Analysis of Composite Wipe Samples for Lead Content

Final Report

For U.S. Environmental Protection Agency
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for Lead Content

Prepared by

Midwest Research Institute

for

Technical Programs Branch
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Office of Pollution Prevention and Toxics
Office of Prevention, Pesticides, and Toxic Substances
U.S. Environmental Protection Agency
Washington, D.C. 20460

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Contributing Organizations

The study described in this report was funded by the U.S. Environmental Protection Agency and the U.S. Department of Housing and Urban Development. The study was managed by the U.S. Environmental Protection Agency. The study was conducted collaboratively by two organizations under contract to the U.S. Environmental Protection Agency, Midwest Research Institute and Battelle Memorial Institute. Each organization's responsibilities are listed below.

Midwest Research Institute

Midwest Research Institute (MRI) was responsible for the study including the overall production of the Test Plan, Quality Assurance Project Plan, the experimental design, the laboratory analysis of the wipe samples, the statistical analysis, and writing the technical report.

Battelle Memorial Institute

Battelle Memorial Institute reviewed the Test Plan and provided input for the experimental design and laboratory analysis, assisted with the development of the report, and provided management oversight for the project.

U.S. Environmental Protection Agency

The U.S. Environmental Agency (EPA) cofunded 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 Work Assignment Manager was Mr. Todd Holderman. Barbara Leczynski and Brad Schultz also provided substantial direction in the study design. The EPA Project Officer was Jill Hacker. Cindy Stroup, Branch Chief of the Technical Programs Branch, initiated this study.

U.S. Department of Housing and Urban Development

The Department of Housing and Urban Development (HUD) cofunded the study.

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Executive Summary

The United States government has responded to the existing hazard posed by the presence of lead-based paint in the nation's housing stock by enacting Title X (the Residential Lead-based Paint Hazard Reduction Act, of federal housing legislation). This Act of Congress also enacted Title IV of the Toxic Substances Control Act (TSCA). Title X directs the U.S. Environmental Protection Agency (EPA), the U.S. Department of Housing and Urban Development (HUD), and other agencies to develop programs that would ultimately reduce lead hazards in housing. It is envisioned that Title X and Title IV programs will result in increased dust sampling in residences across the country. One of the preferred methods for sampling residential dust for lead uses baby or hand wipes. "Compositing" multiple wipes during sample collection has been suggested as a means to reduce the costs of both the sampling and analysis aspects of these activities. In fact, compositing of wipes is mentioned for clearance testing and risk assessment in the HUD *Guidelines for the Evaluation and Control of Lead-Based Paint Hazards in Housing*,[1] and in various EPA documents.

The term *composite* is used when two or more physical samples are combined for laboratory analysis. In the case of wipe sampling, two or more wipes collected from common components (e.g., floors or window sills) in a dwelling are combined in the field and then analyzed as a single sample. Prior to this study, the feasibility of modifying current sample preparation protocols to handle the increased mass of wipes and dust that result from compositing had not been demonstrated. In addition, it was uncertain whether any cost savings could be realized by compositing. Further, no standard method currently exists for the digestion of composite wipe samples for the analysis of lead in dust.

For those reasons, this study was undertaken to (1) investigate the feasibility of developing further the existing sample preparation methods to analyze composite wipe samples while meeting basic data quality objectives for accuracy and precision; and (2) determine whether compositing of wipes, if acceptable on a technical or performance basis, could reduce the cost of sample preparation and analysis relative to single-wipe methods.

From a number of available sample preparation methods, two were selected for evaluation based on discussions with experts in the lead analysis field and a literature review. The modified EPA Method 3050A and the Wisconsin Occupational Health Laboratory (WOHL) method were selected based on their anticipated ability to adequately handle the added mass of materials and dust expected in a composite of wipes. In addition, modified EPA 3050A is a commonly used procedure in many laboratories and is consistent

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[1] *Guidelines for the Evaluation and Control of Lead-based Paint Hazards in Housing*, June 1995. The National Center for Lead-Safe Housing, U.S. Department of Housing and Urban Development, Washington, D.C.

with the ASTM method titled "Emergency Standard Practice for Hot Plate Digestion of Dust Wipe Samples for Determination of Lead by Atomic Spectrometry" (ASTM Designation ES 36-94). The WOHL method is consistent with the method described in Appendix 14.2 of the HUD *Guidelines* and is applicable to composite wipes. The WOHL method also appeared to employ a more rapid digestion procedure, which could provide additional cost savings.

The experimental design for this study involved the following elements: (1) three off-the-shelf wipe materials (one hand-wipe product and two brands of baby wipes) were used; (2) a single-wipe sample, a two-wipe composite sample, and a four-wipe composite sample were used; (3) each wipe was spiked with one standard reference material (NIST SRM 2704) at either a low dust loading level of 0.5 g or a high level of 2 g. These dust loading levels correspond to lead levels of 80.5 µg and 322 µg per wipe, respectively; (4) three separate laboratories involved in lead testing and participating in the National Lead Laboratory Accreditation Program (NLLAP) performed the analysis; (5) each laboratory prepared the samples using both the EPA 3050A and the WOHL preparation methods; and (6) each sample was analyzed with two different analytical techniques, flame atomic absorption spectrometry (FAA) and inductively-coupled plasma spectrometry (ICP).

Performance specifications were met for EPA Method 3050A under all experimental conditions tested (average spike recoveries across all three laboratories ranged from 89% to 101%). Acceptable recoveries were achieved for the WOHL method for single-wipe samples and two-wipe composites at the low and high SRM loading levels, using either FAA or ICP analysis (average spike recoveries across all three laboratories ranged from 82% to 94%). However, difficulties were sometimes encountered with the WOHL method when compositing four wipes. This was the case for FAA and ICP results at the high SRM level (average spike recovery across all three laboratories was 78% for FAA and 65% for ICP). This was also the case for ICP results at the low SRM level (average spike recovery across all three laboratories was 79%). Acceptable recovery (average of 83% across all laboratories) was achieved at the low SRM loading level using the four-wipe WOHL method and FAA.

Based on individual laboratory results, again, all three laboratories met the performance specification for recovery under all experimental conditions using EPA Method 3050A. Acceptable performance was also achieved by each laboratory for the single-wipe samples and the two-wipe composites using the WOHL method and FAA. However, some laboratories experienced difficulties with the four-wipe WOHL method followed by FAA analysis. None of the three laboratories had consistently acceptable performance using the WOHL method followed by ICP analysis, regardless of the type of composite. Thus, it was found that acceptable results using the WOHL method with four-wipe composites could be achieved, although difficulties may be experienced by some laboratories.

On average, EPA Method 3050A yielded consistently higher recoveries than the WOHL method. In addition, FAA yielded higher recoveries on average than ICP for either digestion method. In all cases, intra-laboratory precision, based on the results from the three laboratories in the study, met the data quality objectives of less than 20% relative standard deviation for both the EPA 3050A and WOHL methods. The results from the four-wipe composites were consistently the least precise, although always acceptable.

The time required to perform each digestion procedure and composite type was recorded by each laboratory participating in the investigation. These results indicate that the time requirements for a given digestion method are fairly consistent across each composite type for a given laboratory. The multiwipe composites required slightly more time to digest, relative to the single-wipe samples. However, that increase in time was not proportional to the number of wipes in a composite: the two- and four-wipe composites did not take two and four times as long to digest as the single-wipe samples, respectively.

A sample preparation and analysis cost comparison was performed based on information provided by the three laboratories. Each laboratory also evaluated the cost aspects of compositing from a retrospective viewpoint. Using the single wipe as the basis for cost comparisons, an approximate cost reduction of 64% was estimated for the four-wipe composites using the EPA Method 3050A. An approximate cost reduction of 67% was estimated for the four-wipe composites using the WOHL method. That is, laboratory analysis of a four-wipe composite was approximately 65% less expensive than that of four single-wipe analyses for each method. Across all laboratories and composite types, approximate cost reductions of 25% to 70% were estimated for the WOHL method as compared to the EPA Method 3050A.

Acceptable performance can be achieved using the EPA Method 3050A for composites containing two or four wipes based on the recovery and precision results obtained with the standard reference material used in this study. The results were especially promising using FAA analysis. The WOHL method was found to provide acceptable performance for single wipes and two-wipe composites. It is possible to achieve acceptable performance with the WOHL method for four-wipe composites, although some difficulties might be encountered. Furthermore, the WOHL method was affected by the SRM loading level when ICP was used. A statistically significant, estimated loss in recovery of 1.75% for each gram of SRM was found when preparing the samples with the WOHL method and analyzing them by ICP. This limitation of the WOHL method may be a consideration in sampling applications where high dust loading levels are expected, such as for window sills and troughs in risk assessment. It is recommended that every laboratory would need to validate its performance with any method for the analysis of composite wipes as well as single-wipe samples. A significant cost benefit was derived from compositing. In summary, this study found that compositing two or four wipes into a single sample is a viable alternative for the analysis of lead in dust wipe samples and does result in cost savings.

Section 1
Introduction

1.1 Background

Household dust is recognized as a major pathway for conveying lead to children and adults. Numerous studies in residential settings have collected dust and analyzed it for lead to determine the lead exposure faced by the occupants in response to mandates cited in Title X.[1] The collection of dust in selected areas of the microenvironment using wipes and then compositing these wipes for analysis is a general methodology with potentially broad application because it is technically feasible and may result in cost savings. Compositing of wipes is mentioned for dust sampling in risk assessments, lead hazard screens, and clearance testing in Chapter 5 and Chapter 15 of the HUD *Guidelines*,[2] and in various EPA documents. There is, however, essentially no standard method available for the analysis of composited wipes. The present work assignment is intended to assess such a protocol.

Specific criteria on composite wipe sampling and analysis relative to clearance testing and other uses need to be set. A number of wipe sampling and analysis methods focus on single-wipe samples. The analysis of a composite wipe sample encompasses sample preparation and subsequent spectrometric analysis. Essentially no difference exists in the instrumental analysis of a single and a composite wipe sample, provided the same analytical method is used. The difference lies in the preparation of the sample.

Although several sample preparation methods are available, to our knowledge, none have been thoroughly evaluated for composite wipe samples. For example, the Wisconsin Occupational Health Laboratory (WOHL) developed a method for composite samples containing up to four wipes, but published evaluations of this method have been limited. It should be noted that the WOHL method was included as Appendix 14.2 in both the February 1995 draft and June 1995 versions of the HUD *Guidelines*.[4,2]

Prior to this study, it was expected that with further development, modified EPA SW-846 Method 3050A,[3] a hot plate digestion method, could produce an acceptable, practical method for preparing composite samples of up to four wipes. This method is a commonly used laboratory method for single wipes but is not typically used for composites wipes. As part of this study, modified SW-846 Method 3050A was further modified for two- and four-wipe composites.

1.2 Objectives

The two objectives of this investigation were to evaluate whether (a) preparation methods can be developed for composite wipes so that acceptable recoveries can be achieved and (b) these methods will reduce costs relative to the single-wipe methods.

This exploratory project was limited to investigating the feasibility of further developing the existing modified EPA SW-846 Method 3050A to achieve a recovery of 80% to 120% and a precision of 20% relative standard deviation, and estimating the precision and recovery of the WOHL method. National Institute of Standards and Technology (NIST) Standard Reference Material (SRM) 2704 (Buffalo River Sediment) with a certified value of 161 µg Pb/g of material was used to evaluate both digestion methods.

1.3   Scope of the Investigation

The primary efforts for this study were conducted at the contractor's main laboratories, where staff prepared all samples. The sample digestion and analysis efforts were conducted at a total of three laboratories. The inclusion of two commercial laboratories in this investigation provided for an assessment of digestion and analysis variability across multiple laboratories.

Two laboratories selected for participation in the study were involved in lead testing with the EPA National Lead Laboratory Accreditation Program (NLLAP).[5] The third laboratory was in the process of seeking NLLAP recognition at the time of the study. As part of the NLLAP, all three laboratories had successfully participated in the quarterly Environmental Lead Proficiency Analytical Testing Program (ELPAT) and had maintained a proficient rating for several years. At the onset of this study, two of the three laboratories had been accredited by the American Industrial Hygiene Association (AIHA), a professional association. The third laboratory applied for the Environmental Lead Analysis Program (ELAP) accreditation from the American Association for Laboratory Accreditation (A2LA), and recently received final approval from the A2LA Accreditation Council for NLLAP recognition.

The investigation of the two preparation methods was limited to testing three off-the-shelf wipe materials under two dust/lead-loading conditions using a single NIST SRM. Two brands of baby wipes and one brand of moist disposable towelettes were evaluated in the study. These types of products are not intended for this application, but historically have been suitable for lead analysis. Refer to Section 3.3 for more information on the selection of the wipes.

The digestion methods were evaluated using two different analytical methods each. The first analytical method (EPA SW-846 Method 6010A) determines metals in the digestates using inductively coupled plasma atomic emission spectrometry (ICP). The determination of lead by direct aspiration flame atomic absorption spectrometry (FAA) was the second analytical method (EPA SW-846 Methods 7000A [general] and 7420).

1.4 Overview of Report

The audience for this report are technical management personnel who are responsible for activities involving lead dust sampling and analysis. This may include laboratory management, risk assessors, inspectors, representatives of public housing authorities, and/or state/local health department representatives. The remaining sections of this report, which are described briefly below, present the results, the study design and objectives, and the methods.

Section 2 presents the discussion and conclusions made from the results of the study.

Section 3 presents the study objectives, experimental design, and discusses the selection of the methods, wipe materials, SRM, and SRM loadings used in the study.

Section 4 describes the procedure used for loading the wipe samples with SRM, the sample identifiers, sample containers, and sample storage/shipment.

Section 5 presents the sample analysis instrumentation, calibration standard sources, and method references used for the analysis. The laboratory observations made during the conduct of sample preparation and analysis also are presented in this section. The specific sample digestion procedures used are included in the appendices.

Section 6 describes the quality assurance and quality control (QA/QC) aspects of the study, including the data audit and data assessment. The results of the gravimetric data generated from loading the wipes with SRM, along with the QC sample results, also are presented in this section.

Section 7 describes the procedures used for processing the data. This section presents the results for the analytical data and the statistical analysis procedures. The method accuracy, method precision, and process parameter data also are included in this section.

The appendices contain the following information:

A   Method Review Summary—Protocols for Sample Preparation and Analysis of Lead in Single-Surface and Composite Wipe Samples
B   Modified SW-846 Method 3050A Acid Digestion Procedure for Single-Wipe Samples
C   Modified SW-846 Method 3050A Acid Digestion Procedure for Two-Wipe Composite Samples
D   Modified SW-846 Method 3050A Acid Digestion Procedure for Four-Wipe Composite Samples
E   WOHL Acid Digestion Procedure for Single-Wipe Samples

Section 2
Conclusions

This exploratory study was undertaken to investigate the feasibility of preparing and analyzing wipe composites from a performance and cost standpoint. Three laboratories were recruited to digest and analyze single-wipe samples, two-wipe composite samples, and four-wipe composite samples, using two digestion methods and two analytical methods. The three laboratories provided information regarding time, cost, and effort required for sample preparation and analysis, using the two digestion methods tested. Highlights of the results obtained from the statistical analysis of the analytical data, and tentative conclusions based on an evaluation of the process parameter information provided by the three laboratories are presented below.

2.1 Method Performance

The data quality objectives (DQOs) for the analytical results were those specified for the EPA Method 3050A: (1) the accuracy must be between 80% and 120% of the known spiked amount; and (2) the intra-laboratory precision must be no more than 20% relative standard deviation (%RSD). No DQOs are established for the WOHL method.

The results of this study, using NIST SRM 2704, show that the modified EPA Method 3050A met the DQOs for accuracy and precision, and yielded superior results over the WOHL method, regardless of SRM level, composite type, or analytical method. It was found to be possible that the DQO for recovery could be met using the WOHL method for single wipes and two-wipe composites. The WOHL method met the DQO for accuracy (80% to 120%) for the single wipes and two-wipe composites, regardless of SRM level or analytical method. However, for the four-wipe composites, average accuracies met the DQO only at the low SRM level and with FAA analysis. Otherwise, the method accuracy fell short of the DQO at the high SRM level (78% using FAA; 65% using ICP) and at the low SRM level using ICP (79%). Furthermore, the WOHL method was affected by the SRM loading level when ICP was used. A statistically significant, estimated loss in recovery of 1.75% for each gram of SRM was found when preparing the samples with the WOHL method and analyzing them by ICP. This limitation of the WOHL method may be a consideration in sampling applications where high dust loading levels are expected, such as for window sills and troughs in risk assessment. The WOHL method provided acceptable intra-laboratory precision results. Both preparation methods resulted in %RSD below 10% (compared to the 20% RSD DQO) in all cases.

While the modified EPA Method 3050A is an established procedure (for single-wipe samples only), the WOHL method is relatively new and the laboratories may not have been familiar with this method prior to the study. Furthermore, digesting composite wipes may have been a new procedure for these laboratories.

2.2 Effect of Number of Wipes

The EPA Method could be performed acceptably regardless of the number of wipes per sample: average recoveries ranged from 88% to 100% for single-wipe samples; from 91% to 101% for two-wipe composites; and from 94% to 100% for four-wipe composites. Similarly, the WOHL method provided acceptable results for single-wipe samples and two-wipe composites. Average recoveries ranged from 82% to 91% for single-wipe samples and from 82% to 94% for two-wipe composites. However, difficulties were sometimes encountered with the WOHL method when compositing four wipes. This was the case for both analytical methods (ICP and FAA) at the high SRM level (average recoveries of 65% and 78%, respectively) and at the low SRM level for ICP (average recovery of 79%). The WOHL method performed acceptably only for FAA at the low SRM level, with an average recovery of 83%. On average, the four-wipe composites provided percent recoveries approximately 12% lower than both the single-wipe and the two-wipe composites for the WOHL digestion method. In addition, the four-wipe composites consistently yielded the least precise intra-laboratory results, although in all cases the %RSD was less than 9%.

2.3 Analytical Method

In general, recovery differences between the two analytical methods were affected only slightly by the two digestion methods, with no apparent pattern between the two. Overall, across all factors in the study design, slightly higher recoveries were obtained with FAA than with ICP (average difference of approximately 7%, though not statistically significant). However, in most cases, differences in recoveries between FAA and ICP were larger when using the WOHL method (average difference of 8%) than when using the EPA 3050A Method (average difference of 3%), all other conditions held constant. In addition, ICP recoveries showed a slight decrease with increasing SRM loading for both digestion methods, while SRM loading levels had no effect on FAA recoveries under similar conditions. The implication is that either method will suffice, but there may be modest advantages of using FAA over ICP. Further, since the FAA instrument is generally less expensive to operate, it may be the preferred instrument when analyzing composites with these methods, especially if modified EPA 3050A is being used.

2.4 Cost

The time required to perform each digestion procedure and composite type combination was recorded by each laboratory participating in the investigation. The three laboratories also evaluated their costs associated with digestion and analysis of composite samples from a retrospective viewpoint. A limited sample preparation and analysis cost comparison was performed based on this information.

Based on this cost comparison, a cost-benefit can be derived from compositing. Using the single wipe as the basis for cost comparisons, an average cost reduction of 64% per wipe was estimated for the four-wipe composites using EPA Method 3050A. For example, suppose that a single wipe costs $20 to analyze. Based on the reduction of effort obtained from this study, a four-wipe composite would cost $28.50, a 64% cost reduction over $80, the cost of four single wipes. Similarly, a reduction of 67% was estimated for the four-wipe composites using the WOHL method. In general, the multiwipe composites required slightly more time to digest relative to the single-wipe samples; however, that increase was not proportional (i.e., the two- and four-wipe composites did not take two and four times as long to digest as the single-wipe samples, respectively).

The three laboratories involved in the study also estimated per-wipe cost reductions ranging from 25% to 70% for the WOHL method as compared to the EPA Method 3050A. Some laboratories may not achieve acceptable performance with the four-wipe composites using the WOHL method; in this case, the per-wipe cost reduction would not be realized. In addition, all three laboratories indicated that FAA required less time than ICP analysis, reporting an estimated 2 min per sample for FAA versus 3 to 5 min per sample for ICP. Finally, since FAA instruments are less expensive to purchase than ICP instruments, an additional cost reduction can be achieved from using FAA rather than ICP.

Section 3
Study Design

This section provides the overall study design for the development of the composite wipe method. The study objectives and the selection of the methods, wipe materials, and standard reference material (SRM) are discussed, along with the experimental design. A detailed description of the laboratory methods and data analysis is provided in subsequent sections and the appendices.

3.1 Study Objectives

Compositing of wipes, if acceptable, could have important cost containment implications in the effort to reduce childhood lead poisoning. Cost containment is important if lead hazard control and clearance testing following those efforts are to take place in millions of dwellings with lead-based paint hazards. However, specific criteria on composite wipe sampling and analysis relative to clearance testing and other uses need to be set. As part of this work, two digestion methods were investigated: (a) the modified EPA SW-846 Method 3050A, a hot plate digestion method commonly used for single wipes, and (b) the Wisconsin Occupational Health Laboratory (WOHL) method developed for up to four-wipe composites.

The two objectives of this investigation were to evaluate whether (a) preparation methods can be developed for composite wipes so that acceptable recoveries can be achieved and (b) these methods will reduce costs relative to the single-wipe methods.

The scope of this project was limited. First, the feasibility of further modifying the EPA SW-846 Method 3050A for two- and four-wipe composites to achieve a recovery of 80% to 120% and a precision of 20% relative standard deviation (RSD) was investigated. Second, the precision and recovery of the WOHL method were estimated when using composite wipe samples. Only a single reference material, NIST SRM 2704 with a certified value of 161 µg Pb/g of material, was used to evaluate both methods. The optimization or validation of a composite wipe preparation method was not included in the scope of this project.

During the planning phase of the study, a Test Plan was prepared that addressed the background, objective, and scope of the investigation. The Test Plan also presented the experimental design, laboratory methodology, and the QA/QC aspects for the study. The Test Plan was then submitted to six organizations for peer review, and comments were received from nine reviewers. Their comments fell into the following general categories:

- Statistical design and analysis

- SRM selection and loading levels

- Wipe brands and the number of brands to be tested

- Analytical methods and potential interferences

- Digestion methodology (e.g., beaker sizes, final digest volumes, and reagent volumes)

- Types of composites to include in the investigation

- Type and number of quality control samples to be included in the sample batches

- Number of laboratories to be included in the study

All review comments were discussed in detail during the planning phase of the study. Where appropriate and feasible, the Test Plan was revised to reflect the reviewers' comments and suggestions.

As a result, the investigation of the two preparation methods encompassed the following:

- Testing three off-the-shelf wipe materials under two dust/lead-loading conditions using a single NIST standard reference material. An attempt was made to obtain wipes from two different manufacturers' lots for each brand.

- Performing all sample digestion and analysis work at three laboratories to capture digestion and analysis variability across multiple laboratories.

- Using two analytical methods—inductively coupled plasma atomic emission spectroscopy (ICP) and flame atomic absorption spectroscopy (FAA)—to analyze the wipes for lead at each laboratory.

- Estimating the digestion times and relative cost factors for each composite type and each digestion procedure at the three laboratories.

- Documenting all equipment, facilities, and materials used at each laboratory.

- Comparing the performance and relative costs of the two methods within and among the laboratories.

Figure 3-1 shows, in a schematic form, the layout of this study.

- Selected three brands of wipes

- Defined sample types:
  - single wipe
  - two-wipe composite
  - four-wipe composite

- Spiked individual wipes with SRM 2704 at one of two levels:
  - 0.5 g (low)
  - 2 g (high)

- Selected three laboratories

- Prepared samples using two different methods at each laboratory

- Analyzed each sample using two different analytical techniques at each laboratory
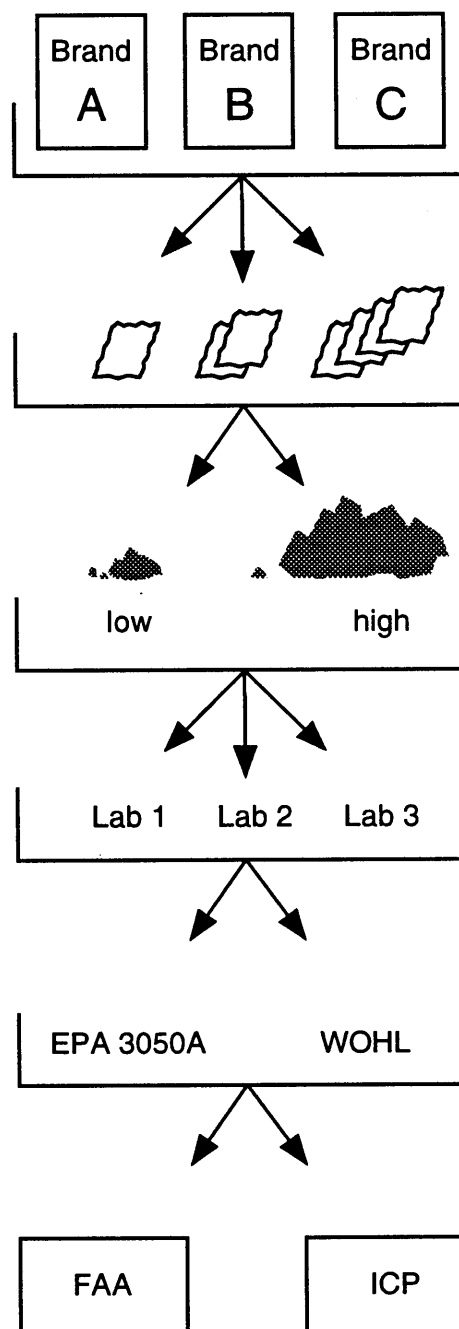
Figure 3-1. Design Layout

3.2 Method Selection

A literature search was previously conducted to identify existing sample preparation and analysis methods for lead in single surface and composite wipes. The methods found were presented in a report titled, "Protocols for the Collection and Analysis of Composite Wipe Samples."[6] A summary of this report, along with other methods evaluated, is presented in Appendix A. The advantages and disadvantages of each method were weighed before method selection and performance of this investigation. The methods found during the literature search are briefly described below:

- Hot plate digestion using nitric acid (HNO_3) and hydrogen peroxide (H_2O_2): This is a commonly used digestion procedure that could be adapted to multiwipe composites and provide suitable lead recoveries. Existing methods that are applicable to or have been adapted to digesting single-wipe samples include the modified EPA SW-846 Method 3050A, ASTM Method ES 36–94, NIOSH Method 7082, and a method used at Azimuth Laboratories.

- Hot plate digestion using HNO_3 and hydrochloric acid (HCl): This method is applicable to single surface and multiwipe composites. Existing methods include the WOHL method, which is equivalent to the method included in Appendix 14.2 of the HUD *Guidelines* (February 1995 draft and June 1995 versions).[4,2]

- Hot plate digestion using HNO_3 and perchloric acid ($HClO_4$): NIOSH Method 7300 exists, but a perchloric acid fume hood is required for this type of digestion due to the potential explosion hazard associated with $HClO_4$.

- Room temperature extraction and hot plate digestion using 1 M HNO_3: This method is used by the University of Cincinnati Medical Center, Department of Environmental Health, for hand wipe composites for the analysis of lead and arsenic.

- Hot plate and microwave digestion: Two methods were found that used a combination of hot plate and microwave oven for heating the digests. These methods also used perchloric acid, which is potentially explosive.

- Room temperature extraction: This extraction method uses 0.1 N HCl at room temperature.

- Ultrasonification: This procedure uses 10% HNO_3 and a commercial ultrasonic cleaner to extract the wipes.

- Dry-ashing: Dry-ashing procedures use furnace heating to oxidize the sample. The resulting ash is solubilized in HNO_3 or HCl and is diluted to volume with water.

The methods were reviewed to determine if they could be adapted to multiwipe composites. Experts in lead analysis were consulted from NIOSH, Azimuth Laboratories, the University of Cincinnati Medical Center Department of Environmental Health, and EPA\Atmospheric Research Exposure Assessment Laboratory (AREAL) to discuss the basic characteristics of a wipe method that must respond to the challenge of handling an added mass of wipe material and dust. Based on the literature review and further discussions, two types of hot plate digestion methods were selected.

The first method selected for inclusion in the study was the modified EPA SW-846 Method 3050A. This method was selected because it is a commonly used procedure that had a strong likelihood of achieving the accuracy and precision objectives of the study. The ASTM method titled "Emergency Standard Practice for Hot Plate Digestion of Dust Wipe Samples for Determination of Lead by Atomic Spectrometry" (ASTM Designation ES 36-94) is consistent with the modified Method 3050A for single surface samples. The modified Method 3050A is also very similar to a method used by Azimuth Laboratories.

The modified EPA SW-846 Method 3050A was routinely used for digesting single-wipe samples using HNO_3 and H_2O_2. As part of this study, further modifications were made to the method to accommodate the two- and four-wipe composite samples. Additional safety measures were required to deal with the larger quantities of acids needed to perform composite sample digestion. The modified EPA SW-846 3050A methods used for single surface, two-, and four-wipe composites are presented in Appendices B, C, and D, respectively.

The second method selected was the WOHL method, which is the method presented in Appendix 14.2 of the HUD *Guidelines* (February 1995 draft and June 1995 versions). As presented in the HUD *Guidelines*, this method is applicable to the analysis of single-wipe samples or composite samples of up to four wipes. This method was selected because it appeared to be a more rapid digestion procedure; however, it has been only used for a limited number of tests. The WOHL method uses HNO_3 and HCl for the digestion. The WOHL digestion procedures for single surface and multiwipe composites are presented in Appendices E and F, respectively.

It should be noted that approximately 70% to 80% of the laboratories participating in the ELPAT program routinely use hotplate digestion methods. These laboratories could thus readily adopt a proposed composite sample digestion technique. Most of the remaining participating laboratories use microwave digestion techniques. To our knowledge, there is no commercially available microwave digestion vessel that can handle multiple wipes. Therefore, microwave digestion would be precluded as an alternative digestion procedure for composite sample digestion unless some method involving predigestion of the wipe media could be developed.

The two types of digestion methods were evaluated using two different analytical methods each. The first analytical method (EPA SW-846 Method 6010A) determines metals in the digestates using ICP. The determination of lead by direct aspiration FAA

(EPA SW-846 Methods 7000A [general] and 7420) was the second analytical method. These methods of analysis were selected because they are techniques commonly used by commercial laboratories for lead analysis. The two methods were included in the study to capture the analysis variability for each digestion method and analysis method combination. It should be noted that about 60% of ELPAT participating laboratories use FAA analysis and about 35% of them use ICP-AES analysis, so the two selected instrumental techniques cover the most commonly used instrumental method used in the ELPAT program for lead wipes.

3.3 Wipe Selection

The evaluation criteria used for the selection of candidate wipes included in the composite wipe study were taken from Appendix 13.1 of the HUD *Guidelines*, and are summarized below:

- The wipes should contain low background lead levels (< 5 μg/wipe). The analysis of wipe blanks was incorporated into the test design to provide background levels for each brand and each lot of wipes.

- The wipes must be durable, of a single thickness, and should not tear easily.

- Aloe vera must be avoided to minimize lead background levels.

- The wipes must be the premoistened type for field sampling purposes; dry materials such as gauze and filter paper are excluded (wipes containing alcohol may be used as long as they do not dry out).

- The wipes selected should yield 80% to 120% recovery rates from samples spiked with leaded dust.

It should be mentioned that a specification for wipes was recently adopted by ASTM. The Standard Specification for Wipe Sampling Materials for Lead in Surface Dust (ASTM E1792-96) establishes limits for background lead in the wipe, ruggedness when used on various typical surfaces, lead recovery, and collection efficiency. The specification is presently undergoing further change in ASTM to improve the specification in regard to the uniformity of moisture and the wipe thickness range specified.

The three brands of wipes selected for inclusion in the composite wipe investigation were Wash a-bye Baby wipes (Scott Paper Company), Baby Wipes with Lanolin (Rockline, Inc.), and Wash'n Dri moist disposable towelettes (Colgate-Palmolive). These wipes were selected based on the above criteria. The brands were also selected because they historically have been used by practitioners in the field for sample collection of settled leaded dust, and/or are listed in the HUD *Guidelines*.

The wipe manufacturers were contacted during the planning phase of the study to gather information on manufacturers' lot designations and other characteristics for each brand of wipe. The inquiries were made because several sources involved in lead testing suggested that potential differences in background lead levels in wipes may exist, since the chemical constitution of the wipes is not exactly the same.

In an effort to capture data on potential differences among different manufacturers' lots for wipes of the same brand, the contractor purchased two brands from different geographical regions of the United States because lot designations were not obtainable from the manufacturers. One set of Wash a-bye Baby wipes and one set of Wash'n Dri moist disposable towelettes were purchased in the metropolitan Kansas City area, and these sets were assigned a lot code of 1. Another set of wipes for these two brands was purchased in North Carolina, and these sets were assigned a lot code of 2. This approach was used following one wipe manufacturer's suggestion to purchase the product from different stores and locations to acquire different lots.

The third brand, Baby Wipes with Lanolin, was a replacement product for a brand formerly known as Little Ones Baby Wash Cloths, supplied from a mass merchandiser. The replacement brand was used because the original supplier discontinued distribution of Little Ones Baby Wash Cloths in many regions of the country. The replacement brand was selected because it was a brand often used by HUD in public housing dust sampling. Different lots could not be evaluated since this brand could only be purchased from a single supplier.

3.4   Standard Reference Material (SRM) Selection

NIST SRM 2704 (Buffalo River Sediment) with a certified lead concentration of 161 μg Pb/g was selected for applying lead onto the wipes. NIST SRM 2704 was selected because a dust matrix SRM was not available for purchase at the time of the study, and the schedule for the study did not permit time for preparing evaluation materials for lead in dust. In addition, a large database of lead percent recovery results for NIST SRM 2704 and single-wipe samples existed for reference to the results of this study.

The wipes were loaded with NIST SRM 2704 at one of two levels, either 0.50 g or 2 g per wipe. The resulting lead-loading levels were 80.5 and 322 μg/wipe. The EPA has the same interim dust clearance standards as the HUD *Guidelines*, which range from 100 to 800 μg/ft^2, depending on the surface sampled (Interim 403 Guidance, Memorandum of July 14, 1994, from Lynn Goldman to Regional Division Directors). Assuming a 1 ft^2 area is wiped during sample collection, the lower of the two loading levels selected is approximately 80% of the 100 μg/ft^2 value, and the higher of the two is in the mid-range of the interim HUD dust clearance standards. Two loading levels were selected to assess the impact of the quantity of material applied to the wipe on percent recovery of lead. The high loading level also provided a dust loading challenge to the methods for all sampling applications (i.e., risk assessments, lead hazard screens, and clearance testing).

3.5 Experimental Design

To meet the study objectives, a laboratory experiment was designed based on a number of parameters. However, due to the exploratory nature of this project, only a small number of parameters were varied, while other experimental conditions were defined and held constant. Furthermore, since this investigation only considers the sample preparation steps for each method, the only relevant parameters are those pertaining to the sample preparation activities. These parameters can be classified into two major categories: method and process parameters. A description of the parameters held constant throughout the experiment, and the method and process parameters that were varied are discussed next.

Design constants: To minimize the size of the experiment, the following parameters were held constant throughout the study. Therefore, the effect of these parameters on method performance was not tested.

- A single SRM was used for spiking. NIST SRM 2704 with a concentration of 161 µg Pb/g of material was selected for the purpose of comparing the study results with past study results.

- A single dust-loading procedure (see Appendix G, 3.6) for applying the SRM on the wipes was followed.

- Work at each laboratory was performed by laboratory technicians with comparable skill levels. It was assumed that the skill level required for the WOHL method was the same as that required for the EPA SW-846 Method 3050A.

- Three commercially available wipe brands were tested: Wash a-bye Baby wipes, Baby Wipes with Lanolin, and Wash'n Dri moist disposable towelettes. These brands of wipes were selected because they are commonly used by practitioners in the field.

Method parameters: The effect of the following parameters (along with their levels, which are indicated parenthetically) on lead recovery from wipe composites was investigated:

- Sample preparation method (2)
- Number of wipe(s) per composite (3)
- Dust loading (2)
- Laboratory (3)

Sample preparation method (2): Two methods were tested: the modified EPA SW-846 Method 3050A for single-wipe samples, further modified to accommodate two- and four-wipe composites, and the WOHL methods for single and multiwipe composites. The modified EPA SW-846 Method 3050A is described in Appendix B. This method,

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further modified for two- and four-wipe composites, is described in Appendices C and D, respectively. The WOHL methods for single and multiwipe composites are provided in Appendices E and F, respectively.

Number of wipe(s) per composite (3): Three levels were considered in this exploratory study: a single wipe, a composite of two wipes, and a composite of four wipes. This applies to each of the three brands of wipes. The single-wipe samples were included in the study to provide a reference base. The maximum of four wipes per composite was chosen for practical purposes. In addition, four wipes are the maximum number recommended for compositing according to the HUD *Guidelines*.

Dust loading (2): Two <u>dust</u> loading levels were tested: one standard dust material, NIST SRM 2704 with a lead concentration of 161 μg Pb/g, was spiked onto the wipes in amounts of 0.5 g/wipe or 2 g/wipe. This resulted in <u>lead</u> loadings of approximately 80.5 μg and 322 μg per single wipe. The maximum composite lead loading was therefore 1,288 μg for the four-wipe composite with a 2 g/wipe SRM loading.

Laboratory (3): Three laboratories were selected to perform the work in an identical fashion. A contractor prepared and shipped the wipes to two commercial laboratories and then participated in the study as the third laboratory performing the analyses. This approach incorporated the variability associated with digestion and analysis procedures among laboratories during the final data evaluation. A comparison of overall performance among laboratories per se was not the intent of this study.

Process parameters: To evaluate the effectiveness of using wipe composites, information on the following process parameters was recorded:

- Material costs (e.g., reagents and waste disposal)
- Time of sample preparation and relative cost factors
- Time of sample analysis
- Equipment and facility needs

Information on these process parameters was recorded and was an integral part of the set of evaluation factors, along with method precision and accuracy which were determined from the chemical analysis results.

A full factorial experimental design was used to assess the performance of the two digestion methods. This design was based on the experimental parameters discussed above. In addition, the following laboratory/technician constraints were carefully considered:

- Each method was tested separately in order to keep different digestion procedures separate for the laboratory technician.

- Only one type of composite was handled in a given batch. This facilitated the addition of different volumes of reagents to different composite types.

- Batch size considerations included the number of beakers that could fit on the hot plates. For example, Figures 3-2 and 3-3 show the number of beakers that could fit on a single hot plate (1 ft × 2 ft) for the modified EPA SW-846 Method 3050A and the WOHL method, respectively.

- All samples within a given hood would be handled by a single laboratory technician.

- Each batch included the required number and type of QC samples, as described in Section 6.

Number of replicates: Considering the three experimental parameters (sample preparation method, number of wipes per composite, and dust loading) and their respective levels, a total of $2 \times 3 \times 2 = 12$ unique combinations were tested for each of the three types of wipes at the three laboratories. To assess method variability, a number of replicate runs were performed. The number of replicates were two or four, depending on the method used and the number of wipes per composite. Tables 3-1 and 3-2 provide the complete layout for the two methods. The number of replicates in each case was guided by the size of each batch and a minimum of two replicate runs for each unique combination of parameters. The size of digestion beakers and hot plates determined how many beakers could fit on one hot plate (1 ft × 2 ft). Two hot plates could be used simultaneously in one hood; therefore, one sample batch consisted of samples processed by a single technician using one or two hot plates (see Figures 3-2 and 3-3).

Lots within wipe brands: As mentioned earlier (see Section 3.3), wipes from two different lots were only obtained for two of the three wipe brands (i.e., because one brand was only available from one location). This was done to account for potential lot-to-lot variability. This decision did not affect the design layouts shown in Tables 3-1 and 3-2. The wipes that were loaded with SRM were selected from the two lots in a balanced fashion; that is, equal numbers of wipes from each lot were assigned to the two loading treatments (0.5 g and 2 g). In addition, all wipe samples in a given composite sample consisted of wipes from a same lot. Wipe blanks were randomly selected from the two lots so as to obtain approximately equal numbers of blank wipes of each lot.

The three brands of wipes were selected based on the HUD *Guidelines* and other criteria listed in Section 3.3. These three brands of wipes were included in the study to take into account variability among wipes. The intent was not to compare the brands but to include a replication scheme encompassing replicates within a brand and across qualifying brands available on the market. Therefore, for analysis purposes, the type of wipe was considered a replicate level rather than a parameter in the design. In summary, from Tables 3-1 and 3-2, the following number of samples were prepared for each laboratory, requiring the following number of wipes per laboratory:

Method/QC	Number of samples	Number of wipes
EPA SW-846 Method 3050A	60[a]	156[b]
QC	24[c]	33[d]
Total	84	189
WOHL Method	72[a]	168[b]
QC	18[c]	21[d]
Total	90	189
Grand total	174	378

[a] This number is the sum of the 3 entries in row r5 in Table 3-1 or Table 3-2, respectively, for the two methods.
[b] This number is the sum of the 3 entries in row r6 in Table 3-1 or Table 3-2, respectively, for the two methods.
[c] This number is the sum of the 3 entries in row r15 in Table 3-1 or Table 3-2, respectively, for the two methods.
[d] This number is the sum of the 3 entries in row r17 in Table 3-1 or Table 3-2, respectively, for the two methods.

