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Prepared for:
U.S. Department of Housing and Urban Development
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ABSTRACT

Previously published laboratory studies have indicated that ultrasonic extraction/anodic stripping voltammetry (UE/ASV) may be suitable for quantitative field analysis of dust wipe samples. Nevertheless, on-site lead extraction and analysis of dust wipes by UE/ASV are not currently used in federal programs for controlling and abating lead hazards in housing. A reservation to adopting UE/ASV is that the effect of the field operator (i.e., the analyst) is unknown. The availability of a reliable field test procedure for determining the amount of lead in dust could allow for on-site extraction and analysis. Thus, the U.S. Department of Housing and Urban Development (HUD) sponsored a study to evaluate the effect of operator when certified lead risk assessors or inspectors, trained to conduct UE/ASV analyses, performed such analyses of laboratory-prepared dust wipe specimens using commercial, field-portable apparatus.

Four operators analyzed 640 dust wipe specimens following a test protocol developed in accordance with the UE/ASV apparatus instructions. These specimens were prepared using four commercial wipes spiked with one of six lead-containing certified reference materials (CRMs). Six lead levels spanned a range from 0 µg to 2000 µg. After UE extraction, the solutions were either filtered or not filtered before conducting the ASV analyses. Key findings from these analyses were that lead recoveries were quite variable, ranging from < 20 % to > 100 % depending upon the combination of experimental variables, and only 42 % of the specimens spiked with a CRM afforded quantitative recovery defined as falling within the range of 100 % ± 20 %. When the entire data set was analyzed, all experimental variables had a significant effect on recovery. The majority of the two-way interaction effects were also significant. The operator effect was essentially associated with three of the four operators determining higher lead recoveries than the fourth. The three operators reporting the higher recoveries followed a test protocol that was a slight modification of that used by the fourth operator. It was not determined whether this modification accounted for the observed operator effect.

To provide data regarding the role of extraction and ASV measurement in the low recoveries, the lead concentrations of a limited number of UE extracts were analyzed using inductively coupled plasma (ICP) atomic emission spectrometry. A key finding was that wipe type had an effect on lead recovery when determined using either ICP or ASV analyses of the UE extract solutions. In addition, one wipe may have produced an interferant(s) to ASV analysis.

Based on the study results, a main conclusion is that the UE/ASV test procedure lacked robustness for analyzing dust wipe specimens. That is, it was found that changes in the experimental variables incorporated in the study design could lead to low lead recoveries. Future investigations should address improving robustness.

Key Words: analysis; anodic stripping voltammetry (ASV); building technology; dust wipes; lead-containing dust; lead recovery; operator effect; testing; ultrasonic extraction (UE)

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1. INTRODUCTION

1.1 Background

Lead in dust is a major source of lead exposure in young children [1]. Consequently, sampling and lead analysis of settled dust is integral to identifying, abating, and controlling hazards associated with the presence of lead in and around housing. Sampling is generally performed by manually wiping a surface with a disposable towellette, called a dust wipe, that has been moistened with a wetting agent. ASTM E 1728, "Standard Practice for Collection of Settled Dust Samples Using Wipe Sampling Methods for Subsequent Lead Determination," describes standardized surface-wiping methods [2]. ASTM E 1792, "Standard Specification for Wipe Sampling Materials for Lead in Surface Dust," sets performance requirements for dust wipes [3].

Analytical methods currently used for extraction and analysis of lead in dust wipes are not readily field-portable. Thus, there is a need for a method that provides on-site, quantitative results. For example, in a situation where a housing unit has been vacated during lead-abatement activities, re-occupancy might be allowed sooner if on-site dust analyses, among other indicators, show the unit to be safe for reentry than if the wipes were sent to a laboratory.

On-site lead extraction and analysis of dust wipes are not currently used in federal programs for identifying, abating, and controlling lead hazards in housing. The availability of a reliable field test procedure for determining the amount of lead in dust could allow for re-examination of this limitation. Results of previous research have suggested that field portable ultrasonic extraction/anodic stripping voltammetry (UE/ASV) has the potential for such use [4-7]. In 1999, Ashley, Song, Esch, Schlecht, Baron, and Wise [4] directed an interlaboratory evaluation of the suitability of UE/ASV for measuring lead in environmental media including dust. A main conclusion was that the UE/ASV procedure was effective in quantifying lead in the media examined. In a follow-up 2001study, Ashley, Wise, Mercado, and Parry [5] spiked four commercial dust wipes with one of four lead-containing powdered Certified Reference Materials* (CRMs) including three NIST† Standard Reference Materials (SRMs). After extracting lead from the wipes using ultrasonication, the UE extracts, either with or without filtering through 0.45 µm hydrophilic polytetrafluoroethylene (PTFE) filters, were subjected to ASV analyses. Filtration was included as an experimental variable to examine whether it enhanced lead recovery through removal of organic material (introduced from the wipe), including surfactants or other potential ASV interferants‡ that were possibly present in the wipes. Ashley et al. [5] found that all four CRMs without dust wipes present provided quantitative lead recovery (i.e., within the range of 100 % ± 20 %) either with or without filtration of the extract solutions. In contrast, lead recovery from dust wipe specimens varied with the combinations of wipe, CRM, and filter treatment. Specifically, they found that, for one wipe, recoveries varied depending upon the combination of wipe and CRM, were generally less than quantitative, and were not improved by filtering. For two other wipes, recoveries also varied depending upon the combination of wipe and CRM, and were generally less than quantitative unless filtering was performed. For the fourth wipe, no matter the CRM used, recovery was always quantitative with or without filtering. The authors [5] concluded that three

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* A Certified Reference Material is "a reference material accompanied by a certificate, one or more of whose property values are certified by a procedure that establishes its traceability to an accurate realization of the unit in which the property values are expressed" [8]. CRM is the generic term used in this report to denote all certified reference materials including NIST Standard Reference Materials (SRMs).

† National Institute of Standards and Technology

‡ An interferant is a species in the sample that affects the results (i.e., causes interference) of the analyte measurement. The interferant may not prevent the analyte measurement, but causes a bias to the measurement of the analyte concentration.

wipes were suitable for UE/ASV provided that filtering of the UE extract was always included in the test procedure. The recommendation for universal filtering was made as a precautionary measure to maximize the chance of quantitative recovery.

Recently, the U.S. Environmental Protection Agency (EPA) published the results of two similar studies that examined the performance of ASV methods for analyzing lead in dust wipe specimens [6,7]. These studies were conducted under EPA's Environmental Technology Verification (ETV) Program, which has as its mission "to facilitate the deployment of innovative or improved environmental technologies through performance verification..." In these studies, directed by Oak Ridge National Laboratory (ORNL), two commercial ASV instruments were used to determine recoveries from wipes spiked with lead-containing CRMs. The ASV-measured recoveries were compared with those measured by a National Lead Laboratory Accreditation Program (NLLAP) laboratory [9] using a referee measurement method. Other parameters used to evaluate performance included precision, and the determination of false positive and false negative results at regulatory-mandated lead levels. For one apparatus, it was reported that "the overall performance [of the instrument] for the analysis of lead in dust wipe samples was characterized as having an acceptable amount of negative bias, very precise, and has a strong linear relationship with the NLLAP-laboratory results" [6]. For the other apparatus, it was reported that "the overall performance [of the instrument] for the analysis of lead in dust wipe samples was characterized as biased low but within acceptable levels of bias, having greater than acceptable levels of variability, and in good linear agreement with the average results reported by the NLLAP laboratory" [7].

The U.S. Department of Housing and Urban Development (HUD) has interest in test methods that may be used to determine lead contents at housing sites. One reservation to adopting field portable test methods is that the effect of operator (i.e., the analyst) on the reliability of the field analyses is generally not known or demonstrated in analytical studies carried out to evaluate the technology. Lead risk assessors and inspectors, who are potential users of field methods (i.e., the operators), may have less skill in conducting lead analyses than chemists and laboratory technicians who participate in evaluation studies. In response to concerns over operator effects on field test reliability, NIST has performed HUD-sponsored studies to examine operator effects on spot test kits and on ASV analyses for detecting and determining lead in paint in the field [10-13]. Until the present study, operator effect on the reliability of ASV analyses of dust wipe specimens had not been addressed in an experiment in which lead risk assessors or inspectors performed the analyses. Operators conducting the dust-wipe tests in the studies cited above [4-7] were generally trained professionals who may be arguably atypical of the risk assessors and inspectors who would use the ASV dust wipe method in practice. For example, one of the ETV reports [6] indicated "the analyst who operated the instrument during the verification test was a … expert." The other ETV report [7] stated that "during the test, one analyst that operated the [instrument] was an expert, and the other was a novice."

1.2 Objective and Scope of the Study

The objective of the study was to evaluate the effect of operator when commercial, field-portable ultrasonic extraction/anodic stripping voltammetry (UE/ASV) was used for quantitatively determining the lead level of laboratory-prepared dust wipe specimens in testing performed by certified lead risk assessors or inspectors trained to conduct UE/ASV analyses. Four other experimental variables were also examined: dust wipe, CRM type, lead level, and filter treatment of the UE extract before ASV analysis. Table 1 lists the five experimental variables. In this report, the phase of the study in which lead risk assessors or inspectors conducted the analyses of the dust wipe specimens is referred to as the "Main Experiment." In addition to the Main Experiment, a limited

Table 1. Experimental variables

Experimental Variable	Description	Comment
Operator	Four operators: • Operator 1 (Op 1) • Operator 2 (Op 2) • Operator 3 (Op 3) • Operator 4 (Op 4)	Four certified lead inspectors and risk assessors, previously trained to perform UE/ASV analyses of paint samples, conducted the dust wipe analyses. They were further trained at NIST to conduct dust wipe analyses.
Dust Wipe	Four commercial dust wipes: • Wipe 1 • Wipe 2 • Wipe 3 • Wipe 4	The dust wipes were reported by the suppliers to meet ASTM Specification E 1792-96, "Wipe Sampling Materials for Lead in Surface Dust" [3]. E 1792-96 was the version of the standard specification that was in effect at the time when the study was designed.
Certified Reference Material (CRM)	Six certified reference materials: • SRM 2580 • SRM 2581 • SRM 2582 • SRM 2584 • SRM 2586 • CRM 01450	In designing the study, it was originally intended to include four CRMs (i.e., SRM 2581, SRM 2584, SRM 2586, and CRM 01450). However, it was not practical to use only four CRMs. With the wide range of lead levels targeted in the experimental design (i.e., 40 µg to 2000 µg), the amount of CRM to be spiked onto a wipe would be too little in the case of SRM 2584 at the lead level of 40 µg, and excessive in the case of SRM 2586 at the lead level of 2000 µg. Table 2 summarizes the specimen mass at which each CRM was spiked onto the wipes for the various lead levels. Table 3 lists the CRM types, the percent lead by mass fraction, and a summary of the results of multiple UE/ASV analyses of each CRM.
Lead Level	Six lead levels: • 0 µg • 40 µg • 90 µg • 250 µg • 500 µg • 2000 µg	Lead levels spanned the range from 0 µg to 2000 µg, with the 0 µg level being the designation assigned to wipes that were not spiked with CRM. These lead levels bracketed the range of interest in federal regulations on lead hazard abatement. In designing the study, it was originally planned to include five lead levels (no 90 µg level). However, it was considered that the small amount of SRM 2581 (i.e., ≈ 9 mg) that would have been weighed on a wipe at the 20 µg level might result in mass measurements having relatively large uncertainty. Thus, for SRM 2581, the 90 µg lead level was included in lieu of the 40 µg lead level (Table 2).
Filter Treatment Before Extract Analysis	Two treatments of the UE extract before ASV analysis: • No filtering (i.e., common practice) • Filtering	Ashley et al. [5] have recommended that filtering of the UE extract solution should be performed before ASV analysis, because it may enhance lead recovery by removing ASV interferants from the extract solution. Consistent with their recommendation, filtering through a 0.45 µm hydrophilic PTFE filter was included in the experimental design.

number of UE extracts was analyzed using inductively coupled plasma (ICP) atomic emission spectrometry. The results of these measurements were compared with the ASV results obtained in the Main Experiment. This phase of the study is referred to as the "ASV/ICP Comparison."

2. EXPERIMENTAL DESIGN

A full factorial experimental design that included four operators, four dust wipes, four CRM Types, five lead levels, and two filtering treatments of the UE extract solution was planned for the investigation. Practical constraints such as availability and percent lead were taken into account in selecting the CRMs. The four CRMs initially considered for the experiments were SRM 2581, SRM 2584, and SRM 2586, and CRM 01450. These included two dust samples (SRMs 2584 and CRM 01450), a powdered paint sample (SRM 2581) and a powdered soil sample (SRM 2586). Lead-containing paints and soils can be a source of lead in dust in housing. Of the four CRMs considered for the design, only CRM 01450 had a lead content (i.e., percent lead by mass fraction) that made it practical to prepare dust wipe specimens having lead levels spanning the selected range of 40 µg to 2000 µg (Table 1). If SRM 2584 were used at the 40 µg lead level, only about 4 mg would have been spiked onto the wipes. Consequently, SRM 2582 was included in the design for the 40 µg lead level. Similarly, use of SRM 2586 at the 2000 µg lead level would have required depositing about 4.5 g onto a wipe. In this case, SRM 2580 was included for the 2000 µg lead level. In addition, if SRM 2581 were used at the 40 µg lead level, less than 10 mg would have been deposited onto a wipe. Consequently, 90 µg was the lowest lead level incorporated in the design for SRM 2581. Table 2 summaries the amount of CRM spiked onto a wipe for each combination of CRM type and lead level. Note that 20 mg was the least amount of CRM used in the design.

The number of UE/ASV analyses conducted by one operator in a test series was 186. Each series was comprised of 160 randomly sequenced dust wipe specimens, 13 control specimens, and 13 method blanks. A set of seven dust wipe specimens could be simultaneously extracted using the sonicator supplied with the ASV instrument. For each test series, every set of seven specimens included either a control or a method blank, with the first sonication set having a control. The controls were samples of CRM 01450 (about 100 mg) that were placed in sonicator tubes without a wipe present. They were included as a periodic check that analyses of specimens without a wipe present provided recoveries that were within acceptable bounds, when the tests were performed using the UE/ASV dust wipe protocol (Section 3.3). Method blanks consisted of performing the UE/ASV procedure without a specimen. They were used to monitor background levels and check for contamination.

Table 2. Mass of CRM spiked onto the wipes at each lead level

CRM Designation	Targeted Mass of CRM Spiked onto the Wipes at Each Lead Level				
	Lead Level 40 µg	Lead Level 90 µg	Lead Level 250 µg	Lead Level 500 µg	Lead Level 2000 µg
SRM 2580	---[a]	---	---	---	46 mg
SRM 2581	---	20 mg	56 mg	111 mg	445 mg
SRM 2582	192 mg	---	---	---	---
SRM 2584	---	---	26 mg	51 mg	205 mg
SRM 2586	93 mg	---	579 mg	1157 mg	---
CRM 01450	21 mg	---	131 mg	261 mg	1045 mg

[a] The dash indicates that specimens were not prepared for the combination of CRM and lead level.

3. EXPERIMENTAL

3.1 UE/ASV Apparatus

A commercial, field-portable UE/ASV apparatus* was used in the study. It was designated Apparatus 1 in the initial NIST UE/ASV study [11].

