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Walter J. Rossiter, Jr.
Blaza Toman
Mary E. McKnight
Ijeoma Emenanjo

NIST

National Institute of Standards and Technology
Technology Administration, U.S. Department of Commerce

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Walter J. Rossiter, Jr.[*]
Blaza Toman[**]
Mary E. McKnight[*]
Ijeoma Emenanjo[*]

[*]Building and Fire Research Laboratory
[**]Information Technology Laboratory
National Institute of Standards and Technology
Gaithersburg, MD 20899-8615

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ABSTRACT

Knowledge of the amount of lead in settled dust is important in clearance examinations, risk assessments, and related activities wherein decisions are made regarding lead hazards in houses and related buildings. At present, analyses of lead in dust are normally performed in laboratories, since quantitative methods are not readily field-portable. In recent years, field portable ultrasonic extraction/anodic stripping voltammetry (UE/ASV) has been suggested as a candidate procedure for field analysis of dust wipe specimens. However, the reliability of UE/ASV analyses of field dust wipe specimens has not been demonstrated. The present study compared the results of ASV analyses of UE extracted field-sampled dust wipes against those of inductively coupled plasma (ICP) atomic emission spectrometry analyses of the same extract solutions. The main objective of the study was to investigate whether lead in dust wipe specimens obtained in the field can be efficiently and effectively extracted, and reliably quantified using common UE/ASV field procedures when the analyses are performed by certified risk assessors. The U.S. Department of Housing and Urban Development (HUD) sponsored the research because of the benefits to be gained in having available a reliable, practical method for on-site analysis of lead in dust in houses and related buildings.

In a preliminary laboratory phase, data on the use of an ICP procedure for estimating the true values of lead in dust wipe specimens were obtained. Experimental variables were: dust wipe, lead-containing certified reference material (CRM), specimen lead level, and filter treatment (i.e., filtering versus no filtering) of the UE extracts before ASV analyses. Lead recoveries determined by both ASV and ICP analyses were compared. The effects of wipe and lead level were significant, whereas the effects of CRM and filter treatment were insignificant. All ICP recoveries were quantitative (100 % ± 20 % of the lead applied to the wipe), whereas only 72 % of the ASV measurements were quantitative.

In the field phase, NIST research staff along with three certified lead risk assessors, who had participated in a previous NIST dust wipe study, sampled lead-containing dust from houses using the same dust wipe products used in the laboratory phase. Three experimental variables were examined: operator, wipe, and filter treatment of the UE extract. Percent lead recoveries by ASV analysis were calculated relative to the lead contents determined by ICP; 88 % of the recoveries were quantitative. None of the three variables was significant when the entire data set was considered. A filter treatment effect was present for a data subset comprised of specimens found to have less-than-500 µg of lead. Based on analyses of the entire data set and also on the less-than-500 µg data subset, the probabilities of a future ASV analysis yielding a result within ± 25 % of an ICP result were estimated to be 0.93 and 0.96, respectively. A recommendation for increasing the efficacy of the UE/ASV protocol is given.

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

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1. INTRODUCTION
1.1 Background

A major potential source of lead exposure, particularly for young children, is lead in dust [1]. Consequently, quantitative analysis to determine the amount of lead in dust is important for identifying and controlling lead hazards in houses and related buildings. At present, analyses of lead in dust are normally performed in laboratories, since quantitative methods are not readily field-portable. The availability of a reliable, practical field test procedure for analyzing lead in dust would be beneficial in expediting decisions regarding identification of dust lead hazards or safe reentry of housing subjected to lead hazard abatement.

Sampling of settled dust is generally performed by manually wiping a surface with a disposable towellette, called a dust wipe, that has been pre-moistened with a wetting agent. The dust wipe is then sent to a laboratory. Soluble lead is extracted and the extract solution is analyzed for lead using a method such as inductively coupled plasma atomic emission spectrometry (ICP-AES) or atomic absorption spectrometry (AAS). 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].

Field portable ultrasonic extraction/anodic stripping voltammetry (UE/ASV) has been suggested as a candidate procedure for field analysis of dust wipe specimens. UE/ASV studies performed using laboratory-prepared dust wipe specimens generally have shown that lead was quantitatively analyzed (i.e., recoveries were in the range of 100 % ± 20 %) [4-7]. However, the study by Ashley, Wise, Mercado, and Parry [5] also demonstrated limitations to the UE/ASV dust wipe methodology. Specifically, they found that only three of the four wipes studied were suitable for analysis by UE/ASV. Also, for some wipes, the UE extract solutions needed to be filtered before ASV analysis.

Recently, the National Institute of Standards and Technology (NIST) reported on the results of an UE/ASV study [8] that was conducted mainly to examine the effect of the operator on lead recovery from dust wipe specimens. In addition to studying operator effect, four other variables were examined: wipe, the lead-containing Certified Reference Material (CRM)[*] applied to the wipe, lead level of the dust wipe specimen, and filter treatment of the UE extract. A key finding was that lead recoveries were variable, ranging from < 20 % to quantitative depending upon the combination of experimental variables. All experimental variables had a significant effect on recovery, as did many of the two-way interactions. Three of the four operators obtained significantly higher lead recoveries than did the fourth operator. In this case, the fourth operator used a different test procedure than the other three. Two of the four wipes provided significantly higher recoveries than the other two. These results were consistent with those of Ashley et al. [5], who found variable lead recovery depending upon the combination of wipe and CRM.

UE/ASV analyses of field-sampled dust wipes have not been validated against measurements performed using a reference method such as ICP. This present study compares the results of ASV and ICP measurements of solutions obtained by ultrasonic extraction of field-sampled dust wipes.

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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" [9]. CRM is the generic term used in this report to denote all certified reference materials including NIST Standard Reference Materials (SRMs).

The majority of the field sampling and UE/ASV analyses were performed by certified risk assessors as such individuals would be expected to perform UE/ASV field analyses. The research was sponsored by the U.S. Department of Housing and Urban Development (HUD). At present, on-site dust analysis is not integral to federal programs for identifying, abating, and controlling lead hazards associated with lead in paint, dust, and soil in housing. This limitation could be reviewed if a practical field test procedure for determining lead in dust is available.

1.2 Objective and Scope

The main objective of the study was to investigate whether lead in dust wipe specimens sampled from housing can be efficiently and effectively extracted, and reliably quantified using common UE/ASV field procedures when the analyses are performed by certified risk assessors. A second objective was to compare the results of ASV and ICP measurements performed on UE extracts of dust wipe specimens. The study was performed in two phases: a laboratory phase and a field phase.

The laboratory phase was conducted to develop experience under idealized experimental conditions on using ICP analysis for estimating the true value of lead in dust wipe specimens, because ICP was selected as the reference test method to estimate the true lead values of the field specimens. In the laboratory phase, lead-containing dust wipe specimens were prepared using some of the same commercial dust wipe products and lead-containing CRMs used in the previous NIST dust wipe study [8] (See Section 2.3.1). NIST research staff conducted the UE/ASV analyses on these laboratory specimens. In the field phase, NIST research staff along with three certified lead risk assessors, who had participated in the previous NIST dust wipe study, sampled dust from houses with the same dust wipe products that were used in the laboratory phase of the study. The field dust wipe samples were brought to the NIST laboratories for UE/ASV analysis by the individual performing the field sampling. For both the laboratory and field specimens, the reliability of the UE/ASV method for determining lead in dust wipe specimens was examined by evaluating whether measured lead recoveries were quantitative; that is, were the lead recoveries within $\pm$ 20 % of the estimated true lead values. Determination of quantitative recovery has been used by others in judging the reliability of UE/ASV for analysis of lead in environmental media [5,10]. Also for the field specimens, UE/ASV reliability was examined vis-à-vis a National Institute for Occupational Safety and Health (NIOSH) criterion that "a method must give a result that is within $\pm$ 25 % of the true concentration value at least 95 % of the time" [11].

2. EXPERIMENTAL
2.1 UE/ASV Apparatus

A commercial, field-portable UE/ASV apparatus was used in the study.

2.1.1 ASV Instrument. The field-portable ASV instrument is battery operated, uses disposable electrodes, and was factory-calibrated. The lead content output is in units of micrograms (μg). The range of detection for lead in dust wipe specimens reported in the ASV instruction booklet is 25 μg to 1500 μg. For UE extracts found to have lead levels exceeding 1500 μg, dilution of the extract using 7.5 % nitric acid is performed before ASV re-analysis. The uncertainty of the ASV measurements, taken as the pooled standard deviation for mean lead recoveries determined for specimens prepared using six CRMs[*] but without a dust wipe present, was estimated to be 7 % [8].

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[*] A minimum of six specimens was analyzed for each CRM.

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Factory calibration of the ASV instrument was checked at the beginning of the study against eight standardized lead nitrate solutions of 7.5 % volume fraction nitric acid having lead contents ranging from 1 mg/L to 50 mg/L. The instrument remained in calibration, and 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 rechecked the instrument calibration 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 throughout the study, and that instrument drift over the course of the day did not occur.

2.1.2 <u>Sonicator</u>. The 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).

2.2 <u>UE/ASV Dust Wipe Protocol</u>

All UE/ASV analyses were conducted in the NIST laboratories. The dust wipe test protocol was in accordance with the UE/ASV apparatus instructions. This protocol, which has been previously described [8], involves the following steps:
- pushing the dust wipe specimen into 15 mL of 25 % volume fraction nitric acid in a 50 mL plastic screw-capped centrifuge tube using a disposable plastic stirring rod,
- rotating and tumbling the dust wipe specimen in the acid with the plastic stirring rod until trapped air bubbles in the wipe are apparently released and the wipe is well wetted,
- extracting the specimen for 30 min using the sonicator at an initial water bath temperature of 45 °C ± 3 °C,
- briefly suspending sonication to re-rotate and re-tumble the dust wipe specimen in the acid using the plastic stirring rod,
- extracting the specimen for an additional 15 min at 45 °C ± 3 °C using the sonicator,
- diluting the extract to 50 mL with water, and
- analyzing the resultant solution for lead using ASV.