This experimental design applied to all three laboratories participating in the study. Each laboratory followed identical digestion and analysis procedures. Aliquots of each prepared sample were analyzed at each laboratory by both ICP and FAA.
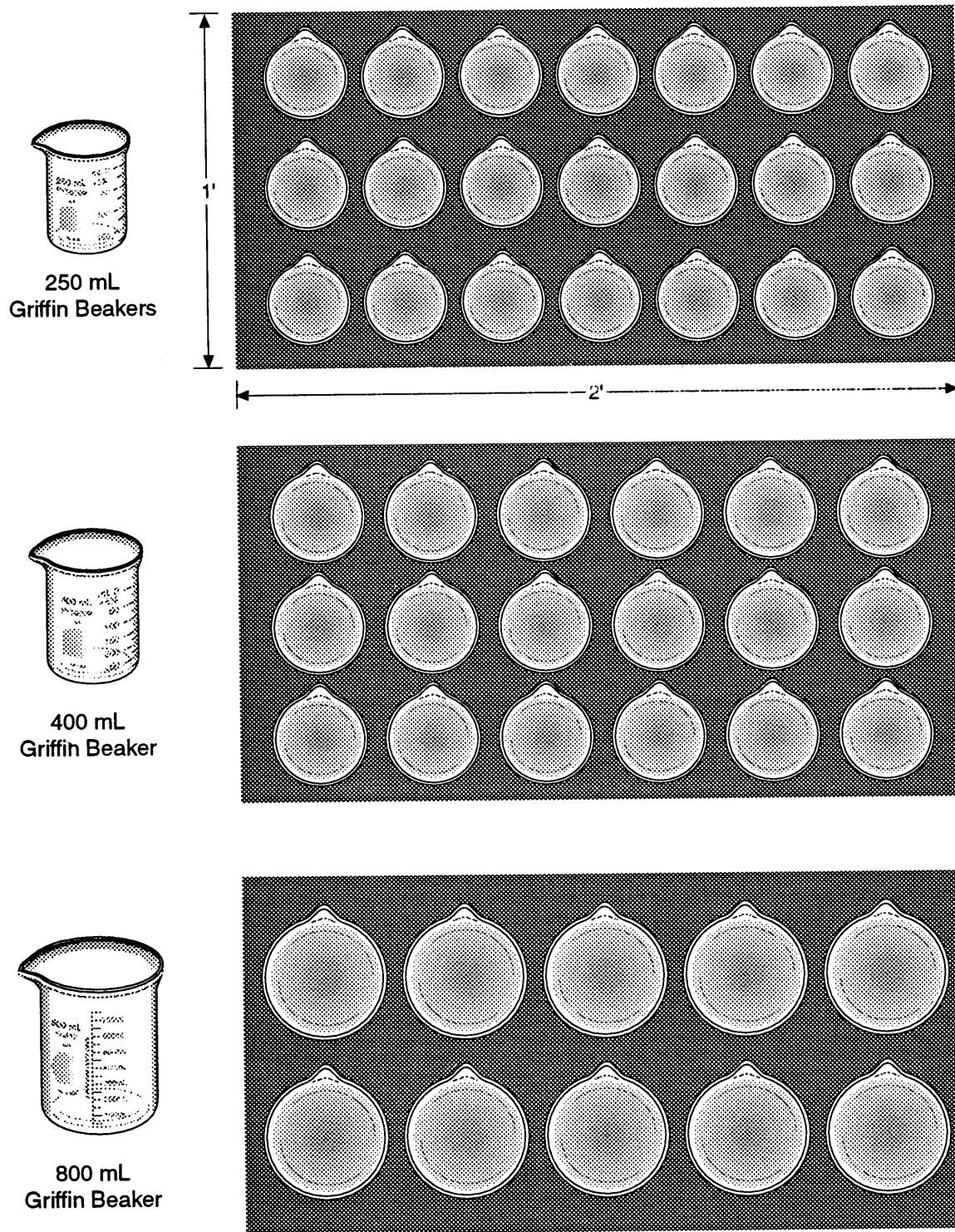
Figure 3-2. Hot Plate Configuration for the Modified EPA SW-846 Method 3050A

250 mL
Phillips Beaker
Single Wipe,
2-Wipe Composite,
or 4-Wipe Composite
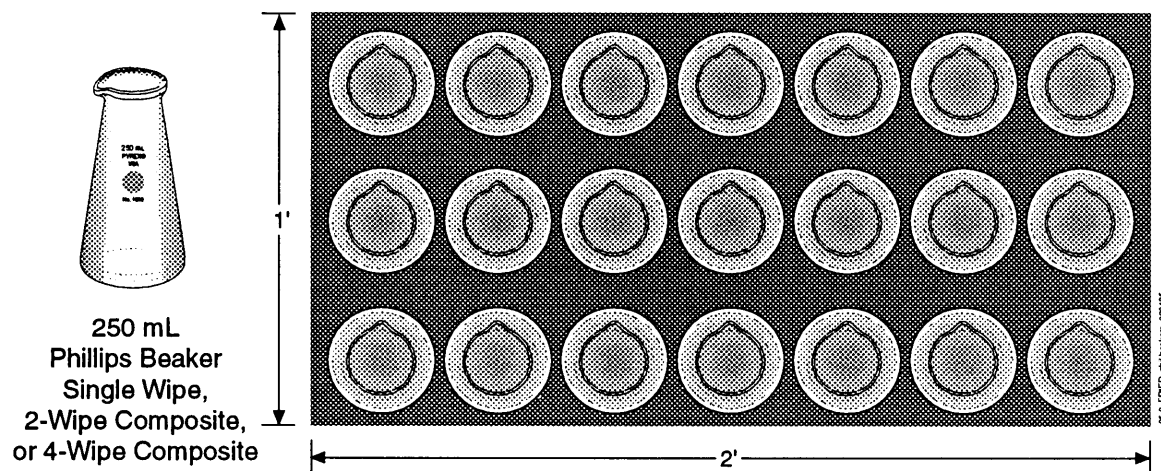
Figure 3-3. Hot Plate Configuration for the Wisconsin Occupational Health Laboratory (WOHL) Method

Table 3-1.   Design Layout for the Modified EPA SW-846 Method 3050A

Row No.	SRM-loaded wipe samples			
r1	Number of wipes per composite	1 wipe	2-wipe composite	4-wipe composite
r2	Type of wipe (brand)	A, B, and C	A, B, and C	A, B, and C
r3	SRM loading per wipe	0.5 g and 2 g	0.5 g and 2 g	0.5 g and 2 g
r4	**Number of replicate runs**	**2**	**4**	**4**
r5	Number of composite wipe samples (r2xr3xr4)	12	24	24
r6	Number of wipes (r5xr1)	12	48	96
	Laboratory configuration			
r7	Size of digestion beaker	250 mL	400 mL	800 mL
r8	Maximum number of beakers per hot plate	21	18	10
r9	Number of hot plates needed	1	2	4
r10	Number of batches (maximum 2 hot plates/batch)	1	1	2
	Quality control samples per batch (see Section 6)			
r11	Method blank (MB)	1[a]	1	1
r12	Wipe blank (WB)	A, B, and C	A, B, and C	A, B, and C
r13	Laboratory control sample (LCS)	2[b]	2	2
r14	Number of QC samples per batch (r11+...+r13)	6	6	6
r15	Total number of QC samples (r10xr14)	6	6	12
r16	Number of QC wipes per batch (r1xr12)	3	6	12
r17	Total number of QC wipes (r16xr10)	3	6	24
	Number of wipes and samples (analyses)			
r18	Total number of wipes needed (r6+r17)	15	54	120
r19	**Total number of samples (i.e., beakers)** r5+r15	**18**	**30**	**36**

[a] One laboratory prepared one extra method blank. See Section 6.2.2.
[b] One laboratory prepared two extra laboratory control samples. See Section 6.2.2.

Table 3-2. Design Layout for the Wisconsin Occupational Health Laboratory (WOHL) Method

Row No.	SRM-loaded wipe samples			
r1 r2 r3	Number of wipes per composite Type of wipe (brand) SRM loading per wipe	1 wipe A, B, and C 0.5 g and 2 g	2-wipe composite A, B, and C 0.5 g and 2 g	4-wipe composite A, B, and C 0.5 g and 2 g
r4	**Number of replicate runs**	**4**	**4**	**4**
r5 r6	Number of composite wipe samples (r2xr3xr4) Number of wipes (r5xr1)	24 24	24 48	24 96
	Laboratory configuration			
r7 r8 r9 r10	Size of digestion beaker Maximum number of beakers per hot plate Number of hot plates needed Number of batches (maximum 2 hot plates/batch)	250 mL 21 2 1	250 mL 21 2 1	250 mL 21 2 1
	Quality control samples per batch (see Section 6)			
r11 r12 r13	Method blank (MB) Wipe blank (WB) Laboratory control sample (LCS)	1[a] A, B, and C 2[b]	1 A, B, and C 2	1 A, B, and C 2
r14 r15	Number of QC samples per batch (r11+...+r13) Total number of QC samples (r10xr14)	6 6	6 6	6 6
r16 r17	Number of QC wipes per batch (r1xr12) Total number of QC wipes (r16xr10)	3 3	6 6	12 12
	Number of wipes and samples (analyses)			
r18	Total number of wipes needed (r6+r17)	27	54	108
r19	**Total number of samples (i.e., beakers)** r5+r15	**30**	**30**	**30**

[a] One laboratory prepared one extra method blank. See Section 6.2.2.
[b] One laboratory prepared two extra laboratory control samples. See Section 6.2.2.

Section 4
Sample Handling and Identification

This section addresses the aspects of loading the wipes with NIST SRM, laboratory sample handling, identification, containers, storage, and shipping. The sample handling procedure was carried out in accordance with the sequence specified by the test design outlined in Section 3. All samples analyzed in this investigation were either wipes spiked with a NIST SRM or various QC samples. All wipe samples were loaded with NIST 2704 (Buffalo River Sediment) at the contractor laboratory.

4.1 Wipe Loading Procedure

A planning session was held with project personnel to determine an efficient order, or loading sequence, that would reduce the chance for error in loading the wipes. This planning session was important because approximately 1,000 wipes were loaded with NIST SRM for the study. The test design was then sorted into the optimum order, and the bar code numbers were sequentially assigned. NIST SRM 2704 (Buffalo River Sediment) with a certified lead concentration of 161 µg Pb/g was used for loading lead onto the wipes.

A Mettler Model AE200 analytical balance was used for weighing the NIST SRM aliquots that were applied to the wipes. The wipes were loaded with NIST SRM 2704 at one of two levels, either 0.50 g or 2 g per wipe, as specified in the test design. The resulting lead-loading levels were 80.5 and 322 µg Pb/wipe. The weighed portion of reference material was placed on the wipe, the wipe was folded, and the SRM was pressed into the wipe as it was folded, and the loaded wipes were transferred to labeled Nalgene bottles for storage until sample preparation. The specific procedure used for loading the reference material onto the wipe(s) is presented in Appendix G.

The samples were randomly assigned to the three laboratories. This was done within the strata defined by SRM levels, composite types, wipe brands, digestion methods, and number of replicates. As a result, the make-up of samples with respect to these factors was identical for the three laboratories. The samples that were loaded with NIST SRM 2704 were segregated into the appropriate batches, incorporating the required number of wipe blanks into each batch, as specified in the test design. The samples were batched and placed in secure storage, pending transfer to the laboratories for sample digestion and analysis. Each laboratory participating in the study was responsible for incorporating the method blank and laboratory control samples into the batches as specified in the digestion procedures. The resulting wipe samples were digested using either the modified EPA SW-846 Method 3050A or the WOHL method.

4.2   Sample Identification

Each sample generated for the study was assigned a unique bar code identifier for laboratory processing. The bar code identifier was correlated to components of the experimental design as presented below:

- Laboratory code (Nos. 1, 2, and 3)
- Preparation method (EPA 3050A and WOHL)
- Type of composite (one-, two-, and four-wipe composite)
- Sample type (loaded wipe, QC sample type: laboratory control sample, method blank, wipe blank)
- Nominal SRM loading (0.5 g or 2 g per wipe)
- Wipe brand (A, B, or C)
- Lot code (1 and 2 for brands A and B only)

4.3   Sample Containers, Storage, and Shipment

As shown in Table 4-1, the sample containers used for storing the wipe samples were wide-mouth, high-density polyethylene Nalgene bottles. These containers were selected based on the volume capacity needed for the two- and four-wipe composite samples. A hard-walled container was used instead of a plastic bag for sample storage because this type of container can be rinsed to ensure a quantitative transfer of the dust sample to the digestion vessel.

Table 4-1. Wipe Sample Containers

Number of wipes per sample	Nalgene bottle size
1	125 mL
2	250 mL
4	250 mL

The wipe samples and resulting digests were stored at room temperature. The holding time from the date of loading the wipes with NIST SRM 2704 through instrumental analysis was 6 months, as specified in EPA SW-846 for metals analysis. The holding time was met for all analyses.

The wipe samples were packaged and shipped to the designated laboratories via next day delivery. Each laboratory participating in the study was provided with the documentation for each method and a work plan describing the project overview, scope of work, schedule, and required deliverables for the study.

Section 5
Sample Digestion and Analysis

The sample preparation and analysis efforts were conducted according to the methods listed in the Quality Assurance Project Plan (QAPjP).[7] The sample preparation methods are presented in the appendices, as outlined in Section 1.4. The digestion methods were evaluated using two different analytical methods each. The first analytical method (EPA SW-846 Method 6010A) determines metals in the digestates using ICP. The determination of lead by direct aspiration FAA (EPA SW-846 Methods 7000A [general] and 7420) was the second analytical method.

5.1 Instrumentation

The spectrometers of each laboratory used for the ICP and FAA analyses are listed below.

ICP Analysis Spectrometers

- Laboratory 1: Thermo Jarrell Ash ICAP 61E simultaneous vacuum path spectrometer with computer-controlled background and interelement corrections and an analytical wavelength of 220.353 nm.

- Laboratory 2: Perkin Elmer Optima 3000 XL ICP simultaneous nitrogen purged path spectrometer with computer-controlled background correction and an analytical wavelength of 220.353 nm.

- Laboratory 3: Thermo Jarrell Ash ICAP 61 simultaneous air path spectrometer with computer-controlled background and interelement corrections and an analytical wavelength of 220.353 nm.

FAA Analysis Spectrometers

- Laboratory 1: ARL GBC Model 902 FAA Spectrometer with deuterium background correction and an analytical wavelength of 283.3 nm.

- Laboratory 2: Thermo Jarrell Ash Video 12 E FAA Spectrometer with Smith–Hieftje background correction and an analytical wavelength of 217.0 nm.

- Laboratory 3: Perkin Elmer 5000 FAA Spectrometer with deuterium background correction and an analytical wavelength of 283.3 nm.

5.2 Calibration Standard Sources

The standard stocks used for the analyses at each laboratory were purchased from commercial suppliers. Working calibration standards were prepared by dilution of the concentrated calibration stocks. The standards used for instrument calibration (primary source) and the independent check standard (secondary source) were either prepared from stocks purchased from different suppliers, or were prepared from different lots of stocks purchased from the same supplier. The sources of the standard stocks used for the analyses at each laboratory are listed below. Spex Industries, Inc., is listed twice under instrument calibration standard sources because this supplier was used by two of the three laboratories.

Instrument Calibration Standard Sources (primary)
- Spex Industries, Inc.
- Spex Industries, Inc.
- Plasmachem Associates, Inc.

Independent Check Standard Sources (secondary)
- Inorganic Ventures, Inc.
- Spex Industries, Inc.
- Fisher Scientific Chemical Division

5.3 Laboratory Observations

The following observations were made by the laboratories conducting the sample digestion procedures:

- Although the hot plate time was low for the WOHL digestion methods, these methods were labor-intensive due to several steps in the procedure (e.g., cutting up the wipes, centrifuging, and filtering). The 3050A method had more hot plate time and seemed to provide a more complete digestion of the wipe material.

- The larger volume beakers and volumetric flasks, required for the modified SW-846 Method 3050A for the four-wipe composites, were difficult to handle in the laboratory and also were more difficult to clean.

- Some of the SRM material adhered to the scissors during the cutting step of the WOHL method. Attempts were made to recover and quantitatively transfer this material to the digestion vessel by wiping the scissors with a small piece of clean wipe material contained in the sample.

- An adequate rinse could not be obtained for the WOHL single-wipe method due to the small final dilution volume (50 mL) used for this method.

Additional observations made regarding the digestion time requirements and material costs for each digestion procedure are presented in Section 7.3 (Process Parameter Data).

Section 6
Quality Assurance/Quality Control

An independent evaluation of the sample preparation and analysis results was performed under the direction of the program QA Officer (QAO). In addition, a technical review of the data reported by the outside laboratories was conducted by the task leader, and the final QC data were statistically analyzed by the project statistician.
The results are presented in the following subsections.

6.1 Quality Assurance

Performance audits and complete systems audits were not conducted at all three laboratories because of their involvement with the NLLAP. The ELPAT analysis was considered to be equivalent to a performance audit, thus additional performance audits were deemed unnecessary. The benefit of conducting an on-site systems audit at each of the laboratories was not considered to outweigh the associated travel costs; however, a partial systems audit was conducted on work performed by the contractor laboratory in which all the records were examined.

A complete list of all sample identification and calculated recovery results is shown in Appendix H. These data pertain to all QC samples as well as all loaded wipe samples used during the project. The hard copy and computer records for the work performed by the contractor laboratory, including the tables for the final report, were audited. All sample identification codes and data were verified throughout the data handling process; no errors were found. The items verified during the audit are listed below.

- Accuracy and completeness of the data packet.

- Completeness, compliance, and accuracy of the laboratory notebook pages, including the method of analysis, the project number, date of analysis, the analyst's name, the standards used for calibration, the steps performed in the preparation of the standards, and the dilutions.

- Accuracy of the value used for NIST SRM 2704, based on the Certificate of Analysis information.

- Accuracy of the sample identification codes in the report tables, using the sample preparation inventory listing from the test design.

- Accuracy of the instrument responses presented as µg/mL for ICP and peak height absorbance for FAA in the data calculation tables, using the instrumental raw analysis data.

- Accuracy of the Initial Calibration Verification (ICV) control charts, using the raw data and data calculation tables.

- Accuracy of the computer entry of the weight data and the analytical results in the SAS computer output file.

The data reports provided by the outside laboratories also were examined. Several discrepancies were noted for Laboratory 2 between the results reported in the QC summary tables, the control chart data report, the detection limit interpretation, and the results. The FAA and ICP data columns were interchanged in several preliminary data tables, and a few data discrepancies also were noted. These problems were resolved during the technical review and data audit, and a revised report was submitted. The reported sample identification codes and data for the outside laboratories were verified after entry into the database.

The only deviation from the QAPjP was one laboratory's use of a smaller than specified capacity (rpm) centrifuge for the WOHL methods. The purpose of the centrifuge was to eliminate particles that would clog the nebulizer during analysis. The centrifuge used was set at approximately 4,000 rpm, which was adequate for this purpose, and the deviation did not impact data quality. The deviation was properly documented in the records.

6.2 Quality Control

This section presents the analyses of the gravimetric quality control results; the analytical quality control results for method blanks, wipe blanks, and laboratory control samples; the spiked wipe extreme recovery values; and the detection limits.

6.2.1 Gravimetric Data

NIST SRM 2704 was spiked at two nominal dust levels onto 972 single wipes: low (0.5 g) and high (2 g). Actual SRM weights were measured for each individual wipe. The actual weight of each single wipe and each composite wipe sample was then compared to the nominal levels, for a total of 396 comparisons. The discrepancies between actual and nominal dust levels were computed and compared against the specifications stated in the QAPjP: 0.5 ± 0.05 g for the 0.5-g loading and 2.0 ± 0.1 g for the 2-g loading. The discrepancies between actual and nominal dust levels are shown in the form of box (or box-and-whisker) plots in Figure 6-1. The data distributions are shown separately for each nominal level on a per single-wipe or composite basis (i.e., 0.5 g, 1 g, 2 g, 4 g, and 8 g). Since the loading of 2 g on a single-wipe sample is the same as the total loading of four times 0.5 g on a four-wipe composite sample, the results were further separated into the

two loading levels, low and high, in Figure 6-1. All box plots in this report were generated using a commercially available software package.[8]
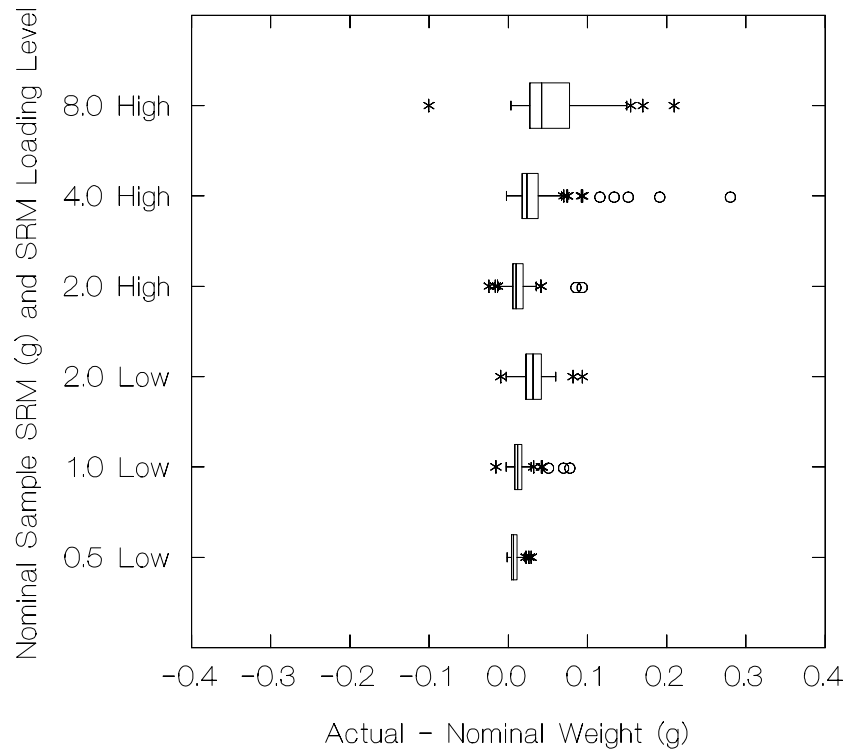
Figure 6-1. Distribution Plots of Gravimetric Data for Single Samples and Composites

Following is a description of how to read the box plots in Figure 6-1:

The left and right edges of a box are located at the sample 25th and 75th percentiles; thus 50% of the data fall within the box. The center vertical line is drawn at the sample median. The horizontal lines, called "whiskers," extend from the box to a distance of at most 1.5 interquartile ranges. (An interquartile range is the distance between the 25th and the 75th percentiles, the length of the box.) Thus, approximately 99% of the values will fall between the left- and right-side ends of the whiskers. Any value more extreme than this is marked with an asterisk "*" if it is within three interquartile ranges of the box, or with a small letter "o" if it is still more extreme.

As shown in Figure 6-1, the accuracy of weighing out the nominal levels decreases with increasing SRM loading at a given loading level because the errors in weighing are additive as the individual wipes are composited. The weight discrepancies exhibit a slight positive bias. However, the specifications stated above were only exceeded on a total of

8 out of 972 individual wipes. After compositing, the specifications were not met for only 1 out of 396 composited wipe samples. The amount of SRM applied to Sample No. 906423 (a two-wipe composite at the high SRM level) was 4.2803 g, when it should have been 4.0 ± 0.2 g. These results confirmed that the weighing procedure achieved the target accuracy called for in the QAPjP.

6.2.2 Quality Control Sample Data

Three types of quality control (QC) samples, for a total of 132 samples, were analyzed by both analytical methods (FAA and ICP) along with the composite wipe samples according to the QAPjP (see Section 4.1.3 of QAPjP). These included:

- 23 method blanks (MBs) to demonstrate absence of laboratory contamination

- 63 wipe blanks (WBs) to determine background levels, if any, in the wipe material

- 46 laboratory control samples (LCSs) to monitor method performance in the absence of any wipe matrix effect

Detailed analytical results for these 132 QC samples are included in Appendix H. It should be noted that Laboratory 1 analyzed more QC samples than the other two laboratories because three samples that had been diluted or spilled during the original preparation were discarded and new samples were prepared. This situation resulted in two additional batches, one each for the EPA Method 3050A and WOHL single-wipe batch types.

Method Blanks. One method blank was analyzed with each sample batch to monitor background levels in the reagents used for digestion and to check for potential laboratory contamination. The QC sample layout and analytical results are shown in Table 6-1. The last two columns provide the results (or range of results) for method blanks when normalized to μg/wipe. Normalized results for each batch type were calculated by multiplying the analytical result (μg/mL) by the digest volume (mL) used for that method/batch type, then dividing by the number of wipes of the corresponding batch. If the analytical result was below the instrument detection limit, then the instrument detection limit (μg/mL) was multiplied by the digest volume and then divided by the number of wipes, and the result was flagged as less than (i.e., <). The digest volume varied by method and composite type.

Table 6-1. Method Blank Sample Layout and Analytical Results

Laboratory code	Digestion method	Composite batch type	Number of samples	Number of detects found using		Normalized results (μg/wipe)[a]	
				FAA	ICP	FAA	ICP
1	EPA 3050A	1-wipe	2	0	0	< 9	< 6
		2-wipe	1	0	0	< 9	< 6
		4-wipe	2	0	0	< 12	< 7
	WOHL	1-wipe	2	0	0	< 3	< 2
		2-wipe	1	0	0	< 6	< 1
		4-wipe	1	0	0	< 3	< 0.4
2	EPA 3050A	1-wipe	1	1	1	5	4
		2-wipe	1	1	1	2	5
		4-wipe	2	1	2	1.3	5[b]
	WOHL	1-wipe	1	0	1	< 1	2
		2-wipe	1	1	1	1.5	1.5
		4-wipe	1	0	1	< 0.3	0.8
3	EPA 3050A	1-wipe	1	0	0	< 4	< 10
		2-wipe	1	0	0	< 4	< 10
		4-wipe	2	0	0	< 5	< 13
	WOHL	1-wipe	1	0	0	< 2	< 5
		2-wipe	1	0	0	< 2	< 5
		4-wipe	1	0	0	< 1	< 2.5

[a] The normalized results indicate detection limits or actual levels detected.
[b] This result is the average of 2 identical values of 20 μg for 4 wipes, normalized to 5 μg/wipe.

Of the three laboratories, only Laboratory 2 reported lead levels above the detection limit in the method blanks. Lead levels were detected by ICP in all seven method blanks (four digested by the EPA Method 3050A and three digested by the WOHL method). When FAA was used, lead levels were detected in four of the seven method blanks (three digested by the EPA 3050A and one digested by the WOHL method).

Laboratory 2 did not use the same approach in establishing detection limits as did Laboratories 1 and 3. This situation was noted during the evaluation of the method blank and wipe blank data; however, as discussed later in Section 6.3.2 (Detection Limits), this is not considered a problem due to the low levels detected.

Wipe Blanks. Wipe blanks were used to determine the background levels, if any, in the wipe material. One blank wipe of each brand was included in each preparation batch, according to the test design. The blank wipes were analyzed by both FAA and ICP.

Table 6-2 summarizes the layout and analytical results for each digestion procedure and analytical method. The last two columns provide the results (or range of results) for the wipe blanks, when normalized to μg/wipe, based on the number of wipes contained in the sample.

A number of wipes were found to have lead levels above the detection limit. A summary of the number of wipe blank detects for each brand of wipe is presented in Table 6-3.

To investigate whether wipe brands vary in their number of wipe blank detects and nondetects, Fisher's exact test was applied to the four 3x2 contingency tables defined by the combinations of digestion and preparation methods and to the two 3x2 contingency tables defined by each analytical method, as shown in Table 6-3. The statistical test results are shown in the last row of Table 6-3. Since none of the tests was significant at the two-sided 95% confidence level, there is no significant correlation between the number of wipe blank detects and wipe brand.

Of the 40 detects out of a total of 126 analyses shown in Table 6-3, 33 were attributed to Laboratory 2. The number of detects can be broken down by laboratory and analytical method as follows:

Laboratory 1 0/21 detect by FAA
 2/21 detects by ICP and WOHL

Laboratory 2 13/21 detects by FAA (5 by EPA 3050A, 8 by WOHL)
 20/21 detects by ICP (12 by EPA 3050A, 8 by WOHL)

Laboratory 3 4/21 detects by FAA (3 by EPA 3050A, 1 by WOHL)
 1/21 detect by ICP and WOHL

As noted earlier for method blanks, a disproportionate number of detects on the wipe blanks were attributed to Laboratory 2. The detects are discussed later in Section 6.3.2, Detection Limits.

Laboratory Control Samples (LCSs). LCSs were used to monitor the method performance in the absence of wipe matrix effects. These samples were prepared at the same NIST SRM 2704 loading levels as the wipe samples for each composite batch type (i.e., one low and one high level). For example, two-wipe composites required a low level loading of 0.5 g/wipe (total of 1.0 g) and a high level loading of 2.0 g/wipe (total of 4.0 g). The two LCSs for this composite type batch consisted of 1.0-g (low) and 4.0-g (high) aliquots of NIST SRM 2704, spiked into empty beakers (i.e., no wipe). The low and high

LCSs were digested with each batch of samples. The sample layout for these LCSs is shown in Table 6-4.

Table 6-2. Wipe Blank Sample Layout and Analytical Results

Laboratory code	Digestion method	Composite type	Number of samples	Number of detects found using		Normalized results (μg/wipe)[a]	
				FAA	ICP	FAA	ICP
1	EPA 3050A	1-wipe	3	0	0	< 9	< 6
		2-wipe	3	0	0	< 9	< 6
		4-wipe	6	0	0	< 12	< 7
	WOHL	1-wipe	3	0	0	< 3	< 2
		2-wipe	3	0	0	< 6	< 1
		4-wipe	3	0	2	< 3	< 0.4 to 1.2[b]
2	EPA 3050A	1-wipe	3	1	3	< 1 to 3	4 to 5
		2-wipe	3	1	3	< 0.5 to 6	4 to 5
		4-wipe	6	3	6	< 0.25 to 4	4 to 5
	WOHL	1-wipe	3	2	3	< 1 to 2	1 to 2
		2-wipe	3	3	2	1 to 2	0.5
		4-wipe	3	3	3	0.3 to 1	0.5 to 3
3	EPA 3050A	1-wipe	3	1	0	< 4 to 5	< 10
		2-wipe	3	0	0	< 4	< 10
		4-wipe	6	2	0	< 5 to 7.5	< 13
	WOHL	1-wipe	3	0	0	< 2	< 5
		2-wipe	3	0	0	< 2	< 5
		4-wipe	3	1	1	< 1 to 4.8	< 2.5 to 4.3

[a] The normalized results indicate detection limits or actual levels detected.
[b] The range of results obtained is reported for several method/composite types. The actual values, prior to normalization, are provided in Appendix H.

Table 6-3.   Number of Wipe Blank Detects by Wipe Brand

Wipe brand	FAA method			ICP method			All
	EPA 3050A	WOHL	Both	EPA 3050A	WOHL	Both	
A	1/12[a]	2/9	3/21	4/12	2/9	6/21	9/42
B	2/12	3/9	5/21	4/12	4/9	8/21	13/42
C	5/12	4/9	9/21	4/12	5/9	9/21	18/42
All brands	8/36	9/27	17/63	12/36	11/27	23/63	40-/126
Fisher's exact test: p-value	0.21	0.87	0.14	1.0	0.49	0.72	

[a] Number of detects/number of samples analyzed.

Table 6-4. Laboratory Control Sample Layout

Laboratory code	Digestion method	Nominal SRM loading (g)	Nominal Pb loading (µg)	Number of samples
1	EPA 3050A	0.5	80.5	2
		1.0	161	1
		2.0	322	4
		4.0	644	1
		8.0	1,288	2
	WOHL	0.5	80.5	2
		1.0	161	1
		2.0	322	3
		4.0	644	1
		8.0	1,288	1
2	EPA 3050A	0.5	80.5	1
		1.0	161	1
		2.0	322	3
		4.0	644	1
		8.0	1,288	2
	WOHL	0.5	80.5	1
		1.0	161	1
		2.0	322	2
		4.0	644	1
		8.0	1,288	1
3	EPA 3050A	0.5	80.5	1
		1.0	161	1
		2.0	322	3
		4.0	644	1
		8.0	1,288	2
	WOHL	0.5	80.5	1
		1.0	161	1
		2.0	322	2
		4.0	644	1
		8.0	1,288	1

The data quality objective for these samples was to obtain a recovery of 80% to 120%. Percent recovery statistics for the 46 LCSs are plotted in Figures 6-2 and 6-3 for FAA and ICP, respectively. Within each figure, the results are shown separately for the two digestion methods, using the laboratory code to represent the data.

All results obtained by FAA fall within the range of 80% to 120% recovery. When the samples were analyzed by ICP, all recovery results obtained from the samples digested by the EPA 3050A method met the recovery objectives. Of the results obtained by ICP on the samples digested by the WOHL method, three results (two from Laboratory 3 and one from Laboratory 2) fell below the lower limit of 80%. Two of these results were slightly above 79%, while the third result was approximately 73%. The plot at the bottom of Figure 6-3 shows a decreasing trend of percent recovery with increasing SRM for ICP results when

using the WOHL digestion method. This estimated trend is statistically significant
($p = 0.06$) with a decrease of 1.75% recovery for each gram of SRM (slope = 1.75), and
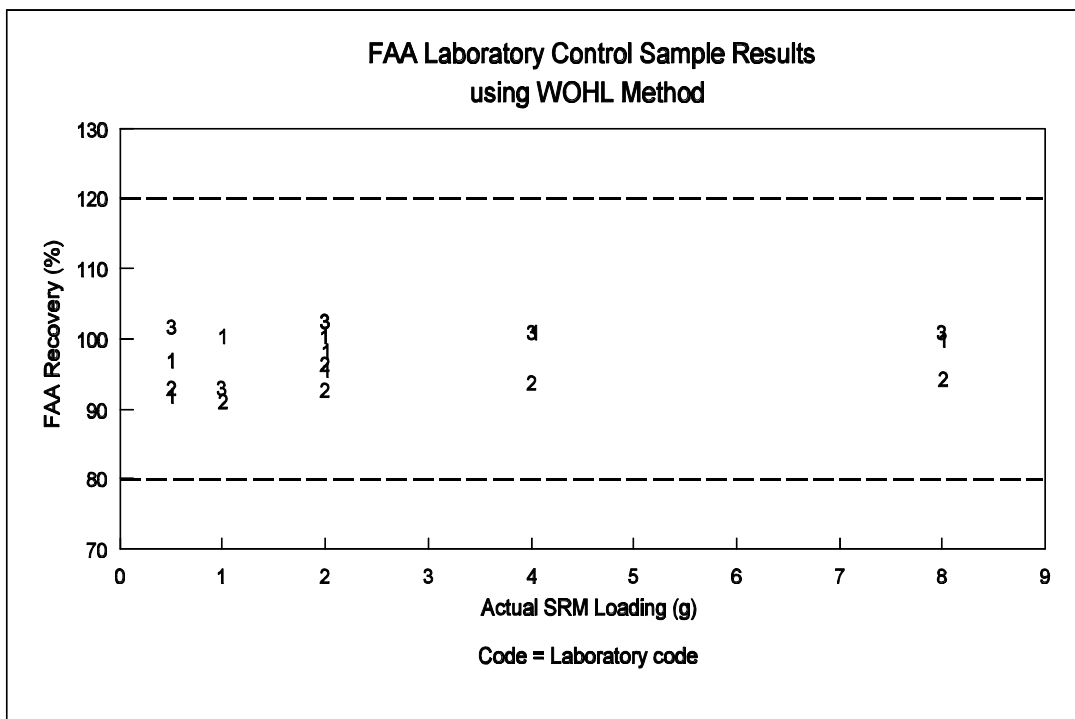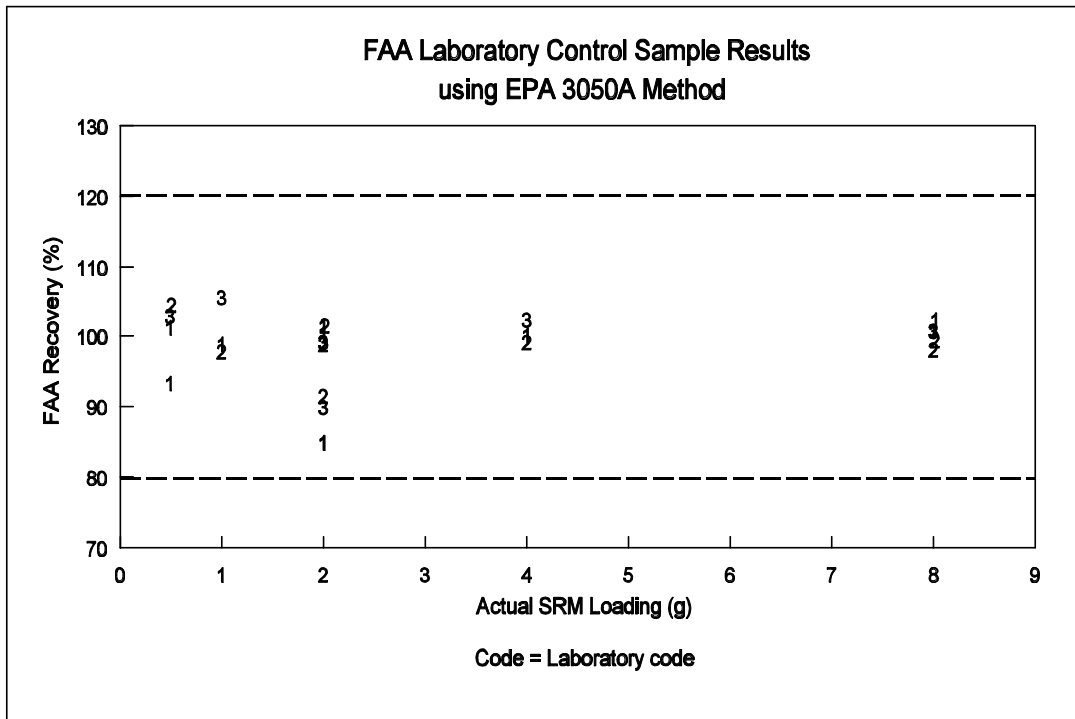
Figure 6-2. LCS Percent Recovery by FAA for EPA 3050A and WOHL Methods
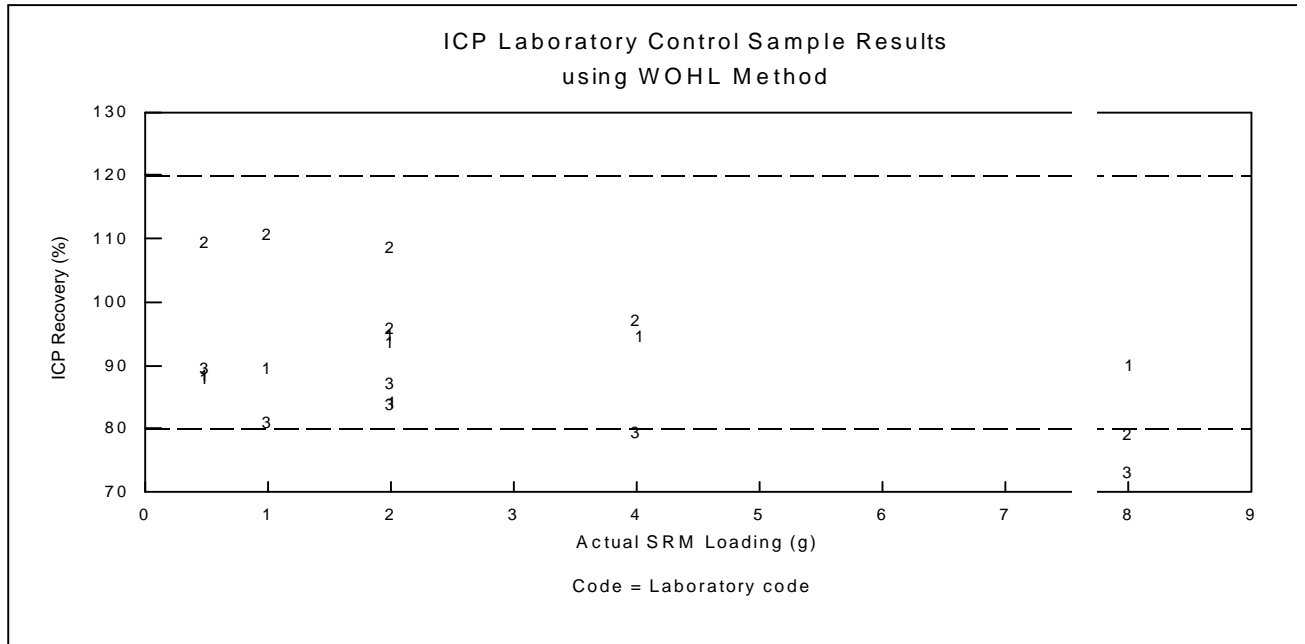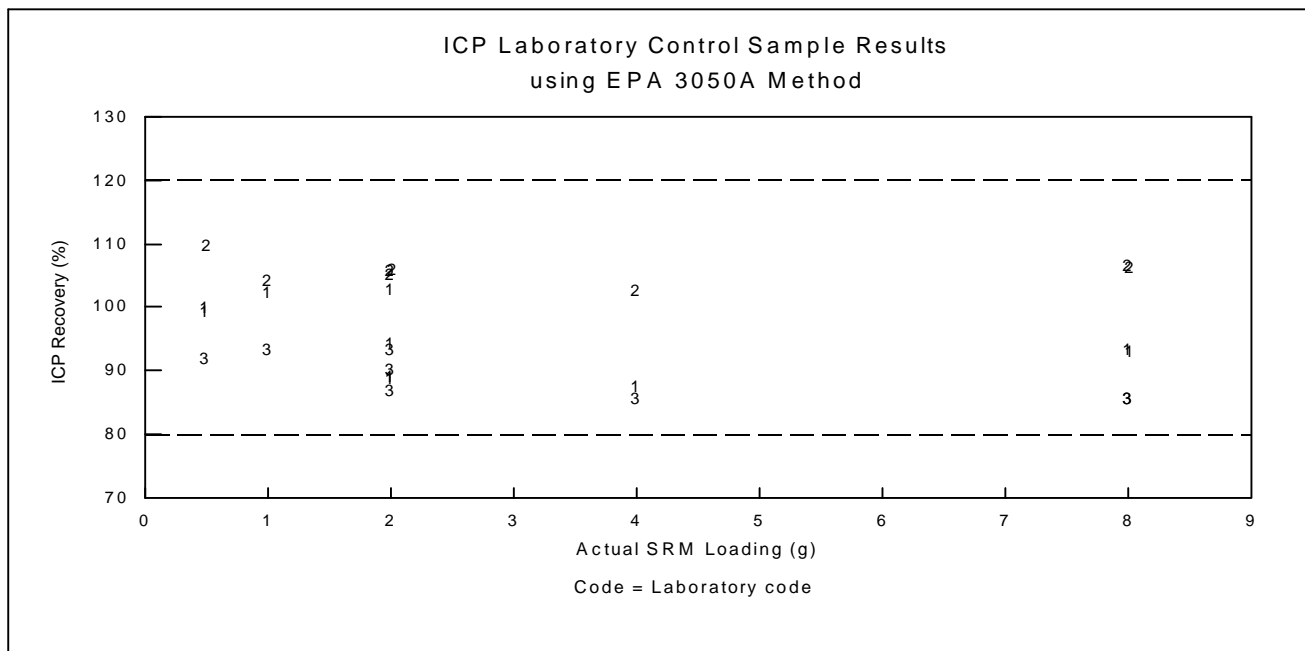
Figure 6-3. LCS Percent Recovery by ICP for EPA 3050A and WOHL Methods

may be a consideration in sampling applications where high dust loading levels are expected, such as for window sills and troughs in risk assessment.