3.1.1 ASV Instrument. The field-portable ASV instrument is battery operated, uses disposable electrodes, and is factory-calibrated. Results of analyses of dust wipe specimens are given in units of micrograms (µg). The range of detection for lead in dust wipe specimens reported in the instrument instruction booklet is 25 µg to 1500 µg. For specimens found to have lead levels exceeding this range, dilution of the UE extract is performed before ASV re-analysis. The uncertainty of the ASV measurements based on the pooled standard deviation of the results of all analyses of CRM specimens using the protocol in Section 3.3 but without a dust wipe present (summary given in Table 3) was estimated to be 7.3 %. Lead recovery (LR %) measured in an UE/ASV analysis of a dust wipe specimen was calculated using the following formula:

$$LR\ \% = (\text{UE/ASV Result/Estimated True Lead Value}) \times 100.$$

Factory calibration of the ASV instrument was checked in the previous NIST UE/ASV paint studies [11-13]. A repeat calibration check performed at the beginning of the current study using six standardized lead nitrate solutions (7.5 % volume fraction nitric acid with lead contents ranging from 1 mg/L to 28 mg/L) confirmed that the instrument has remained in calibration. The ASV response versus lead concentration was linear ($r^2 = 0.998$). In addition, at the beginning and end of each day's analyses, operators performed two calibration checks using two standardized solutions of lead nitrate (7.5 % volume fraction nitric acid with lead contents of 1 mg/L and 28 mg/L). These daily checks indicated that the ASV instrument remained in calibration, and that instrument drift over the course of the day did not occur.

3.1.2 Sonicator. The commercial sonicator is specified as having an average power of 45 W. The bath dimensions are 146 mm by 133 mm by 100 mm (length, width, and depth).

Table 3. Certified reference materials (CRMs) used in the study

Certified Reference Material			Results of UE/ASV Analyses[a]					
		Lead Mass Fraction		Lead Recovery, %				CoV[d]
Designation	Material Type	%	n[b]	Min	Max	Mean	SD[c]	%
SRM 2580	Powdered Paint	4.34	6	90.1	96.3	93.0	2.1	2.2
SRM 2581	Powdered Paint	0.449	8	57.9	96.7	85.6	12.1	14.2
SRM 2582	Powdered Paint	0.02088	6	92.2	97.7	94.4	1.8	1.9
SRM 2584	Indoor Dust	0.9761	8	74.2	92.6	83.7	6.5	7.8
SRM 2586	Powdered Soil	0.0432	6	89.4	104.6	95.5	5.3	5.5
CRM 01450	Bag House Dust	0.1914	8	85.6	109.6	100.2	8.3	8.3

[a] The analyses were performed using the protocol in Section 3.3; the CRM specimens were prepared without dust wipes.
[b] Number of analyses.
[c] SD indicates standard deviation.
[d] CoV indicates coefficient of variation (standard deviation/mean*100)

* In this report, the term, "apparatus," refers to the combination of UE sonicator and ASV electrochemical instrument that was purchased from a single supplier as part of a field-portable kit. For the individual pieces of equipment, the terms, "[UE] sonicator" and "[ASV] instrument" are used.

3.2 Dust Wipe Specimens

The dust wipe specimens were prepared by the Research Triangle Institute (RTI)* using a gravimetric procedure in which one of the six CRMs (Table 2) was loaded onto one of four commercial, individually packaged dust wipe products. RTI has used the gravimetric procedure for preparing dust wipe specimens for use in the Environmental Lead Proficiency Analytical Testing (ELPAT) Program [14]. Gravimetrically prepared specimens were also used in the EPA ETV studies on UE/ASV analysis of dust wipe specimens [6,7].

Table 3 lists the CRM types, which were in general different from those used in the previously referenced studies [4-7]. Table 3 also summarizes the results of UE/ASV analyses conducted on specimens prepared without dust wipes and analyzed without filtering of the UE extracts. The mean recovery for each of the six CRMs was quantitative.

RTI furnished 640 dust wipe specimens for analysis by the 4 operators in the Main Experiment. An additional 35 specimens were prepared for quality control analysis by RTI. In preparing a dust wipe specimen, RTI deposited onto a wipe the appropriate amount of CRM as specified in the experimental design (Table 2). Only one analytical balance was used for all CRM weighings. A single 0.1 g Class 1 weight was used for a daily calibration check before and after each weighing period. Prior to weighing, the bulk container of CRM was tumbled several times. Floating dust in the container was then allowed to settle before opening it. CRM was taken using a spatula and transferred to a tared sheet of weighing paper. If more CRM was needed, it was taken from the bulk container and added to the material on the weighing paper. If excess CRM was placed on the paper, it was carefully removed with the tip of the spatula and discarded. Wipes were prepared for receiving the CRM by opening the packaging, removing the wet folded wipe, and squeezing excess moisture out by hand over a trash can. The wipe was then unfolded and briefly set on a laboratory paper towel that soaked up excess moisture. The wipe was then transferred to a flat plastic board to await the CRM. The weighing paper containing the pre-weighed CRM was removed from the balance and the CRM was gently tapped onto the center of the wipe. The wipe was then folded and placed in a 50 mL plastic centrifuge tube, which was capped and labeled. The amount of lead in each dust wipe specimen (i.e., estimated true value) was taken as the mass of CRM weighed onto a wipe multiplied by the certified % mass fraction lead in the CRM.

All tubes containing the dust wipe specimens were stored at RTI in a cold room to retard possible mold growth until shipment to NIST. Upon arrival at NIST, they were kept in a refrigerator until about 48 h before use during which time they warmed to room temperature.

The 35 dust wipe specimens analyzed by RTI were typical combinations of wipe, CRM type, and lead level used to prepare the specimens analyzed by the four operators in the Main Experiment. The RTI analyses were performed in accordance with the acid digestion process and inductively coupled plasma (ICP) atomic emission spectrometry method by which RTI has measured lead in dust wipe specimens included in the ELPAT Program [14]. Based on experience from the ELPAT Program, expected lead recoveries from such analysis of gravimetrically prepared dust wipe specimens are, on the average, about 87 % or 88 %†. The results of the present RTI analyses are

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* Certain trade names or company products are mentioned in the text to specify adequately the test specimens, experimental procedure and equipment used. In no case does such identification imply recommendation or endorsement by the National Institute of Standards and Technology, nor does it imply that the equipment is the best available for the purpose.

† Personal communication from RTI's Dr. David Binstock.

given in Table 4, which shows that the lead recoveries ranged from 75 % to 118 %. The mean recovery for all analyses was 92 % (11.5 % CoV*), which was consistent with past ELPAT experience.

Table 4. Results of RTI analyses of selected dust wipe specimens

Specimen ID	Wipe No.	CRM No.	Lead Level µg	Lead Recovery %
RTI S01	1	SRM 2581	90	75.3
RTI S02	1	SRM 2581	500	86.6
RTI S03	1	SRM 2584	500	102.2
RTI S04	1	SRM 2584	2000	85.6
RTI S05	1	SRM 2586	40	93.5
RTI S06	1	SRM 2586	250	76.0
RTI S07	1	CRM 01450	40	89.8
RTI S08	1	CRM 01450	500	111.8
RTI S09	1	CRM 01450	2000	101.9
RTI S10	2	SRM 2580	2000	84.6
RTI S11	2	SRM 2581	90	87.8
RTI S12	2	SRM 2581	500	102.7
RTI S13	2	SRM 2581	2000	98.3
RTI S14	2	SRM 2582	40	78.0
RTI S15	2	SRM 2584	250	76.7
RTI S16	2	SRM 2586	250	79.6
RTI S17	2	CRM 01450	40	102.0
RTI S18	2	CRM 01450	2000	103.5
RTI S19	3	SRM 2580	2000	79.5
RTI S20	3	SRM 2581	90	87.8
RTI S21	3	SRM 2581	250	82.7
RTI S22	3	SRM 2584	250	78.6
RTI S23	3	SRM 2584	500	97.2
RTI S24	3	SRM 2586	500	97.2
RTI S25	3	CRM 01450	250	101.5
RTI S26	3	CRM 01450	500	118.7
RTI S27	4	SRM 2581	90	88.4
RTI S28	4	SRM 2581	250	82.1
RTI S29	4	SRM 2581	2000	92.6
RTI S30	4	SRM 2582	40	75.1
RTI S31	4	SRM 2584	2000	97.1
RTI S32	4	SRM 2586	40	110.1
RTI S33	4	SRM 2586	500	91.9
RTI S34	4	CRM 01450	40	104.0
RTI S35	4	CRM 01450	250	101.3

* Coefficient of variation (std. dev./mean x 100).

3.3 UE/ASV Protocol

The operators, who were lead risk assessors and inspectors, performed the analyses at NIST. Their work schedules determined the sequence in which each came to NIST. They performed the analyses according to a written protocol developed from the instructions for the UE/ASV apparatus (Table 5). The protocol was reviewed by the UE/ASV supplier to ensure that the extraction and analysis procedures were consistent with the apparatus instructions. Additional steps were included for completeness. For example, the supplier's instructions were not specific regarding steps to be taken in selecting and labeling the test specimens, and did not address formats for recording data. One deviation from the apparatus instructions was that the extraction was performed with warm tap water (≈ 45 °C) in the sonicator bath. Use of warm water was based on the results of previous NIST studies showing that lead recovery from laboratory-prepared paint film specimens was enhanced at higher bath temperatures [12,13]. Another deviation was that, for half of the specimens tested by an operator, the extract solutions were filtered using a Millipore PTFE 0.45 mm filter. This procedure was incorporated to be consistent with the recommendation of Ashley et al. [5] on filtering UE extracts to help ensure reliable ASV analysis.

All operators had been previously trained (receiving a certificate) to conduct UE/ASV analyses. For the current study, before beginning a series of tests, NIST research staff reviewed the dust wipe protocol with each operator. Importance was placed on the procedures in the protocol that differed from those for paint analysis such as Step 2d and Step 3e (Table 5) that focused on mixing the specimen in the acid solution. "Training specimens" were provided to the operators.

3.4 NLLAP Analysis

During the course of the operators' analyses, aliquots (≈ 20 mL) of selected UE extract solutions were sent to a National Lead Laboratory Accreditation Program (NLLAP) [9] laboratory that was accredited for lead analysis. Lead concentration was determined using inductively coupled plasma (ICP) atomic emission spectrometry according to EPA Method 6010B [15].

4. RESULTS, ANALYSIS, AND DISCUSSION

4.1 Controls and Blanks

Figure 1 shows the UE/ASV results for the CRM 01450 control specimens in the sequence in which the operators performed the analyses. All recoveries were quantitative with slightly more than 80 % of the tests yielding recoveries between 100 % to 110 %. The mean recovery was 106 % (5.3 % CoV), which was not statistically different (0.05 level) from the mean value for CRM 01450 (i.e., 103 %) determined in the preliminary stage of the investigation (Table 3). Analyses of all method blanks gave results that were "below the detection limit," indicating that measurable contamination had not occurred with these specimens.

Table 5. Summary of the steps of the UE/ASV protocol used for analyzing dust wipe specimens

	Step[a]		Summary of the Activities for Each Step
1.	Specimen Selection	a.	Obtain a sample tube from the storage rack following the sequence of "Test Sample ID Numbers" on the data form.
		b.	Check that the "Test Sample ID Number" on the data form is the same as that on the tube.
		c.	Write the "Test Sample ID Number" and the "Test Sample Letter Code" on the tube cap.
2.	Specimen Acidification	a.	Place a new (clean) disposable tip on the 5 mL pipettor.
		b.	Using the pipettor, add 15 mL (3 x 5 mL) of 25 % volume fraction nitric acid to the sample tube; dispose of the pipettor tip.
		c.	Write the "Test Sample ID Number" on a new (clean) plastic sample-stirring rod.
		d.	Use this stirring rod to push the dust wipe down into the sonicator tube. Ensure that the dust wipe is covered with acid solution. Keep pushing the wipe down into the acid solution until trapped air bubbles in the wipe have been released and until the acid solution appears cloudy from the dust residue.
		e.	Set aside the stirring rod for re-use ensuring that it is kept clean.
		f.	Re-cap the sonicator tube.
3.	Specimen Extraction Using the Sonicator	a.	Add warm (≈ 45 °C) tap water to the ridge (i.e. mark) in the sonicator bath.
		b.	Position the cover on the sonicator bath, and place the specimen tube into the randomly selected (i.e., numbered) hole such that the tube cap rests on the bath cover.
		c.	Place a weight on top of the sonicator tube to prevent floating.
		d.	Sonicate the specimen for 30 min and remove it from the bath.
		e.	Take the previously used stirring rod (ensure that it correctly corresponds to the tube sample number) and repeat the stirring of the dust wipe in the sample tube.
		f.	Re-cap the sample tube and replace it in the bath; set the stirring rod aside such that it will not be contaminated.
		g.	Sonicate for an additional 15 min.
		h.	Take the sonicated specimen/tube and, after removing the cap, carefully add distilled water to the 50 mL mark; do not fill over this mark during dilution.
		i.	Replace the cap on the sample tube and mix the diluted extract well by shaking.
		j.	Take the same stirring rod as previously used for the given specimen and thoroughly stir the wipe and solution to ensure mixing of the diluted extract.
		k.	Replace the cap on the sonicator tube and shake the tube vigorously for at least 15 s.
		l.	Allow the diluted extract solution to cool to room temperature before ASV analysis.
4.	Preparation of Extract Solution for Analysis	a.	Add an electrolyte tablet into a 5 mL analysis vial; crush the tablet with a tablet-crushing rod; using an indelible marking pen, mark the "Test Sample ID Number" (written on the sample tube) on the outside of the analysis vial.
		b.	Take a 10 mL disposable plastic syringe and draw 5 mL of the diluted extract solution from the sample tube into the syringe.
		c.	Check the data form to determine whether the diluted extract solution is to be filtered.
		d.	If the solution is not to be filtered, dispense the diluted extract solution directly from the syringe into the ID-marked analysis vial, filling to the 5 mL mark.
		e.	If the solution is to be filtered, place a Millipore PTFE 0.45 mm filter on the tip of the syringe; dispense the diluted extract solution from the syringe through the filter and into the ID-marked analysis vial, filling to the 5 mL mark.
		f.	Re-cap the analysis vial and gently shake to ensure that the crushed tablet is dissolved.
5.	Extract Analysis	a.	Place a disposable electrode into the electrode connector; grip the electrode by the edges or through the foil packaging only; do not touch exposed electrodes.
		b.	Perform the lead analysis according to the instructions for operation of the ASV instrument.
		c.	Record the analysis result (in µg) on the NIST-provided data form along with the analysis ID number assigned by the ASV instrument.
		d.	If the analysis result is "above the instrument range," using the 1 mL pipettor, dilute the extract solution (1 mL with 4 mL of 7.5 % volume fraction nitric acid), either with or without filtering as indicated on the data form, and repeat the analysis; record the result of this repeated analysis on the data form.

[a] In following the protocol, each step was performed on a set of seven dust wipe specimens, because seven specimens can be extracted simultaneously using the sonicator supplied with the ASV instrument.