Note that the initial 45 °C sonicator bath temperature deviated from the UE/ASV apparatus instructions, which prescribed water at room temperature. Water heated to 45 °C (taken from a laboratory tap) was used based on previous NIST results showing that UE/ASV lead recovery from laboratory-prepared paint film specimens is enhanced at higher bath temperatures [12].

2.3 <u>Dust Wipe Specimens</u>

2.3.1 <u>Laboratory-Prepared Specimens</u>. Two CRMs, each at three lead levels, and three commercial, individually packaged dust wipe products were used in preparing the laboratory specimens. The CRMs were SRM 2581 (NIST) at lead levels of 90 μg, 250 μg, and 500 μg, and CRM 01450 (Resource Technology Corp, Laramie, WY; available through Fisher)[†] at lead levels of 40 μg,

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[*] In a previous NIST UE/ASV study [12], aqueous acidic extractions of lead-containing specimens from a limited number of paint-film panels, and also from reference material samples, gave comparable lead recoveries when performed with and without ultrasound under the same conditions of temperature and time. It has been recommended [12] that sonicators be used for lead extraction in the field, because they provide a practical and affordable means for agitating and heating specimens during lead extraction. Consequently, a sonicator was used in this study. Studies have not been reported comparing the results of aqueous acidic extractions of lead-containing dust-wipe specimens performed with and without sonication.
[†] 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

250 µg, and 500 µg. These two CRMs were included in the previous NIST UE/ASV dust wipe study [8]. Both CRMs have been found to provide quantitative recovery when analyzed neat (i.e., no dust wipe was present) without filtering of the extract UE solution (Table 1).

The selection of the three dust wipes in this study was based on their performance in the previous NIST study [8]. Specifically, for three of the four operators, 96 % of the Wipe 2, 3, and 4 specimens subjected to UE followed by ICP analysis afforded quantitative lead recovery[*][†]. In contrast, only 13 % of the Wipe 1 specimens analyzed using UE/ICP showed quantitative lead recovery. Wipe 1 was not included in the present study. The suppliers of Wipes 2, 3, and 4 had reported [8] that these products conformed to the requirements of ASTM E 1792.

In the laboratory phase of the study, four replicate specimens were prepared for each combination of CRM, wipe, and lead level (Table 2) for a total of 72 specimens. The dust wipe preparation procedure was developed by the Research Triangle Institute (RTI) and used in the previous NIST study [8]. Thus, an appropriate amount of CRM as specified in the experimental design was deposited onto a wipe. One analytical balance was used for all weighing. The calibration of the balance was checked before and after each weighing period using either a 50 µg or a 100 µg Class 1 weight. Rubber gloves were worn throughout the procedure. In initiating the procedure, the bulk

Table 1. Certified reference materials (CRMs) used in the laboratory phase

| Certified Reference Material | | Lead Mass Fraction | | Results of UE/ASV Analyses[a] | | | | |
| | | | | | Lead Recovery, % | | | CoV[d] |
Designation	Material Type	%	n[b]	Min	Max	Mean	SD[c]	%
SRM 2581	Powdered Paint	0.449	6	83.9	92.8	89.3	3.0	3.3
CRM 01450	Bag House Dust	0.1914	11	92.3	111.2	103.8	6.2	5.9

[a] Analyses were performed on the CRM specimens prepared without dust wipes; UE extracts were not filtered.
[b] Number of analyses.
[c] Standard deviation.
[d] Coefficient of variation (standard deviation/mean*100).

Table 2. Combinations of CRMs, wipes, and lead levels used in the preparation of the laboratory dust wipe specimens[a]

| CRM ID | Lead in CRM % Mass Fraction | Wipe No. | Lead Level, µg | | |
			LL1	LL2	LL3
SRM 2581	0.449	2	90	250	500
		3	90	250	500
		4	90	250	500
CRM 01450	0.1914	2	40	250	500
		3	40	250	500
		4	40	250	500

[a] Four specimens were prepared for each combination.

container of CRM was tumbled several times, and floating dust in the container was allowed to settle before opening it. CRM was removed from the container using a spatula and transferred to a tared sheet of weighing paper. Wipes were prepared for receiving the CRM by opening the wipe packaging, removing the wet folded wipe, and reducing excess moisture in the wipe by hand squeezing. The wipe was then unfolded and briefly placed on a dry laboratory paper towel to soak up moisture. The wipe was then transferred to a flat plastic board for deposition of the CRM. The weighing paper containing the pre-weighed CRM was removed from the balance using tweezers and held over the unfolded wipe. By tilting the weighing paper, the CRM was allowed to slide onto the center of the wipe after which, while holding the weighing paper manually, it was tapped with the tweezers. The wipe was folded and placed in a 50 mL plastic centrifuge tube that was capped and labeled[*].

The amount of lead in each dust wipe specimen (i.e., estimated true lead value) was taken as the mass of CRM deposited onto a wipe multiplied by the certified % mass fraction of lead in the CRM. Lead recovery (LR %) determined by UE/ASV (or UE/ICP) analyses of a laboratory-prepared dust wipe specimen was calculated as:

$$LR \% \ = \ (UE/ASV \ Result/Estimated \ True \ Lead \ Value) \ x \ 100. \hspace{2cm} (Eq. \ 1a)$$

2.3.2 <u>Field Specimens</u>. NIST research staff along with three certified lead risk assessors (referred to as operators) followed the procedure in ASTM E 1728 [2] to obtain the field specimens from four vacant row houses in Baltimore, MD[†]. The houses were known to contain substantial amounts of lead and were awaiting rehabilitation. Little maintenance had been performed on the houses in recent years so that it was expected that wiping surfaces would pick up measurable amounts of lead. A total of 108 specimens were obtained: 36 by NIST research staff and 24 each by the three operators. Specimens were mainly sampled from interior window troughs and sills, and interior floors beneath windows and doors.

The three operators had participated in the previous NIST dust wipe study and, at the time, had been designated Operator 2, Operator 3, and Operator 4[‡] [8]. For comparison with the previous study, these operator designations were retained in the current study. When conducting the analyses of the 24 field specimens, each operator also analyzed at least two control specimens prepared using CRM 01450 without a dust wipe present.

2.3.2.1 <u>Lead Content</u>. The amount of lead (i.e., estimated true value) in each field specimen was unknown and needed to be determined by a reference test method. In this regard, after the UE/ASV analysis of a field specimen was performed, ICP lead analyses of both the UE extract solutions and the UE residues remaining in the centrifuge tubes (see Section 2.2) were conducted[§]. For the ICP analyses of the UE extracts, a 20 mL aliquot of each extract solution was decanted from the residue and filtered through a disposable 0.45 μm hydrophilic polytetrafluoroethylene (PTFE) filter cassette (Millipore Millex LCR) using a disposable polypropylene syringe. The filtrate (i.e., UE extract solution) was placed in a capped vial and sent to a National Lead Laboratory Accreditation Program (NLLAP) [13] laboratory. The NLLAP laboratory performed the lead analysis using the ICP procedure prescribed by EPA Method 6010B [14].

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[*] For specimens prepared without dust wipes, the weighed CRM was deposited directly into the centrifuge tube.
[†] The dust specimens were collected according to the procedure in a 2003 proposed revision of ASTM E 1728. Subsequent to the field sampling, the revision was approved by ASTM International [2].
[‡] The Operator designated as No. 1 in the previous study was not available to participate in the current study.
[§] The UE/ASV specimens in the laboratory-phase of the study were also subjected to the ICP reference procedure.

For the ICP analyses of the UE residues, the extract solution remaining in each centrifuge tube after PTFE filtering of the 20 mL aliquot was decanted and vacuum-filtered through Whatman No. 40 ashless filter paper. The PTFE filter was removed from its cassette and placed in the centrifuge tube along with the Whatman filter paper. The centrifuge tube was capped and sent to the NLLAP laboratory for lead analysis of its contents (i.e., residue and filters) using the hot plate digestion method prescribed by EPA Method 3050B [15] followed ICP analysis [14] of the digestion solution.

For approximately 40 % of the laboratory and field specimens, the amount of lead in the residue and filters was less than the NLLAP laboratory's ICP method-reporting limit (i.e., 20 μg). When the amounts of lead in the residues were reported by the NLLAP laboratory, 97 % ± 4 % (1 SD) of the lead was in the UE extract solutions. Accordingly, the true values of lead in the field specimens were estimated based on the NLLAP ICP lead contents determined for the UE extract solutions. The percent lead recovery (LR %) measured by a UE/ASV analysis of a field specimen was calculated as:

LR % = (UE/ASV Result/Estimated True Lead Value) x 100,   (Eq. 1b)
        where the estimated true lead value was based on the amount of lead in the
        UE extract solution, as determined by NLLAP ICP analysis.

3. RESULTS, ANALYSIS, AND DISCUSSION
3.1 Laboratory-Prepared Specimens

Tables A1 through A3 present[*] the amount of lead (in μg) determined by ASV and ICP analyses of the UE extract solutions for specimens prepared using Wipes 2, 3, and 4, respectively. The ASV analyses were conducted on unfiltered or filtered UE extracts; the ICP analyses were performed only on filtered UE extracts.