LCS recovery statistics are further summarized in Table 6-5, separately for each laboratory, for each of the four digestion and analytical method combinations, across SRM levels. The precision, expressed as relative standard deviation, was less than 13% in all cases, ranging from approximately 2% to 7% for FAA and from 2% to 12% for ICP.

Table 6-5.   LCS Percent Recovery Statistics

Digestion method	Laboratory code	Number of samples	FAA recovery (%)		ICP recovery (%)	
			Mean	RSD[a]	Mean	RSD[a]
EPA 3050A	1	10	96.99	6.97	94.96	6.02
	2	8	98.99	3.73	105.67	1.95
	3	8	100.23	4.59	88.94	3.97
WOHL	1	8	98.15	3.11	90.24	4.13
	2	6	93.62	1.90	100.02	12.10
	3	6	100.31	3.56	82.20	7.18

[a] Relative standard deviation: RSD (%) = 100 × standard deviation/mean.

6.3   Other Data Quality Indicators

6.3.1 Extreme Percent Recovery Data

Percent recovery data were calculated for each spiked wipe composite sample separately for the FAA and ICP methods as:

$$\text{Recovery } (\%) \; = \; \frac{\text{Amount Found } (\mu g)}{\text{Amount Spiked } (\mu g)} \times 100$$

Figures 6-4 and 6-5 show the recovery data distribution in the form of box plots, for FAA and ICP, respectively. The data (box plots) within each figure are organized from the bottom up by laboratory, digestion method, and number of wipes per composite. For example, 1-3050A2 denotes Laboratory 1, digestion Method 3050A, and a two-wipe composite. Any value falling between 1.5 and 3 times the interquartile range from the outside of the box is marked with an asterisk "*", or with a small letter "o" if it is still more extreme. The 11 FAA recovery values and the 6 ICP recovery values marked as either "*" or "o" were further investigated and are summarized in Table 6-6. The gravimetric and analytical data for each sample were verified, and no data reporting errors or anomalies were noted. As a result, the data were not discarded from the statistical analyses.

6-14
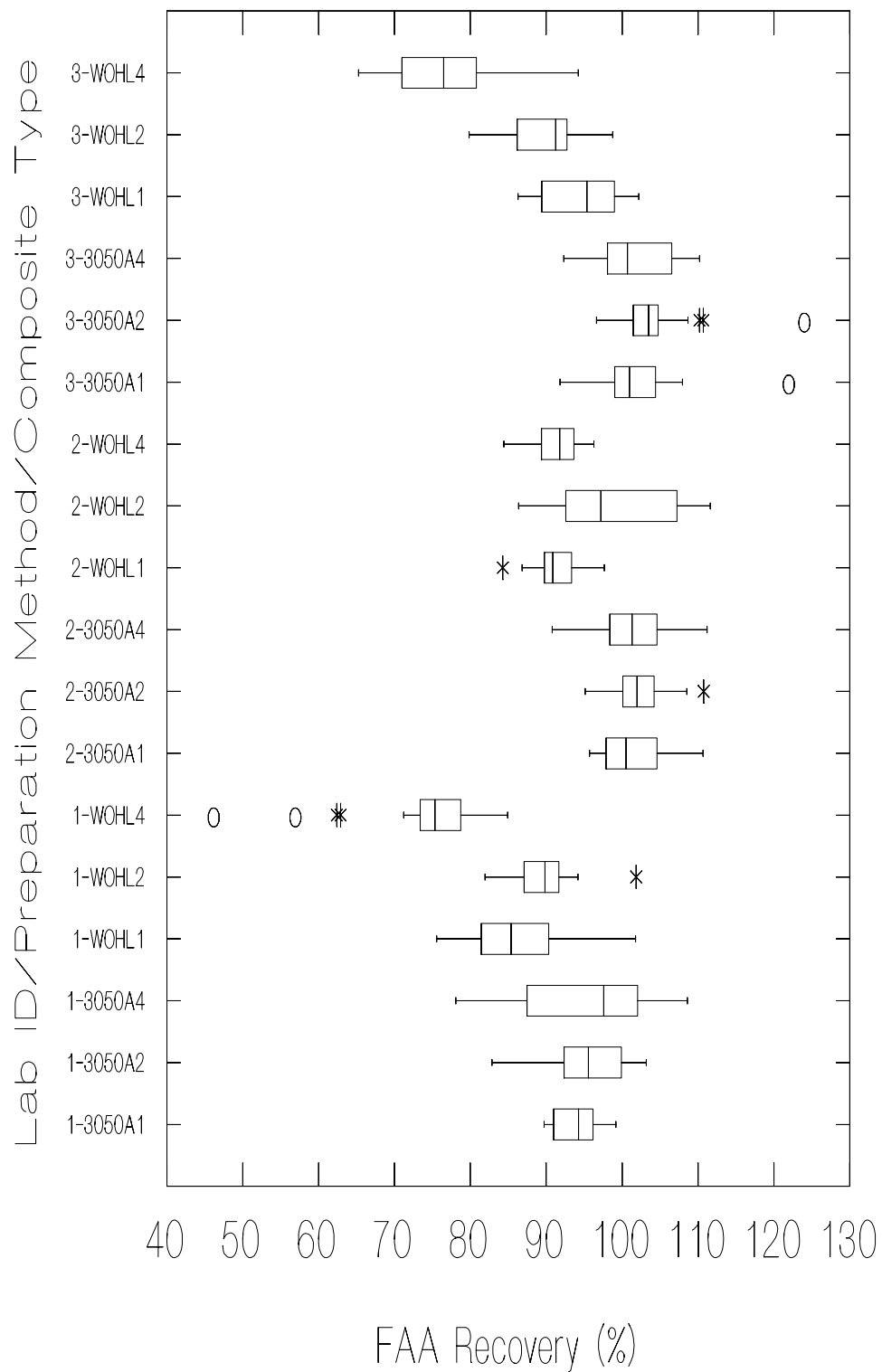
Figure 6-4. Distribution Plots of FAA Percent Recovery Data by Laboratory, Digestion Method, and Type of Composite
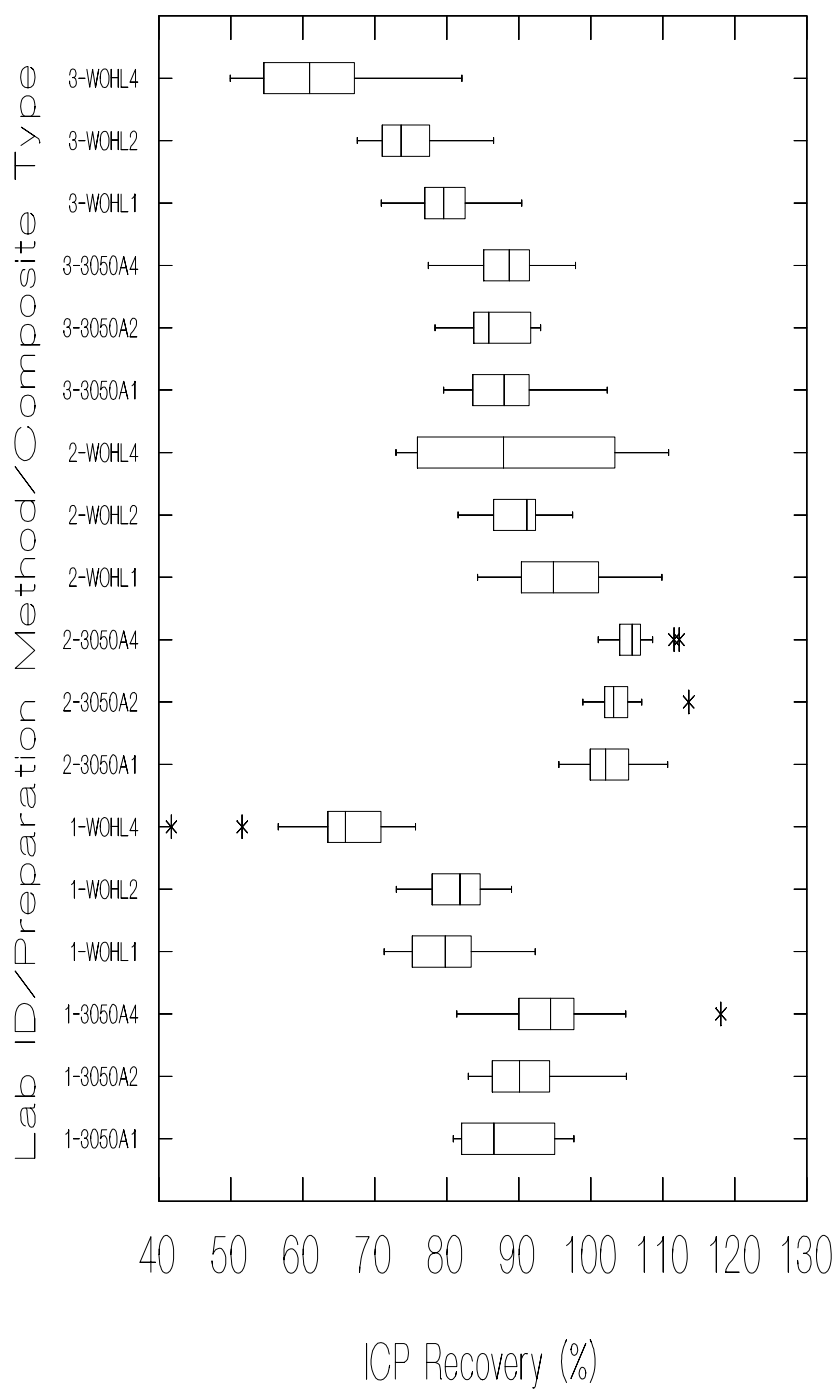
Figure 6-5. Distribution Plots of ICP Percent Recovery Data by Laboratory, Digestion Method, and Type of Composite

Table 6-6.   Wipe Samples with Extreme Recovery Results

Sample bar code	Laboratory code	Digestion method	Composite type	Wipe brand	FAA recovery (%)	ICP recovery (%)
Samples with Recoveries Flagged as "*"						
906223	1	WOHL	4-wipe	B	62.41	
906296		WOHL	4-wipe	B	62.89	
906179		WOHL	2-wipe	B	101.86	
906350	2	3050A	2-wipe	C	110.73	
906279		WOHL	1-wipe	B	84.26	
906101	3	3050A	2-wipe	A	110.68	
906354		3050A	2-wipe	C	110.20	
906389	1	3050A	4-wipe	C		118.03
906224		WOHL	4-wipe	B		41.70
906295		WOHL	4-wipe	B		51.56
906351	2	3050A	2-wipe	C		113.57
906372		3050A	4-wipe	C		112.26
906077		3050A	4-wipe	A		111.52
Samples with Recoveries Flagged as "o"						
906224	1	WOHL	4-wipe	B	46.18	
906295		WOHL	4-wipe	B	56.94	
906333	3	3050A	1-wipe	C	121.96	
906357		3050A	2-wipe	C	124.01	

In addition, 9 of the 17 samples yielded recoveries within the 80% to 120% data objective. Figures 6-4 and 6-5 also show that a considerable portion of recoveries are below the 80% lower limit for Laboratories 1 and 3 when using the WOHL method and either analytical technique (FAA or ICP) for four-wipe composites.

The difference between FAA and ICP percent recoveries was calculated for each individual sample to compare the two analytical methods. These data are shown in the box plots in Figure 6-6. These data are organized in the same fashion as Figures 6-4 and 6-5 and provide a means of comparing the two analytical methods on a per sample basis.

6.3.2 Detection Limits

As mentioned earlier, Laboratory 2 found more method blank and wipe blank detects than the other two laboratories. Laboratory 2 did not use the same approach in establishing detection limits as did Laboratories 1 and 3. The study only allowed for incorporating variability in digestion and analysis procedures among laboratories into the final data evaluation. A comparison of overall performance among laboratories per se was not the intent of this study.

Several reasons why detection limits can vary among laboratories are given:

- The analytical methods used do not elaborate on how to determine a detection limit; therefore, detection limits can be estimated using different procedures

- The use of different instrumentation may provide different detection limits

- Even the same instrumentation will provide variable detection limits over time; for example, many standard procedures specify that instrument detection limits should be determined every three calendar months

- The use of different analytical parameters, such as the analytical wavelength, can provide different detection limits from the same instrument (e.g., for FAA, Laboratory 2 used a more sensitive wavelength than Laboratories 1 and 3)

Of the detection limits reported by the three laboratories, the highest detection limits reported for FAA and ICP were 12 μg/wipe and 13 μg/wipe, respectively. In each case, the highest detection limits reported were those for the EPA 3050A digestion method and the four-wipe composite sample type. This digestion method/composite type combination yielded the highest method detection limits (MDL) because the MDL is dependent on the final digest volume, as discussed in Section 6.2.2.

The EPA has the same interim dust clearance standards as the HUD *Guidelines*, which range from 100 to 800 $\mu g/ft^2$, depending on the surface sampled (Interim 403 Guidance, Memorandum of July 14, 1994, from Lynn Goldman to Regional Division Directors).
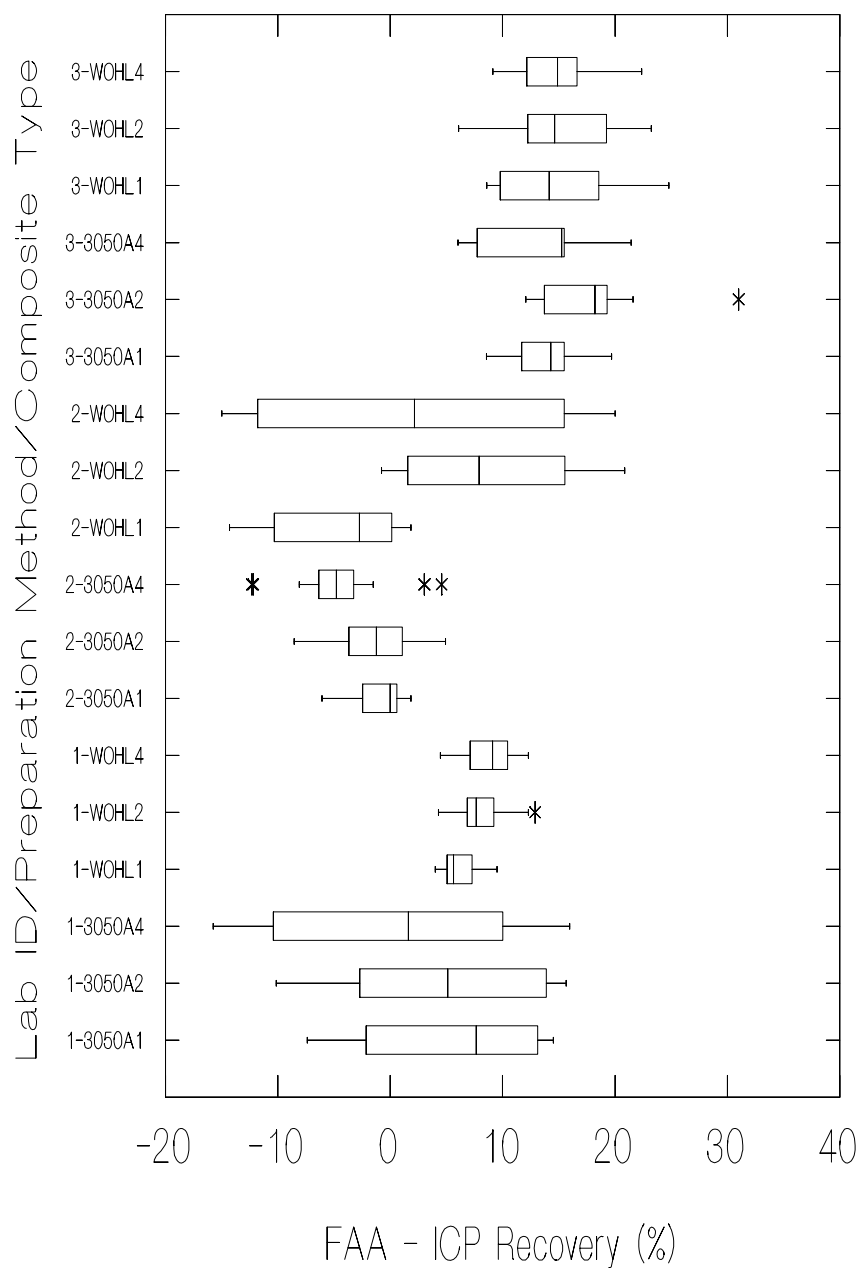
**Figure 6-6. Distribution Plots of FAA—ICP Percent Recovery Data by Laboratory, Digestion Method, and Type of Composite**

Assuming a 1-ft^2 area is wiped during typical sample collection, then the lowest clearance standard can be calculated as 100 µg/wipe. From the results reported by the three laboratories, both digestion methods tested provided sufficient sensitivity for the purpose of clearance applications.

Appendix 13.1 of the HUD *Guidelines* states that "Blank media should contain no more than 25 µg/wipe (the detection limit using Flame Atomic Absorption)." Appendix 13.1 also specifies that wipe media should contain less than 5 µg/wipe. As discussed above, some of the detection limits reported by the laboratories in this study were greater than 5 µg/wipe. For the methods with adequate sensitivity at the 5 µg/wipe level, the highest levels of lead detected in the wipe blanks above 5 µg/wipe were the following:

- Laboratory 2, Method 3050A, FAA analysis, two-wipe composite: 6 µg/wipe
- Laboratory 3, Method 3050A, FAA analysis, four-wipe composite: 7.5 µg/wipe

These values are only slightly above the criterion of 5 µg/wipe. Overall, approximately 68% (86 out of 126) of the wipe blanks were found to have levels below the detection limit. However, Laboratory 2 only found 21% (9 out of 42) of the wipe blanks to have levels below the detection limit. In contrast, Laboratories 1 and 3 combined found 92% (77 out of 84) of the wipe blanks to have levels below the detection limit. It was noted that the wipe blank levels reported as detects by Laboratory 2 were in most cases at or below the detection limits reported by Laboratories 1 and 3. More sensitive analytical techniques, such as graphite furnace atomic absorption spectrometry, could be used for screening blank media. This technique would provide more quantitative data at the 5 µg/wipe level.

The wipe blank results reported by all three laboratories also were evaluated using a worst-case scenario (i.e., using the maximum detection limit reported for each digestion method, composite type, and analytical method combination). If the maximum detection limits are used for the comparison, then the highest values detected are the wipe blanks for the WOHL four-wipe composites listed below:

- Laboratory 2: ICP at 3.0 µg/wipe
- Laboratory 3: ICP at 4.3 µg/wipe
- Laboratory 3: FAA at 4.8 µg/wipe

These values were all less than 5 µg/wipe.

Based on this evaluation, blank correction was not used because all three brands of wipe media used in this study were found to have a minimal number of detects and, in those instances where detects were reported, the lead levels were negligible. For future work, it is recommended that detection limits be established by the same procedure for all laboratories so that the results are more comparable.

Section 7
Statistical Analysis and Results

In accordance with the study design described in Section 3, two types of data were obtained from this study: analytical results, in units of total amount of lead per sample (µg), and information on all relevant process parameters from three selected laboratories. This section describes in detail the data processing, the treatment of each of the two types of data, and an approach to the final assessment of the two preparation methods for composite wipes.

7.1   Data Tracking, Data Entry, and Data Processing

All MRI project records, including sample control, sample weighing data, instrument outputs, wipe lead-loading data, instrumental parameters, and final summary data tables, were assembled and stored in central project files at MRI. The data reports submitted from the two commercial laboratories also were retained in the files. All laboratory results were traceable to the unique sample identifier (bar code). Unusual experimental observations were documented in laboratory notebooks and in the reports received from the two outside laboratories. All project records were technically reviewed and audited as described in Section 6. Checklists were used to assure that the analytical data packets were complete.

The analytical results for the single- and composite-wipe sample analyses (ICP and FAA) conducted at MRI were checked, by separate laboratory personnel, using a double key entry procedure. This provided a 100% check of the raw data entered into spreadsheets. MRI staff also entered all analytical results reported from the other two laboratories into spreadsheets using the double key entry procedure. Following the technical review and the QA audit, all data results were released for statistical analysis.

The individual data spreadsheets from all the laboratories were then uploaded to an IBM mainframe for analysis using SAS,[9] a commercial statistical software package, available to MRI at the University of Missouri-Columbia. Basic error-checking procedures were performed to identify possible data errors. Appendix H provides a complete list of all gravimetric and analytical data generated and statistically analyzed during this project.

7.2   Statistical Analysis of the Analytical Data

At each of three laboratories and for each of two digestion methods, lead measurements, in total µg Pb/sample, were obtained from both the ICP and FAA analyses of each sample as defined in Section 3. Percent recovery, calculated as the ratio of measured over spiked amount of lead per sample, was the dependent variable in all

statistical analyses and provided a measure of accuracy. The lead data obtained by FAA or ICP were analyzed separately, using identical approaches.

Average percent recoveries were calculated for each laboratory, separately for each composite type, SRM loading, digestion method, and analysis method. Upper and lower 95% confidence limits for <u>individual</u> recovery data were computed as well. These statistics (average and confidence limits) are based on 12 measurements except for single-wipe samples prepared by the EPA 3050A method, in which case only 6 measurements were available. These basic statistics are shown in Figures 7-1 and 7-2 for FAA and ICP analysis, respectively.

The aim of this experimental study was to assess the effect of the chosen factors on method recovery. The factors investigated, either fixed or random from a statistical point of view, were:

Fixed effect factors:
1.   Number of wipes per composite (one, two, or four wipes)
2.   SRM loading level (low at 0.5 g or high at 2 g per wipe)
3.   Digestion method (EPA 3050A or WOHL)

Random effect factors:
4.   Laboratory (Laboratory 1, 2, or 3)
5.   Wipe brand (three commercially available brands, A, B, or C)
6.   Wipe brand lot (two lots; does not apply to Brand C because it was purchased from a single location)

The first three factors were selected at the onset of the study which was limited to investigating these main factors and their levels. Statistically speaking, these factors are called fixed factors. On the other hand, the three laboratories can be considered as having been selected from among all laboratories performing comparable lead analyses. The objective of the study was to assess the performance of the two composite methods across laboratories in general, not for these three laboratories in particular. Wipe brand and lot within brands (Brands A and B only) were two additional factors in this study. The three wipe brands represent a sample of commercially available wipe brands adequate for dust sampling. The purpose for including more than one brand of wipes and, to the extent possible, wipes obtained from different manufacturing lots, was to take into account potential variability among lots and/or brands of wipes. Again, the intended purpose of the study was not to assess method performance for individual wipe brands. Statistically speaking, the factors, laboratory, wipe brand, and wipe brand lot are called random factors.

Figure 7-1. Basic FAA Recovery Statistics for Each Laboratory, Preparation Method,
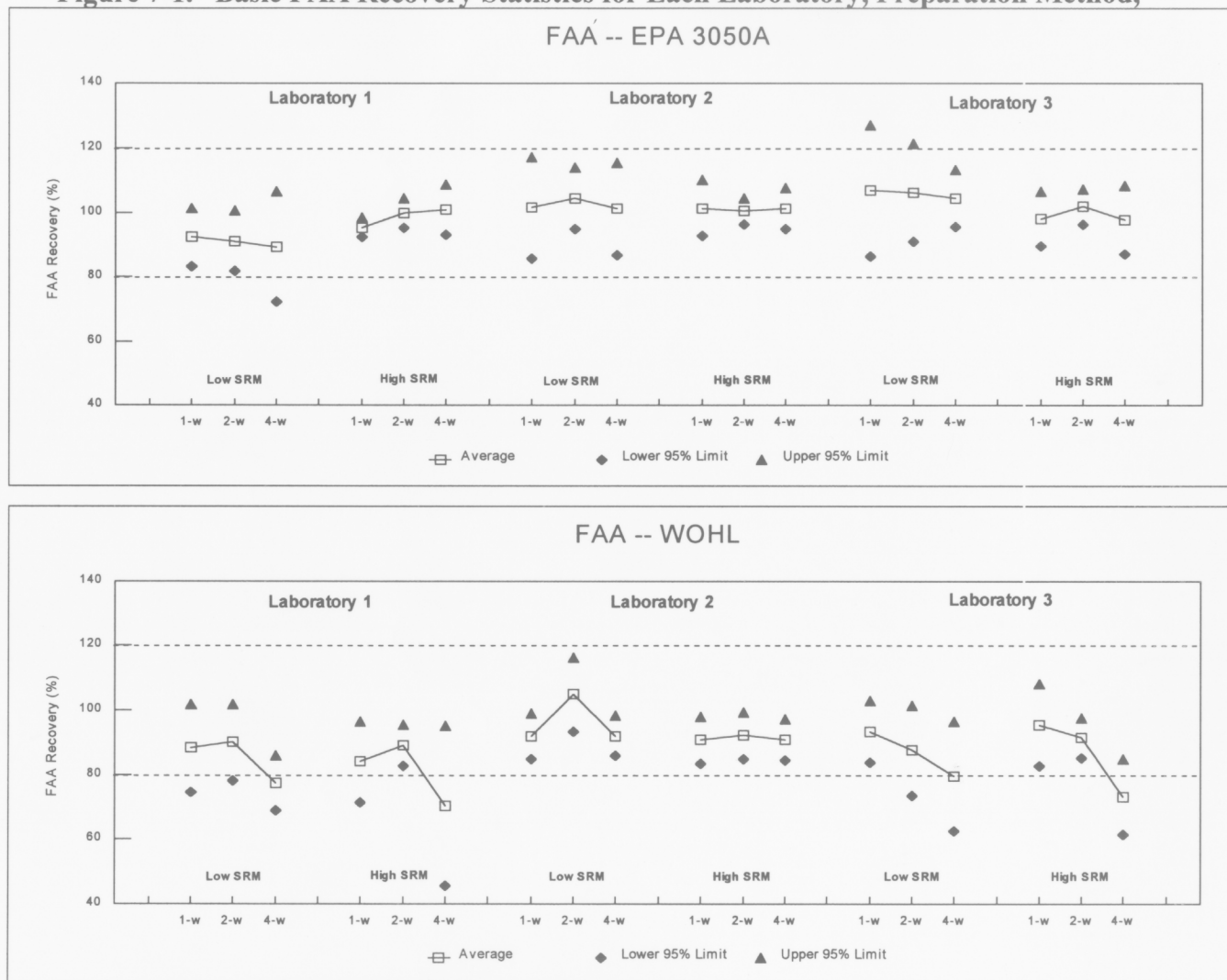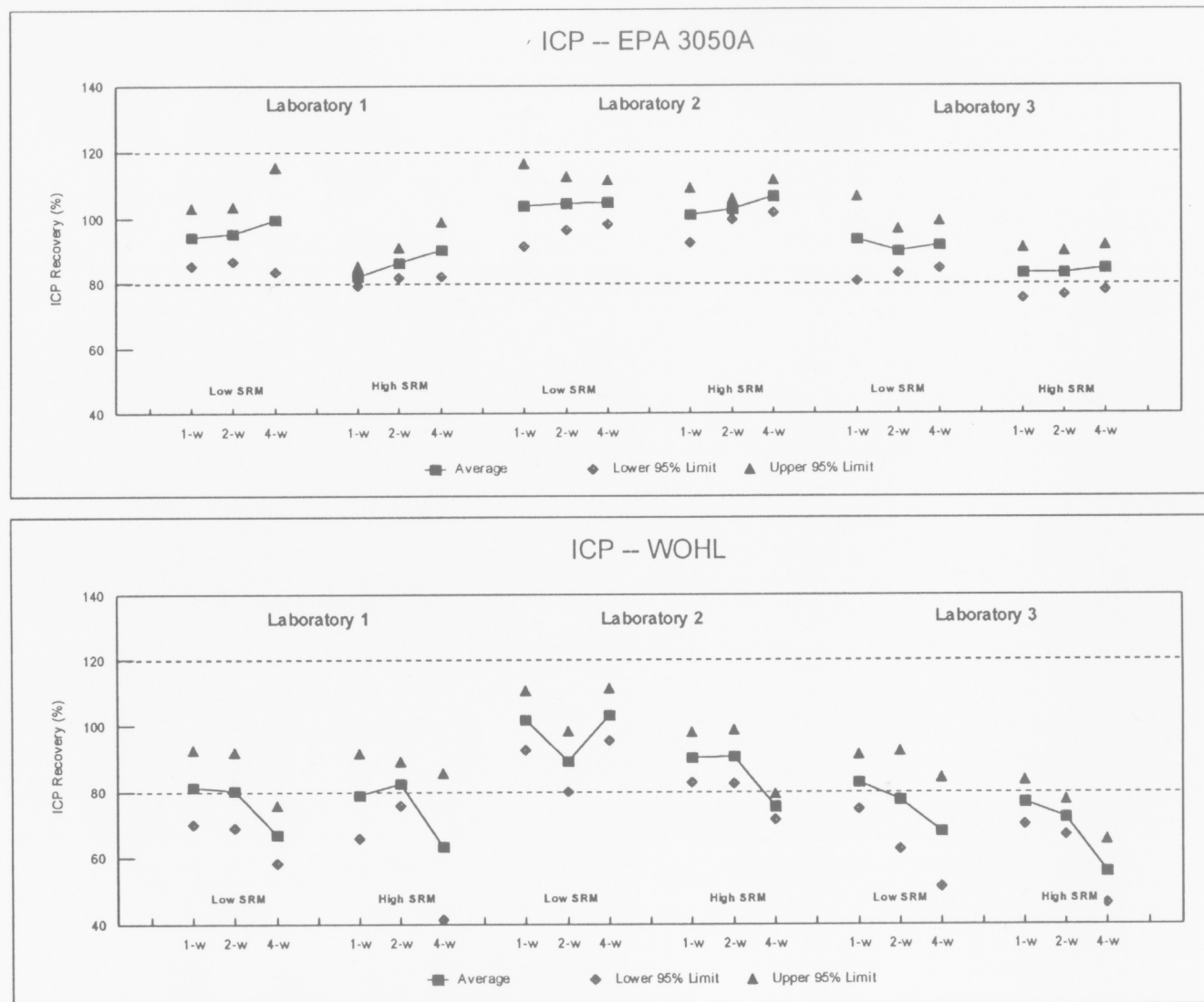
Figure 7-2. Basic ICP Recovery Statistics for Each Laboratory, Preparation Method, Composite Type, and SRM Loading

A model including both fixed and random effects is called a mixed model in which all estimates of the fixed effects will be adjusted for the random effects in the model and broad inference will be made across laboratories. Since only three laboratories were included in the design, treating them as a random factor will result in less sensitive tests of significance as compared to a fixed factor model. This is reflected in the relatively wide confidence intervals computed for means, based on three laboratories only. All mixed model ANOVAs were performed using the PROC MIXED procedure of SAS.[10,11]

An assessment of method accuracy and method precision for both digestion methods is presented next.

7.2.1 Method Accuracy Analysis

A statistical model was developed to estimate the effect of the above main factors (fixed and random) and their interactions on percent recovery (accuracy) using FAA and ICP analyses. As stated earlier, three brands of wipes and two lots within brand (Brands A and B only) were considered in this study in an attempt to broaden the method performance evaluation of the two preparation methods. Various wipe brands are commercially available for the purpose of lead dust sampling. Rather than evaluating the two digestion methods and the two analysis methods based on a single brand of wipe, it was decided that one or two lots of three wipe brands each would provide an appropriate basis for evaluating method performance in the context of this study.

This approach was in no means an attempt to rank commercially available wipe brands with respect to method recovery of lead dust. However, the potential variability between lots within a brand and that among brands were examined to assess the need to incorporate these random factors into subsequent statistical models. The alternative approach chosen in the present case was not to include the two factors (brand and lot), whether random or fixed, into the model, but to simply treat all wipes as replicate samples. This decision is supported by the outcome of the following analyses.

Investigation of Potential Wipe Manufacturers' Lot Differences: Potential differences between lots of Brands A and B were investigated before simply treating brands and lots as design replicates. Since the design was balanced with respect to the fixed factors (number of wipes per composite and SRM loading) across brands, a nested random effects two-way analysis of variance (ANOVA) was performed, where wipe brand and lot nested within brands were the main effects. This analysis was done separately for each digestion method using percent recovery results from FAA and ICP. None of the factors was found to have a statistically significant effect on percent recovery in either case (smallest p-value = 0.38). It was concluded that the variability between lots of Brand A and Brand B was negligible and that the manufacturers' lot information can be ignored in subsequent analyses.

Investigation of Potential Wipe Brand Differences: In light of the results from the above analysis, the wipes of Brands A and B were each pooled across lots. Under this configuration and using the data from all three brands A, B, and C, the design was again balanced with respect to the fixed factors (number of wipes per composite and SRM loading) across brands. A one-way ANOVA was then performed separately for FAA and ICP recoveries to assess whether percent recovery varied significantly among the three wipe brands.

The ANOVA results are shown in Table 7-1. The p-values in the fourth column indicate the significance level of the overall wipe brand effect. The wipe brand effect is significant in all but one case—ICP recovery when using the EPA 3050A digestion method (p=0.2191). Average recoveries were then computed for each wipe brand, and these are shown in columns 5 through 7 in Table 7-1. The F-test for brand effect was followed by a multiple comparison test to identify which brands vary among themselves. In all cases, there was no statistically significant difference between Brands A and B. However, except for ICP and EPA 3050A, percent recovery for Brand C differs significantly from that of either Brand A or B. The last two columns of Table 7-1 summarize the results of comparing Brand C recovery with the average recovery of Brands A and B. Average differences and their standard error are shown.

The outcome of this analysis would suggest that the random effect for brand should be included in the main statistical models analyzed subsequently. However, it was decided not to included brand as a factor in light of the original intention to treat wipes of different brands simply as replicate samples. (It should be noted that the statistical models discussed next were also investigated while including the random effect for brand. This approach had no impact on the mean recoveries but resulted in slightly wider confidence intervals for the estimated means.) In summary, neither manufacturing lot nor type of wipe brand was considered as a design factor in this study.

Statistical Modeling Approach: FAA and ICP recovery results from a total of 396 wipe composites were available for the final evaluation. In light of the above analysis results for wipe brands and wipe lots, the four factors remaining to be analyzed were:

Fixed effect factors:
1. Number of wipes per composite (one, two, or four wipes)
2. SRM loading level (low at 0.5 g or high at 2 g per wipe)
3. Digestion method (EPA 3050A or WOHL)

Random effect factor:
4. Laboratory (Laboratory 1, 2, or 3)

Table 7-1.   Average Percent Recovery by Wipe Brand

Analytical method	Digestion method	Total number of wipes	Overall F-test for brand effect: p-value	Average recovery (%)			Recovery difference (%) C-(A+B)/2[c]	
				Brand A[a]	Brand B[a]	Brand C[b]	Mean	Standard error of mean
FAA	EPA 3050A	180	0.0183	99.33	98.39	101.66	2.80	1.02
	WOHL	216	0.0003	86.06	86.10	91.62	5.54	1.34
ICP	EPA 3050A	180	0.2191	93.94	93.62	96.19	2.41[d]	1.38
	WOHL	216	0.0039	77.23	78.31	83.88	6.11	1.83

[a] Percent recoveries from Brands A and B are not statistically significant from each other.
[b] Percent recovery from Brand C is significantly different from that of Brand A or B, except for ICP and EPA Method 3050A.
[c] Estimated percent recovery difference between Brand C and the average of Brands A and B.
[d] Difference is not significantly different from zero at the 5% significance level.

The resulting design is a full factorial design for both FAA and ICP recovery statistics. In a preliminary analysis, the effect on percent recovery (FAA and ICP) of all main factors and all their interactions was investigated in a four-way mixed model ANOVA. This linear model, in which all fixed effect estimates are adjusted for random effects, relates percent recovery to the following factors and their interactions:

— Digestion Method
— SRM Level
— Number of Wipes

— Digestion Method $\times$ SRM Level
— Digestion Method $\times$ Number of Wipes
— SRM Level $\times$ Number of Wipes

— Digestion Method $\times$ SRM Level $\times$ Number of Wipes

— Laboratory

— Laboratory $\times$ Digestion Method
— Laboratory $\times$ SRM Level
— Laboratory $\times$ Number of Wipes

— Laboratory $\times$ Digestion Method $\times$ SRM Level
— Laboratory $\times$ Digestion Method $\times$ Number of Wipes
— Laboratory $\times$ SRM Level $\times$ Number of Wipes

— Laboratory $\times$ Digestion Method $\times$ SRM Level $\times$ Number of Wipes

All terms involving Laboratory as a factor were treated as random in the model. All other terms were considered fixed.

The results from this analysis, performed separately for FAA and ICP recovery, are shown in Tables I-1 and I-2 in Appendix I, respectively. Of all the fixed effect terms considered in the model, only the following three were statistically significant at the 95% confidence level:

— Digestion method for FAA recovery (p=0.0431)
— Digestion method for ICP recovery (p=0.0191)
— The interaction between digestion method and number of wipes per composite for ICP recovery (p=0.0206)

These analyses clearly show the recovery differences between the two digestion methods, with the EPA Method 3050A yielding higher recoveries, on the average, than the

WOHL method. In addition, FAA yields higher recoveries on the average than ICP for either digestion method. Average recoveries and their standard error are summarized in Table 7-2.

Table 7-2. Overall Recovery Statistics by Analytical and Digestion Method

Analytical method	Digestion method	Mean recovery (%)	Standard error of mean[a] (%)
FAA	EPA 3050A	99.7	2.84
	WOHL	87.9	2.84
	Difference	11.8	2.53
ICP	EPA 3050A	94.3	5.56
	WOHL	79.8	5.56
	Difference	14.5	2.03

[a] Note that these standard errors, computed in a mixed model setting, have only 2 degrees of freedom (the number of laboratories, 3 minus 1).

At a reduced confidence level of 90%, the following additional fixed effect terms would be statistically significant:

— Number of wipes per composite for FAA recovery (p=0.086)
— The interaction between digestion method and number of wipes per composite for FAA recovery (p=0.0849)
— SRM loading level for ICP recovery (p=0.069)

Following these preliminary significant analysis results, it was decided to analyze recovery data separately for each digestion method and each analytical method. This approach provided a means to report method performance separately for each of the methods tested.

Investigation of Fixed Effect Factors—Composite Type and SRM Loading Level: The effect on percent recovery (FAA and ICP) of the remaining main factors and all their interactions was investigated separately for each digestion method in a three-way mixed model ANOVA. This linear model, in which all fixed effect estimates are adjusted for random effects, relates percent recovery to the following factors and their interactions:

— SRM Level
— Number of Wipes

— SRM Level × Number of Wipes

— Laboratory

— Laboratory × SRM Level
— Laboratory × Number of Wipes

— Laboratory × SRM Level × Number of Wipes

As before, all terms involving Laboratory as a factor were treated as random in the model. All other terms were considered fixed. The model parameters were estimated for the four combinations defined by the two digestion methods and the two analytical methods. The results from this analysis performed for FAA and EPA 3050A and WOHL are shown in Tables I-3 and I-4 in Appendix I. The ICP results from analysis using EPA 3050A and WOHL are shown in Tables I-5 and I-6 in Appendix I.

The highlights in Tables I-3 through I-6 pertaining to the effect of the fixed factors (SRM loading level and number of wipes per composite) and their interaction on percent recovery can be summarized as follows:

— The interaction between SRM level and type of composite is never significant

— SRM loading level by itself is never a significant factor

— Composite type (number of wipes per composite) is only significant for the combination ICP and WOHL method (p=0.048). This factor is also significant at the 90% confidence level for the FAA and WOHL combination (p=0.0789). More specifically:

  • The difference of 11.9% between two-wipe and four-wipe composite recoveries (averaged across SRM levels) for FAA and WOHL is statistically significant (p=0.0409). The difference of 10.1% between one-wipe and four-wipe composite recoveries for FAA and WOHL is slightly less statistically significant (p=0.0661) (see contrast estimates in Table I-4 under heading "Estimate Statement Results").