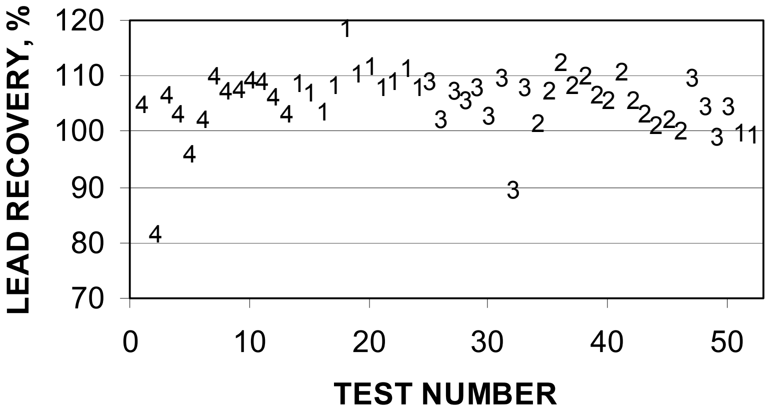

Figure 1. Results of the UE/ASV Analyses of the CRM 01450 Controls, Which Were Prepared Without Dust Wipes Present. The data are given in the sequence in which the operators performed the tests. The data symbol is the operator number.

4.2 The Main Experiment

4.2.1 Results. The results of the UE/ASV analyses of the dust wipe specimens for Operators 1 through 4 are plotted in Figures 2 through 5, respectively. Each plot shows lead recovery (%) versus specimen lead level for a given combination of operator and wipe. The plot symbols denote analyses performed with and without filtering of the extract solution. Tables 6 through 9 include lead recovery for all combinations of experimental variables for each of the four wipes. For those readers who have interest in the effects of combinations of operator, wipe, and CRM type, Appendix A summarizes lead recovery for those combinations.

4.2.2 Analysis and Discussion. The data in Figures 2 through 5, and Tables 6 through 9 indicate that lead recovery was quite variable and depended on the combination of experimental variables. This finding was consistent with that of Ashley et al. [5], who found variable recovery depending upon the combination of wipe, CRM, and filter treatment used in the analysis. Most notable in the current study, lead recovery for a majority of the dust wipe specimens was less than quantitative. This finding was in marked contrast with previously published results [4-7] wherein the majority of the UE/ASV analyses were quantitative, particularly when filtering was used. The low recoveries in the current study also contrasted with the results of the analyses of CRM specimens prepared without wipes that were, on average, always quantitative (Table 3).

For the 512 CRM-containing specimens analyzed by the operators, quantitative recovery occurred for 213 specimens (i.e., 42 %) and varied among the operators (statistical analysis follows below). Operators 1 and 3 had the highest percent quantitative recovery; 52 % of their analyses provided quantitative results. Operator 2 was intermediate at 43 %, while Operator 4 was lowest at 20 %. The mean recovery for all specimens* was 74 % with values for specimens analyzed by Operators 1 through 3 ranging from 75 % to 80 %. In contrast, the mean recovery for Operator 4 was significantly less (0.05 level) at 63 %.

* Not including those having results recorded as "below the detection limit."

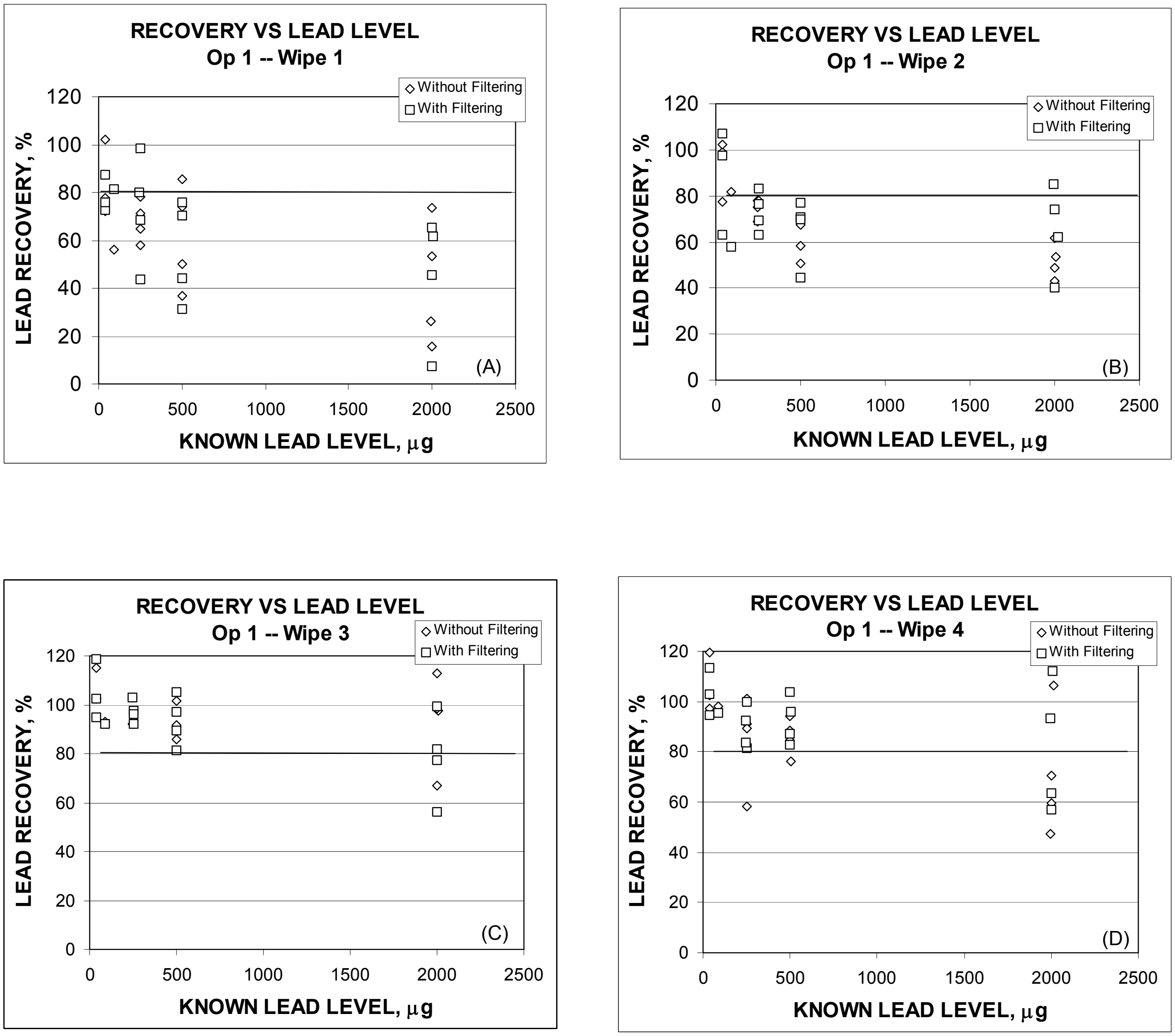

Figure 2. Results for Operator 1 Using: (A) Wipe 1, (B) Wipe 2, (C) Wipe 3, and (D) Wipe 4. Data points above the horizontal bold line at 80 % recovery represent quantitative recovery.

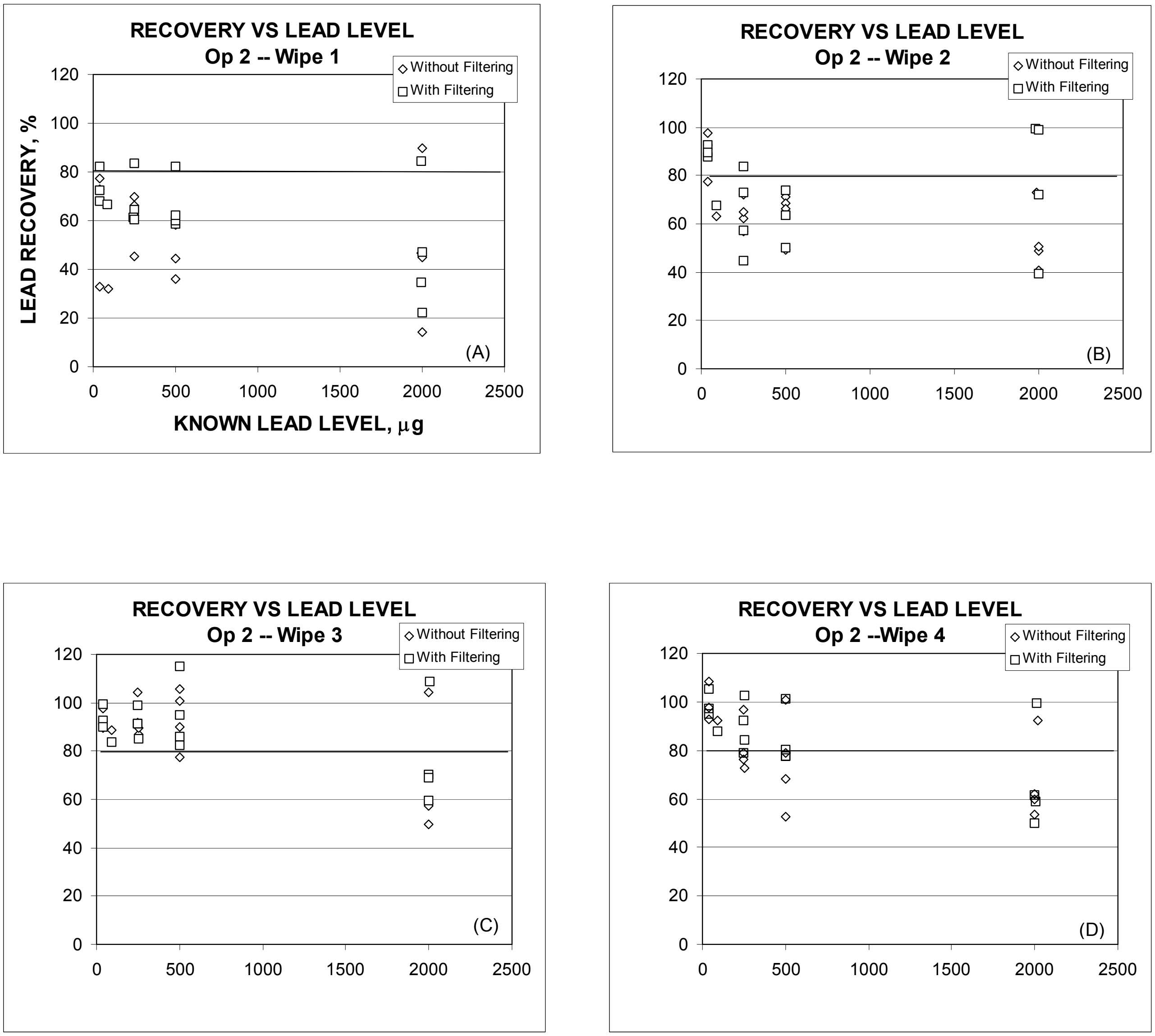

Figure 3. Results for Operator 2 Using: (A) Wipe 1, (B) Wipe 2, (C) Wipe 3, and (D) Wipe 4. Data points above the horizontal bold line at 80 % recovery represent quantitative recovery.

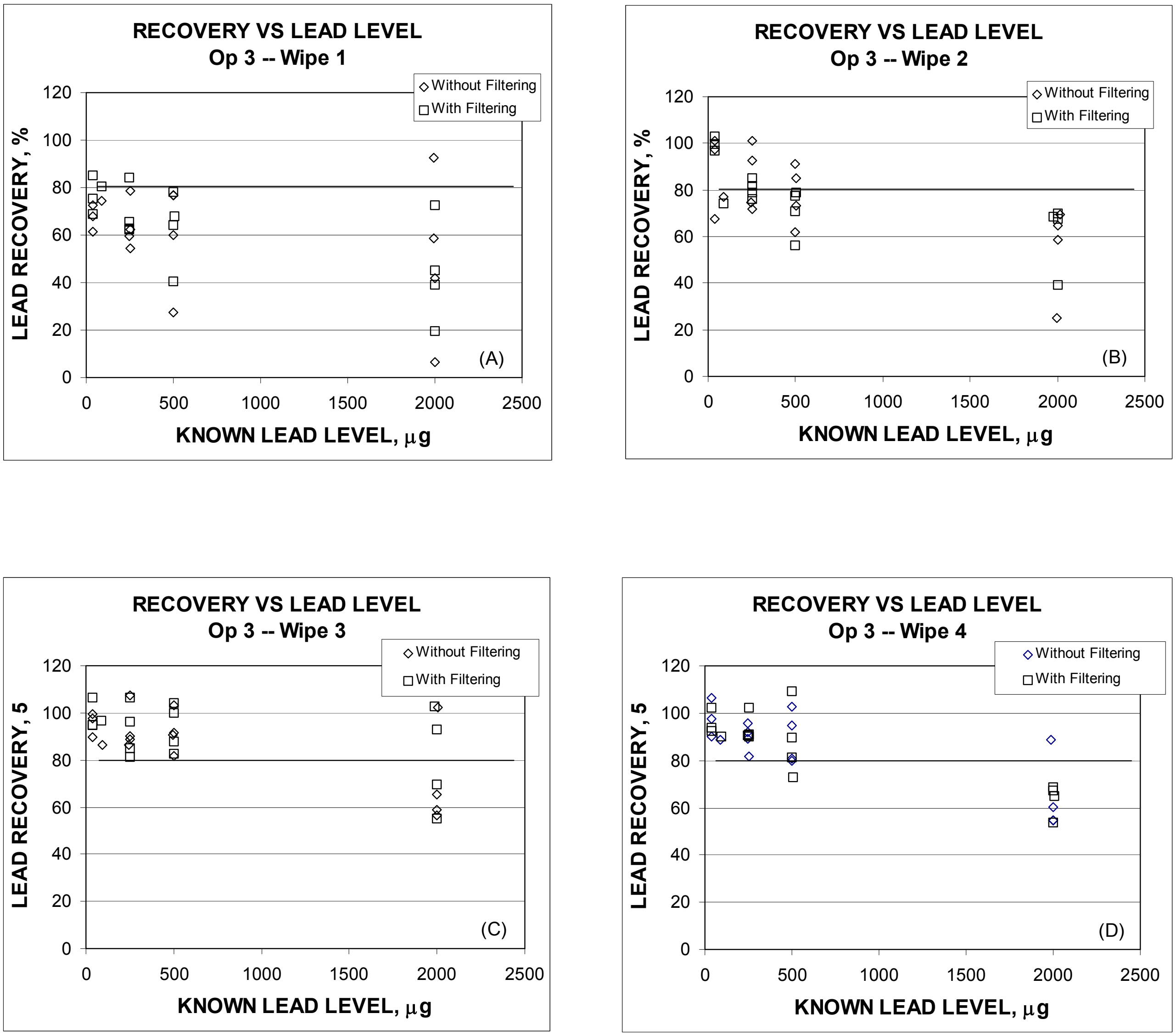

Figure 4. Results for Operator 3 Using: (A) Wipe 1, (B) Wipe 2, (C) Wipe 3, and (D) Wipe 4. Data points above the horizontal bold line at 80 % recovery represent quantitative recovery.

