
[c] Mean of three replicates.
[d] [(standard deviation of three replicates)/(mean recovery)](100)

Because of the inconsistent results obtained from Test 1, decisions were made to modify the two extraction methods with the aim of improving lead recoveries. The modifications made to the methods are shown in Table AAA-5.

2.2 Discussion of Test 2

The purpose of Test 2 was to compare the two selected hot-plate digestion (extraction) methods after modification by examining the lead recoveries from NIST SRM No. 1579a. A summary

of the two extraction procedures used in Test 2 are shown in Table AAA-5.

The effect of variation in sample mass on lead recovery was examined more carefully in Test 2 than in Test 1. Test 2 included a set of triplicate extractions for four (4) different nominal sample masses ranging from 1 to 4 grams for NIST SRM No. 1579a. This set of samples was prepared by a single technician using the modified SW846 method 3050 within a single sample preparation batch to minimize any potential between batch effects. This entire set was duplicated by a second technician and a third technician using the modified SW846 method 3050 and the modified NIOSH method 7082, respectively as summarized in Table AAA-6. The replication of the sample set using the same extraction method, modified SW846 method 3050, was done to help rule out a potential technician processing problem which was proposed as a potential cause of the inconsistent results obtained in Test 1 for this commonly used method of sample preparation.

The following conclusions are suggested from the Test 2 results presented in Table AAA-7:

(1) Mean recoveries of lead in NIST SRM 1579a extracted by modified SW846 method 3050 and the modified NIOSH method 7082 decreased with increases in sample mass.

(2) Lower lead recoveries for the modified SW846 method 3050 were a result of the sample preparation methodology and not a technician processing problem since the two technicians using the same procedure obtained similar results.

(3) Lead recovery using the modified NIOSH method 7082 for the nominal 1 and 2 gram sample mass was higher than for the modified SW846 method 3050 at 98.6% and 79.8% compared to 76.3%, 89.3% and 56.1%, 58.8%, respectively.

(4) Precision of the lead recovery was also better using the modified NIOSH method 7082 for the nominal 1 and 2 gram sample mass than for the modified SW846 method 3050 as measured by the relative standard deviations at 3.0% and 3.5% compared to 19.7%, 4.1% and 8.4%, 17.9%, respectively.

Table AAA-5. Summary of Modifications made to Methods for Tests 2, 3, and 4.

Sample Extraction Method	**Modification to Method**	**Reason for Modification**
EPA SW846 method 3050 (Test 2 only)	For nominal sample mass ≥2 gram: 2.5 fold increase in reagents and final dilution volume.	To allow for the increased sample mass
NIOSH method 7082	References to use 140°C hot-plates were replaced with temperatures of 85-100°C.	To avoid potential losses caused by spattering
	References for evaporation to dryness were replaced by evaporation to near dryness.	
	Increased total concentrated nitric acid volumes from 6 to 22.5 mL, increased total hydrogen peroxide volumes from 3 to 7.5 mL and increase final dilution volume from 10 to 250 mL.	To allow for the increased sample mass.

Table AAA-6. Summary of Design Parameters for Test 2.

Method[a]	**Sample Type**[b]	**Nominal Sample Mass (grams)**	**No. of Replicates at Each Mass**
SW846-A	NIST	1, 2, 3, 4	3
SW846-B	NIST	1, 2, 3, 4	3
NIOSH-D	NIST	1, 2, 3, 4	3

[a] SW846-A = modified SW846 method 3050 performed by technician A
SW846-B = modified SW846 method 3050 performed by technician B
NIOSH-D = modified NIOSH method 7082 performed by technician D
[b] NIST = SRM No. 1579a, lead level of 11.995%

Table AAA-7. Summary Results for Test 2: The Effect of Lead Recovery from NIST SRM 1579a at Variable Sample Mass using modified SW846 method 3050 and modified NIOSH method 7082

Nominal Sample Mass[a] (grams)	Lead Recovery Results for NIST SRM 1579a					
	modified SW846 method 3050			modified NIOSH method 7082		
	Person Code[b]	Mean Recovery[c]	Relative Standard Deviation[d]	Person Code[b]	Mean Recovery[c]	Relative Standard Deviation[d]
1	A B	76.3% 89.3%	19.7% 4.1%	D	98.6%	3.0%
2	A B	56.1% 58.8%	8.4% 17.9%	D	79.8%	3.5%
3	A B	23.7% 44.3%	26.6% 37.2%	D	48.1%	2.7%
4	A B	29.9% 27.6%	10.0% 20.3%	D	34.8%	2.6%

[a] Actual sample mass was within ±12% of the nominal sample mass.
[b] Codes represent preparation of samples by specific technicians.
[c] Mean of three replicates.
[d] [(standard deviation of three replicates)/(mean recovery)](100)

Based on the conclusions presented above, the modified NIOSH method 7082 appeared to be adequate for use in this study as compared to EPA SW846 method 3050. Further experiments, Tests 3 and 4, were performed without including SW846 method 3050 and were used to determine the appropriate sample mass for the modified NIOSH method 7082.