  • The difference of 13.1% between one-wipe and four-wipe composite recoveries (averaged across SRM levels) for ICP and WOHL is statistically significant (p=0.0222). The difference of 9.9% between two-wipe and four-wipe composite recoveries for ICP and WOHL is statistically significant (p=0.0531). (See contrast estimates in Table I-6 under heading "Estimate Statement Results.")

Average (model least squares means) recoveries and their standard errors are shown in Table 7-3 for each analytical method, digestion method, SRM loading level, and composite type. (These and other statistics can be found in Tables I-3 through I-6 under the heading "Least Squares Means.") These recovery results, along with their lower and upper 95% confidence limits, are plotted in Figure 7-3. It should be noted that the confidence

Table 7-3. Recovery Statistics by Analytical Method, Digestion Method, SRM Loading Level, and Composite Type

Analytical method	Digestion method	SRM level	Composite type	Mean recovery (%)	Standard error of mean (%)
FAA	EPA 3050A	Low	1-wipe	100.2	3.40
			2-wipe	100.6	3.31
			4-wipe	98.4	3.31
			All	99.7	3.24
		High	1-wipe	98.3	3.40
			2-wipe	100.8	3.31
			4-wipe	100.0	3.31
			All	99.7	3.24
	WOHL	Low	1-wipe	91.2	4.19
			2-wipe	94.2	4.19
			4-wipe	83.1	4.19
			All	89.5	3.24
		High	1-wipe	90.1	4.19
			2-wipe	90.8	4.19
			4-wipe	78.1	4.19
			All	86.4	3.24
ICP	EPA 3050A	Low	1-wipe	97.3	5.18
			2-wipe	96.5	5.14
			4-wipe	98.7	5.14
			All	97.5	5.08
		High	1-wipe	88.7	5.19
			2-wipe	90.7	5.14
			4-wipe	94.0	5.14
			All	91.1	5.08
	WOHL	Low	1-wipe	88.6	6.81
			2-wipe	82.3	6.81
			4-wipe	79.4	6.81
			All	83.4	6.27
		High	1-wipe	81.9	6.81
			2-wipe	81.7	6.81
			4-wipe	64.9	6.81
			All	76.2	6.27
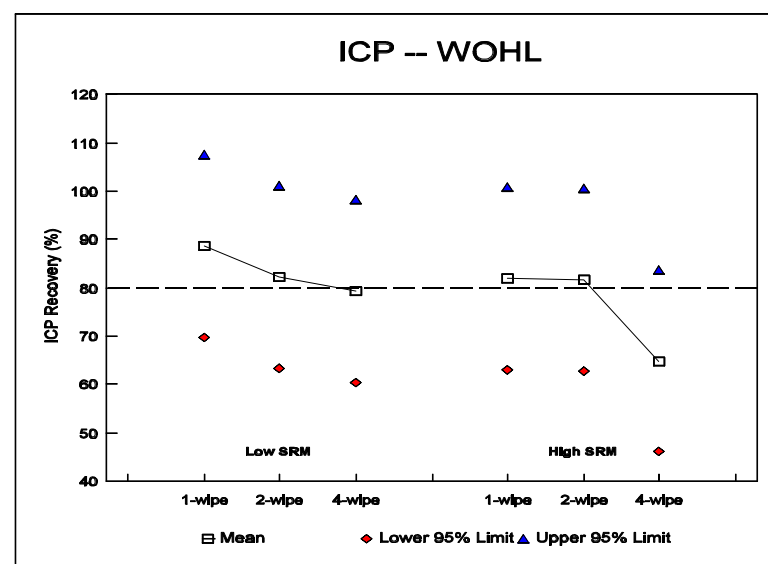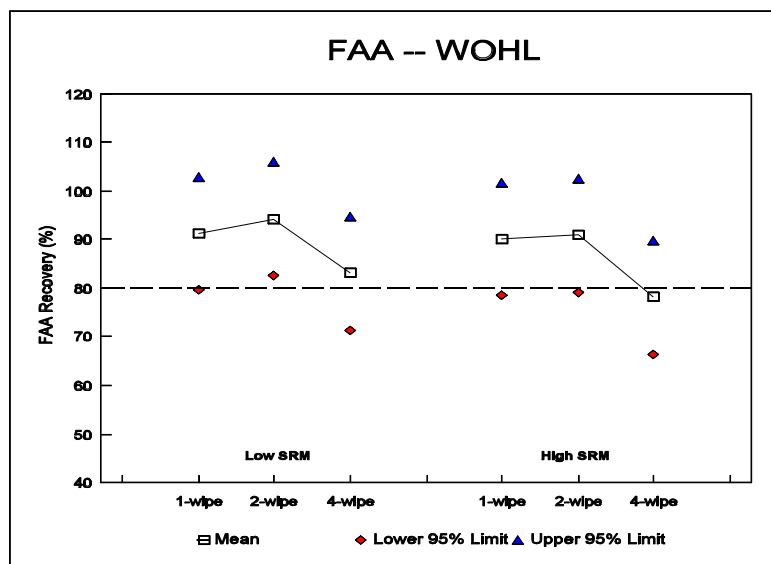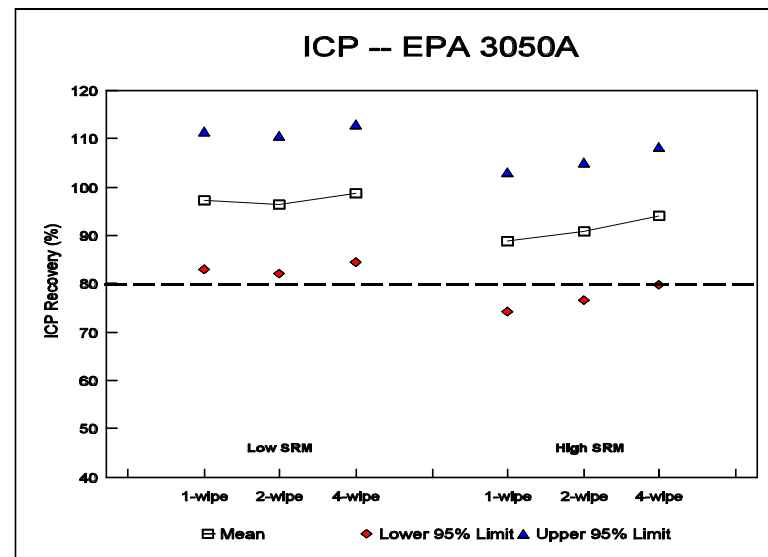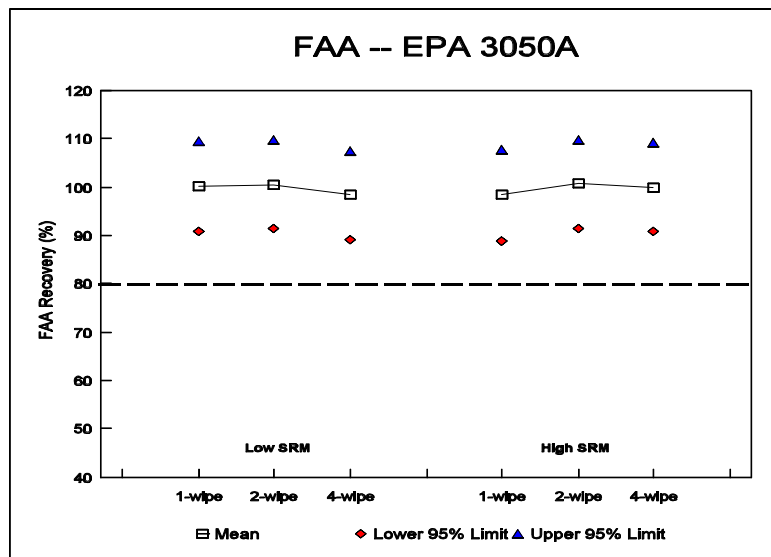
Figure 7-3. Recovery Statistics by Analytical Method, Digestion Method, SRM Loading Level, and Composite Type

intervals, calculated from the means and their standard errors, are relatively large. This is due to the fact that these statistics are based on only 4 degrees of freedom defined by the 3-way interaction: $(3-1)_{\text{Laboratory}} * (2-1)_{\text{SRM level}} * (3-1)_{\text{Composite type}}$, as the model is treated as a mixed model with the random factor, Laboratory.

Comparison of Method Recovery with Data Quality Objectives: One of the objectives of this study was to assess whether, upon compositing and digesting two or four wipes as single samples, acceptable recoveries could still be achieved. The data quality objectives were to obtain recoveries between 80% and 120%. The average recoveries shown in Table 7-3 provide a comparison for each situation tested:

— Regardless of the SRM loading level and the number of wipes per composite, acceptable recoveries were achieved for EPA Method 3050A, using either FAA or ICP analysis. Average recoveries ranged from a low of 98.3% to a high of 100.8% for FAA, and from a low of 88.7% to a high of 98.7% for ICP.

— Acceptable recoveries were achieved for the WOHL method for all three types of composite at the low SRM level (0.5 g per wipe) using FAA analysis. Average recoveries ranged from a low of 83.1% (four-wipe composite) to a high of 94.2% (two-wipe composite).

— Some difficulties were encountered with the WOHL method when compositing four wipes. This was the case for FAA and ICP results at the high SRM level (2 g per wipe), and for ICP results at the low SRM level. The average recoveries in these cases were 79.4% (ICP and low SRM level); 78.1% (FAA and high SRM level); and 64.9% (ICP and high SRM level). Of these three results, only one is considerably outside the data quality objectives.

7.2.2 Method Precision Analysis

Lead recovery results were obtained for various combinations of fixed factors based on the results from three laboratories. The analysis results presented in the previous section pertain to the accuracy of these recovery results, considering the effects of all factors. In this section, the precision of the recovery results is evaluated, with a focus on between and within laboratory precision components. Although some recovery results appeared to be outliers (see Section 6.3.1) for some laboratories, all data were included in the precision analyses as none of the data could be discarded for any plausible reasons (e.g., data reporting errors, laboratory anomalies).

Between and Within Laboratory Precision by Individual Composite Type: To estimate these two types of variability, the following approach was used. A two-way fixed model ANOVA including Laboratory, SRM loading level, and the interaction of these two factors was performed separately for each composite type and digestion method. As in previous analyses, this was done individually for FAA and ICP recovery results. Thus a

total of 12 ANOVAs were performed. This approach deviates from that previously used for assessing method accuracy, where Laboratory was treated as a random effect. In the present case, inference was made for the variability between and within the three laboratories.

For each analysis, the mean square error provided an estimate of the within laboratory variability, and the mean square associated with Laboratory provided an estimate of the between laboratory variability. The square root of each of these quantities provided an estimate of the corresponding standard deviation. Dividing the standard deviation by the mean recovery in each case provided an estimate of the corresponding relative standard deviations. Within and between precision estimates, expressed both in absolute standard deviation and relative standard deviation, are summarized in Table 7-4. In each row, the table shows the average recovery across all samples of a given type analyzed by all three laboratories using the corresponding digestion and analytical methods. Between and within laboratory precision estimated, expressed in percent relative standard deviations (% RSD), are plotted in Figure 7-4 separately for the two analytical methods.

<u>Within laboratory precision</u> is good, with ICP yielding slightly better precision estimates (3.18% to 8.12%) than FAA (4.01% to 7.93%). Except for ICP and WOHL, the two-wipe samples provided the best precision. The four-wipe samples consistently yielded the least precise results, although in all cases, the % RSD was less than 9%. In all cases, within laboratory precision, based on the results from the three laboratories included in the study, met the data quality objectives of being less than 20% RSD for both the EPA 3050A and the WOHL methods.

<u>Between laboratory precision</u> is, in many cases, an order of magnitude worse than within laboratory precision. This is especially true for some of the four-wipe composite samples. Except for FAA and EPA Method 3050A results, between laboratory precision considerably exceeded 20% relative standard deviation. As seen in Figure 7-4, no particular pattern is apparent between the one-, two-, and four-wipe composites.

7.2.3 Comparison of FAA and ICP Recovery Results

Each loaded wipe composite sample was analyzed by both FAA and ICP, two analytical methods commonly used by commercial laboratories for lead determination. In this section, the recoveries of the two methods are compared, separately for each digestion method, and within and between each laboratory.

In total, 396 samples were analyzed under different combinations of SRM levels, composite types, and lead-loading levels. For each individual sample, the paired difference between FAA and ICP recoveries was computed. The distribution of these pairwise differences was plotted previously in Figure 6-6 in Section 6, in the form of box plots, organized by laboratory, digestion method, and composite type. Overall, differences in

Table 7-4. Within and Between Laboratory Precision Results by Digestion Method, Analytical Method, and Composite Type

Digestion method	Composite type	Number of wipes	Mean recovery (%)	Absolute standard deviation (%)		Relative standard deviation[a] (%)	
				Between laboratories	Within laboratories	Between laboratories	Within laborator-ies
FAA Precision results							
EPA 3050A	1-wipe	36	99.25	16.21	4.75	16.33	4.79
	2-wipe	72	100.67	21.99	4.03	21.85	4.01
	4-wipe	72	99.18	16.97	5.22	17.11	5.26
WOHL	1-wipe	72	90.65	20.57	4.87	22.70	5.38
	2-wipe	72	92.52	25.45	4.53	27.51	4.90
	4-wipe	72	80.60	46.52	6.39	57.72	7.93
ICP Precision Results							
EPA 3050A	1-wipe	36	93.01	28.01	3.70	30.12	3.98
	2-wipe	72	93.61	43.25	2.98	46.20	3.18
	4-wipe	72	96.35	42.85	4.11	44.48	4.26
WOHL	1-wipe	72	85.28	45.58	4.34	53.45	5.09
	2-wipe	72	81.99	37.20	4.45	45.37	5.43
	4-wipe	72	72.14	73.51	5.86	101.89	8.12

[a] Relative standard deviation = 100*Absolute standard deviation/mean.
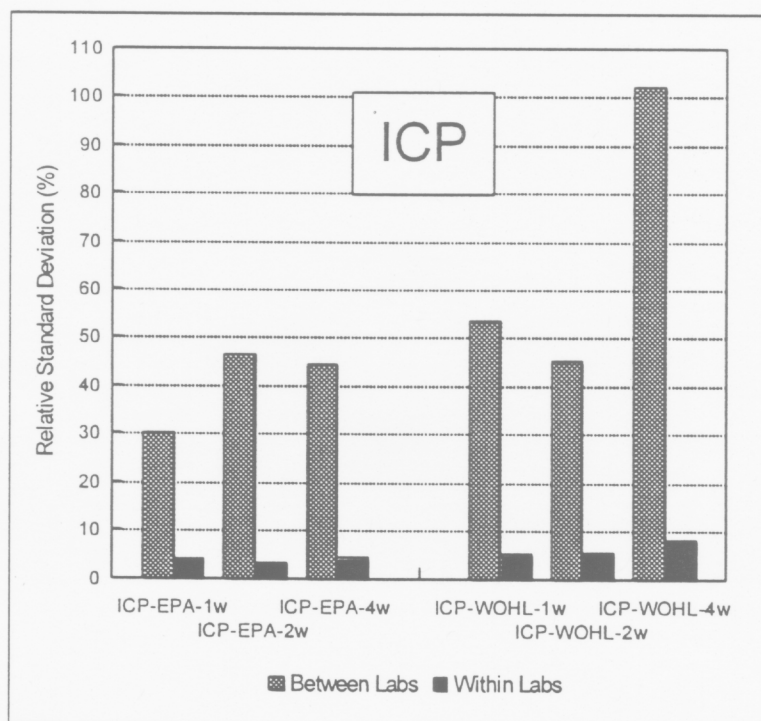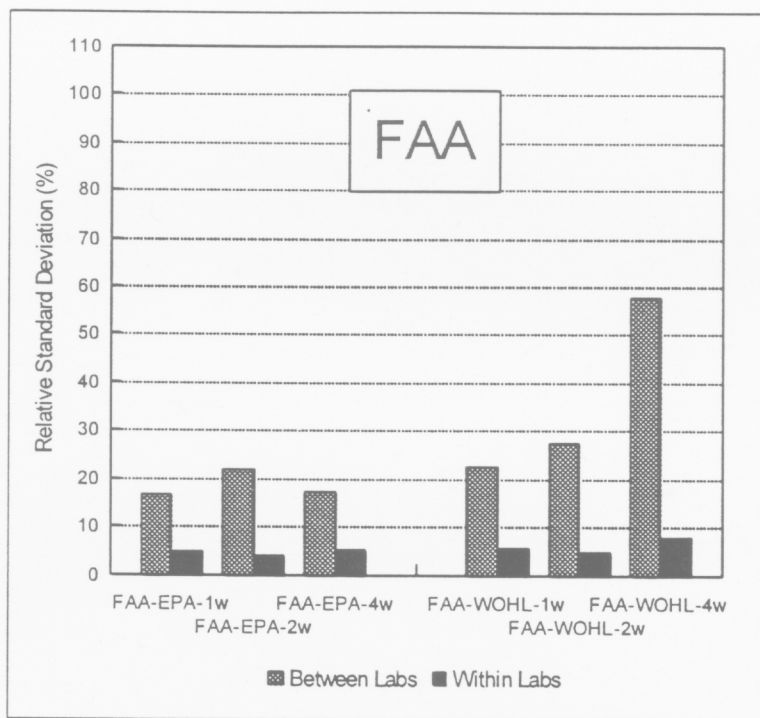
Figure 7-4. Between and Within Laboratory Precision by Analytical Method, Digestion Method, and Composite Type

recoveries between FAA and ICP ranged from -15.73% to 31.00%, with an average of 6.80% and a standard deviation of 9.36%.

The differences between FAA and ICP recovery results were analyzed in a way similar to that used in previous ANOVAs (see section 7.2.1). A mixed ANOVA model was used to investigate the effect of the fixed factors (composite type and SRM loading level) and the random factor (laboratory) on paired differences in analytical recovery results, separately for each digestion method. On the average, higher recoveries were obtained with FAA than with ICP, regardless of the digestion method used. However, in most cases, differences in recoveries were larger when using the WOHL method, all other conditions held constant. Of the two fixed effects and their interaction, only composite type significantly ($p=0.027$) affected the difference in recovery between FAA and ICP when using the EPA 3050A digestion method. For EPA Method 3050A, average (model least square means) differences between FAA and ICP recoveries, across SRM levels, were as follows:

Single-wipes: 6.24%
Two-wipe composites: 7.06%
Four-wipe composites: 2.84%

where the difference for the four-wipe composites is significantly lower (at the 5% level) than either that for the single wipes or the two-wipe composites.

Average differences and their lower and upper 95% confidence limits obtained from the mixed model ANOVAs are shown in Table 7-5, for each level of the fixed factors and their interaction. As shown in the table, none of the average differences is significantly different from zero as none of the relatively wide confidence intervals includes zero. This is in part due to the choice of the mixed model as an analysis approach and the fact that laboratories are the single largest contributor to the variability in these results.

Figure 6-6 clearly shows the variability among laboratories. Although the differences between the recoveries of the two analytical methods were consistently high at Laboratory 3 using either digestion method, these differences exhibit a relatively small variation. In comparison, Laboratory 1 showed consistently higher FAA results with very little variability for the WOHL method. On the other hand, when using the EPA Method 3050A, Laboratory 1 showed no consistent differences between the two analytical methods. This pattern is practically the opposite of that shown by the Laboratory 2 results.

Table 7-5.   Comparison of FAA and ICP Recoveries by Digestion Method, SRM Level, and Composite Type

Digestion method	SRM level	Composite type	FAA-ICP recovery difference (%)		
			Mean	95% lower limit	95% upper limit
EPA 3050A	Low	1-wipe	2.92	-13.50	19.34
		2-wipe	4.12	-12.22	20.45
		4-wipe	-0.37	-16.71	15.96
		All	2.22	-22.87	27.31
	High	1-wipe	9.56	-6.86	25.98
		2-wipe	10.01	-6.33	26.34
		4-wipe	6.05	-10.29	22.38
		All	8.54	-16.55	33.63
	All	1-wipe	6.24	-7.86	20.34
		2-wipe	7.06	-6.99	21.12
		4-wipe	2.84	-11.22	16.89
WOHL	Low	1-wipe	2.61	-12.09	17.32
		2-wipe	11.89	-2.81	26.60
		4-wipe	3.70	-11.01	18.40
		All	6.07	-11.40	23.54
	High	1-wipe	8.13	-6.58	22.84
		2-wipe	9.16	-5.54	23.87
		4-wipe	13.22	-1.48	27.93
		All	10.17	-7.30	27.65
	All	1-wipe	5.37	-6.78	17.52
		2-wipe	10.53	-1.63	22.68
		4-wipe	8.46	-3.69	20.61

7.3 Evaluation of Process Parameter Data

The second objective of this study was to determine whether compositing of wipes, if acceptable on a technical or performance basis, could reduce the cost of sample preparation and analysis relative to single-wipe methods. To help assess the relative costs of the methods, each laboratory was asked to record the time, materials, and equipment requirements for each sample digestion and analytical procedure included in the study. It should be noted that the time required to conduct analytical methods is *not* a parameter typically reported by commercial laboratories.

7.3.1 Initial Process Parameter Data

The cost information initially provided by the laboratories was fairly limited since the digestion times reported by each laboratory were based only on one or two sample batches for each digestion method and composite type. Only one batch of single wipes and one batch of two-wipe composites were prepared using the EPA Method 3050A and the WOHL method. Only one batch of four-wipe composites was prepared using the WOHL method, while two batches of four-wipe composites were prepared using the EPA method. Until new digestion methods become routine, the actual time required to use the methods cannot be defined with certainty.

The approximate time required to perform each digestion method and composite type combination was recorded by each laboratory participating in the investigation. A relative sample preparation and analysis cost comparison was then made. A general evaluation of the results indicated that, within a given laboratory, the time requirements for a given method were fairly consistent across composite types. The multiwipe composites required slightly more time to digest relative to the single-wipe samples; however, that increase was not proportional (i.e., the two- and four-wipe composites did not take two and four times as long to digest as the single-wipe samples, respectively). The single-wipe samples digested using the WOHL method at Laboratory 1 actually required more time to digest than the multiwipe composites, but this is most likely attributable to the lack of familiarity with that method.

An interlaboratory comparison was made for the digestion times reported by each laboratory; it was found that the data were not very comparable. Although each laboratory reported the total hours required for each digestion method and composite type combination, differences in other time parameters were reported. For example, Laboratory 1 reported the total hours to complete the digestions, and reported also the number of labor hours required (e.g., the hours when staff were actively working on the digestion procedures, including glassware cleaning, filtering samples, centrifuging samples, etc.). Laboratory 2 reported the total hours and the digestion times required for each method. Laboratory 3 simply reported the total hours and the number of hours that the samples were on the hot plate for each digestion procedure.

The total hours reported by Laboratory 1 and Laboratory 3 were somewhat comparable; however, the total hours reported by Laboratory 2 were an order of magnitude lower than the digestion times reported by Laboratories 1 and 3. Additional information was requested from Laboratory 2, but the revised data were similar to those originally reported. It was noted that the digestion times reported by Laboratory 2 for the WOHL method corresponded to those specified in the procedures for the digestion. The total hours reported by Laboratory 2 for the EPA Method 3050A were considered to be unrealistic, in comparison to the results from Laboratories 1 and 3. Based on these observations, it was concluded that Laboratory 2 did not accurately record the required digestion times for the study. Thus, the times reported by that laboratory were not included in the evaluation. Because Laboratory 1 recorded all work steps in the necessary detail and since all

laboratories followed the same steps in conducting the digestion procedures, only the total hour and labor hour data provided by Laboratory 1 were evaluated. The data reported by Laboratory 1 for each digestion method and composite type combination were considered representative of all three laboratories and are presented in Table 7-6.

The percent relative effort and estimated percent reduction in effort per wipe were calculated for each digestion method (EPA 3050A and WOHL). These results are shown also in Table 7-6 and provide a means for comparing the digestion times to evaluate the cost efficiency of the composite wipe methods. Using the single-wipe batch type as the basis for cost comparisons, a reduction of 70% was estimated for the four-wipe composites using the EPA Method 3050A. A reduction of 77% was estimated for the four-wipe composites using the WOHL method. That is, laboratory analysis of a four-wipe composite was estimated to be 70% and 77% less time consuming than four single-wipe analyses for the EPA Method 3050A and the WOHL method, respectively. Similarly for the two-wipe composites, time reductions of 39% and 53% were estimated for the EPA 3050A and WOHL methods, respectively.

7.3.2 Supplementary Process Parameter Data

The three laboratories also evaluated the cost aspects of compositing from a retrospective viewpoint, based on the experience gained from the study. Each laboratory was asked to estimate the relative cost factors that could be applied to the digestion methods, assuming someone approached the laboratory with a need to analyze *a sizable number of composite samples for lead*. A limited sample preparation and analysis cost comparison was performed based on the information provided by the three laboratories.

Comparison across composite types, separately for each digestion method: The relative cost factors that could be applied to two- and four-wipe composites for a given digestion method were estimated, assuming the single-wipe methods represented 100%. For example, if two-wipe composites digested with the EPA method required 1.5 times the effort needed to digest single-wipe samples using the same method, then the relative cost factor would be 150% for two-wipe composites. The data reported by each laboratory from this comparison are presented in Table 7-7.

Again, using the single-wipe batch type as the basis for cost comparisons, an average per-wipe cost reduction of 64% was estimated for the four-wipe composites using EPA Method 3050A. For example, suppose that a single wipe costs $20 to analyze. Based on the reduction of effort obtained from this study, a four-wipe composite would cost $28.50, a 64% cost reduction over $80, the cost of four single wipes. A reduction of 67% was estimated for the four-wipe composites using the WOHL method. Similarly for the two-wipe composites, per-wipe cost reductions of 42% and 38% were estimated for the EPA 3050A and WOHL methods, respectively. The estimated cost reductions from the retrospective data are therefore in line with the cost reduction data obtained from Laboratory 1 (Table 7-6).

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Table 7-6.   Relative Effort Comparison Among Types of Composite for Laboratory 1

Digestion method	Composite type	Number of analysis batches	Total hours	Labor hours	Labor hours per batch	Relative effort (%) Per batch[a]	Per wipe[b]	% Reduction in effort per wipe[c]
EPA 3050A	1-wipe	1	18	14	14	100	100	NA
	2-wipe	1	17	17	17	121	61	39
	4-wipe	2	40	34	17	121	30	70
WOHL	1-wipe	1	21	16	16	100	100	NA
	2-wipe	1	19	15	15	94	47	53
	4-wipe	1	19	15	15	94	23	77

[a]   Within each cell, % ratio of labor hours per batch (2- or 4-wipe composite) to labor hours per batch (1 wipe).
[b]   Within each cell, % relative effort per batch divided by the number of wipes in the composite.
[c]   The % reduction in effort per wipe is calculated as 100% minus the % relative effort per wipe.

Table 7-7.   Relative Cost Factor Comparison Across Composite Types, Separately for Each Digestion Method

Method	Laboratory	Single-wipe weighting (%)	2-wipe composite Relative cost factor (%)	% Relative effort per wipe[a]	% Reduction in effort per wipe[b]	4-wipe composite Relative cost factor (%)	% Relative effort per wipe[a]	% Reduction in effort per wipe[b]
EPA 3050A	1	100	120	60	40	150	38	62
	2	100	130	65	35	150	38	62
	3	100	100	50	50	125	31	69
WOHL	1	100	110	55	45	125	31	69
	2	100	110	55	45	120	30	70
	3	100	150	75	25	150	38	63

[a]   The % relative effort per wipe is calculated by dividing the relative cost factor by the number of wipes in the composite.
[b]   The % reduction in effort per wipe is calculated as 100% minus the % relative effort per wipe.

Based on this limited cost comparison, an approximate cost benefit can be derived from compositing.

Comparison between digestion methods, separately for each composite type: The cost-effectiveness of the EPA method versus the WOHL method was also of interest. The relative cost factors that could be applied to the WOHL digestion method, for a given composite type, were estimated assuming the EPA method represented 100% for each composite type. For example, if the WOHL Method for two-wipe composites could be performed in half the time required by the EPA Method for two-wipe composites, then the relative cost factor would be 50% for the WOHL method. The data from this comparison are presented in Table 7-8.

Table 7-8. Relative Cost Factor Comparison Between Digestion Methods, Separately for Each Composite Type

Composite type	Laboratory	EPA method weighting (%)	WOHL method weighting (%)	% Reduction in effort WOHL vs. EPA
1-wipe	1	100	60	40
	2	100	30	70
	3	100	50	50
2-wipe	1	100	60	40
	2	100	40	60
	3	100	75	25
4-wipe	1	100	50	50
	2	100	40	60
	3	100	50	50

Across all laboratories and composite types, cost reductions of 25% to 70% were estimated for the WOHL method as compared to the EPA Method 3050A. Thus, based on these performance data alone, cost saving can be derived from using the WOHL method instead of EPA Method 3050A. It should be noted that the process parameter data do not take into consideration the analytical performance of the methods.

7.3.3 Other Cost Data

The costs of reagents, waste disposal, and associated increased safety were higher for the multiwipe composite methods relative to the single-wipe methods since larger quantities of acids are needed to perform composite sample digestion. However, these costs also were compared to the total cost per sample to determine whether that increase was significant. The reagent and waste disposal costs for the EPA Method 3050A were

estimated at $1, $2, and $4 per sample for the single-, two-, and four-wipe composite types, respectively. If the wipe samples are composited, then the reagent and waste disposal costs can be normalized to $1/wipe. The reagent and waste disposal costs were a negligible part (less than $0.50 per sample) for the WOHL single- and composite-wipe methods. Thus, the WOHL method is less expensive than the EPA Method 3050A in all cases.

The times required for the ICP and FAA analyses were reported also by each laboratory. As expected, the analysis times did not vary by composite type for a given analytical technique. All three laboratories indicated that an FAA analysis required less time than an ICP analysis, with an estimated 2 min per sample for FAA versus 3 to 5 min per sample for ICP. However, since many laboratories use autosamplers, the importance of instrument analysis time is not very important in the context of cost savings. In addition, since FAA instruments are also less expensive to purchase than ICP instruments, a cost advantage can be achieved from using FAA rather than ICP.

Section 8
References and Bibliography

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Method Review Summary—Protocols for Sample Preparation and Analysis of Lead in Single-Surface and Composite Wipe Samples

Method Review Summary—Protocols for Sample Preparation and Analysis of Lead in Single-Surface and Composite Wipe Samples

1   Introduction

The contractor completed a review of available protocols for the preparation and analysis of lead (Pb) in single-surface and composite wipe samples, and found that few protocols were available for composite wipes. Sample preparation techniques typically use acid(s) and water to solubilize (digest) the Pb contained in the samples, prior to analysis. Sample preparation procedures written specifically for single-surface samples may require modifications for multiwipe composites, because of the increased mass of wipe and dust material contained in the samples. As the total mass of a sample increases (composite samples), the total amount of solution needed to solubilize the sample increases. Because sample preparation protocols for lead are designed and validated with specific upper limits for sample mass, most of the currently available preparation protocols are not intended for composite wipes.

The analysis of composite samples is dependent upon the preparation of sample digestate, which is likely the most important step in the analysis process. The effectiveness of a given method is measured by a number of analytical parameters including accuracy and precision. Of course, each laboratory would need to validate its performance with any method for the analysis of composite wipes as well as single-wipe samples.

The mass of the dust collected on a single-wipe sample is generally unknown and cannot be determined in the laboratory, because the tare weight of the wipe prior to sample collection cannot be measured. Using a blank wipe to obtain a tare weight is not effective since the mass between wipes is variable as a result of inconsistencies in manufacturing and varying degrees of wetness among individual wipes. Therefore, research to develop or validate a laboratory method for preparing composite wipe samples should include an assessment of the maximum level of dust loading that can be tolerated by the method, to ensure that the method is suitable for the intended sampling application (e.g., risk assessments, lead hazard screens, or clearance testing).

This appendix is divided into four sections. Section 1 provides an introduction, and Section 2 provides sample preparation and analysis protocols, including descriptions of sample preparation and analytical techniques currently available in the literature for single-surface and composite wipe samples. Section 3 discusses the advantages and disadvantages of the methods, and Section 4 provides a list of references reviewed in preparing this appendix.

2 Protocols

The sample preparation and analysis protocols found in the literature search are summarized in Sections 2.1 and 2.2, respectively. Most of the methods cited apply to single-surface samples, but may be adaptable to composite wipes.

2.1 Sample Preparation Techniques

Several preparation techniques were reviewed; however, few were designed for composite samples. A summary of the available information on sample preparation techniques for the determination of lead in wipe samples is given in Table A-1. The superscript number next to the method type identifies the corresponding document referenced in Section 4.

2.2 Analytical Techniques

The most commonly used techniques to analyze lead in the laboratory are flame and graphite furnace atomic absorption spectrometry (AAS) and inductively coupled plasma atomic emission spectrometry (ICP-AES). Other techniques include the polarographic, colorimetric, and anodic stripping voltammetry methods. The analysis protocols reviewed are discussed below and their advantages and disadvantages are summarized in Table A-2. The superscript refers to the Section 4 references.

Flame Atomic Absorption Spectrometry (FAA).[5,6,11] An oxidizing air-acetylene flame-type is recommended. The working range is dependent on the analytical line selected for analysis. The 217.0-nm line with a slit width of 1.0 nm has an optimum working range of 2.5 to 20 µg/mL with a sensitivity of 0.06 µg/mL. The 283.3-nm line is often preferred for routine analysis using a slit width of 0.5 nm and a working range of 7.0 to 50 µg/mL with a sensitivity of 0.16 µg/mL. Few interferences are reported. The use of deuterium background correction is recommended at the 217.0-nm wavelength to correct for non-atomic absorbances. For method of quantitation, standards, blanks, and samples are aspirated and absorbances are recorded. Standards of known concentration are plotted versus the obtained absorbance. Using the measured sample absorbances, the corresponding sample concentrations (µg/mL) are calculated from the standard curve plotted. The lead concentration ($\mu g/ft^2$) is calculated by multiplying the sample concentration by the sample dilution volume (mL) and then dividing the product by the area sampled in square feet. FAA is a readily available laboratory method that is easy to operate. The method is capable of quantifying lead from 0.1 µg/mL and recoveries (when digested with $HNO_3:H_2O_2$) can be expected to be from 95% to 105%.

Table A-1. Summary of Sample Preparation Methodologies for Wipe Samples

Method type	Summary description
Hot Plate[2,6] (HNO_3:H_2O_2)	**Advantages:** Commonly used laboratory method. Possibly adaptable for composite wipes. Lead recovery is expected to be adequate.
	Disadvantages: Limited to 1 wipe. Method not easily adaptable to field use since it requires a laboratory fume hood.
	Typical Digestion Vessel Size: 150-mL or 250-mL beaker
	Primary Chemicals Used: HNO_3, H_2O_2
	Final Dilution Volume: 100 mL
	Procedure: Transfer the single-wipe sample to a clean empty beaker. Add 25 mL 1:1 HNO_3, gently swirl to mix, and cover with a watch glass. Gently heat the sample at 85° to 100°C and reflux for 10 to 15 min without boiling. Remove from hot plate and allow the sample to cool. Add 10 mL concentrated HNO_3, replace the watch glass and reflux for 30 min. Repeat this last step to ensure complete oxidation. Allow the solution to evaporate to approximately 10 mL without boiling, while maintaining a covering of solution over the bottom of the beaker. Cool the sample, add 5 mL of Type I water and 5 mL of 30% H_2O_2. Cover the beaker with a watch glass and return the covered beaker to the hot plate for warming. Heat until the effervescence subsides and cool the beaker. Continue to add 30% H_2O_2 in 1-mL aliquots with warming until the effervescence is minimal or until the general sample appearance is unchanged. Do not add more than a total of 10 mL of 30% H_2O_2, even if the effervescence has not been reduced to a minimal level. Heat the acid-peroxide digestate carefully until the volume has been reduced to approximately 10 mL. Allow the digestate to cool, rinse the beaker walls and bottom of the watch glass with Type I water, and quantitatively transfer it to a 100-mL volumetric flask. Dilute to volume with Type I water.
Hot Plate[7] (HNO_3:H_2O_2)	**Advantages:** Relatively simple, commonly used laboratory method. Possibly adaptable for composite wipe samples. Lead recovery is expected to be adequate.
	Disadvantages: Currently written for the preparation of air particulate on cassette filters. Modifications must be made to use for wipe samples. Modifications would need to be substantial to use on composite wipe samples. Possibly adaptable for composite wipes. This method is not easily adaptable to field use since it requires a laboratory fume hood.
	Typical Digestion Vessel Size: 50-mL beaker
	Primary Chemicals Used: HNO_3, H_2O_2
	Final Dilution Volume: 10 mL
	Procedure: Transfer one filter sample to a clean, empty beaker. Add 3 mL concentrated HNO_3 acid and 1 mL 30% H_2O_2 and cover with a watch glass. Gently heat the sample at 140°C until the volume is reduced to about 0.5 mL. Repeat two more times using 2 mL HNO_3 and 1 mL 30% H_2O_2 each time. Heat on the hot plate (140°C) until about 0.5 mL remains. When the sample is dry, rinse the beaker walls and bottom of the watch glass with 3 to 5 mL 10% HNO_3. Allow the solution to evaporate to dryness. Cool each beaker and dissolve the residue in 1 mL HNO_3. Transfer the solution to a 10-mL volumetric flask and dilute to volume with distilled water.
Hot Plate and Microwave[3] (HCl:HNO_3:$HClO_4$)	**Advantages:** Relatively simple laboratory method. Possibly adaptable for composite wipes. Lead recovery is expected to be adequate.
	Disadvantages: Limited to 1 wipe. This method not easily adaptable to field use since it requires a laboratory fume hood. The use of $HClO_4$ is potentially explosive.
	Typical Digestion Vessel Size: 50-mL beaker
	Primary Chemicals Used: HNO_3, H_2O_2, $HClO_4$
	Final Dilution Volume: 50 mL
	Procedure: Place each wipe in a 50-mL beaker, add the optimum mixture (15 mL) 6:2:5 HCl:HNO_3:$HClO_4$, and carry out digestion at 100°C until no brown gas evolves. Decant the acid into a beaker after centrifugation at 700 rpm for 10 min, evaporate it just until it is dry, and solubilize the residue in 10 mL 10% HNO_3.

Method type	Summary description
Hot Plate and Microwave Preparation[3]	**Advantages:** Written for multiple wipes. **Disadvantages:** Lead recovery information is not known. This method not easily adaptable to field use since it requires a laboratory fume hood. Evaporation step is expected to be extremely time consuming and is prone to losses caused by spattering of the sample at the indicated temperature. The use of $HClO_4$ is potentially explosive. **Typical Digestion Vessel Size:** 800-mL beaker **Primary Chemicals Used:** HNO_3, $HClO_4$ **Final Dilution Volume:** 10 mL **Procedure:** Up to six wipes are placed in a 800-mL beaker to which 5:4 HNO_3:$HClO_4$ acid (100 mL) was added. The microwave is operated at 10% power for 15 min and at 50% power for an additional 15 min, after which the contents are swirled manually and then processed again for 15 min at 50% power. The beaker is transferred to a hot plate, and the acid evaporated to dryness at 250°C. After cooling, the residue is redissolved in 10 mL 10% HNO_3 for lead analysis.
Room Temp Elution[4] (HCl)	**Advantages:** Simple procedure. Possibly adaptable to field use. Possibly adaptable for composite wipe samples. **Disadvantages:** Limited to 1 wipe sample. Lead recovery information is not known. Extraction using 0.1 N HCl at room temperature is not expected to achieve full lead recoveries from all samples. Contamination potential exists from drying step. **Typical Digestion Vessel Size:** 30-mL test tube **Primary Chemicals Used:** HCl **Final Dilution Volume:** 20 mL **Procedure:** Flatten the sample on a clear surface, allow it to dry, roll it up and insert into a test tube. Add 20 mL of 0.1 N HCl and allow the sample to stand at room temperature for 10 to 15 hours. Decant the acid solution (leachate) and take 15-mL aliquots for analysis.
Ultrasonification[8] (HNO_3) Preparation	**Advantages:** Simple procedure that is readily usable in field. Possibly adaptable for composite wipe samples. **Disadvantages:** Limited to 1 wipe. Lead recovery information is not known. **Typical Digestion Vessel Size:** 50-mL beaker **Primary Chemicals Used:** HNO_3 **Final Dilution Volume:** 30 mL **Procedure:** Transfer wipe samples to a small glass beaker, treat with 30 mL of 10% HNO_3 and then sonicate in a commercial ultrasonic cleaner for 60 minutes.
Dry-Ashing Procedures[9]	**Advantages:** Relatively simple procedure. Not adaptable to field use. Can be used for composite wipe samples. **Disadvantages:** Dry-ashing procedures have potential problems with convection losses, volatility loss of lead, contamination problems, and limited batch sizes. **Typical Digestion Vessel Size:** 100 mL porcelain dish **Primary Chemicals Used:** HNO_3 **Final Dilution Volume:** 100 mL **Procedure:** Dry-ashing procedures employ furnace heating to oxidize a sample and convert sample components to mostly oxides. The resulting ash is solubilized in HNO_3 or HCl and diluted to volume with water. An ashing agent is commonly used to aid in retaining the elements of interest during the heating process.