3.1.1 Effect of Wipe, CRM, Lead Level, and Filter Treatment. Because both filtered and unfiltered specimens of the same UE extracts were analyzed, repeated measures analysis of variance (ANOVA) [16] of the lead content data could be used to evaluate the effect of wipe, CRM, lead level, and filter treatment (Table 3). The effects of wipe and lead level were significant (p-value

Table 3. Analysis of variance examining effect of wipe, CRM, lead level, and filter
treatment for the laboratory-prepared specimens

Parameter	Analysis Result[a]	
	F-Value	P-Value
Wipe	12.65	**< 0.0001**
CRM	1.92	0.1708
Lead Level	93.69	**< 0.0001**
Filter Treatment	1.08	0.3019
Filter Treatment – Wipe	3.75	**0.0289**
Filter Treatment – CRM	0.14	0.7107
Filter Treatment – Lead Level	0.30	0.9128

[a] By convention, p-values less than 0.05 are considered to be statistically significant. Bold font is used to denote significant p-values.

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[*] Raw data from all UE/ASV analyses are given in Appendix A.

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< 0.05). In contrast, those for CRM and filter treatment were not significant. There was a significant two-way interaction between filter treatment and wipe.

The effects of wipe, CRM, lead level, and filter treatment are illustrated in Figure 1, which shows three bar plots representing lead recoveries for specimens prepared with Wipes 2, 3, and 4. As seen in Figure 1, lead recovery varied considerably, ranging from 32 % to quantitative depending on the combination of wipe, CRM and lead level. Seventy-two percent (72 %) of the recoveries were quantitative. In the previous NIST study [8], 71 % of the recoveries were quantitative for specimens prepared with Wipes 2, 3, and 4, and either SRM 2581 or CRM 01450. In each plot in Figure 1, a pair of adjoining bars having light and dark fills represents the effect of filter treatment for the same combination of CRM and lead level. Observe that, as expected from the ANOVA results, adjoining bars in each plot generally have comparable heights indicating the lack of a filter treatment effect. Observe also in Figure 1 that, for Wipe 4, two-thirds of the unfiltered extracts provided higher recoveries than the filtered extracts. This observation could explain the significant two-way interaction found between filter treatment and wipe (Table 3). The finding of higher recoveries for unfiltered extracts contrasts with previous NIST results [8] wherein 58 % of the filtered extracts from Wipe 4 specimens spiked with either SRM 2581 or CRM 01450 provided higher recoveries than the unfiltered extracts

The significant effect of wipe on lead recovery is also observable in Figure 1. As evident, Wipe 3 and Wipe 4 specimens provided generally higher recoveries than Wipe 2 specimens. This wipe effect was present in the previous NIST study [8], wherein Wipe 3 and Wipe 4 specimens had significantly higher recoveries than Wipe 2 specimens. Table 4 provides, as a function of combination of wipe and CRM, a summary of the percentages of the lead recoveries that were quantitative in the present and previous NIST studies. Consistent with the findings that Wipe 3 and Wipe 4 specimens had higher recovery than Wipe 2 specimens, the percentage of analyses providing quantitative recovery for Wipe 3 and Wipe 4 specimens was 75 % or more, whereas that for Wipe 2 specimens was 42 % or less.

The significant effect of lead level on lead recovery of Wipe 2 and CRM 01450 is also exhibited in Figure 1. For the combinations of these two parameters, it is seen that lead level 1 (LL1) specimens had recoveries that were much higher than lead level 2 (LL2) and lead level 3 (LL3) specimens.

Table 4. Percentage of analyses providing quantitative lead recovery in the current and previous NIST studies

| Wipe No. | Current NIST Study | | | | Previous NIST Study[a] | | | |
| | SRM 2581 | | CRM 01450 | | SRM 2581 | | CRM 01450 | |
	No. of Analyses	Quant.[b] %	No. of Analyses	Quant.[b] %	No. of Analyses	Quant.[b] %	No. of Analyses	Quant.[b] %
2	24	42	24	42	18	17	18	39
3	24	75	24	100	18	94	18	100
4	24	83	24	88	18	83	18	94

[a] The summary is for data from three of the four operators in the study, because the fourth operator used a different UE/ASV test protocol than the other three [8].

[b] This column indicates the percentage of the analyses for which the ASV lead recovery was quantitative.
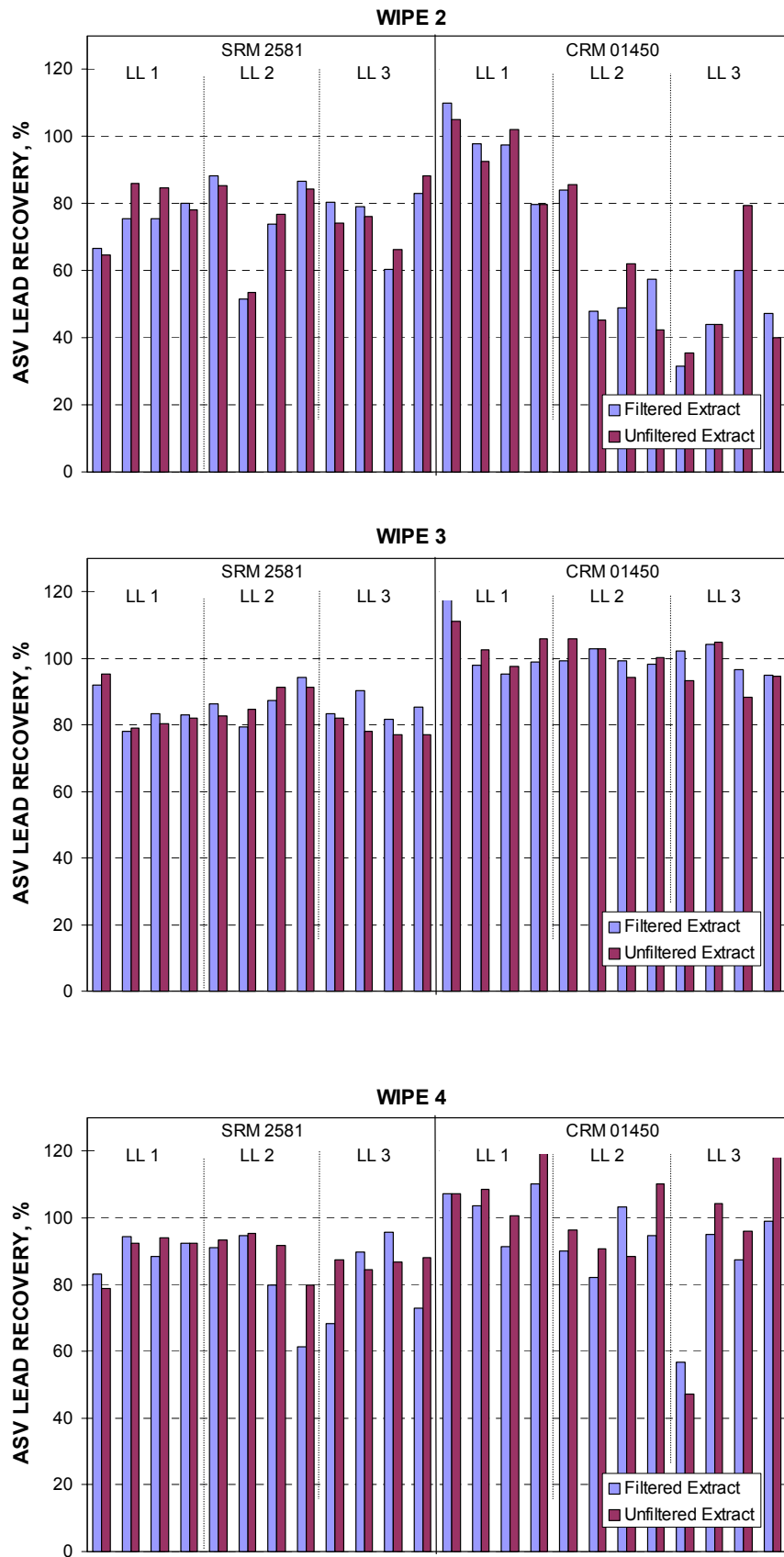
Figure 1. Laboratory-Prepared Specimens: The Effect of Filtering of the UE Extract on ASV Lead Recovery by Wipe, CRM, and Lead Level (LL). Recoveries are relative to the amount of lead applied on the dust wipes.

Specifically, whereas at LL1 all recoveries were quantitative with an average of about 95 %, at LL2 and LL3 most recoveries (88 %) were not quantitative with the majority ranging between 40 % and 60 %. This lead level effect for Wipe 2 and CRM 01450 specimens was also found in the previous NIST dust wipe study [8].

3.1.2 <u>Effect of Analytical Method</u>. ANOVA was conducted to determine the significance of the analytical method, i.e., ASV versus ICP, on lead recovery. From Table 5, the analytical method was significant (p-value < 0.05), as were the two-way interactions between analytical method and the three specimen variables, wipe, CRM, and lead level. Figure 2 illustrates the effect of analytical method on lead recovery. In each plot, a pair of adjoining bars having light and dark fills represents ASV and ICP recoveries, respectively, for the same combination of CRM and lead level. Figure 2 shows that all ICP recoveries were quantitative whereas, as previously mentioned, only 72 % of the ASV recoveries was quantitative. Specifically observe, for Wipe 2, the higher recoveries determined by ICP for specimens prepared using CRM 01450 at LL2 and LL3. The finding that all ICP measurements were quantitative supports the results from the NLLAP analyses (Section 2.3.2.1) that the UE procedure successfully extracted the lead from the laboratory-prepared dust wipe specimens [5].

In the previous NIST study [8], ASV and ICP comparative analyses were performed on UE extracts from three specimens prepared with Wipe 2 and CRM 01450 at the lead levels used in the present study. Two of these specimens were at LL3 and the other was at LL1. The two LL3 specimens in the previous study provided ASV and ICP recoveries of about 45 % and 95 %, respectively; whereas the LL1 specimen gave 104 % and 101 % recovery for the ASV and ICP analyses, respectively. That is, the trends for the ASV and ICP analyses for specimens prepared with Wipe 2 and CRM 01450 at various lead levels were similar for the present and previous NIST studies.