2.3 Discussion of Test 3

The purpose of Test 3 was to determine the appropriate sample mass for the modified NIOSH method 7082 by examining the lead recoveries from NIST SRM No. 1579a and ELPAT samples. A summary of the modified NIOSH method 7082 used in Test 3 is shown in Table AAA-5.

Test 3 included a set of triplicate extractions for five (5) different nominal sample masses ranging from 0.25 to 1.25 grams for NIST SRM No. 1579a as summarized in Table AAA-8. This set of samples was prepared by a single technician using the modified NIOSH method 7082 within a single sample preparation batch to minimize any potential between-batch effects. This entire set was duplicated by a second technician and a third technician to provide multiple data sets on the effect of sample mass on lead

recovery using the modified NIOSH method 7082 and to identify differences in recoveries associated with individual technicians.

Table AAA-8. Summary of Design Parameters for Test 3.

Method[a]	Sample Type[b]	Nominal Sample Mass (grams)	No. of Replicates at Each Mass
NIOSH-A	NIST	0.25, 0.5, 0.75, 1, 1.25	3
NIOSH-B	NIST	0.25, 0.5, 0.75, 1, 1.25	3
NIOSH-D	NIST	0.25, 0.5, 0.75, 1, 1.25	3

[a] NIOSH-B = modified method 7082 performed by technician A
NIOSH-D = modified method 7082 performed by technician B
NIOSH-A = modified method 7082 performed by technician D
[b] NIST = SRM No. 1579a, lead level of 11.995%

The following conclusions are suggested from the Test 3 results presented in Table AAA-9:

(1) Mean recoveries of lead in NIST SRM 1579a extracted by the modified NIOSH method 7082 decreased with increases in sample mass. This conclusion is consistent with results from Test 2.
(2) Lead recoveries for the 0.25 gram sample mass gave the highest lead recoveries for all three technicians ranging from 99.0% to 100.3%.
(3) Lead recoveries for the 0.25 gram and 0.5 gram sample masswere above the 90% level for all three technicians ranging from 92.8% to 100.3%.
(4) For two out of three technicians, lead recoveries for the 0.75 gram sample mass dropped below 80% at 74.4% and 75.4% compared to 94.6%, respectively.
(5) Lead recoveries for the 1.0 and 1.25 gram sample mass gave the lowest lead recoveries for all three technicians ranging from 50.2% to 87.9%.
(6) Precision of the lead recovery, as measured by the relative standard deviations from triplicate samples, was below 15% for all three technicians at all sample masses, ranging from 0.6% to 13.0%, with one exception at 49.6%.

Data presented above suggest that sample mass should not exceed 0.5 grams.

Table AAA-9. Summary Results for Test 3: The Effect of Lead Recovery from NIST SRM 1579a at Variable Sample Mass using modified NIOSH method 7082.

[a] Nominal Sample Mass (grams)	Lead Recovery Results for NIST SRM 1579a modified NIOSH method 7082		
	[b] Person Code	[c] Mean Recovery	[d] Relative Standard Deviation
0.25	A	100.3%	0.4%
	B	99.0%	3.1%
	D	99.6%	2.2%
0.5	A	92.8%	13.0%
	B	97.9%	0.6%
	D	93.7%	1.6%
0.75	A	75.4%	8.9%
	B	94.6%	8.8%
	D	74.4%	7.1%
1.0	A	68.9%	5.7%
	B	87.9%	10.5%
	D	83.9%	49.6%
1.25	A	56.6%	6.0%
	B	73.7%	8.1%
	D	50.2%	10.6%

[a] Actual sample mass was within ±10% of the nominal sample mass.
[b] Codes represent preparation of samples by specific technicians.
[c] Mean of three replicates
[d] [(standard deviation of three replicates)/(mean recovery)](100)

2.4 Discussion of Test 4

The purpose of Test 4 was to examine the extraction efficiency for the 0.5 and 0.25 gram sample mass using the modified NIOSH method 7082 in more detail than Test 3. Because the contribution to variability from sample inhomogeneity increases with decreasing aliquot mass, as discussed in subsection 3.3.1.2.3, use of a 0.5 gram mass is more desirable than a 0.25 gram sample mass even though data from Test 3 show that the 0.25 gram mass produces the highest lead recoveries from the NIST SRM No. 1579a. Data from Test 3 suggests that a 0.5 gram aliquot should provide lead recoveries greater than 90% from field samples assuming that recovery from field samples is at least as high as that from NIST SRM No. 1579a. Test 4 was performed to provide additional confidence that the use of the 0.5 gram sample mass would achieve high recovery of lead from field samples.