Table A-1 (Continued)

Method type	Summary description
Room Temperature and Hot Plate[13] (1 M HNO$_3$)	**Advantages:** Relatively simple laboratory method. Applicable to six-wipe composites from children's hands. **Disadvantages:** This method may not accommodate the increased sample mass and particle sizes expected from the collection of settled dust. **Typical Digestion Vessel Size:** 800-, 250-, and 100-mL beakers **Primary Chemicals Used:** 1 M HNO$_3$ **Final Dilution Volume:** 10 mL **Procedure:** Transfer each sample to a labeled 800-mL beaker. Add 100 mL of 1 M HNO$_3$ to each sample, and swirl each sample for 10 sec with a glass stirring rod to thoroughly wet the wipes. Cover the samples with watch glasses and allow the samples to extract at room temperature for 2 hr. Decant the acid solution from the 800-mL beaker into a clean, labeled 250-mL beaker. Add 50 mL of 1 M HNO$_3$ to the hand wipes in the 800-mL beaker, and swirl the wipes with a glass rod for 10 sec. Decant the acid solution into the same 250-mL beaker to composite the acid rinse. Again, add 50 mL of 1 M HNO$_3$ to the hand wipes in the 800-mL beaker and swirl the wipes with a glass rod for 10 sec. Decant the acid rinse solution into the same 250-mL beaker to composite the acid rinse for a total volume of about 200 mL. Place the samples with watch glass covers on a hot plate at a setting that does not exceed 100°C (if lead is the only analyte the temperature can be 140°C). Rinse the stirring rods into the beakers and discard them. Evaporate the samples until the sample volume is approximately 25 mL. Remove the samples from the hot plate, cool, and filter the samples collecting the filtrate in labeled 100-mL beakers. Place the 100-mL beakers with watch glass covers on a hot plate and reduce the volume to approximately 5 mL. Remove the samples from the hot plate and cool. Transfer each sample to a graduated test tube with stopper and dilute to 10 mL with distilled deionized water.

Table A-2.   Laboratory Analytical Protocols

Protocol	Function	Field or lab use	Advantages and disadvantages
Flame Atomic Absorption Spectrometry[5,6,11]	Analysis	Lab	A (Advantage): Readily available and capable of quantifying lead from 0.1 µg/mL; recoveries can be expected to be from 95% to 105%. D (Disadvantage): Interferences: high concentrations of calcium, sulfate, carbonate, phosphate, iodine, fluoride, or acetate.
Inductively Coupled Argon Plasma Atomic Emission Spectrometry[6,9]	Analysis	Lab	A: Readily available and capable of quantifying lead from 0.03 µg/mL; recoveries can be expected to be from 95% to 105%. D: Spectral interferences.
Graphite Furnace[1,6]	Analysis	Lab	A: Readily available and capable of quantifying lead from 0.002 µg/mL; recoveries can be expected to be from 95% to 105%. D: High concentrations of calcium sulfate, phosphate, iodide, fluoride, or acetate can interfere with lead determination.
Polarographic[4]	Analysis	Lab	A: Recoveries of soluble lead standard from towels using the HCl room temperature elution method gave recoveries of approximately 90%. D: Selective and sensitive method. Not widely used.
Colorimetric[8]	Analysis	Field/ Lab	A: Method can be used in the field. Lead can be extracted from the samples using the ultrasonic technique, then measured with the Hach method. D: Limited sample size.
Anodic Stripping Voltammetry[10]	Analysis	Field	A: Method can be used in the field. D: Not widely available.

Inductively Coupled Argon Plasma Atomic Emission Spectrometry (ICP).[6,9] The working range (at the lead wavelength of 220.3 nm) for this method is 0.03 to 20.0 µg/mL. Spectral interferences are the primary interferences encountered in ICP analysis. These are minimized by interelement correction factors and background correction. For method of quantitation, standards, blanks, and samples are aspirated and emissions are recorded. Standards of known concentration are plotted versus the obtained emission. Using the measured sample emissions, the corresponding sample concentrations (µg/mL) are calculated from the standard curve plotted. The lead concentration ($µg/ft^2$) is calculated by multiplying the sample concentration by the sample dilution volume (mL), then dividing the product by the area sampled in square feet. The ICP method is a readily available laboratory application that is easy to operate. The method is capable of quantifying lead from 0.03 µg/mL and recoveries (when samples are digested with $HNO_3:H_2O_2$) can be expected to be from 95% to 105%.

Graphite Furnace Atomic Absorption Spectrometry (GFAAS).[1,6] The instrument is equipped with background correction and a lead hollow cathode lamp or equivalent and is capable of making lead absorption measurements at the 283.3-nm absorption line. For instrument setup, the GFAAS spectrometer should be set for the analysis of lead at 283.3 nm according to the instructions given by the manufacturer. The hollow cathode lamp should have at least a 30-minute warmup prior to starting calibration and analysis. The calibration curve for GFAAS should consist of a minimum of three calibration standards and a blank. Calibration and quantitative lead measurement of sample digestates and instrumental quality control are performed in sequential order. A calibration curve is prepared to convert instrument response to concentration using a linear regression fit. All instrumental measurements are converted on instrumental quality control standards and sample digests to lead concentration using the calibration.

Polarographic.[4] Polarographic waves are recorded from -0.24 to -0.69 volts against a saturated calomel electrode (SCE). A 0.4-mL portion of 0.2% gelatin solution is added for suppression of the polarographic maxima. For method quantitation, standards of known concentration made from stock solutions are analyzed, and a calibration curve is drawn relating the lead concentration (µg/mL) and the limiting current (µA) of polarographic wave. Sample concentrations are calculated from the current (µA) of polarographic wave measured versus the calibration curve (µg/mL). The lead concentration ($µg/ft^2$) is calculated by multiplying the sample concentration by the sample dilution volume (mL) and then dividing the product by the area sampled in square feet. Polarography is a selective and sensitive method for the determination of lead. It is not widely used.

Colorimetric[8] (Hach Method). An aliquot of the extract is added to 100 mL deionized water. The solution is passed through an extraction column and lead is removed from the solution. Lead is then eluted from the column, and a color-forming reagent is added. The resultant solution is divided in two; in one of the halves, the colored product of the lead and color-forming reagent are dispersed by the addition of a decolorizing reagent. The colorimeter is blanked with the decolorized sample, and the other half is placed in the

colorimeter to read concentration. The linear range of the method is approximately 1 to 150 µg Pb/L.

Metals that have been reported to interfere with lead analysis by this method are: aluminum, barium, calcium, copper, iron, magnesium, manganese, and zinc. Several other inorganic ions are also reported to interfere with lead analysis including: chloride, fluoride, ammonium, nitrate, and sulfate. For method of quantitation, standards, blanks, and samples are treated with the color-forming reagent, and the percent transmittance of the samples is recorded. Standards of known concentration are plotted versus the obtained percent transmittance. Using the measured sample percent transmittances, the corresponding sample concentrations (µg/mL) are calculated from the standard curve plotted. The lead concentration (µg/ft^2) is calculated by multiplying the sample concentration by the sample dilution volume and then dividing the product by the area in square feet. This method can be taken into the field for measurement of lead in samples collected. Lead can be extracted from the samples using the microwave technique and then measured with the Hach Method. Recoveries of 90% to 110% can be expected for samples using this method.

Anodic Stripping Voltammetry (ASV).[10] The ASV technique employs disposable electrodes to measure lead in solutions. Voltametric methods are based on the current-voltage curves arising from a microelectrode when diffusion is the rate-determining step in the electrochemical reaction. The ASV device is compact and portable, making it ideal for field analysis.

3   Summary of Advantages and Disadvantages of Methods

There are advantages and disadvantages to the sample preparation and analytical methods discussed in Section 2 (see Tables A-1 and A-2) for use with composite wipe samples. Note that most of these methods and protocols were originally designed for the preparation and analysis of single-wipe samples.

3.1 Laboratory Preparation Methods

Hot plate digestion methods are widely used procedures in which lead is solubilized with liquid oxidizing reagents facilitated by heat using a hot plate. Hot plate procedures, which are generally not subject to the loss and contamination problems common to other preparation procedures, can only be performed in a laboratory. Although the room temperature elution and ultrasonification preparation methods can be performed in the field, the sample size is limited.

A-8

3.2 Laboratory Analytical Methods or Protocols

Flame atomic absorption spectrometry is readily available and capable of quantifying lead from 0.1 μg/mL and recoveries can be expected to be from 95% to 105%. However, interferences that can affect results include high concentrations of calcium, sulfate, carbonate, phosphate, iodine, fluoride, or acetate. Inductively coupled argon plasma atomic emission spectrometry is readily available and capable of quantifying lead from 0.03 μg/mL. Recoveries can be expected to be from 95% to 105%. There are, however, spectral interferences. Polarographic analysis yielded recoveries for towels spiked with soluble lead standard of approximately 90% when using the HCl room temperature elution method. This analytical method, which is selective and sensitive, is not in wide use. Although colorimetric analysis can be used in the field, the method was originally designed for water testing.

4 References

MRI reviewed the documents listed below. The materials listed contained valuable and useful information.

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Modified SW-846 Method 3050A Acid Digestion Procedure for Single-Wipe Samples

Modified SW-846 Method 3050A Acid Digestion Procedure for Single-Wipe Samples

1   Summary of Method

This method is an acid digestion procedure based on SW-846 Method 3050A. The published method was revised to accommodate dust samples collected on a single wipe. This method is used for the acid digestion of dust samples and associated quality control (QC) samples for analysis of lead. It should be noted that this procedure does not use hydrochloric acid for digestion. The entire wipe sample is digested in nitric acid (HNO_3) and hydrogen peroxide (H_2O_2). The digestates are diluted to a final volume of 100 mL following the final cookdown step. The sample digestate will contain approximately 10% (v/v) HNO_3 following digestion.

2   Apparatus and Materials

2.1   Beakers: 250-mL Griffin

2.2   Watch glasses appropriate for designated beakers

2.3   Forceps: polyethylene, or equivalent

2.4   Volumetric flasks with stoppers: 100-mL, Class A

2.5   Funnels: plastic or glass, sized to fit into the 100-mL volumetric flasks

2.6   Thermometers: red alcohol, range of 0° to 110°C

2.7   Hot plates: capable of maintaining a temperature of approximately 95°C

2.8   Centrifuge (optional)

2.9   Centrifuge tubes: polyethylene with screw caps, 50-mL capacity

2.10   Kimwipes, or equivalent

2.11   Syringes: plastic, 10-mL disposable with Luer-lok fittings

2.12   Gelman Acrodisc filters, female Luer lok, 0.45-µm pore size, or equivalent

2.13   Filter paper: Whatman No. 541, or equivalent

2.14   Food service towels

2.15   Gloves: disposable, powderless, vinyl

2.16   Pipettes with disposable tips

2.17   Repipet reagent dispensers

2.18   Analytical balance: Capable of weighing to the nearest 0.0001 g

2.19   Drying oven: Capable of maintaining a temperature of ~ 110°C

2.20   Desiccator

3   Reagents/Standard Stock

3.1 Milli-Q reagent water: minimum resistance of 16.67 MΩ-cm, or equivalent

3.2 Concentrated nitric acid (HNO_3)—70% to 71%: Baker Instra-Analyzed grade, or
     equivalent

3.3 Hydrogen peroxide (H_2O_2)—30%: A.C.S. reagent grade

3.4 NIST SRM 2704—Buffalo River Sediment

4   Quality Control

Quality control (QC) samples are processed with each digestion batch. The QC
samples to be included in each batch are summarized in Table B-1.

Table B-1. Quality Control Samples Prepared With Each Batch

Description	Abbre-viation	Purpose	Frequency
Method blank	MB	Demonstrate absence of laboratory contamination	One with each sample batch
Wipe Blanks	WB	Determine background levels in the wipe material	Incorporated into the batch as specified in the test design. One of each wipe type per batch.
Laboratory control sample (NIST SRM 2704 spiked into an empty beaker, i.e., no wipe matrix)	LCSL LCSH	Monitor method performance in the absence of wipe matrix effects	One low and one high NIST SRM 2704 loading level per batch. The levels are equivalent to the single-wipe loadings.

5   Procedure

5.1   Record all reagent sources, lot numbers, and expiration dates used for sample preparation in the laboratory notebook. In addition, record any inadvertent deviations to this procedure, unusual happenings, or observations on a real-time basis as the samples are processed. If any deviation or unusual happenings occur, notify the Work Assignment Leader or Facility Manager in a manner that permits corrective action as close to real-time as possible.

5.2   Treat all samples (including QC samples) in a processing batch equally. For example, if one sample requires additional hydrogen peroxide, then *all* samples in that batch must receive the same volume of additional hydrogen peroxide.

5.3   Take care during the execution of each step of the following procedure to ensure that sample losses do not occur due to splattering or spillage and that samples are not contaminated or cross-contaminated.

5.4   Document the condition of the samples received in a laboratory notebook.

5.5   Label 250-mL Griffin beakers for each wipe sample and associated quality control sample to be processed.

5.6   Do not weigh the wipe samples. Open the sample container and carefully transfer the wipe to a labeled Griffin beaker, using a new pair of plastic gloves and\or plastic forceps.

5.7   Using a pipette or repipet dispenser, transfer 5 mL of Milli-Q reagent water to the empty sample container. Cap the bottle with the original cap, shake the bottle to remove any remaining dust residue from the inside surface of the bottle and cap. Open the bottle and transfer the Milli-Q rinse solution to the beaker.

5.8   Repeat step 5.7 two times to ensure a quantitative transfer of the dust sample to the digestion beaker.

5.9   Transfer all wipe samples in the batch to labeled beakers. A low-level and a high-level laboratory control sample are processed with each sample batch. The control samples are designated LCSL and LCSH, respectively. Using an analytical balance transfer 0.5 ± 0.05 g and 2.0 ± 0.1 g aliquots of NIST SRM 2704 to tared beakers designated for the LCSL and LCSH samples, respectively. Record the actual sample weight used.

        NOTE: NIST SRM 2704 must be dried according to the procedure listed on the Certificate of Analysis, and allowed to cool to room temperature in a desiccator prior to use.

5.10   Carefully, add 25 mL of 1:1 HNO_3 to each beaker, gently swirl to mix, and cover with a watch glass. Gently heat the sample to approximately 95°C and reflux for 10 to 15 min without boiling.

5.11   Allow the sample to cool.

5.12   Add 10 mL of concentrated HNO_3, replace the watch glass, and reflux for 30 min without boiling.

5.13   Repeat step 5.12 to ensure complete oxidation of the sample.

5.14   Remove each watch glass and allow the digests to evaporate to approximately 10 mL without boiling, while maintaining a covering of solution over the bottom of the beaker.

        NOTE: As the watch glasses are removed, rinse the condensate on the watch glass into the respective sample beaker with a minimum amount of Milli-Q reagent water. Place the rinsed watch glasses upside down on new clean food service towels or Kimwipes.

5.15   Following the cookdown step (5.14), replace the watch glass cover, and allow the sample to cool.

5.16   Add 5 mL of Milli-Q reagent water and 5 mL of 30% hydrogen peroxide (H_2O_2). Cover the beaker with a watch glass and return the covered beaker to the hot plate for warming and to start the peroxide reaction. Care must be taken to ensure that losses do not occur due to excessively vigorous effervescence. Heat until the effervescence subsides, then allow the digests to cool.

5.17   Continue to add 30% H_2O_2 in 1-mL aliquots with warming and cooling, as needed, until the effervescence is minimal or until the general sample appearance is unchanged. Do not add more than a total of 10 mL of 30% H_2O_2, even if effervescence is still observed.

5.18 Carefully remove the watch glass and continue heating the acid-peroxide digestate until the volume has been reduced to approximately 10 mL (see note in step 5.14).

5.19 Allow the digestate to cool; rinse the beaker walls and underside of the watch glass with Milli-Q reagent water into the beaker. Using a funnel, quantitatively transfer the digestate to a 100-mL volumetric flask. Dilute to volume with Milli-Q reagent water and mix thoroughly. Do not filter at this step.

5.20 Remove the particulates in the digestate by filtration, by centrifugation, or by allowing the sample to settle prior to analysis. A disposable syringe equipped with an Acrodisc filter can be used to filter a portion of the sample digest prior to analysis.

5.21 The diluted digest contains approximately 10% (v/v) HNO_3. The calibration standards used for analysis should be made in 10% (v/v) HNO_3 to match the sample digests.

Appendix C

Modified SW-846 Method 3050A Acid Digestion Procedure for Two-Wipe Composite Samples

Modified SW-846 Method 3050A Acid Digestion Procedure for Two-Wipe Composite Samples

1 Summary of Method

This method is an acid digestion procedure based on SW-846 Method 3050A. The published method was revised to accommodate composite dust samples containing two wipes. This method is used for the acid digestion of dust samples and associated quality control (QC) samples for analysis of lead. It should be noted that this procedure does not use hydrochloric acid for digestion. The entire composite wipe sample is digested in nitric acid (HNO_3) and hydrogen peroxide (H_2O_2). The digestates are diluted to a final volume of 200 mL following the final cookdown step. The sample digestate will contain approximately 10% (v/v) HNO_3 following digestion.

2 Apparatus and Materials

2.1   Beakers: 400-mL Griffin

2.2   Watch glasses appropriate for designated beakers

2.3   Forceps: polyethylene, or equivalent

2.4   Volumetric flasks with stoppers: 200-mL, Class A

2.5   Funnels: plastic or glass, sized to fit into the 200-mL volumetric flasks

2.6   Thermometers: red alcohol, range of $0°$ to $110°C$

2.7   Hot plates: capable of maintaining a temperature of approximately $95°C$

2.8   Centrifuge (optional)

2.9   Centrifuge tubes: polyethylene with screw caps, 50-mL capacity

2.10   Kimwipes, or equivalent

2.11   Syringes: plastic, 10-mL disposable with Luer-lok fittings

2.12   Gelman Acrodisc filters, female Luer lok, 0.45-µm pore size, or equivalent.

2.13   Filter paper: Whatman No. 541, or equivalent

2.14   Food service towels

2.15   Gloves: disposable, powderless, vinyl

2.16   Pipettes with disposable tips

2.17   Repipet reagent dispensers

2.18   Analytical balance: Capable of weighing to the nearest 0.0001 g

2.19   Drying oven: Capable of maintaining a temperature of ~ 110°C

2.20   Desiccator

3   Reagents/Standard Stock

3.1 Milli-Q reagent water: minimum resistance of 16.67 MΩ-cm, or equivalent

3.2 Concentrated nitric acid (HNO_3)—70% to 71%: Baker Instra-Analyzed grade, or equivalent

3.3 Hydrogen peroxide (H_2O_2)—30%: A.C.S. reagent grade

3.4 NIST SRM 2704—Buffalo River Sediment

4   Quality Control

Quality control (QC) samples are processed with each digestion batch. The QC samples to be included in each batch are summarized in Table C-1.

Table C-1. Quality Control Samples Prepared With Each Batch

Description	Abbre-viation	Purpose	Frequency
Method blank	MB	Demonstrate absence of laboratory contamination	One with each sample batch
Wipe Blanks	WB	Determine background levels in the wipe material	Incorporated into the batch as specified in the test design. One of each wipe type per batch.
Laboratory control sample (NIST SRM 2704 spiked into an empty beaker, i.e., no wipe matrix)	LCSL LCSH	Monitor method performance in the absence of wipe matrix effects	One low and one high NIST SRM 2704 loading level per batch. The levels are equivalent to the two-wipe composite loadings.

5 Procedures

5.1   Record all reagent sources, lot numbers, and expiration dates used for sample preparation in the laboratory notebook. In addition, record any inadvertent deviations to this procedure, unusual happenings, or observations on a real-time basis as the samples are processed. If any deviation or unusual happenings occur, notify the Work Assignment Leader or Facility Manager in a manner that permits corrective action as close to real-time as possible.

5.2   Treat all samples (including QC samples) in a processing batch equally. For example, if one sample requires additional hydrogen peroxide, then *all* samples in that batch must receive the same volume of additional hydrogen peroxide.

5.3   Take care during the execution of each step of the following procedure to ensure that sample losses do not occur due to splattering or spillage and that samples are not contaminated or cross-contaminated.

5.4   Document the condition of the samples received in a laboratory notebook.

5.5   Label 400-mL Griffin beakers for each composite wipe sample and associated quality control sample to be processed.

5.6   Do not weigh the composite wipe samples. Open the sample container and carefully transfer both wipes to a single labeled Griffin beaker, using a new pair of plastic gloves and\or plastic forceps.

5.7   Using a pipette or repipet dispenser, transfer 5 mL of Milli-Q reagent water to the empty sample container. Cap the bottle with the original cap, shake the bottle to remove any remaining dust residue from the inside surface of the bottle and cap. Open the bottle and transfer the Milli-Q rinse solution to the beaker.

5.8   Repeat step 5.7 two times to ensure a quantitative transfer of the dust sample to the digestion beaker.

5.9   Transfer all wipe samples in the batch to labeled beakers. A low-level and a high-level laboratory control sample are processed with each sample batch. The control samples are designated LCSL and LCSH, respectively. Using an analytical balance transfer 1.0 ± 0.05 g and 4.0 ± 0.1 g aliquots of NIST SRM 2704 to tared beakers designated for the LCSL and LCSH samples, respectively. Record the actual sample weight used.

NOTE: NIST SRM 2704 must be dried according to the procedure listed on the Certificate of Analysis, and allowed to cool to room temperature in a desiccator prior to use.

5.10   Carefully, add 50 mL of 1:1 HNO_3 to each beaker, gently swirl to mix, and cover with a watch glass. Gently heat the sample to approximately 95°C and reflux for 10 to 15 min without boiling.

5.11   Allow the sample to cool.

5.12   Add 20 mL of concentrated HNO_3, replace the watch glass, and reflux for 30 min without boiling.

5.13   Repeat step 5.12 to ensure complete oxidation of the sample.

5.14   Remove each watch glass and allow the digests to evaporate to approximately 20 mL without boiling, while maintaining a covering of solution over the bottom of the beaker.

   NOTE: As the watch glasses are removed, rinse the condensate on the watch glass into the respective sample beaker with a minimum amount of Milli-Q reagent water. Place the rinsed watch glasses upside down on new clean food service towels or Kimwipes.

5.15   Following the cookdown step (5.14), replace the watch glass cover, and allow the sample to cool.

5.16   Add 10 mL of Milli-Q reagent water and 10 mL of 30% hydrogen peroxide (H_2O_2). Cover the beaker with a watch glass and return the covered beaker to the hot plate for warming and to start the peroxide reaction. Care must be taken to ensure that losses do not occur due to excessively vigorous effervescence. Heat until the effervescence subsides, then allow the digests to cool.

5.17   Continue to add 30% H_2O_2 in 2-mL aliquots with warming and cooling, as needed, until the effervescence is minimal or until the general sample appearance is unchanged. Do not add more than a total of 20 mL of 30% H_2O_2, even if effervescence is still observed.

5.18   Carefully remove the watch glass and continue heating the acid-peroxide digestate until the volume has been reduced to approximately 20 mL (see note in step 5.14).

5.19   Allow the digestate to cool; rinse the beaker walls and underside of the watch glass with Milli-Q reagent water into the beaker. Using a funnel, quantitatively transfer the digestate to a 200-mL volumetric flask. Dilute to volume with Milli-Q reagent water and mix thoroughly. Do not filter at this step.

5.20   Remove the particulates in the digestate by filtration, by centrifugation, or by allowing the sample to settle prior to analysis. A disposable syringe equipped with an Acrodisc filter can be used to filter a portion of the sample digest prior to analysis.

5.21   The diluted digest contains approximately 10% (v/v) HNO_3. The calibration standards used for analysis should be made in 10% (v/v) HNO_3 to match the sample digests.

Modified SW-846 Method 3050A Acid Digestion Procedure for Four-Wipe Composite Samples

Modified SW-846 Method 3050A Acid Digestion Procedure for Four-Wipe Composite Samples

1   Summary of Method

This method is an acid digestion procedure based on SW-846 Method 3050A. The published method was revised to accommodate composite dust samples containing four wipes. This method is used for the acid digestion of dust samples and associated quality control (QC) samples for analysis of lead. It should be noted that this procedure does not use hydrochloric acid for digestion. The entire composite wipe sample is digested in nitric acid (HNO_3) and hydrogen peroxide (H_2O_2). The digestates are diluted to a final volume of 500 mL following the final cookdown step. The sample digestate will contain approximately 10% (v/v) HNO_3 following digestion.

2   Apparatus and Materials

2.1   Beakers: 800-mL Griffin

2.2   Watch glasses appropriate for designated beakers

2.3   Forceps: polyethylene, or equivalent

2.4   Volumetric flasks with stoppers: 500-mL, Class A

2.5   Funnels: plastic or glass, sized to fit into the 500-mL volumetric flasks

2.6   Thermometers: red alcohol, range of 0° to 110°C

2.7   Hot plates: capable of maintaining a temperature of approximately 95°C

2.8   Centrifuge (optional)

2.9   Centrifuge tubes: polyethylene with screw caps, 50-mL capacity

2.10   Kimwipes, or equivalent

2.11   Syringes: plastic, 10-mL disposable with Luer-lok fittings

2.12   Gelman Acrodisc filters, female Luer lok, 0.45-µm pore size, or equivalent

2.13   Filter paper: Whatman No. 541, or equivalent

2.14   Food service towels

2.15   Gloves: disposable, powderless, vinyl

2.16   Pipettes with disposable tips

2.17   Repipet reagent dispensers

2.18   Analytical balance: Capable of weighing to the nearest 0.0001 g

2.19   Drying oven: Capable of maintaining a temperature of ~ 110°C

2.20   Desiccator

3   Reagents/Standard Stock

3.1 Milli-Q reagent water: minimum resistance of 16.67 MΩ-cm, or equivalent

3.2 Concentrated nitric acid (HNO_3)—70% to 71%: Baker Instra-Analyzed grade, or equivalent

3.3 Hydrogen peroxide (H_2O_2)—30%: A.C.S. reagent grade

3.4 NIST SRM 2704—Buffalo River Sediment

4   Quality Control

Quality control (QC) samples are processed with each digestion batch. The QC samples to be included in each batch are summarized in Table D-1.

Table D-1. Quality Control Samples Prepared With Each Batch

Description	Abbr e- viatio n	Purpose	Frequency
Method blank	MB	Demonstrate absence of laboratory contamination	One with each sample batch
Wipe Blanks	WB	Determine background levels in the wipe material	Incorporated into the batch as specified in the test design. One of each wipe type per batch.
Laboratory control sample (NIST SRM 2704 spiked into an empty beaker, i.e., no wipe matrix)	LCSL LCSH	Monitor method performance in the absence of wipe matrix effects	One low and one high NIST SRM 2704 loading level per batch. The levels are equivalent to the four-wipe composite loadings.

5   Procedure

5.1   Record all reagent sources, lot numbers, and expiration dates used for sample preparation in the laboratory notebook. In addition, record any inadvertent deviations to this procedure, unusual happenings, or observations on a real-time basis as the samples are processed. If any deviation or unusual happenings occur, notify the Work Assignment Leader or Facility Manager in a manner that permits corrective action as close to real-time as possible.

5.2   Treat all samples (including QC samples) in a processing batch equally. For example, if one sample requires additional hydrogen peroxide, then *all* samples in that batch must receive the same volume of additional hydrogen peroxide.

5.3   Take care during the execution of each step of the following procedure to ensure that sample losses do not occur due to splattering or spillage and that samples are not contaminated or cross-contaminated.

5.4   Document the condition of the samples received in a laboratory notebook.

5.5   Label 800-mL Griffin beakers for each composite wipe sample and associated quality control sample to be processed.

5.6   Do not weigh the composite wipe samples. Open the sample container and carefully transfer the four wipes to a single labeled Griffin beaker, using a new pair of plastic gloves and\or plastic forceps.

5.7   Using a pipette or repipet dispenser, transfer 10 mL of Milli-Q reagent water to the empty sample container. Cap the bottle with the original cap, shake the bottle to remove any remaining dust residue from the inside surface of the bottle and cap. Open the bottle and transfer the Milli-Q rinse solution to the beaker.

5.8   Repeat step 5.7 two times to ensure a quantitative transfer of the dust sample to the digestion beaker.

5.9   Transfer all wipe samples in the batch to labeled beakers. A low-level and a high-level laboratory control sample are processed with each sample batch, which are designated LCSL and LCSH, respectively. Using an analytical balance transfer 2.0 ± 0.05 g and 8.0 ± 0.1 g aliquots of NIST SRM 2704 to tared beakers designated for the LCSL and LCSH samples, respectively. Record the actual sample weight used.

        NOTE: NIST SRM 2704 must be dried according to the procedure listed on the Certificate of Analysis, and allowed to cool to room temperature in a desiccator prior to use.

5.10   Carefully, add 100 mL of 1:1 HNO_3 to each beaker, gently swirl to mix, and cover with a watch glass. Gently heat the sample to approximately 95°C and reflux for 10 to 15 min without boiling.

5.11   Allow the sample to cool.

5.12   Add 40 mL of concentrated HNO_3, replace the watch glass, and reflux for 30 min without boiling.

5.13   Repeat step 5.12 to ensure complete oxidation of the sample.

5.14   Remove each watch glass and allow the digests to evaporate to approximately 50 mL without boiling, while maintaining a covering of solution over the bottom of the beaker.

        NOTE: As the watch glasses are removed, rinse the condensate on the watch glass into the respective sample beaker with a minimum amount of Milli-Q reagent water. Place the rinsed watch glasses upside down on new clean food service towels or Kimwipes.

5.15   Following the cookdown step (5.14), replace the watch glass cover, and allow the sample to cool.

5.16   Add 20 mL of Milli-Q reagent water and 20 mL of 30% hydrogen peroxide (H_2O_2). Cover the beaker with a watch glass and return the covered beaker to the hot plate for warming and to start the peroxide reaction. Care must be taken to ensure that losses do not occur due to excessively vigorous effervescence. Heat until the effervescence subsides, then allow the digests to cool.

5.17   Continue to add 30% H_2O_2 in 4-mL aliquots with warming and cooling, as needed, until the effervescence is minimal or until the general sample appearance is unchanged. Do not add more than a total of 40 mL of 30% H_2O_2, even if effervescence is still observed.

5.18   Carefully remove the watch glass and continue heating the acid-peroxide digestate until the volume has been reduced to approximately 50 mL (see note in step 5.14).

5.19   Allow the digestate to cool; rinse the beaker walls and underside of the watch glass with Milli-Q reagent water into the beaker. Using a funnel, quantitatively transfer the digestate to a 500-mL volumetric flask. Dilute to volume with Milli-Q reagent water and mix thoroughly. Do not filter at this step.

5.20   Remove particulates in the digestate by filtration, by centrifugation, or by allowing the sample to settle prior to analysis. A disposable syringe equipped with an Acrodisc filter can be used to filter a portion of the sample digest prior to analysis.

5.21   The diluted digest contains approximately 10% (v/v) HNO_3. The calibration standards used for analysis should be made in 10% (v/v) HNO_3 to match the sample digests.

Appendix E

WOHL Acid Digestion Procedure for Single-Wipe Samples

WOHL Acid Digestion Procedure for Single-Wipe Samples

1 Summary of Method

This digestion procedure is based on the method used by the Wisconsin Occupational Health Laboratory (WOHL) and Appendix 14.2 of the HUD *Guidelines* for digesting samples containing a single wipe. This method is used for the acid digestion of dust samples and associated quality control (QC) samples for analysis of lead. The wipe samples are digested in nitric acid (HNO_3) and hydrochloric acid (HCl). The digestates are diluted to a final volume of 50 mL following a 20- to 30-min reflux at 100°C and a cooling step. The sample digestates contain approximately 4% (v/v) HNO_3 and 4% (v/v) HCl.

2 Apparatus and Materials

2.1   Beakers: 125-mL, Phillips conical

2.2   Watch glasses appropriate for designated beakers

2.3   Scissors: stainless steel, or equivalent

2.4   Volumetric flasks with stoppers: 50-mL, Class A

2.5   Funnels: plastic or glass, sized to fit into the 50-mL volumetric flasks

2.6   Thermometers: red alcohol, range of 0° to 110°C

2.7   Stirring rods: glass or plastic

2.8   Hot plates: capable of maintaining a temperature of approximately 100°C

2.9   Centrifuge

2.10   Centrifuge tubes: polyethylene with screw caps, 50-mL capacity

2.11   Filter paper: Whatman No. 541, or equivalent

2.12   Kimwipes, or equivalent

2.13   Food service towels

2.14   Gloves: disposable, powderless, vinyl

2.15   Pipettes with disposable tips

2.16   Repipet reagent dispensers

2.17   Analytical balance: Capable of weighing to the nearest 0.0001 g

2.18   Drying oven: Capable of maintaining a temperature of ~ 110°C

2.19   Desiccator

3   Reagents/Standard Stock

3.1   Milli-Q reagent water: minimum resistance of 16.67 MΩ-cm, or equivalent

3.2   Concentrated nitric acid (HNO_3)—70% to 71%: Baker Instra-Analyzed grade, or equivalent

3.3   Hydrochloric acid (HCl)—36.5% to 38%: Baker Instra-Analyzed grade, or equivalent

3.4   NIST SRM 2704—Buffalo River Sediment

4   Quality Control

Quality control (QC) samples are processed with each digestion batch. The QC samples to be included in each batch are summarized in Table E-1.

Table E-1. Quality Control Samples Prepared With Each Batch

Description	Abbre-viation	Purpose	Frequency
Method blank	MB	Demonstrate absence of laboratory contamination	One with each sample batch
Wipe blanks	WB	Determine background levels in the wipe material	Incorporated into the batch as specified in the test design. One of each wipe type per batch.
Laboratory control sample (NIST SRM 2704 spiked into an empty beaker, i.e., no wipe matrix)	LCSL LCSH	Monitor method performance in the absence of sample matrix effects	One low and one high NIST SRM 2704 loading level per batch.

5   Procedure

5.1   Record all reagent sources, lot numbers, and expiration dates used for sample preparation in the laboratory notebook. In addition, record any inadvertent deviations to this procedure, unusual happenings, or observations on a real-time basis as the samples are processed. If any deviation or unusual happenings occur, notify the Work Assignment Leader or Facility Manager in a manner that permits corrective action as close to real-time as possible.

5.2   Treat all samples (including QC samples) in a processing batch equally. Care should be taken during the execution of each step of the following procedure to ensure the sample losses do not occur due to splattering or spillage and that samples are not contaminated or cross-contaminated.

5.3   Document the condition of the samples received in a laboratory notebook.

5.4   Label 125-mL Phillips conical beakers for each wipe sample and associated quality control sample to be processed.

5.5   Do not weigh the wipe samples. Open the sample container and remove the wipe using a new pair of plastic gloves and/or plastic forceps. Hold the wipe over the neck of a 125-mL Phillips conical beaker designated for the sample, and using scissors cut the wipe into smaller pieces collecting the wipe pieces in the labeled beaker.

5.6   Clean and dry the scissors between samples using Kimwipes and Milli-Q reagent water.

5.7   Quantitatively rinse the inside surfaces of the sample bottle and cap with Milli-Q water, transferring the rinses into the respective sample beaker.

5.8   Transfer all wipe samples in the batch to labeled beakers as described above. A low-level and a high-level laboratory control sample are processed with each sample batch. The control samples are designated LCSL and LCSH, respectively. Aliquots of NIST SRM 2704 are transferred to tared beakers designated for the LCSL and LCSH samples, respectively. The amount of NIST SRM used for the LCSL and LCSH are shown in the following table. Record the actual sample weight used.

Amount of NIST SRM 2704 spiked LCSL (g)	Amount of NIST SRM 2704 spiked LCSH (g)
0.5 ± 0.05	2.0 ± 0.1

NOTE: NIST SRM 2704 must be dried according to the procedure listed on the Certificate of Analysis, and allowed to cool to room temperature in a desiccator prior to use.

5.9   Add 10 mL of Milli-Q water to each sample covering the wipe.

5.10   Add 2 mL of concentrated HNO_3 and 2 mL of concentrated HCl to each beaker, gently swirl to mix, and cover with a watch glass. Gently heat the sample to approximately $100°C$ and reflux for 20 to 30 min.

5.11   Allow the digestates to cool; rinse the beaker walls and underside of the watch glass with Milli-Q reagent water into the beaker. Using a stirring rod, squeeze the wipe material, and then quantitatively transfer the acid digestate and the bulk material to a 50-mL volumetric flask.

5.12   To help ensure a quantitative transfer of the sample digest, if there is too much bulk material remaining in the beaker, rinse the material remaining with Milli-Q water, stir and squeeze the wipe material with the stirring rod, and transfer this rinsate to the 50-mL volumetric flask. Dilute to volume with Milli-Q reagent water and mix thoroughly. Do not filter at this step.

5.13   Filter a portion of the digest through a Whatman 541 filter paper. Then centrifuge this portion at 9,000 rpm for 20 min. The supernatant is drawn off and analyzed by AA, ICP, or other equivalent methodology.

5.14   The diluted digest contains approximately 4% (v/v) HNO_3 and 4% (v/v) HCl. The calibration standards used for analysis should be made in 4% (v/v) HNO_3 to match the sample digests.

Appendix F

WOHL Acid Digestion Procedure for Two- and Four-Wipe Composite Samples

WOHL Acid Digestion Procedure for Two- and Four-Wipe Composite Samples

1 Summary of Method

This digestion procedure is based on the method used by the Wisconsin Occupational Health Laboratory (WOHL) and Appendix 14.2 of the Hud *Guidelines* for digesting composite samples containing up to four wipes. This method is used for the acid digestion of dust samples and associated quality control (QC) samples for analysis of lead. The wipe composites are digested in nitric acid (HNO_3) and hydrochloric acid (HCl). The digestates are diluted to a final volume of 100 mL following a 5-min reflux at 100°C and cooling step. The sample digestates contain approximately 8% (v/v) HNO_3 and 8% (v/v) HCl.

2 Apparatus and Materials

2.1   Beakers: 250-mL, Phillips conical

2.2   Watch glasses appropriate for designated beakers

2.3   Scissors: stainless steel, or equivalent

2.4   Volumetric flasks with stoppers: 100-mL, Class A

2.5   Funnels: plastic or glass, sized to fit into the 100-mL volumetric flasks

2.6   Thermometers: red alcohol, range of 0° to 110°C

2.7   Stirring rods: glass or plastic

2.8   Hot plates: capable of maintaining a temperature of approximately 100°C

2.9   Centrifuge

2.10   Centrifuge tubes: polyethylene with screw caps, 50-mL capacity

2.11   Filter paper: Whatman No. 541, or equivalent

2.12   Kimwipes, or equivalent

2.13   Food service towels

2.14   Gloves: disposable, powderless, vinyl

2.15   Pipettes with disposable tips

2.16   Repipet reagent dispensers

2.17   Analytical balance: Capable of weighing to the nearest 0.0001 g

2.18   Drying oven: Capable of maintaining a temperature of ~ 110°C

2.19   Desiccator

3   Reagents/Standard Stock

3.1   Milli-Q reagent water: minimum resistance of 16.67 MΩ-cm, or equivalent

3.2   Concentrated nitric acid (HNO$_3$)—70% to 71%: Baker Instra-Analyzed grade, or
       equivalent

3.3   Hydrochloric acid (HCl)—36.5% to 38%: Baker Instra-Analyzed grade, or
       equivalent

3.4   NIST SRM 2704—Buffalo River Sediment

4   Quality Control

Quality control (QC) samples are processed with each digestion batch. The QC
samples to be included in each batch are summarized in Table F-1.

Table F-1. Quality Control Samples Prepared With Each Batch

Description	Abbreviation	Purpose	Frequency
Method blank	MB	Demonstrate absence of laboratory contamination	One with each sample batch
Wipe blanks	WB	Determine background levels in the wipe material	Incorporated into the batch as specified in the test design. One of each wipe type per batch.
Laboratory control sample (NIST SRM 2704 spiked into an empty beaker, i.e., no wipe matrix)	LCSL LCSH	Monitor method performance in the absence of sample matrix effects	One low and one high NIST SRM 2704 loading level per batch. The levels are equivalent to the two- and four-wipe loadings.

5 Procedure

5.1   Record all reagent sources, lot numbers, and expiration dates used for sample preparation in the laboratory notebook. In addition, also record any inadvertent deviations to this procedure, unusual happenings, or observations on a real-time basis as the samples are processed. If any deviation or unusual happenings occur, notify the Work Assignment Leader or Facility Manager in a manner that permits corrective action as close to real-time as possible.

5.2   Treat all samples (including QC samples) in a processing batch equally. Care should be taken during the execution of each step of the following procedure to ensure the sample losses do not occur due to splattering or spillage and that samples are not contaminated or cross-contaminated.

5.3   Document the condition of the samples received in a laboratory notebook.

5.4   Label 250-mL Phillips conical beakers for each wipe sample and associated quality control sample to be processed.

5.5   Do not weigh the wipe samples. Open the sample container and remove the wipe using a new pair of plastic gloves and/or plastic forceps. Hold the wipe over the neck of a 250-mL Phillips conical beaker designated for the sample, and using scissors cut the wipe into smaller pieces collecting the wipe pieces in the labeled beaker. Repeat this procedure for each wipe contained in the two- and four-wipe composite samples.

5.6   Clean and dry the scissors between samples using Kimwipes and Milli-Q reagent water.

5.7   Using a pipette or repipet dispenser, transfer 10 mL of Milli-Q reagent water to the empty sample container. Cap the bottle with the original cap, shake the bottle to remove any remaining dust residue from the inside surface of the bottle and cap. Open the bottle and transfer the Milli-Q rinse solution to the beaker.

5.8   Repeat step 5.7 two times to ensure a quantitative transfer of the dust sample to the digestion beaker. Then add an additional 10 mL of Milli-Q reagent water to each beaker.

5.9   Transfer all wipe samples in the batch to labeled beakers. A low-level and a high-level laboratory control sample are processed with each sample batch. The control samples are designated LCSL or LCSH, respectively. Aliquots of NIST SRM 2704 are transferred to tared beakers designated for the LCSL and LCSH samples, respectively. The amount of NIST SRM used for the LCSL and LCSH is shown in the following table. Record the actual sample weight used.

