REVISED FINAL REPORT

TASK ORDER 1026

ROUND ROBIN STUDY OF *IN-VITRO* BIOACCESSIBILITY ASSAY (IVBA) FOR LEAD IN SOIL AND EPA METHOD 3051A FOR LEAD AND ARSENIC: FLAT CREEK SOIL REFERENCE MATERIAL

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Statistical Analysis of Round Robin Results

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Note: This document is the Revised Final Report for the “Round Robin Study of *In-Vitro* Bioaccessibility Assay (IVBA) for Lead in Soil and EPA Method 3051a for Lead and Arsenic: Flat Creek Soil Reference Material”. This revised report was prepared under Task Order 1026 of the EPA Quality Assurance Technical Support Contract Number EP-W-16-016. This Study was conducted in 2012, and the original final report was submitted to EPA on November 14, 2012. The Study was conducted under Task Order 1026 of the EPA Quality Assurance Technical Support Contract Number EP-W-10-033, managed by Shaw Environmental, Inc. In 2013, Shaw Environmental Inc. was acquired by CB&I Federal Services LLC. All references to “Shaw” or “Shaw Environmental, Inc.” in this revised final report should be considered to be the same as “CB&I Federal Services LLC”. All electronic mail addresses in the report with the domain @shawgrp.com are now @cbifederalservices.com.

Subsequent to the submission of the original report, Syracuse Research Corporation (SRC), under EPA Contract Number EP-W-12-003, conducted an independent statistical analysis of the Study results. Under EPA Contract EP-W-16-016, Task Order 1026, Task 1, CB&I Federal Services LLC has been directed to prepare this revised final report of the 2012 Study to include the independent statistical analysis of the Study results performed by SRC. The results of the SRC independent statistical analysis, in narrative, tabular, and graphic format, have been included in this revised report on Pages 12 through 16 in Section V.E., with the beginning and ending of the SRC independent analysis clearly defined.

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ACRONYMS AND ABBREVIATIONS

ANOVA	Analysis of Variance
dF	Degrees of Freedom
F	Value Calculated
F-C rit	Critical Value
FCRM	Flat Creek Soil Reference Material
HSD	Honestly Significant Difference
ICP-MS	Inductively Coupled Plasma - Mass Spectrometry
ICP-AES	Inductively Coupled Plasma - Atomic Emission Spectrometry
IDP	Initial Demonstration of Proficiency
IVBA	In-Vitro Bioaccessibility Assay
MS	Mean Square
NERL	National Exposure Research Laboratory
NIST	National Institute of Standards and Technology
P-Value	Probability Value
RM	Reference Material
RSD	Relative Standard Deviation
RTP	Research Triangle Park
SAS\STAT	Statistical Analysis Software
SD	Standard Deviation
SEM	Standard Error of the Mean
Shaw	Shaw Environmental, Inc.
Sm	Standard Deviation of the Mean
SOP	Standard Operating Procedure
SOW	Statement of Work
SRC	Syracuse Research Corporation, Inc.
SRM	Standard Reference Material
SS	Sum of Squares
TRW	Technical Review Workgroup
USEPA	United States Environmental Protection Agency
USGS	United States Geological Survey
QATS	Quality Assurance Technical Support Program
QC	Quality Control

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ROUND ROBIN STUDY OF *IN-VITRO* BIOACCESSIBILITY ASSAY (IVBA) FOR LEAD IN SOIL AND SOIL-LIKE MATERIALS AND EPA METHOD 3051A FOR LEAD AND ARSENIC: FLAT CREEK SOIL REFERENCE MATERIAL

I. SUMMARY

The Bioavailability Committee of the USEPA Technical Review Workgroup for Metals and Asbestos (http://epa.gov/superfund/bioavailability/trw.htm) conducted a Round Robin Study (herein referred to as Study) of the *In Vitro* Bioaccessibility Assay (IVBA) for Lead[1] and EPA Method 3051A for Lead and Arsenic on the Flat Creek Soil Reference Material (FCRM).

Objectives:

- To derive a mean consensus value for the Lead IVBA for the FCRM, using EPA SOP 9200.2-86
- To report the total amount of lead and arsenic in the FCRM as measured by EPA Method 3051A

This Study included the participation of eight (8) laboratories, each reporting five (5) replicate analysis results for the FCRM Lead IVBA, as well as the Lead and Arsenic EPA Method 3051A digestion, with results totaling three (3) data sets of forty (40) results each. The EPA "Standard Operating Procedure for an *In Vitro* Bioaccessibility Assay" (EPA 9200.2-86) and EPA Method 3051A were provided for the participating laboratories as well as the Scope of Work to be performed. The results were statistically evaluated for IVBA Lead, and total Lead and Arsenic to derive the final consensus values provided in Table 1. No outlying sample results were identified using the Grubb's test either within each laboratory (n=5), or collectively (n=40) for the entire data set.

The associated quality control (QC) sample results provided by the laboratories for the reagent blank, bottle blank, spiked blank, and matrix spike were all within the control limits presented in the standard operating procedure (SOP) EPA 9200.2-86, with the exception of the Lead IVBA control soil. Although there were no outlier sample results identified using the Grubb's test, two (2) of the eight (8) laboratory results for the control soil NIST SRM 2710a exceeded the control limits specified in the EPA SOP 9200.2-86. A statistical comparison (t-test) was performed for the data set for one of the laboratories that had a control soil Lead IVBA result that was outside the control limits specified in EPA SOP 9200.2-86 and could be excluded. The Lead IVBA results for this one (1) laboratory were excluded and the statistical analysis was repeated for the Lead IVBA data set. Similarly, the EPA Method 3051A lead results from another laboratory were statistically evaluated using the t-test, which indicated that these EPA Method 3051A lead results could also be excluded. The revised statistical calculations resulted in lower standard deviation values for the individual and combined Lead IVBA and EPA Method 3051A lead results from the remaining laboratories, thus resulting in narrower Lead IVBA and EPA Method 3051A lead concentration 99-percentile prediction intervals for the new FCRM compared to using the total data set.

[1] This method has been incorporated into the SW846 Compendium as Method 1340: https://www.epa.gov/hw-sw846/sw-846-test-method-1340-vitro-bioaccessibility-assay-lead-soil.

Table 1: FCRM Summary of Results (Rounded Values)

FCRM	Low 99% PI	Mean	High 99% PI	RSD
Lead Method 3051A (mg/Kg)	5490	6440	7400	5.4%
Arsenic Method 3051A (mg/Kg)	550	730	910	8.9%
Lead IVBA Extracted (mg/Kg)	3990	4620	5250	4.9%

II. INTRODUCTION

Utilization of IVBA assays as an estimator of the bioaccessibility and bioavailability of lead in soil has been studied and recognized by the bioavailability scientific community. A comparison of the *in vivo* and *in vitro* assays for lead was conducted in 2007 and the results exhibited a high correlation between the two assays. (2007, EPA OSWER 9285.7-77). The IVBA assay is a viable and less cost prohibitive alternative to an *in vivo* assay (e.g., juvenile swine).

This report provides the Study results for the analysis of the FCRM. The objective of this Study is twofold: (1) derive a mean consensus value for the Lead IVBA for the FCRM, using EPA SOP 9200.2-86, and (2) report the total amount of lead and arsenic in the FCRM as measured by EPA Method 3051A. This report provides the data and statistical analysis of the lead and arsenic results from the Study conducted by the United States Environmental Protection Agency (USEPA), which validates its use as an additional soil reference material for EPA SOP 9200.2-86 and EPA Method 3051A. The FCRM was developed by the United States Geological Survey (USGS) from soil containing high concentrations of metals due to mining activity near an abandoned lead mine in Montana.

The Bioavailability Committee of the USEPA Technical Review Workgroup for Metals and Asbestos initiated the task of verification of the Lead IVBA values for the new FCRM in July, 2011. This Study was coordinated, evaluated, and reported by the USEPA Quality Assurance Technical Support (QATS) Program. The QATS Program was tasked to provide support that included a Study design, the development of the Study instructions in the form of a Statement of Work (SOW), reference material (RM) bottling and shipping, laboratory coordination, statistical analysis of results, and report preparation. Each of the eight (8) laboratories participating in the Study was requested to analyze each of the reference materials in five (5) replicate analyses, along with the EPA SOP 9200.2-86 required QC samples, including blank, matrix spike, and control soil.

III. BACKGROUND

The utilization of IVBA methods as an estimator of the bioavailability of lead in soil matrices has been studied and adopted by the bioavailability community. The IVBA technique is utilized because it is a less expensive method for the estimation of the bioavailability of lead in soil for humans than the previous method of choice, which involved juvenile swine assays. A comparison of the *in vivo* and *in vitro* methods is presented in USEPA OSWER 9285.7-77 (USEPA, 2007). This Study employed two (2) methods: EPA Method 3051A for the determination of total lead and total arsenic, and EPA SOP 9200.2-86 for Lead IVBA determination. These methods are summarized below.

III.A. EPA Method 3051A

EPA Method 3051A was used for the determination of total lead and total arsenic concentrations in the Study samples. Using EPA Method 3051A, solid samples are digested in concentrated nitric acid and concentrated hydrochloric acid using microwave heating with a suitable laboratory

microwave unit. In this Study, 0.5 ± 0.001 grams of sample, 9 ± 0.1 mL of concentrated nitric acid, and 3 ± 0.1 mL of concentrated hydrochloric acid were added to a fluorocarbon polymer microwave vessel. The vessel was then sealed and heated in the microwave unit with power setting(s) that cause the mixture within the vessels to rise to a temperature of 175º C ± 5º C in approximately 5.5 ± 0.25 minutes, and remain at 175º C ± 5º C for 4.5 minutes, or for the remainder of the 10 minute digestion period. After cooling, the vessel contents are either filtered, centrifuged, or allowed to settle, and then diluted to a suitable volume and analyzed using either EPA SW-846 Method 6010C (ICP-AES) or EPA SW-846 Method 6020A (ICP-MS).

III.B. EPA SOP 9200.2-86

EPA SOP 9200.2-86 was the method used for the determination of Lead IVBA results for the Study samples. Throughout this report, the term "Lead IVBA" is used synonymously with EPA SOP 9200.2-86, unless the SOP is specifically referenced. The IVBA method is performed by first retrieving the soil to be assessed for *in vitro* bioaccessibility assay, drying the soil at less than 40º C, and passing the dried material through a sieve to obtain the soil particles that are less than 250 μm[2]. One (1) gram of the soil is placed in a plastic bottle, and 100 mL of 0.4 M glycine, at a pH of 1.5, is added. The sample bottle(s), and associated quality control sample bottles, are then placed on a rotary extractor (30 ± 2 RPM) for one (1) hour while being heated at a constant temperature of 37ºC ± 2ºC. The heating of the bottles and rotary extraction apparatus is accomplished by immersion in a temperature controlled water bath (aquarium style), or alternatively, the apparatus can be heated by the flow of temperature controlled air (incubator style). After the prescribed extraction period, the bottles are removed from the extraction apparatus and the supernatant removed using an in-line filter and a 20 mL syringe. The filtered supernatant is then analyzed for lead (or other analytes) by ICP-AES or ICP-MS using the analytical methods cited above.

The Lead IVBA value for the FCRM is expressed as the ratio of the Lead IVBA result divided by EPA Method 3051A lead result, multiplied by 100.

$$IVBA\ (\%) = \frac{EPA\ SOP\ 9200.2-86\ Result}{Method\ 3051A\ Result} \times 100$$

EPA Method 3051A and EPA SOP 9200.2-86 can be accessed using the following USEPA website hyperlinks:

https://www.epa.gov/hw-sw846/sw-846-test-method-3051a-microwave-assisted-acid-digestion-sediments-sludges-soils-and-oils

https://nepis.epa.gov/Exe/ZyPDF.cgi/P100GESL.PDF?Dockey=P100GESL.PDF

IV. TECHNICAL APPROACH

Shaw's QATS Program support included the following subtasks:

- Contacting candidate laboratories with previous IVBA experience;
- Requesting laboratories to complete an Initial Demonstration of Proficiency (IDP) form, if they had not done so in a previous Lead IVBA Round Robin Study;
- Bioavailability Committee of the USEPA Technical Review Workgroup for Metals and Asbestos review of the completed IDP forms and selection of laboratories to participate

[2] After this round robin was completed, the recommended sieve size for this method was revised to <150 μm.

in the Study;

- Study Design;
- Development of a Statement of Work (SOW), including IVBA data reporting forms;
- Shipment of the IVBA samples and associated QC samples; and
- Statistical analysis of the Study results and report preparation.

IV.A. Contacting Laboratories, IDP Form, and Participating Laboratory Selection

To identify qualified candidates to participate in the Study, Shaw first contacted laboratories with previous IVBA experience. Most of the laboratories in this Study were participants in a previous Lead IVBA Round Robin Study conducted by the Bioavailability Committee of the USEPA Technical Review Workgroup for Metals and Asbestos and coordinated by Shaw, which was completed in 2011. The laboratories were asked to complete an IDP form to determine their level of experience with the IVBA procedures. The information requested on the IDP form included the total number of IVBA analyses performed by the laboratory, as well as the QC sample results for the most recent ten (10) batches of IVBA analyses conducted at their facility. Only those laboratories that had not been participants in the previous Study were asked to complete the IDP form. From previous participation and the IDP form response, the Bioavailability Committee of the USEPA Technical Review Workgroup for Metals and Asbestos selected a total of eight (8) laboratories for participation in the Study, which are presented in Table 2. In order to maintain the anonymity of the Study participants, the IDP forms provided by the laboratories are presented in Appendix F in redacted format, with an alphanumeric letter used as an identifier, in an order inconsistent with the order presented in Table 2. The IDP forms, without redaction, are available from USEPA HQ Co-Chair for the Technical Review Workgroup (TRW), http://epa.gov/superfund/bioavailability/trw.htm.

Table 2. Laboratories Selected to Participate in the Study

Laboratory		Address	Contact Name and e-mail Address
1	ACZ Laboratories	2773 Downhill Drive Steamboat Springs, CO 80487	Mr. Tim VanWyngarden (timv@acz.com) Ms. Sue Webber (suew@acz.com)
2	USEPA National Exposure Research Laboratory (NERL) Research Triangle Park (RTP)	109 T.W. Alexander Dr. Research Triangle Park, NC 27709	Dr. Karen Bradham (bradham.karen@epa.gov)
3	Ohio State University	School of Environment and Natural Resources 2021 Coffey Rd. 410 C Kottman Hall Columbus, OH 43210-1043	Dr. Nicholas Basta (basta.4@osu.edu)
4	PRIMA Environmental	5070 Robert J Mathews Pkwy, Suite 300 El Dorado Hills, CA 95762	Dr. Cindy Schreier (cschreier@primaenvironmental.com)
5	USEPA Region 7 Laboratory	300 Minnesota Avenue Kansas City, KS 66101	Michael Davis (davis.michael@epa.gov)

6	USEPA Region 9 Laboratory	1337 South 46th Street, Bldg 201 Richmond, CA 94804	Richard Bauer (bauer.richard@epa.gov)
7	Royal Roads University (Canada)	Royal Roads University 2005 Sooke Road Victoria, BC, Canada V9B 5Y2	Dr. Matt Dodd (Matt.Dodd@RoyalRoads.ca)
8	University of Colorado	Benson Earth Science 2200 Colorado Avenue Boulder, CO 80309	Dr. John Drexler (Drexlerj@Colorado.edu)

IV.B. Study Design

IV.B.1. FCRM and Number of Replicates

The FCRM used in this Study was sent to the QATS Laboratory for sub-aliquoting and shipment on February 24, 2012 by USEPA National Exposure Research Laboratory (NERL) personnel, who had previously received the material from the USGS Associate Project Chief. The FCRM was provided in a 500 mL glass bottle, and sufficient FCRM material was mixed before sub-aliquots were bottled for Study sample analysis. The standard reference material (SRM) NIST SRM 2710a used as a control soil in this Study was provided by the NIST Analytical Chemistry Division from a previous Study conducted at the QATS Laboratory in 2009. The QATS Laboratory was provided with 50 grams of NIST SRM 2710a, and a sufficient amount of the material for the Study was combined and mixed before sub-aliquots were bottled for distribution to the laboratories.

The moisture content of the FCRM was <0.5%, and was determined by heating a 5 gram sample in an oven at 105º C for twelve (12) hours. The NIST SRM 2710a moisture content is approximately 2%, and the particle size is <74 µm, as reported on the NIST SRM 2710a Certificate of Analysis.

The Certificate of Analysis for the NIST SRM 2710a is presented in Appendix F. Table 3 provides the lead and arsenic concentration, particle size, and moisture content for this NIST SRM 2710a, derived from the Certificates of Analysis.

Table 3. NIST SRM 2710a Certificate of Analysis Parameters

Element	Total Concentration (mg/Kg)	Leachable Concentration (mg/Kg)	Particle Size	Moisture Content
Pb	5520	5100	<74 µm	~2%
As	1540	1400		

The Bioavailability Committee of the USEPA Technical Review Workgroup for Metals and Asbestos determined that five (5) replicate analyses of the FCRM would be conducted by each laboratory participating in the Study. Five (5) replicate analyses were chosen to ensure that a sufficient number of results were available for establishing a statistically sound Lead IVBA mean value and control limits for the new FCRM.

IV.B.2. QC Samples

In this Study, the laboratories were instructed to analyze the samples in strict accordance with the EPA SOP 9200.2-86 including all of the associated quality control samples, with noted exceptions. Table 4 below provides the EPA SOP 9200.2-86 required QC samples and associated control limits used in this Study.

Table 4. EPA SOP 9200.2-86 Required QC Samples and Control Limits

QC Sample	Control Limits
Reagent Blank	<25 µg/L Lead
Bottle Blank	<50 µg/L Lead
Blank Spike (10 mg/L)	85% -115% Recovery
Matrix Spike (10 mg/L)	75% -125% Recovery
Duplicate Sample	± 20% RPD
Control Soil (NIST SRM 2710a)	IVBA Mean = 67.5% Acceptable Range 60.7% - 74.2%

NIST SRM 2710a was used as the control soil for both the Lead IVBA and EPA Method 3051A portions of the Study, followed by analysis. Both the lead and arsenic mean values and range appear in the Addendum to the NIST SRM 2710a Certificate of Analysis titled "Leachable Concentrations Determined Using EPA Methods 200.7 and 3050B." Five (5) replicate aliquots of the FCRM were subjected to the Lead IVBA procedure; therefore, there was no additional duplicate sample analysis requirement in this Study as a measure of analytical precision.

The laboratories were instructed to perform the analysis of one set of QC samples with each RM batch for both the Lead IVBA and the EPA Method 3051A methods.

IV.C. Statement of Work for the Study

An SOW was developed by QATS personnel and the Bioavailability Committee of the USEPA Technical Review Workgroup for Metals and Asbestos which provided instructions to the participating laboratories on the analysis and reporting of the Study samples. The SOW provided a list of samples for each Lead IVBA batch and a recommended sequence of instrumental analysis of the Lead IVBA samples. The SOW also provided a list of the required associated QC sample analysis and QC sample control limits derived from the EPA SOP 9200.2-86.

IV.D. Shipment of the Study Samples and Associated QC Samples

The Study samples were shipped to the eight (8) participating laboratories in April, 2012. The laboratories were provided a 30 day turnaround time for submitting the sample results. The Study sample shipments also included hardcopies and CDs of the SOW and the EPA SOP 9200.2-86.

IV.E. Statistical Analysis of the Study Results

Conventional statistical analysis techniques were used to analyze the data collected from the Study. The statistical analyses were performed in Microsoft® Excel, using Analysis of Variance (ANOVA) and t-test data analysis tools provided by the Excel Analysis Tool Pac add-in package.

The statistical tool ANOVA, single factor (e.g., lead), was used to discern the intralaboratory versus the interlaboratory sources of variance of each FCRM data set derived from the Study. The statistical t-test was used to analyze the data from the different laboratories to determine, for example, if one set of data is statistically different than the others. Specifically, the t-test employed was the two (2) sample, assuming equal variances t-test.

The QC samples, including the reagent blank, bottle bank, spiked blank, matrix spike, and NIST SRM 2710a, were also processed with the Lead IVBA / EPA Method 3051A digested FCRM samples. The results were evaluated to determine if there were any anomalous data submitted by a participating laboratory that should be excluded from the composite results in the course of setting the FCRM statistical values and control limits.

V. RESULTS AND DISCUSSION

V.A. Initial Demonstration of Proficiency

The IDP forms provided by the laboratories selected for the Study are presented in Appendix G. As discussed in a previous section, these forms have been redacted to preserve anonymity. The original unredacted forms are available from the USEPA HQ Co-Chair for the TRW. Out of the ten (10) candidate laboratories submitting IDP forms, eight (8) laboratories were selected to be participants in the Study.