Test 4 included a set of duplicate extractions for 10 homogenized field samples at two (2) different nominal sample masses, 0.25 gram and 0.5 gram as summarized in Table AAA-10. In addition, one extraction for NIST SRM No. 1579a at 0.25 gram mass and duplicate extractions for ELPAT samples at the 0.5 gram mass were included to assess the processing control for the sample set. This set of samples was prepared by a single technician using the modified NIOSH method 7082 within a single sample preparation batch to minimize any potential between-batch effects. A similar set was prepared by a second technician using a different set of 10 homogenized field samples to generate additional data. A different set of field samples was required due to limits in the total mass of sample material available for individual samples.

The following conclusions are suggested from the Test 4 results presented in Tables AAA-11, AAA-12 and AAA-13:

(1) Recoveries greater than 90% suggest efficient extraction occurred in each batch using the modified NIOSH method 7082 for the NIST SRM No. 1579a and ELPAT samples at the 0.25 gram and 0.5 gram sample mass, consistent with the recoveries observed in Test 3 for the extraction of 0.25 gram and 0.5 gram NIST SRM No. 1579a.

(2) There is no significant difference in variability between pairs of samples weighing 0.25 grams and pairs weighing 0.5 grams. The root-mean-square relative percent difference between duplicates weighing 0.25 grams in Table AAA-12 is

21.9% as compared to 25.9% for duplicates weighing 0.5 grams. The difference is not statistically significant.

(3) Variability between laboratory duplicate samples, as measured by relative % differences in lead results for subsamples taken from the same homogenized parent sample, is inconsistent. Relative % differences between like sample masses and between different samples masses ranged from 0.1% to 47.5% and 0.2 and 66.2%, respectively.

Table AAA-10. Summary of Design Parameters for Test 4.

Method[a]	Sample Type[b]	Nominal Sample Mass (grams)	No. of Replicates at Each Mass
NIOSH-B	NIST	0.25, 0.5	1
	ELPAT	0.25	2
	10 FIELD SAMPLES	0.25, 0.5	2
NIOSH-D	NIST	0.25, 0.5	1
	ELPAT	0.25	2
	10 FIELD SAMPLES	0.25, 0.5	2

[a] NIOSH-B = modified method 7082 performed by technician B
NIOSH-D = modified method 7082 performed by technician D
[b] NIST = SRM No. 1579a, lead level of 11.995%
ELPAT = samples from round 1, sample 2, reference value of 0.5568%
FIELD SAMPLES = field samples from Louisville

Table AAA-11. Summary Results for Test 4: The Effect of Lead Recovery from NIST SRM 1579a and ELPAT samples at Variable Sample Mass using modified NIOSH method 7082.

Nominal Sample Mass[a] (grams)	Lead Recovery Results using modified NIOSH method 7082			
	NIST SRM No. 1579a		ELPAT[b]	
	Person Code[c]	Lead Recovery	Person Code[c]	Lead Recovery
0.25	B	97.9%	B	100.9% 94.1%
	D	na[d]	D	95.7% 93.9%
0.5	B	93.0%	B	na[e]
	D	91.0%	D	na[e]

[a] Actual sample mass was within ±13% of the nominal sample mass.
[b] ELPAT samples from round 1, sample 2, reference value of 0.5568%
[c] Codes represent preparation of samples by specific technicians.
[d] Not available, sample was inadvertently missed by technician.
[e] Not available - not planned for extraction at this mass because of insufficient material

Table AAA-12. Summary Results for Test 4: The Effect of Lead Results from Filed Samples at Variable Sample Mass using modified NIOSH method 7082 Performed by Technician B[a].