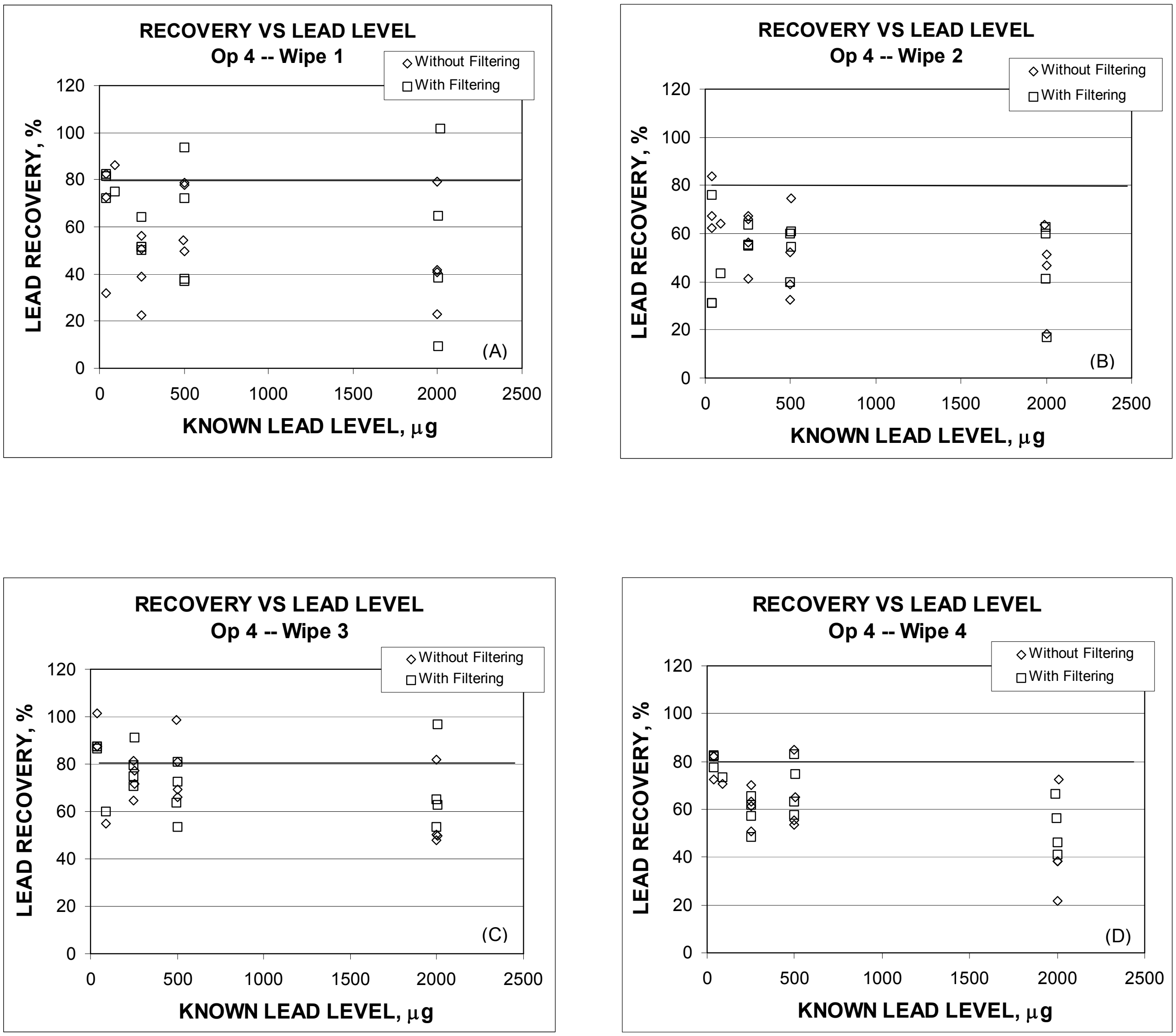

Figure 5. Results for Operator 4 Using: (A) Wipe 1, (B) Wipe 2, (C) Wipe 3, and (D) Wipe 4. Data points above the horizontal bold line at 80 % recovery represent quantitative recovery.

Table 6. Lead recovery for each combination of experimental variables for Wipe No. 1

Wipe No.	CRM No.	Lead Level[a] µg	Filter Treatment	UE/ASV Lead Recovery[b], %			
				Op 1	Op 2	Op 3	Op 4
1	SRM 2580	2000	With Filtering	65.3	84.7	92.4	79.4
1	SRM 2580	2000	Without Filtering	73.6	90.0	72.7	101.6
1	SRM 2581	0	With Filtering	<	<	<	<
1	SRM 2581	0	Without Filtering	<	<	<	<
1	SRM 2581	90	With Filtering	81.3	32.0	80.7	86.2
1	SRM 2581	90	Without Filtering	56.0	66.7	74.2	75.0
1	SRM 2581	250	With Filtering	98.2	45.4	54.6	51.4
1	SRM 2581	250	Without Filtering	78.3	83.8	84.1	38.7
1	SRM 2581	500	With Filtering	70.4	82.0	78.3	54.5
1	SRM 2581	500	Without Filtering	85.4	58.1	76.6	93.5
1	SRM 2581	2000	With Filtering	45.5	46.9	45.3	41.5
1	SRM 2581	2000	Without Filtering	53.5	44.8	41.9	64.6
1	SRM 2582	40	With Filtering	87.3	72.4	84.9	82.3
1	SRM 2582	40	Without Filtering	72.4	<	72.5	82.4
1	SRM 2584	0	With Filtering	<	<	<	<
1	SRM 2584	0	Without Filtering	<	<	<	<
1	SRM 2584	250	With Filtering	79.8	66.3	62.2	64.2
1	SRM 2584	250	Without Filtering	64.9	61.4	78.7	56.3
1	SRM 2584	500	With Filtering	75.7	44.3	76.8	78.7
1	SRM 2584	500	Without Filtering	74.0	58.6	67.7	72.1
1	SRM 2584	2000	With Filtering	61.6	46.6	39.3	38.6
1	SRM 2584	2000	Without Filtering	26.0	34.6	58.6	40.7
1	SRM 2586	0	With Filtering	<	<	<	<
1	SRM 2586	0	Without Filtering	<	<	<	<
1	SRM 2586	40	With Filtering	72.7	67.8	67.7	72.3
1	SRM 2586	40	Without Filtering	77.9	77.2	75.2	72.5
1	SRM 2586	250	With Filtering	68.4	60.4	59.6	50.0
1	SRM 2586	250	Without Filtering	58.0	62.4	65.6	50.8
1	SRM 2586	500	Without Filtering	50.0	59.8	40.4	49.6
1	SRM 2586	500	With Filtering	44.0	59.0	60.2	38.2
1	CRM 01450	0	With Filtering	<	<	<	<
1	CRM 01450	0	Without Filtering	<	<	<	<
1	CRM 01450	40	With Filtering	76.0	82.1	61.3	<
1	CRM 01450	40	Without Filtering	102.0	72.8	68.8	81.7
1	CRM 01450	250	With Filtering	43.5	69.9	62.8	51.7
1	CRM 01450	250	Without Filtering	71.3	64.3	62.3	22.7
1	CRM 01450	500	Without Filtering	36.9	36.1	27.6	37.0
1	CRM 01450	500	With Filtering	31.4	62.1	64.1	78.0
1	CRM 01450	2000	With Filtering	7.2	14.4	6.3	9.3
1	CRM 01450	2000	Without Filtering	15.6	22.2	19.6	23.1

[a] Wipe specimens with designated lead levels of 0 µg were not spiked with a CRM.
[b] The less-than symbol (<) indicates that the results were "below the detection limit" of the instrument.

Table 7. Lead recovery for each combination of experimental variables for Wipe No. 2

Wipe No.	CRM No.	Lead Level[a] µg	Filter Treatment	UE/ASV Lead Recovery[b], % Op 1	Op 2	Op 3	Op 4
2	SRM 2580	2000	With Filtering	62.0	72.8	69.3	63.6
2	SRM 2580	2000	Without Filtering	53.7	99.3	68.7	62.7
2	SRM 2581	0	With Filtering	<	<	<	<
2	SRM 2581	0	Without Filtering	<	<	<	<
2	SRM 2581	90	With Filtering	57.7	67.6	74.2	43.6
2	SRM 2581	90	Without Filtering	81.9	63.2	76.8	64.1
2	SRM 2581	250	With Filtering	76.6	83.7	71.7	55.6
2	SRM 2581	250	Without Filtering	69.0	72.2	84.8	67.1
2	SRM 2581	500	With Filtering	70.6	73.7	77.4	32.7
2	SRM 2581	500	Without Filtering	58.3	66.1	73.2	54.6
2	SRM 2581	2000	With Filtering	74.0	50.8	64.7	60.1
2	SRM 2581	2000	Without Filtering	49.0	72.1	69.9	51.2
2	SRM 2582	40	With Filtering	107.3	92.6	99.8	62.4
2	SRM 2582	40	Without Filtering	77.4	77.4	67.7	<
2	SRM 2584	0	With Filtering	<	<	<	<
2	SRM 2584	0	Without Filtering	<	<	<	<
2	SRM 2584	250	With Filtering	63.3	56.9	74.6	54.8
2	SRM 2584	250	Without Filtering	77.9	44.6	75.8	41.2
2	SRM 2584	500	With Filtering	76.9	68.6	85.3	74.7
2	SRM 2584	500	Without Filtering	67.4	63.7	78.9	61.0
2	SRM 2584	2000	With Filtering	85.0	40.7	58.4	41.4
2	SRM 2584	2000	Without Filtering	61.6	98.9	67.7	46.8
2	SRM 2586	0	With Filtering	<	<	<	<
2	SRM 2586	0	Without Filtering	<	<	<	<
2	SRM 2586	40	With Filtering	97.6	92.8	97.4	<
2	SRM 2586	40	Without Filtering	102.3	89.5	97.1	67.1
2	SRM 2586	250	With Filtering	83.1	62.0	78.8	63.6
2	SRM 2586	250	Without Filtering	74.8	73.1	92.8	66.0
2	SRM 2586	500	With Filtering	70.0	63.4	70.8	52.4
2	SRM 2586	500	Without Filtering	69.6	71.2	91.2	59.8
2	CRM 01450	0	With Filtering	<	<	<	<
2	CRM 01450	0	Without Filtering	<	<	<	<
2	CRM 01450	40	With Filtering	63.1	97.5	101.0	76.0
2	CRM 01450	40	Without Filtering	98.9	87.9	103.0	83.7
2	CRM 01450	250	With Filtering	69.1	64.8	100.9	54.8
2	CRM 01450	250	Without Filtering	78.1	57.3	81.6	56.5
2	CRM 01450	500	With Filtering	44.5	49.3	56.5	39.0
2	CRM 01450	500	Without Filtering	50.4	50.0	61.9	39.8
2	CRM 01450	2000	With Filtering	40.1	39.4	24.9	18.1
2	CRM 01450	2000	Without Filtering	42.9	48.7	39.2	17.2

[a] Wipe specimens with designated lead levels of 0 µg were not spiked with a CRM.
[b] The less-than symbol (<) indicates that the results were "below the detection limit" of the instrument.

Table 8. Lead recovery for each combination of experimental variables for Wipe No. 3

Wipe No.	CRM No.	Lead Level[a] µg	Filter Treatment	UE/ASV Lead Recovery[b], %			
				Op 1	Op 2	Op 3	Op 4
3	SRM 2580	2000	With Filtering	99.5	104.2	102.6	96.8
3	SRM 2580	2000	Without Filtering	97.5	109.0	102.5	81.9
3	SRM 2581	0	With Filtering	<	<	<	<
3	SRM 2581	0	Without Filtering	<	<	<	<
3	SRM 2581	90	With Filtering	92.2	83.5	96.6	54.8
3	SRM 2581	90	Without Filtering	92.9	88.9	86.2	59.9
3	SRM 2581	250	With Filtering	97.7	91.3	88.9	77.4
3	SRM 2581	250	Without Filtering	92.1	89.7	96.0	70.9
3	SRM 2581	500	With Filtering	97.1	77.4	99.7	72.6
3	SRM 2581	500	Without Filtering	91.9	82.3	81.8	69.4
3	SRM 2581	2000	With Filtering	81.7	68.8	92.7	47.8
3	SRM 2581	2000	Without Filtering	113.0	49.6	56.7	65.0
3	SRM 2582	40	With Filtering	102.4	92.3	97.5	87.6
3	SRM 2582	40	Without Filtering	102.5	92.6	94.9	87.5
3	SRM 2584	0	With Filtering	<	<	<	<
3	SRM 2584	0	Without Filtering	<	<	<	<
3	SRM 2584	250	With Filtering	96.1	85.0	86.2	71.8
3	SRM 2584	250	Without Filtering	97.3	87.0	81.5	91.3
3	SRM 2584	500	With Filtering	89.5	90.1	87.9	81.0
3	SRM 2584	500	Without Filtering	90.7	94.9	90.6	63.9
3	SRM 2584	2000	With Filtering	77.3	58.3	69.6	62.6
3	SRM 2584	2000	Without Filtering	98.5	70.3	65.3	50.4
3	SRM 2586	0	With Filtering	<	<	<	<
3	SRM 2586	0	Without Filtering	<	<	<	<
3	SRM 2586	40	With Filtering	95.0	90.2	89.6	87.4
3	SRM 2586	40	Without Filtering	102.3	97.5	94.9	87.4
3	SRM 2586	250	With Filtering	92.0	92.0	84.8	64.4
3	SRM 2586	250	Without Filtering	93.6	91.1	90.0	74.8
3	SRM 2586	500	With Filtering	81.4	105.6	103.0	53.6
3	SRM 2586	500	Without Filtering	85.8	85.8	82.6	65.8
3	CRM 01450	0	With Filtering	<	<	<	<
3	CRM 01450	0	Without Filtering	<	<	<	<
3	CRM 01450	40	With Filtering	118.6	99.5	106.5	101.5
3	CRM 01450	40	Without Filtering	115.2	89.6	99.5	86.7
3	CRM 01450	250	With Filtering	102.8	98.9	107.5	79.4
3	CRM 01450	250	Without Filtering	102.8	104.2	106.7	81.4
3	CRM 01450	500	With Filtering	105.0	115.2	104.3	98.8
3	CRM 01450	500	Without Filtering	101.6	100.8	91.5	81.0
3	CRM 01450	2000	With Filtering	56.0	59.7	58.8	53.4
3	CRM 01450	2000	Without Filtering	66.8	57.5	55.1	49.7

[a] Wipe specimens with designated lead levels of 0 µg were not spiked with a CRM.
[b] The less-than symbol (<) indicates that the results were "below the detection limit" of the instrument.