V.B. Study Results

Each of the eight (8) laboratories participating in the Study analyzed the FCRM using five (5) replicate aliquots, providing a total of 40 results for the Lead IVBA procedure, and 40 lead and arsenic results for the EPA Method 3051A procedure. The SOW provided to the laboratories contained several tables that allowed laboratory reporting of the Study sample analysis results using Microsoft® Word. The participating laboratories were asked to submit the results to the QATS Laboratory via electronic mail, and provide hard copies of the results that could not be converted to electronic files. The results provided by the laboratories in the SOW tables are presented in Appendix H in redacted form. The original unredacted SOW forms completed by the laboratories are available from the USEPA HQ Co-Chair for the TRW.

V.C. FCRM Results and Statistical Analysis

V.C.1. Lead Results, EPA Method 3051A

Results of EPA Method 3051A for lead for the FCRM are presented in Appendix A. Table A-1 presents the EPA Method 3051A lead results for the FCRM. The mean lead result from all eight (8) laboratories (n=40) is 6,634 mg/Kg, with a pooled RSD value of 9.1%. The calculated lead 99 percentile prediction interval, based on the EPA Method 3051A results (n=40) alone, is ± 25.0%. As shown in Table A-1, the calculated percent standard deviation of the mean is 1.4%, and the calculated 99 percentile confidence interval of the mean is ± 3.9%. Note that the Laboratory D EPA Method 3051A mean lead result of 7,963 mg/Kg was higher than the results from the other laboratories, and 20% higher than the EPA Method 3051A mean result of 6,634 mg/Kg.

The formulas used for the prediction interval and confidence interval of the mean are provided below in Exhibit 1.

Exhibit 1:

Prediction Interval: $\bar{x} \pm \left(sd * t \left(\sqrt{1 + 1/n} \right) \right)$

Confidence Interval: $\bar{x} \pm (sm * t) \quad where\ sm = sd / \sqrt{n}$

Where:

sd = standard deviation (n-1)
t = Student's t; for n = 40, df=39, t = 2.708, for 99 percentile
sm = standard deviation of the mean

The mean Pb IVBA value is the mean Pb extraction result / the mean Pb EPA Method 3051A digestion result * 100.

The pooled standard deviation resulting from division is based on the square root of the sum of the squares formula for two independent variables with unequal means formula. Please note that the sd are normalized to percentiles before squaring.

$$sd\ Pb\ IVBAratio = \left(Pb\ IVBAratio * \left(\sqrt{((sd/mean Extraction)^{\wedge 2} + (sd/mean Digestion)^{\wedge 2}} \right) \right)$$

Note: The square root of the sum of variances squared method was used as an estimator of the combined variance for the final lead IVBA result, as the expected means and variances of the IVBA extraction and digestion results are not expected to be equal. The IVBA extraction and the digestion results are not subsets of the same population, and therefore their respective variances are additive, even during the division operation.

Table A-2 presents the EPA Method 3051A lead results for the associated QC samples that were determined with the FCRM. These results include the blank spike recovery, matrix spike recovery, and the NIST SRM 2710a results and percent recovery. All results are within the control limits presented in the EPA SOP 9200.2-86 and in Table 4 above, with the exception of the EPA Method 3051A NIST SRM 2710a lead result of 4,537 mg/Kg for Laboratory E, which is slightly below the lower limit of 4,700 mg/Kg. Although the Laboratory E, EPA Method 3051A NIST SRM 2710a lead result is slightly below the lower control limit for lead based on the Addendum to the NIST SRM 2710a Certificate of Analysis, this did not translate into lower results for the FCRM when compared to the other laboratory results. The EPA Method 3051A NIST SRM 2710a mean lead result for all eight (8) laboratories is 100.8% of the value presented in the Addendum to the NIST SRM 2710a Certificate of Analysis, which indicates good overall accuracy. The Laboratory D NIST SRM 2710a matrix spike recovery of 57% is outside the 75% to 125% matrix spike recovery range; however, because the spiking ratio was less than 1:4 spike to sample concentration, this spike result is not a reliable predictor of accuracy.

Table A-3 presents the ANOVA for the FCRM EPA Method 3051A lead results. For each set of laboratory data, Table A-3 presents the number of sample replicates (n), as well as the sum, mean, and variance (square of the data set standard deviation) values. The table also provides the various statistical calculation values that are used by the ANOVA algorithm to test the variance of all of the results for both within a laboratory, and between laboratories. These calculation results include: sum of squares (SS), degrees of freedom (df), mean square (MS), value calculated (F), critical value of (F-Crit), and probability value (P-value).

The results of the ANOVA assessment which are presented in Table A-3 indicate that the intralaboratory variance is low compared to interlaboratory variance. This is reflected by the large MS value for the interlaboratory group results (1,807,266) compared to the lower intralaboratory group results MS value (49,325). The variance uses the null hypothesis that the data sets provided by the laboratories represent the same samples, analyzed by the same method. The ANOVA

assessment allows the user to select the probability of error of falsely rejecting the hypothesis that all results are from the same population (same samples and method). The error significance level is typically set at 95%, which translates to a 5% chance of wrongly rejecting the hypothesis. The data comparison performed by the algorithm is referred to as a two-tail test, which means that both the upper and the lower ends of data distribution are tested. The ANOVA algorithm calculates (or selects from an algorithm table) the f-critical value, based on the assumption of normal distributions of the intralaboratory results and the composite results. If the calculated f-value, which is based on the ratio of variances displayed by the between laboratory results to the variance of individual laboratory results, is greater than the f-critical value, then the null hypothesis is rejected, which is the case for the lead extraction data sets. The ANOVA results presented in Table A-3 indicate that the variance in interlaboratory data is large relative to the intralaboratory data variances; therefore the null hypothesis is rejected with a high degree of confidence (low P-value). The rejection of the null hypothesis could indicate: 1) different methods were used in the analysis, 2) different samples were being analyzed, or 3) the intralaboratory variance is small compared to what might be expected. The latter choice must be accepted as correct, considering the RSDs for the FCRM for the intralaboratory (n=5) results all quite low (less than 8% RSD for three (3) data sets and less than 3% RSD for the remaining five (5) sets of results).

Appendix B provides the statistical t-test comparison of the Laboratory D EPA Method 3051A lead results. Table B-1 presents the t-test statistical comparison for the FCRM EPA Method 3051A lead results from Laboratory D and the results from the other seven (7) laboratories. This t-test was performed because the EPA Method 3051A lead results from Laboratory D as shown in Table A-1 were higher than the other laboratory results.

The t-test was employed to evaluate if there was a statistical difference between the results from Laboratory D versus the other reported results. The t-test function in Microsoft® Excel was used, which is the 2-sample (assuming equal variances, alpha 0.01, 99-percentile) t-test. The t-test results presented in Table B-1 shows there is a significant difference between the data from Laboratory D compared to the results derived from the other laboratories collectively, as indicated by a P ($T \leq t$) value that is less than 0.01 for the t-tests performed on the data set. A t-Stat value that is greater that the t-critical value also indicates a significant difference between the Laboratory D data and the remaining data sets. The t-test comparison results, which are presented in Table B-1, indicate that the extraction results for Laboratory D can reasonably be excluded with a less than 1% chance of being incorrect. The EPA Method 3051A lead results for this laboratory were omitted and the statistical analysis was repeated for the EPA Method 3051A data set.

Table B-2 presents the revised statistical analysis of the EPA Method 3051A lead results for the FCRM, excluding the results from Laboratory D. The mean of the pooled EPA Method 3051A lead results (n=35) from the FCRM is 6,444 mg/Kg. The calculated pooled standard deviation of the FCRM EPA Method 3051A lead results is 345 mg/Kg, which provides a pooled RSD of 5.4%. The calculated lead 99 percentile prediction interval, based on the EPA Method 3051A lead results (n=35) alone, is ±14.8% of the mean value of 6,444 mg/Kg. The calculated percent standard deviation of the mean for the FCRM EPA Method 3051A lead results is 0.91%. The calculated 99 percentile confidence interval of the mean EPA Method 3051A lead results for the FCRM is 6,444 ± 2.5%.

V.C.2. Arsenic Results, Method 3051A

Appendix C presents the FCRM EPA Method 3051A arsenic results. Table C-1 presents the EPA Method 3051A arsenic results, prediction intervals, and confidence intervals for the FCRM. The mean arsenic result is 728 mg/Kg, with a standard deviation of 65 mg/Kg and RSD of 8.9%. The calculated arsenic 99 percentile prediction interval, based on the EPA Method 3051A arsenic results (n=40) alone, is 728 mg/Kg ± 24.2%. The calculated 99 percentile confidence interval of the EPA Method 3051A mean arsenic result for the FCRM is 728 mg/Kg ± 3.8%.

Table C-2 presents results for the associated arsenic QC samples that were also determined by EPA Method 3051A along with the FCRM. The table includes the blank spike recovery, matrix spike recovery, and the NIST SRM 2710a results and percent recovery. Using the NIST SRM 2710a Certificate of Analysis as a guideline, the NIST SRM 2710a arsenic results are within the range presented in the Certificate of Analysis, with the exception of the results from Laboratories B and F. The arsenic results from Laboratories B and F, at 1,650 mg/Kg and 1,684 mg/Kg, respectively, slightly exceed the 1,600 mg/Kg upper range for arsenic listed in the Certificate of Analysis. The NIST SRM 2710a mean arsenic result for all eight (8) laboratories is 1,592 mg/Kg. This value is 113.7% of the NIST SRM 2710a strong leach value of 1,400 mg/Kg presented in the Addendum to the NIST SRM 2710a Certificate of Analysis, and indicates a high bias in the recovery of arsenic from the EPA Method 3051A digestion relative to the CLP digestion procedures referenced in the Certificate of Analysis. In comparison, the CLP digestions are usually open beaker or block digestions and may result in incomplete digestion, or possible increased losses of arsenic acid vapor during digestion.

Table C-3 presents the ANOVA for the FCRM EPA Method 3051A arsenic results. As with the ANOVA of the FCRM EPA Method 3051A lead results, the ANOVA of the FCRM EPA Method 3051A arsenic results show the intralaboratory variance to be low compared to interlaboratory variance. This is indicated by the large MS value of 20,732 for the interlaboratory group results compared to the lower intralaboratory group results MS value of 585. The RSD values for the FCRM EPA Method 3051A intralaboratory results (n=5) are all 6% or less.

V.C.3. Lead IVBA Results (EPA SOP 9200.2-86)

Appendix D presents the FCRM Lead IVBA results. Table D-1 presents the Study Lead IVBA results for the FCRM along with the mean, standard deviation (n-1 weighting), and RSD values for each sample set. Please note that the values presented in these tables are not rounded. The pertinent rounded values are presented in the Conclusions and Recommendations section of this report. Table D-1 also presents the 99 percentile prediction interval for the Lead IVBA result, in mg/Kg. This 99 percentile prediction interval for the Lead IVBA result was converted to the Lead IVBA prediction interval by dividing the statistically combined Lead IVBA results with the statistically combined EPA Method 3051A lead results. Table D-1 also provides the confidence interval of the mean for the FCRM Lead IVBA value.

Table D-1 presents the mean concentration (n=40) of the FCRM Lead IVBA at 4,700 mg/Kg. This is 70.8% of the EPA Method 3051A mean lead concentration of 6,634 mg/Kg, which is presented in Table A-1, and represents a Lead IVBA value of 70.8%. The calculated pooled RSD value of the FCRM Lead IVBA results is 6.5%. The calculated lead 99 percentile prediction interval based on the Lead IVBA results alone (n=40) is ± 17.7%. The calculated lead 99 percentile predication interval for the Lead IVBA result, which includes the variance of the EPA Method 3051A results (n=80), is significantly higher at ± 29.7%. The calculated percent standard deviation of the mean for the FCRM is 1.24%. The calculated 99 percentile confidence interval of the Lead IVBA mean result for the FCRM is 70.8 ± 3.3%. The Laboratory B results in Table D-1 were observed to be higher than the results from the other laboratories.

Table D-2 presents the Lead IVBA results for the associated QC samples that were processed with the FCRM, as well as the EPA Method 3051A lead QC results. These include results for the reagent blank, bottle blank, blank spike, matrix spike, and the NIST SRM 2710a. All results are within the control limits presented in the EPA SOP 9200.2-86 and in Table 4 above, with the exception of the Lead IVBA NIST SRM 2710a results from laboratories B and C and the reagent blank result for laboratory C.

In Table D-2, row 1, Laboratory C reported a reagent blank result of <40 ug/L, which is greater

than the EPA SOP 9200.2-86 required detection limit of 25 ug/L. The laboratory was contacted after a review of the laboratory's reagent blank results, and confirmed the reported <40 ug/L blank result was correct. However, because the actual analytical sample results are approximately 100 times greater in concentration, this elevated blank result does not impact the Study results.

Row 6 of Table D-2 presents the NIST SRM 2710a Lead IVBA percent recovery based on the Lead IVBA mean recovery of 3,440 mg/Kg from a previous Study. All percent recovery results are within ± 20% of the mean value, with the Laboratory B recovery the highest at 116.3%.

Row 8 of Table D-2 presents the NIST SRM 2710a EPA Method 3051A percent recovery for lead based on the NIST SRM 2710a Certificate of Analysis mean result for the strong leach acid digestion (EPA Method 3050B) of 5,100 mg/Kg, with the acceptance range of (4,700 - 5,800 mg/Kg). All of the lead digestion result recoveries are 100 ± 20%; however, compared with the Certificate of Analysis acceptance range (4,700 – 5,800 mg/Kg), the EPA Method 3051A result for Laboratory E was slightly below the lower limit at 4,537 mg/Kg. However, because the FCRM Lead IVBA results for Laboratory E were not low biased, the results were retained.

Row 9 of Table D-2 presents the Lead IVBA values for the NIST SRM 2710a derived from both the Lead IVBA and EPA Method 3051A results from this Study. The results are within the previously established EPA SOP 9200.2-86 control limits of 60.7% to 74.2% with the exception of the results from Laboratories B and C, which exceeded the 99 percentile control limits at 78.4% and 77.3%, respectively. The FCRM Lead IVBA results from these two (2) laboratories seem to correlate with these high NIST SRM 2710a Lead IVBA results.

Table D-3 presents the statistical summary for the ANOVA for the FCRM Lead IVBA results. The results of the ANOVA assessment, which are presented in Table D-3, indicate that the intralaboratory variance is low compared to interlaboratory variance. This is indicated by the large MS value of 485,588 for the interlaboratory group compared to the substantially lower MS value of 6,469 for the intralaboratory group. The ANOVA results presented in Table D-3 indicate that the variance in interlaboratory data is large relative to the intralaboratory data variances; therefore the null hypothesis is rejected with a high degree of confidence (low P-value). The RSD values for the FCRM for the intralaboratory (n=5) results are all quite low (less than 4% for one (1) of the sets, and less than 2% for the remaining seven (7) sets of results).

Because of the higher than acceptable Lead IVBA results for the NIST SRM 2710a from Laboratories B and C presented in row 9 of Table D-2, a statistical comparison (t-test) was performed between the FCRM data set for Laboratory B (the highest results) and the remaining FCRM data. The t-test was employed to evaluate if there was a statistical difference between the results from Laboratory B versus the results from the remaining seven (7) laboratories. The Excel t-test output for this exercise is presented in Appendix E. The t-test results presented in Table E-1 shows there is a significant difference between the data from Laboratory B compared to the results derived from the other laboratories collectively, as indicated by a P ($T \leq t$) value that is less than 0.01 for the t-tests performed on the data set. The t-test comparison results, which are presented in Table E-1, indicate that the Lead IVBA results for Laboratory B can reasonably be excluded with a less than 1% chance of being incorrect. The Lead IVBA extraction results for this one (1) laboratory were omitted and the statistical analysis was repeated for the remaining Lead IVBA data set.

Table E-2 presents the revised statistical analysis for the FCRM Lead IVBA results, excluding the results from Laboratory B. The FCRM Lead IVBA mean value minus the results from Laboratory B (n=35) is 4,619 mg/Kg. The Lead IVBA value is 69.6% of the EPA Method 3051A mean lead value of 6,634 mg/Kg, presented in Table E-2, and represents a Lead IVBA value of 69.6. The calculated pooled RSD of the FCRM Lead IVBA results is 4.9%. The calculated lead 99 percentile prediction interval based on the Lead IVBA results (n=35) alone, is ± 13.6%. The calculated 99 percentile predication interval for the Lead IVBA value, which includes the variance of the EPA Method 3051A results (n=75), is ± 27.5%. The calculated percent standard deviation of the mean for the FCRM Lead IVBA results is 1.2%. The calculated 99 percentile confidence interval of the mean Lead IVBA result for the FCRM is 69.6 ± 3.2%. Overall, the statistical results for the FCRM, excluding the Laboratory B data set, exhibit slightly increased precision when compared to the full data set.

Table D-4 presents the resulting calculated 99 percentile prediction interval for the Lead IVBA, which includes the variance of the EPA Method 3051A results, but has excluded both the Laboratory B Lead IVBA results and Laboratory D EPA Method 3051A lead results. The Lead IVBA value (n=70) is 71.7%, the SD is 5.2, and the RSD is 7.3%. The calculated 99 percentile prediction interval for the Lead IVBA, which includes the variance for the EPA Method 3051A results (n=70) is 71.7% ± 19.4%. The calculated percent standard deviation of the mean for the FCRM Lead IVBA result is 0.87%. The calculated 99 percentile confidence interval of the Lead IVBA mean result for the FCRM is 71.7% ± 2.3%. Overall, the statistical results for the FCRM, excluding the Laboratory B Lead IVBA data set and the Laboratory D EPA Method 3051A lead data set, exhibit increased precision as compared to the full data set.

V.D. Summary of FCRM Study Results and Prediction Intervals

Table 5 provides the EPA Method 3051A lead and arsenic results, statistics, and 99 percentile prediction intervals for the FCRM. Table 6 provides the Lead IVBA results, statistics, and 99 percentile prediction intervals for the FCRM.

Table 5. FCRM EPA Method 3051A Results and Statistics

Analyte	Low 99% PI	Mean	High 99% PI	RSD	N
Pb	5490	6440	7400	5.4%	35
As	550	730	910	8.9%	40

Table 6. FCRM Lead IVBA Results and Statistics

Analyte	Mean Result Lead IVBA (%)	Lead IVBA (%) (± 99% PI)	RSD*	Mean Result Lead IVBA (mg/Kg)	Lead IVBA (mg/Kg) (± 99% PI)	RSD*	N
Pb	71.7	(57.8 – 85.6)	7.3%	4619	(3991 – 5247)	4.9%	35

* RSD was derived from the replicate Lead IVBA results.

V.E. Independent Statistical Analysis of the Round Robin Study Results by Syracuse Research Corporation (SRC) and Comparison to Shaw's Standard Statistical Analysis Results

SRC, Inc. under contract EP-W-12-003 analyzed the Round Robin Study data using Tukey's Studentized Range (also known as the Honestly Significant Difference [HSD] test). This test evaluates whether the data from each laboratory are significantly different from the others while controlling the type 1 error rate (at $\alpha = 0.05$) when multiple statistical comparisons are performed. When data from one or more laboratories were identified as different from the others, these datasets were further evaluated visually to determine if they should be excluded from the final dataset used to calculate prediction intervals (PIs) and confidence intervals (CIs). If the Tukey's HSD test or visual examination of the data did not identify any datasets that differed significantly from the others, or if the test indicated many differences among the datasets with no clear grouping, then all laboratory results were included in the final dataset.

The final dataset for each measurement endpoint included all laboratories that were not excluded for QC issues and were not identified as significantly different from the other laboratories by Tukey's HSD or visual inspection of the data. These data were used to calculate the PIs and CIs for the measurement endpoint. The PI for a Reference Material (RM) refers to a specific measurement and is used to determine if a laboratory result is acceptable, while the CI is an estimated range of values that is likely (with probability of α) to include the mean of a population. The formulas used for the prediction and CI are provided in Exhibit 2.

Exhibit 2.

99% Prediction Interval: $\bar{x} \pm \left(sd * t\left(\sqrt{1 + 1/n} \right) \right)$

99% Confidence Interval: $\bar{x} \pm (sem * t) \quad where\ sem = sd/\sqrt{n}$

Where:

SD = standard deviation
t = Student's critical t value for α =0.01 (two-tailed)
n = number of samples
SEM = standard error of the mean

The final datasets for lead by EPA method 3051A and by IVBA Method SOP 9200.2-86 were used to estimate the IVBA as a percentage of the total lead (i.e., IVBA by Method EPA SOP 9200.2-86 / lead by Method 3051A). Fieller confidence intervals for the ratio of bi-variate normal random variables were calculated (Fieller, 1954; Dilba et al., 2006; Tamhane and Logan, 2004) using the T-

Test procedure in SAS (Exhibit 3; SAS/STAT software, Version 9.3 of the SAS System for Windows).