3.1.2.1 <u>ASV Versus ICP Plots</u>. Complementary to the bar charts (Figure 2) used to illustrate the effect of analytical method, Figure 3 provides plots of ASV lead recovery versus ICP lead recovery for Wipes 2, 3, and 4. In each plot, the data symbol denotes the CRM, and the 45° solid line represents perfect agreement between the two analytical methods. The Wipe 2 plot illustrates the effect of lead level for CRM 01450 specimens, as it is evident that almost all the data points for the Wipe 2 CRM 01450 specimens having about 250 µg of lead (LL2) and 500 µg of lead (LL3) fall below the perfect agreement line. Moreover, a couple of the data points for Wipe 2 specimens prepared with SRM 2518 at LL2 and LL3 also fall below the perfect agreement line.

Table 5. Analysis of variance examining effect of analytical method on lead recovery for the laboratory-prepared specimens, as determined by ASV and ICP

Parameter	Analysis Result[a]	
	F-Value	P-Value
Analytical Method (i.e., ASV versus ICP)	23.07	< **0.0001**
Analytical Method – Wipe	11.13	< **0.0001**
Analytical Method – CRM	4.36	**0.0408**
Analytical Method – Lead Level	5.81	**0.0002**

[a] By convention, p-values less than 0.05 were considered to be statistically significant. Bold font is used to denote significant p-values.

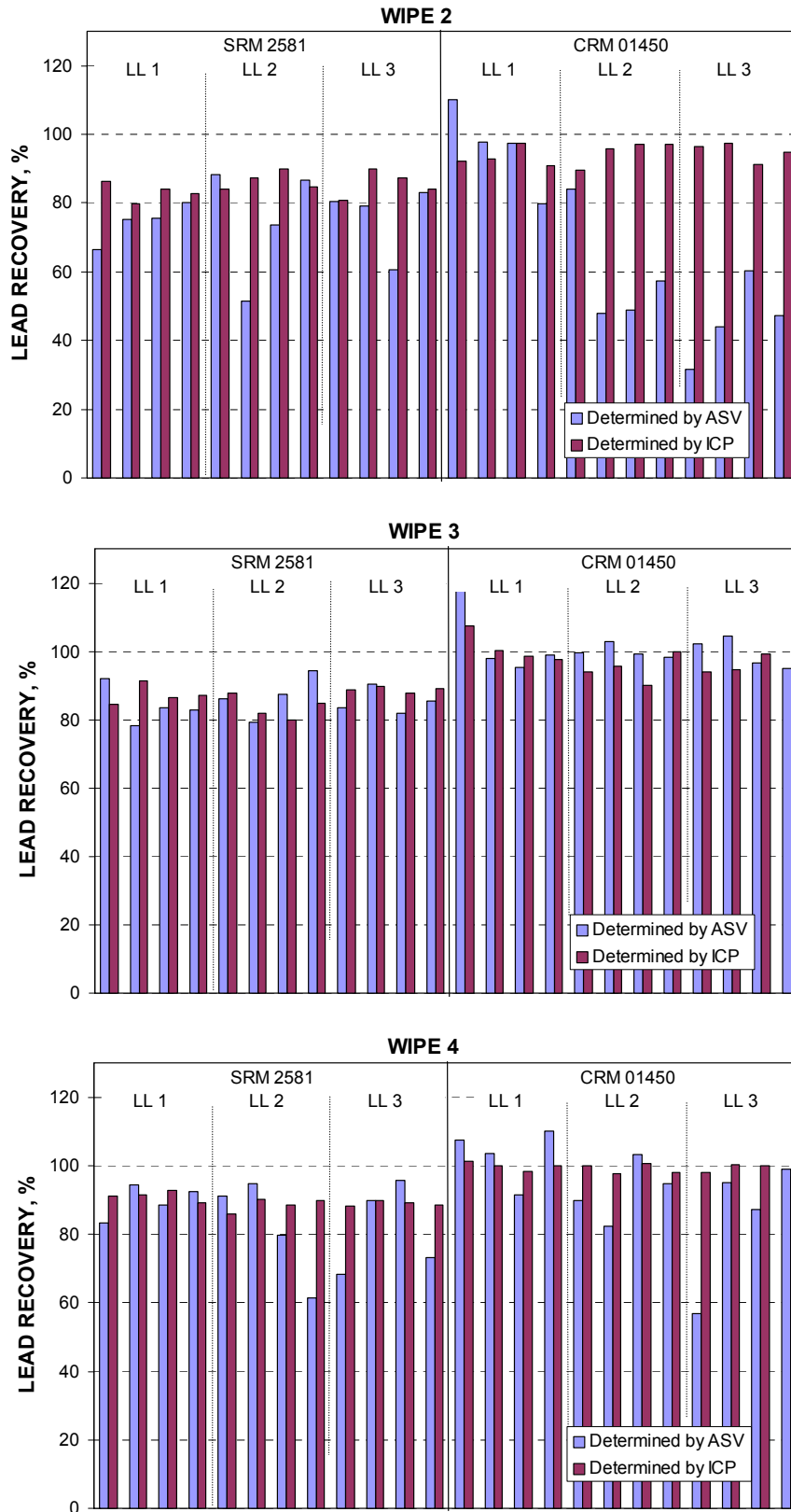Figure 2. Laboratory-Prepared Specimens: ASV Recovery Versus ICP Recovery by Wipe, CRM, and Lead Level (LL). For each method, recoveries are relative to the amount of lead applied on the dust wipes.
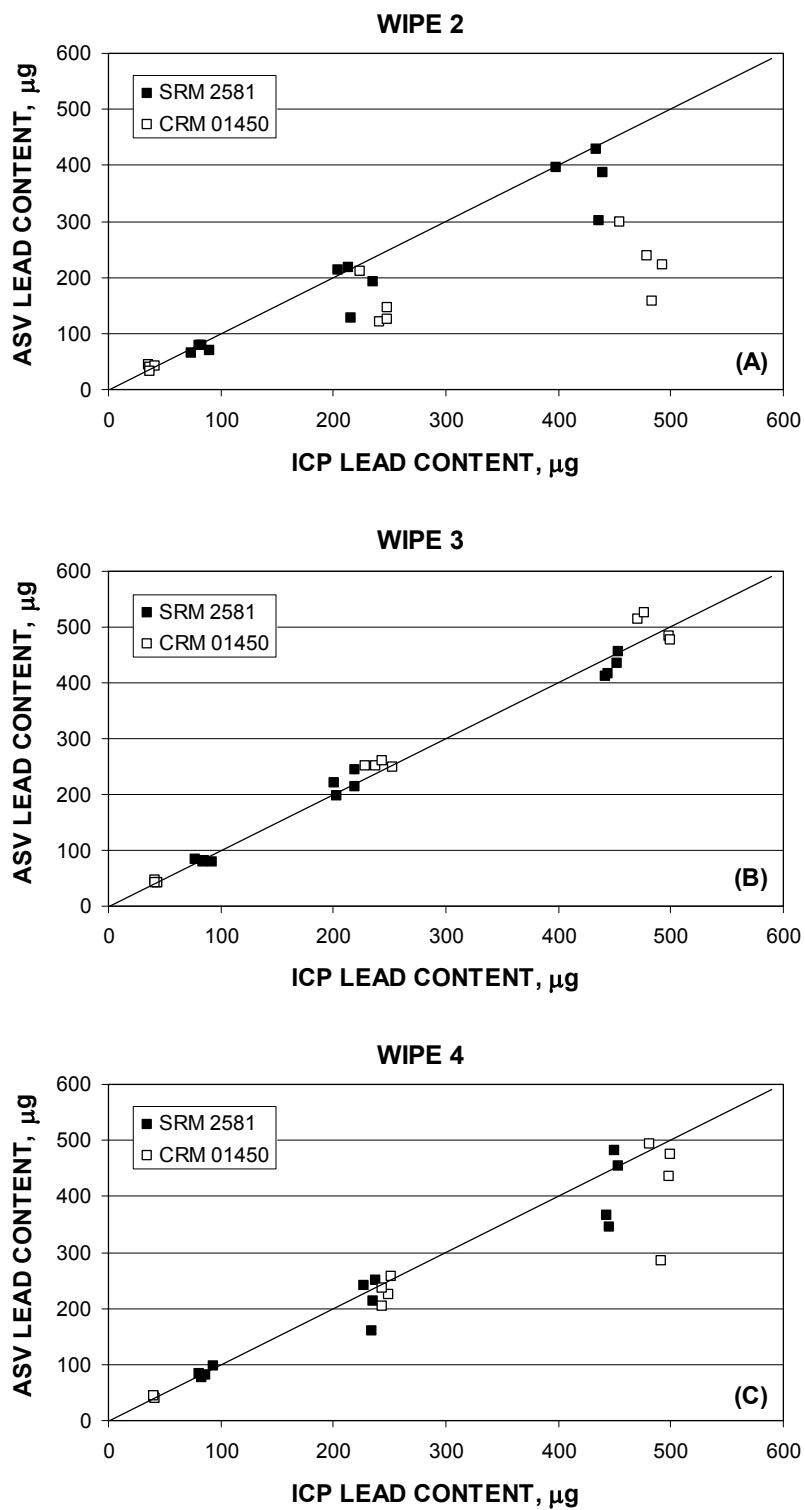
Figure 3. Laboratory-Prepared Specimens: Amount of Lead Measured by ASV Versus That Determined by ICP for Specimens Prepared Using Wipes 2, 3, and 4. In each plot, the 45° line represents perfect agreement between the two measurements.