Table 9. Lead recovery for each combination of experimental variables for Wipe No. 4

Wipe No.	CRM No.	Lead Level[a] µg	Filter Treatment	UE/ASV Lead Recovery[b], % Op 1	Op 2	Op 3	Op 4
4	SRM 2580	2000	With Filtering	112.2	92.4	88.5	72.6
4	SRM 2580	2000	Without Filtering	106.5	99.6	64.8	66.4
4	SRM 2581	0	With Filtering	<	<	<	<
4	SRM 2581	0	Without Filtering	<	<	<	<
4	SRM 2581	90	With Filtering	95.6	92.4	88.9	70.5
4	SRM 2581	90	Without Filtering	98.1	88.0	90.2	73.5
4	SRM 2581	250	With Filtering	83.6	84.3	90.8	63.1
4	SRM 2581	250	Without Filtering	89.5	72.5	89.2	65.6
4	SRM 2581	500	With Filtering	95.9	77.6	89.6	63.1
4	SRM 2581	500	Without Filtering	84.5	79.1	80.6	53.7
4	SRM 2581	2000	With Filtering	93.1	61.5	60.0	56.5
4	SRM 2581	2000	Without Filtering	70.7	62.1	67.3	21.5
4	SRM 2582	40	With Filtering	102.8	95.0	102.4	82.3
4	SRM 2582	40	Without Filtering	102.6	97.7	97.5	72.6
4	SRM 2584	0	With Filtering	<	<	<	<
4	SRM 2584	0	Without Filtering	<	<	<	<
4	SRM 2584	250	With Filtering	92.3	92.4	90.4	57.2
4	SRM 2584	250	Without Filtering	58.4	76.3	81.8	70.0
4	SRM 2584	500	With Filtering	82.8	77.6	72.9	74.5
4	SRM 2584	500	Without Filtering	76.4	52.8	80.0	65.2
4	SRM 2584	2000	With Filtering	57.1	58.7	54.8	46.1
4	SRM 2584	2000	Without Filtering	47.2	53.3	68.5	38.5
4	SRM 2586	0	With Filtering	<	<	<	<
4	SRM 2586	0	Without Filtering	<	<	<	<
4	SRM 2586	40	With Filtering	94.8	92.6	90.1	82.1
4	SRM 2586	40	Without Filtering	97.3	97.2	92.6	77.7
4	SRM 2586	250	With Filtering	81.5	78.8	92.0	61.2
4	SRM 2586	250	Without Filtering	91.2	78.9	90.3	62.0
4	SRM 2586	500	With Filtering	87.0	80.2	81.2	55.6
4	SRM 2586	500	Without Filtering	88.6	100.6	102.6	57.8
4	CRM 01450	0	With Filtering	<	<	<	<
4	CRM 01450	0	Without Filtering	<	<	<	<
4	CRM 01450	40	With Filtering	113.6	108.5	93.6	82.5
4	CRM 01450	40	Without Filtering	119.6	105.5	106.5	82.1
4	CRM 01450	250	With Filtering	99.8	96.9	95.7	48.4
4	CRM 01450	250	Without Filtering	101.4	102.8	102.3	50.8
4	CRM 01450	500	With Filtering	103.9	68.2	109.1	84.9
4	CRM 01450	500	Without Filtering	94.3	101.5	94.8	83.0
4	CRM 01450	2000	With Filtering	63.5	59.6	54.8	38.3
4	CRM 01450	2000	Without Filtering	59.4	49.9	53.7	41.1

[a] Wipe specimens with designated lead levels of 0 µg were not spiked with a CRM.
[b] The less-than symbol (<) indicates that the results were "below the detection limit" of the instrument.

Only four of the 512 CRM-containing specimens (i.e., ≈ 1 %) had results that were "below the detection limit." All of these four specimens had lead levels of 40 μg. In addition, whenever a dust wipe specimen did not contain lead, the analyses indicated that the results were "below the detection limit."

Reasons why Operator 4 determined, on the average, lower recoveries than the other three operators were not investigated, although one possible explanation is offered. Due to scheduling, Operator 4 completed the series of analyses before any other operator began. This provided NIST research staff opportunity to consider reasons for Operator 4's low recoveries in advance of the other operators' beginning testing. Departures from the test protocol followed by Operator 4 were regarded as unlikely. Observations of this operator during testing gave no indication that the test protocol was not being followed. For example, note in Figure 1 that Operator 4's analyses of the CRM 01450 controls afforded quantitative recovery. Ruling out non-conformance to the test protocol, NIST research staff asked whether the protocol itself played a role. One consideration was whether lead in the dust wipe specimens was satisfactorily extracted. For example, Step 2d (Table 5) on first mixing the specimen in the acid directs the operator to "keep pushing the wipe down into the acid solution until trapped air bubbles in the wipe have been released and until the solution appears cloudy from the dust residue." This mixing step is subjective. As a supposition, if the step is not carried out to the fullest, perhaps dust in a wipe might not be adequately exposed to the acid during extraction. As a consequence of considering the effect of mixing on lead recovery, NIST research staff made a slight modification to the original test protocol used by Operator 4. Specifically, the modification required more rigorous mixing of the dust wipe in the acid. That is, after completing either Step 2d or 3e, the operator was to repeat the step.* A possible result of this change in protocol might have been that Operators 1 through 3 better mixed the specimens in the acid than did Operator 4.

Analysis of variance (ANOVA) was performed on the full data set to evaluate the effect of the five main experimental variables—operator, dust wipe, CRM type, lead level, and filter treatment. An examination of the effect of CRM mass was also performed, as mass varied considerably among the specimens. Appendix B shows the effect of increasing mass on lead recovery. The ANOVA results are summarized in Table 10. A measure of how unlikely an observed effect is due to chance is provided by the p-value. Conventionally, effects that have p-values less than 0.05 are considered to be statistically significant. The summary in Table 10 shows that all of the main effects were significant, although the filter treatment effect was considered to be less significant in comparison to the effects of operator, dust wipe, CRM type, lead level, and CRM mass. In addition, many of the two-way interactions were highly significant.

Because the two-way interaction effects with CRM type were generally highly significant†, analysis of variance was next conducted to examine separately for each CRM the effect of the other four variables—operator, dust wipe, lead level, and filter treatment. The effect of mass was not included in these analyses. When the data set is split and examined separately for each CRM type, the group of observations for each lead level is the same as the group of observations for each mass level. That is, the lead level and CRM type factors are confounded. The results of the analyses for each CRM are given in Appendix C. Trends on the factors affecting recovery, as gleaned from these analyses, are presented below (Sections 4.2.2.1 through 4.2.2.4) along with observations from the analysis of variance (Table 10) for the entire data set.

* This revised mixing directive was still subjective. Nevertheless, it resulted in Operators 1 through 3 mixing the specimens in acid for longer periods of time than Operator 4.

† The insignificance of the Operator & CRM Type interaction was considered to be marginal ($p = 0.0512$).

Table 10. Analysis of variance of the entire data set

Main Effects and Interactions[a]	Analysis Result F-Statistic	P-Value
Main Effects		
Operator	27.852	**0.0000**
Wipe	81.778	**0.0000**
CRM Type	52.585	**0.0000**
Lead Level	142.630	**0.0000**
CRM Mass	7.597	**0.0000**
Filter Treatment	6.327	**0.0123**
Two-Way Interactions		
Operator — Wipe	7.028	**0.0000**
Operator — CRM Type	1.685	0.0512
Operator — Lead Level	2.009	**0.0222**
Operator — CRM Mass	1.085	0.3712
Operator — Filter Treatment	1.082	0.3567
Wipe — CRM Type	9.277	**0.0000**
Wipe — Lead Level	4.644	**0.0000**
Wipe — CRM Mass	1.426	0.1514
Wipe — Filter Treatment	0.395	0.7567
CRM Type — Lead Level	10.869	**0.0000**
CRM Type — Filter Treatment	4.714	**0.0003**
Lead Level — Filter Treatment	0.399	0.8089
Lead Level — CRM Mass	11.90	**0.0000**
CRM Mass — Filter Treatment	1.967	0.0989

[a] Because of the one-to-one correspondence between lead level and CRM mass, the interaction between the CRM type and CRM mass could not be estimated by the ANOVA model used in the analysis. The interaction between CRM type and CRM mass is identical to that for lead level and CRM mass. Bold font is used to denote significant p-values (i.e., <0.05).

The analyses given in Appendix C for each CRM type again shows that lead recovery was quite variable and generally affected by all experimental factors. No matter the CRM type, the effect of operator, dust wipe, and lead level was always significant. Additionally, for half the CRM types, filter treatment of the extract solution was also significant.

4.2.2.1 Operator Effect

- The highest recoveries were for Operators 1 through 3 and, in the majority of cases, the values for each of these three operators were not significantly different from each other. In particular, for the 18 comparisons made among Operators 1 through 3 for the six CRMs, a significant difference in recovery was only observed between Operator 1 and Operator 2 for SRM 2581. This represents 6 % of those between-operator comparisons.
- Although Operators 1 through 3 had the highest recoveries, the median values were not always quantitative; for example, for SRM 2584 and CRM 01450, median recoveries for the four operators were never quantitative.

- The lowest recoveries were for Operator 4. In all cases, the median recoveries for Operator 4 were significantly lower than those for at least one of the other operators.
- Operator 4 median recoveries were less than quantitative with the exception of SRM 2582.

Discussion. The results of the analysis of the entire data set indicate that the operator effect was essentially associated with Operators 1 through 3 achieving higher lead recovery than Operator 4. Recall that Operators 1 through 3 performed the analyses using a slight variation of the original test protocol that was followed by Operator 4, although it was not determined whether this modification accounted for the observed operator effect.* An operator effect was still present when the data for Operator 4 were excluded from the analysis, even though no significant differences in recovery were generally observed among Operators 1 through 3 when the entire data set was analyzed. The finding of an operator effect in the current study was in contrast with the results of Ashley et al.'s interlaboratory study [5], wherein acceptable UE/ASV lead recovery was found for the participating laboratories (i.e., tests with different operators).

4.2.2.2 Wipe Effect

- The highest recoveries were generally found for Wipe 3 although, in most cases, they were not significantly different than those observed for Wipe 4.
- The lowest recoveries were generally found for Wipe 1 although, in most cases, they were not significantly different than those observed for Wipe 2.
- The median recoveries for Wipes 3 and 4 were generally quantitative; whereas those for Wipes 1 and 2 were generally not quantitative.

Discussion. The analyses indicated that two of the four wipes (i.e., Wipes 3 and 4) had higher lead recovery. The observance of a wipe effect is consistent with the findings of Ashley et al. [5], who previously reported differences in lead recovery due to the wipe. Factors contributing to the wipe effect observed in this current study were not examined and should be the subject of future investigation. Further discussion of the wipe effect is given in Section 4.3.

4.2.2.3 Lead Level Effect

- The highest recoveries were generally found for the lower lead levels of 40 µg and 90 µg, whereas lower recoveries were generally found for the 2000 µg lead levels.
- The 40 µg and 90 µg lead levels had median recoveries that were all quantitative.
- The 250 µg, 500 µg, and 2000 µg lead levels generally had median recoveries that were not quantitative. An exception was SRM 2580, which had a median recovery of 90 % at the 2000 µg lead level (data not shown). The mass of the SRM 2580 specimens was 46 mg.

Discussion. The results indicate that lead was more readily recovered from specimens having low lead levels than those with high lead levels. However, because the data for lead level and CRM mass were confounded, it may be that increased specimen mass was responsible for the observed lower recoveries at higher lead levels. Note in Figure B1 that specimens with higher CRM mass had significant lower median recoveries. The suggestion that CRM mass may be responsible for the observed lead level effect is supported by the observation that SRM 2580 at the 2000 µg lead level, but having low CRM mass (46 mg), provided quantitative recovery. However, other data conflict with this observation. In this regard, many of the specimens,

* Plans have been proposed to perform a repeat set of dust wipe analyses by Operator 4 using the slightly modified test protocol. The results of such tests would be published when completed.

which were prepared at relatively high lead levels of 250 µg, 500 µg, and 2000 µg with relatively low CRM mass (i.e., 26 mg to 205 mg), afforded less than quantitative recovery. Future research is needed to elucidate the effects of lead level and CRM mass on recovery.

4.2.2.4 Filter Treatment Effect

- An effect due to filter treatment was found for three of the six certified reference materials: SRM 2581, SRM 2582, and CRM 01450.
- For SRM 2581 and SRM 2582, recovery was higher for filtered extracts than for non-filtered extracts. In contrast, for CRM 01450, non-filtering provided higher recovery. However, in this case, the difference between the median recoveries of the filtered and unfiltered extracts was slight (≈ 2 %), and no practical significance was attributed to the observed filter treatment effect.

Discussion. Analysis of variance on the entire data set (Table 10) indicated a main effect due to filter treatment with the only significant two-way interaction being that between filter treatment and CRM type. Further analysis of the CRM data sets (Appendix C) showed that the filter treatment-CRM type effect was not universal to the six CRMs in the study. Reasons for the observed filter treatment-CRM type effect are not clear. Although in the cases of SRM 2581 and SRM 2582 it might be asked whether potential ASV interferants were removed by filtering which, in turn, increased lead recovery, recall (Table 3) that these two CRMs provided quantitative recovery for specimens prepared without wipes and analyzed without filtering. The lack of a filter treatment-wipe interaction was in contrast with Ashley et al.'s results [5] that showed recovery for some wipes was enhanced by filtering. The data in the current study suggest that the wipes used did not contain ASV interferants that could be removed by filtering. The wipe-effect inconsistencies between the current results and those previously published need to be resolved.

Although reasons for the filter treatment-CRM type interaction have not been identified, the finding that filtering enhanced lead recovery for two CRMs has implications for UE/ASV analyses of field samples. It may be that UE/ASV analysis of some "real-world" dusts would be improved by filtering. In this regard, the finding that filtering can promote recovery for some CRMs (i.e., the "dust" in the wipes) supports the recommendation [5] that filtering be used in the UE/ASV analysis of dust wipe specimens to help to ensure reliable analysis.

4.2.3 Summary of the Main Experiment. Four operators conducted UE/ASV analyses of 640 dust wipe samples. The results were variable with lead recoveries ranging from < 20 % to > 100 % depending upon the combination of experimental variables. Only 42 % of the specimens spiked with a CRM afforded quantitative recovery. Statistical analyses of the entire data set showed that all experimental variables had a significant effect on recovery. Many of the two-way interaction effects were also significant. The operator effect was essentially associated with Operators 1 through 3 achieving higher lead recovery than Operator 4. Although no significant differences in recovery were generally observed among Operators 1 through 3 when the entire data set was analyzed, an operator effect was still present when the data for Operator 4 were excluded from the analysis. Reasons why the results varied widely with the combinations of experimental factors are not known, but such variability is consistent with the observations from a previous study [5]. The low lead recoveries in the current study were in contrast to the findings of the previous studies [4-7] wherein generally quantitative recoveries were achieved particularly when filtering was used. Knowing that the analysis is dependent on many factors including wipe and CRM, it is interesting to speculate whether the combinations of the wipe and CRM in the previous studies by chance provided dust wipe specimens that afforded higher recovery than some of the combinations of wipe and CRM in the current study. Based on the results of the Main Experiment, a main conclusion is that the test procedure lacked robustness. That is, it was found that changes in the experimental variables incorporated in the study design could lead to low lead recoveries. Investigations for improving the robustness of the test procedure were beyond the scope of this study and need to be addressed in future research.

4.3 The ASV/ICP Comparison

Investigations into the reason(s) why the lead recoveries from many dust wipe specimens were low were beyond the scope of the study. Nevertheless, it may be broadly hypothesized that the low recoveries were associated with (1) operator error in performing the UE and ASV procedures, (2) incomplete lead extraction during sonication, (3) ASV measurement error perhaps due to interferants introduced from the dust wipes or CRMs, but not generally removed by filtering, or (4) some combination of the three. Although not ruled out by experimentation, operator error is not considered in the present discussions because the operators consistently attained quantitative lead recovery from the control specimens (Figure 1).

To provide initial data regarding the role of extraction and ASV measurement in the low recoveries, aliquots (≈ 20 mL) of a limited number of UE extracts (after ASV analysis) were shipped overnight to an NLLAP laboratory for lead analyses using ICP. For each operator, the extracts selected were those prepared during one or two day's UE/ASV analyses. These extracts were filtered before shipment to remove solids (that possibly contained lead)*. The extract solutions were analyzed by the NLLAP laboratory on the same day that they arrived.