Exhibit 3.

99% Confidence Interval (unequal variance): $-\frac{bu}{a_u} + \frac{\sqrt{(b_u^2 - b_u c_u)}}{a_u}, -\frac{bu}{a_u} - \frac{\sqrt{(b_u^2 - b_u c_u)}}{a_u}$

Where:

$a_u = \frac{s_p t^2_{1-\frac{\alpha}{2}df_u}}{n_2} - \bar{x}_2^2$

$b_u = \bar{x}_1 * \bar{x}_2$

$c_u = \frac{s_p t^2_{1-\frac{\alpha}{2}df_u}}{n_1} - \bar{x}_1^2$

$df_u = (\frac{s_1^2}{n_1} + \frac{s_2^2(\frac{\bar{x}_1}{\bar{x}_2})^2}{n_2})^2 / (\frac{s_1^4}{n_1^2(n_1-1)} + \frac{s_2^4(\frac{\bar{x}_1}{\bar{x}_2})^4}{n_2^2(n_2-1)})$ (Satterthwaite approximate degrees of freedom; Tamhane and Logan, 2004)

x_1 = average of IVBA lead (mg/kg)
x_2 = average of lead by 3051A
s_1 = standard deviation of IVBA lead (mg/kg)
s_2 = standard deviation of lead by 3051A

Prediction intervals account for the variability among individual measurements as well as the uncertainty in the estimate of mean. The prediction interval was estimated by extending the Fieller confidence limits to account for the estimated variability of the ratio (Exhibit 4).

Exhibit 4.

99% Prediction Interval (unequal variance): $LL - s_r * t_{1-\frac{\alpha}{2}df_u} UL + s_r * t_{1-\alpha.df_u}$

Where:

$LL, UL = lower\ limit, upper\ limit\ of\ the\ 99\%\ Fieller\ type\ CI (unequal\ variance)\ for\ the\ ratio\ (Exhibit\ 2)$

$s_r = \frac{\bar{x}}{\bar{y}} * \sqrt{(\frac{s_1}{\bar{x}_1})^2 + (\frac{s_2}{\bar{x}_2})^2}$ (first order Taylor series approximation; Stuart & Ord, 1986)

$df_u = (\frac{s_1^2}{n_1} + \frac{s_2^2(\frac{\bar{x}_1}{\bar{x}_2})^2}{n_2})^2 / (\frac{s_1^4}{n_1^2(n_1-1)} + \frac{s_2^4(\frac{\bar{x}_1}{\bar{x}_2})^4}{n_2^2(n_2-1)})$ (Welch-Satterthwaite degrees of freedom; Tamhane and Logan, 2004)

x_1 = average of IVBA (mg/kg)
x_2 = average of lead by 3051A
s_1 = standard deviation of IVBA (mg/kg)
s_2 = standard deviation of lead by 3051A

The SRC estimates based on the methods described above are presented in Table 7 below. The results of Pb 3051A and As 3051A are identical to what was determined using the standard statistical tests, compare to results in Tables B-2 and C-1, respectively. The SRC estimates of Pb IVBA (mg/kg) differ from the standard statistical results (compare Table 7 with Table E-2) because different laboratories were selected for inclusion in the estimate of Pb IVBA (mg/kg).

For the Pb 3051A data, SRC found the results of the ANOVA assessment indicated that data from at least one laboratory were significantly ($p < 0.05$) different from the other data. Analysis of the data using Tukey's HSD test showed that the data from Laboratory D were different from all of the remaining laboratories; this difference is also evident in Figure 1 (below). As data from Laboratory D were significantly different from all other laboratories, these data were omitted from the final dataset. Laboratory D was also eliminated from the estimate of Pb 3051A in the analysis using the standard statistical approach (see Table B-2).

For the As 3051A data, SRC found the results of the ANOVA assessment indicated a significant ($p < 0.05$) difference among the laboratories. Analysis of the data using Tukey's Studentized Range Test indicated many differences among the datasets with no clear majority grouping (see Figure 2 below). Therefore, all laboratories were included in the final dataset. All laboratories were also included in the estimate of As 3051A in the standard statistical analysis (see Table C-1).

For the Pb IVBA data, SRC removed the results from laboratories B and C because these laboratories exceeded the previously established EPA SOP 9200.2-86 control limits of 60.7% to 74.2% on the NIST SRM 2710a control soil. In addition, the results of the ANOVA assessment indicated a significant ($p < 0.05$) difference among the laboratories. While Tukey's HSD found the data from laboratory E to be significantly different from all of the remaining laboratories, visual inspection of Figure 3 (below) does not indicate the mean IVBA from laboratory E is substantially different from the mean IVBAs from the other laboratories; therefore, the data from laboratory E were retained in the final dataset.

Since SRC eliminated both laboratories B and C data for the Pb IVBA (mg/kg) results but the Shaw analysis only eliminated laboratory B (see section 'V.C.3 Lead IVBA Results (EPA SOP 9200.2-86)' for a discussion on why laboratory B data was eliminated but laboratory C was retained), there is a difference in the Pb IVBA results with 4562 ± 183 mg/kg for the SRC analysis versus 4619 ± 227 mg/kg from Table E-2. Consequently, the Pb % IVBA results differ as well, 71 ± 4.7% from the SRC analysis versus 71.7 ± 5.2% from Table D-4.

Table 7: SRC Statistical Results for FCRM

Results	Mean	SD	99% PI	99% CI of mean	n
Pb 3051A (mg/kg)	6444	345	5489-7399	6285-6603	35
As 3051A (mg/kg)	728	65	550-905	700-756	40
Pb IVBA (mg/kg)	4562	183	4049-5076	4470-4654	30
Pb IVBA (%)	71	4.7	56-86	69-73[1]	65[2]

[1]A Fieller's method modified by Dilba was used to calculate the 99% confidence interval for the % IVBA.
[2]Based on thirty (30) IVBA extraction results and thirty-five (35) Method 3051A digestion results.

Figure 1. All Laboratory Results, Lead by Method 3051a
(Mean ± 2 SEM)

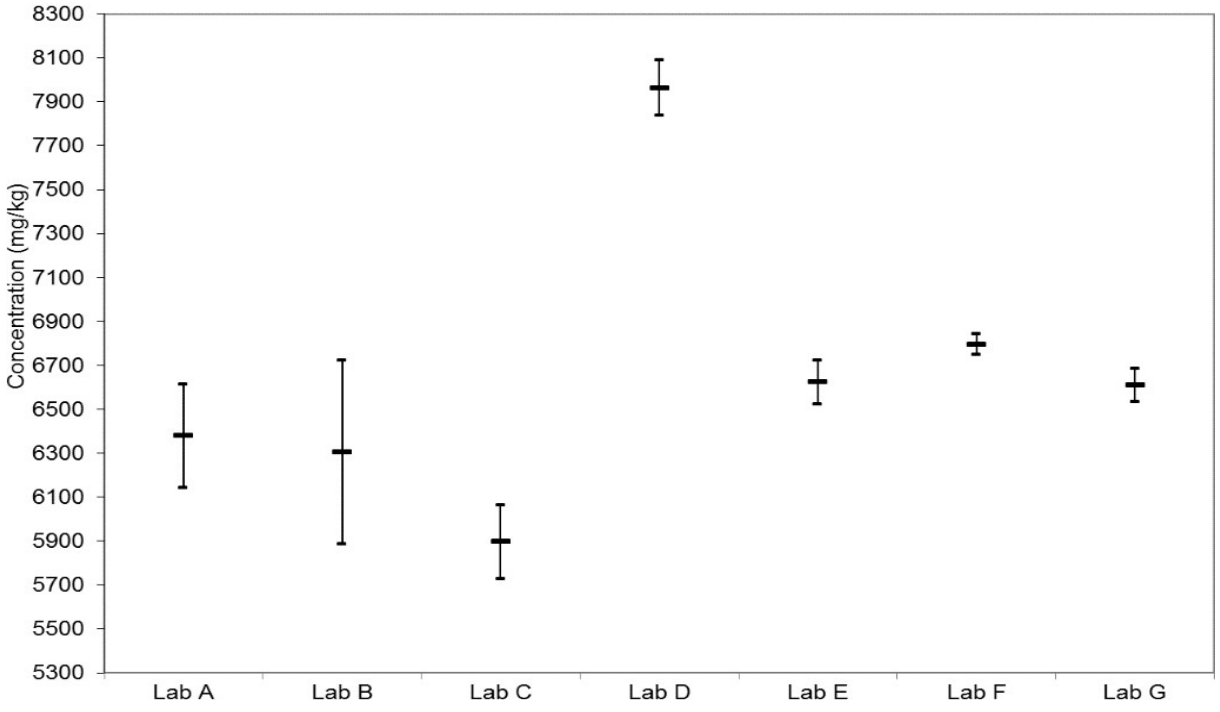


Figure 2. All Laboratory Results, Arsenic by Method 3051a
(Mean ± 2 SEM)

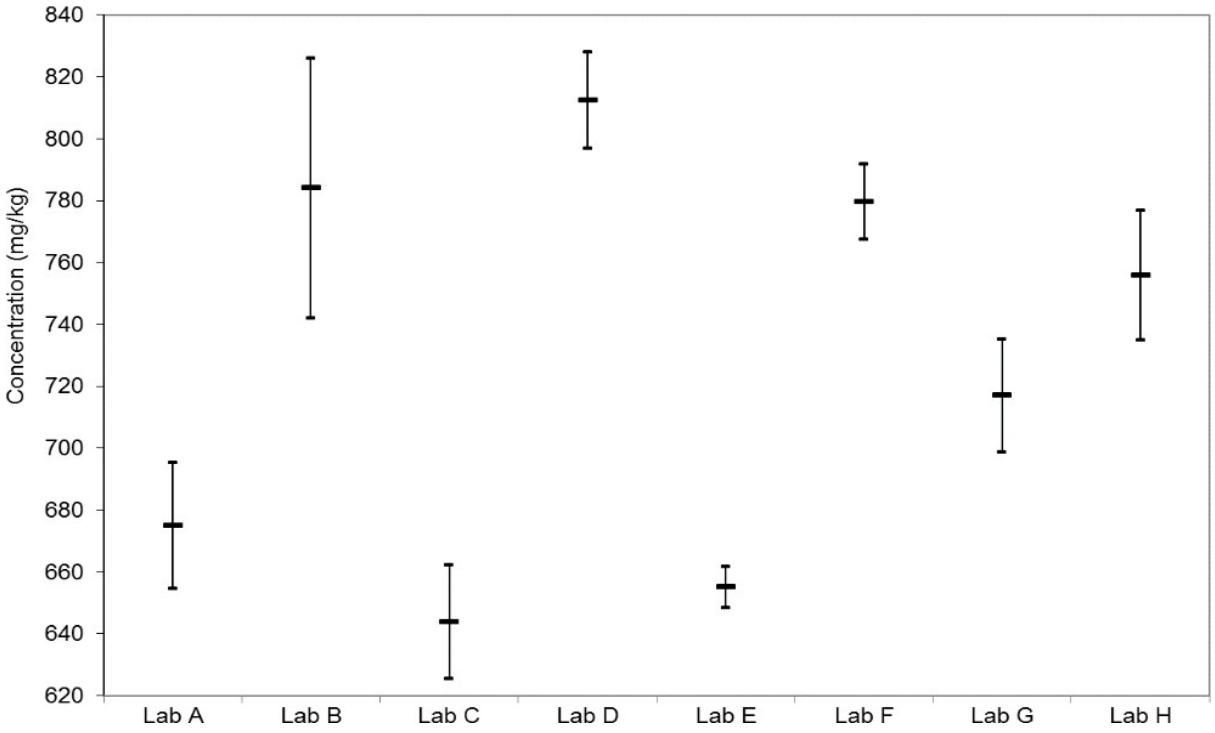
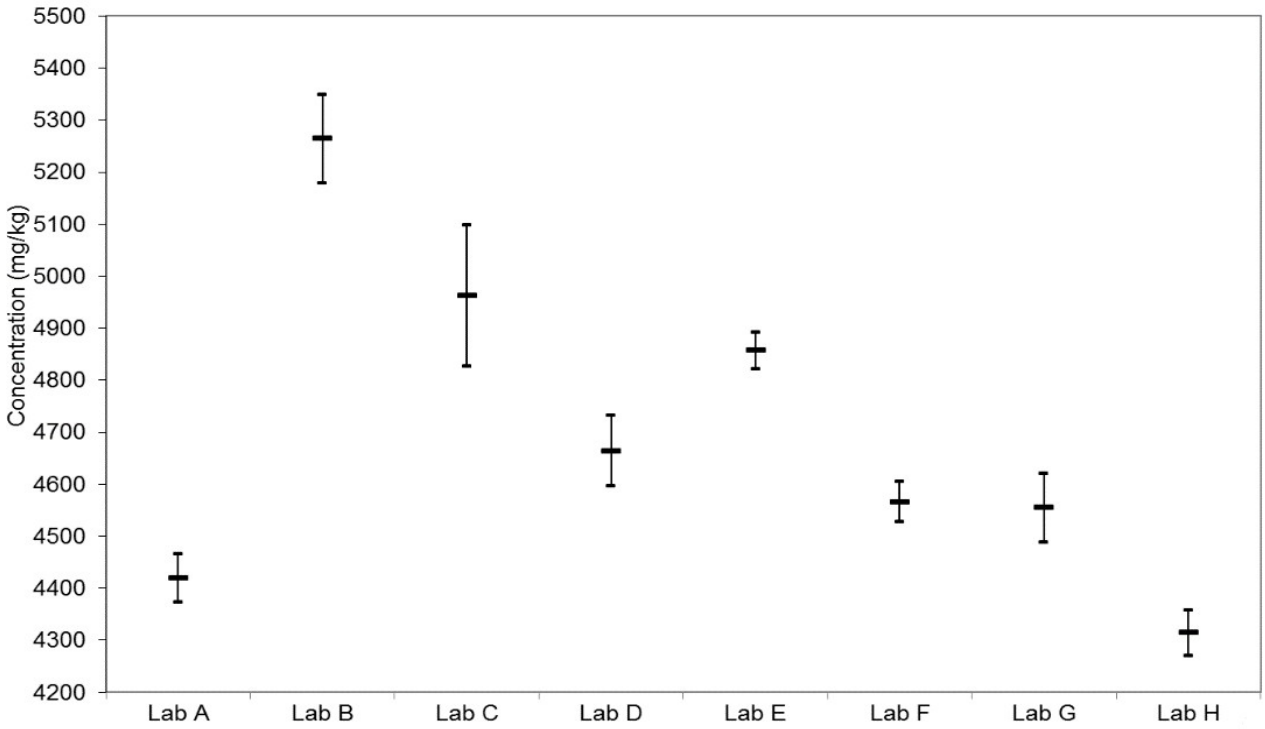


Figure 3. All Laboratory Results, Lead IVBA
(Mean ± 2 SEM)



Note: Conclusion of Section V.E. – "Independent Statistical Analysis of the Round Robin Study Results by Syracuse Research Corporation (SRC) and Comparison to Shaw's Standard Statistical Analysis Results"

The TRW IVBA committee selected the Shaw results provided in this report as the final results for the Round Robin Study, noting that there is little difference between the statistical results provided by the Shaw standard statistical approach and the results from the SRC statistical approach.

VI. CONCLUSIONS AND RECOMMENDATIONS

The main objectives of this Study were to derive a Lead IVBA mean with known confidence for the FCRM, as well as to estimate the 99 percentile prediction interval. Another objective was to derive a mean value with known confidence for the lead and arsenic concentrations for the FCRM based on the EPA Method 3051A results from this Study. The Study results from the eight (8) participating laboratories were all determined to be acceptable using conventional statistics and the Grubb's test for outliers. However, the t-test allowed for the exclusion of one laboratory's Lead IVBA results and another laboratory's EPA Method 3051A lead results, which allowed for the establishment of a Lead IVBA value for the FCRM with known and acceptable precision. This Study also provided for the determination of the lead and arsenic concentrations of the FCRM with known and acceptable precision. The associated QC results provided by the participating laboratories were all within the EPA SOP 9200.2-86 defined control limits, with a few noted exceptions.

Table 8 presents the final rounded values for the mean result and 99 percentile prediction intervals for the FCRM Lead IVBA results, as well as the EPA Method 3051A lead and arsenic values and

prediction intervals based on the pooled Study results. The prediction intervals for the EPA Method 3051A lead and arsenic values are presented in mg/Kg, and the Lead IVBA prediction intervals are presented in both mg/Kg and as Lead IVBA values.

Table 8. Rounded Values for the FCRM Lead IVBA and EPA Method 3051A Lead and Arsenic Results

FCRM	Low 99% PI	Mean	High 99% PI
Lead Method 3051A (mg/Kg)	5490	6440	7400
Arsenic Method 3051A (mg/Kg)	550	730	910
Lead IVBA (mg/Kg)	3990	4620	5250
Lead IVBA (%)	57.8	71.7	85.6

VII. REFERENCES

Dilba, G., Bretz, F. and V.Guiard. 2006. Simultaneous Confidence Sets and Confidence Intervals for Multiple Ratios. Journal of Statistical Planning and Inference. 136, 2640-2658.

Fieller, E.C. 1954. Some Problems in Interval Estimation. Journal of the Royal Statistical Society. Series B (Methodological), Vol. 16, No. 2, 175-185.

NFESC (2003). *Guide for Incorporating Bioavailability Adjustments into Human Health and Ecological Risk Assessments at U. S. Department of Defense Facilities*, Naval Facilities Engineering Service Center (NFESC), Air Force Center for Environmental Excellence (AFCEE), Army Environmental Center (AEC), Part 2, Appendix C, June 2003.

Ruby, M., Drexler, J., "*In Vitro Method for Determination of Lead Bioaccessibility Standard Operating Procedure for Stomach Phase Extraction*", June 2003.

Tamhane, A.C. and Logan, B.R. 2004. Finding the Maximum Safe Dose Level for Heteroscedastic Data. J. Biopharmaceutical Statistics, Vol.14, No. 4, 843-856.

US EPA (2012) *Standard Operating Procedure for an In Vitro Bioaccessibility Assay for Lead in Soil*, USEPA OSWER 9200.2-86, July 2012. On-line at: http://epa.gov/superfund/bioavailability/pdfs/EPA_Pb_IVBA_SOP_040412_FINAL_SRC.pdf

USEPA SW-846 EPA *Method 3051A Microwave Assisted Acid Digestion of Sediments, Sludges, Soils, and Oils*, USEPA Rev. 1, February, 2007. On-line at: http://www.epa.gov/osw/hazard/testmethods/sw846/pdfs/3051a.pdf

US EPA (2007). *Estimation of Relative Bioavailability of Lead in Soil and Soil-like Materials using In Vivo and In Vitro methods*, USEPA OSWER 9285.7-77, May 2007.

Appendix A
FCRM EPA Method 3051A Lead Results and Statistics

Table A-1. FCRM EPA Method 3051A Lead Results With Prediction Intervals and Confidence Intervals – All Labs

FCRM EPA Method 3051A Lead Results (mg/Kg)								
Laboratory >	**A**	**B**	**C**	**D**	**E**	**F**	**G**	**H**
Replicate 1	6180	5600	5762	7812	6788	6838	6670	6246
Replicate 2	6036	6500	6019	8141	6543	6742	6470	6513
Replicate 3	6657	6350	6156	8087	6687	6815	6605	6471
Replicate 4	6579	6870	5845	7878	6566	6739	6670	6538
Replicate 5	6439	6200	5699	7898	6533	6844	6630	6737
Mean	6378	6304	5896	7963	6623	6796	6609	6501
SD	264	466	188	143	111	51	82	175
RSD	4.1%	7.4%	3.2%	1.8%	1.7%	0.8%	1.2%	2.7%

Pooled Results (n-1) n=40	
Mean	6634
SD	604
RSD	9.1%

FCRM EPA Method 3051A Lead – 99 Percentile Prediction Interval (mg/Kg)		
Low 99 % PI	**Mean**	**High 99 % PI**
4978	6634	8290
± 99 % Prediction Interval = 25.0% of the Mean Value		

The range above should be used to determine if a laboratory EPA Method 3051A lead result is acceptable.