No. of wipes per sample for the batch	Amount of NIST SRM 2704 spiked LCSL (g)	Amount of NIST SRM 2704 spiked LCSH (g)
2	1.0 ± 0.05	4.0 ± 0.1
4	2.0 ± 0.05	8.0 ± 0.1

NOTE: NIST SRM 2704 must be dried according to the procedure listed on the Certificate of Analysis, and allowed to cool to room temperature in a desiccator prior to use.

5.10   Add 8 mL of concentrated HNO_3 and 8 mL of concentrated HCl to each beaker, gently swirl to mix, and cover with a watch glass. Gently heat the sample to approximately 100°C and reflux for 50 min.

5.11   Allow the digestates to cool; rinse the beaker walls and underside of the watch glass with Milli-Q reagent water into the beaker. Using a stirring rod, squeeze the wipe material, and then quantitatively transfer the acid digestate to a 100-mL volumetric flask.

5.12   To help ensure a quantitative transfer of the sample digest, add approximately 20 mL of Milli-Q reagent water to the wipe material remaining in the beaker, stir and squeeze the wipe material with the stirring rod, and transfer this rinsate to the 100-mL volumetric flask. Dilute to volume with Milli-Q reagent water and mix thoroughly. Do not filter at this step.

5.13   Filter a portion of the digest through a Whatman 541 filter paper. Then centrifuge this portion at 9,000 rpm for 20 min. The supernatant is drawn off and analyzed by AA, ICP, or other equivalent methodology.

5.14   The diluted digest contains approximately 8% (v/v) HNO_3 and 8% (v/v) HCl. The calibration standards used for analysis should be made in 8% (v/v) HNO_3 to match the sample digests.

Preparation of Reference Material Wipe Samples for the Composite Wipe Investigation

1 Summary of Method

This procedure is used for loading lead (Pb) using a standard reference material (SRM) onto wipes for the composite wipe study. The National Institute of Standards and Technology (NIST) SRM 2704 Buffalo River Sediment with a certified Pb concentration of 161 µg/g is used for this loading. Three types of wipes are used: (1) Wash a-bye Baby wipes, (2) Baby Wipes with Lanolin, and (3) Wash'n Dri wipes.

The wipes are loaded with NIST SRM 2704 at one of two levels, either 0.50 g or 2 g. The weighed portion of SRM is placed on the wipe, and the wipe is carefully folded and transferred to a labeled Nalgene bottle for storage until sample preparation.

2 Apparatus and Materials

2.1   Gloves: disposable, powderless, vinyl, or equivalent

2.2   Ziplock bags: quart and gallon size

2.3   Sample labels

2.4   NIST SRM 2704 Buffalo River Sediment

2.5   Wash a-bye Baby wipes

2.6   Baby Wipes with Lanolin

2.7   Wash'n Dri wipes

2.8   Analytical balance: capable of weighing to the nearest 0.0001 g

2.9   Weighing paper

2.10   Drying oven: capable of maintaining a temperature of ~ 110 °C

2.11   Beaker: appropriate size for drying NIST SRM 2704

2.12   Nalgene bottles: polyethylene, 125 mL and 250 mL

2.13   Scissors: stainless steel, or equivalent

2.14   Desiccator

3 Procedure

3.1   Transfer a sufficient amount of NIST SRM 2704 to a beaker for drying. The amount of NIST SRM 2704 required for a given weighing session is based on the number of wipes to be loaded and the loading level (0.50 g or 2 g) for each wipe. Place the beaker containing NIST SRM 2704 in a drying oven and dry the material for a minimum of 2 h at ~ 110 °C.

3.2   Remove the beaker from the drying oven, place the beaker in a desiccator, and allow the SRM to cool to room temperature.

3.3   Select the appropriate wipe material, as designated in the test design, which is targeted for loading with the reference material.

3.4   Using clean gloves and a pair of scissors, cut open a new Ziplock bag (quart or gallon size, whichever is appropriate based on the dimensions of the wipe). Open the bag exposing the inside surface, and place the bag inside surface up on the lab bench. Unfold a new wipe and place the wipe on the inside surface of the Ziplock bag located on the lab bench.

3.5   Remove the pre-dried portion of NIST SRM 2704 from the desiccator and weigh the designated aliquot, either 0.50 ± 0.05 g or 2.0 ± 0.1 g (as designated in the test design), of the pre-dried NIST SRM 2704 onto a clean piece of tared weighing paper. Record the weight of SRM used.

   NOTE: Return the beaker containing the pre-dried NIST SRM 2704 to the desiccator between each respective sample weighing.

3.6   With gloves on, carefully tap the weighed portion of the reference material from the weighing paper into the center of the wipe. Carefully fold the wipe in half several times, pressing the NIST SRM into the wipe while folding. Wipe off any residue remaining on the weighing paper using the folded wipe. Care should be taken to ensure that losses of the SRM do not occur during the folding process.

3.7   Transfer the folded wipe to a labeled Nalgene bottle according to the table below:

Number of wipes per sample	Nalgene bottle size
1	125 mL
2	250 mL
4	250 mL

Place each Nalgene bottle and the additional bar code labels in a Ziplock bag for secondary containment and seal the bag. **NOTE**: For multiwipe composites, the same Ziplock bag used for the first wipe in the composite (Step 3.4) should be reused for all wipes (2 or 4) contained in the composite.

3.8   When preparing a composite wipe sample of two or four wipes, repeat steps 3.3 through 3.7 for each wipe contained in the composite. It should be noted that for the two- and four-wipe composite samples, all wipes that comprise the composite sample should be transferred to the same labeled Nalgene bottle.

3.9   Record any unusual observations noted in the laboratory notebook.

3.10   Store the wipe samples loaded with the reference material at room temperature until sample digestion.

Laboratory Data and Lead (Pb) Recovery Results

Laboratory Data and Lead (Pb) Recovery Results

All laboratory data and recovery results for 528 samples are presented in this appendix. The data are organized in the following order:

- laboratory code (Nos. 1, 2, and 3)
- preparation method (EPA 3050A and WOHL)
- type of composite (one-, two-, and four-wipe composite)
- sample type (loaded wipe, QC sample type: laboratory control sample, method blank, wipe blank)
- total sample target (nominal) SRM loading (0.5 g to 8.0 g)
- wipe brand (A, B, or C)
- lot code (1 and 2 for brands A and B only)

Page breaks occur for each combination of laboratory, preparation method, and composite type. The data tables were generated in SAS with the following headers:

SAS variable name	Definition (unit)
OBS	Running observation number (1 through 528)
LAB_ID	Laboratory code
METHOD	Preparation method
COMPOSIT	Composite type
BRAND_W	Wipe brand
LOT	Wipe brand lot code
SPLETYPE	Sample type
BARCODE	Sample barcode ID
TOT_SRM	Target (nominal) SRM loading (g)
TOT_LAB	Actual SRM loading (g)
TOT_PB	Total lead (Pb) loading (μg)
ICP	ICP lead (Pb) result (μg)
ICP_Q	ICP qualifier (ND = nondetect, Q = quantified)
FAA	FAA lead (Pb) result (μg)
FAA_Q	FAA qualifier (ND = nondetect, Q = quantified)
ICP_REC	ICP recovery (%)
FAA_REC	FAA recovery (%)

The values at the bottom of each page are column totals corresponding to the variable(s) on the left-hand side of each page. The values are sequentially totaled across composite type, preparation method, and laboratory.

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LABORATORY DATA AND LEAD RECOVERY RESULTS 1

--- LAB_ID=1 METHOD=3050A COMPOSIT=1-WIPE ---

OBS	LAB ID	METHOD	COMPOSIT	BRAND	LOT	SPLE TYPE	BARCODE	TOT SRM	TOT LAB	TOT PB	ICP	ICP Q	FAA	FAA Q	ICP REC	FAA REC
1	1	3050A	1-WIPE	A	1	LOAD	906013	0.5	0.5006	80.60	73.8	Q	73.5	Q	91.57	91.19
2	1	3050A	1-WIPE	A	2	LOAD	906460	0.5	0.5032	81.02	78.0	Q	73.5	Q	96.28	90.72
3	1	3050A	1-WIPE	B	1	LOAD	906163	0.5	0.5059	81.45	79.5	Q	73.5	Q	97.61	90.24
4	1	3050A	1-WIPE	B	2	LOAD	906241	0.5	0.5054	81.37	72.6	Q	75.6	Q	89.22	92.91
5	1	3050A	1-WIPE	C	.	LOAD	906328	0.5	0.5122	82.46	80.4	Q	81.8	Q	97.50	99.19
6	1	3050A	1-WIPE	C	.	LOAD	906329	0.5	0.5088	81.92	76.7	Q	73.5	Q	93.63	89.73
7	1	3050A	1-WIPE	A	1	LOAD	906046	2.0	2.0075	323.21	263.0	Q	305.0	Q	81.37	94.37
8	1	3050A	1-WIPE	A	2	LOAD	906121	2.0	2.0082	323.32	271.0	Q	313.0	Q	83.82	96.81
9	1	3050A	1-WIPE	B	1	LOAD	906196	2.0	2.0079	323.27	264.0	Q	311.0	Q	81.67	96.20
10	1	3050A	1-WIPE	B	2	LOAD	906274	2.0	1.9833	319.31	266.0	Q	303.0	Q	83.30	94.89
11	1	3050A	1-WIPE	C	.	LOAD	906394	2.0	2.0351	327.65	270.0	Q	315.0	Q	82.40	96.14
12	1	3050A	1-WIPE	C	.	LOAD	906395	2.0	2.0128	324.06	262.0	Q	305.0	Q	80.85	94.12
13	1	3050A	1-WIPE	QC	.	QC-LCS	905710	0.5	0.5017	80.77	80.2	Q	75.6	Q	99.29	93.59
14	1	3050A	1-WIPE	QC	.	QC-LCS	905770	0.5	0.5015	80.74	80.6	Q	81.8	Q	99.82	101.31
15	1	3050A	1-WIPE	QC	.	QC-LCS	905711	2.0	2.0053	322.85	287.0	Q	328.0	Q	88.89	101.59
16	1	3050A	1-WIPE	QC	.	QC-LCS	905771	2.0	2.0004	322.06	286.0	Q	324.0	Q	88.80	100.60
17	1	3050A	1-WIPE	QC	.	QC-MB	905706	.	.	.	.	ND	.	ND	.	.
18	1	3050A	1-WIPE	QC	.	QC-MB	905769	.	.	.	.	ND	.	ND	.	.
19	1	3050A	1-WIPE	A	1	QC-WB	906001	.	.	.	.	ND	.	ND	.	.
20	1	3050A	1-WIPE	B	2	QC-WB	906229	.	.	.	.	ND	.	ND	.	.
21	1	3050A	1-WIPE	C	.	QC-WB	906307	.	.	.	.	ND	.	ND	.	.
COMPOSIT								20.0	20.0998	3236.07	2790.8		3112.8			

-- LAB_ID=1 METHOD=3050A COMPOSIT=2-WIPE --

OBS	LAB ID	METHOD	COMPOSIT	BRAND	LOT $\overline{W}$	SPLE TYPE	BARCODE	TOT $\overline{S}$RM	TOT $\overline{L}$AB	TOT $\overline{P}$B	ICP	ICP $\overline{Q}$	FAA	FAA $\overline{Q}$	ICP $\overline{R}$EC	FAA $\overline{R}$EC
22	1	3050A	2-WIPE	A	1	LOAD	906022	1.0	1.0098	162.58	158.0	Q	155.0	Q	97.18	95.34
23	1	3050A	2-WIPE	A	1	LOAD	906023	1.0	1.0110	162.77	151.0	Q	143.0	Q	92.77	87.85
24	1	3050A	2-WIPE	A	2	LOAD	906097	1.0	1.0141	163.27	157.0	Q	155.0	Q	96.16	94.93
25	1	3050A	2-WIPE	A	2	LOAD	906098	1.0	1.0106	162.71	150.0	Q	147.0	Q	92.19	90.35
26	1	3050A	2-WIPE	B	1	LOAD	906172	1.0	1.0320	166.15	156.0	Q	143.0	Q	93.89	86.07
27	1	3050A	2-WIPE	B	1	LOAD	906173	1.0	1.0224	164.61	159.0	Q	155.0	Q	96.59	94.16
28	1	3050A	2-WIPE	B	2	LOAD	906250	1.0	1.0155	163.50	160.0	Q	155.0	Q	97.86	94.80
29	1	3050A	2-WIPE	B	2	LOAD	906251	1.0	1.0197	164.17	152.0	Q	151.0	Q	92.59	91.98
30	1	3050A	2-WIPE	C	.	LOAD	906346	1.0	1.0418	167.73	156.0	Q	139.0	Q	93.01	82.87
31	1	3050A	2-WIPE	C	.	LOAD	906347	1.0	1.0064	162.03	170.0	Q	155.0	Q	104.92	95.66
32	1	3050A	2-WIPE	C	.	LOAD	906348	1.0	1.0114	162.84	154.0	Q	151.0	Q	94.57	92.73
33	1	3050A	2-WIPE	C	.	LOAD	906349	1.0	1.0428	167.89	152.0	Q	147.0	Q	90.54	87.56
34	1	3050A	2-WIPE	A	1	LOAD	906055	4.0	4.0082	645.32	551.0	Q	652.0	Q	85.38	101.04
35	1	3050A	2-WIPE	A	1	LOAD	906056	4.0	4.0671	654.80	554.0	Q	652.0	Q	84.61	99.57
36	1	3050A	2-WIPE	A	2	LOAD	906130	4.0	4.0605	653.74	577.0	Q	652.0	Q	88.26	99.73
37	1	3050A	2-WIPE	A	2	LOAD	906131	4.0	4.0363	649.84	555.0	Q	643.0	Q	85.41	98.95
38	1	3050A	2-WIPE	B	1	LOAD	906205	4.0	4.0169	646.72	562.0	Q	647.0	Q	86.90	100.04
39	1	3050A	2-WIPE	B	1	LOAD	906206	4.0	4.0193	647.11	541.0	Q	631.0	Q	83.60	97.51
40	1	3050A	2-WIPE	B	2	LOAD	906283	4.0	4.1336	665.51	552.0	Q	635.0	Q	82.94	95.42
41	1	3050A	2-WIPE	B	2	LOAD	906284	4.0	4.1156	662.61	568.0	Q	660.0	Q	85.72	99.61
42	1	3050A	2-WIPE	C	.	LOAD	906412	4.0	4.0204	647.28	578.0	Q	668.0	Q	89.30	103.20
43	1	3050A	2-WIPE	C	.	LOAD	906413	4.0	4.0008	644.13	560.0	Q	660.0	Q	86.94	102.46
44	1	3050A	2-WIPE	C	.	LOAD	906414	4.0	3.9977	643.63	577.0	Q	647.0	Q	89.65	100.52
45	1	3050A	2-WIPE	C	.	LOAD	906415	4.0	4.0205	647.30	566.0	Q	656.0	Q	87.44	101.34
46	1	3050A	2-WIPE	QC	.	QC-LCS	905712	1.0	1.0026	161.42	165.0	Q	160.0	Q	102.22	99.12
47	1	3050A	2-WIPE	QC	.	QC-LCS	905713	4.0	4.0008	644.13	563.0	Q	647.0	Q	87.40	100.45
48	1	3050A	2-WIPE	QC	.	QC-MB	905707	.	.	.	.	ND	.	ND	.	.
49	1	3050A	2-WIPE	A	2	QC-WB	906079	.	.	.	.	ND	.	ND	.	.
50	1	3050A	2-WIPE	B	1	QC-WB	906154	.	.	.	.	ND	.	ND	.	.
51	1	3050A	2-WIPE	C	.	QC-WB	906313	.	.	.	.	ND	.	ND	.	.
COMPOSIT								65.0	65.7378	10583.79	9344.0		10406			

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-- LAB_ID=1 METHOD=3050A COMPOSIT=4-WIPE --

OBS	LAB_ID	METHOD	COMPOSIT	BRAND_W	LOT	SPLE TYPE	BAR CODE	TOT_SRM	TOT_LAB	TOT_PB	ICP	IP_Q	FAA	FAA_Q	ICP_REC	FAA_REC
52	1	3050A	4-WIPE	A	1	LOAD	906034	2.0	2.0125	324.01	292.0	Q	253.0	Q	90.12	78.08
53	1	3050A	4-WIPE	A	1	LOAD	906043	2.0	2.0264	326.25	342.0	Q	305.0	Q	104.83	93.49
54	1	3050A	4-WIPE	A	2	LOAD	906109	2.0	2.0291	326.69	318.0	Q	294.0	Q	97.34	89.99
55	1	3050A	4-WIPE	A	2	LOAD	906118	2.0	2.0264	326.25	309.0	Q	284.0	Q	94.71	87.05
56	1	3050A	4-WIPE	B	1	LOAD	906184	2.0	2.0072	323.16	304.0	Q	284.0	Q	94.07	87.88
57	1	3050A	4-WIPE	B	1	LOAD	906193	2.0	2.0527	330.48	336.0	Q	284.0	Q	101.67	85.93
58	1	3050A	4-WIPE	B	2	LOAD	906262	2.0	1.9971	321.53	309.0	Q	263.0	Q	96.10	81.80
59	1	3050A	4-WIPE	B	2	LOAD	906271	2.0	2.0321	327.17	338.0	Q	294.0	Q	103.31	89.86
60	1	3050A	4-WIPE	C	.	LOAD	906370	2.0	2.0270	326.35	312.0	Q	284.0	Q	95.60	87.02
61	1	3050A	4-WIPE	C	.	LOAD	906371	2.0	2.0236	325.80	327.0	Q	315.0	Q	100.37	96.69
62	1	3050A	4-WIPE	C	.	LOAD	906388	2.0	2.0428	328.89	322.0	Q	284.0	Q	97.90	86.35
63	1	3050A	4-WIPE	C	.	LOAD	906389	2.0	2.0418	328.73	388.0	Q	357.0	Q	118.03	108.60
64	1	3050A	4-WIPE	A	1	LOAD	906067	8.0	8.0149	1290.40	1050.0	Q	1190.0	Q	81.37	92.22
65	1	3050A	4-WIPE	A	1	LOAD	906076	8.0	8.0444	1295.15	1160.0	Q	1300.0	Q	89.57	100.37
66	1	3050A	4-WIPE	A	2	LOAD	906142	8.0	8.0380	1294.12	1170.0	Q	1300.0	Q	90.41	100.45
67	1	3050A	4-WIPE	A	2	LOAD	906151	8.0	8.0403	1294.49	1200.0	Q	1320.0	Q	92.70	101.97
68	1	3050A	4-WIPE	B	1	LOAD	906217	8.0	8.0696	1299.21	1210.0	Q	1360.0	Q	93.13	104.68
69	1	3050A	4-WIPE	B	1	LOAD	906218	8.0	8.0072	1289.16	1160.0	Q	1270.0	Q	89.98	98.51
70	1	3050A	4-WIPE	B	2	LOAD	906301	8.0	8.0144	1290.32	1150.0	Q	1270.0	Q	89.13	98.43
71	1	3050A	4-WIPE	B	2	LOAD	906302	8.0	8.0794	1300.78	1170.0	Q	1300.0	Q	89.95	99.94
72	1	3050A	4-WIPE	C	.	LOAD	906436	8.0	8.1699	1315.35	1170.0	Q	1380.0	Q	88.95	104.91
73	1	3050A	4-WIPE	C	.	LOAD	906437	8.0	8.0875	1302.09	1160.0	Q	1330.0	Q	89.09	102.14
74	1	3050A	4-WIPE	C	.	LOAD	906454	8.0	8.0953	1303.34	1260.0	Q	1350.0	Q	96.67	103.58
75	1	3050A	4-WIPE	C	.	LOAD	906455	8.0	8.0274	1292.41	1230.0	Q	1350.0	Q	95.17	104.46
76	1	3050A	4-WIPE	QC	.	QC-LCS	905714	2.0	2.0017	322.27	304.0	Q	274.0	Q	94.33	85.02
77	1	3050A	4-WIPE	QC	.	QC-LCS	905715	2.0	2.0020	322.32	331.0	Q	274.0	Q	102.69	85.01
78	1	3050A	4-WIPE	QC	.	QC-LCS	905716	8.0	8.0103	1289.66	1200.0	Q	1320.0	Q	93.05	102.35
79	1	3050A	4-WIPE	QC	.	QC-LCS	905717	8.0	8.0031	1288.50	1200.0	Q	1300.0	Q	93.13	100.89
80	1	3050A	4-WIPE	QC	.	QC-MB	905708	.	.	.	.	ND	.	ND	.	.
81	1	3050A	4-WIPE	QC	.	QC-MB	905709	.	.	.	.	ND	.	ND	.	.
82	1	3050A	4-WIPE	A	1	QC-WB	906007	.	.	.	.	ND	.	ND	.	.
83	1	3050A	4-WIPE	A	2	QC-WB	906085	.	.	.	.	ND	.	ND	.	.
84	1	3050A	4-WIPE	B	1	QC-WB	906160	.	.	.	.	ND	.	ND	.	.
85	1	3050A	4-WIPE	B	2	QC-WB	906238	.	.	.	.	ND	.	ND	.	.
86	1	3050A	4-WIPE	C	.	QC-WB	906319	.	.	.	.	ND	.	ND	.	.
87	1	3050A	4-WIPE	C	.	QC-WB	906325	.	.	.	.	ND	.	ND	.	.
COMPOSIT								140.0	141.0241	22704.88	21022		22389			
METHOD								225.0	226.8617	36524.73	33157		35908			

-- LAB_ID=1 METHOD=WOHL COMPOSIT=1-WIPE --

OBS	LAB_ID	METHOD	COMPOSIT	BRAND	LOT	SPLE TYPE	BAR CODE	TOT_SRM	TOT_LAB	TOT_PB	ICP	ICP_Q	FAA	FAA_Q	ICP_REC	FAA_REC
88	1	WOHL	1-WIPE	A	1	LOAD	906016	0.5	0.5040	81.14	66.7	Q	73.3	Q	82.20	90.33
89	1	WOHL	1-WIPE	A	1	LOAD	906017	0.5	0.5063	81.51	61.4	Q	66.2	Q	75.32	81.21
90	1	WOHL	1-WIPE	A	2	LOAD	906091	0.5	0.5116	82.37	61.5	Q	68.3	Q	74.67	82.92
91	1	WOHL	1-WIPE	A	2	LOAD	906092	0.5	0.5059	81.45	61.0	Q	65.2	Q	74.89	80.05
92	1	WOHL	1-WIPE	B	1	LOAD	906166	0.5	0.5127	82.54	68.8	Q	76.4	Q	83.35	92.56
93	1	WOHL	1-WIPE	B	1	LOAD	906167	0.5	0.5129	82.58	69.4	Q	76.4	Q	84.04	92.52
94	1	WOHL	1-WIPE	B	2	LOAD	906244	0.5	0.5036	81.08	61.7	Q	66.2	Q	76.10	81.65
95	1	WOHL	1-WIPE	B	2	LOAD	906245	0.5	0.5090	81.95	67.5	Q	72.3	Q	82.37	88.23
96	1	WOHL	1-WIPE	C	.	LOAD	906334	0.5	0.5154	82.98	68.6	Q	73.3	Q	82.67	88.34
97	1	WOHL	1-WIPE	C	.	LOAD	906335	0.5	0.5158	83.04	76.6	Q	84.5	Q	92.24	101.75
98	1	WOHL	1-WIPE	C	.	LOAD	906336	0.5	0.5111	82.29	69.1	Q	74.3	Q	83.97	90.29
99	1	WOHL	1-WIPE	C	.	LOAD	906337	0.5	0.5258	84.65	70.6	Q	76.4	Q	83.40	90.25
100	1	WOHL	1-WIPE	A	1	LOAD	906049	2.0	2.0096	323.55	237.0	Q	255.0	Q	73.25	78.81
101	1	WOHL	1-WIPE	A	1	LOAD	906050	2.0	2.0035	322.56	242.0	Q	258.0	Q	75.02	79.98
102	1	WOHL	1-WIPE	A	2	LOAD	906125	2.0	2.0051	322.82	230.0	Q	244.0	Q	71.25	75.58
103	1	WOHL	1-WIPE	A	2	LOAD	906461	2.0	2.0118	323.90	233.0	Q	251.0	Q	71.94	77.49
104	1	WOHL	1-WIPE	B	1	LOAD	906199	2.0	2.0165	324.66	257.0	Q	270.0	Q	79.16	83.16
105	1	WOHL	1-WIPE	B	1	LOAD	906200	2.0	2.0061	322.98	250.0	Q	270.0	Q	77.40	83.60
106	1	WOHL	1-WIPE	B	2	LOAD	906278	2.0	2.0853	335.73	261.0	Q	287.0	Q	77.74	85.48
107	1	WOHL	1-WIPE	B	2	LOAD	906462	2.0	2.0129	324.08	295.0	Q	308.0	Q	91.03	95.04
108	1	WOHL	1-WIPE	C	.	LOAD	906400	2.0	2.0085	323.37	269.0	Q	287.0	Q	83.19	88.75
109	1	WOHL	1-WIPE	C	.	LOAD	906401	2.0	2.0147	324.37	259.0	Q	272.0	Q	79.85	83.86
110	1	WOHL	1-WIPE	C	.	LOAD	906402	2.0	2.0227	325.65	281.0	Q	295.0	Q	86.29	90.59
111	1	WOHL	1-WIPE	C	.	LOAD	906403	2.0	2.0032	322.52	257.0	Q	275.0	Q	79.69	85.27
112	1	WOHL	1-WIPE	QC	.	QC-LCS	905721	0.5	0.5018	80.79	70.9	Q	78.4	Q	87.76	97.04
113	1	WOHL	1-WIPE	QC	.	QC-LCS	905773	0.5	0.5010	80.66	71.2	Q	74.3	Q	88.27	92.11
114	1	WOHL	1-WIPE	QC	.	QC-LCS	905722	2.0	2.0028	322.45	302.0	Q	324.0	Q	93.66	100.48
115	1	WOHL	1-WIPE	QC	.	QC-LCS	905774	2.0	2.0067	323.08	306.0	Q	318.0	Q	94.71	98.43
116	1	WOHL	1-WIPE	QC	.	QC-MB	905718	.	.	.	.	ND	.	ND	.	.
117	1	WOHL	1-WIPE	QC	.	QC-MB	905772	.	.	.	.	ND	.	ND	.	.
118	1	WOHL	1-WIPE	A	1	QC-WB	906004	.	.	.	.	ND	.	ND	.	.
119	1	WOHL	1-WIPE	B	2	QC-WB	906232	.	.	.	.	ND	.	ND	.	.
120	1	WOHL	1-WIPE	C	.	QC-WB	906310	.	.	.	.	ND	.	ND	.	.
COMPOSIT								35.0	35.3463	5690.75	4624.0		4939.5			

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-- LAB_ID=1 METHOD=WOHL COMPOSIT=2-WIPE --

OBS	LAB_ID	METHOD	COMPOSIT	BRAND_W	LOT	SPLE TYPE	BARCODE	TOT_SRM	TOT_LAB	TOT_PB	ICP_Q	FAA_A	FAA_Q	ICP_REC	FAA_REC
121	1	WOHL	2-WIPE	A	1	LOAD	906028	1.0	1.0092	162.48	122.0	Q	136.0	Q	75.09	83.70
122	1	WOHL	2-WIPE	A	1	LOAD	906029	1.0	1.0133	163.14	119.0	Q	138.0	Q	72.94	84.59
123	1	WOHL	2-WIPE	A	2	LOAD	906103	1.0	1.0107	162.72	130.0	Q	150.0	Q	79.89	92.18
124	1	WOHL	2-WIPE	A	2	LOAD	906104	1.0	1.0131	163.11	127.0	Q	146.0	Q	77.86	89.51
125	1	WOHL	2-WIPE	B	1	LOAD	906178	1.0	1.0158	163.54	120.0	Q	134.0	Q	73.37	81.94
126	1	WOHL	2-WIPE	B	1	LOAD	906179	1.0	1.0122	162.96	145.0	Q	166.0	Q	88.98	101.86
127	1	WOHL	2-WIPE	B	2	LOAD	906256	1.0	1.0136	163.19	127.0	Q	144.0	Q	77.82	88.24
128	1	WOHL	2-WIPE	B	2	LOAD	906257	1.0	1.0109	162.75	131.0	Q	144.0	Q	80.49	88.48
129	1	WOHL	2-WIPE	C	.	LOAD	906358	1.0	1.0697	172.22	144.0	Q	158.0	Q	83.61	91.74
130	1	WOHL	2-WIPE	C	.	LOAD	906359	1.0	1.0157	163.53	141.0	Q	154.0	Q	86.22	94.17
131	1	WOHL	2-WIPE	C	.	LOAD	906360	1.0	1.0113	162.82	141.0	Q	152.0	Q	86.60	93.36
132	1	WOHL	2-WIPE	C	.	LOAD	906361	1.0	1.0079	162.27	132.0	Q	148.0	Q	81.34	91.20
133	1	WOHL	2-WIPE	A	1	LOAD	906061	4.0	4.0101	645.63	509.0	Q	556.0	Q	78.84	86.12
134	1	WOHL	2-WIPE	A	1	LOAD	906062	4.0	4.0342	649.51	522.0	Q	572.0	Q	80.37	88.07
135	1	WOHL	2-WIPE	A	2	LOAD	906136	4.0	4.0213	647.43	547.0	Q	592.0	Q	84.49	91.44
136	1	WOHL	2-WIPE	A	2	LOAD	906137	4.0	4.0104	645.67	538.0	Q	580.0	Q	83.32	89.83
137	1	WOHL	2-WIPE	B	1	LOAD	906211	4.0	4.0175	646.82	548.0	Q	594.0	Q	84.72	91.83
138	1	WOHL	2-WIPE	B	1	LOAD	906212	4.0	4.0258	648.15	540.0	Q	584.0	Q	83.31	90.10
139	1	WOHL	2-WIPE	B	2	LOAD	906289	4.0	4.0286	648.60	501.0	Q	538.0	Q	77.24	82.95
140	1	WOHL	2-WIPE	B	2	LOAD	906290	4.0	4.0222	647.57	505.0	Q	550.0	Q	77.98	84.93
141	1	WOHL	2-WIPE	C	.	LOAD	906424	4.0	4.0700	655.27	555.0	Q	588.0	Q	84.70	89.73
142	1	WOHL	2-WIPE	C	.	LOAD	906425	4.0	4.1911	674.77	589.0	Q	618.0	Q	87.29	91.59
143	1	WOHL	2-WIPE	C	.	LOAD	906426	4.0	4.0937	659.09	542.0	Q	592.0	Q	82.24	89.82
144	1	WOHL	2-WIPE	C	.	LOAD	906427	4.0	4.0301	648.85	546.0	Q	592.0	Q	84.15	91.24
145	1	WOHL	2-WIPE	QC	.	QC-LCS	905723	1.0	1.0008	161.13	144.0	Q	162.0	Q	89.37	100.54
146	1	WOHL	2-WIPE	QC	.	QC-LCS	905724	4.0	4.0330	649.31	612.0	Q	655.0	Q	94.25	100.88
147	1	WOHL	2-WIPE	QC	.	QC-MB	905719	.	.	.	.	ND	.	ND	.	.
148	1	WOHL	2-WIPE	A	2	QC-WB	906082	.	.	.	.	ND	.	ND	.	.
149	1	WOHL	2-WIPE	B	1	QC-WB	906157	.	.	.	.	ND	.	ND	.	.
150	1	WOHL	2-WIPE	C	.	QC-WB	906316	.	.	.	.	ND	.	ND	.	.

COMPOSIT   65.0   65.7922   10592.54   8777.0   9543.0

--------------------------------------- LAB_ID=1 METHOD=WOHL COMPOSIT=4-WIPE ---------------------------------------

OBS	LAB ID	METHOD	COMPOSIT	BRAND	LOT	SPL TYPE	BARCODE	TOT $\bar{S}$RM	TOT $\bar{L}$AB	TOT $\bar{P}$B	ICP	ICP $\bar{Q}$	FAA	FAA $\bar{Q}$	ICP $\bar{R}$EC	FAA $\bar{R}$EC
151	1	WOHL	4-WIPE	A	1	LOAD	906037	2.0	2.0209	325.36	214.0	Q	243.0	Q	65.77	74.69
152	1	WOHL	4-WIPE	A	1	LOAD	906038	2.0	2.0138	324.22	210.0	Q	243.0	Q	64.77	74.95
153	1	WOHL	4-WIPE	A	2	LOAD	906112	2.0	2.0188	325.03	207.0	Q	247.0	Q	63.69	75.99
154	1	WOHL	4-WIPE	A	2	LOAD	906113	2.0	2.0287	326.62	204.0	Q	239.0	Q	62.46	73.17
155	1	WOHL	4-WIPE	B	1	LOAD	906187	2.0	2.0310	326.99	216.0	Q	249.0	Q	66.06	76.15
156	1	WOHL	4-WIPE	B	1	LOAD	906188	2.0	2.0257	326.14	211.0	Q	249.0	Q	64.70	76.35
157	1	WOHL	4-WIPE	B	2	LOAD	906265	2.0	2.0192	325.09	211.0	Q	247.0	Q	64.90	75.98
158	1	WOHL	4-WIPE	B	2	LOAD	906266	2.0	2.0435	329.00	208.0	Q	243.0	Q	63.22	73.86
159	1	WOHL	4-WIPE	C	.	LOAD	906376	2.0	2.0818	335.17	247.0	Q	281.0	Q	73.69	83.84
160	1	WOHL	4-WIPE	C	.	LOAD	906377	2.0	2.0328	327.28	236.0	Q	275.0	Q	72.11	84.03
161	1	WOHL	4-WIPE	C	.	LOAD	906378	2.0	2.0598	331.63	239.0	Q	273.0	Q	72.07	82.32
162	1	WOHL	4-WIPE	C	.	LOAD	906379	2.0	2.0362	327.83	229.0	Q	261.0	Q	69.85	79.61
163	1	WOHL	4-WIPE	A	1	LOAD	906070	8.0	8.0217	1291.49	879.0	Q	978.0	Q	68.06	75.73
164	1	WOHL	4-WIPE	A	1	LOAD	906071	8.0	8.0433	1294.97	827.0	Q	922.0	Q	63.86	71.20
165	1	WOHL	4-WIPE	A	2	LOAD	906145	8.0	8.0253	1292.07	877.0	Q	966.0	Q	67.88	74.76
166	1	WOHL	4-WIPE	A	2	LOAD	906146	8.0	8.0455	1295.33	858.0	Q	958.0	Q	66.24	73.96
167	1	WOHL	4-WIPE	B	1	LOAD	906223	8.0	8.0515	1296.29	733.0	Q	809.0	Q	56.55	62.41
168	1	WOHL	4-WIPE	B	1	LOAD	906224	8.0	8.0435	1295.00	540.0	Q	598.0	Q	41.70	46.18
169	1	WOHL	4-WIPE	B	2	LOAD	906295	8.0	8.0716	1299.53	670.0	Q	740.0	Q	51.56	56.94
170	1	WOHL	4-WIPE	B	2	LOAD	906296	8.0	8.0296	1292.77	742.0	Q	813.0	Q	57.40	62.89
171	1	WOHL	4-WIPE	C	.	LOAD	906442	8.0	8.1496	1312.09	868.0	Q	966.0	Q	66.15	73.62
172	1	WOHL	4-WIPE	C	.	LOAD	906443	8.0	8.1349	1309.72	940.0	Q	1020.0	Q	71.77	77.88
173	1	WOHL	4-WIPE	C	.	LOAD	906444	8.0	8.1482	1311.86	981.0	Q	1090.0	Q	74.78	83.09
174	1	WOHL	4-WIPE	C	.	LOAD	906445	8.0	7.8996	1271.84	962.0	Q	1080.0	Q	75.64	84.92
175	1	WOHL	4-WIPE	QC	.	QC-LCS	905725	2.0	2.0179	324.88	273.0	Q	311.0	Q	84.03	95.73
176	1	WOHL	4-WIPE	QC	.	QC-LCS	905726	8.0	8.0165	1290.66	1160.0	Q	1290.0	Q	89.88	99.95
177	1	WOHL	4-WIPE	QC	.	QC-MB	905720	.	.	.	.	ND	.	ND	.	.
178	1	WOHL	4-WIPE	A	1	QC-WB	906010	.	.	.	.	ND	.	ND	.	.
179	1	WOHL	4-WIPE	B	2	QC-WB	906235	.	.	.	2.2	Q	.	ND	.	.
180	1	WOHL	4-WIPE	C	.	QC-WB	906322	.	.	.	4.7	Q	.	ND	.	.
COMPOSIT								130.0	131.1109	21108.85	13949		15591			
METHOD								230.0	232.2494	37392.15	27350		30074			
LAB_ID								455.0	459.1111	73916.89	60507		65981			

LABORATORY DATA AND LEAD RECOVERY RESULTS   7

-- LAB_ID=2 METHOD=3050A COMPOSIT=1-WIPE --

OBS	LAB_ID	METHOD	COMPOSIT	BRAND	LOT	SAMPLE TYPE	BARCODE	TOT SRM	TOT LAB	TOT PB	ICP	ICP_Q	FAA	FAA_Q	ICP REC	FAA REC
181	2	3050A	1-WIPE	A	1	LOAD	906014	0.5	0.5020	80.82	81.0	Q	78.0	Q	100.22	96.51
182	2	3050A	1-WIPE	A	2	LOAD	906089	0.5	0.5062	81.50	79.0	Q	78.0	Q	96.93	95.71
183	2	3050A	1-WIPE	B	1	LOAD	906164	0.5	0.5128	82.56	87.0	Q	82.0	Q	105.38	99.32
184	2	3050A	1-WIPE	B	2	LOAD	906242	0.5	0.5060	81.47	84.0	Q	81.0	Q	103.11	99.43
185	2	3050A	1-WIPE	C	.	LOAD	906330	0.5	0.5164	83.14	92.0	Q	92.0	Q	110.66	110.66
186	2	3050A	1-WIPE	C	.	LOAD	906331	0.5	0.5041	81.16	87.0	Q	87.0	Q	107.20	107.20
187	2	3050A	1-WIPE	A	1	LOAD	906047	2.0	2.0029	322.47	321.0	Q	320.0	Q	99.55	99.23
188	2	3050A	1-WIPE	A	2	LOAD	906122	2.0	2.0242	325.90	327.0	Q	333.0	Q	100.34	102.18
189	2	3050A	1-WIPE	B	1	LOAD	906197	2.0	2.0117	323.88	327.0	Q	329.0	Q	100.96	101.58
190	2	3050A	1-WIPE	B	2	LOAD	906275	2.0	2.0040	322.64	339.0	Q	341.0	Q	105.07	105.69
191	2	3050A	1-WIPE	C	.	LOAD	906396	2.0	2.0932	337.01	322.0	Q	324.0	Q	95.55	96.14
192	2	3050A	1-WIPE	C	.	LOAD	906397	2.0	1.9871	319.92	330.0	Q	331.0	Q	103.15	103.46
193	2	3050A	1-WIPE	QC	.	QC-LCS	905731	0.5	0.5046	81.24	89.0	Q	85.0	Q	109.55	104.63
194	2	3050A	1-WIPE	QC	.	QC-LCS	905732	2.0	2.0172	324.77	344.0	Q	330.0	Q	105.92	101.61
195	2	3050A	1-WIPE	QC	.	QC-MB	905727	.	.	.	4.0	Q	5.0	Q	.	.
196	2	3050A	1-WIPE	A	1	QC-WB	906002	.	.	.	4.0	Q	.	ND	.	.
197	2	3050A	1-WIPE	B	2	QC-WB	906230	.	.	.	4.0	Q	.	ND	.	.
198	2	3050A	1-WIPE	C	.	QC-WB	906308	.	.	.	5.0	Q	3.0	Q	.	.
COMPOSIT								17.5	17.6924	2848.48	2926.0		2899.0			