Figure 6 presents plots comparing lead recoveries measured by each operator using ASV with those determined by the NLLAP laboratory using ICP. The 45° line in each plot represents agreement between the results of the two measurements. The vertical lines denote whether the results of the NLLAP ICP analyses were quantitative; i.e., recoveries of ≥ 80 %. The plot symbols signify Wipes 1 through 4. Bold, italicized plot symbols (in the upper right corner of the plots) represent data points for which the ASV and ICP measurements were both quantitative. Linear regression analyses of the data in each plot provided r^2-values of ≤ 0.50 for any operator.

Table 11 summarizes by wipe the percent of specimens that provided quantitative recovery as determined by ICP measurement, ASV measurement, and both methods. Review of Figure 6 and Table 11 provides some initial insight into the role of extraction and ASV analyses on the low recoveries. The observed trends may help direct the design of future experimentation on furthering the understanding of factors affecting lead extraction and analysis using UE and ASV, respectively.

* Ultrasonic extraction does not totally digest the dust wipe specimens.

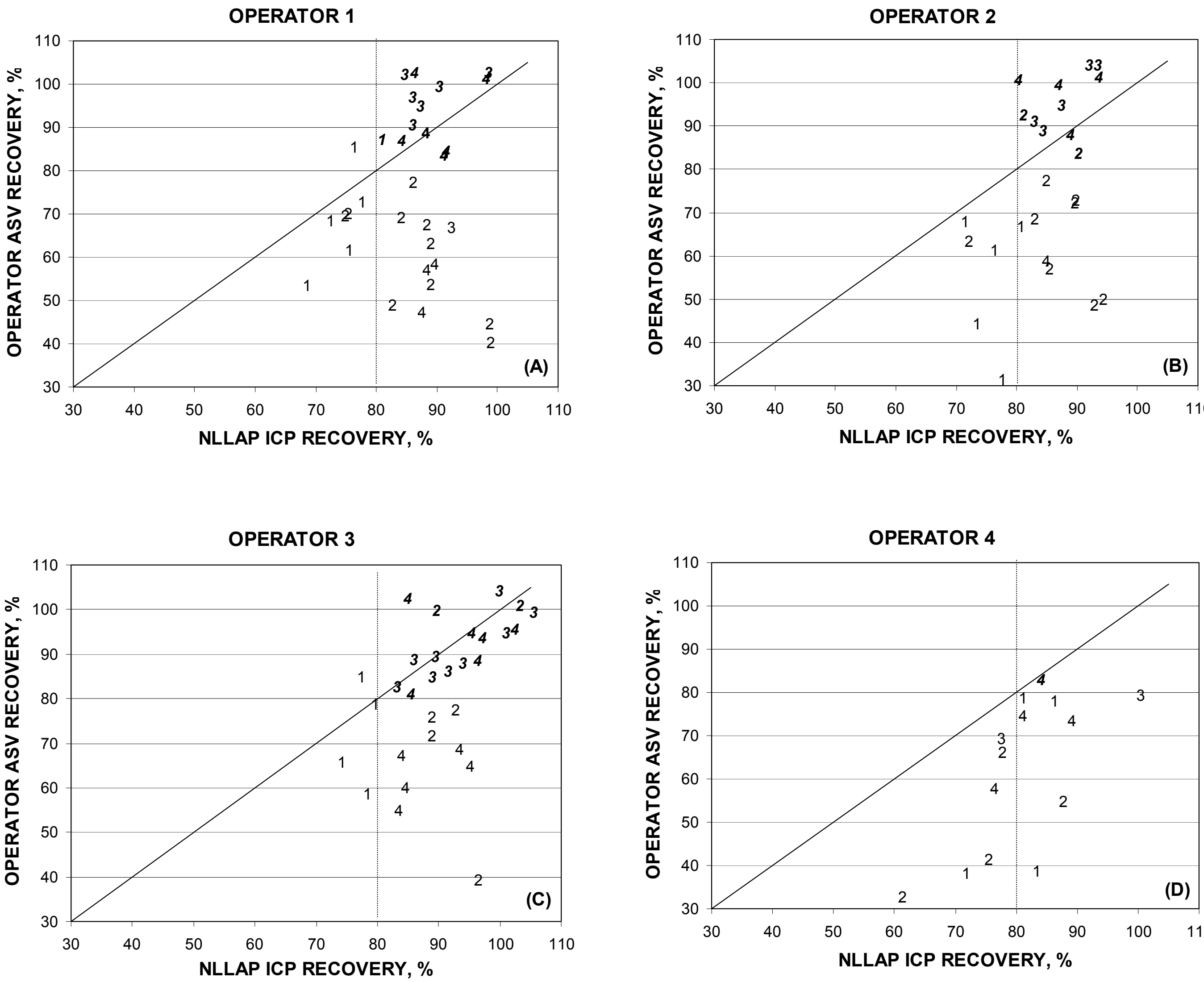

Figure 6. Comparison of Lead Recovery Measured by the Operators Using ASV with That Measured by the NLLAP Laboratory Using ICP: (A) Operator 1, (B) Operator 2, (C) Operator 3, and (D) Operator 4. For a given specimen, the ASV and ICP measurements were made on the same UE extract solution. In the plots, the 45° lines represent agreement between the results of the two measurements. The vertical lines denote whether the results of the NLLAP ICP analyses were quantitative; i.e., recoveries of ≥ 80 %. The plot symbols represent Wipes 1 through 4. Bold, italicized plot symbols (in the upper right corner of the plots) represent data points for which the ASV and ICP measurements were both quantitative.

Table 11. Comparison of recoveries as measured by ASV and ICP methods

Wipe No.	Percent of Specimens Providing Quantitative Recovery		
	ICP Measured %	ASV Measured %	Both Methods %
1	26	16	5
2	80	13	13
3	96	87	87
4	96	59	59

- *Operators 1 through 3 generally had higher recoveries than Operator 4*. For Operator 1, Operator 2, and Operator 3, the percents of recoveries that were quantitative by both ASV and ICP were 41 %, 44 %, and 57 %, respectively. In contrast, for Operator 4, only one specimen (i.e., 7 % of the total) was quantitatively analyzed by both measurements.
- *For Operators 1 through 3, the percent of ICP measurements that were quantitative ranged from about 80 % to 87 %.* This suggests that UE extraction was generally adequate for Operators 1 through 3, and that some step(s) in the ASV analysis adversely affected recovery. For Operator 4, only 57 % of the ICP measurements were quantitative. In this case, inadequate extraction may have played more of a role in the low recoveries. Remember that it was Operator 4 who followed a test protocol that included less mixing of the specimen in acid than that used by Operators 1 through 3 (Section 4.2.2). For Operator 4, the majority of the eight specimens showing quantitative ICP recovery yielded less-than-quantitative ASV recovery.
- *Wipes 1 and 2 provided lower lead recovery than Wipes 3 and 4.* The percents of quantitative recovery for both ASV and ICP were only 5 % and 13 % for Wipes 1 and 2, respectively. In contrast, the percents of quantitative recovery for both ASV and ICP were 87 % and 59 % for Wipes 3 and 4, respectively.
- *Wipe 1 specimens afforded fewer ICP analyses having quantitative recovery than specimens prepared with Wipes 2 through 4.* For Wipe 1, the percent of ICP quantitative recoveries was only 26 %. A possible explanation for this observation may be that lead extraction from Wipe 1 specimens using the ultrasonic procedure was more difficult than for specimens prepared with Wipes 2 through 4. This possibility has support in the literature. Specifically, Ashley et al. [5] reported that ultrasonic extraction (using a procedure similar to that of the present study) did not successfully solubilize lead from one of four wipes spiked with SRM 1648 or SRM 2704. Another explanation may be that there is an interaction between Wipe 1 and solubilized lead that resulted in lowered lead recovery when determined using ICP. A third possibility is that Wipe 1 released interferants to the ICP analysis. Regarding adverse interactions or interferants, if such damaging factors came into play, they seemingly are not present when the dust wipe analyses involves hot plate acid digestion followed by ICP. Note from Table 4 that the mean recovery determined by RTI for Wipe 1 specimens was 91 % (12 % CoV). Also, recall from Table 1 that the Wipe 1 supplier indicated that its wipe met the provisions of ASTM Standard Specification E 1792 [3]. This specification includes a requirement for efficacy of lead recovery from CRM-spiked wipes. In showing conformance to this requirement, lead extraction is performed using hot plate digestion. Lead analysis of the resulting digestate is performed using ICP or Flame Atomic Absorption Spectrometry (FAAS), or Graphite Furnace Atomic Absorption Spectrometry (GFAAS).

- *Wipes 2 through 4 were in general adequately extracted using UE.* For these three wipes, ICP recoveries were generally quantitative, with percents ranging from 80 % to 96 %.
- *Wipe 2 specimens may have produced interferants to ASV analysis.* Although 80 % of the Wipe 2 extracts analyzed using ICP provided quantitative recovery, only 13 % of those same extracts analyzed by ASV gave quantitative recovery.
- *Wipe 3 specimens showed the most efficient extraction and ASV measurement.* Wipe 3 specimens displayed quantitative recoveries for 96 % of the ICP analyses, and for 87 % of the ASV analyses.
- *Wipe 4 specimens showed efficient extraction, but intermediate ASV performance.* Like Wipe 3 specimens, Wipe 4 specimens gave quantitative recoveries for 96 % of the ICP analyses. Unlike Wipe 3, the Wipe 4 specimens provided quantitative ASV recoveries only 59 % of the time.

4.3.1 Summary of the ASV/ICP Comparison. The comparison of the ASV and ICP data showed, consistent with the statistical analyses of the data in the Main Experiment, that:

- Operator had an effect on recovery as one of the four generally measured lower recoveries than the other three.
- Wipe type had an effect on lead recovery when determined using either ICP or ASV analyses of the UE extract solutions.

5. SUMMARY, CONCLUSIONS, AND RECOMMENDATIONS

Lead in dust can pose a serious risk for young children. Consequently, lead analysis of dust wipe samples collected in the field is an integral part of programs for controlling and abating potential lead hazards in housing. Normally this analysis is not performed in the field. Previous laboratory studies have indicated that ultrasonic extraction/anodic stripping voltammetry (UE/ASV) may be suitable for quantitative field analysis of dust wipe samples. A reservation to adopting UE/ASV is that the effect of the field operator (i.e., the analyst) is unknown. Lead risk assessors and inspectors, who are potential users of field methods (i.e., the operators), may have less skill in conducting the lead analyses than chemists and laboratory technicians who participate in evaluation studies. On-site lead extraction and analysis of dust wipes are not currently used in federal programs for controlling and abating lead hazards in housing. The U.S. Department of Housing and Urban Development (HUD) has interest in test methods that may be used to determine lead in dust at housing sites. The availability of a reliable field test procedure could allow for on-site lead extraction and analysis of dust wipe specimens after field collection. Thus, HUD requested that NIST conduct a study to evaluate the effect of operator when certified lead risk assessors or inspectors, trained to conduct UE/ASV analyses, performed such analyses of laboratory-prepared dust wipe specimens using commercial, field-portable apparatus. Four other experimental variables were also examined: dust wipe, CRM type, lead level, and filter treatment of the UE extract solution before ASV analysis. All dust wipes were commercial products that were reported by the suppliers as conforming to the requirements of ASTM E 1792, “Standard Specification for Wipe Sampling Materials for Lead in Surface Dust.” All CRMs provided quantitative lead recovery (i.e., 100 % ± 20 %) when specimens without dust wipes were analyzed by UE/ASV.

A five-factor full factorial design guided the experimental program referred to as the Main Experiment. Using commercial apparatus, four operators performed UE/ASV analyses of 640 dust wipe specimens following a test protocol that was developed in accordance with the UE/ASV apparatus instructions. These specimens were prepared using four commercial wipes spiked with one of six lead-containing CRMs*. Six lead levels spanned a range from 0 µg to 2000 µg. After UE extraction, the extract solutions were either filtered or not filtered before the ASV analyses were conducted. Statistical analysis was performed to determine the effects of the experimental factors on lead recovery. The key conclusions of the Main Experiment are:

- Lead recoveries were variable, ranging from < 20 % to > 100 % depending upon the combination of experimental variables. Only 42 % of the specimens spiked with a CRM afforded quantitative recovery.
- All experimental factors had a significant effect on recovery; in addition, the majority of the two-way interactions were also significant.
- The operator effect was essentially associated with three of the four operators determining higher lead recoveries than the other one. The three operators reporting the higher recoveries followed a test protocol than was a slight modification of that used by the fourth operator. It was not determined whether this modification accounted for the observed operator effect. Only in one case was a significant difference in recovery observed among the three operators achieving higher recoveries; nevertheless, the operator effect was present when the data for the fourth operator were excluded from the analysis.
- The wipe effect was associated with two of the four wipes providing lower recoveries than the other two. Median recoveries for the two wipes providing the higher recoveries were generally quantitative.
- The lead level effect was associated with lower lead levels providing greater recovery than the higher lead levels. A CRM mass effect was also present with lower mass resulting in greater recovery. Because, for a given CRM, increases in mass are accompanied by increases in lead level, the lead level and CRM mass effects could not be distinguished.
- The filter treatment effect was primarily associated with two of the six CRMs providing significantly greater recoveries when the UE extract was filtered before ASV analysis.
- A filter treatment-CRM type interaction was found; whereas a filter treatment-wipe interaction was not observed.

Investigations as to why the lead recoveries from many dust wipe specimens were low were beyond the scope of the study. Nevertheless, to provide data regarding the role played by ultrasonic extraction and ASV measurement on the low recoveries, the lead concentrations of a limited number of UE extracts were analyzed by an NLLAP laboratory using ICP. For each operator, a comparison was made between the ICP and ASV results. Key findings from these comparisons include:

- Wipe type had an effect on lead recovery when determined using either ICP or ASV analyses of the UE extract solutions.
- One wipe may have produced interferants to ASV analysis.

Based on the results of the current study, a main conclusion is that the UE/ASV test procedure lacked robustness for analyzing dust wipe specimens. That is, it was found that changes in the experimental variables incorporated in the study design could lead to low lead recoveries. This variability in recovery was consistent with the results of a previous study in which it was found that recovery depended upon the combination of experiment factors examined. Investigations for

* Four CRMs were initially considered for the experiments; however, of the four, only one had a lead content (i.e., percent lead by mass fraction) that made it practical to prepare dust wipe specimens having lead levels spanning the selected range. To help overcome this limitation, two additional CRMs were included in the study.

improving the robustness of the test procedure were beyond the scope of this study. It is recommended that such investigations be conducted in future research.

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APPENDIX A. LEAD RECOVERY FOR COMBINATIONS OF WIPE AND CRM

This Appendix presents, for each operator, a summary of lead recovery for each combination of wipe and CRM (Tables A1 through A4). This summary is a further illustration of the variability in the lead recovery data developed in the Main Experiment. Table A5 lists, for each operator, the number of the six combinations of wipe and CRM that provided mean quantitative recovery. Note from Table A5 that Operators 1 through 3 had many more instances of mean quantitative recovery than Operator 4. Note also that Wipes 3 and 4 provided more cases of mean quantitative recovery than Wipes 1 and 2.