FCRM EPA Method 3051A Lead – 99 Percentile Confidence Interval of the Mean		
6634 = Mean	96 = SD of the Mean	1.4% = RSD of the Mean
Low 99 % CI	**Mean**	**High 99 % CI**
6375	6634	6892
± 99 Percentile of the Confidence Interval of the Mean = 3.9% of the Mean Value		

The range above can be used to statistically assess the confidence in the accuracy of the mean result.

SD = Standard Deviation
RSD = Relative Standard Deviation
CI = Confidence Interval
PI = Prediction Interval

Appendix A
FCRM EPA Method 3051A Lead Results and Statistics

Table A-2. FCRM EPA Method 3051A Batch QC Sample Lead Results

Laboratory>	A	B	C	D	E	F	G	H	Mean
Blank Spike Recovery (Nominal: 10 mg/L) (Range: 85% to 115%)	93.2%	111.8%	96.3%	108.0%	96.9%	104.0%	101.0%	105.0%	102.0%
FCRM Matrix Spike Recovery (Nominal: 10 mg/L) (Range: 75% to 125%)	101.9%	88%	83.2%	57.0%	98.1%	76.5%	80.0%	81.2%	83.2%
NIST SRM 2710a Digestion Lead Results NIST Certificate (Nominal: 5100 mg/Kg) (Range: 4700-5800 mg/kg)	5554	5370	4882	4912	(4537)	5491	5195	5181	5140
Lead IVBA NIST SRM 2710a Recovery Based on NIST Certificate Leachable Value of 5100 mg/Kg	108.9%	105.3%	95.7%	96.3%	89.0%	107.7%	101.9%	101.6%	100.8%

Values in parentheses are outside the associated control limits.

Table A-3. FCRM EPA Method 3051A Lead Digestion Analysis of Variance

Excel ANOVA: Single Factor (Lead)
Note: alpha at 0.05 (95 percentile)

SUMMARY

Groups	*Count*	*Sum*	*Mean*	*Variance*
Laboratory A	5	31891	6378	69533
Laboratory B	5	31520	6304	216830
Laboratory C	5	29480	5896	35459
Laboratory D	5	39816	7963	20328
Laboratory E	5	33117	6623	12215
Laboratory F	5	33978	6796	2648
Laboratory G	5	33045	6609	6805
Laboratory H	5	32505	6501	30784

ANOVA

Source of	*SS*	*df*	*MS*	*F*	*P-value*	*F-Crit*
Interlaboratory	12650866	7	1807266	36.6	1.59 E-13	2.31
Intralaboratory	1578406	32	49325			
Total	14229273	39				

SS = Sum of Squares
Df = Degrees of Freedom
MS = Mean Square
F = F Value Calculated
F-Crit = Critical Value of F
P-value = Probability Value

Appendix B
FCRM EPA Method 3051A Lead Results and t-Test

Table B-1. FCRM Lead Results and t-Test for Laboratory D Digestion Data

FCRM EPA Method 3051A Lead Results t-Test (mg/Kg)									
Laboratory>	**A**	**B**	**C**	**E**	**F**	**G**	**H**	**Laboratory>**	**D**
Replicate 1	6180	5600	5762	6788	6838	6670	6246	**Replicate 1**	7812
Replicate 2	6036	6500	6019	6543	6742	6470	6513	**Replicate 2**	8141
Replicate 3	6657	6350	6156	6687	6815	6605	6471	**Replicate 3**	8087
Replicate 4	6579	6870	5845	6566	6739	6670	6538	**Replicate 4**	7878
Replicate 5	6439	6200	5699	6533	6844	6630	6737	**Replicate 5**	7898
Mean	6378	6304	5896	6623	6796	6609	6501	**Mean**	7963
SD	264	466	188	111	51	82	175	**SD**	143
RSD	4.1%	7.4%	3.2%	1.7%	0.76%	1.2%	2.7%	**RSD**	1.8%

Labs A-H		**Percent Difference**	**Lab D**	
	n=35			**N=5**
Mean	6444	21.1%	**Mean**	7963
SD	345		**SD**	143
RSD	5.4%		**RSD**	1.8%

Excel t-Test: Two-Sample Assuming Equal Variances
alpha = 0.01 1 in 99)

	Lab A-H	**Lab D**
Mean	6444	7963
Variance	119094	20328
Observations	35	5
Pooled Variance	108698	
Hypothesized Mean Difference	0	
Df	38	
t-Stat	9.64	
P(T ≤ t) (two-tail)	9.40 E-12	
t-Critical (two-tail)	2.71	

The t-Stat value of 9.64 is greater than t-Critical two-tail value of 2.71; therefore, the null hypothesis that the means are not significantly different (zero difference, same population), can be rejected.

A P(T ≤ t) two tail value of less than 0.01 indicates a greater than 99% probability that the means of the two groups do not come from the same population.

Based on the above statements and t-test results, the results from Laboratory D can be excluded.

Appendix B
FCRM EPA Method 3051A Lead Results and t-Test

Table B-2. FCRM EPA Method 3051A Lead Results With Prediction Intervals and Confidence – Minus Laboratory D

FCRM EPA Method 3051A Lead Results (mg/Kg)								
Laboratory >	**A**	**B**	**C**	**D**	**E**	**F**	**G**	**H**
Replicate 1	6180	5600	5762		6788	6838	6670	6246
Replicate 2	6036	6500	6019		6543	6742	6470	6513
Replicate 3	6657	6350	6156		6687	6815	6605	6471
Replicate 4	6579	6870	5845		6566	6739	6670	6538
Replicate 5	6439	6200	5699		6533	6844	6630	6737
Mean	6378	6304	5896		6623	6796	6609	6501
SD	264	466	188		111	51	82	175
RSD	4.1%	7.4%	3.2%		1.7%	0.8%	1.2%	2.7%

Pooled Results (n-1) n=35	
Mean	6444
SD	345
RSD	5.4%

FCRM EPA Method 3051A Lead Results – 99 Percentile Prediction Interval (mg/Kg)		
Low 99 % PI	**Mean**	**High 99 % PI**
5489	6444	7399
± 99 % Prediction Interval = 14.8% of the Mean Value		

The range above should be used to determine if a laboratory EPA Method 3051A *lead result is acceptable.*

FCRM EPA Method 3051A Lead Results – 99 Percentile Confidence Interval of the Mean		
6444 = Mean	58 =SD of the Mean	0.91% = RSD of the Mean
Low 99 % CI	**Mean**	**High 99 % CI**
6285	6444	6603
± 99 Percentile of the Confidence Interval of the Mean = 2.5% of the Mean Value		

The range above can be used to statistically assess the confidence in the accuracy of the mean result.

SD = Standard Deviation
RSD = Relative Standard Deviation
CI = Confidence Interval
PI = Prediction Interval

Appendix C
FCRM EPA Method 3051A Arsenic Results and Statistics

Table C-1. FCRM Laboratory Arsenic Results With Prediction Intervals and Confidence Intervals – All Labs

FCRM EPA Method 3051A Arsenic Results (mg/Kg)								
Laboratory >	**A**	**B**	**C**	**D**	**E**	**F**	**G**	**H**
Replicate 1	652	716	621	800	649	776	715	748
Replicate 2	653	760	673	841	656	789	700	731
Replicate 3	700	792	655	816	649	770	712	742
Replicate 4	696	830	639	799	654	765	706	790
Replicate 5	674	822	631	806	667	798	752	768
Mean	675	784	644	812	655	779	717	756
SD	23	47	21	17	7	14	21	24
RSD	3.4%	6.0%	3.2%	2.1%	1.1%	1.8%	2.9%	3.1%

Pooled Results n=40	
Mean	728
Std Dev	65
RSD	8.9%

FCRM EPA Method 3051A Arsenic – 99 Percentile Prediction Interval (mg/Kg)		
Low 99 % PI	**Mean**	**High 99 % PI**
550	728	905
± 99 Prediction Interval = 24.2% of the Mean Value		

The range above should be used to determine if a laboratory EPA Method 3051A *arsenic result is acceptable.*

FCRM EPA Method 3051A Arsenic – 99 Percentile Confidence Interval of the Mean (mg/Kg)		
728 = Mean	10.2 SD of the Mean	1.41% = RSD of the Mean
Low 99 % CI	**Mean**	**High 99 % CI**
700	728	756
± 99 Percentile of the Confidence Interval of the Mean = 3.8% of the Mean Value		

The range above can be used to assess the confidence in the accuracy of the mean result.

SD = Standard Deviation
RSD = Relative Standard Deviation
CI = Confidence Interval
PI = Prediction Interval

Appendix C
FCRM EPA Method 3051A Arsenic Results and Statistics

Table C-2. FCRM EPA Method 3051A Batch QC Sample Arsenic Results

Laboratory>	A	B	C	D	E	F	G	H	Mean
Blank Spike Recovery (Range: 85% to 115%)	90.8%	114.8%	90.5%	110.0%	95.8%	102.0%	99.9%	104.0%	101.0%
RM Matrix Spike Recovery (Range: 75% to 125%)	103.8%	NA	106.7%	106.0%	120.7%	97.9%	100.0%	97.1%	104.5%
NIST SRM 2710a Arsenic (Mean: 1400 mg/Kg) (Range: 1300-1600 mg/Kg)	1592	(1650)	1460	1546	1322	(1684)	1505	1577	1592

Values in parentheses are outside the associated control limits.

Table C-3. FCRM EPA Method 3051A Arsenic Results Analysis of Variance

Excel ANOVA: Single Factor (Arsenic)
Note: alpha at 0.05 (95 percentile)

SUMMARY

Groups	*Count*	*Sum*	*Mean*	*Variance*
Laboratory A	5	3375	675	520
Laboratory B	5	3920	784	2206
Laboratory C	5	3220	644	430
Laboratory D	5	4062	812	301
Laboratory E	5	3274	655	56.4
Laboratory F	5	3897	779	187
Laboratory G	5	3584	717	424
Laboratory H	5	3778	756	553

ANOVA

Source of Variation	*SS*	*Df*	*MS*	*F*	*P-value*	*F-Crit*
Interlaboratory	145124	7	20732	35.5	2.51 E-13	2.31
Intralaboratory	18708	32	585			
Total	163833	39				

SS = Sum of Squares
df = Degrees of Freedom
MS = Mean Square
F = F Value Calculated
F-Crit = Critical Value of F
P-value = Probability Value

Appendix D
FCRM Lead IVBA Results and Statistics

Table D-1. FCRM Lead IVBA Results
With Prediction Intervals and Confidence Intervals

FCRM Lead IVBA Results (mg/Kg)								
Laboratory >	**A**	**B**	**C**	**D**	**E**	**F**	**G**	**H**
Replicate 1	4360	5210	4870	4762	4921	4609	4538	4314
Replicate 2	4491	5420	5000	4639	4840	4604	4434	4285
Replicate 3	4387	5260	5060	4622	4849	4549	4584	4267
Replicate 4	4448	5260	5130	4576	4857	4563	4589	4393
Replicate 5	4409	5170	4750	4720	4816	4505	4626	4310
Mean	4419	5264	4962	4664	4856	4566	4554	4314
SD	52	95	152	76	39	43	74	48
RSD	1.17%	1.81%	3.07%	1.62%	0.81%	0.94%	1.63%	1.12%

Pooled Results (n-1) n=40	
Mean	4700
SD	304
RSD	6.5%

FCRM Lead IVBA – 99 Percentile Prediction Interval (mg/Kg)		
Low 99 % PI	**Mean**	**High 99 % PI**
3866	4700	5534
± 99 % Prediction Interval = 17.7% of the Mean Value		
The range above should be used to determine if a laboratory EPA SOP 9200.2-86 IVBA extracted lead result is acceptable.		

FCRM Lead IVBA – 99 Percentile Prediction Interval		
Low 99 % PI	**Mean**	**High 99 % PI**
49.8	70.8	91.9
± 99 % Prediction Interval = 29.7% of the Mean Value		
The pooled EPA SOP 9200.2-86 lead extraction results have been divided by the pooled EPA Method 3051A digestion results to derive an EPA 9200.2-86 Lead IVBA value that includes the variance of both extraction and digestion results		
IVBA = 70.8 or 70.8%	SD = 7.9	RSD = 11.2%
The range above should be used to determine if a laboratory EPA SOP 9200.2-86 lead IVBA result is acceptable.		

FCRM Lead IVBA – Confidence Interval of the Mean		
70.8 = Mean	0.88 SD of the Mean	1.24% = RSD of the Mean
Low 99 % PI	**Mean**	**High 99 % PI**
68.5	70.8	73.2
± 99 Percentile of the Confidence Interval of the Mean = 3.3% of the Mean Value		
The range above can be used to statistically assess the confidence in the accuracy of the mean result.		

SD = Standard Deviation
RSD = Relative Standard Deviation
CI = Confidence Interval
PI = Prediction Interval

Appendix D
FCRM Lead IVBA Results and Statistics

Table D-2. FCRM Lead IVBA and EPA Method 3051A Lead Batch QC Sample Results

	Laboratory>	A	B	C	D	E	F	G	H	Mean
1	**Reagent Blank <25 ug/L**	1	<3	<40	<1	1.8	<2.5	<11	4.5	NA
2	**Bottle Blank ug/L <50 ug/L**	1	<3	<40	<1	1.6	<2.5	<11	4.1	NA
3	**Blank Lead Spike Recovery (Control Limits: 85% to 115%)**	91.4%	104.0%	108.3%	100.0%	98.0%	113.0%	102.0%	98.4%	101.9%
4	**FCRM Lead Matrix Spike Recovery (Control Limits: 75% to 125%)**	100.2%	117.6%	116.2%	62%	103.1%	62%	122%	92.2%	96.9%
5	**NIST SRM 2710a mg/Kg Lead IVBA Results (Nominal = 3440 mg/Kg)**	3325	4000	3943	3595	3615	3400	3393	3332	3575
6	**NIST SRM 2710a Lead IVBA Percent Recovery (Nominal: 3440 mg/Kg) (Control Limits: 80% to 120%)**	96.7%	116.3%	114.6%	104.5%	105.1%	98.8%	98.6%	96.9%	103.9%
7	**NIST SRM 2710a EPA Method 3051A Digestion Lead Results (mg/Kg) NIST Certificate (Nominal: 5100 mg/Kg) (Range: 4700 to 5800 mg/Kg)**	5554	5370	4882	4912	(4537)	5491	5195	5181	5140
8	**NIST SRM 2710a EPA Method 3051A Lead Percent Recovery Based on NIST Certificate Leachable Value of 5100 mg/Kg (Control Limits: 80% to 120%)**	108.9%	105.3%	95.7%	96.3%	89.0%	107.7%	101.9%	101.6%	100.8%
9	**Lead IVBA value for NIST SRM 2710a, based on the mean EPA 3051A lead value using EPA SOP 9200.2-86 criteria. (Mean 67.5%: Control Limits: 60.7% - 74.2%)**	65.2	(78.4)	(77.3)	70.5	70.9	66.7	66.5	65.3	70.1
10	**NIST SRM 2710a Lead IVBA Results Based on both the IVBA Lead Extraction and EPA 3051A Digestion of NIST SRM 2710a During this Study Lead IVBA: (Mean 67.5%: Control Limits: 60.7% to 74.2%)**	59.9	(74.5)	(80.8)	73.2	(79.7)	61.9	65.3	64.3	69.9
11	**Lead IVBA value for NIST SRM 2710a based on the Study Lead IVBA and EPA Method 3051A results. (i.e. Row 10 divided by IVBA 67.5%)**	88.7%	110.4%	119.7%	108.4%	118.0%	91.7%	96.8%	95.3%	103.6%

NA = Not Applicable

Values in parentheses are outside the associated control limits.

Appendix D
FCRM Lead IVBA Results and Statistics

Table D-3. FCRM Lead IVBA - Analysis of Variance Results

Excel ANOVA: Single Factor (Lead)
Note: alpha at 0.05 (95 percentile)

SUMMARY

Groups	*Count*	*Sum*	*Mean*	*Variance*
Laboratory A	5	22095	4419	2658
Laboratory B	5	26320	5264	9030
Laboratory C	5	24810	4962	23170
Laboratory D	5	23319	4664	5718
Laboratory E	5	24282	4856	1534
Laboratory F	5	22830	4566	1828
Laboratory G	5	22771	4554	5491
Laboratory H	5	21569	4314	2327

ANOVA

Source of	*SS*	*df*	*MS*	*F*	*P-value*	*F-Crit*
Interlaboratory	3399119	7	485588	75.1	4.83 E-18	2.31
Intralaboratory	207022	32	6469			
Total	3606142	39				

SS = Sum of Squares
df = Degrees of Freedom
MS = Mean Square
F = F Value Calculated
F-Crit = Critical Value of F
P-value = Probability Value

Appendix D
FCRM Lead IVBA Results and Statistics

Table D-4. FCRM Lead IVBA Prediction and Confidence Intervals Minus Lab B Lead IVBA and Lab D EPA Method 3051A Lead Results

FCRM Lead IVBA – 99 Percentile Prediction Interval		
Low 99 % PI	**Mean**	**High 99 % PI**
57.8	71.7	85.6
± 99 Prediction Interval = 19.4% of the Mean Value		
The pooled extraction results been divided by the pooled digestion results to derive a Lead IVBA and includes the variance of both extraction and digestion results		
IVBA = 71.7 or 71.7%	SD = 5.2	RSD = 7.3%
The range above should be used to determine if a laboratory lead IVBA result is acceptable.		
FCRM Lead IVBA – 99 Percentile Confidence Interval of the Mean		
71.7 = Mean	0.62 SD of the Mean	0.87% = RSD of the Mean
Low 99 % CI	**Mean**	**High 99 % CI**
70.0	71.7	73.3
± 99 Percentile of the Confidence Interval of the Mean = 2.3% of the Mean Value		
The range above can be used to statistically assess the confidence in the accuracy of the mean result.		

SD = Standard Deviation
RSD = Relative Standard Deviation
CI = Confidence Interval
PI = Prediction Interval

Appendix E
FCRM Lead IVBA Results and t-Test

Table E-1. FCRM Lead IVBA Results and t-Test for Laboratory B

FCRM Lead IVBA Results t-Test (mg/Kg)									
Laboratory >	**A**	**C**	**D**	**E**	**F**	**G**	**H**	**Laboratory >**	**B**
Replicate 1	4360	4870	4762	4921	4609	4538	4314	**Replicate 1**	5210
Replicate 2	4491	5000	4639	4840	4604	4434	4285	**Replicate 2**	5420
Replicate 3	4387	5060	4622	4849	4549	4584	4267	**Replicate 3**	5260
Replicate 4	4448	5130	4576	4857	4563	4589	4393	**Replicate 4**	5260
Replicate 5	4409	4750	4720	4816	4505	4626	4310	**Replicate 5**	5170
Mean	4419	4962	4664	4856	4566	4554	4314	**Mean**	5264
SD	52	152	76	39	43	74	48	**SD**	95
RSD	1.2%	3.1%	1.6%	0.8%	0.94%	1.6%	1.1%	**RSD**	1.8%

Labs A-H		**Percent Difference**	**Lab B**	
	n=35			**n=5**
Mean	4619	13.1%	**Mean**	5264
SD	227		**SD**	95
RSD	4.9%		**RSD**	1.8%

Excel t-Test: Two-Sample Assuming Equal Variances

alpha = 0.01 (1 in 99)

	Labs A-H	**Lab B**
Mean	4619	5264
Variance	51519	9030
Observations	35	5
Pooled Variance	47047	
Hypothesized Mean Difference	0	
Df	38	
t-Stat	6.22	
P(T ≤ t) two-tail	2.87 E-07	
t-Critical two-tail	2.71	

The t-Stat value of 6.22 is greater than t-Critical two-tail value of 2.71; therefore, the null hypothesis that the means are not significantly different (zero difference, same population), can be rejected.

A P(T ≤ t) two tail value of less than 0.01 indicates a greater than 99% probability that the means of the two groups do not come from the same population.

Based on the above statements and t-test results, the results from Laboratory B can be excluded.

SD = Standard Deviation
RSD = Relative Standard Deviation

Appendix E
FCRM Lead IVBA Results and t-Test

Table E-2. FCRM Lead IVBA Results
With Prediction Intervals and Confidence Intervals - Minus Lab B

FCRM Lead IVBA Results (mg/Kg)								
Laboratory >	**A**	**B**	**C**	**D**	**E**	**F**	**G**	**H**
Replicate 1	4360		4870	4762	4921	4609	4538	4314
Replicate 2	4491		5000	4639	4840	4604	4434	4285
Replicate 3	4387		5060	4622	4849	4549	4584	4267
Replicate 4	4448		5130	4576	4857	4563	4589	4393
Replicate 5	4409		4750	4720	4816	4505	4626	4310
Mean	4419		4962	4664	4856	4566	4554	4314
SD	52		152	76	39	43	74	48
RSD	1.2%		3.1%	1.6%	0.8%	0.94%	1.6%	1.1%

Pooled Results (n-1) n=35	
Mean	4619
SD	227
RSD	4.9%

FCRM Lead IVBA – 99 Percentile Prediction Interval (mg/Kg)		
Low 99 % PI	**Mean**	**High 99 % PI**
3991	4619	5247
± 99 % Prediction Interval = 13.6% of the Mean Value		
The range above should be used to determine if a laboratory IVBA extracted lead result is acceptable.		