-- LAB_ID=2 METHOD=3050A COMPOSIT=2-WIPE --

OBS	LAB ID	METHOD	COMPOSIT	BRAND	LOT W̄	SPLE TYPE	BARCODE	TOT S̄RM	TOT L̄AB	TOT P̄B	ICP	ICP Q̄	FAA	FAA Q̄	ICP R̄EC	FAA R̄EC
199	2	3050A	2-WIPE	A	1	LOAD	906024	1.0	1.0098	162.58	168.0	Q	176.0	Q	103.34	108.26
200	2	3050A	2-WIPE	A	1	LOAD	906025	1.0	1.0035	161.56	162.0	Q	164.0	Q	100.27	101.51
201	2	3050A	2-WIPE	A	2	LOAD	906099	1.0	1.0103	162.66	168.0	Q	174.0	Q	103.28	106.97
202	2	3050A	2-WIPE	A	2	LOAD	906100	1.0	1.0080	162.29	172.0	Q	164.0	Q	105.98	101.05
203	2	3050A	2-WIPE	B	1	LOAD	906174	1.0	1.0185	163.98	170.0	Q	156.0	Q	103.67	95.13
204	2	3050A	2-WIPE	B	1	LOAD	906175	1.0	1.0065	162.05	168.0	Q	166.0	Q	103.67	102.44
205	2	3050A	2-WIPE	B	2	LOAD	906252	1.0	1.0176	163.83	162.0	Q	168.0	Q	98.88	102.54
206	2	3050A	2-WIPE	B	2	LOAD	906253	1.0	1.0172	163.77	168.0	Q	168.0	Q	102.58	102.58
207	2	3050A	2-WIPE	C	.	LOAD	906350	1.0	1.0097	162.56	174.0	Q	180.0	Q	107.04	110.73
208	2	3050A	2-WIPE	C	.	LOAD	906351	1.0	0.9844	158.49	180.0	Q	172.0	Q	113.57	108.53
209	2	3050A	2-WIPE	C	.	LOAD	906352	1.0	1.0049	161.79	170.0	Q	170.0	Q	105.08	105.08
210	2	3050A	2-WIPE	C	.	LOAD	906353	1.0	1.0160	163.58	172.0	Q	176.0	Q	105.15	107.60
211	2	3050A	2-WIPE	A	1	LOAD	906057	4.0	4.0116	645.87	658.0	Q	664.0	Q	101.88	102.81
212	2	3050A	2-WIPE	A	1	LOAD	906058	4.0	4.0004	644.06	662.0	Q	646.0	Q	102.78	100.30
213	2	3050A	2-WIPE	A	2	LOAD	906132	4.0	4.0246	647.96	660.0	Q	666.0	Q	101.86	102.78
214	2	3050A	2-WIPE	A	2	LOAD	906133	4.0	4.0202	647.25	658.0	Q	646.0	Q	101.66	99.81
215	2	3050A	2-WIPE	B	1	LOAD	906207	4.0	4.0216	647.48	650.0	Q	656.0	Q	100.39	101.32
216	2	3050A	2-WIPE	B	1	LOAD	906208	4.0	4.0174	646.80	666.0	Q	648.0	Q	102.97	100.19
217	2	3050A	2-WIPE	B	2	LOAD	906285	4.0	4.0337	649.43	662.0	Q	654.0	Q	101.94	100.70
218	2	3050A	2-WIPE	B	2	LOAD	906286	4.0	4.0138	646.22	660.0	Q	646.0	Q	102.13	99.97
219	2	3050A	2-WIPE	C	.	LOAD	906416	4.0	4.0263	648.23	670.0	Q	634.0	Q	103.36	97.80
220	2	3050A	2-WIPE	C	.	LOAD	906417	4.0	4.0460	651.41	664.0	Q	634.0	Q	101.93	97.33
221	2	3050A	2-WIPE	C	.	LOAD	906418	4.0	4.0393	650.33	684.0	Q	672.0	Q	105.18	103.33
222	2	3050A	2-WIPE	C	.	LOAD	906419	4.0	4.0212	647.41	684.0	Q	644.0	Q	105.65	99.47
223	2	3050A	2-WIPE	QC	.	QC-LCS	905733	1.0	1.0025	161.40	168.0	Q	158.0	Q	104.09	97.89
224	2	3050A	2-WIPE	QC	.	QC-LCS	905734	4.0	4.0050	644.81	660.0	Q	640.0	Q	102.36	99.25
225	2	3050A	2-WIPE	QC	.	QC-MB	905728	.	.	.	10.0	Q	4.0	Q	.	.
226	2	3050A	2-WIPE	A	2	QC-WB	906080	.	.	.	8.0	Q	.	ND	.	.
227	2	3050A	2-WIPE	B	1	QC-WB	906155	.	.	.	8.0	Q	.	ND	.	.
228	2	3050A	2-WIPE	C	.	QC-WB	906314	.	.	.	10.0	Q	12.0	Q	.	.
COMPOSIT								65.0	65.3900	10527.79	10876		10658			

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-- LAB_ID=2 METHOD=3050A COMPOSIT=4-WIPE --

OBS	LAB ID	METHOD	COMPOSIT	BRAND	LOT	SPLE TYPE	BARCODE	TOT SRM	TOT LAB	TOT PB	ICP	ICP Q	FAA	FAA Q	ICP REC	FAA REC
229	2	3050A	4-WIPE	A	1	LOAD	906035	2.0	2.0116	323.87	330.0	Q	315.0	Q	101.89	97.26
230	2	3050A	4-WIPE	A	1	LOAD	906044	2.0	2.0183	324.95	335.0	Q	295.0	Q	103.09	90.78
231	2	3050A	4-WIPE	A	2	LOAD	906110	2.0	2.0205	325.30	340.0	Q	335.0	Q	104.52	102.98
232	2	3050A	4-WIPE	A	2	LOAD	906119	2.0	2.0546	330.79	345.0	Q	335.0	Q	104.30	101.27
233	2	3050A	4-WIPE	B	1	LOAD	906185	2.0	2.0295	326.75	330.0	Q	310.0	Q	100.99	94.87
234	2	3050A	4-WIPE	B	1	LOAD	906194	2.0	2.0357	327.75	335.0	Q	345.0	Q	102.21	105.26
235	2	3050A	4-WIPE	B	2	LOAD	906263	2.0	2.0372	327.99	340.0	Q	300.0	Q	103.66	91.47
236	2	3050A	4-WIPE	B	2	LOAD	906272	2.0	2.0475	329.65	350.0	Q	345.0	Q	106.17	104.66
237	2	3050A	4-WIPE	C	.	LOAD	906372	2.0	2.0472	329.60	370.0	Q	350.0	Q	112.26	106.19
238	2	3050A	4-WIPE	C	.	LOAD	906373	2.0	2.0297	326.78	350.0	Q	325.0	Q	107.11	99.45
239	2	3050A	4-WIPE	C	.	LOAD	906390	2.0	2.0391	328.30	350.0	Q	365.0	Q	106.61	111.18
240	2	3050A	4-WIPE	C	.	LOAD	906391	2.0	2.0347	327.59	345.0	Q	355.0	Q	105.32	108.37
241	2	3050A	4-WIPE	A	1	LOAD	906068	8.0	8.0384	1294.18	1360.0	Q	1275.0	Q	105.09	98.52
242	2	3050A	4-WIPE	A	1	LOAD	906077	8.0	8.0199	1291.20	1440.0	Q	1385.0	Q	111.52	107.26
243	2	3050A	4-WIPE	A	2	LOAD	906143	8.0	8.0277	1292.46	1375.0	Q	1330.0	Q	106.39	102.90
244	2	3050A	4-WIPE	A	2	LOAD	906152	8.0	8.0313	1293.04	1380.0	Q	1305.0	Q	106.73	100.93
245	2	3050A	4-WIPE	B	1	LOAD	906219	8.0	8.0425	1294.84	1370.0	Q	1320.0	Q	105.80	101.94
246	2	3050A	4-WIPE	B	1	LOAD	906220	8.0	8.0591	1297.52	1325.0	Q	1260.0	Q	102.12	97.11
247	2	3050A	4-WIPE	B	2	LOAD	906303	8.0	8.0280	1292.51	1365.0	Q	1290.0	Q	105.61	99.81
248	2	3050A	4-WIPE	B	2	LOAD	906304	8.0	8.0571	1297.19	1365.0	Q	1315.0	Q	105.23	101.37
249	2	3050A	4-WIPE	C	.	LOAD	906438	8.0	8.0614	1297.89	1380.0	Q	1275.0	Q	106.33	98.24
250	2	3050A	4-WIPE	C	.	LOAD	906439	8.0	8.0372	1293.99	1385.0	Q	1295.0	Q	107.03	100.08
251	2	3050A	4-WIPE	C	.	LOAD	906456	8.0	8.0829	1301.35	1405.0	Q	1360.0	Q	107.97	104.51
252	2	3050A	4-WIPE	C	.	LOAD	906457	8.0	8.1545	1312.87	1425.0	Q	1360.0	Q	108.54	103.59
253	2	3050A	4-WIPE	QC	.	QC-LCS	905735	2.0	2.0003	322.05	340.0	Q	295.0	Q	105.57	91.60
254	2	3050A	4-WIPE	QC	.	QC-LCS	905736	2.0	2.0068	323.09	340.0	Q	320.0	Q	105.23	99.04
255	2	3050A	4-WIPE	QC	.	QC-LCS	905737	8.0	8.0008	1288.13	1370.0	Q	1265.0	Q	106.36	98.20
256	2	3050A	4-WIPE	QC	.	QC-LCS	905738	8.0	8.0077	1289.24	1370.0	Q	1285.0	Q	106.26	99.67
257	2	3050A	4-WIPE	QC	.	QC-MB	905729	.	.	.	20.0	Q	5.0	Q	.	.
258	2	3050A	4-WIPE	QC	.	QC-MB	905730	.	.	.	20.0	Q	.	ND	.	.
259	2	3050A	4-WIPE	A	1	QC-WB	906008	.	.	.	20.0	Q	15.0	Q	.	.
260	2	3050A	4-WIPE	A	2	QC-WB	906086	.	.	.	20.0	Q	.	ND	.	.
261	2	3050A	4-WIPE	B	1	QC-WB	906161	.	.	.	15.0	Q	15.0	Q	.	.
262	2	3050A	4-WIPE	B	2	QC-WB	906239	.	.	.	20.0	Q	.	ND	.	.
263	2	3050A	4-WIPE	C	.	QC-WB	906320	.	.	.	20.0	Q	.	ND	.	.
264	2	3050A	4-WIPE	C	.	QC-WB	906326	.	.	.	20.0	Q	10.0	Q	.	.

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COMPOSIT                                            140.0    141.0612   22710.85   24270      22955
METHOD                                              222.5    224.1436   36087.12   38072      36512
```

--- LAB_ID=2 METHOD=WOHL COMPOSIT=1-WIPE ---

OBS	LAB_ID	METHOD	COMPOSIT	BRAND	LOT (W̄)	SPLE TYPE	BARCODE	TOT S̄RM	TOT L̄AB	TOT P̄B	ICP	ICP Q̄	FAA	FAA Q̄	ICP R̄EC	FAA R̄EC
265	2	WOHL	1-WIPE	A	1	LOAD	906018	0.5	0.5007	80.61	77.0	Q	70.0	Q	95.52	86.83
266	2	WOHL	1-WIPE	A	1	LOAD	906019	0.5	0.5026	80.92	82.0	Q	73.0	Q	101.34	90.21
267	2	WOHL	1-WIPE	A	2	LOAD	906093	0.5	0.5027	80.93	78.0	Q	73.0	Q	96.37	90.20
268	2	WOHL	1-WIPE	A	2	LOAD	906094	0.5	0.5059	81.45	84.0	Q	74.0	Q	103.13	90.85
269	2	WOHL	1-WIPE	B	1	LOAD	906168	0.5	0.5061	81.48	81.0	Q	77.0	Q	99.41	94.50
270	2	WOHL	1-WIPE	B	1	LOAD	906169	0.5	0.5109	82.25	84.0	Q	80.0	Q	102.12	97.26
271	2	WOHL	1-WIPE	B	2	LOAD	906246	0.5	0.5062	81.50	82.0	Q	75.0	Q	100.62	92.03
272	2	WOHL	1-WIPE	B	2	LOAD	906247	0.5	0.5057	81.42	82.0	Q	74.0	Q	100.72	90.89
273	2	WOHL	1-WIPE	C	.	LOAD	906338	0.5	0.5289	85.15	89.0	Q	77.0	Q	104.52	90.43
274	2	WOHL	1-WIPE	C	.	LOAD	906339	0.5	0.5192	83.59	84.0	Q	75.0	Q	100.49	89.72
275	2	WOHL	1-WIPE	C	.	LOAD	906340	0.5	0.5210	83.88	90.0	Q	78.0	Q	107.29	92.99
276	2	WOHL	1-WIPE	C	.	LOAD	906341	0.5	0.5088	81.92	90.0	Q	80.0	Q	109.87	97.66
277	2	WOHL	1-WIPE	A	1	LOAD	906051	2.0	2.0024	322.39	289.0	Q	287.0	Q	89.64	89.02
278	2	WOHL	1-WIPE	A	1	LOAD	906052	2.0	2.0103	323.66	276.0	Q	281.0	Q	85.28	86.82
279	2	WOHL	1-WIPE	A	2	LOAD	906126	2.0	2.0097	323.56	290.0	Q	292.0	Q	89.63	90.25
280	2	WOHL	1-WIPE	A	2	LOAD	906127	2.0	2.0156	324.51	281.0	Q	287.0	Q	86.59	88.44
281	2	WOHL	1-WIPE	B	1	LOAD	906201	2.0	2.0085	323.37	303.0	Q	307.0	Q	93.70	94.94
282	2	WOHL	1-WIPE	B	1	LOAD	906202	2.0	2.0090	323.45	294.0	Q	295.0	Q	90.90	91.20
283	2	WOHL	1-WIPE	B	2	LOAD	906279	2.0	1.9756	318.07	268.0	Q	268.0	Q	84.26	84.26
284	2	WOHL	1-WIPE	B	2	LOAD	906280	2.0	2.0132	324.13	298.0	Q	297.0	Q	91.94	91.63
285	2	WOHL	1-WIPE	C	.	LOAD	906404	2.0	2.0409	328.58	295.0	Q	295.0	Q	89.78	89.78
286	2	WOHL	1-WIPE	C	.	LOAD	906405	2.0	2.0413	328.65	309.0	Q	314.0	Q	94.02	95.54
287	2	WOHL	1-WIPE	C	.	LOAD	906406	2.0	2.0260	326.19	305.0	Q	303.0	Q	93.50	92.89
288	2	WOHL	1-WIPE	C	.	LOAD	906407	2.0	2.0093	323.50	304.0	Q	303.0	Q	93.97	93.66
289	2	WOHL	1-WIPE	QC	.	QC-LCS	905742	0.5	0.5001	80.52	88.0	Q	75.0	Q	109.29	93.15
290	2	WOHL	1-WIPE	QC	.	QC-LCS	905743	2.0	2.0005	322.08	308.0	Q	311.0	Q	95.63	96.56
291	2	WOHL	1-WIPE	QC	.	QC-MB	905739	.	.	.	2.0	Q	.	ND	.	.
292	2	WOHL	1-WIPE	A	1	QC-WB	906005	.	.	.	1.0	Q	.	ND	.	.
293	2	WOHL	1-WIPE	B	2	QC-WB	906233	.	.	.	1.0	Q	2.0	Q	.	.
294	2	WOHL	1-WIPE	C	.	QC-WB	906311	.	.	.	2.0	Q	2.0	Q	.	.

COMPOSIT 32.5 32.7811 5277.76 4917.0 4825.0

LABORATORY DATA AND LEAD RECOVERY RESULTS

------------------------------------- LAB_ID=2 METHOD=WOHL COMPOSIT=2-WIPE -------------------------------------

OBS	LAB ID	METHOD	COMPOSIT	BRAND W	LOT	SPLE TYPE	BARCODE	TOT S̄RM	TOT L̄AB	TOT P̄B	ICP	ICP Q̄	FAA	FAA Q̄	ICP R̄EC	FAA R̄EC
295	2	WOHL	2-WIPE	A	1	LOAD	906030	1.0	1.0000	161.00	139.0	Q	159.0	Q	86.34	98.76
296	2	WOHL	2-WIPE	A	1	LOAD	906031	1.0	1.0042	161.68	138.0	Q	159.0	Q	85.36	98.34
297	2	WOHL	2-WIPE	A	2	LOAD	906105	1.0	1.0038	161.61	149.0	Q	173.0	Q	92.20	107.05
298	2	WOHL	2-WIPE	A	2	LOAD	906106	1.0	1.0135	163.17	138.0	Q	165.0	Q	84.57	101.12
299	2	WOHL	2-WIPE	B	1	LOAD	906180	1.0	1.0209	164.36	134.0	Q	159.0	Q	81.53	96.74
300	2	WOHL	2-WIPE	B	1	LOAD	906181	1.0	1.0072	162.16	152.0	Q	181.0	Q	93.74	111.62
301	2	WOHL	2-WIPE	B	2	LOAD	906258	1.0	1.0091	162.47	144.0	Q	165.0	Q	88.63	101.56
302	2	WOHL	2-WIPE	B	2	LOAD	906259	1.0	1.0118	162.90	141.0	Q	175.0	Q	86.56	107.43
303	2	WOHL	2-WIPE	C	.	LOAD	906362	1.0	1.0507	169.16	159.0	Q	184.0	Q	93.99	108.77
304	2	WOHL	2-WIPE	C	.	LOAD	906363	1.0	1.0190	164.06	151.0	Q	177.0	Q	92.04	107.89
305	2	WOHL	2-WIPE	C	.	LOAD	906364	1.0	1.0142	163.29	152.0	Q	178.0	Q	93.09	109.01
306	2	WOHL	2-WIPE	C	.	LOAD	906365	1.0	1.0172	163.77	150.0	Q	181.0	Q	91.59	110.52
307	2	WOHL	2-WIPE	A	1	LOAD	906063	4.0	4.0091	645.47	551.0	Q	563.0	Q	85.36	87.22
308	2	WOHL	2-WIPE	A	1	LOAD	906064	4.0	4.0189	647.04	597.0	Q	592.0	Q	92.27	91.49
309	2	WOHL	2-WIPE	A	2	LOAD	906138	4.0	4.0118	645.90	576.0	Q	584.0	Q	89.18	90.42
310	2	WOHL	2-WIPE	A	2	LOAD	906139	4.0	4.0153	646.46	578.0	Q	583.0	Q	89.41	90.18
311	2	WOHL	2-WIPE	B	1	LOAD	906213	4.0	4.0131	646.11	576.0	Q	598.0	Q	89.15	92.55
312	2	WOHL	2-WIPE	B	1	LOAD	906214	4.0	4.0317	649.10	591.0	Q	604.0	Q	91.05	93.05
313	2	WOHL	2-WIPE	B	2	LOAD	906291	4.0	4.0265	648.27	542.0	Q	560.0	Q	83.61	86.38
314	2	WOHL	2-WIPE	B	2	LOAD	906292	4.0	4.0227	647.65	596.0	Q	618.0	Q	92.02	95.42
315	2	WOHL	2-WIPE	C	.	LOAD	906428	4.0	4.0208	647.35	631.0	Q	632.0	Q	97.47	97.63
316	2	WOHL	2-WIPE	C	.	LOAD	906429	4.0	4.0334	649.38	613.0	Q	619.0	Q	94.40	95.32
317	2	WOHL	2-WIPE	C	.	LOAD	906430	4.0	4.0067	645.08	588.0	Q	602.0	Q	91.15	93.32
318	2	WOHL	2-WIPE	C	.	LOAD	906431	4.0	4.0165	646.66	597.0	Q	599.0	Q	92.32	92.63
319	2	WOHL	2-WIPE	QC	.	QC-LCS	905744	1.0	1.0006	161.10	178.0	Q	147.0	Q	110.49	91.25
320	2	WOHL	2-WIPE	QC	.	QC-LCS	905745	4.0	4.0010	644.16	625.0	Q	604.0	Q	97.03	93.77
321	2	WOHL	2-WIPE	QC	.	QC-MB	905740	.	.	.	3.0	Q	3.0	Q	.	.
322	2	WOHL	2-WIPE	A	2	QC-WB	906083	.	.	.	.	ND	3.0	Q	.	.
323	2	WOHL	2-WIPE	B	1	QC-WB	906158	.	.	.	1.0	Q	2.0	Q	.	.
324	2	WOHL	2-WIPE	C	.	QC-WB	906317	.	.	.	1.0	Q	4.0	Q	.	.
COMPOSIT								65.0	65.3997	10529.35	9591.0		9973.0			

-- LAB_ID=2 METHOD=WOHL COMPOSIT=4-WIPE --

OBS	LAB ID	METHOD	COMPOSIT	BRAND	LOT W̄T	SAMPLE TYPE	BARCODE	TOT S̄RM	TOT L̄AB	TOT P̄B	ICP	ICP Q̄	FAA Ā A	FAA Q̄	ICP R̄EC	FAA R̄EC
325	2	WOHL	4-WIPE	A	1	LOAD	906039	2.0	2.0156	324.51	324.0	Q	292.0	Q	99.84	89.98
326	2	WOHL	4-WIPE	A	1	LOAD	906040	2.0	2.0196	325.16	322.0	Q	292.0	Q	99.03	89.80
327	2	WOHL	4-WIPE	A	2	LOAD	906114	2.0	2.0195	325.14	332.0	Q	313.0	Q	102.11	96.27
328	2	WOHL	4-WIPE	A	2	LOAD	906115	2.0	2.0207	325.33	319.0	Q	283.0	Q	98.05	86.99
329	2	WOHL	4-WIPE	B	1	LOAD	906189	2.0	2.0287	326.62	341.0	Q	302.0	Q	104.40	92.46
330	2	WOHL	4-WIPE	B	1	LOAD	906190	2.0	2.0325	327.23	338.0	Q	300.0	Q	103.29	91.68
331	2	WOHL	4-WIPE	B	2	LOAD	906267	2.0	2.0264	326.25	330.0	Q	290.0	Q	101.15	88.89
332	2	WOHL	4-WIPE	B	2	LOAD	906268	2.0	2.0379	328.10	339.0	Q	299.0	Q	103.32	91.13
333	2	WOHL	4-WIPE	C	.	LOAD	906380	2.0	1.9903	320.44	355.0	Q	307.0	Q	110.79	95.81
334	2	WOHL	4-WIPE	C	.	LOAD	906381	2.0	2.0405	328.52	350.0	Q	307.0	Q	106.54	93.45
335	2	WOHL	4-WIPE	C	.	LOAD	906382	2.0	2.0341	327.49	349.0	Q	309.0	Q	106.57	94.35
336	2	WOHL	4-WIPE	C	.	LOAD	906383	2.0	2.0435	329.00	344.0	Q	309.0	Q	104.56	93.92
337	2	WOHL	4-WIPE	A	1	LOAD	906072	8.0	8.0188	1291.03	942.0	Q	1186.0	Q	72.97	91.86
338	2	WOHL	4-WIPE	A	1	LOAD	906073	8.0	8.0131	1290.11	967.0	Q	1225.0	Q	74.95	94.95
339	2	WOHL	4-WIPE	A	2	LOAD	906147	8.0	8.0598	1297.63	946.0	Q	1196.0	Q	72.90	92.17
340	2	WOHL	4-WIPE	A	2	LOAD	906148	8.0	8.0361	1293.81	946.0	Q	1200.0	Q	73.12	92.75
341	2	WOHL	4-WIPE	B	1	LOAD	906225	8.0	8.0392	1294.31	988.0	Q	1151.0	Q	76.33	88.93
342	2	WOHL	4-WIPE	B	1	LOAD	906226	8.0	8.0870	1302.01	990.0	Q	1158.0	Q	76.04	88.94
343	2	WOHL	4-WIPE	B	2	LOAD	906297	8.0	8.0363	1293.84	993.0	Q	1137.0	Q	76.75	87.88
344	2	WOHL	4-WIPE	B	2	LOAD	906298	8.0	8.1291	1308.79	972.0	Q	1105.0	Q	74.27	84.43
345	2	WOHL	4-WIPE	C	.	LOAD	906446	8.0	8.0727	1299.70	1009.0	Q	1220.0	Q	77.63	93.87
346	2	WOHL	4-WIPE	C	.	LOAD	906447	8.0	8.2092	1321.68	1001.0	Q	1201.0	Q	75.74	90.87
347	2	WOHL	4-WIPE	C	.	LOAD	906448	8.0	8.0661	1298.64	1001.0	Q	1182.0	Q	77.08	91.02
348	2	WOHL	4-WIPE	C	.	LOAD	906449	8.0	8.0031	1288.50	994.0	Q	1198.0	Q	77.14	92.98
349	2	WOHL	4-WIPE	QC	.	QC-LCS	905746	2.0	2.0030	322.48	350.0	Q	299.0	Q	108.53	92.72
350	2	WOHL	4-WIPE	QC	.	QC-LCS	905747	8.0	8.0051	1288.82	1020.0	Q	1215.0	Q	79.14	94.27
351	2	WOHL	4-WIPE	QC	.	QC-MB	905741	.	.	.	3.0	Q	.	ND	.	.
352	2	WOHL	4-WIPE	A	1	QC-WB	906011	.	.	.	2.0	Q	2.0	Q	.	.
353	2	WOHL	4-WIPE	B	2	QC-WB	906236	.	.	.	2.0	Q	1.0	Q	.	.
354	2	WOHL	4-WIPE	C	.	QC-WB	906323	.	.	.	9.0	Q	4.0	Q	.	.
COMPOSIT								130.0	131.0879	21105.15	17178		19283			
METHOD								227.5	229.2687	36912.26	31686		34081			
LAB_ID								450.0	453.4123	72999.38	69758		70593			

LABORATORY DATA AND LEAD RECOVERY RESULTS

------------------------------------ LAB_ID=3 METHOD=3050A COMPOSIT=1-WIPE ------------------------------------

OBS	LAB $\bar{I}$D	METHOD	COMPOSIT	BRAND $\bar{W}$	LOT	SPLE TYPE	BARCODE	TOT $\bar{S}$RM	TOT $\bar{L}$AB	TOT $\bar{P}$B	ICP	ICP $\bar{Q}$	FAA	FAA $\bar{Q}$	ICP $\bar{R}$EC	FAA $\bar{R}$EC
355	3	3050A	1-WIPE	A	1	LOAD	906015	0.5	0.5014	80.73	72.0	Q	83.0	Q	89.19	102.82
356	3	3050A	1-WIPE	A	2	LOAD	906090	0.5	0.5063	81.51	79.0	Q	88.0	Q	96.92	107.96
357	3	3050A	1-WIPE	B	1	LOAD	906165	0.5	0.5073	81.68	75.0	Q	82.0	Q	91.83	100.40
358	3	3050A	1-WIPE	B	2	LOAD	906243	0.5	0.5012	80.69	73.0	Q	82.0	Q	90.47	101.62
359	3	3050A	1-WIPE	C	.	LOAD	906332	0.5	0.4983	80.23	73.0	Q	85.0	Q	90.99	105.95
360	3	3050A	1-WIPE	C	.	LOAD	906333	0.5	0.5042	81.18	83.0	Q	99.0	Q	102.25	121.96
361	3	3050A	1-WIPE	A	1	LOAD	906048	2.0	2.0074	323.19	280.0	Q	320.0	Q	86.64	99.01
362	3	3050A	1-WIPE	A	2	LOAD	906123	2.0	2.0068	323.09	270.0	Q	320.0	Q	83.57	99.04
363	3	3050A	1-WIPE	B	1	LOAD	906198	2.0	2.0070	323.13	270.0	Q	320.0	Q	83.56	99.03
364	3	3050A	1-WIPE	B	2	LOAD	906276	2.0	2.0245	325.94	260.0	Q	320.0	Q	79.77	98.18
365	3	3050A	1-WIPE	C	.	LOAD	906398	2.0	2.0188	325.03	280.0	Q	330.0	Q	86.15	101.53
366	3	3050A	1-WIPE	C	.	LOAD	906399	2.0	2.0299	326.81	260.0	Q	300.0	Q	79.56	91.80
367	3	3050A	1-WIPE	QC	.	QC-LCS	905752	0.5	0.5000	80.50	74.0	Q	83.0	Q	91.93	103.11
368	3	3050A	1-WIPE	QC	.	QC-LCS	905753	2.0	2.0000	322.00	280.0	Q	320.0	Q	86.96	99.38
369	3	3050A	1-WIPE	QC	.	QC-MB	905748	.	.	.	.	ND	.	ND	.	.
370	3	3050A	1-WIPE	A	1	QC-WB	906003	.	.	.	.	ND	.	ND	.	.
371	3	3050A	1-WIPE	B	2	QC-WB	906231	.	.	.	.	ND	.	ND	.	.
372	3	3050A	1-WIPE	C	.	QC-WB	906309	.	.	.	.	ND	5.0	Q	.	.
COMPOSIT								17.5	17.6131	2835.71	2429.0		2837.0			

OBS	LAB ID	METHOD	COMPOSIT	BRAND W̄	LOT	SPLET TYPE	BARCODE	TOT S̄RM	TOT L̄AB	TOT P̄B	ICP	ICP Q̄	FAA	FAA Q̄	ICP R̄EC	FAA R̄EC
373	3	3050A	2-WIPE	A	1	LOAD	906026	1.0	1.0104	162.67	140.0	Q	170.0	Q	86.06	104.50
374	3	3050A	2-WIPE	A	1	LOAD	906027	1.0	1.0145	163.33	150.0	Q	170.0	Q	91.84	104.08
375	3	3050A	2-WIPE	A	2	LOAD	906101	1.0	1.0101	162.63	150.0	Q	180.0	Q	92.24	110.68
376	3	3050A	2-WIPE	A	2	LOAD	906102	1.0	1.0097	162.56	150.0	Q	170.0	Q	92.27	104.58
377	3	3050A	2-WIPE	B	1	LOAD	906176	1.0	0.9974	160.58	140.0	Q	160.0	Q	87.18	99.64
378	3	3050A	2-WIPE	B	1	LOAD	906177	1.0	1.0159	163.56	140.0	Q	170.0	Q	85.60	103.94
379	3	3050A	2-WIPE	B	2	LOAD	906254	1.0	1.0153	163.46	150.0	Q	170.0	Q	91.76	104.00
380	3	3050A	2-WIPE	B	2	LOAD	906255	1.0	1.0179	163.88	150.0	Q	170.0	Q	91.53	103.73
381	3	3050A	2-WIPE	C	.	LOAD	906354	1.0	1.0145	163.33	150.0	Q	180.0	Q	91.84	110.20
382	3	3050A	2-WIPE	C	.	LOAD	906355	1.0	1.0288	165.64	150.0	Q	180.0	Q	90.56	108.67
383	3	3050A	2-WIPE	C	.	LOAD	906356	1.0	1.0286	165.60	140.0	Q	160.0	Q	84.54	96.62
384	3	3050A	2-WIPE	C	.	LOAD	906357	1.0	1.0017	161.27	150.0	Q	200.0	Q	93.01	124.01
385	3	3050A	2-WIPE	A	1	LOAD	906059	4.0	4.0232	647.74	560.0	Q	660.0	Q	86.46	101.89
386	3	3050A	2-WIPE	A	1	LOAD	906060	4.0	4.0173	646.79	530.0	Q	640.0	Q	81.94	98.95
387	3	3050A	2-WIPE	A	2	LOAD	906134	4.0	4.0271	648.36	550.0	Q	680.0	Q	84.83	104.88
388	3	3050A	2-WIPE	A	2	LOAD	906135	4.0	4.0234	647.77	550.0	Q	690.0	Q	84.91	106.52
389	3	3050A	2-WIPE	B	1	LOAD	906209	4.0	4.0923	658.86	540.0	Q	670.0	Q	81.96	101.69
390	3	3050A	2-WIPE	B	1	LOAD	906210	4.0	4.0295	648.75	550.0	Q	660.0	Q	84.78	101.73
391	3	3050A	2-WIPE	B	2	LOAD	906287	4.0	4.0176	646.83	550.0	Q	660.0	Q	85.03	102.04
392	3	3050A	2-WIPE	B	2	LOAD	906288	4.0	4.0741	655.93	520.0	Q	660.0	Q	79.28	100.62
393	3	3050A	2-WIPE	C	.	LOAD	906420	4.0	4.0484	651.79	540.0	Q	660.0	Q	82.85	101.26
394	3	3050A	2-WIPE	C	.	LOAD	906421	4.0	4.1514	668.38	590.0	Q	690.0	Q	88.27	103.24
395	3	3050A	2-WIPE	C	.	LOAD	906422	4.0	4.0267	648.30	520.0	Q	660.0	Q	80.21	101.80
396	3	3050A	2-WIPE	C	.	LOAD	906423	4.0	4.2803	689.13	540.0	Q	670.0	Q	78.36	97.22
397	3	3050A	2-WIPE	QC	.	QC-LCS	905754	1.0	1.0000	161.00	150.0	Q	170.0	Q	93.17	105.59
398	3	3050A	2-WIPE	QC	.	QC-LCS	905755	4.0	4.0000	644.00	550.0	Q	660.0	Q	85.40	102.48
399	3	3050A	2-WIPE	QC	.	QC-MB	905749	.	.	.	.	ND	.	ND	.	.
400	3	3050A	2-WIPE	A	2	QC-WB	906081	.	.	.	.	ND	.	ND	.	.
401	3	3050A	2-WIPE	B	1	QC-WB	906156	.	.	.	.	ND	.	ND	.	.
402	3	3050A	2-WIPE	C	.	QC-WB	906315	.	.	.	.	ND	.	ND	.	.

COMPOSIT   65.0   65.9761   10622.15   9000.0   10910

------------------------------------- LAB_ID=3 METHOD=3050A COMPOSIT=4-WIPE -------------------------------------

OBS	LAB ID	METHOD	COMPOSIT	BRAND	LOT	SPLE TYPE	BARCODE	TOT SRM	TOT LAB	TOT PB	ICP	Q	FAA	Q	ICP REC	FAA REC
403	3	3050A	4-WIPE	A	1	LOAD	906036	2.0	2.0250	326.03	290.0	Q	330.0	Q	88.95	101.22
404	3	3050A	4-WIPE	A	1	LOAD	906045	2.0	2.0077	323.24	290.0	Q	340.0	Q	89.72	105.19
405	3	3050A	4-WIPE	A	2	LOAD	906111	2.0	2.0288	326.64	290.0	Q	360.0	Q	88.78	110.21
406	3	3050A	4-WIPE	A	2	LOAD	906120	2.0	2.0296	326.77	310.0	Q	330.0	Q	94.87	100.99
407	3	3050A	4-WIPE	B	1	LOAD	906186	2.0	2.0361	327.81	300.0	Q	350.0	Q	91.52	106.77
408	3	3050A	4-WIPE	B	1	LOAD	906195	2.0	2.0486	329.82	300.0	Q	320.0	Q	90.96	97.02
409	3	3050A	4-WIPE	B	2	LOAD	906264	2.0	2.0935	337.05	300.0	Q	360.0	Q	89.01	106.81
410	3	3050A	4-WIPE	B	2	LOAD	906273	2.0	2.0349	327.62	290.0	Q	330.0	Q	88.52	100.73
411	3	3050A	4-WIPE	C	.	LOAD	906374	2.0	2.0446	329.18	310.0	Q	350.0	Q	94.17	106.32
412	3	3050A	4-WIPE	C	.	LOAD	906375	2.0	2.0400	328.44	300.0	Q	360.0	Q	91.34	109.61
413	3	3050A	4-WIPE	C	.	LOAD	906392	2.0	2.0311	327.01	320.0	Q	350.0	Q	97.86	107.03
414	3	3050A	4-WIPE	C	.	LOAD	906393	2.0	2.0564	331.08	320.0	Q	340.0	Q	96.65	102.69
415	3	3050A	4-WIPE	A	1	LOAD	906069	8.0	8.0300	1292.83	1100.0	Q	1300.0	Q	85.08	100.55
416	3	3050A	4-WIPE	A	1	LOAD	906078	8.0	8.0208	1291.35	1100.0	Q	1300.0	Q	85.18	100.67
417	3	3050A	4-WIPE	A	2	LOAD	906144	8.0	8.1043	1304.79	1100.0	Q	1300.0	Q	84.30	99.63
418	3	3050A	4-WIPE	A	2	LOAD	906153	8.0	8.0317	1293.10	1100.0	Q	1200.0	Q	85.07	92.80
419	3	3050A	4-WIPE	B	1	LOAD	906221	8.0	8.0213	1291.43	1100.0	Q	1300.0	Q	85.18	100.66
420	3	3050A	4-WIPE	B	1	LOAD	906222	8.0	8.0303	1292.88	1100.0	Q	1300.0	Q	85.08	100.55
421	3	3050A	4-WIPE	B	2	LOAD	906305	8.0	8.0259	1292.17	1100.0	Q	1200.0	Q	85.13	92.87
422	3	3050A	4-WIPE	B	2	LOAD	906306	8.0	8.0255	1292.11	1000.0	Q	1200.0	Q	77.39	92.87
423	3	3050A	4-WIPE	C	.	LOAD	906440	8.0	8.1479	1311.81	1100.0	Q	1300.0	Q	83.85	99.10
424	3	3050A	4-WIPE	C	.	LOAD	906441	8.0	8.0892	1302.36	1200.0	Q	1400.0	Q	92.14	107.50
425	3	3050A	4-WIPE	C	.	LOAD	906458	8.0	8.0416	1294.70	1100.0	Q	1200.0	Q	84.96	92.69
426	3	3050A	4-WIPE	C	.	LOAD	906459	8.0	8.0753	1300.12	1100.0	Q	1200.0	Q	84.61	92.30
427	3	3050A	4-WIPE	QC	.	QC-LCS	905756	2.0	2.0000	322.00	290.0	Q	290.0	Q	90.06	90.06
428	3	3050A	4-WIPE	QC	.	QC-LCS	905757	2.0	2.0000	322.00	300.0	Q	320.0	Q	93.17	99.38
429	3	3050A	4-WIPE	QC	.	QC-LCS	905758	8.0	8.0000	1288.00	1100.0	Q	1300.0	Q	85.40	100.93
430	3	3050A	4-WIPE	QC	.	QC-LCS	905759	8.0	8.0000	1288.00	1100.0	Q	1300.0	Q	85.40	100.93
431	3	3050A	4-WIPE	QC	.	QC-MB	905750	.	.	.	.	ND	.	ND	.	.
432	3	3050A	4-WIPE	QC	.	QC-MB	905751	.	.	.	.	ND	.	ND	.	.
433	3	3050A	4-WIPE	A	1	QC-WB	906009	.	.	.	.	ND	.	ND	.	.
434	3	3050A	4-WIPE	A	2	QC-WB	906087	.	.	.	.	ND	.	ND	.	.
435	3	3050A	4-WIPE	B	1	QC-WB	906162	.	.	.	.	ND	30.0	Q	.	.
436	3	3050A	4-WIPE	B	2	QC-WB	906240	.	.	.	.	ND	.	ND	.	.
437	3	3050A	4-WIPE	C	.	QC-WB	906321	.	.	.	.	ND	30.0	Q	.	.
438	3	3050A	4-WIPE	C	.	QC-WB	906327	.	.	.	.	ND	.	ND	.	.
COMPOSIT								140.0	141.1201	22720.34	19610		22590			
METHOD								222.5	224.7093	36178.20	31039		36337			

-- LAB_ID=3 METHOD=WOHL COMPOSIT=1-WIPE --

OBS	LAB ID	METHOD	COMPOSIT	BRAND	LOT	SPLE TYPE	BARCODE	TOT SRM	TOT LAB	TOT PB	ICP Q̄	FAA Q̄	ICP REC	FAA REC
439	3	WOHL	1-WIPE	A	1	LOAD	906020	0.5	0.5038	81.11	64.0 Q	72.0 Q	78.90	88.77
440	3	WOHL	1-WIPE	A	1	LOAD	906021	0.5	0.5025	80.90	66.0 Q	78.0 Q	81.58	96.41
441	3	WOHL	1-WIPE	A	2	LOAD	906095	0.5	0.5099	82.09	65.0 Q	73.0 Q	79.18	88.92
442	3	WOHL	1-WIPE	A	2	LOAD	906096	0.5	0.5094	82.01	64.0 Q	72.0 Q	78.04	87.79
443	3	WOHL	1-WIPE	B	1	LOAD	906170	0.5	0.5061	81.48	67.0 Q	74.0 Q	82.23	90.82
444	3	WOHL	1-WIPE	B	1	LOAD	906171	0.5	0.5102	82.14	68.0 Q	76.0 Q	82.78	92.52
445	3	WOHL	1-WIPE	B	2	LOAD	906248	0.5	0.5071	81.64	69.0 Q	78.0 Q	84.51	95.54
446	3	WOHL	1-WIPE	B	2	LOAD	906249	0.5	0.5092	81.98	65.0 Q	73.0 Q	79.29	89.04
447	3	WOHL	1-WIPE	C	.	LOAD	906342	0.5	0.5222	84.07	76.0 Q	84.0 Q	90.40	99.91
448	3	WOHL	1-WIPE	C	.	LOAD	906343	0.5	0.5078	81.76	70.0 Q	81.0 Q	85.62	99.08
449	3	WOHL	1-WIPE	C	.	LOAD	906344	0.5	0.5067	81.58	71.0 Q	79.0 Q	87.03	96.84
450	3	WOHL	1-WIPE	C	.	LOAD	906345	0.5	0.5089	81.93	69.0 Q	78.0 Q	84.22	95.20
451	3	WOHL	1-WIPE	A	1	LOAD	906053	2.0	2.0002	322.03	240.0 Q	280.0 Q	74.53	86.95
452	3	WOHL	1-WIPE	A	1	LOAD	906054	2.0	2.0056	322.90	250.0 Q	330.0 Q	77.42	102.20
453	3	WOHL	1-WIPE	A	2	LOAD	906128	2.0	2.0226	325.64	250.0 Q	320.0 Q	76.77	98.27
454	3	WOHL	1-WIPE	A	2	LOAD	906129	2.0	2.0148	324.38	250.0 Q	330.0 Q	77.07	101.73
455	3	WOHL	1-WIPE	B	1	LOAD	906203	2.0	2.0151	324.43	230.0 Q	280.0 Q	70.89	86.30
456	3	WOHL	1-WIPE	B	1	LOAD	906204	2.0	2.0058	322.93	240.0 Q	290.0 Q	74.32	89.80
457	3	WOHL	1-WIPE	B	2	LOAD	906281	2.0	2.0233	325.75	260.0 Q	310.0 Q	79.82	95.16
458	3	WOHL	1-WIPE	B	2	LOAD	906282	2.0	2.0069	323.11	240.0 Q	310.0 Q	74.28	95.94
459	3	WOHL	1-WIPE	C	.	LOAD	906408	2.0	1.9862	319.78	240.0 Q	290.0 Q	75.05	90.69
460	3	WOHL	1-WIPE	C	.	LOAD	906409	2.0	2.0054	322.87	260.0 Q	320.0 Q	80.53	99.11
461	3	WOHL	1-WIPE	C	.	LOAD	906410	2.0	2.0200	325.22	260.0 Q	330.0 Q	79.95	101.47
462	3	WOHL	1-WIPE	C	.	LOAD	906411	2.0	2.0107	323.72	260.0 Q	320.0 Q	80.32	98.85
463	3	WOHL	1-WIPE	QC	.	QC-LCS	905763	0.5	0.5000	80.50	72.0 Q	82.0 Q	89.44	101.86
464	3	WOHL	1-WIPE	QC	.	QC-LCS	905764	2.0	2.0000	322.00	270.0 Q	330.0 Q	83.85	102.48
465	3	WOHL	1-WIPE	QC	.	QC-MB	905760	.	.	.	. ND	. ND	.	.
466	3	WOHL	1-WIPE	A	1	QC-WB	906006	.	.	.	. ND	. ND	.	.
467	3	WOHL	1-WIPE	B	2	QC-WB	906234	.	.	.	. ND	. ND	.	.
468	3	WOHL	1-WIPE	C	.	QC-WB	906312	.	.	.	. ND	. ND	.	.
COMPOSIT								32.5	32.7204	5267.98	4136.0	5040.0		

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--- LAB_ID=3 METHOD=WOHL COMPOSIT=2-WIPE ---

OBS	LAB_ID	METHOD	COMPOSIT	BRAND	LOT	SPLE TYPE	BARCODE	TOT_SRM	TOT_LAB	TOT_PB	ICP	ICP_Q	FAA	FAA_Q	ICP_REC	FAA_REC
469	3	WOHL	2-WIPE	A	1	LOAD	906032	1.0	1.0072	162.16	120.0	Q	140.0	Q	74.00	86.33
470	3	WOHL	2-WIPE	A	1	LOAD	906033	1.0	1.0073	162.18	120.0	Q	130.0	Q	73.99	80.16
471	3	WOHL	2-WIPE	A	2	LOAD	906107	1.0	1.0053	161.85	110.0	Q	130.0	Q	67.96	80.32
472	3	WOHL	2-WIPE	A	2	LOAD	906108	1.0	1.0112	162.80	110.0	Q	130.0	Q	67.57	79.85
473	3	WOHL	2-WIPE	B	1	LOAD	906182	1.0	1.0112	162.80	120.0	Q	140.0	Q	73.71	85.99
474	3	WOHL	2-WIPE	B	1	LOAD	906183	1.0	1.0179	163.88	120.0	Q	140.0	Q	73.22	85.43
475	3	WOHL	2-WIPE	B	2	LOAD	906260	1.0	1.0009	161.14	130.0	Q	140.0	Q	80.67	86.88
476	3	WOHL	2-WIPE	B	2	LOAD	906261	1.0	1.0017	161.27	130.0	Q	140.0	Q	80.61	86.81
477	3	WOHL	2-WIPE	C	.	LOAD	906366	1.0	1.0204	164.28	140.0	Q	150.0	Q	85.22	91.31
478	3	WOHL	2-WIPE	C	.	LOAD	906367	1.0	1.0062	162.00	140.0	Q	160.0	Q	86.42	98.77
479	3	WOHL	2-WIPE	C	.	LOAD	906368	1.0	1.0150	163.42	130.0	Q	150.0	Q	79.55	91.79
480	3	WOHL	2-WIPE	C	.	LOAD	906369	1.0	1.0777	173.51	150.0	Q	170.0	Q	86.45	97.98
481	3	WOHL	2-WIPE	A	1	LOAD	906065	4.0	4.0122	645.96	450.0	Q	600.0	Q	69.66	92.88
482	3	WOHL	2-WIPE	A	1	LOAD	906066	4.0	4.0474	651.63	440.0	Q	560.0	Q	67.52	85.94
483	3	WOHL	2-WIPE	A	2	LOAD	906140	4.0	4.0191	647.08	460.0	Q	590.0	Q	71.09	91.18
484	3	WOHL	2-WIPE	A	2	LOAD	906141	4.0	4.0204	647.28	470.0	Q	600.0	Q	72.61	92.69
485	3	WOHL	2-WIPE	B	1	LOAD	906215	4.0	4.0233	647.75	460.0	Q	570.0	Q	71.01	88.00
486	3	WOHL	2-WIPE	B	1	LOAD	906216	4.0	4.0157	646.53	460.0	Q	600.0	Q	71.15	92.80
487	3	WOHL	2-WIPE	B	2	LOAD	906293	4.0	4.0323	649.20	460.0	Q	570.0	Q	70.86	87.80
488	3	WOHL	2-WIPE	B	2	LOAD	906294	4.0	4.0250	648.03	490.0	Q	620.0	Q	75.61	95.68
489	3	WOHL	2-WIPE	C	.	LOAD	906432	4.0	4.0524	652.44	480.0	Q	600.0	Q	73.57	91.96
490	3	WOHL	2-WIPE	C	.	LOAD	906433	4.0	4.0415	650.68	490.0	Q	600.0	Q	75.31	92.21
491	3	WOHL	2-WIPE	C	.	LOAD	906434	4.0	4.0261	648.20	460.0	Q	600.0	Q	70.97	92.56
492	3	WOHL	2-WIPE	C	.	LOAD	906435	4.0	4.0749	656.06	490.0	Q	610.0	Q	74.69	92.98
493	3	WOHL	2-WIPE	QC	.	QC-LCS	905765	1.0	1.0000	161.00	130.0	Q	150.0	Q	80.75	93.17
494	3	WOHL	2-WIPE	QC	.	QC-LCS	905766	4.0	4.0000	644.00	510.0	Q	650.0	Q	79.19	100.93
495	3	WOHL	2-WIPE	QC	.	QC-MB	905761	.	.	.	.	ND	.	ND	.	.
496	3	WOHL	2-WIPE	A	2	QC-WB	906084	.	.	.	.	ND	.	ND	.	.
497	3	WOHL	2-WIPE	B	1	QC-WB	906159	.	.	.	.	ND	.	ND	.	.
498	3	WOHL	2-WIPE	C	.	QC-WB	906318	.	.	.	.	ND	.	ND	.	.

COMPOSIT
65.0   65.5723   10557.14   7770.0   9640.0

--- LAB_ID=3 METHOD=WOHL COMPOSIT=4-WIPE ---

OBS	LAB ĪD	METHOD	COMPOSIT	BRAND	LOT	SPLETYPE	BARCODE	TOT S̄RM	TOT L̄AB	TOT P̄B	ICP	ICP_Q	FAA	FAA_Q	ICP R̄EC	FAA R̄EC
499	3	WOHL	4-WIPE	A	1	LOAD	906041	2.0	2.0201	325.24	210.0	Q	250.0	Q	64.57	76.87
500	3	WOHL	4-WIPE	A	1	LOAD	906042	2.0	2.0202	325.25	180.0	Q	220.0	Q	55.34	67.64
501	3	WOHL	4-WIPE	A	2	LOAD	906116	2.0	2.0521	330.39	220.0	Q	260.0	Q	66.59	78.70
502	3	WOHL	4-WIPE	A	2	LOAD	906117	2.0	2.0402	328.47	220.0	Q	270.0	Q	66.98	82.20
503	3	WOHL	4-WIPE	B	1	LOAD	906191	2.0	2.0299	326.81	220.0	Q	250.0	Q	67.32	76.50
504	3	WOHL	4-WIPE	B	1	LOAD	906192	2.0	2.0310	326.99	220.0	Q	260.0	Q	67.28	79.51
505	3	WOHL	4-WIPE	B	2	LOAD	906269	2.0	2.0417	328.71	200.0	Q	230.0	Q	60.84	69.97
506	3	WOHL	4-WIPE	B	2	LOAD	906270	2.0	2.0243	325.91	200.0	Q	240.0	Q	61.37	73.64
507	3	WOHL	4-WIPE	C	.	LOAD	906384	2.0	2.0441	329.10	230.0	Q	270.0	Q	69.89	82.04
508	3	WOHL	4-WIPE	C	.	LOAD	906385	2.0	2.0434	328.99	270.0	Q	310.0	Q	82.07	94.23
509	3	WOHL	4-WIPE	C	.	LOAD	906386	2.0	2.0337	327.43	240.0	Q	270.0	Q	73.30	82.46
510	3	WOHL	4-WIPE	C	.	LOAD	906387	2.0	2.0539	330.68	260.0	Q	300.0	Q	78.63	90.72
511	3	WOHL	4-WIPE	A	1	LOAD	906074	8.0	8.0268	1292.31	670.0	Q	880.0	Q	51.84	68.09
512	3	WOHL	4-WIPE	A	1	LOAD	906075	8.0	8.0114	1289.84	680.0	Q	940.0	Q	52.72	72.88
513	3	WOHL	4-WIPE	A	2	LOAD	906149	8.0	8.0465	1295.49	790.0	Q	1000.0	Q	60.98	77.19
514	3	WOHL	4-WIPE	A	2	LOAD	906150	8.0	8.0404	1294.50	700.0	Q	960.0	Q	54.07	74.16
515	3	WOHL	4-WIPE	B	1	LOAD	906227	8.0	8.0425	1294.84	660.0	Q	880.0	Q	50.97	67.96
516	3	WOHL	4-WIPE	B	1	LOAD	906228	8.0	8.0909	1302.63	650.0	Q	850.0	Q	49.90	65.25
517	3	WOHL	4-WIPE	B	2	LOAD	906299	8.0	8.0234	1291.77	710.0	Q	940.0	Q	54.96	72.77
518	3	WOHL	4-WIPE	B	2	LOAD	906300	8.0	8.0295	1292.75	700.0	Q	900.0	Q	54.15	69.62
519	3	WOHL	4-WIPE	C	.	LOAD	906450	8.0	8.0154	1290.48	780.0	Q	1000.0	Q	60.44	77.49
520	3	WOHL	4-WIPE	C	.	LOAD	906451	8.0	8.1075	1305.31	750.0	Q	940.0	Q	57.46	72.01
521	3	WOHL	4-WIPE	C	.	LOAD	906452	8.0	8.1224	1307.71	790.0	Q	1000.0	Q	60.41	76.47
522	3	WOHL	4-WIPE	C	.	LOAD	906453	8.0	8.0462	1295.44	810.0	Q	1100.0	Q	62.53	84.91
523	3	WOHL	4-WIPE	QC	.	QC-LCS	905767	2.0	2.0000	322.00	280.0	Q	330.0	Q	86.96	102.48
524	3	WOHL	4-WIPE	QC	.	QC-LCS	905768	8.0	8.0000	1288.00	940.0	Q	1300.0	Q	72.98	100.93
525	3	WOHL	4-WIPE	QC	.	QC-MB	905762	.	.	.	.	ND	.	ND	.	.
526	3	WOHL	4-WIPE	A	1	QC-WB	906012	.	.	.	.	ND	.	ND	.	.
527	3	WOHL	4-WIPE	B	2	QC-WB	906237	.	.	.	.	ND	.	ND	.	.
528	3	WOHL	4-WIPE	C	.	QC-WB	906324	.	.	.	17.0	Q	19.0	Q	.	.
COMPOSIT								130.0	131.0375	21097.04	12597		16169			
METHOD								227.5	229.3302	36922.16	24503		30849			
LAB_ID								450.0	454.0395	73100.36	55542		67186			
								1355.0	1366.5629	220016.63	185807		203760			

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Appendix I

ANOVA Results for Four-way and Three-way Mixed Models

Organization of Statistical Tables I-1 Through I-6

The following tables are reproduced computer outputs obtained when using the PROC MIXED procedure of SAS.[10,11] Each table presents the following information:

1. **Class Level Information**: Lists the class variables (fixed and random factors) and their levels included in the model.

2. **REML (REstricted Maximum Likelihood) Iteration History**: These estimates are the values that maximize the restricted likelihood function of the data, or alternatively (as on this output), minimize the function, -2 times the logarithm of this likelihood function, using a Newton-Raphson algorithm. For example, in Table I-1, 3 iterations were needed to obtain the estimates. A criterion of zero indicates that the algorithm converged.

3. **Covariance Parameter Estimates (REML)**: For each random factor, interaction, and residual (under header "Cov Parm"), this tables provides estimates of variance components under the heading "Estimate". The column "Ratio" is the ratio of these variance components over the residual variance component. The "STD Error" provides approximate standard errors of the variance components, with associated Z-statistics to test that the variance components equal zero, and the associated p-level.

4. **Model Fitting Information for Dependent Variable (e.g., FAA_REC, ICP_REC)**: This output provides various information about the fitted mixed model.

5. **Tests of Fixed Effects**: This table lists all fixed effect factors and their interaction, numerator and denominator degrees of freedom (NDF and DDF), and p-level associated with their F-test for significance. A p-level below 0.05 indicates that the corresponding factor or interaction is significant at the 5% level.

6. **Estimate Statement Results**: This table lists estimates and associated statistics for selected contrasts. For example, the estimate for "FAA: 3050A vs WOHL" of 11.777% (Table I-1) is the estimated difference of FAA recoveries between the EPA 3050A and the WOHL methods. Its standard error is 2.527% with 2 degrees of freedom. This difference is statistically significant at the 5% level as indicated by the p-level of 0.0431. Lower and upper 95% (alpha of 5%) are given in the last two columns.

7. **Least Squares Means**: This information is shown in Tables I-3 through I-6 and provides model least squares means for all main fixed effect factors and their interactions. Identical information to that shown in item No. 6 is provided. Parameter estimates in item No. 6 can be obtained from those in this table.

Table I-1. Analysis of Variance Results for the 4-way Mixed Model for FAA Recovery