Table A1. Lead recovery determined by Operator 1 for each combination of wipe and CRM

Operator No.	Wipe No.	CRM No.	n[a]	Lead Recovery, % Min	Max	Mean	CoV[b]
1	1	SRM 2580	2	65.3	73.6	69.4	8.5
		SRM 2581	8	45.5	98.2	71.1	25.4
		SRM 2582	2	72.4	87.3	79.9	13.2
		SRM 2584	6	26.0	79.8	63.7	30.9
		SRM 2586	6	44.0	77.9	61.8	21.6
		CRM 01450	8	7.2	102.0	48.0	67.6
1	2	SRM 2580	2	53.7	62.0	57.9	10.1
		SRM 2581	8	49.0	81.9	67.1	16.6
		SRM 2582	2	77.4	107.3	92.3	22.9
		SRM 2584	6	61.6	85.0	72.0	12.9
		SRM 2586	6	69.6	102.3	82.9	17.1
		CRM 01450	8	40.1	98.9	60.9	33.7
1	3	SRM 2580	2	97.5	99.5	98.5	1.4
		SRM 2581	8	81.7	113.0	94.8	9.3
		SRM 2582	2	102.4	102.5	102.4	0.1
		SRM 2584	6	77.3	98.5	91.6	8.6
		SRM 2586	6	81.4	102.3	91.7	8.0
		CRM 01450	8	56.0	118.6	96.1	23.4
1	4	SRM 2580	2	106.5	112.2	109.4	3.7
		SRM 2581	8	70.7	98.1	88.9	10.2
		SRM 2582	2	102.6	102.8	102.7	0.1
		SRM 2584	6	47.2	92.3	69.0	25.2
		SRM 2586	6	81.5	97.3	90.1	6.3
		CRM 01450	8	59.4	119.6	94.4	23.2

[a] n indicates number of specimens.
[b] CoV indicates coefficient of variation (std deviation/mean x 100).

Table A2. Lead recovery determined by Operator 2 for each combination of wipe and CRM

Operator No.	Wipe No.	CRM No.	n[a]	Lead Recovery, % Min	Max	Mean	CoV[b]
2	1	SRM 2580	2	84.7	90.0	87.3	4.3
		SRM 2581	8	32.0	83.8	57.5	32.5
		SRM 2582	1[c]	---	72.4	---	---
		SRM 2584	6	34.6	66.3	52.0	23.2
		SRM 2586	6	59.0	77.2	64.4	10.9
		CRM 01450	8	14.4	82.1	53.0	47.6
2	2	SRM 2580	2	72.8	99.3	86.1	21.8
		SRM 2581	8	50.8	83.7	68.7	39.4
		SRM 2582	2	77.4	92.6	85.0	12.6
		SRM 2584	6	40.7	98.9	62.2	33.6
		SRM 2586	6	62.0	92.8	75.3	17.3
		CRM 01450	8	39.4	97.5	61.9	33.2
2	3	SRM 2580	2	104.2	109.0	106.6	3.2
		SRM 2581	8	49.6	91.3	78.9	17.7
		SRM 2582	2	92.3	92.6	92.5	0.3
		SRM 2584	6	58.3	94.9	80.9	17.1
		SRM 2586	6	85.8	105.6	93.7	7.4
		CRM 01450	8	57.5	115.2	90.7	23.2
2	4	SRM 2580	2	92.4	99.6	96.0	5.3
		SRM 2581	8	61.5	92.4	77.2	14.7
		SRM 2582	2	95.0	97.7	96.4	2.0
		SRM 2584	6	52.8	92.4	68.5	23.4
		SRM 2586	6	78.8	100.6	88.0	11.3
		CRM 01450	8	49.9	108.5	86.6	27.1

[a] n indicates number of specimens.
[b] CoV indicates coefficient of variation (std deviation/mean x 100).
[c] One analysis for this combination of wipe and CRM had a result that was "below the detection limit."

Table A3. Lead recovery determined by Operator 3 for each combination of wipe and CRM

Operator No.	Wipe No.	CRM No.	n[a]	Lead Recovery, % Min	Max	Mean	CoV[b]
3	1	SRM 2580	2	72.7	92.4	82.5	16.9
		SRM 2581	8	41.9	84.1	67.0	25.3
		SRM 2582	2	72.5	84.9	78.7	11.2
		SRM 2584	6	39.3	78.7	63.9	22.5
		SRM 2586	6	40.4	75.2	61.4	19.2
		CRM 01450	8	6.3	68.8	46.6	52.7
3	2	SRM 2580	2	68.7	69.3	69.0	0.6
		SRM 2581	8	64.7	84.8	74.1	8.0
		SRM 2582	2	67.7	99.8	83.7	27.1
		SRM 2584	6	58.4	85.3	73.5	12.7
		SRM 2586	6	70.8	97.4	88.0	12.3
		CRM 01450	8	24.9	103.0	71.1	42.4
3	3	SRM 2580	2	102.5	102.6	102.5	0.1
		SRM 2581	8	56.7	99.7	87.3	15.7
		SRM 2582	2	94.9	97.5	96.2	1.9
		SRM 2584	6	65.3	90.6	80.2	12.9
		SRM 2586	6	82.6	103.0	90.8	8.1
		CRM 01450	8	55.1	107.5	91.2	23.9
3	4	SRM 2580	2	64.8	88.5	76.7	21.9
		SRM 2581	8	60.0	90.8	82.1	14.6
		SRM 2582	2	97.5	102.4	100.0	3.5
		SRM 2584	6	54.8	90.4	74.7	16.5
		SRM 2586	6	81.2	102.6	91.5	7.5
		CRM 01450	8	53.7	109.1	88.8	24.8

[a] n indicates number of specimens.
[b] CoV indicates coefficient of variation (std deviation/mean x 100).

Table A4. Lead recovery determined by Operator 4 for each combination of wipe and CRM

Operator No.	Wipe No.	CRM No.	n[a]	Lead Recovery, % Min	Max	Mean	CoV[b]
4	1	SRM 2580	2	79.4	101.6	90.5	17.3
		SRM 2581	8	38.7	93.5	63.2	32.1
		SRM 2582	2	82.3	82.4	82.4	0.1
		SRM 2584	6	38.6	78.7	58.4	28.0
		SRM 2586	6	38.2	72.5	55.6	24.9
		CRM 01450	7[c]	9.3	81.7	43.4	65.0
4	2	SRM 2580	2	62.7	63.6	63.1	1.0
		SRM 2581	8	32.7	67.1	53.6	20.9
		SRM 2582	1[c]	---	62.4	---	---
		SRM 2584	6	41.2	74.7	53.3	24.4
		SRM 2586	5[c]	52.4	67.1	61.8	9.7
		CRM 01450	8	17.2	83.7	48.1	50.7
4	3	SRM 2580	2	81.9	96.8	89.3	11.8
		SRM 2581	8	47.8	77.4	64.7	15.3
		SRM 2582	2	87.5	87.6	87.6	0.0
		SRM 2584	6	50.4	91.3	70.2	20.7
		SRM 2586	6	53.6	87.4	72.2	18.7
		CRM 01450	8	49.7	101.5	79.0	23.8
4	4	SRM 2580	2	66.4	72.6	69.5	4.4
		SRM 2581	8	21.5	73.5	58.4	27.9
		SRM 2582	2	72.6	82.3	77.5	8.9
		SRM 2584	6	38.5	74.5	58.6	24.0
		SRM 2586	6	55.6	82.1	66.1	16.7
		CRM 01450	8	38.3	84.9	63.9	32.7

[a] n indicates number of specimens.
[b] CoV indicates coefficient of variation (std deviation/mean x 100).
[c] One analysis for this combination of wipe and CRM had a result that was “below the detection limit.”

Table A5. Number of combinations of wipe and CRM providing mean quantitative recovery for each operator

Wipe No.	Mean Quantitative Recovery: number of cases per operator[a] Op 1	Op 2	Op 3	Op 4
1	0	1	1	2
2	2	2	2	0
3	6	5	6	2
4	5	4	4	0

[a] There were six combinations of wipe and CRM for each operator.

APPENDIX B. CRM MASS EFFECT ON LEAD RECOVERY

This Appendix illustrates the effect of CRM mass on lead recovery. Mass varied considerably among the specimens because of the varying lead content (by % mass fraction) of the CRMs. Figure B1 shows plots of lead recovery versus CRM mass for each of the four operators. In this figure, the plot symbol distinguishes the data points by wipe.

A box plot was prepared to illustrate the mass effect. To simplify this analysis, the CRM masses of the specimens were grouped into six levels, each having specimens with comparable amounts of mass (Table B1). In a box plot, the shaded boxes represent the range of recovery for 50 % of the data points. The white line in each shaded box indicates the median recovery. The brackets (often called "whiskers") above and below the box represent the smallest and largest points except for the outliers, which are represented by solid lines. The size of the box and spread of the whiskers indicate the influence of the other experimental variables (i.e., those factors that are not the focus of the given box plot) on lead recovery. Relatively small boxes indicate that the result was generally not affected by the other experimental variables.

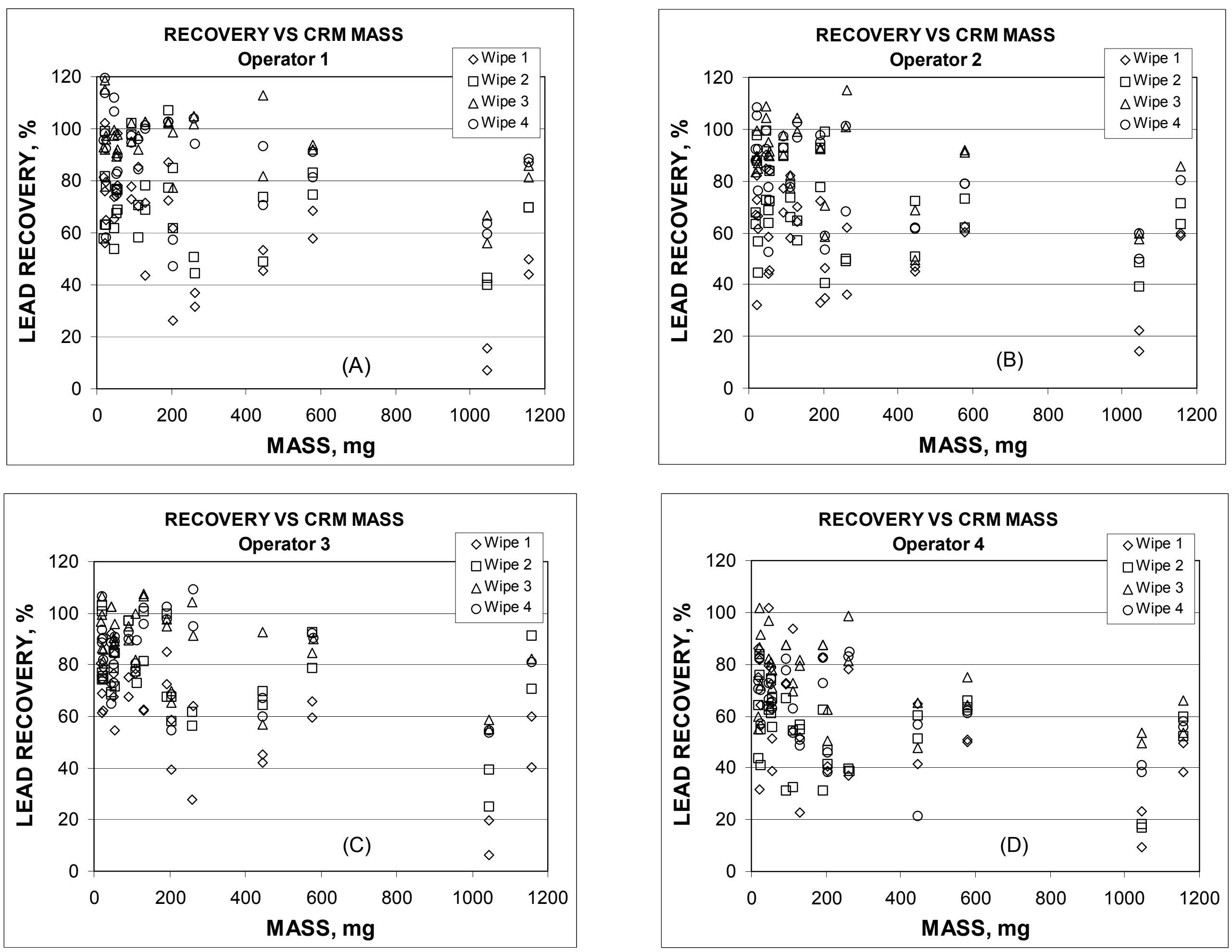


Figure B1. Lead Recovery as Related to CRM Mass and Wipe Type: (A) Operator 1, (B) Operator 2, (C) Operator 3, and (D) Operator 4.

Figure B2 presents the box plot showing the effect of mass on lead recovery for the six groupings of mass level (Table B1). As mass level increased, median lead recovery decreased, particularly for Mass Levels 4 through 6. Analysis using simultaneous 95 % confidence intervals (data not shown) was performed to identify whether an observed difference in lead recovery between a given pair of factor levels (e.g., Mass Level 1 vs Mass Level 2) was significant. This analysis indicated that there was no significant difference between Mass Level 1 and Mass Level 2. All other pair-wise comparisons were significantly different.

Table B1. Groupings of mass levels for use in the analysis

Specimen CRM Mass Level	Design CRM Mass, mg
1	20
	21
	26
2	46
	51
	56
3	93
	111
	131
4	192
	205
	261
5	445
	579
6	1045
	1157

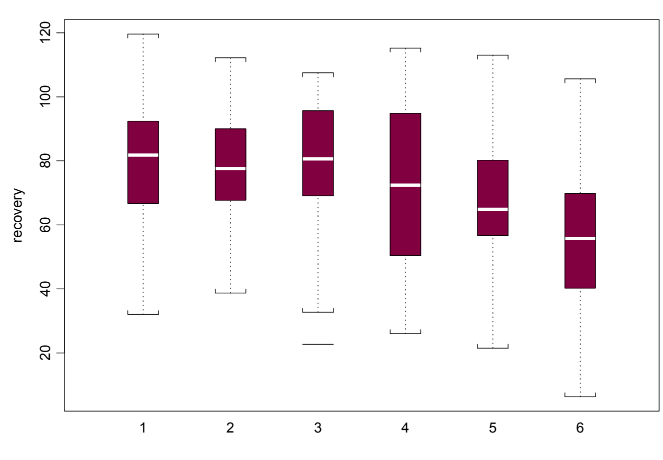


Figure B2. Effect of CRM Mass on Lead Recovery

APPENDIX C. ANALYSIS BY CRM TYPE

C1. INTRODUCTION

Many of the two-way interaction terms with CRM type were highly significant. Thus, analysis of variance was performed to examine separately the effect of operator, dust wipe, lead level, and filter treatment for each CRM. Table C1 summarizes the results of this analysis of variance and indicates whether the combination of a given CRM with one of the other experimental variables had a significant effect (p-value > 0.05) on lead recovery. Analysis using simultaneous 95 % confidence intervals (data not shown) was next performed for each combination of CRM type and those experimental variables found to be significant in the analysis of variance. The simultaneous confidence intervals identified whether a measured difference in lead recovery for a given pair of factor levels (e.g., Operator 1 vs Operator 2) was significant.

Box plots were prepared for each combination of CRM type and significant variable to illustrate the magnitude of the effects found in these analyses. The sections that follow (C2 through C7) present the box plots. Major observations from each analysis are given to the right of each box plot.