FCRM Lead IVBA – 99 Percentile Prediction Interval		
Low 99 % PI	**Mean**	**High 99 % PI**
50.5	69.6	88.8
± 99 % Prediction Interval = 27.5% of the Mean Value		
The pooled extraction results been divided by the pooled digestion results to derive an Lead IVBA and includes the variance of both extraction and digestion results		
IVBA = 69.6 or 69.6%	SD = 7.2	RSD = 10.3%
The range above should be used to determine if a laboratory lead IVBA result is acceptable.		

FCRM Lead IVBA – 99 Percentile Confidence Interval of the Mean		
69.6 = Mean	0.83 = SD of the Mean	1.2% = RSD of the Mean
Low 99 % CI	**Mean**	**High 99 % CI**
67.4	69.6	71.8
± 99 Percentile of the Confidence Interval of the Mean = 3.2% of the Mean Value		
The range above can be used to statistically assess the confidence in the accuracy of the mean result.		

SD = Standard Deviation
RSD = Relative Standard Deviation
CI = Confidence Interval
PI = Prediction Interval

National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 2710a

Montana I Soil

Highly Elevated Trace Element Concentrations

This Standard Reference Material (SRM) is intended primarily for use in the analysis of soils, sediments, or other materials of a similar matrix. One unit of SRM 2710a consists of 50 g of the dried, powdered soil, blended with lead oxide.

Certified Values: The certified concentrations for 22 elements, expressed as mass fractions [1] on a dry-mass basis, are provided in Table 1. Certified values are based on results obtained from critically evaluated independent analytical techniques. A NIST certified value is a value for which NIST has the highest confidence in its accuracy in that all known or suspected sources of bias have been investigated or taken into account [2].

Reference Values: The reference values for 13 constituents, expressed as mass fractions on a dry-mass basis, are provided in Table 2. Ten reference values are based on results obtained from a single NIST analytical method, and three are based on results form two NIST analytical methods. Reference values are non-certified values that are the best estimate of the true value; however, the values do not meet NIST criteria for certification and are provided with associated uncertainties that may not include all sources of uncertainty [2].

Information Values: The values for 13 elements are provided in Table 3 for information purposes only. These are non-certified values with no uncertainty assessed. The information values included in this certificate are based on results obtained from one NIST method.

Expiration of Certification: The certification of SRM 2710a is valid, within the measurement uncertainties specified, until **1 January 2019**, provided the SRM is handled in accordance with the instructions given in this certificate (see "Instructions for Use"). This certification is nullified if the SRM is damaged, contaminated, or otherwise modified.

Maintenance of SRM Certification: NIST will monitor this SRM over the period of its certification. If substantive technical changes occur that affect the certification before the expiration of this certificate, NIST will notify the purchaser. Registration (see attached sheet) will facilitate notification.

E.A. Mackey and R.R. Greenberg of the NIST Analytical Chemistry Division were responsible for coordination of the technical measurements leading to certification.

Statistical analyses were performed by J.H. Yen of the NIST Statistical Engineering Division.

Support aspects involved in the issuance of this SRM were coordinated through the NIST Measurement Services Division.

Stephen A. Wise, Chief
Analytical Chemistry Division

Gaithersburg, MD 20899
Certificate Issue Date: 7 April 2009

Robert L. Watters, Jr., Chief
Measurement Services Division

Appendix F
NIST SRM 2710a Certificate of Analysis

INSTRUCTIONS FOR USE

Sampling: The SRM should be thoroughly mixed by repeatedly inverting and rotating the bottle horizontally before removing a test portion for analysis. A minimum mass of 250 mg (dry mass - see *Instructions for Drying*) should be used for analytical determinations to be related to the mass fraction values in this Certificate of Analysis.

To obtain the certified values, sample preparation procedures should be designed to effect complete dissolution. If volatile elements (i.e., arsenic, mercury, selenium) will be determined, precautions should be taken in the dissolution of SRM 2710a to avoid volatilization losses.

Drying: To relate measurements to the certified, reference, and information values that are expressed on a dry-mass basis, users should determine a drying correction at the time of each analysis. The recommended drying procedure is oven drying for 2 h at 110 °C. Note that analytical determination of volatile elements (i.e., arsenic, mercury, selenium) should be determined on samples as received; separate samples should be dried as previously described to obtain a correction factor for moisture. Correction for moisture must be made to the data for volatile elements before comparing to the certified values. This procedure ensures that these elements are not lost during drying. The mass loss on drying for this material as bottled was approximately 2 %, but this value may change once the bottle is opened and the soil is exposed to air.

SOURCE, PREPARATION, AND ANALYSIS

Source and Preparation of Material[1]: The U.S. Geological Survey (USGS), under contract to NIST, collected and processed the material for SRM 2710a. The original collection site used for SRM 2710 was no longer available due to remediation efforts by the Montana Department of Environmental Quality. An alternative nearby site, located within the flood plain of the Silver Bow Creek, was selected. The site is approximately five miles west of Butte, Montana. Soil for SRM 2710a was placed in 22 plastic-lined five-gallon buckets using a common garden spade. The buckets were sealed and transferred to the USGS using a commercial freight carrier. At the USGS, the SRM 2710a soil was dried at room temperature, disaggregated, and sieved to remove coarse material (≥2 mm). The resulting soil was ball-milled in 50 kg portions together with an amount of lead oxide sufficient to achieve a mass fraction of 0.55 % lead in the final product. The entire ball-milled batch of soil was transferred to a cross-flow V-blender for mixing. The blended soil was radiation sterilized prior to bottling. In the final preparation step the blended material was split into containers using a custom-designed spinning riffler, which was used to divide the material into smaller batches, and then used to apportion approximately 50 g into each pre-cleaned bottle.

Every 100th bottle was set aside for chemical analyses designed to assess material homogeneity using X-ray fluorescence spectrometry (XRF), inductively coupled plasma optical emission spectrometry (ICP-OES), and inductively coupled plasma mass spectrometry (ICP-MS) at the USGS. Homogeneity assessments were performed at NIST as well, and results indicated that additional processing was needed to achieve optimum homogeneity. The material from all bottles was combined, and then ground in batches between stainless steel plates for a time sufficient to produce a powder of which ≥95 %, by mass, passed through a 200 mesh (74 µm) sieve. The resulting powder was blended, and 50 g portions were dispensed into bottles using the spinning riffler. Results from additional analyses indicated material homogeneity was acceptable (see below).

Analysis: The homogeneity was assessed for selected elements in the bottled material using X-ray fluorescence spectrometry and instrumental neutron activation analysis (INAA). The estimated relative standard deviation for material inhomogeneity is <1 % and no component for inhomogeneity was included in the expanded uncertainties of the certified or reference values.

Analyses of this material were performed at NIST and at the USGS (Denver, CO). Results from NIST were used to provide the certified, reference, and information values shown in Tables 1, 2, and 3 respectively. Results from the USGS were used to confirm those values. The analytical techniques used for each element are listed in Table 4; the analysts are listed in Tables 5 and 6.

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[1] Certain commercial equipment, instruments, or materials are identified in this certificate in order to specify adequately the experimental procedure. Such identification does not imply recommendation or endorsement by the National Institute of Standards and Technology, nor does it imply that the materials or equipment identified are necessarily the best available for the purpose.

Appendix F
NIST SRM 2710a Certificate of Analysis

Table 1. Certified Values[a,b] (Dry-Mass Basis) for Selected Elements in SRM 2710a

Element	Mass Fraction (%)			Element	Mass Fraction (mg/kg)		
Aluminum	5.95	±	0.05	Antimony	52.5	±	1.6
Arsenic	0.154	±	0.010	Barium	792	±	36
Calcium	0.964	±	0.045	Cadmium	12.3	±	0.3
Copper	0.342	±	0.005	Cobalt	5.99	±	0.14
Iron	4.32	±	0.08	Lanthanum	30.6	±	1.2
Lead	0.552	±	0.003	Mercury	9.88	±	0.21
Magnesium	0.734	±	0.038	Strontium	255	±	7
Manganese	0.214	±	0.006	Uranium	9.11	±	0.30
Phosphorus	0.105	±	0.004				
Potassium	2.17	±	0.13				
Silicon	31.1	±	0.4				
Sodium	0.894	±	0.019				
Titanium	0.311	±	0.007				
Zinc	0.418	±	0.015				

[a] Certified values for all elements except lead and mercury are the equally weighted means of results from two or three analytical methods. The uncertainty listed with each value is an expanded uncertainty about the mean. The expanded uncertainty is calculated as $U = ku_c$, where u_c is intended to represent, at the level of one standard deviation, the combined effect of between-method and within-method components of uncertainty, following the ISO Guide [3,4]. The coverage factor (k) is determined from the Student's t-distribution corresponding to the appropriate associated degrees of freedom and approximately 95 % confidence for each analyte.

[b] The certified values for lead and mercury are each results from a single NIST method (see Table 4) for which a complete evaluation of all sources of uncertainty has been performed. The uncertainties for the certified values for these elements represent expanded uncertainties with a coverage factor of 2, with uncertainty components combined following the ISO Guide [4].

Appendix F
NIST SRM 2710a Certificate of Analysis

Table 2. Reference Values[a,b,c] (Dry-Mass Basis) for Selected Elements in SRM 2710a

Element	Mass Fraction (mg/kg)
Cesium	8.25 ± 0.11
Chromium	23 ± 6
Europium	0.82 ± 0.01
Gadolinium	3.0 ± 0.1
Lutetium	0.31 ± 0.01
Neodymium	22 ± 2
Nickel	8 ± 1
Rubidium	117 ± 3
Samarium	4.0 ± 0.2
Scandium	9.9 ± 0.1
Thallium	1.52 ± 0.02
Thorium	18.1 ± 0.3
Vanadium	82 ± 9

[a] Reference values for all elements except chromium, nickel, samarium, and vanadium are based on results from one analytical method at NIST (see Table 4) and the uncertainties represent the expanded uncertainties, which include the combined Type A and Type B with a coverage factor of 2, following the ISO Guide [4].

[b] Reference values for nickel and samarium are the equally weighted means of results from two analytical methods for nickel and two INAA experiments for samarium. The uncertainty listed with each value is an expanded uncertainty about the mean. The expanded uncertainty is calculated as $U = ku_c$, where u_c is intended to represent, at the level of one standard deviation, the combined effect of between-method and within-method components of uncertainty, following the ISO Guide [3,4]. The coverage factor (k) is determined from the Student's t-distribution corresponding to the appropriate associated degrees of freedom and approximately 95 % confidence for each analyte.

[c] Reference values for chromium and vanadium are based on a weighted mean calculated based on the Dersimonian-Laird method [5], which incorporates an estimate of the between-method variance into the weights. The expanded uncertainty listed with these values is calculated as $U = ku_c$, where $k = 2$, and u_c is intended to represent, at the level of one standard deviation, the combined effect of between-method and within-method components of uncertainty.

Table 3. Information Values[a] (Dry-Mass Basis) for Selected Elements in SRM 2710a

Element	Mass Fraction (mg/kg)
Boron	20
Cerium	60
Dysprosium	3
Gold	0.2
Hafnium	7
Indium	7
Selenium	1
Silver	40
Tantalum	0.9
Terbium	0.5
Tungsten	190
Ytterbium	2
Zirconium	200

[a] Information values are based on results from one analytical method at NIST

Appendix F
NIST SRM 2710a Certificate of Analysis

Table 4. NIST Methods Used for the Analysis of SRM 2710a

Element	Methods	Element	Methods
Ag	INAA	Na	INAA; XRF
Al	INAA; XRF	Nd	INAA
As	CCT-ICP-MS; INAA; XRF	Ni	ICP-MS; ICP-OES
Au	INAA	P	ICP-OES; XRF
B	PGAA	Pb	ID-ICP-MS
Ba	INAA: XRF	Rb	INAA
Ca	INAA; XRF	Sb	ICP-MS; INAA
Cd	ID-ICP-MS; PGAA	Sc	INAA
Ce	INAA	Se	CCT-ICP-MS
Co	INAA; ICP-OES	Si	PGAA; XRF
Cr	INAA; XRF	Sm	INAA[a]
Cs	INAA	Sr	ICP-OES; XRF
Cu	INAA; XRF	Ta	INAA
Dy	INAA	Tb	INAA
Eu	INAA	Th	INAA
Fe	INAA; PGAA; XRF	Ti	PGAA; XRF
Gd	PGAA	Tl	ICP-MS
Hf	INAA	U	ICP-MS; INAA
Hg	CV-ID-ICPMS	V	INAA; XRF
K	INAA; PGAA; XRF	W	INAA
La	INAA[a]	Yb	INAA
Lu	INAA	Zn	INAA; XRF
Mg	INAA; XRF	Zr	XRF
Mn	INAA; PGAA; XRF		

NIST Methods of Analysis

CCT-ICP-MS	Collision cell inductively coupled plasma mass spectrometry
CV-ID-ICP-MS	Cold vapor isotope dilution inductively coupled plasma mass spectrometry
ICP-MS	Inductively coupled plasma mass spectrometry
ICP-OES	Inductively coupled plasma optical emission spectrometry
ID-ICP-MS	Isotope dilution inductively coupled plasma mass spectrometry
INAA	Instrumental neutron activation analysis
PGAA	Prompt gamma-ray activation analysis
XRF	X-ray fluorescence spectrometry

USGS Methods of Analysis[b]

WD-XRF-2	Wavelength dispersive X-ray fluorescence spectrometry at USGS
ICP-OES-2	Inductively coupled plasma optical emission spectrometry at USGS
ICP-MS-2	Inductively coupled plasma mass spectrometry at USGS

[a]Two different INAA experiments, performed using different sub-samples and different analytical conditions, were used to provide certified and reference values for lanthanum and samarium, respectively.
[b]USGS Methods of Analysis were used to confirm results from certification methods.

Appendix F
NIST SRM 2710a Certificate of Analysis

Table 5. Participating NIST Analysts:

S.J. Christopher	S.A. Rabb
R.D. Day	J.R. Sieber
S.E. Long	R.O. Spatz
E.A. Mackey	R.S. Popelka-Filcoff
A.F. Marlow	B.E. Tomlin
J.L. Molloy	L.J. Wood
K.E. Murphy	L.L. Yu
R.L. Paul	R. Zeisler

Table 6. Participating USGS Laboratory and Analysts

Laboratory	Analysts
U.S. Geological Survey	M.G. Adams
Branch of Geochemistry	Z.A. Brown
Denver, CO, USA	P.L. Lamothe
	J.E. Taggart
	S.A. Wilson

REFERENCES

[1] Thompson, A.; Taylor, B.N.; *Guide for the Use of the International System of Units (SI)*, NIST Special Publication 811 (2008); available at http://www.physics.nist.gov/Pubs/contents.html.

[2] May, W.E.; Gills, T.E.; Parris, *R.; Beck, II, C.M.; Fassett, J.D.; Gettings, R.J.; Green*berg, R.R.; Guenther, F.R.; Kramer, G.; MacDonald, B.S.; Wise, S.A.; *Definitions of Terms and Modes Used at NIST for Value-Assignment of Reference Materials for Chemical Measurements*, NIST Special *Publication 260-136* (1999); available at http://www.cstl.nist.gov/nist839/NIST_special_publications.htm.

[3] Levenson, M.S.; Banks, D.L.; Eberhardt, K.R.; Gill, L.M.; Guthrie, W.F.; Liu, H.K.; Vangel, M.G.; Yen, J.H.; Zhang, N.F.; J. Res. NIST 105, pp. 571-579 (2000).

[4] ISO; *Guide to the Expression of Uncertainty in Measurement*, ISBN 92-67-10188-9, 1st ed.; International Organization for Standardization: Geneva, Switzerland (1993); see also Taylor, B.N.; Kuyatt, C.E.; *Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurement Results*, NIST Technical Note 1297, U.S. Government Printing Office, Washington, DC (1994); available at http://www.physics.nist.gov/Pubs/contents.html.

[5] DerSimonian, R.; Laird, N.; Controlled Clinical Trials 7, 177-188 (1986).

Users of this SRM should ensure that the certificate in their possession is current. This can be accomplished by contacting the SRM Program at: telephone (301) 975-2200; fax (301) 926-4751; e-mail srminfo@nist.gov; or via the Internet at http://www.nist.gov/srm.

Addendum to Certificate

Standard Reference Material® 2710a

Montana I Soil

Highly Elevated Trace Element Concentrations

Leachable Concentrations Determined Using USEPA Methods 200.7 and 3050B

The mass fraction values contained in the NIST Certificate of Analysis for SRM 2710a represent the total element content of the material. The measurement results used to provide the certified, reference or information values are obtained from methods that require complete sample decomposition, or from nondestructive analytical methods such as instrumental neutron activation analysis or prompt gamma-ray activation analysis. Where complete sample decomposition is required, it can be accomplished by digestion with mixed acids or by fusion. For mixed-acid decomposition, hydrofluoric acid must be included in the acid mixture used to totally decompose siliceous materials such as soils and sediments.

In its monitoring programs, the U.S. Environmental Protection Agency (USEPA) has established a number of leach methods for the preparation of soil samples for the determination of extractable elements. Six laboratories participated, five of which used USEPA Method 200.7; the remaining laboratory used USEPA SW-846 Method 3050B for preparation of soil samples. All elements were determined in leachates by inductively coupled plasma optical emission spectrometry. All laboratories provided individual results from duplicate portions, and these results were averaged together to provide one result for each element from each participating laboratory. Results rejected as outliers by the USEPA Contract Laboratory Program (CLP) officials were not included. Results are summarized in Table A1. The ranges of mass fraction values, median values (to two significant figures), and the number of results included for each are given for 23 elements. The percent recovery values based on the ratios of the median values to the total element content (from the certified, reference, or information values in the Certificate of Analysis) are listed in the last column of Table A1. **Note that the certified values provided as total mass fractions in the Certificate of Analysis are the best estimate of the true mass fraction values for this material.**

This USEPA CLP Study was coordinated by Clifton Jones, Quality Assurance and Technical Support Program (QATS), Shaw Environmental & Infrastructure Group, Las Vegas, NV, under the direction of John Nebelsick, USEPA, Analytical Services Branch. The participating laboratories are listed in Table A2.

Appendix F
NIST SRM 2710a Certificate of Analysis

Table A1. Results from Laboratories Participating in the EPA Contract Laboratory Program Study.

Element	n	Range (mg/kg)	Median (mg/kg)	Recovery (%)
Aluminum	6	8200 - 12000	10000	17
Antimony	6	5.0 - 12	9.6	18
Arsenic	6	1300 - 1600	1400	92
Barium	6	490 - 540	510	65
Beryllium	6	0.24 - 0.51	0.48	--
Cadmium	5	9.6 - 12	11	86
Calcium	6	1700 - 2000	1800	19
Chromium	6	9.2 - 11	10	41
Cobalt	6	2.8 - 5.2	3.8	64
Copper	6	3100 - 3500	3300	95
Iron	6	30000 - 36000	34000	79
Lead	6	4700 - 5800	5100	93
Magnesium	6	3200 - 3600	3500	48
Manganese	6	1500 - 1800	1700	77
Mercury	6	9.3 - 11.7	10	104
Nickel	5	4.8 - 6.1	5.5	69
Potassium	6	3800 - 4700	4100	19
Selenium	2	1.5 - 2.6	2.0	200
Silver	6	31 - 39	36	91
Sodium	6	550 - 650	590	7
Thallium	3	1.3 - 3.6	3.2	213
Vanadium	6	35 - 43	38	48
Zinc	6	3300 - 4400	3800	90

Table A2. List of CLP and non-CLP Participating Laboratories

A4 Scientific, Inc.
Bonner Analytical Testing Co.
Chem Tech Consulting Group
Datachem Laboratories, Inc.
Liberty Analytical Corporation
SVL Analytical, Inc.