3.2 Field Specimens

3.2.1 Ultrasonic Extraction. The finding that after sonication of the field specimens essentially all the lead was found by NLLAP ICP analysis to be in the UE extract (Section 2.3.2.1) indicated that the sonicator operation conditions were successful in extracting lead from these specimens. Successful sonicator extraction of lead in field-sampled dust, which has generally small particle size (sub-millimeter range), was consistent with the results of previous NIST research on UE/ASV analysis of lead-containing paints [12,17]. In those studies, lead in paint specimens was successfully extracted by sonication at temperatures and times similar to those used in the present study provided that the paint specimens were ground sufficiently small (i.e., generally < 425 μm particle size). Similarly, Drake et al. [11] found that lead in air particulate is readily extracted using UE (provided that lead-containing compounds that may need to be extracted using hydrofluoric acid are not present). Drake et al.'s sonication temperature and time were similar to those used in the present dust wipe study, although the extraction solution was 10 % (volume fraction) nitric acid as opposed to the 25 % nitric acid solution used here. On the other hand, in the previous NIST [8] and the Ashley et al. [5] dust wipe studies, it was observed that, for some combinations of wipe and CRMs (which are powdered), the ultrasonic extraction did not successfully solubilize lead in the dust wipe specimens.

3.2.2 UE/ASV Lead Content. Table A4 contains the results of the UE/ASV analyses of the field specimens sampled by NIST research staff. Similarly, Tables A5, A6, and A7 give the results of the UE/ASV analyses of the field specimens sampled by Operators 2, 3, and 4, respectively. The amount of lead measured in the field specimens varied considerably, ranging from about 25 μg to more than 17 000 μg, although 58 % had less than 500 μg. These findings were consistent with the experimental plan that the houses chosen for sampling would provide specimens having measurable amounts of lead. Nevertheless, four specimens sampled by NIST research staff (Table A4) afforded UE/ASV results that were "below the detection limit" (i.e., 25 μg). The amount of lead found in the UE extracts for each of these four specimens by NLLAP ICP analysis was 25 μg or less.

The results of the UE/ASV analyses of the field specimens are plotted in Figure 4 for each operator as a function of filter treatment of the UE extracts before ASV analyses. For clarity in presenting the data for specimens having low lead contents (e.g., < 100 μg), the y-axis scale for lead content is truncated at 2000 μg. Note that many of the pairs of adjacent bars have comparable height suggesting little difference between the ASV analyses of the filtered and unfiltered extracts. Repeated measures ANOVA [16] was performed on the entire data set to examine the effect of filter treatment, operator, and wipe on lead recovery (Table 6). Consistent with Figure 4, the ANOVA results for the entire data set indicated that filter treatment and the two-way interactions of filter treatment with operator and wipe were not significant (p-value > 0.05). Additionally, the effects of operator and wipe were also not significant. The lack of a wipe effect in the field phase was in contrast with the wipe effect observed in the laboratory phase of the present study (Section 3.1.3) and also in the previous NIST dust wipe study [8].

Repeated measures ANOVA was also conducted on a data subset that contained only those points for which the amount of lead determined by ICP analysis was < 500 μg. Analyses of dust wipe specimens found to have low lead contents play a role in identifying lead hazards in residential properties, as defined in federal regulations [18]. For example, dust clean-up after lead abatement activities is considered to be inadequate if lead levels in dust on floors, interior windowsills, and
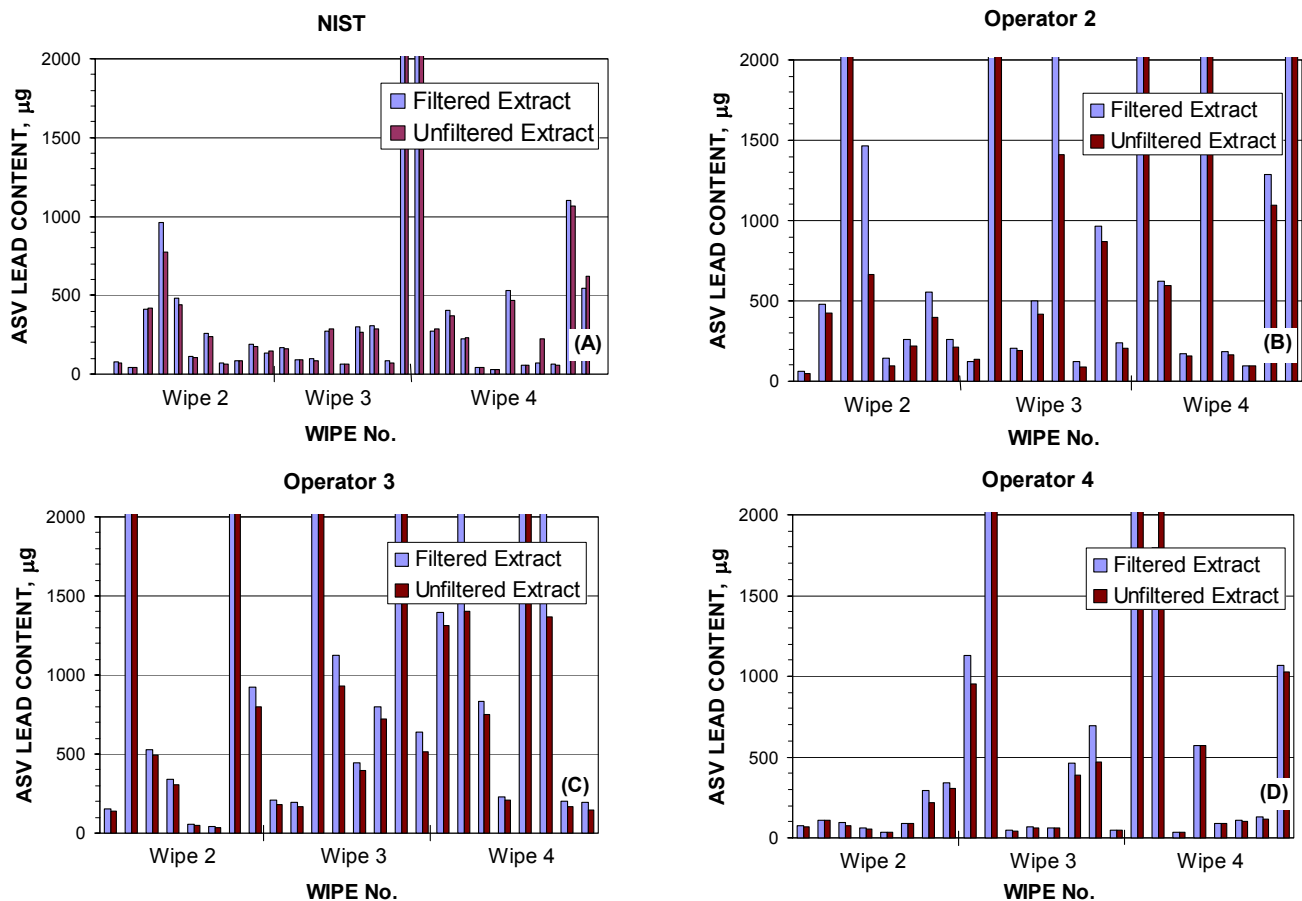
Figure 4. Field Specimens: Lead Content Determined by ASV Analyses of Filtered and Unfiltered UE Extracts for Each Operator.

Table 6. Analysis of variance examining effect of operator, wipe, and filter treatment for the field specimens

Data Set Analyzed[a]	Parameter	Analysis Result[b]	
		F-Value	P-Value
All Data	Operator	2.51	0.0631
	Wipe	0.08	0.9205
	Filter Treatment	1.59	0.2109
	Filter Treatment – Operator	0.54	0.6569
	Filter Treatment – Wipe	0.81	0.4476
Less-Than-500 μg	Operator	1.41	0.2507
	Wipe	0.05	0.9468
	Filter Treatment	9.50	**0.0032**
	Filter Treatment – Operator	2.87	**0.0447**
	Filter Treatment – Wipe	1.65	0.2015

[a] ANOVAs of the field data were performed twice: (1) using all points in the data set, and (2) using a data subset containing only those points for which the amount of lead determined by ICP analysis was < 500 μg.

[b] By convention, p-values less than 0.05 are considered to be statistically significant. Bold font is used to denote significant p-values.

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window troughs are equal to or exceed 40 µg/ft^2, 250 µg/ft^2 and 400 µg/ft^2, respectively.[*] For the less-than-500 µg data subset, a filter treatment effect was found (Table 6), with the filtered UE extracts providing greater recovery than the unfiltered extracts. In addition, an interaction between filter treatment and operator was observed. The filter treatment effect for the less-than-500 µg subset supports previous recommendations [5] that filtering of UE dust wipe extracts should be performed before conducting ASV analyses to enhance lead recovery.