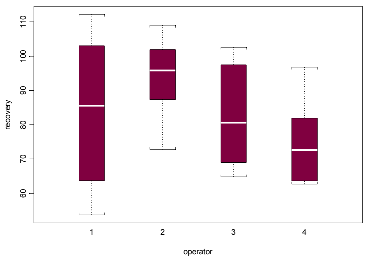
Table C1. Analysis of variance for each Certified Reference Material data set

Certified Reference Material	Experimental Variable	Analysis Result		
		F-Statistic	P-Value	Sign. Effect ?
SRM 2580	Operator	3.632	0.0279	Yes
	Wipe	9.095	0.0004	Yes
	Filter Treatment	0.287	0.5973	No
SRM 2581	Operator	19.904	0.0000	Yes
	Wipe	16.111	0.0000	Yes
	Lead Level	13.987	0.0000	Yes
	Filter Treatment	14.575	0.0002	Yes
SRM 2582	Operator	3.859	0.0233	Yes
	Wipe	6.623	0.00245	Yes
	Filter Treatment	6.572	0.0177	Yes
SRM 2584	Operator	7.979	0.0001	Yes
	Wipe	14.984	0.0000	Yes
	Lead Level	24.089	0.0000	Yes
	Filter Treatment	2.623	0.1087	No
SRM 2586	Operator	28.313	0.0000	Yes
	Wipe	52.839	0.0000	Yes
	Lead Level	33.516	0.0000	Yes
	Filter Treatment	4.549	0.0358	Yes
CRM 01450	Operator	10.960	0.0000	Yes
	Wipe	72.082	0.0000	Yes
	Lead Level	95.844	0.0000	Yes
	Filter Treatment	0.050	0.8233	No

[a] This column indicates whether the given combination of CRM and experimental variable had a significant effect on lead recovery.

C2. Statistical Analysis for SRM 2580. For SRM 2580, there was only one lead level used in the experiment so lead level was not a factor in the analysis. Operator and dust wipe were found to have a significant effect on lead recovery; whereas filter treatment had an insignificant effect (Table C1). The major observations are:

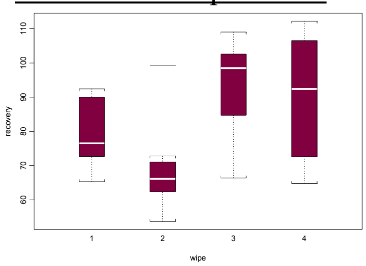
SRM 2580: Operator Effect



Observations from the Analysis:

- Operator 2 achieved the highest recovery (median ≈ 95 %), which was significantly higher than Operator 4, who had the lowest recovery (median ≈ 72 %).
- All other pair-wise comparisons were not significantly different.
- The results for Operators 1 and 3 were quite variable.

SRM 2580: Wipe Effect

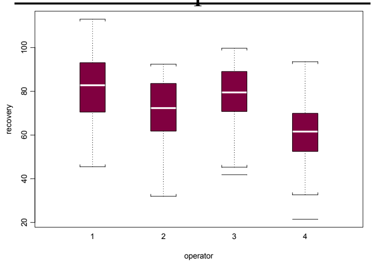


Observations from the Analysis:

- Wipe 2 had the lowest recovery (median ≈ 66 %), which was significantly worse than Wipe 3 or Wipe 4 (medians ≈ 101 % and ≈ 90 %, respectively).
- Wipe 3 had the highest recovery, which was not significantly different from that of Wipe 4.
- All other pair-wise comparisons were not significantly different.
- The results for Wipe 4 were quite variable.

C3. Statistical Analysis for SRM 2581. For SRM 2581, all variables—operator, dust wipe, filter treatment, and lead level—had a significant effect on lead recovery (Table C1). The major observations are:

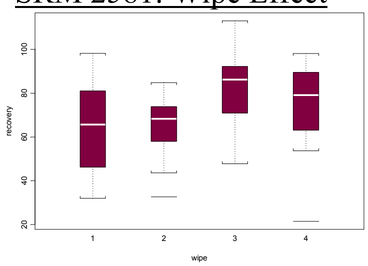
SRM 2581: Operator Effect



Observations from the Analysis:

- Operator 4 (median ≈ 60 %) was significantly lower than the other three operators (median range ≈ 71 % to ≈ 82 %).
- Operator 1 (median ≈ 82 %) was significantly higher than Operator 2 (median ≈ 71 %), but not Operator 3 (median ≈ 80 %).
- All other pair-wise comparisons were not significantly different.

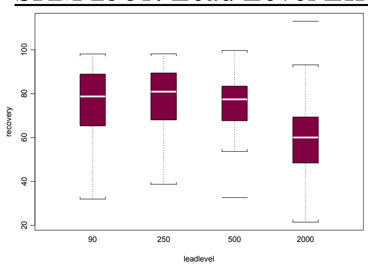
SRM 2581: Wipe Effect



Observations from the Analysis:

- Wipes 3 and 4 had significantly higher recovery than Wipes 1 and 2.
- Wipe 3 had the highest recovery (median ≈ 84 %), which was not significantly different from that of Wipe 4 (median ≈ 80 %).
- Wipe 1 had the lowest recovery (median ≈ 65 %), which was not significantly different from Wipe 2 (median ≈ 70 %).

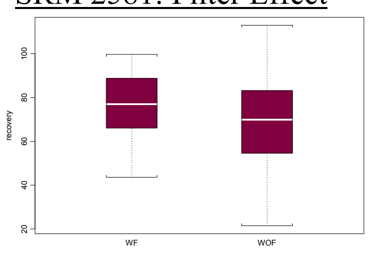
SRM 2581: Lead Level Effect



Observations from the Analysis:

- The 2000 μg Lead Level had the lowest recovery (median ≈ 60 %), which was significantly different from that of the other three lead levels (median range ≈ 77 % to ≈ 81 %).
- All other pair-wise comparisons were not significantly different.

SRM 2581: Filter Effect

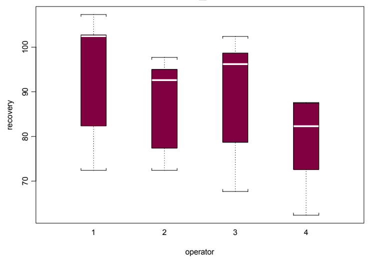


Observations from the Analysis:

- Analyses with filtering (WF) provided significantly higher recovery than those without filtering (WOF).

C4. <u>Statistical Analysis for SRM 2582</u>. For SRM 2582, there was only one lead level used in the experiment so lead level was not a factor in the analysis. Operator, dust wipe, and filter treatment all had a significant effect on lead recovery (Table C1). The major observations are:

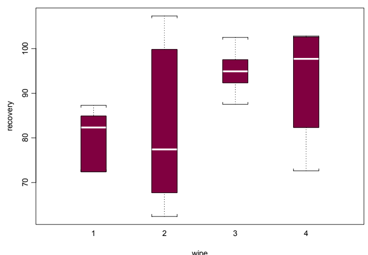
<u>SRM 2582: Operator Effect</u>



<u>Observations from the Analysis</u>:

- Operator 1 had the highest recovery (median ≈ 103 %), which was significantly different from that of Operator 4, who had the lowest recovery (median ≈ 82 %).
- Recoveries for Operators 1 through 3 ranged from ≈ 93 % to ≈ 103 %, (median values). Differences were not significant.
- All other pair-wise comparisons were not significantly different.

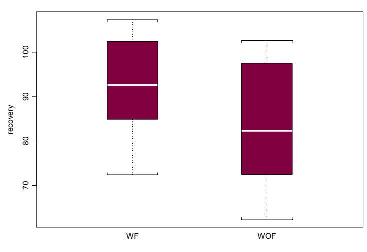
<u>SRM 2582: Wipe Effect</u>



<u>Observations from the Analysis</u>:

- Wipe 1 was significantly lower than Wipes 3 and 4.
- The highest recovery was for Wipe 4 (median ≈ 97 %), which was not significantly different from that of Wipe 3 (median ≈ 93 %).
- The lowest recovery was for Wipe 2 (median ≈ 77 %), which was not significantly different from that of Wipe 1 (median ≈ 82 %).
- All other pair-wise comparisons were not significantly different.
- Recovery for Wipe 2 was extremely variable.

<u>SRM 2582: Filter Effect</u>

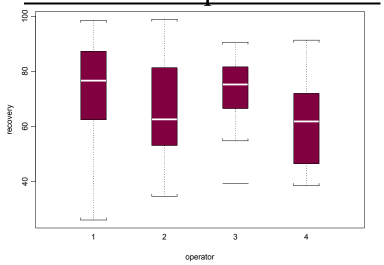


<u>Observations from the Analysis</u>:

- Analyses with filtering (WF) provided significantly higher recovery than those without filtering (WOF).

C5. Statistical Analysis for SRM 2584. For SRM 2584, three variables—operator, dust wipe, and lead level—had a significant effect on lead recovery (Table C1). The major observations are:

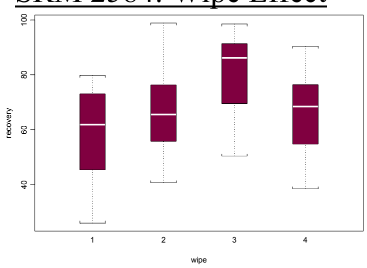
SRM 2584: Operator Effect



Observations from the Analysis:

- Operator 1 (median ≈ 78 %) and Operator 3 (median ≈ 77 %) had significantly higher recoveries than Operator 4 (median ≈ 60 %).
- The lowest median recovery was for Operator 4, which was not significantly different from that of Operator 2 (median ≈ 61 %).
- All other pair-wise comparisons were not significantly different.

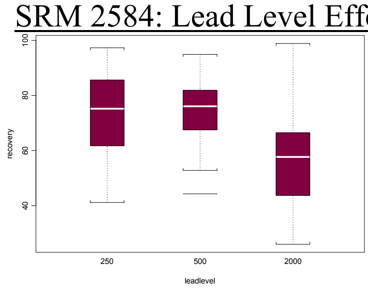
SRM 2584: Wipe Effect



Observations from the Analysis:

- Wipe 3 had the highest recovery (median ≈ 85 %), which was significantly different from the other three wipes (median range ≈ 61 % to ≈ 69 %).
- Wipe 1, Wipe 2, and Wipe 4 were not significantly different from each other.

SRM 2584: Lead Level Effect

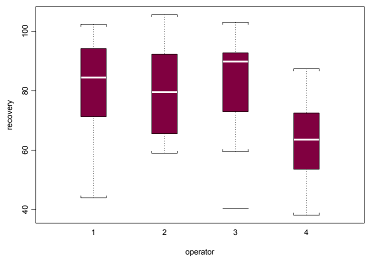


Observations from the Analysis:

- Recovery for the 2000 µg lead level (median ≈ 58 %) was significantly lower than that for the 250 µg and 500 µg lead levels (both medians ≈ 76 %).

C6. Statistical Analysis for SRM 2586. For SRM 2586, all variables—operator, dust wipe, filter treatment, and lead level—had a significant effect on lead recovery (Table C1). The major observations are:

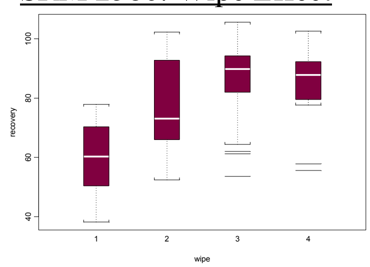
SRM 2586: Operator Effect



Observations from the Analysis:

- Operator 4 (median ≈ 62 %) had the lowest recovery, which was significantly different from that of Operators 1 through 3 (median range ≈ 80 % to≈ 90 %)
- Operator 3 had the highest recovery (median ≈ 90 %), which was not significantly different from that of Operators 1 and 2.

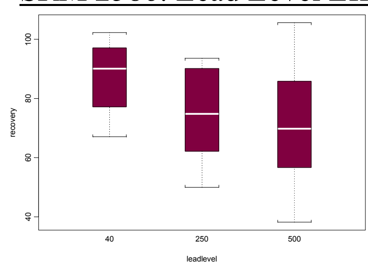
SRM 2586: Wipe Effect



Observations from the Analysis:

- Wipe 3 had the highest recovery (median ≈ 90 %), which was not significantly different from that of Wipe 4 (median ≈ 88 %).
- All other pair-wise comparisons were significantly different, with Wipe 1 having the lowest recovery (median ≈ 60 %).

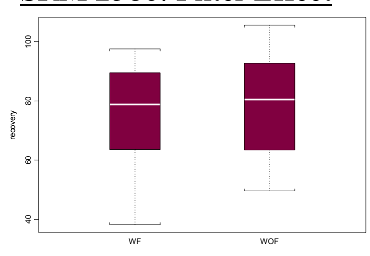
SRM 2586: Lead Level Effect



Observations from the Analysis:

- The 40 µg lead level had the highest recovery (median ≈ 90 %), which was significantly higher than that of the other two lead levels.
- The 250 µg lead level (median ≈ 75 %) and the 500 µg lead level (median ≈ 70 %) were not significantly different from each other.

SRM 2586: Filter Effect

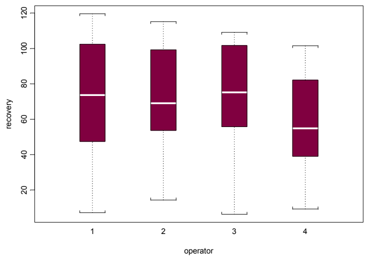


Observations from the Analysis:

- The analyses without filtering (WOF) provided significantly higher recovery than those with filtering (WF). Nevertheless, the median recovery for the unfiltered extracts was ≈ 81 % versus ≈ 79 % for the filtered extracts.

C7. Statistical Analysis for CRM 01450. For CRM 01450, three variables—operator, dust wipe, and lead level—had a significant effect on lead recovery (Table C1). The major observations are:

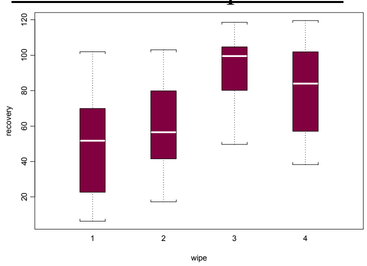
CRM 01450: Operator Effect



Observations from the Analysis:

- Operator 4 had the lowest recovery (median ≈ 53 %), which was significantly different from that of the other three operators (median range ≈ 68 % to ≈ 73 %).
- The recoveries for Operator 1 through Operator 3 were not significantly different from each other.

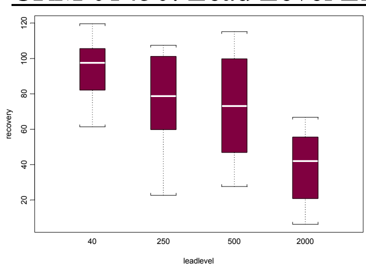
CRM 01450: Wipe Effect



Observations from the Analysis:

- Wipe 3 had the highest recovery (median ≈ 100 %), which was not significantly different from that of Wipe 4 (median ≈ 95 %).
- Wipe 1 had the lowest recovery (median ≈ 51 %), which was significantly different from that of Wipe 2 (median ≈ 56 %).
- All other pair-wise comparisons were significantly different.

CRM 01450: Lead Level Eff.



Observations from the Analysis:

- The 40 µg lead level (median ≈ 97 %) was significantly higher than the other three lead levels.
- The 2000 µg lead level (median ≈ 41 %) was significantly lower than the other three lead levels.
- The 250 µg lead level (median ≈ 79 %) and the 500 µg lead level (median ≈ 72 %) were not significantly different.