APPENDIX G

Laboratory Submitted Initial Demonstration of Proficiency Forms

Laboratory A

Appendix G
Laboratory Submitted IDP Forms

LAB A Initial Demonstration of Proficiency (IDP) Form For IVBA Round Robin of NIST 2710a and 2711a (ver. 06-30-10)

Before the USEPA initiates the Round Robin analysis of the NIST 2710a and 2711a materials they have requested that each of the laboratories that wish to participate in the study complete the following Initial Demonstration of Proficiency (IDP) Form, Clifton Jones (Quality Assurance Technical Support Laboratory) US (702 895-8713) clifton.jones@shawgrp.com

General and Facility Questions

1	**Number of IVBA analyses your facility has performed for lead using the attached SOP?**	
2	**Will your facility conduct the extraction? (Yes/No)**	Yes
3	**If the answer to question 2 is no, please provide the name of the laboratory that will be conducting the extraction. (Lab Name)**	
4	**Will your facility conduct the extract analysis? (Yes/No)**	No
5	**If the answer to question 4 is no, please provide the name of the laboratory that will be conducting the analysis. (Lab Name)**	Other lab name was here
6	**Will your facility be able to conduct the attached IVBA Method EPA 9200.1-86 as written? (air controlled temperature is OK) (Yes, or Provide comment Below in 7)**	No
7	**If the answer the question 6 is no, please provide the deviation from the EPA 9200.1-86 method in the field provided here. Comment-** The apparatus we use is different from the one described in the SOP. It consists of a locally built Plexiglas/LPDE basket attached to via a pulley to a motor that operates at 30 rpm in an end over end rotation. The basket holds up to ten 125 ml HPDE bottles. The basket containing the bottles is immersed in a water bath maintained at a temperature of 37 ± 2°C. We have been using this apparatus for IVBA determination since 2002 (mostly for arsenic and lead) but have compiled relevant data for lead since 2007. We will use the protocol as written including matrix spikes which we have not included frequently in the past.	

Apparatus

8	**Does the IVBA apparatus your facility has use air or water as the 37°C thermal conducting/controlling medium. (Air, Water)**	Water
9	**How many bottle positions does your apparatus have?**	10

Analytical

10	**Type of analytical instrument use for the final Determination (ICP-AES) (ICP-MS) (GFAA) or specify other instrument type.**	ICP-MS
11	**Please provide the instrumental detection limit for the procedure that you currently use for the IVBA method. (□g/L)**	0.1 µg/L
12	**Name of Control Soil - Reference Material typically used by your facility for the IVBA extraction. (e.g., NIST 2710 or 2711, or other)**	NIST 2711
13	**Blank spike amount (mg/L) used in your procedure.**	10 mg/L
14	**Matrix spike amount (mg/L) used in your procedure.**	10 mg/L

Appendix G
Laboratory Submitted IDP Forms

Analytical (continued)

Table of Batch IVBA Results

No	Batch Date	Reagent Blank µg/L	Bottle Blank µg/L	Spiked Blank Result	Spike Blank Percent Recovery	Matrix Spike Percent Recovery	Duplicate Relative Percent Difference	Reference Material Name	Control Soil Result (mg/L) (analytical solution)	Control Soil RPD	Control Soil IVBA
A	**Date**	**<25 µg/L**	**<50 µg/L**	**(mg/L)**	**85-115%**	**75-125%**	**<20%RPD**			**<10%RPD**	**IVBA%**
B	**mm/dd/yyyy**	**<25 µg/L**	**<50 µg/L**	**9.2**	**92.4%**	**87.3%**	**7.4%**	**NIST 2711**	**9.12 mg/L**	**7.1%**	**75.5%**
1	01/15/2007	<50	<50	9.5	95.0	N/A	11.9	NIST 2711	10.1	9.54	84.6
2	10/02/2007	<50	<50	9.5	95.0		0.0	NIST 2711	9.42	2.17	81.4
3	11/26/2007	<50	<50	9.5	94.7		9.5	NIST 2711	9.85	6.83	84.8
4	11/28/2007	<50	<50	9.3	93.3		2.4	NIST 2711	9.59	4.01	82.5
5	12/03/2007	<50	<50	9.4	94.1		27.0	NIST 2711	9.44	2.39	81.2
6	12/04/2007	<50	<50	9.5	94.7		6.9	NIST 2711	9.75	5.75	83.9
7	12/05/2007	<50	<50	9.5	95.0		31.6	NIST 2711	9.85	6.83	84.8
8	12/05/2007	<50	<50	9.5	95.2		14	NIST 2711	10.0	8.46	86.1
9	12/05/2007	<50	<50	9.5	95.2		5.1	NIST 2711	10.1	9.54	86.9
10	12/09/2007	<50	<50	9.8	97.8		10.0	NIST 2711	9.42	2.17	81.1
11	03/25/2008	<0.1	1.1	9.3	93.0		6.2	NIST 2711	10.2	10.6	89.2
12	03/28/2008	<0.1	0.7	10.5	105		2.3	NIST 2711	10.7	16.1	90.6
13	05/08/2008	<0.1	0.7	9.8	98		0.4	NIST 2711	10.9	18.2	91.5
14	05/08/2008	<0.1	0.8	10.2	102		2.7	NIST 2711	10.1	9.54	86.8
15	11/26/2008	<0.1	4.2	9.8	98		2.8	NIST 2711	9.79	6.18	84.3
16	11/27/2008	<0.1	1.0	10.5	105		4.2	NIST 2711	10.4	12.8	89.5
17	02/03/2009	<0.1	3.5	9.9	99		3.6	NIST 2711	9.50	3.04	80.6
18	02/04/2009	<0.1	0.9	9.9	99		7.7	NIST 2711	9.77	5.97	82.6
19	02/05/2009	<0.1	0.5	10.5	105		2.3	NIST 2711	8.97	-2.71	75.7
20	02/05/2009	<0.1	0.5	9.9	99		1.5	NIST 2711	9.34	1.30	79.3
21	02/05/2009	<0.1	0.4	9.9	99		1.5	NIST 2711	8.97	-2.71	75.7
22	02/10/2009	<0.1	12.5	11.1	111		14.5	NIST 2711	10.0	8.46	84.4
23	02/11/2009	<0.1	0.2	10.3	103		1.6	NIST 2711	10.1	9.54	86.2
24	02/12/2009	<0.1	0.2	10.2	102		2.2	NIST 2711	10.2	10.6	87.1
25	02/12/2009	<50	<50	10.3	103		2.9	NIST 2711	9.34	1.30	78.4

26	10/05/2009	<50	<50	9.5	95.0		4.5	NIST 2711	10.6	15.0	91.6
27	11/23/2009	<0.1	<0.5	10.3	103		11.9	NIST 2711	10.4	12.8	88.6
28	01/12/2010	<0.1	2.3	10.7	107		0.1	NIST 2711	10.40	12.8	88.2
29	01/13/2010	<0.1	1.0	10.8	108		0.9	NIST 2711	10.80	17.1	91.2
30	02/09/2010	<0.1	0.6	10.7	107		1.7	NIST 2711	8.10	-12.1	69.7
31	02/09/2010	<0.1	0.6	10.3	103		8.0	NIST 2711	9.57	3.80	82.4
32	02/13/2010	<0.2	<0.2	10.3	103		9.2	NIST 2711	10.0	8.46	86.1
33	02/15/2010	<5	<5	9.5	95.0		12.2	NIST 2711	10.4	12.8	86.7
34	02/15/2010	<5	<5	9.8	98.2		0.0	NIST 2711	10.0	8.46	85.3
35	02/17/2010	<0.2	0.5	10.4	104		2.4	NIST 2711	9.19	-0.33	78.6
36	02/19/2010	<0.1	0.4	10.3	103		2.3	NIST 2711	9.82	6.51	82.1
37	02/23/2010	<0.1	0.3	10.5	105		2.0	NIST 2711	9.57	3.80	80.1
38	02/23/2010	<0.1	1.4	10.2	102		2.6	NIST 2711	9.66	4.77	80.1
39	02/24/2010	<0.1	0.6	10.2	102		0.1	NIST 2711	9.24	0.22	79.5
40	03/01/2010	<0.1	0.6	10.2	102		1.2	NIST 2711	9.52	3.25	80.8
41	03/02/2010	<0.1	0.4	10.3	103		1.9	NIST 2711	9.36	1.52	80.3
42	03/03/2010	<0.1	1.1	10.4	104		8.5	NIST 2711	9.76	5.86	81.0
43	03/04/2010	<0.1	0.3	10.3	103		3.5	NIST 2711	9.48	2.82	81.0
44	03/05/2010	<0.1	0.3	10.4	104		2.2	NIST 2711	9.74	5.64	82.9
45	03/09/2010	<0.1	0.8	10.4	104		79	NIST 2711	9.96	8.03	82.4
46	03/09/2010	<0.1	0.5	10.5	105		0.7	NIST 2711	9.84	6.72	83.1

Note Row A presents the quality control acceptance criteria from the USEPA IVBA Method EPA 9200.1-86, and Row B provides an example.

Laboratory B

Appendix G
Laboratory Submitted IDP Forms

Lab B Initial Demonstration of Proficiency (IDP) Form For IVBA Round Robin of NIST 2710a and 2711a (ver. 06-30-10) (submitted 7-08-2010)

Before the USEPA initiates the Round Robin analysis of the NIST 2710a and 2711a materials they have requested that each of the laboratories that wish to participate in the study complete the following Initial Demonstration of Proficiency (IDP) Form, Clifton Jones (Quality Assurance Technical Support Laboratory) US (702 895-8713) clifton.jones@shawgrp.com

General and Facility Questions

1	**Number of IVBA analyses your facility has performed for lead using the attached SOP?**	**~ 50 for Pb (> 150 for As)**
2	**Will your facility conduct the extraction? (Yes/No)**	**Yes**
3	**If the answer to question 2 is no, please provide the name of the laboratory that will be conducting the extraction. (Lab Name)**	
4	**Will your facility conduct the extract analysis? (Yes/No)**	**No**
5	**If the answer to question 4 is no, please provide the name of the laboratory that will be conducting the analysis. (Lab Name)**	**Other lab name was here.**
6	**Will your facility be able to conduct the attached IVBA Method EPA 9200.1-86 as written? (air controlled temperature is OK) (Yes, or Provide comment Below in 7)**	**Yes**
7	**If the answer the question 6 is no, please provide the deviation from the EPA 9200.1-86 method in the field provided here. Comment-**	

Apparatus

8	**Does the IVBA apparatus your facility has use air or water as the 37^{O}C thermal conducting/controlling medium. (Air, Water)**	**water**
9	**How many bottle positions does your apparatus have?**	**10**

Analytical

10	**Type of analytical instrument use for the final Determination (ICP-AES) (ICP-MS) (GFAA) or specify other instrument type**	**ICP-MS**
11	**Please provide the instrumental detection limit for the procedure that you currently use for the IVBA method. (□g/L)**	**0.106 □g/L**
12	**Name of Control Soil - Reference Material typically used by your facility for the IVBA extraction. (e.g., NIST 2710 or 2711, or other)**	**NIST 2711**
13	**Blank spike amount (mg/L) used in your procedure.**	**10.0 mg/L**
14	**Matrix spike amount (mg/L) used in your procedure.**	**n.a.**

Appendix G
Laboratory Submitted IDP Forms

Analytical (continued)

Table of Batch IVBA Results

No	Batch Date	Reagent Blank µg/L	Bottle Blank µg/L	Spiked Blank Result	Spike Blank Percent Recovery	Matrix Spike Percent Recovery	Duplicate Relative Percent Difference	Reference Material Name	Control Soil Result (mg/L) (analytical solution)	Control Soil RPD	Control Soil IVBA
A	Date	<25 µg/L	<50 µg/L	(mg/L)	85-115%	75-125%	<20%RPD			<10%RPD	IVBA%
B	mm/dd/yyyy	<25 µg/L	<50 µg/L	9.2	92%	87%	7%	NIST 2711	9.12 mg/L	7.1%	75.5%
1	4/26/2005	n.m.	< 5	9.6	96%	n.m.	n.m.	NIST 2711	11	n.m.	95%^
2	8/22/2005	< 5	< 5	1.0*	100	n.m.	0	NIST 2711	12	n.m.	103%^
3	8/30/2005	n.m.	< 5	11	110	n.m.	10%	NIST 2711	10, 10, 10, 11**	10%	86%^
4	9/1/2005	n.m.	< 5	8.9	89	n.m.	3%	NIST 2711	9.6, 9.5, 9.8, 9.6**	3%	83%^
5	9/12/2005	n.m.	< 5	11	110	n.m.	1%	NIST 2711	10,10, 9.9, 10**	1%	86%^
6	9/19/2005	n.m.	< 5	11	110	n.m.	9.5%	NIST 2711	10, 10, 11, 11**	9.5%	91%^
7	9/21/06	< 5	8	11	110	n.m.	n.m.	NIST 2711	9.5	n.m.	82%^
8	9/22/2006	< 5	9	11	110	n.m.	n.m.	NIST 2711	15	n.m.	130%^
9	8/22/2008	< 5	< 5	11	110	n.m.	0	NIST 2711	10	n.m.	86%^
10											

*** Spiked to 1.0 mg/L Pb.**
**** NIST soil extracted 4 times during this data set.**
^ Assumes concentration of lead in NIST 2711 soil is 1162 mg/kg, per certificate of analysis.

Note Row A presents the quality control acceptance criteria from the USEPA IVBA Method EPA 9200.1-86, and Row B provides an example.

Laboratory C

Appendix G
Laboratory Submitted IDP Forms

LAB C Initial Demonstration of Proficiency (IDP) Form For IVBA Round Robin of NIST 2710a and 2711a (ver. 07-02-10)

(Submitted 7-26-2010)

Before the USEPA initiates the Round Robin analysis of the NIST 2710a and 2711a materials they have requested that each of the laboratories that wish to participate in the study complete the following Initial Demonstration of Proficiency (IDP) Form, Clifton Jones (Quality Assurance Technical Support Laboratory) US (702 895-8713) clifton.jones@shawgrp.com

General and Facility Questions

1	Number of IVBA analyses your facility has performed for lead using the attached SOP?	1,926 (MS Access data base query, includes QC)
2	Will your facility conduct the extraction? (Yes/No)	yes
3	If the answer to question 2 is no, please provide the name of the laboratory that will be conducting the extraction. (Lab Name)	
4	Will your facility conduct the extract analysis? (Yes/No)	yes
5	If the answer to question 4 is no, please provide the name of the laboratory that will be conducting the analysis. (Lab Name)	
6	Will your facility be able to conduct the attached IVBA Method EPA 9200.1-86 as written? (air controlled temperature is OK) (Yes, or Provide comment Below in 7)	Yes
7	If the answer the question 6 is no, please provide the deviation from the EPA 9200.1-86 method in the field provided here. Comment-	

Apparatus

8	Does the IVBA apparatus your facility has use air or water as the 37^{O}C thermal conducting/controlling medium. (Air, Water)	Water
9	How many bottle positions does your apparatus have?	10

Analytical

10	Type of analytical instrument use for the final Determination (ICP-AES) (ICP-MS) (GFAA) or specify other instrument type.	ICP-AES or ICP-MS (We have both)
11	Please provide the aqueous method detection limit for the procedure that you currently use for the IVBA method. (□g/L)	ICP 40 ug/L & ICP-MS 0.1 ug
12	Name of Control Soil - Reference Material typically used by your facility for the IVBA extraction. (e.g., NIST 2710 or 2711, or other)	NIST 2711
13	Blank spike amount (mg/L) used in your procedure.	High 10 mg/L Low is 1 mg/L
14	Matrix spike amount (mg/L) used in your procedure.	High 10 mg/L Low is 1 mg/L

Appendix G
Laboratory Submitted IDP Forms

Analytical (continued)

Table of Batch IVBA Results

No	Batch Date	Reagent Blank µg/L	Bottle Blank µg/L	Spiked Blank Result	Spike Blank Percent Recovery	Matrix Spike Percent Recovery	Duplicate Relative Percent Difference	Reference Material Name	Control Soil Result (mg/L) (analytical solution)	Control Soil RPD	Control Soil IVBA
A	Date	<25 µg/L	<50 µg/L	(mg/L)	85-115%	75-125%	<20%RPD			<10%RPD	IVBA%
B	mm/dd/yyyy	<25 µg/L	<50 µg/L	9.2	92.4%	87.3%	7.4%	NIST 2711	9.12 mg/L	7.1%	75.5%
1	06/04/2009	<25ug/L	<40 ug/L	10.42	104.3	121.8	2.2	NIST 2711	9.48	2.4	82
2	06/29-2008	<25ug/L	<40 ug/L	9.62	96.2	92.5	0.6	NIST 2711	9.13	0.4	79
3	06/28/2008	<25ug/L	<40 ug/L	9.69	96.9	95.7	3.2	NIST 2711	9.36	0.1	81
4	02//05/2008	<25ug/L	<40 ug/L	9.81	98.1	84.2	0.8	NIST 2711	9.47	2.6	81
5	02/07/2008	<25ug/L	<40 ug/L	9.94	99.4	85.5	0.2	NIST 2711	8.21	2.6	71
6	02/07/2008	<25ug/L	<40 ug/L	9.53	95.3	89.2	0.1	NIST 2711	9.20	2.5	79
7	02/07/2008	<25ug/L	<40 ug/L	9.43	94.3	89.00	1.8	NIST 2711	9.11	0.6	78
8	04/24/2008	<25ug/L	<40 ug/L	9.89	98.9	92.3	1.1	NIST 2711	9.66	2.2	83
9	05/16/2008	<25ug/L	<40 ug/L	9.43	94.3	Lab C-M3 FLAG* SEE Below	0.7	NIST 2711	9.10	0.8	78
10	08/08/2009	<25ug/L	<40 ug/L	9.28	92.8	Lab C-M3 FLAG* SEE Below	2.5	NIST 2711	8.92	2.7	77

Note Row A presents the quality control acceptance criteria from the USEPA IVBA Method EPA 9200.1-86, and Row B provides an example.

M3 Flag on Lab -X's reports. M3 = The Spike Recovery value is unusable since the analyte concentration in the sample was disproportionate to the spike level. The recovery of associated control samples (LFB & LCS) was acceptable. In this case the samples were so high in Pb the spike values were unusable

Control Soil IVBA % were based on TV of 1162, which is the value used by the EPA in the 2007b validation document, (Drexler and Brattin 2007: EPA 2007b)

Laboratory D

Appendix G
Laboratory Submitted IDP Forms

LAB D Initial Demonstration of Proficiency (IDP) Form For IVBA Round Robin of NIST 2710a and 2711a (ver. 07-02-10)

(Submitted 7-21-2010)

Before the USEPA initiates the Round Robin analysis of the NIST 2710a and 2711a materials they have requested that each of the laboratories that wish to participate in the study complete the following Initial Demonstration of Proficiency (IDP) Form, Clifton Jones (Quality Assurance Technical Support Laboratory) US (702 895-8713) clifton.jones@shawgrp.com

General and Facility Questions

1	Number of IVBA analyses your facility has performed for lead using the attached SOP?	~9-10,000
2	Will your facility conduct the extraction? (Yes/No)	Yes
3	If the answer to question 2 is no, please provide the name of the laboratory that will be conducting the extraction. (Lab Name)	
4	Will your facility conduct the extract analysis? (Yes/No)	Yes
5	If the answer to question 4 is no, please provide the name of the laboratory that will be conducting the analysis. (Lab Name)	
6	Will your facility be able to conduct the attached IVBA Method EPA 9200.1-86 as written? (air controlled temperature is OK) (Yes, or Provide comment Below in 7)	Yes
7	If the answer the question 6 is no, please provide the deviation from the EPA 9200.1-86 method in the field provided here. Comment-	

Apparatus

8	Does the IVBA apparatus your facility has use air or water as the 37^{O}C thermal conducting/controlling medium. (Air, Water)	Either
9	How many bottle positions does your apparatus have?	We have two 10 position

Analytical

10	Type of analytical instrument use for the final Determination (ICP-AES) (ICP-MS) (GFAA) or specify other instrument type.	ICP/MS
11	Please provide the aqueous method detection limit for the procedure that you currently use for the IVBA method. (□g/L)	.02 ug/l
12	Name of Control Soil - Reference Material typically used by your facility for the IVBA extraction. (e.g., NIST 2710 or 2711, or other)	NIST 2710 , 2711, or 2710A
13	Blank spike amount (mg/L) used in your procedure.	1 mg/l
14	Matrix spike amount (mg/L) used in your procedure.	1 mg/l

Appendix G
Laboratory Submitted IDP Forms

Analytical (continued)

Table of Batch IVBA Results

No	Batch Date	Reagent Blank µg/L	Bottle Blank µg/L	Spiked Blank Result	Spike Blank Percent Recovery	Matrix Spike Percent Recovery	Duplicate Relative Percent Difference	Reference Material Name	Control Soil Result (mg/L) (analytical solution)	Control Soil RPD	Control Soil IVBA
A	Date	<25 µg/L	<50 µg/L	(mg/L)	85-115%	75-125%	<20%RPD			<10%RPD	IVBA%
B	mm/dd/yyyy	<25 µg/L	<50 µg/L	9.2	92.4%	87.3%	7.4%	NIST 2711	9.12 mg/L	7.1%	75.5%
1	05/19/10	0.05	0.05	2603	100	112	6				
2	03/19/10	-.09	-.09	2669	107	99	3				
3	03/07/10	-.08	-.08	2789	111	108	9				
4	02/03/10	.07	.07	2658	106	107	34	2711	0.611	2.2	
5	12/03/09	.23	.23	2744	110	102	6				
6	12/02/09	.1	.1	2614	105	102	16	2711	0.567	8.9	
7	11/09/09	.17	.17	2497	100	94	23				
8	12.03/09	.08	.08	2667	107	93	3				
9	12/04/09	.1	.1	2737	109	101	12				
10	12/01/09	-.04	-.04	2615	105	102	1				

Note Row A presents the quality control acceptance criteria from the USEPA IVBA Method EPA 9200.1-86, and Row B provides an example.