3.2.2.1 <u>ASV Versus ICP Plots</u>. Figure 5 compares the results of the ASV and ICP lead analyses determined by all operators[†]. The left linear regression plot (A) shows all data with the exception of the data point of about 17 000 µg of lead determined by Operator 2 (Table A5)[‡]. The right linear regression plot (B) presents the less-than-500 µg data subset. In both the (A) and (B) plots, the plot character denotes the wipe number. The dashed line is that from the linear regression analysis. The 45° solid line represents perfect agreement between the two measurement methods. Table 7 gives the coefficients for the linear regression analyses. For the entire data set, the slope and intercept were not significantly different from 1 and 0, respectively. For the less-than-500 µg data subset, the slope was significantly different from 1, but it was only about 12 % less. The intercept was again not significantly different from 0. For both data sets, as evident in Figure 5, the variability around the regression line was small. This observation is quantified by r^2-values (Table 7) of 0.99 and 0.95 for the entire data set and for the less-than-500 µg data subset, respectively.
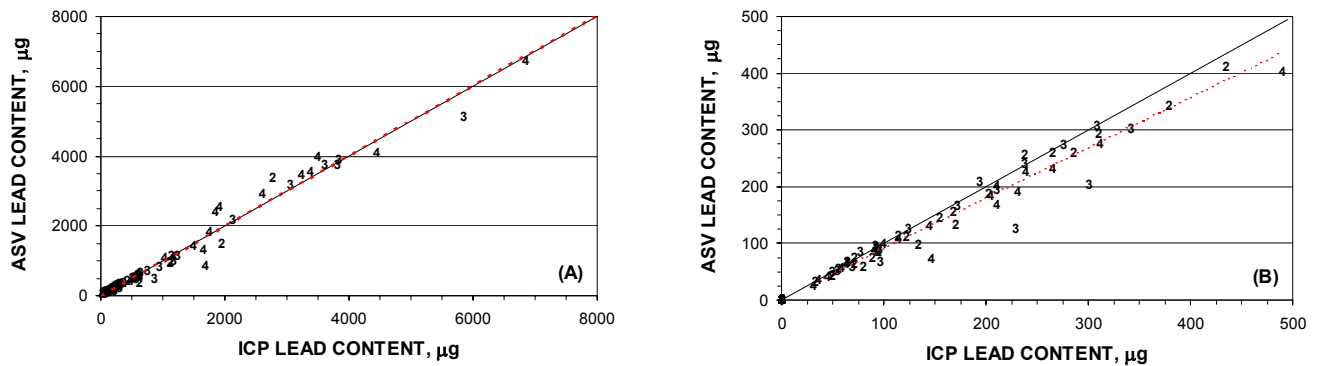
Figure 5. Field Specimens: ASV Results Versus ICP Results. The left plot (A) shows all data with exception of one point with a lead content of ≈ 17 000 µg that was not presented for purposes of clarity. The right plot (B) is for the less-than-500 µg data subset. In each plot, the plot character represents the wipe number. The dashed line is that from the linear regression analysis. The solid 45° line represents perfect agreement between the two measurements.

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[*] Micrograms per squared foot (µg/ft^2) is the unit of measurement for dust levels prescribed in federal regulations [18]; sampling is often performed on a 0.09 m^2 (1 ft^2) area.
[†] Comparison of the ASV and ICP lead recoveries by operator is a main objective of the study so regression plots of the ASV and ICP results by operator are presented in Appendix B.
[‡] The data point was included in the regression analysis.

3.2.3 <u>Lead Recovery</u>. Figure 6 shows the percent lead recoveries (using filtered extracts) by operator relative to the amount of lead determined by ICP analyses. Eighty-eight percent (88 %) of the 104 UE/ASV field specimen analyses providing results greater than the ASV instrument detection limit were quantitative. In comparison, in the laboratory phase of the study, 72 % of the UE/ASV analyses were quantitative.

Table 7. Linear regression analysis coefficients for the ASV versus ICP comparison for all field data

Operator	Data Set Analyzed[a]	Coefficients				r^2-value	Significant[c]	
		Slope[b]		Intercept[b]			Slope	Intercept
All Ops.	All Data	1.008	(0.010)	- 40.553	(22.937)	0.991	No	No
	< 500 µg	0.883	(0.026)	3.531	(4.972)	0.953	Yes	No

[a] Linear regression analyses were performed twice: (1) using all points in the data set, and (2) using a data subset containing only those points for which the amount of lead determined by ICP analysis was < 500 µg.
[b] The value in parentheses is the standard error.
[c] These two columns indicate whether the slope and the intercept were significantly different from 1 and 0, respectively.
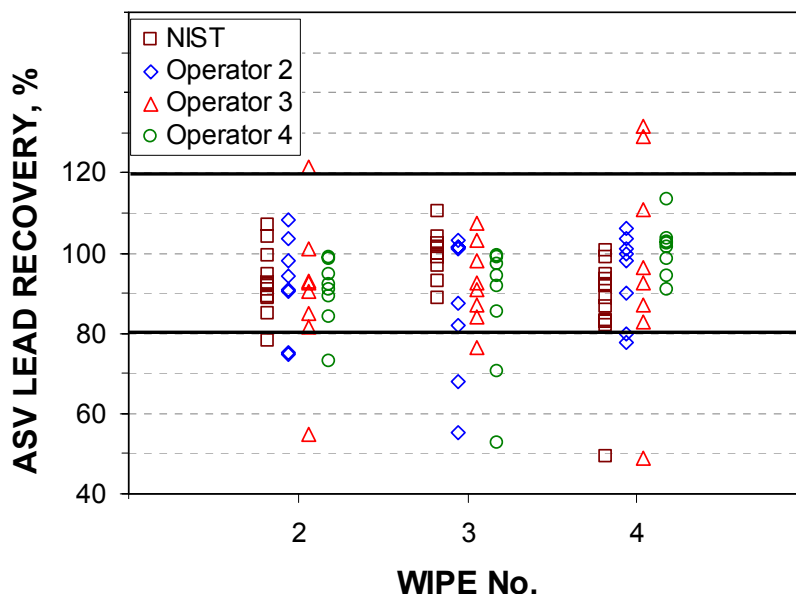
Figure 6. Field Specimens: Percent ASV Lead Recovery. The percent ASV lead recovery is relative to the amount of lead determined by ICP analysis. A recovery falling on or between the two horizontal bold lines is quantitative, i.e., recovery is 100 % ± 20 % of the estimated true lead value.

3.2.4 <u>Comparison with the NIOSH Criterion</u>. The National Institute for Occupational Safety and Health (NIOSH) prescribes that "a method must give a result that is within ± 25 % of the true concentration value at least 95 % of the time" [11]. Statistical analysis of the field specimen results was performed to examine the reliability of the UE/ASV method vis-à-vis the NIOSH criterion. The calculation was based on the predictive probability distribution of a future observation [19], and used ICP results as the estimated true lead values of the field specimens. Using the entire NIST data set for all operators and an estimated 8 % uncertainty for the ICP measurements, the probability of a future ASV analysis yielding a result within ± 25 % of an ICP result was estimated to be 0.93. This value was low in comparison with the NIOSH criterion that, for at least 95 % of the time, the result should be within ± 25 % of the true concentration. For the less-than-500 µg data set for all operators, the probability of a future ASV analysis yielding a result within ± 25 % of an ICP result was estimated to be 0.96, which was in agreement with the NIOSH criterion.

3.3 <u>Future Work</u>.

The evidence from this and previous studies [4-8] suggests that UE/ASV can be a viable procedure for the analysis of lead in dust. However, an important factoring affecting the reliability of UE/ASV analysis is the wipe used for the dust sampling, as this and other studies [5,8] using specimens prepared from commercial dust wipes and a variety of CRMs have shown a significant dust wipe effect. Not all wipes have been found to be suitable for use with UE/ASV analyses. A need exists to identify the attributes of wipes that make them exceptionally effective in the UE/ASV protocol. Once these attributes have been identified and their effectiveness verified, then it would be possible to publish these attributes and amend ASTM Standards such as E 1792 [3] or E 1775 [20] so that only effective wipes are used in the UE/ASV protocol—thus increasing the efficacy of the UE/ASV protocol.

4. SUMMARY AND CONCLUSIONS

Knowledge of the amount of lead in settled dust is important in clearance examinations, risk assessments, and related activities wherein decisions are made regarding the identification of lead hazards in houses and related buildings. For example, under certain circumstances such as re-occupancy of the living space after lead abatement, it could be worthwhile having a test procedure for on-site dust analysis so that quick decisions regarding any hazards due to lead in dust could be made. At present, dust analyses are performed in laboratories, because quantitative analysis methods generally used for analysis of lead in dust are not readily field-portable. In recent years, field portable ultrasonic extraction/anodic stripping voltammetry (UE/ASV) has been suggested as a method for field analysis of dust wipe specimens. A reservation to accepting UE/ASV for field use is that the method's response in analyzing field-sampled dust wipe specimens has not been demonstrated. As a step toward overcoming this limitation, this study sponsored by the U.S. Department of Housing and Urban Development (HUD) compared the results of ASV analyses of UE extracted field-sampled dust wipes with the results of ICP analyses of the same extract solutions. The main objective was to investigate whether lead in dust wipe specimens obtained in the field can be efficiently and effectively extracted and reliably quantified using common UE/ASV field procedures when the sampling and analyses are performed by certified risk assessors.

In the laboratory phase, data on the use of an ICP procedure for estimating the true value of lead in dust wipe specimens were obtained, because ICP was selected as the reference test method for estimating the true lead values of the field specimens. Seventy-two (72) specimens including four replicates per wipe, CRM and lead level combination were prepared by applying two lead-

containing CRMs at three lead levels on three commercial dust wipe products. Lead in the specimens was extracted using ultrasonication, and ASV analyses were performed on both filtered and unfiltered UE extracts. In addition, ICP analyses were conducted on filtered UE extracts, and compared with the ASV analyses. The reliability of the UE/ASV method for determining lead in these dust wipe specimens was examined by evaluating whether the lead recoveries were quantitative; that is 100 % ± 20 % of the estimated true lead value. For the laboratory specimens, the estimated true lead values were taken relative to the amounts of CRM deposited on the wipes.

Key findings of the laboratory phase were:
- The wipe effect was significant; two of the three wipes provided higher recovery than the third wipe.
- The lead level effect was significant; one of the three lead levels incorporated in specimens prepared with one wipe and one CRM provided higher recovery than the other two lead levels.
- The CRM effect was insignificant.
- The filter treatment effect was insignificant, although the two-way interaction between filter treatment and wipe was significant. For one wipe, the unfiltered extracts provided higher recoveries than the filtered extracts.
- ICP recoveries were significantly higher than ASV recoveries. All ICP recoveries were quantitative, whereas only 72 % of the ASV recoveries were quantitative.
- The quantitative ICP analyses indicated that the sonicator operating conditions successfully extracted essentially all the lead from the specimens.