Field Sample ID No.	Nominal Sample Mass[b]	Mean Lead Results (mg/g)[c]	Relative % Difference between same Mass[d]	Relative % Difference between different Mass[e]
905545	0.25	3.884	1.2	2.1
	0.5	3.804	24.6	
905541	0.25	3.384	43.5	66.2
	0.5	6.735	44.2	
905533	0.25	1.709	33.7	na[f]
	0.5	na[f]	na[f]	
905597	0.25	1.962	4.9	5.1
	0.5	1.864	0.7	
905604	0.25	2.038	0.1	4.5
	0.5	1.949	0.4	
905524	0.25	4.249	1.3	0.4
	0.5	4.266	0.4	
905605	0.25	1.495	33.7	9.5
	0.5	1.644	26.5	
905564	0.25	131.213	16.9	4.3
	0.5	137.033	23.1	
905592	0.25	21.672	8.0	37.1
	0.5	31.556	46.9	
905501	0.25	67.722	16.0	27.4
	0.5	89.186	5.1	

[a] Codes represent preparation of samples by specific technicians.
[b] Actual sample mass was within ±13% of the nominal sample mass.
[c] Mean of two replicates
[d] Absolute value calculated using the following:

$$\frac{(\text{mg/g of 1st duplicate - mg/g for 2nd duplicate})}{(\text{mean mg/g for both duplicates})}(100)$$

[e] Absolute value calculated using the following:

$$\frac{(\text{mean mg/g for 0.25g - mean mg/g for 0.5g})}{(\text{mean mg/g for both 0.25g and 0.5g})}(100)$$

[f] na = not available, sample was inadvertently spilled by a technician.

Table AAA-13. Summary Results for Test 4: The Effect of Lead Results from Filed Samples at Variable Sample Mass using modified NIOSH method 7082 Performed by Technician D[a].

Field Sample ID No.	Nominal Sample Mass[b]	Mean Lead Results (mg/g)[c]	Relative % Difference Between Same Mass[d]	Relative % Difference Between Different Mass[e]
905591	0.25	2.253	30.8	7.1
	0.5	2.418	3.1	
905593	0.25	1.255	5.7	5.3
	0.5	1.324	0.1	
905507	0.25	4.224	19.7	0.2
	0.5	4.215	21.0	
905527	0.25	3.380	4.2	3.6
	0.5	3.504	8.2	
905528	0.25	44.385	4.9	8.8
	0.5	48.468	0.5	
905587	0.25	63.907	40.3	45.4
	0.5	40.253	17.3	
905531	0.25	2.275	3.4	15.7
	0.5	2.663	4.5	
905521	0.25	38.138	27.2	11.6
	0.5	42.845	8.9	
905523	0.25	32.319	1.2	14.5
	0.5	37.360	3.6	
905590	0.25	34.178	47.5	34.5
	0.5	48.429	8.4	

[a] Codes represent preparation of samples by specific technicians.
[b] Actual sample mass was within ±13% of the nominal sample mass.
[c] Mean of two replicates
[d] Absolute value calculated using the following:

$$\frac{(\text{mg/g of 1st duplicate - mg/g for 2nd duplicate})}{(\text{mean mg/g for both duplicates})}(100)$$

[e] Absolute value calculated using the following:

$$\frac{(\text{mean mg/g for 0.25g - mean mg/g for 0.5g})}{(\text{mean mg/g for both 0.25g and 0.5g})}(100)$$

Inconsistencies are suspected to be a result of matrix variations and sample homogeneity variations among the field samples.

Based on the conclusions obtained from Tests 3 and 4 combined with the logical assumption that the contribution to variability from sample inhomogeneity increases with decreasing aliquot mass, a decision was made to limit sample aliquots to a nominal 0.5 gram sample mass for processing paint chip samples using the modified NIOSH method 7082 summarized in Table AAA-5. If a homogenized individual paint chip sample was less than 0.5 gram, then all of the sample was extracted. Otherwise, a nominal 0.5 gram subsample was extracted.

The following errors have been discovered in the Mega-Doc.

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Incorrect For example, there are 31 error rates exceeding 10%; of these, only two were reduced below 10% by use of the average, and both improvements were small (11.1 % versus 10.2% false negative rates for the Lead Analyzer K-shell on concrete and 7.4% versus 6.8% false negative rates for the Microlead I on plaster).

Corrected For example, there are 31 error rates exceeding 10%; of these, only two were reduced below 10% by use of the average, and both improvements were small (11.1 % versus 7.4% false negative rates for the Lead Analyzer K-shell on concrete and 10.2% versus 6.8% false negative rates for the Microlead I on plaster).

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Incorrect: Section of table missing and incorrect ratio2 formula.