Laboratory E

Appendix G
Laboratory Submitted IDP Forms

Lab E Initial Demonstration of Proficiency (IDP) Form For IVBA Round Robin of NIST 2710a and 2711a (ver. 07-02-10)

Before the USEPA initiates the Round Robin analysis of the NIST 2710a and 2711a materials they have requested that each of the laboratories that wish to participate in the study complete the following Initial Demonstration of Proficiency (IDP) Form, Clifton Jones (Quality Assurance Technical Support Laboratory) US (702 895-8713) clifton.jones@shawgrp.com

General and Facility Questions

1	Number of IVBA analyses your facility has performed for lead using the attached SOP?	~ 420 analyses
2	Will your facility conduct the extraction? (Yes/No)	Yes
3	If the answer to question 2 is no, please provide the name of the laboratory that will be conducting the extraction. (Lab Name)	
4	Will your facility conduct the extract analysis? (Yes/No)	Yes
5	If the answer to question 4 is no, please provide the name of the laboratory that will be conducting the analysis. (Lab Name)	
6	Will your facility be able to conduct the attached IVBA Method EPA 9200.1-86 as written? (air controlled temperature is OK) (Yes, or Provide comment Below in 7)	Yes
7	If the answer the question 6 is no, please provide the deviation from the EPA 9200.1-86 method in the field provided here. Comment-	

Apparatus

8	Does the IVBA apparatus your facility has use air or water as the 37°C thermal conducting/controlling medium. (Air, Water)	Water
9	How many bottle positions does your apparatus have?	12

Analytical

10	Type of analytical instrument use for the final Determination (ICP-AES) (ICP-MS) (GFAA) or specify other instrument type.	ICP-MS
11	Please provide the aqueous method detection limit for the procedure that you currently use for the IVBA method. (µg/L)	0.08 µg/L
12	Name of Control Soil - Reference Material typically used by your facility for the IVBA extraction. (e.g., NIST 2710 or 2711, or other)	2710 (used through 2/10/09 when we ran out of this SRM)
13	Blank spike amount (mg/L) used in your procedure.	10 mg/L
14	Matrix spike amount (mg/L) used in your procedure.	10 mg/L

Appendix G
Laboratory Submitted IDP Forms

Analytical (continued)

Table of Batch IVBA Results for Pb

No	Batch Date	Reagent Blank µg/L	Bottle Blank µg/L	Spiked Blank Result	Spike Blank Percent Recovery	Matrix Spike Percent Recovery	Duplicate Relative Percent Difference	Reference Material Name	Control Soil Result (mg/L) (analytical solution)	Control Soil RPD	Control Soil IVBA
A	Date	<25 µg/L	<50 µg/L	(mg/L)	85-115%	75-125%	<20%RPD			<10%RPD	IVBA%
B	mm/dd/yyyy	<25 µg/L	<50 µg/L	9.2	92.4%	87.3%	7.4%	NIST 2711	9.12 mg/L	7.1%	75.5%
1	2/4/09	<5 µg/L	<5 µg/L	9.9	99.5	100	0.0	NIST 2710	40.8	1.9	73.6
2	2/4/09	<5 µg/L	<5 µg/L	10.2	101.8	99.0	1.0	NIST 2710	40.7	1.2	74.1
3	2/5/09	<5 µg/L	<5 µg/L	10.2	101.6	105	4.6	NIST 2710	46.3	6.0	79.5
4	2/5/09	<5 µg/L	<5 µg/L	10.2	102.3	103	3.2	NIST 2710	NA	NA	NA
5	2/9/09	<5 µg/L	<5 µg/L	10.0	100.5	NA	NA	NIST 2710	NA	NA	NA
6	2/10/09	<5 µg/L	<5 µg/L	10.1	101.4	99.3	0.7	NIST 2710	42.3	0.4	75.3
7	2/10/09	<5 µg/L	<5 µg/L	NA	NA	96.6	3.4	NIST 2710	43.5	3.45	77.6
8											
9											
10											

Note Row A presents the quality control acceptance criteria from the USEPA IVBA Method EPA 9200.1-86, and Row B provides an example.

Laboratory F

Appendix G
Laboratory Submitted IDP Forms

LAB F Initial Demonstration of Proficiency (IDP) Form For IVBA Round Robin of NIST 2710a and 2711a (ver. 07-02-10)

(Submitted 7-13-2010)

Before the USEPA initiates the Round Robin analysis of the NIST 2710a and 2711a materials they have requested that each of the laboratories that wish to participate in the study complete the following Initial Demonstration of Proficiency (IDP) Form, Clifton Jones (Quality Assurance Technical Support Laboratory) US (702 895-8713) clifton.jones@shawgrp.com

General and Facility Questions

1	**Number of IVBA analyses your facility has performed for lead using the attached SOP?**	**60**
2	**Will your facility conduct the extraction? (Yes/No)**	**Yes**
3	**If the answer to question 2 is no, please provide the name of the laboratory that will be conducting the extraction. (Lab Name)**	
4	**Will your facility conduct the extract analysis? (Yes/No)**	**Yes**
5	**If the answer to question 4 is no, please provide the name of the laboratory that will be conducting the analysis. (Lab Name)**	
6	**Will your facility be able to conduct the attached IVBA Method EPA 9200.1-86 as written? (air controlled temperature is OK) (Yes, or Provide comment Below in 7)**	**Yes, air controlled**
7	**If the answer the question 6 is no, please provide the deviation from the EPA 9200.1-86 method in the field provided here. Comment-**	

Apparatus

8	**Does the IVBA apparatus your facility has use air or water as the 37^{O}C thermal conducting/controlling medium. (Air, Water)**	**Air**
9	**How many bottle positions does your apparatus have?**	**12**

Analytical

10	**Type of analytical instrument use for the final Determination (ICP-AES) (ICP-MS) (GFAA) or specify other instrument type.**	**ICP-AES**
11	**Please provide the aqueous method detection limit for the procedure that you currently use for the IVBA method. (□g/L)**	**25 ug/L**
12	**Name of Control Soil - Reference Material typically used by your facility for the IVBA extraction. (e.g., NIST 2710 or 2711, or other)**	**2711**
13	**Blank spike amount (mg/L) used in your procedure.**	**10 mg/L**
14	**Matrix spike amount (mg/L) used in your procedure.**	**10 mg/L**

Appendix G
Laboratory Submitted IDP Forms

Analytical (continued)

Table of Batch IVBA Results

No	Batch Date	Reagent Blank ug/L	Bottle Blank ug/L	Spiked Blank Result	Spike Blank Percent Recovery	Matrix Spike Percent Recovery	Duplicate Relative Percent Difference	Reference Material Name	Control Soil Result (mg/L) (analytical solution)	Control Soil RPD	Control Soil IVBA
A	Date	<25 ug/L	<50 ug/L	(mg/L)	85-115%	75-125%	<20%RPD			<10%RPD	IVBA%
B	mm/dd/yyyy	<25 ug/L	<50 ug/L	9.2	92.4%	87.3%	7.4%	NIST 2711	9.12 mg/L	7.1%	75.5%
1	06/16/2010	<25 ug/L	NA	9.6	95.5%	92.3	2.8	NIST 2711	9.36 mg/L	0.7%	84.4%
2	06/28/2010	<25 ug/L	NA	9.6	95.9%	91.6	1.8	NIST 2711	9.20 mg/L	-0.8%	84.4%
3	06/30/2010	<25 ug/L	NA	9.6	96.5%	96.0	2.2	NIST 2711	9.42 mg/L	1.2%	84.4%
4	07/06/2010	<25 ug/L	NA	9.5	94.8%	94.2	3.1	NIST 2711	9.31 mg/L	0.2%	84.4%
5	07/07/2010	<25 ug/L	NA	9.5	94.8%	89.1	1.2	NIST 2711	9.19 mg/L	-0.8%	84.4%
6											
7											
8											
9											
10											

Note Row A presents the quality control acceptance criteria from the USEPA IVBA Method EPA 9200.1-86, and Row B provides an example.

Note (LAB F): 75.5% IVBA listed in example should be for NIST 2710 according to USEPA IVBA Method EPA 9200.

Laboratory G

Appendix G
Laboratory Submitted IDP Forms

LAB G Demonstration of Proficiency (IDP) Form
For IVBA Round Robin of NIST 2710a and 2711a (ver. 07-02-10)

Before the USEPA initiates the Round Robin analysis of the NIST 2710a and 2711a materials they have requested that each of the laboratories that wish to participate in the study complete the following Initial Demonstration of Proficiency (IDP) Form, Clifton Jones (Quality Assurance Technical Support Laboratory) US (702 895-8713) clifton.jones@shawgrp.com

General and Facility Questions

1	Number of IVBA analyses your facility has performed for lead using the attached SOP?	228
2	Will your facility conduct the extraction? (Yes/No)	Yes
3	If the answer to question 2 is no, please provide the name of the laboratory that will be conducting the extraction. (Lab Name)	
4	Will your facility conduct the extract analysis? (Yes/No)	Yes
5	If the answer to question 4 is no, please provide the name of the laboratory that will be conducting the analysis. (Lab Name)	
6	Will your facility be able to conduct the attached IVBA Method EPA 9200.1-86 as written? (air controlled temperature is OK) (Yes, or Provide comment Below in 7)	Yes. * However, we do not have riffle splitter to mix and split the samples. We use in air incubator set at 37 C.
7	If the answer the question 6 is no, please provide the deviation from the EPA 9200.1-86 method in the field provided here. Comment- 1. Per method comparison, We normally dry our samples at 105 deg. Celsius instead recommended <40 deg. Celsius per item # 6. May need some clarification. 2. Cost of splitter is $500- recommended but not required per specified method.	

Apparatus

8	Does the IVBA apparatus your facility has use air or water as the 37^{O}C thermal conducting/controlling medium. (Air, Water)	Air
9	How many bottle positions does your apparatus have?	8 per each

Analytical

10	Type of analytical instrument use for the final Determination (ICP-AES) (ICP-MS) (GFAA) or specify other instrument type.	ICP-AES
11	Please provide the aqueous method detection limit for the procedure that you currently use for the IVBA method. (□g/L)	50 □g/L
12	Name of Control Soil - Reference Material typically used by your facility for the IVBA extraction. (e.g., NIST 2710 or 2711, or other)	2711
13	Blank spike amount (mg/L) used in your procedure.	100 □g/L
14	Matrix spike amount (mg/L) used in your procedure.	100 □g/L

Appendix G
Laboratory Submitted IDP Forms

Analytical (continued)

Table of Batch IVBA Results

No	Batch Date	Reagent Blank µg/L	Bottle Blank µg/L	Spiked Blank Result	Spike Blank Percent Recovery	Matrix Spike Percent Recovery	Duplicate Relative Percent Difference	Reference Material Name	Control Soil Result (mg/L) (analytical solution)	Control Soil RPD	Control Soil IVBA
A	**Date**	<25 µg/L	<50 µg/L	**(mg/L)**	**85-115%**	**75-125%**	**<20%RPD**			**<10%RPD**	**IVBA%**
B	**mm/dd/yyyy**	<25 µg/L	<50 µg/L	**9.2**	**92.4%**	**87.3%**	**7.4%**	**NIST 2711**	**9.12 mg/L**	**7.1%**	**75.5%**
1	06/14/2010 (1)		<50 ug/L	4.37	83.2%	-132, -266	2.3, 4.6, 10.4, 4.8, 4.1	NIST 2711	8.39 mg/L		77.7%
2	06/14/2010 (2)		<50 ug/L	4.36	83.0%	80.1, 76.7	2.6, 6.6, 6.3, 0.9	NIST 2711	8.89 mg/L		80.8%
3	06/14/2010 (3)		<50 ug/L	4.35	82.9%	81.3, 72.9	1.6	NIST 2711	8.51 mg/L		76.7%
4	01/28/2010	<50 ug/L		4.21	84.2%	406, 403	0.6, 4.4, 2.1	NIST 2711	8.78 mg/L		85.0%
5	12/14/2009			0.745	74.5%	596, 287		NIST 2711	2.12 mg/L		67.5%
6	08/27/2009		<50 ug/L	3.75	75.0%	76.6, 86.5	6.3, 9.2, 5.8, 8.6	NIST 2711	8.10 mg/L		74.3%
7	06/30/2009		<50 ug/L	3.88	77.6%	73.7, 71.5	86.0, 0.9, 0.5, 4.8	NIST 2711	8.78 mg/L		81.3%
8	06/25/2009		<50 ug/L	4.16	83.2%	69.4, 57.0	70.2, 16.3, 1.0	NIST 2711	8.50 mg/L		78.0%
9	06/02/2009		<50 ug/L	4.02	80.4%	77.0, 91.0	3.2, 3.0, 6.5	unknown	8.20 mg/L		74.5%
10	05/26/2009		<50 ug/L			25.5, 38.9	16.4, 0.8, 8.8, 0.4, 18.8	unknown	5.70 mg/L		52.8%

Note Row A presents the quality control acceptance criteria from the USEPA IVBA Method EPA 9200.1-86, and Row B provides an example.

Data Notes: All batches had a matrix spike and matrix spike duplicate. Most batches had duplicate analyses on multiple samples.
Row 1 MS/MSD were spiked at <10% of native concentration.
Row 4 MS/MSD were spiked at ~30% of native concentration.
Row 5 MS/MSD were spiked at <15% of native concentration.
Row 10 MS/MSD were spiked at ~15% of native concentration.

Laboratory H

Appendix G
Laboratory Submitted IDP Forms

LAB H Demonstration of Proficiency (IDP) Form For Lead IVBA Round Robin of new RM, with Microwave Digestion of RM for Lead and Arsenic using EPA Method 3051A (ver. 09-22-11)

Before the USEPA initiates the Round Robin analysis the new RM they have requested that each of the laboratories that wish to participate in the study complete the following Initial Demonstration of Proficiency (IDP) Form, Clifton Jones (Quality Assurance Technical Support Laboratory) US (702 895-8713) clifton.jones@shawgrp.com

General and Facility Questions

	IVBA	
1	Number of IVBA analyses your facility has performed for lead using the attached IVBA SOP EPA 9200.1-86?	* See below
2	Will your facility conduct the extraction? (Yes/No)	Yes
3	If the answer to question 2 is no, please provide the name of the laboratory that will be conducting the Extraction. (Lab Name)	
4	Will your facility conduct the extract analysis? (Yes/No)	Yes
5	If the answer to question 4 is no, please provide the name of the laboratory that will be conducting the Analysis. (Lab Name)	
6	Will your facility be able to conduct the attached IVBA Method EPA 9200.1-86 as written? (air controlled temperature is OK) (Yes, or Provide comment Below in 7)	Yes, see LAB H Lab SOP 256.
7	If the answer the question 6 is no, please provide the deviation from the EPA 9200.1-86 method in the field provided here. Comment-	

*** The LAB H has performed 9200.1-86 on 143 client samples. 80 of these were tested for lead and the remaining 63 were tested for arsenic. These numbers do not count laboratory QC samples or work performed during method development and documentation of acceptable performance prior to running client samples.**

	Microwave Digestion using 3051A	
8	Total number of analyses your facility has performed for lead and arsenic using the attached EPA Method 3051A.	Typically has been used for oil or tissue matrix only, not soil or sediment. Currently performing MDLs and precision and accuracy studies for soil and recently updated soil procedure in SOP 420. No client soil samples in several years.
9	Will your facility conduct the digestion? (Yes/No)	Yes
10	If the answer to question 2 is no, please provide the name of the laboratory that will be conducting the Digestion. (Lab Name)	
11	Will your facility conduct the digest analysis? (Yes/No)	Yes
12	If the answer to question 4 is no, please provide the name of the laboratory that will be conducting the analysis. (Lab Name)	
13	Will your facility be able to conduct the attached EPA Method 3051A as written? (Yes, or Provide comment Below in 7)	See below.
14	If the answer the question 6 is no, please provide the deviation from EPA Method 3051A in the field provided here. Comment- See appendix A of LAB H Lab SOP 420 for deviations.	

Apparatus IVBA

16	Does the IVBA apparatus your facility has use air or water as the 37^{O}C thermal conducting/controlling medium. (Air, Water)	Air
17	How many bottle positions does your apparatus have?	It holds 12 x 2L bottles. Each 2L bottle can hold about ten 125 mL IVBA extraction bottles. Total = 120
18	Does your usual protocol allow for the pre-incubation of the extraction solution to 37°C before initiation of the IVBA extraction.	Yes
19	How do you measure temperature of the controlling apparatus?	Digital thermometer with data logger.
20	If your lab uses air control, are you using a commercially available extraction apparatus? If possible, please provide the vendor and part number, or Model number.	Associated Designs 3740-12BRE (12 place TCLP rotary

		agitator)
21	If your lab uses air control, what type of temperature control device is being used (i.e., benchtop, upright, or walk-in incubator)?	Walk-in
22	Does your lab use a pH probe which compensates for temperature (i.e., Automatic Temperature Control (ATC probe))?	Yes
23	If not, how does your lab control for temperature when measuring the pH?	

Analytical IVBA

24	Type of analytical instrument typically used for the final Determination (ICP-AES) (ICP-MS) (GFAA) or specify other instrument type.	Typically use ICP/AES. ICP/MS could be used if necessary.
25	Please provide the aqueous method detection limit for the procedure that you currently use for the IVBA method for both Lead and Arsenic. (□g/L)	As = 10 ug/L Pb = 15 ug/L
26	Name of Control Soil - Reference Material typically used by your facility for the IVBA extraction. (e.g., NIST 2710 or 2711, or other)	NIST 2711A
27	Blank spike amount (mg/L) used in your procedure.	1 mg/L
28	Matrix spike amount (mg/L) used in your procedure.	5 mg/L

Apparatus Microwave

29	Does the Microwave apparatus at your facility have temperature or pressure control.	temperature
30	How many vessel positions does your apparatus have?	12
31	Please provide the manufacturer and model of your microwave Apparatus.	CEM MARS Xpress
32	What procedure do you use for the microwave power calibration?	Not performed, use temp control
33	When was your microwave apparatus last power calibrated?	N/A

Appendix G
Laboratory Submitted IDP Forms

Table of Batch Lead IVBA Results Table modified by CLJ - QATS

No	Batch Date	Reagent Blank µg/L	Bottle Blank µg/L	Spiked Blank Result	Spike Blank Percent Recovery	Matrix Spike Percent Recovery	Duplicate Relative Percent Difference	Reference Material Name	Control Soil Result (mg/L) (analytical solution)	Control Soil RPD	Control Soil IVBA	Determination by ICP-AES or ICP-MS
A	**Date**	**<25 µg/L**	**<50 µg/L**	**(mg/L)**	**85-115%**	**75-125%**	**<20%RPD**			**<10%RPD**	**IVBA%**	
B	**Mm/dd/yyyy**	**<25 µg/L**	**<50 µg/L**	**9.2**	**92.4%**	**87.3%**	**7.4%**	**NIST 2711**	**9.12 mg/L**	**7.1%**	**82.9%**	
1	04/11/2011	<15	?	0.99	99	93	0.1	NIST 2711a	10.5	3.7	75	ICP-AES
2	04/13/2011	<15	?	5.21	104	104	7	NIST 2711a	11.6	6.2	83	ICP-AES
3	10/03/2011	<15	?	0.98	98	80	4	NIST 2711a	11	2.8	76	ICP-AES
4												
5												
6												
7												
8												
9												
10												

Note Row A presents the quality control acceptance criteria from the USEPA IVBA Method EPA 9200.1-86, and Row B provides an example.