```
FAA: 4-WAY MIXED MODEL ANOVA WITH ALL INTERACTIONS
          LABORATORY: RANDOM FACTOR


               The MIXED Procedure

              Class Level Information

        Class     Levels  Values

        LAB_ID      3     1 2 3
        METHOD      2     EPA3050A WOHL
        SRMLEVEL    2     HIGH LOW
        COMPOSIT    3     1-WIPE 2-WIPE 4-WIPE


           REML Estimation Iteration History

   Iteration  Evaluations    Objective     Criterion

        0          1      1957.8188777
        1          3      1726.4612877    0.00003545
        2          1      1726.4289827    0.00000045
        3          1      1726.4285906    0.00000000

            Convergence criteria met.
```

Covariance Parameter Estimates (REML)

Cov Parm	Ratio	Estimate	Std Error	Z	Pr > |Z|
LAB_ID	0.54402482	13.84447502	19.55222070	0.71	0.4789
LAB_ID*METHOD	0.00000000	0.00000000	.	.	.
LAB_ID*SRMLEVEL	0.00000000	0.00000000	.	.	.
LAB_ID*COMPOSIT	0.00000000	0.00000000	.	.	.
LAB_ID*METHOD*SRMLEV	0.46894918	11.93393183	9.17062097	1.30	0.1931
LAB_ID*METHOD*COMPOS	0.29911154	7.61186263	6.20050721	1.23	0.2196
LAB_ID*SRMLEV*COMPOS	0.17744576	4.51568263	4.95242324	0.91	0.3619
LAB_*METH*SRML*COMPO	0.15616808	3.97420288	4.63559043	0.86	0.3913
Residual	1.00000000	25.44824142	1.89510158	13.43	0.0001

Model Fitting Information for FAA_REC

Description	Value
Observations	396.0000
Variance Estimate	25.4482
Standard Deviation Estimate	5.0446
REML Log Likelihood	-1216.09
Akaike's Information Criterion	-1225.09
Schwarz's Bayesian Criterion	-1242.86
-2 REML Log Likelihood	2432.173

Tests of Fixed Effects

Source	NDF	DDF	Type III F	Pr > F
METHOD	1	2	21.72	0.0431
SRMLEVEL	1	2	0.45	0.5733
COMPOSIT	2	4	4.82	0.0860
METHOD*SRMLEVEL	1	2	0.52	0.5463
METHOD*COMPOSIT	2	4	4.86	0.0849
SRMLEVEL*COMPOSIT	2	4	0.00	0.9994
METHOD*SRMLEV*COMPOS	2	4	1.51	0.3252

ESTIMATE Statement Results

Parameter	Estimate	Std Error	DDF	T	Pr > |T|	Alpha	Lower	Upper
FAA: 3050A VS WOHL	11.77720504	2.52705050	2	4.66	0.0431	0.05	0.9042	22.6502
FAA: 3050A MEAN	99.70114814	2.84223932	2	35.08	0.0008	0.05	87.4720	111.9303
FAA: WOHL MEAN	87.92394310	2.83532227	2	31.01	0.0010	0.05	75.7245	100.1234

Table I-2. Analysis of Variance Results for the 4-way Mixed Model for ICP Recovery

```
ICP: 4-WAY MIXED MODEL ANOVA WITH ALL INTERACTIONS
            LABORATORY: RANDOM FACTOR
```

The MIXED Procedure

Class Level Information

Class	Levels	Values
LAB_ID	3	1 2 3
METHOD	2	EPA3050A WOHL
SRMLEVEL	2	HIGH LOW
COMPOSIT	3	1-WIPE 2-WIPE 4-WIPE

REML Estimation Iteration History

Iteration	Evaluations	Objective	Criterion
0	1	2175.7735891	
1	4	1627.2329038	.
2	2	1626.9787910	0.00004732
3	2	1626.9369787	0.00000105
4	1	1626.9361081	0.00000000

Convergence criteria met.

Covariance Parameter Estimates (REML)

Cov Parm	Ratio	Estimate	Std Error	Z	Pr > |Z|
LAB_ID	4.48046522	86.11838349	89.73612688	0.96	0.3372
LAB_ID*METHOD	0.00000000	0.00000000	.	.	.
LAB_ID*SRMLEVEL	0.00000000	0.00000000	.	.	.
LAB_ID*COMPOSIT	0.07596925	1.46019416	5.18508680	0.28	0.7782
LAB_ID*METHOD*SRMLEV	0.32401634	6.22787195	6.75406839	0.92	0.3565
LAB_ID*METHOD*COMPOS	0.13790059	2.65056763	6.77970587	0.39	0.6958
LAB_ID*SRMLEV*COMPOS	0.00000000	0.00000000	.	.	.
LAB_*METH*SRML*COMPO	0.59765443	11.48743067	6.99246107	1.64	0.1004
Residual	1.00000000	19.22085750	1.43178133	13.42	0.0001

Model Fitting Information for ICP_REC

Description	Value
Observations	396.0000
Variance Estimate	19.2209
Standard Deviation Estimate	4.3842
REML Log Likelihood	-1166.34
Akaike's Information Criterion	-1175.34
Schwarz's Bayesian Criterion	-1193.12
-2 REML Log Likelihood	2332.681

Tests of Fixed Effects

Source	NDF	DDF	Type III F	Pr > F
METHOD	1	2	50.79	0.0191
SRMLEVEL	1	2	13.00	0.0690
COMPOSIT	2	4	3.14	0.1513
METHOD*SRMLEVEL	1	2	0.06	0.8327
METHOD*COMPOSIT	2	4	11.95	0.0206
SRMLEVEL*COMPOSIT	2	4	2.47	0.1999
METHOD*SRMLEV*COMPOS	2	4	3.44	0.1353

ESTIMATE Statement Results

Parameter	Estimate	Std Error	DDF	T	Pr > |T|	Alpha	Lower	Upper
ICP: 3050A VS WOHL	14.51673604	2.03690605	2	7.13	0.0191	0.05	5.7526	23.2808
ICP: 3050A MEAN	94.32092798	5.56396402	2	16.95	0.0035	0.05	70.3811	118.2607
ICP: WOHL MEAN	79.80419193	5.56129786	2	14.35	0.0048	0.05	55.8759	103.7325

Table I-3. Analysis of Variance Results for the 3-way Mixed Model for FAA Recovery using EPA Method 3050A

```
FAA: 3-WAY MIXED MODEL ANOVA WITH ALL INTERACTIONS
          LABORATORY: RANDOM FACTOR

---------------------------- METHOD=EPA3050A ----------------------------------

                    The MIXED Procedure

                 Class Level Information

         Class      Levels  Values

         LAB_ID        3    1 2 3
         SRMLEVEL      2    HIGH LOW
         COMPOSIT      3    1-WIPE 2-WIPE 4-WIPE


               REML Estimation Iteration History

        Iteration  Evaluations     Objective      Criterion

            0            1       850.08630832
            1            4       748.21339847
            2            2       748.02978880     0.00004734
            3            1       748.01067270     0.00000083
            4            1       748.01035961     0.00000000

                Convergence criteria met.


              Covariance Parameter Estimates (REML)

   Cov Parm              Ratio       Estimate     Std Error      Z    Pr > |Z|

   LAB_ID              0.11331120    2.48280130   22.29316410   0.11   0.9113
   LAB_ID*SRMLEVEL     1.27797303   28.00211443   28.96455200   0.97   0.3337
   LAB_ID*COMPOSIT     0.00000000    0.00000000       .           .
   LAB_ID*SRMLEV*COMPOS 0.02519946   0.55215415    1.42823131   0.39   0.6991
   Residual            1.00000000   21.91135012    2.43164949   9.01   0.0001


            Model Fitting Information for FAA_REC

         Description                          Value

         Observations                        180.0000
         Variance Estimate                    21.9114
         Standard Deviation Estimate           4.6810
         REML Log Likelihood                -533.900
         Akaike's Information Criterion     -538.900
         Schwarz's Bayesian Criterion       -546.798
         -2 REML Log Likelihood             1067.801
```

Table I-3. (Continued)

FAA: 3-WAY MIXED MODEL ANOVA WITH ALL INTERACTIONS
LABORATORY: RANDOM FACTOR

-- METHOD=EPA3050A --

Tests of Fixed Effects

Source	NDF	DDF	Type III F	Pr > F
SRMLEVEL	1	2	0.00	0.9948
COMPOSIT	2	4	1.66	0.2992
SRMLEVEL*COMPOSIT	2	4	1.43	0.3398

ESTIMATE Statement Results

Parameter	Estimate	Std Error	DDF	T	Pr > |T|	Alpha	Lower	Upper
FAA: SRM HIGH VS LOW	-0.03241493	4.39679408	2	-0.01	0.9948	0.05	-18.9503	18.8855
FAA: SRM LOW MEAN	99.71735561	3.23936715	2	30.78	0.0011	0.05	85.7795	113.6552
FAA: SRM HIGH MEAN	99.68494067	3.23936715	2	30.77	0.0011	0.05	85.7471	113.6228
FAA: 1-W VS 2-W	-1.42520511	1.04738928	4	-1.36	0.2452	0.05	-4.3332	1.4828
FAA: 1-W VS 4-W	0.06386256	1.04738928	4	0.06	0.9543	0.05	-2.8442	2.9719
FAA: 2-W VS 4-W	1.48906766	0.89033701	4	1.67	0.1697	0.05	-0.9829	3.9610
FAA: 1-WIPE MEAN	99.24736729	2.48903471	4	39.87	0.0001	0.05	92.3367	106.1580
FAA: 2-WIPE MEAN	100.67257240	2.42713195	4	41.48	0.0001	0.05	93.9338	107.4114
FAA: 4-WIPE MEAN	99.18350474	2.42713195	4	40.86	0.0001	0.05	92.4447	105.9223

Least Squares Means

Level	LSMEAN	Std Error	DDF	T	Pr > |T|	Alpha	Lower	Upper
SRMLEVEL HIGH	99.68494067	3.23936715	2	30.77	0.0011	0.05	85.7471	113.6228
SRMLEVEL LOW	99.71735561	3.23936715	2	30.78	0.0011	0.05	85.7795	113.6552
COMPOSIT 1-WIPE	99.24736729	2.48903471	4	39.87	0.0001	0.05	92.3367	106.1580
COMPOSIT 2-WIPE	100.67257240	2.42713195	4	41.48	0.0001	0.05	93.9338	107.4114
COMPOSIT 4-WIPE	99.18350474	2.42713195	4	40.86	0.0001	0.05	92.4447	105.9223
SRMLEVEL*COMPOSIT HIGH 1-WIPE	98.30015829	3.40043926	4	28.91	0.0001	0.05	88.8590	107.7413
SRMLEVEL*COMPOSIT HIGH 2-WIPE	100.75141735	3.30973391	4	30.44	0.0001	0.05	91.5621	109.9407
SRMLEVEL*COMPOSIT HIGH 4-WIPE	100.00324639	3.30973391	4	30.21	0.0001	0.05	90.8140	109.1925
SRMLEVEL*COMPOSIT LOW 1-WIPE	100.19457629	3.40043926	4	29.47	0.0001	0.05	90.7534	109.6357
SRMLEVEL*COMPOSIT LOW 2-WIPE	100.59372745	3.30973391	4	30.39	0.0001	0.05	91.4044	109.7830
SRMLEVEL*COMPOSIT LOW 4-WIPE	98.36376308	3.30973391	4	29.72	0.0001	0.05	89.1745	107.5531

Table I-4. Analysis of Variance Results for the 3-way Mixed Model for FAA Recovery using the WOHL Method

```
                FAA: 3-WAY MIXED MODEL ANOVA WITH ALL INTERACTIONS
                          LABORATORY: RANDOM FACTOR
-------------------------------------- METHOD=WOHL -------------------------------------


                              The MIXED Procedure

                             Class Level Information

                   Class     Levels  Values

                   LAB_ID       3    1 2 3
                   SRMLEVEL     2    HIGH LOW
                   COMPOSIT     3    1-WIPE 2-WIPE 4-WIPE


                        REML Estimation Iteration History

            Iteration  Evaluations    Objective     Criterion

                 0          1       1101.2677265
                 1          3        966.05875487   0.00002386
                 2          1        966.04641441   0.00000032
                 3          1        966.04625725   0.00000000

                         Convergence criteria met.


                    Covariance Parameter Estimates (REML)

      Cov Parm                 Ratio       Estimate     Std Error      Z  Pr > |Z|

      LAB_ID                0.73515523   20.86376901   29.44089379   0.71   0.4785
      LAB_ID*SRMLEVEL       0.00000000    0.00000000        .          .       .
      LAB_ID*COMPOSIT       0.57398455   16.28973121   17.60699292   0.93   0.3549
      LAB_ID*SRMLEV*COMPOS  0.46531735   13.20574648    8.99200342   1.47   0.1419
      Residual              1.00000000   28.38008639    2.85230600   9.95   0.0001


                    Model Fitting Information for FAA_REC

            Description                          Value

            Observations                       216.0000
            Variance Estimate                   28.3801
            Standard Deviation Estimate          5.3273
            REML Log Likelihood               -676.000
            Akaike's Information Criterion    -681.000
            Schwarz's Bayesian Criterion      -689.368
            -2 REML Log Likelihood            1352.000
```

Table I-4. (Continued)

```
                    FAA: 3-WAY MIXED MODEL ANOVA WITH ALL INTERACTIONS
                               LABORATORY: RANDOM FACTOR

------------------------------------------------ METHOD=WOHL ------------------------------------------------


                                   Tests of Fixed Effects

                       Source              NDF   DDF   Type III F   Pr > F

                       SRMLEVEL             1     2       2.87      0.2322
                       COMPOSIT             2     4       5.12      0.0789
                       SRMLEVEL*COMPOSIT    2     4       0.34      0.7287


                               ESTIMATE Statement Results

       Parameter               Estimate      Std Error   DDF      T    Pr > |T|  Alpha    Lower      Upper

       FAA: SRM HIGH VS LOW   -3.15278030    1.86015254   2    -1.69   0.2322    0.05   -11.1564    4.8508
       FAA: SRM LOW MEAN      89.50033325    3.23954373   2    27.63   0.0013    0.05    75.5617  103.4390
       FAA: SRM HIGH MEAN     86.34755295    3.23954373   2    26.65   0.0014    0.05    72.4089  100.2862
       FAA: 1-W VS 2-W        -1.86546788    4.00625412   4    -0.47   0.6657    0.05   -12.9886    9.2577
       FAA: 1-W VS 4-W        10.05395272    4.00625412   4     2.51   0.0661    0.05    -1.0692   21.1771
       FAA: 2-W VS 4-W        11.91942059    4.00625412   4     2.98   0.0409    0.05     0.7963   23.0426
       FAA: 1-WIPE MEAN       90.65343804    3.87035214   4    23.42   0.0001    0.05    79.9076  101.3993
       FAA: 2-WIPE MEAN       92.51890592    3.87035214   4    23.90   0.0001    0.05    81.7731  103.2647
       FAA: 4-WIPE MEAN       80.59948533    3.87035214   4    20.82   0.0001    0.05    69.8537   91.3453


                                    Least Squares Means

  Level                           LSMEAN      Std Error   DDF      T    Pr > |T|  Alpha    Lower      Upper

  SRMLEVEL HIGH                 86.34755295   3.23954373   2    26.65   0.0014    0.05    72.4089  100.2862
  SRMLEVEL LOW                  89.50033325   3.23954373   2    27.63   0.0013    0.05    75.5617  103.4390
  COMPOSIT 1-WIPE               90.65343804   3.87035214   4    23.42   0.0001    0.05    79.9076  101.3993
  COMPOSIT 2-WIPE               92.51890592   3.87035214   4    23.90   0.0001    0.05    81.7731  103.2647
  COMPOSIT 4-WIPE               80.59948533   3.87035214   4    20.82   0.0001    0.05    69.8537   91.3453
  SRMLEVEL*COMPOSIT HIGH 1-WIPE 90.07055275   4.19222510   4    21.49   0.0001    0.05    78.4311  101.7100
  SRMLEVEL*COMPOSIT HIGH 2-WIPE 90.83250605   4.19222510   4    21.67   0.0001    0.05    79.1930  102.4720
  SRMLEVEL*COMPOSIT HIGH 4-WIPE 78.13960005   4.19222510   4    18.64   0.0001    0.05    66.5001   89.7791
  SRMLEVEL*COMPOSIT LOW 1-WIPE  91.23632334   4.19222510   4    21.76   0.0001    0.05    79.5968  102.8758
  SRMLEVEL*COMPOSIT LOW 2-WIPE  94.20530579   4.19222510   4    22.47   0.0001    0.05    82.5658  105.8448
  SRMLEVEL*COMPOSIT LOW 4-WIPE  83.05937061   4.19222510   4    19.81   0.0001    0.05    71.4199   94.6989
```

Table I-5. Analysis of Variance Results for the 3-way Mixed Model for ICP using EPA Method 3050A

```
            ICP: 3-WAY MIXED MODEL ANOVA WITH ALL INTERACTIONS
                      LABORATORY: RANDOM FACTOR

------------------------------ METHOD=EPA3050A --------------------------------------


                        The MIXED Procedure

                     Class Level Information

           Class      Levels  Values

           LAB_ID        3    1 2 3
           SRMLEVEL      2    HIGH LOW
           COMPOSIT      3    1-WIPE 2-WIPE 4-WIPE


                  REML Estimation Iteration History

        Iteration  Evaluations    Objective    Criterion

              0           1    927.43114177
              1           3    662.43820134   0.00002202
              2           1    662.43129462   0.00000023
              3           1    662.43121927   0.00000000

                   Convergence criteria met.


                 Covariance Parameter Estimates (REML)

     Cov Parm              Ratio       Estimate    Std Error      Z    Pr > |Z|

     LAB_ID             5.09913705   65.66017473  72.02885617   0.91   0.3620
     LAB_ID*SRMLEVEL    0.81293052   10.46788110  10.89665288   0.96   0.3367
     LAB_ID*COMPOSIT    0.16184772    2.08406822   2.06566234   1.01   0.3130
     LAB_ID*SRMLEV*COMPOS  0.00000000  0.00000000        .       .        .
     Residual           1.00000000   12.87672289   1.41488270   9.10   0.0001


                 Model Fitting Information for ICP_REC

            Description                       Value

            Observations                    180.0000
            Variance Estimate                12.8767
            Standard Deviation Estimate       3.5884
            REML Log Likelihood            -491.111
            Akaike's Information Criterion -496.111
            Schwarz's Bayesian Criterion   -504.009
            -2 REML Log Likelihood          982.2218
```

Table I-5. (Continued)

ICP: 3-WAY MIXED MODEL ANOVA WITH ALL INTERACTIONS
LABORATORY: RANDOM FACTOR

-- METHOD=EPA3050A --

Tests of Fixed Effects

Source	NDF	DDF	Type III F	Pr > F
SRMLEVEL	1	2	5.53	0.1431
COMPOSIT	2	4	3.45	0.1346
SRMLEVEL*COMPOSIT	2	4	3.33	0.1410

ESTIMATE Statement Results

Parameter	Estimate	Std Error	DDF	T	Pr > |T|	Alpha	Lower	Upper
ICP: SRM HIGH VS LOW	-6.35108986	2.70120921	2	-2.35	0.1431	0.05	-17.9735	5.2713
ICP: SRM LOW MEAN	97.49647291	5.07607660	2	19.21	0.0027	0.05	75.6559	119.3371
ICP: SRM HIGH MEAN	91.14538304	5.07607660	2	17.96	0.0031	0.05	69.3048	112.9860
ICP: 1-W VS 2-W	-0.60513353	1.38777121	4	-0.44	0.6853	0.05	-4.4582	3.2479
ICP: 1-W VS 4-W	-3.34154735	1.38777121	4	-2.41	0.0737	0.05	-7.1946	0.5115
ICP: 2-W VS 4-W	-2.73641382	1.32176608	4	-2.07	0.1072	0.05	-6.4062	0.9334
ICP: 1-WIPE MEAN	93.00536768	4.96827414	4	18.72	0.0001	0.05	79.2112	106.7995
ICP: 2-WIPE MEAN	93.61050121	4.95024288	4	18.91	0.0001	0.05	79.8664	107.3546
ICP: 4-WIPE MEAN	96.34691503	4.95024288	4	19.46	0.0001	0.05	82.6028	110.0910

Least Squares Means

Level	LSMEAN	Std Error	DDF	T	Pr > |T|	Alpha	Lower	Upper
SRMLEVEL HIGH	91.14538304	5.07607660	2	17.96	0.0031	0.05	69.3048	112.9860
SRMLEVEL LOW	97.49647291	5.07607660	2	19.21	0.0027	0.05	75.6559	119.3371
COMPOSIT 1-WIPE	93.00536768	4.96827414	4	18.72	0.0001	0.05	79.2112	106.7995
COMPOSIT 2-WIPE	93.61050121	4.95024288	4	18.91	0.0001	0.05	79.8664	107.3546
COMPOSIT 4-WIPE	96.34691503	4.95024288	4	19.46	0.0001	0.05	82.6028	110.0910
SRMLEVEL*COMPOSIT HIGH 1-WIPE	88.73652837	5.17552717	4	17.15	0.0001	0.05	74.3670	103.1061
SRMLEVEL*COMPOSIT HIGH 2-WIPE	90.74296245	5.14085545	4	17.65	0.0001	0.05	76.4697	105.0163
SRMLEVEL*COMPOSIT HIGH 4-WIPE	93.95665832	5.14085545	4	18.28	0.0001	0.05	79.6834	108.2300
SRMLEVEL*COMPOSIT LOW 1-WIPE	97.27420700	5.17552717	4	18.80	0.0001	0.05	82.9046	111.6438
SRMLEVEL*COMPOSIT LOW 2-WIPE	96.47803997	5.14085545	4	18.77	0.0001	0.05	82.2047	110.7513
SRMLEVEL*COMPOSIT LOW 4-WIPE	98.73717175	5.14085545	4	19.21	0.0001	0.05	84.4639	113.0105

Table I-6. Analysis of Variance Results for the 3-way Mixed Model for ICP Recovery using the WOHL Method

```
        ICP: 3-WAY MIXED MODEL ANOVA WITH ALL INTERACTIONS
               LABORATORY: RANDOM FACTOR
-------------------------------------- METHOD=WOHL ----------------------------------------
```

The MIXED Procedure

Class Level Information

Class	Levels	Values
LAB_ID	3	1 2 3
SRMLEVEL	2	HIGH LOW
COMPOSIT	3	1-WIPE 2-WIPE 4-WIPE

REML Estimation Iteration History

Iteration	Evaluations	Objective	Criterion
0	1	1233.9150460	
1	1	941.32300026	0.00000000

Convergence criteria met.

Covariance Parameter Estimates (REML)

Cov Parm	Ratio	Estimate	Std Error	Z	Pr > |Z|
LAB_ID	4.08088497	99.26100538	110.42337665	0.90	0.3687
LAB_ID*SRMLEVEL	0.34261207	8.33349097	17.14295083	0.49	0.6269
LAB_ID*COMPOSIT	0.32377314	7.87526425	16.24058601	0.48	0.6277
LAB_ID*SRMLEV*COMPOS	0.88843638	21.60979519	16.71494438	1.29	0.1961
Residual	1.00000000	24.32340194	2.44459387	9.95	0.0001

Model Fitting Information for ICP_REC

Description	Value
Observations	216.0000
Variance Estimate	24.3234
Standard Deviation Estimate	4.9319
REML Log Likelihood	-663.639
Akaike's Information Criterion	-668.639
Schwarz's Bayesian Criterion	-677.006
-2 REML Log Likelihood	1327.277

Table I-6. (Continued)

```
                    ICP: 3-WAY MIXED MODEL ANOVA WITH ALL INTERACTIONS
                           LABORATORY: RANDOM FACTOR
-------------------------------------------------- METHOD=WOHL --------------------------------------------------
```

Tests of Fixed Effects

Source	NDF	DDF	Type III F	Pr > F

Tests of Fixed Effects

Source	NDF	DDF	Type III F	Pr > F
SRMLEVEL	1	2	4.87	0.1580
COMPOSIT	2	4	7.12	0.0480
SRMLEVEL*COMPOSIT	2	4	3.04	0.1575

ESTIMATE Statement Results

Parameter	Estimate	Std Error	DDF	T	Pr > |T|	Alpha	Lower	Upper
ICP: SRM HIGH VS LOW	-7.25684385	3.28759346	2	-2.21	0.1580	0.05	-21.4022	6.8885
ICP: SRM LOW MEAN	83.43261385	6.27424629	2	13.30	0.0056	0.05	56.4367	110.4285
ICP: SRM HIGH MEAN	76.17577001	6.27424629	2	12.14	0.0067	0.05	49.1799	103.1717
ICP: 1-W VS 2-W	3.29175757	3.62340879	4	0.91	0.4150	0.05	-6.7684	13.3520
ICP: 1-W VS 4-W	13.14287877	3.62340879	4	3.63	0.0222	0.05	3.0827	23.2031
ICP: 2-W VS 4-W	9.85112120	3.62340879	4	2.72	0.0531	0.05	-0.2091	19.9113
ICP: 1-WIPE MEAN	85.28240404	6.40628306	4	13.31	0.0002	0.05	67.4957	103.0691
ICP: 2-WIPE MEAN	81.99064647	6.40628306	4	12.80	0.0002	0.05	64.2040	99.7773
ICP: 4-WIPE MEAN	72.13952528	6.40628306	4	11.26	0.0004	0.05	54.3528	89.9262

Least Squares Means

Level	LSMEAN	Std Error	DDF	T	Pr > |T|	Alpha	Lower	Upper
SRMLEVEL HIGH	76.17577001	6.27424629	2	12.14	0.0067	0.05	49.1799	103.1717
SRMLEVEL LOW	83.43261385	6.27424629	2	13.30	0.0056	0.05	56.4367	110.4285
COMPOSIT 1-WIPE	85.28240404	6.40628306	4	13.31	0.0002	0.05	67.4957	103.0691
COMPOSIT 2-WIPE	81.99064647	6.40628306	4	12.80	0.0002	0.05	64.2040	99.7773
COMPOSIT 4-WIPE	72.13952528	6.40628306	4	11.26	0.0004	0.05	54.3528	89.9262
SRMLEVEL*COMPOSIT HIGH 1-WIPE	81.94296926	6.80946660	4	12.03	0.0003	0.05	63.0369	100.8491
SRMLEVEL*COMPOSIT HIGH 2-WIPE	81.66936157	6.80946660	4	11.99	0.0003	0.05	62.7633	100.5755
SRMLEVEL*COMPOSIT HIGH 4-WIPE	64.91497920	6.80946660	4	9.53	0.0007	0.05	46.0089	83.8211
SRMLEVEL*COMPOSIT LOW 1-WIPE	88.62183883	6.80946660	4	13.01	0.0002	0.05	69.7157	107.5279
SRMLEVEL*COMPOSIT LOW 2-WIPE	82.31193138	6.80946660	4	12.09	0.0003	0.05	63.4058	101.2180
SRMLEVEL*COMPOSIT LOW 4-WIPE	79.36407135	6.80946660	4	11.65	0.0003	0.05	60.4580	98.2702

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15. Supplementary Notes

16. Abstract (Limit 200 words): This study was undertaken to (1) investigate the feasibility of developing further the existing sample preparation methods analyze composite wipe samples while meeting basic data quality objectives for accuracy and precision; and (2) determine whether compositing of wipes, acceptable on a technical or performance basis, could reduce the cost of sample preparation and analysis relative to single-wipe methods. From a number of available sample preparation methods, two were selected: the modified EPA Method 3050A and the Wisconsin Occupational Health Laboratory (WOH method.

The study found that acceptable performance can be achieved using the EPA Method 3050A for composites containing two or four wipes based the recovery and precision results obtained with the standard reference material used in this study. The results were especially promising using FAA analysis. The WOHL method was found to provide acceptable performance for single wipes and two-wipe composites. It is possible to achieve acceptable performance with the WOHL method for four-wipe composites, although some difficulties might be encountered. Furthermore, the WOHL method was affected by the SRM loading level when ICP was used. A statistically significant, estimated loss in recovery of 1.75% for each gram of SRM was found when preparing the samples with the WOHL method and analyzing them by ICP. This limitation of the WOHL method may be a consideration in sampling applications where high dust loading levels are expected, such as for window sills and troughs in risk assessment. It is recommended that every laboratory would need to validate its performance with any method for the analysis of composite wipes as well as single-wipe samples. A significant cost benefit was derived from compositing. In summary, this study found that compositing two or four wipes into a single sample is a viable alternative for the analysis of lead in dust wipe samples and does result in cost savings.

17. Document Analysis   a. Descriptors

Lead, laboratory analysis, compositing, dust, wipes

b. Identifiers/Open-Ended Terms

Lead, Lead-Based Paint, Lead-Based Paint Risk Assessment, Lead-Based Paint Clearance, Analysis Costs

c. COSATI Field/Group

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