Corrected: Present, and RATIO2 = AREA2 ÷ AREA1

Table 4-17 (cont). Lead Levels in Field Duplicate Samples in **Area Units (mg/cm²)**, Sorted by RATIO1.

SAMPLE ID	AREA1	AREA2	SUBSTRATE	CITY	LOG(AREA1)	LOG(AREA2)	RATIO1	RATIO2

81256	0.2627	0.2479	Plaster	Philadelphia	-1.33682	-1.39473	1.05962	0.94373
80421	0.0265	0.0281	Wood	Denver	-3.63174	-3.57270	1.06082	1.06082
905594	0.1019	0.0960	Plaster	Louisville	-2.28412	-2.34388	1.06157	0.94200
905543	0.0042	0.0040	Metal	Louisville	-5.46894	-5.52956	1.06250	0.94118
81824	0.2982	0.2799	Plaster	Philadelphia	-1.21006	-1.27343	1.06542	0.93859
905508	5.3777	5.0434	Wood	Louisville	1.68226	1.61809	1.06628	0.93784
905511	10.2236	9.5797	Wood	Louisville	2.32469	2.25964	1.06721	0.93702
80706	2.0836	2.2249	Metal	Denver	0.73409	0.79970	1.06781	1.06781
905593	0.1269	0.1359	Plaster	Louisville	-2.06436	-1.99595	1.07081	1.07081
80375	0.0021	0.0023	Plaster	Denver	-6.16582	-6.09683	1.07143	1.07143
905545	0.4320	0.4024	Metal	Louisville	-0.83927	-0.91027	1.07358	0.93147
80612	0.0013	0.0014	Metal	Denver	-6.63773	-6.56417	1.07634	1.07634
905550	0.6764	0.7285	Metal	Louisville	-0.39094	-0.31675	1.07702	1.07702
81731	0.5362	0.5795	Concrete	Philadelphia	-0.62319	-0.54564	1.08064	1.08064
905570	0.1341	0.1239	Wood	Louisville	-2.00913	-2.08855	1.08266	0.92365
905564	14.0468	15.2450	Wood	Louisville	2.64239	2.72425	1.08530	1.08530
81833	0.2160	0.1990	Plaster	Philadelphia	-1.53266	-1.61460	1.08539	0.92133
905531	0.2561	0.2780	Concrete	Louisville	-1.36233	-1.28014	1.08566	1.08566
80529	0.0177	0.0163	Wood	Denver	-4.03589	-4.11905	1.08672	0.92020
81752	0.5324	0.5789	Plaster	Philadelphia	-0.63030	-0.54656	1.08735	1.08735
80323	0.0099	0.0108	Wood	Denver	-4.61826	-4.53285	1.08916	1.08916
905514	0.2103	0.1930	Wood	Louisville	-1.55943	-1.64517	1.08953	0.91783
81945	0.1976	0.1813	Metal	Philadelphia	-1.62136	-1.70771	1.09019	0.91727
80778	7.4628	6.8423	Plaster	Denver	2.00993	1.92313	1.09068	0.91686
905506	11.2622	12.2941	Wood	Louisville	2.42145	2.50912	1.09163	1.09163
905521	3.7888	4.1370	Plaster	Louisville	1.33206	1.41998	1.09190	1.09190
81427	0.6456	0.5907	Plaster	Philadelphia	-0.43756	-0.52640	1.09290	0.91500
905516	2.4268	2.6531	Wood	Louisville	0.88659	0.97571	1.09322	1.09322
905529	0.3228	0.3537	Plaster	Louisville	-1.13066	-1.03924	1.09572	1.09572
80444	0.1031	0.1130	Wood	Denver	-2.27167	-2.18019	1.09579	1.09579
80219	0.0681	0.0622	Wood	Denver	-2.68648	-2.77820	1.09606	0.91236
905580	0.0010	0.0009	Drywall	Louisville	-6.90114	-6.99464	1.09800	0.91074
905509	0.1567	0.1425	Wood	Louisville	-1.85346	-1.94829	1.09948	0.90952
80915	0.0588	0.0534	Brick	Denver	-2.83429	-2.92957	1.09996	0.90912
80071	1.3858	1.2537	Plaster	Denver	0.32626	0.22611	1.10534	0.90470

AREA1 = Lead level in primary sample (mg/cm^2) AREA2 = Lead level in field duplicate sample (mg/cm^2)
RATIO1 = max(AREA1, AREA2) ÷ min(AREA1, AREA2) RATIO2 = AREA2 ÷ AREA1