In the field phase, three experimental variables were examined for their effect on dust wipe lead recovery: operator, wipe, and filter treatment of the UE extract before ASV analysis. NIST research staff along with three certified lead risk assessors, who had participated in a previous NIST dust wipe study, obtained 108 dust wipe specimens from houses using the three dust wipe products incorporated in the laboratory phase of the study. The individual who performed the field sampling conducted the UE/ASV analyses at NIST. ASV analyses were conducted on filtered and unfiltered UE extracts. The true lead values of the specimens were estimated using the results of ICP analyses. UE/ASV lead recoveries were calculated relative to the lead contents determined by the ICP. As in the laboratory phase, the reliability of the UE/ASV method for determining lead in these dust wipe specimens was examined by evaluating whether the lead recoveries were quantitative. Additionally, UE/ASV reliability was analyzed vis-à-vis a National Institute for Occupational Safety and Health (NIOSH) criterion that "a method must give a result that is within ± 25 % of the true concentration value at least 95 % of the time." Statistical analyses of the field specimen results were performed for the set of all data collected by the operators, and also for a data subset consisting of those points for which the ICP lead results were less than 500 µg.

Key findings of the field phase were:
- None of the three variables incorporated in the field phase, operator, wipe, and filter treatment, was significant when the entire data set was analyzed. A filter treatment effect was present for the less-than-500 µg data subset.
- The lack of a wipe effect contrasted with the results of the laboratory phase in which two wipes afforded significantly higher recoveries than the third wipe.
- Eighty-eight percent (88 %) of the field-specimen analyses provided quantitative recovery.
- Ultrasonic extraction was successful in solubilizing the lead from the field specimens.
- Based on analyses of the entire data set and also of the less-than-500 µg data subset, the probabilities of a future ASV analysis yielding a result within ± 25 % of an ICP result were estimated to be 0.93 and 0.96, respectively.

The results of this and previous studies suggest that UE/ASV lead analysis of dust wipes can be a viable procedure provided that suitable wipes are selected for dust sampling and subsequent analysis. Not all wipes have been found to be suitable for use with UE/ASV analyses. A need exists to identify and verify those attributes that make wipes exceptionally effective in the UE/ASV protocol. Future work should be directed toward this goal.

5. ACKNOWLEDGMENTS

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APPENDIX A. UE/ASV DATA

This appendix presents tables of data from all UE/ASV and UE/ICP analyses performed during the study. Tables A1 through A3 summarize the data for the laboratory-prepared specimens, while Tables A4 through A7 summarize the data for the field specimens.

Table A1. Results of lead analyses of laboratory specimens prepared using Wipe 2

| Wipe No. | CRM ID | Target Lead Level μg | Replicate No. | Lead Spiked on Wipe μg | Lead Content of UE Extract | | |
					ASV-UFE[b] μg	ASV-FE[c] μg	ICP-FE[d] μg
2	SRM 2581	90	1	104	67	69	90
			2	103	89	78	83
			3	87	74	66	74
			4	97	76	78	81
2	SRM 2581	250	1	243	207	214	204
			2	247	132	127	216
			3	262	201	193	236
			4	252	212	218	213
2	SRM 2581	500	1	493	365	396	399
			2	489	373	387	440
			3	500	332	302	437
			4	517	456	429	434
2	CRM 01450	40	1	39	41	43	36
			2	40	37	39	37
			3	42	43	41	41
			4	40	32	32	37
2	CRM 01450	250	1	250	214	210	224
			2	252	114	121	241
			3	255	158	125	248
			4	256	108	147	248
2	CRM 01450	500	1	501	177	158	484
			2	505	223	223	493
			3	499	396	300	455
			4	505	202	238	479

[a] ASV-UFE indicates ASV measurement of the unfiltered extract.
[b] ASV-FE indicates ASV measurement of the filtered extract.
[c] ICP-FE indicates ICP measurement of the filtered extract.

A1

Table A2. Results of lead analyses of laboratory specimens prepared using Wipe 3

Wipe No.	CRM ID	Target Lead Level μg	Replicate No.	Lead Spiked on Wipe μg	Lead Content of UE Extract		
					ASV-UFE[b] μg	ASV-FE[c] μg	ICP-FE[d] μg
3	SRM 2581	90	1	91	87	84	77
			2	101	80	79	93
			3	98	79	82	85
			4	96	79	80	84
3	SRM 2581	250	1	249	206	215	219
			2	248	210	197	203
			3	252	230	220	201
			4	259	236	244	220
3	SRM 2581	500	1	499	410	417	444
			2	505	395	456	454
			3	504	389	412	442
			4	508	391	434	452
3	CRM 01450	40	1	39	43	46	42
			2	44	45	43	44
			3	44	43	42	44
			4	42	45	42	42
3	CRM 01450	250	1	253	267	251	238
			2	254	261	261	243
			3	254	239	252	229
			4	254	254	249	253
3	CRM 01450	500	1	502	468	513	471
			2	503	527	525	476
			3	501	443	484	498
			4	502	474	476	500

[a] ASV-UFE indicates ASV measurement of the unfiltered extract.
[b] ASV-FE indicates ASV measurement of the filtered extract.
[c] ICP-FE indicates ICP measurement of the filtered extract.

Table A3. Results of lead analyses of laboratory specimens prepared using Wipe 4

Wipe No.	CRM ID	Target Lead Level µg	Replicate No.	Lead Spiked on Wipe µg	Lead Content of UE Extract		
					ASV-UFE[b] µg	ASV-FE[c] µg	ICP-FE[d] µg
4	SRM 2581	90	1	91	72	76	83
			2	103	95	97	94
			3	93	87	82	86
			4	91	84	84	81
4	SRM 2581	250	1	264	246	240	227
			2	264	252	250	238
			3	266	244	212	235
			4	261	208	160	235
4	SRM 2581	500	1	505	442	345	446
			2	505	426	453	454
			3	504	438	482	451
			4	500	440	365	443
4	CRM 01450	40	1	40	43	43	41
			2	41	44	42	41
			3	43	43	39	42
			4	40	49	44	40
4	CRM 01450	250	1	249	240	224	249
			2	249	226	205	243
			3	249	220	257	251
			4	249	275	236	244
4	CRM 01450	500	1	501	236	285	492
			2	498	519	474	500
			3	498	478	435	498
			4	498	595	493	481

[a] ASV-UFE indicates ASV measurement of the unfiltered extract.
[b] ASV-FE indicates ASV measurement of the filtered extract.
[c] ICP-FE indicates ICP measurement of the filtered extract.

Table A4. Results of lead analyses of NIST field specimens

| Specimen No. | Operator ID | Wipe No. | Lead Content of UE Extract | | | Lead Recovery[c] |
			ASV−UFE[a] μg	ASV−FE[b] μg	ICP−FE[b] μg	%
1	NIST	2	68	74	71	104.2
2		2	39	43	49	88.7
3		2	417	412	435	94.8
4		2	777	960	1130	85.0
5		2	439	480	520	92.3
6		2	105	113	114	99.6
7		2	234	259	285	90.9
8		2	63	68	64	107.1
9		2	84	84	94	89.4
10		2	173	187	202	92.6
11		2	143	133	170	78.2
12		2	< ADL[d]	< ADL	25	--
13	NIST	3	160	166	172	96.8
14		3	89	89	96	93.2
15		3	84	95	92	103.8
16		3	288	273	276	99.1
17		3	63	65	64	102.4
18		3	265	303	341	88.9
19		3	284	307	308	99.7
20		3	72	84	76	110.5
21		3	2890	2160	2130	101.4
22		3	< ADL	< ADL	20	--
23		3	< ADL	< ADL	11	--
24		3	< ADL	< ADL	18	--
25	NIST	4	4315	4080	4450	91.7
26		4	285	275	311	88.6
27		4	367	402	490	82.1
28		4	232	226	239	94.8
29		4	41	41	45	92.1
30		4	29	25	30	83.3
31		4	468	527	635	83.0
32		4	59	57	58	99.1
33		4	221	72	147	49.1
34		4	59	63	63	100.8
35		4	1069	1103	1180	93.5
36		4	620	542	625	86.7

[a] ASV-UFE indicates ASV measurement of the unfiltered extract.
[b] ASV-FE and ICP-FE indicate ASV and ICP measurements, respectively, of the filtered extract.
[c] Lead recovery (%) = [(ASV−FE/ICP−FE) x 100].
[d] < ADL indicates the result was below the ASV detection limit (i.e., 25 μg).

Table A5. Results of lead analyses of Operator 2 field specimens

Specimen No.	Operator ID	Wipe No.	Lead Content of UE Extract			Lead Recovery[c] %
			ASV–UFE[a] μg	ASV–FE[b] μg	ICP–FE[b] μg	
1	2	2	47	59	79	74.7
2		2	423	478	525	91.0
3		2	19 550	17 300	16 700	103.6
4		2	663	1467	1950	75.2
5		2	93	145	154	94.2
6		2	219	257	237	108.4
7		2	400	556	615	90.4
8		2	215	259	265	97.9
9	2	3	139	126	229	55.1
10		3	3730	3885	3835	101.3
11		3	194	204	300	68.0
12		3	419	499	570	87.5
13		3	1412	3165	3065	103.3
14		3	91	126	124	101.6
15		3	868	969	1180	82.1
16		3	207	240	237	101.3
17	2	4	6330	6715	6850	98.0
18		4	595	626	620	101.0
19		4	156	168	210	80.0
20		4	3295	3515	3395	103.5
21		4	162	183	204	89.9
22		4	95	99	99	100.0
23		4	1099	1287	1660	77.5
24		4	3455	3435	3235	106.2

[a] ASV-UFE indicates ASV measurement of the unfiltered extract.
[b] ASV-FE and ICP-FE indicate ASV and ICP measurements, respectively, of the filtered extract.
[c] Lead recovery (%) = [(ASV-FE/ICP-FE) x 100].

Table A6. Results of lead analyses of Operator 3 field specimens

| Specimen No. | Operator ID | Wipe No. | Lead Content of UE Extract | | | Lead Recovery[c] |
			ASV–UFE[a] µg	ASV–FE[b] µg	ICP–FE[b] µg	%
1	3	2	141	156	168	92.9
2		2	2985	3365	2775	121.3
3		2	495	525	580	90.5
4		2	309	343	625	54.9
5		2	47	55	55	100.9
6		2	36	42	50	84.8
7		2	8150	7575	8200	92.4
8		2	796	924	1135	81.4
9	3	3	183	208	194	107.5
10		3	168	194	210	92.4
11		3	4060	3720	3610	103.0
12		3	928	1124	1235	91.0
13		3	394	447	585	76.4
14		3	725	796	945	84.2
15		3	5240	5090	5850	87.0
16		3	515	638	650	98.2
17	3	4	1311	1394	1505	92.6
18		4	1403	2535	1925	131.7
19		4	752	833	1700	49.0
20		4	210	231	265	87.2
21		4	3155	2890	2605	110.9
22		4	1365	2390	1850	129.2
23		4	168	202	210	96.4
24		4	149	191	231	82.9

[a] ASV-UFE indicates ASV measurement of the unfiltered extract.
[b] ASV-FE and ICP-FE indicate ASV and ICP measurements, respectively, of the filtered extract.
[c] Lead recovery (%) = [(ASV-FE/ICP-FE) x 100].

Table A7. Results of lead analyses of Operator 4 field specimens

| Specimen No. | Operator ID | Wipe No. | Lead Content of UE Extract | | | Lead Recovery[c] |
			ASV–UFE[a] μg	ASV–FE[b] μg	ICP–FE[b] μg	%
1	4	2	68	74	88	84.1
2		2	107	112	122	92.2
3		2	74	97	133	72.9
4		2	53	63	71	89.4
5		2	32	32	33	98.5
6		2	89	91	92	98.9
7		2	221	294	310	94.8
8		2	307	343	378	90.7
9	4	3	949	1131	1140	99.2
10		3	3505	3720	3820	97.4
11		3	42	51	54	94.4
12		3	59	68	97	70.5
13		3	63	59	69	85.5
14		3	390	461	875	52.7
15		3	468	692	755	91.7
16		3	47	49	50	99.0
17	4	4	2720	3970	3500	113.4
18		4	2430	1795	1755	102.3
19		4	34	36	35	102.9
20		4	569	574	565	101.6
21		4	91	89	91	98.3
22		4	99	107	114	94.3
23		4	116	131	144	91.0
24		4	1029	1069	1030	103.8

[a] ASV-UFE indicates ASV measurement of the unfiltered extract.
[b] ASV-FE and ICP-FE indicate ASV and ICP measurements, respectively, of the filtered extract.
[c] Lead recovery (%) = [(ASV-FE/ICP-FE) x 100].

APPENDIX B. REGRESSION PLOTS COMPARING ASV AND ICP FIELD SPECIMEN
              RESULTS BY OPERATOR

This Appendix provides linear regression plots (Figure B1) comparing the ASV and ICP field
specimen results by operator. Although the effect of operator on the UE/ASV lead analyses of the
field specimens was not statistically significant (Section 3.2.2), some readers may have interest in
observing the individual operator data because an examination of operator effect was a main
objective of the study.

In Figure B1, the plots in the left column show all data for each operator with the exception of the
17 000 µg data point determined by Operator 2 (Table A5); this data point was included in the
regression analyses. The right plots in the figure present the less-than-500 µg data subset for each
operator. In each plot, the plot character denotes the wipe number. The dashed line is that from the
linear regression analyses. The 45° solid line represents perfect agreement between the two
measurement methods. Table B1 gives the coefficients for these linear regression analyses.

Table B1. Linear regression analysis coefficients for the ASV versus ICP comparisons by
           operator

Operator	Data Set Analyzed[a]	Coefficients				r^2-value	Significant[c]	
		Slope[b]		Intercept[b]			Slope	Intercept
NIST	All Data	0.930	(0.009)	-4.212	(8.926)	0.997	Yes	No
	< 500 µg	0.885	(0.030)	4.970	(6.404)	0.974	Yes	No
2	All Data	1.033	(0.009)	-88.534	(37.033)	0.998	Yes	Yes
	< 500 µg	0.773	(0.173)	19.510	(35.670)	0.688	No	No
3	All Data	0.943	(0.037)	34.939	(91.033)	0.968	No	No
	< 500 µg	0.885	(0.077)	7.161	(14.439)	0.957	No	No
4	All Data	1.043	(0.026)	-36.316	(31.145)	0.987	No	No
	< 500 µg	0.919	(0.026)	-1.806	(3.901)	0.989	Yes	No

[a] For each operator's data set, regression analyses were performed twice: (1) using all points in the data set, and (2)
   using a data subset containing only those points for which the amount of lead determined by ICP analysis was
   < 500 µg.
[b] The value in parentheses is the standard error.
[c] These two columns indicate whether the slope and the intercept were significantly different from 1 and 0,
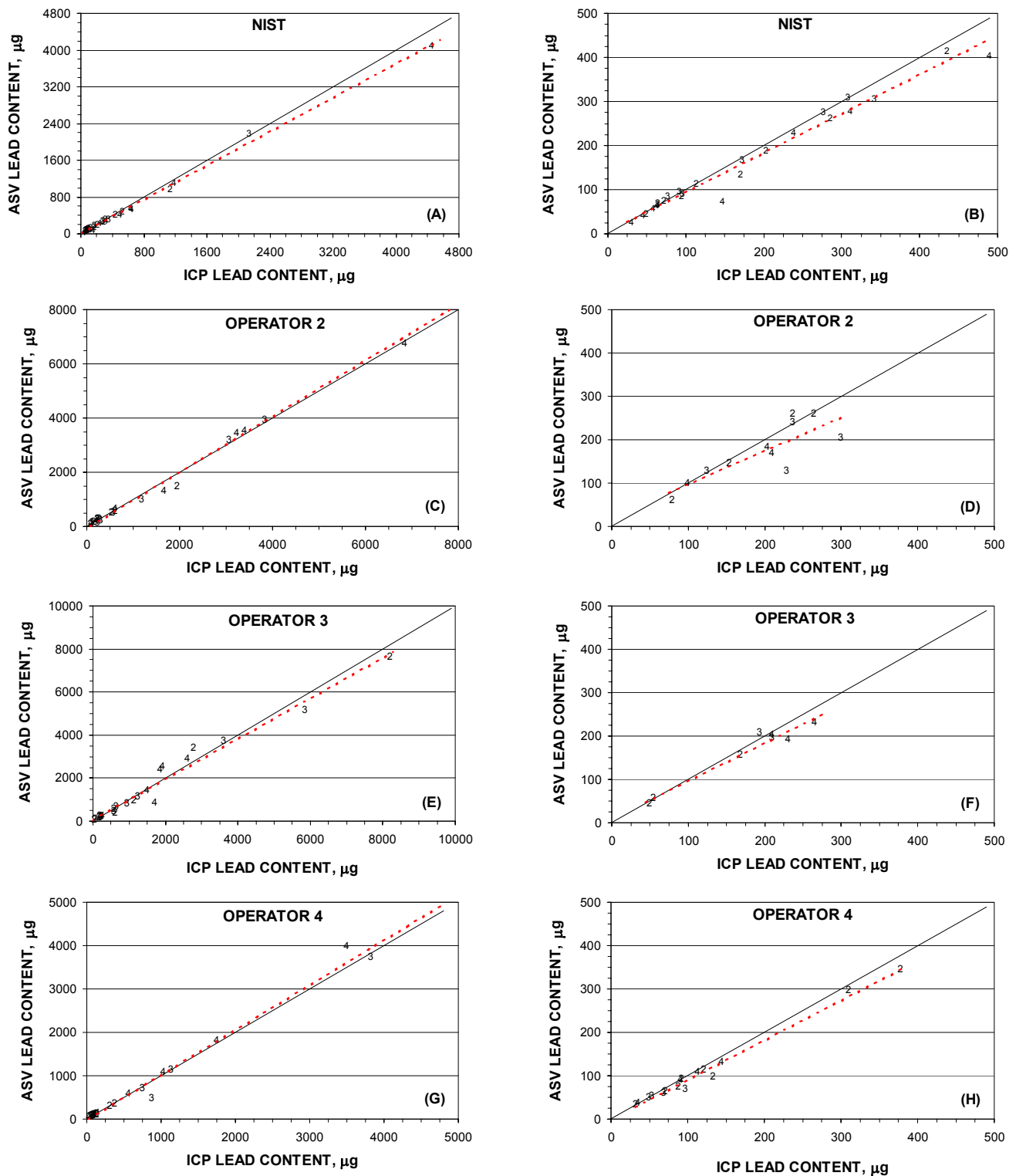   respectively.

Figure B1. Field Specimens: ASV Results Versus ICP Results by Operator. The plots in the left
column show all data for each operator, except one point with a lead content of
≈ 17 000 μg (Operator 2). The plots in the right column give data for specimens found
by ICP analyses to have less than 500 μg of lead. In each plot, the plot character
represents the wipe number. The dashed line is that from the linear regression analysis.
The 45° solid line represents perfect agreement between the two measurements.

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