Appendix G
Laboratory Submitted IDP Forms

Table of EPA Microwave Method 3051a Soil Batch Lead Results

No	Batch Date	Reagent Blank µg/L	Matrix Spike Percent Recovery	Duplicate Sample Relative Percent Difference	LCS or Reference Material Name	LCS or Reference Material Nominal Value (mg/Kg)	Reference Material Result (mg/ Kg)	Reference Material Result Percent Recovery	Determination by ICP-AES or ICP-MS
A	**Date**	**<25 µg/L**	**75-125%**	**<20%RPD**					
B	**Mm/dd/yyyy**	**<25 µg/L**	**87.3%**	**7.4%**	**NIST 2711**	**1100 mg/kg**	**912 mg/L**	**82.9%**	
1									
2									
3									
4									
5									
6									
7									
8									
9									
10									

Note: Row A presents typical quality control acceptance criteria from the USEPA Method 6010, and Row B provides an example.

Appendix G
Laboratory Submitted IDP Forms

Table of EPA Microwave Method 3051a Soil Batch Arsenic Results

No	Batch Date	Reagent Blank µg/L	Matrix Spike Percent Recovery	Duplicate Sample Relative Percent Difference	LCS or Reference Material Name	LCS or Reference Material Nominal Value (mg/Kg)	Reference Material Result (mg/ Kg)	Reference Material Result Percent Recovery	Determination by ICP-AES or ICP-MS
A	**Date**	**<25 µg/L**	**75-125%**	**<20%RPD**					
B	**Mm/dd/yyyy**	**<25 µg/L**	**87.3%**	**7.4%**	**NIST 2711**	**90 mg/kg**	**81 mg/kg**	**90.0%**	
1									
2									
3									
4									
5									
6									
7									
8									
9									
10									

Note: Row A presents typical quality control acceptance criteria from the USEPA Method 6010, and Row B provides an example.

APPENDIX H

Laboratory Submitted Round Robin Study Sample Results

Laboratory A

Appendix H
Laboratory Submitted Study Results

LAB A- Statement of Work for the Lead IVBA and EPA Method 3051A (for Lead and Arsenic) Round Robin Analyses of a New Reference Material (RM)

(version 4, April 9, 2012)

Introduction: The purpose of this Statement of Work (SOW) is to provide specific information and procedures for the analysis and reporting for (1) EPA SOP EPA 9200.2-86 (the lead only IVBA) and (2) EPA method 3051A (for analysis lead and arsenic) Round Robin analyses for the New EPA IVBA Reference Material (RM). Please read carefully. Analyses of the New RM must be performed in strict accordance with the EPA SOP EPA 9200.2-86 (see attachment 1) and EPA Method 3051A (see attachment 2). Any exceptions to the SOP procedures will be provided in this Statement of Work. **Please note the sample extraction, analyses, and reporting are to be completed within a thirty (30) day-turn-around time**.

Suggestions or Edits to the EPA SOP: If you have any suggested changes to the Lead IVBA SOP 9200.2-86 that might be helpful, ether editorial or technical, it would be great if you could e-mail (or provide on CD) a word document with the suggested changes along with the Round Robin Study results. If you wish, you could use the "Tools - Tract Changes" feature of WORD to provide the suggested changes to the WORD copy of the Lead IVBA SOP 9200.2-86 that you received on the CD with the Round Robin Study samples.

Sample Receipt: Two 30 mL Nalgene (polyethylene) wide mouth bottles will be provided to you. One bottle will contain approximately twenty (20) grams of the **New IVBA RM Sample**, and the second bottle will also contain approximately five (5) grams of **NIST SRM 2710a**. The bottles will be logged in to your usual sample receipt login system; however, these soil materials will not require refrigeration.

(1) EPA SOP EPA 9200.2-86 (Lead IVBA)

Required Quality Assurance/Quality Control: During the EPA review of the Initial Demonstration of Proficiency Forms (IDP) Forms submitted by the laboratories participating in the Round Robin Study, it was noted that not all laboratories performed each of the Quality Control samples that are presented in the SOP EPA 9200.2-86. **It is imperative for this study that all of the required quality control samples are prepared and analyzed as specified in the SOP EPA 9200.2-86.** It was also noted during the reviewed of the IDP Forms that different laboratories use varying acceptance criteria for the quality control parameters. **It is a requirement for this study** that the acceptance criteria presented in the SOP EPA 9200.2-86 be used for quality control sample results. Below is a table of the required quality control samples and the control limits, which was derived from Section 9 of the SOP EPA 9200.2-86. Limits that are lower than those specified for the Reagent Blank and the Bottle Blank are acceptable. Please note that a designated duplicate sample is **not** required for these analyses.

QC Sample	Control Limits
Reagent blank	<25 µg/L lead
Bottle blank	<50 µg/L lead
Blank spike (10 mg/L)	85-115% recovery
Matrix spike (10 mg/L)	75-125% recovery
Duplicate sample	±20% RPD
Control soil (NIST 2710a)	NIST 2710a Mean 67.5% (Acceptable Range 60.7- 74.2%)

All quality Control Samples must be run on every batch extraction of the NIST materials. **The New RM must each be extracted in a batch with a complete set of quality control QC samples.** Please note that the control soil NIST SRM 2710a range is based on the NIST Addendum to the Certificate of Analysis leachable median lead value of 5100 mg/kg, **not** the total dissolution Certificate of Analysis lead value 5520 mg/kg.

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Sample preparation: The provided **New IVBA RM Sample** and **NIST SRM 2710a** should be used as is. The oven drying and the sieving to less than 250um should **not** be performed. Also, riffle splitting should **not** be performed on these SRM materials. The New RM must be extracted in a single extraction batch, with five (5) replicate RM samples, along with complete associated QC samples for each batch. To insure homogeneity, the New RM and NIST SRM 2710a bottles **must** be rotated along the x, y, and z axes for at least one minute before sub-sampling for extraction. **Note: the New RM and NIST SRM 2710a materials used in this study must be weighted out to 1.000 +/- 0.001 g, which is a more precise weighing that the SOP requirement.** The extraction apparatus may have the extraction temperature controlled to 37 ± 2 ^{O}C by either air (incubator type) or water (aquarium type). For either incubator or aquarium type of extractor, the sample rotation speed must be 28 RPM as specified in the SOP.

The batch sequence that **must** be used for this study is provided in Table 1 below. Again, please note that a designated duplicate sample is not required. The sample extraction will proceed as presented in the SOP.

Table 1. IVBA Lead Extraction Batch for Round Robin Analysis of the New RM

IVBA Extraction Batch for Lead		
Extractor Position	**Sample Name**	**Comment**
1	**New RM**	
2	**New RM**	
3	**New RM**	
4	**New RM**	
5	**New RM**	
6	**New RM Matrix Spike**	**10 mg/L Pb**
7	**Bottle Blank**	
8	**Blank Spike**	**10 mg/L Pb**
9	**Control Soil NIST SRM 2710a**	

Sample Filtering and Analysis: Sample filtering and analysis should proceed as indicated in the SOP. The analysis will be performed using either EPA SW-846 method 6010C (ICP-AES) or 6020A (ICP-MS); however, the analytical sequence should be exactly as specified in Table 2.

Reporting: Tables 3 and 4 **must** be used for reporting the IVBA analysis results for the New RM and the associated QC samples results. The laboratory **must** provide copies of the calibration and the raw data print out from the instrumental analysis for both batches as part of the data submission.

Please complete the Results Tables 3 and 4 and e-mail to clifton.jones@shawgrp.com, followed by a 2nd day Fed-Ex mailing of the Results Tables 3 and 4, along with the copies of the calibration and the raw data print outs from the instrumental analysis to the address provided below. Please provide any other pertinent information regarding the RM extraction and analysis with the data submission.

Clifton Jones
Shaw Environmental - QATS
2700 Chandler Avenue, Bldg C
Las Vegas, Nevada, USA
89120
Tel. (702) 895-8713

Table 2. Analytical Sequence for Lead IVBA Extraction Batch for the New RM

Position	Sample Name	Comment
Initial Standard Calibration and Beginning QC Samples	Initial Standard Calibration	
	Interference Check Sample (s)	
	Initial Calibration Verification and/or Continuing Calibration Standards and Blanks, as per EPA Methods 6010C or 6020A.	
10(<<proxy position no.)	Reagent Blank	
11	Bottle Blank	(from Extractor Position 7)
12	New RM (Extractor Position 1)	
13	New RM (Extractor Position 2)	
14	New RM (Extractor Position 3)	
15	New RM (Extractor Position 4)	
16	New RM (Extractor Position 5)	
17	Control Soil NIST SRM 2710a	(from Extractor Position 9)
18	Blank Spike	10 mg/L (from Extractor Position 8)
19	New RM Matrix Spike	10 mg/L (from Extractor Position 6)
20	Continuing Calibration Verification Standard	
21	Continuing Calibration Verification Blank	
Analytical Run Closing QC Samples-	Interference Check Sample etc. as required by either EPA Methods 6010C or 6020A.	

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(1) EPA SOP EPA 9200.2-86 (Lead IVBA)

Table3: Laboratory, Instrument, Instrumental MDL, and IVBA Extraction Batch Data Reporting Form for new RM

Laboratory Performing Extraction	**Lab A**			
Laboratory Performing Analysis	**Lab A**			
IVBA Extraction Batch Results new RM: Lead				
Instrument Type? (ICP-AES or ICP-MS)	ICP-MS	**Instrument Method Detection Limit (MDL) (ug/L)**	2.0 ug/L	
Extraction Date	May 10, 2012			
Extraction Lead Standard Manufacturer and Lot #	Ultra Scientific ICP-082, Lot# L00394			
Analysis Date(s)	May 18, 2012			
Analysis Lead Standard Manufacturer and Lot #	Inorganic Ventures, E2-MEB393062			
Initial Calibration Verification Standard Source and Lot #	High Purity Standards, 1125704			
Interference Check Sample Source and Lot #	n/a			
Sample Name	**Instrument result for the analytical solution (ug/L)**	**Dilution Factor**	**Final Instrumental result analytical solution (corrected for dilution) (ug/L)**	**Result in mg/Kg (corrected for 1g/100mL extraction) (i.e., ug/L times 100/1000 = mg/kg)**
EXAMPLE SOIL (NIST 2710a)	**70**	**10**	**700**	**70**
Reagent Blank	1.01	1	1.01	
Bottle Blank	0.72	1	0.72	
RM (Extractor Position 1)	43600	1	43600	4360
RM (Extractor Position 2)	45100	1	45100	4491
RM (Extractor Position 3)	43900	1	43900	4387
RM (Extractor Position 4)	44700	1	44700	4448
RM (Extractor Position 5)	44500	1	44500	4409
Control Soil SRM 2710a	33300	1	3300	3325
Blank Spike	9140	1	9140	914
RM Matrix Spike	56400	1	56400	5547

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Laboratory Submitted Study Results

Table 4. Lead Extraction Batch Spiked Blank and Spiked Sample Results for new RM

Laboratory Performing Extraction	**Lab A**
Laboratory Performing Analysis	**Lab A**
IVBA Extraction Batch Spiked Blank and Spiked Sample Results for New RM: Lead	
Bottle Blank Result (mg/L)	0.0010
Blank Spike Result (mg/L)	0.00072 (9140) CLJ
Blank Spike Percent Recovery	91.4%
Average (5) Result RM (mg/L)	44.2
RM Matrix Spike Result (mg/L)	55.4
RM Matrix Spike Percent Recovery	100.2

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Laboratory Submitted Study Results

(2) SW-846 METHOD 3051A MICROWAVE ASSISTED ACID DIGESTION OF SEDIMENTS, SLUDGES, SOILS, AND OILS

Table 3. Laboratory, Instrument, Instrumental MDL, and 3051A Digestion Data Reporting Form for new RM: Results for Lead

Laboratory Performing Extraction	**Lab A**			
Laboratory Performing Analysis	**Lab A**			
Digestion Batch Results new RM: Lead				
Instrument Type? (ICP-AES or ICP-MS)	ICP-MS	**Instrument Method Detection Limit (MDL) (ug/L)**		
Digestion Date	May 9, 2012			
Digestion Lead Standard Manufacturer and Lot #	Ultra Scientific ICP-082, Lot# L00394			
Analysis Date(s)	May 18, 2012			
Analysis Lead Standard Manufacturer and Lot #	n/a			
Initial Calibration Verification Standard Source and Lot #	n/a			
Interference Check Sample Source and Lot #	n/a			
Sample Name	**Instrument result for the analytical solution (ug/L)**	**Dilution Factor**	**Final Instrumental result analytical solution (corrected for dilution) (ug/L)**	**Result in mg/Kg (corrected for 0.5g/100mL extraction)(i.e ug/L times 200/1000 = mg/kg)**
EXAMPLE SOIL (NIST 2710a)	**35**	**10**	**350**	**70**
Reagent Blank	0.94	1	0.94	
RM (Sample 1)	30900	1	30900	6180
RM (Sample 2)	30300	1	30300	6036
RM (Sample 3)	33300	1	33300	6657
RM (Sample 4)	32900	1	32900	6579
RM (Sample 5)	32600	1	32600	6439
Control Soil SRM 2710a	27800	1	27800	5554
Blank Spike	9320	1	9320	
RM Matrix Spike	42800	1	42800	

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Laboratory Submitted Study Results

Table 4. 3051A Digestion Spiked Blank and Spiked Sample Results for new RM: Results for Lead

Laboratory Performing Extraction	**Lab A**
Laboratory Performing Analysis	**Lab A**
3051A Digestion Spiked Blank and Spiked Sample Results for New RM: Lead	
Blank Spike Result (mg/L)	0.00094 mg/L(9.320) CLJ
Blank Spike Percent Recovery	93.2%
Average (5) Result RM (mg/L)	32.0
RM Matrix Spike Result (mg/L)	42.8
RM Matrix Spike Percent Recovery	101.9%

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Laboratory Submitted Study Results

Table 5. Laboratory, Instrument, Instrumental MDL, and 3051A Digestion Data Reporting Form for new RM: Results for Arsenic

Laboratory Performing Extraction	**Lab A**			
Laboratory Performing Analysis	**Lab A**			
Digestion Batch Results new RM: Arsenic				
Instrument Type? (ICP-AES or ICP-MS)	ICP-MS	**Instrument Method Detection Limit (MDL) (ug/L)**		
Digestion Date				
Digestion Arsenic Standard Manufacturer and Lot #	Ultra Scientific ICP-033, Lot# L00431A			
Analysis Date(s)				
Analysis Arsenic Standard Manufacturer and Lot #	Inorganic Ventures, E2-MEB393062			
Initial Calibration Verification Standard Source and Lot #	High Purity Standards, 1125704			
Interference Check Sample Source and Lot #	n/a			
Sample Name	**Instrument result for the analytical solution (ug/L)**	**Dilution Factor**	**Final Instrumental result analytical solution (corrected for dilution) (ug/L)**	**Result in mg/Kg (corrected for 0.5g/100mL extraction)(i.e ug/L times 200/1000 = mg/kg)**
EXAMPLE SOIL (NIST 2710a)	**35**	**10**	**350**	**70**
Reagent Blank	0.80	1	0.80	
RM (Sample 1)	3260	1	3260	652
RM (Sample 2)	3280	1	3280	653
RM (Sample 3)	3500	1	3500	700
RM (Sample 4)	3480	1	3480	696
RM (Sample 5)	3410	1	3410	674
Control Soil SRM 2710a	7970	1	7970	1592
Blank Spike	9080	1	9080	
RM Matrix Spike	13900	1	13900	

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Laboratory Submitted Study Results

Table 6. 3051A Digestion Spiked Blank and Spiked Sample Results for new RM: Results for Arsenic

Laboratory Performing Extraction	**Lab A**
Laboratory Performing Analysis	**Lab A**
3051A Digestion Spiked Blank and Spiked Sample Results for New RM : Arsenic	
Blank Spike Result (mg/L)	0.00080 mg/L (9.08) CLJ
Blank Spike Percent Recovery	90.8%
Average (5) Result RM (mg/L)	3.386 mg/L
RM Matrix Spike Result (mg/L)	13.9
RM Matrix Spike Percent Recovery	103.8%

Laboratory B

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Laboratory Submitted Study Results

(1) EPA SOP EPA 9200.2-86 (Lead IVBA)

Table 3. Laboratory, Instrument, Instrumental MDL, and IVBA Extraction Batch Data Reporting Form for New RM

Laboratory Performing Extraction	**Lab B**			
Laboratory Performing Analysis	**Lab B**			
IVBA Extraction Batch Results New RM: Lead				
Instrument Type? (ICP-AES or ICP-MS)	**ICP-MS**	**Instrument Method Detection Limit (MDL) (ug/L)**	**333, 0.17 (CLJ)**	
Extraction Date	**05/14/2012**			
Extraction Lead Standard Manufacturer and Lot #	**CPI INT'L**	**#11L036**		
Analysis Date(s)	**5/16/2012**			
Analysis Lead Standard Manufacturer and Lot #	**Inorganic Ventures**		**E2-MEB373122**	
Initial Calibration Verification Standard Source and Lot #	**ACCUSTANDARD**		**211055033**	
Interference Check Sample Source and Lot #	**Inorganic Ventures**		**E2-MEB348034**	
Sample Name	**Instrument result for the analytical solution (ug/L)**	**Dilution Factor**	**Final Instrumental result analytical solution (corrected for dilution) (ug/L)**	**Result in mg/Kg (corrected for 1g/100mL extraction) (i.e ug/L times 100/1000 = mg/kg)**
EXAMPLE SOIL	**70**	**10**	**700**	**70**
Reagent Blank	**0.44**	**5**	**2.2**	**0**
Bottle Blank	**0.88**	**5**	**4.4**	**0**
New RM (Extractor Position 1)	**521**	**100**	**52100**	**5210**
New RM (Extractor Position 2)	**542**	**100**	**54200**	**5420**
New RM (Extractor Position 3)	**526**	**100**	**52600**	**5260**
New RM (Extractor Position 4)	**526**	**100**	**52600**	**5260**
New RM (Extractor Position 5)	**517**	**100**	**51700**	**5170**
Control Soil NIST SRM 2710a	**400**	**100**	**40000**	**4000**
Blank Spike	**2075**	**5**	**10375**	**1040**
New RM Matrix Spike	**644**	**100**	**64400**	**6440**

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Laboratory Submitted Study Results

Table 4. Lead Extraction Batch Spiked Blank and Spiked Sample Results for New RM

Laboratory Performing Extraction	**Lab B**
Laboratory Performing Analysis	**Lab B**
IVBA Extraction Batch Spiked Blank and Spiked Sample Results for New RM: Lead	
Bottle Blank Result (mg/L)	0
Blank Spike Result (mg/L)	10375
Blank Spike Percent Recovery	104%
Average (5) Result New RM (mg/L)	5264
New RM Matrix Spike Result (mg/L)	6440
New RM Matrix Spike Percent Recovery	117.6%

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Laboratory Submitted Study Results

(2) SW-846 METHOD 3051A MICROWAVE ASSISTED ACID DIGESTION OF SEDIMENTS, SLUDGES, SOILS, AND OILS

Table 3. Laboratory, Instrument, Instrumental MDL, and 3051A Digestion Data Reporting Form for New RM: Results for Lead

Laboratory Performing Extraction	**Lab B**			
Laboratory Performing Analysis	**Lab B**			
Digestion Batch Results New RM: Lead				
Instrument Type? (ICP-AES or ICP-MS)		**Instrument Method Detection Limit (MDL) (ug/L)**		**333 , 0.17 (CLJ)**
Digestion Date	**05/14/2012**			
Digestion Lead Standard Manufacturer and Lot #	**CPI INT'L**	**#11L036**		
Analysis Date(s)	**5/16/2012**			
Analysis Lead Standard Manufacturer and Lot #	**Inorganic Ventures**		**E2-MEB373122**	
Initial Calibration Verification Standard Source and Lot #	**ACCUSTANDARD**		**211055033**	
Interference Check Sample Source and Lot #	**Inorganic Ventures**		**E2-MEB348034**	
Sample Name	**Instrument result for the analytical solution (ug/L)**	**Dilution Factor**	**Final Instrumental result analytical solution (corrected for dilution) (ug/L)**	**Result in mg/Kg (corrected for 0.5g/100mL extraction)(i.e ug/L times 200/1000 = mg/kg)**
EXAMPLE SOIL	**35**	**10**	**350**	**70**
Reagent Blank	**0.128**	**2000**	**256**	**ND@1.0**
New RM (Sample 1)	**279.8**	**20000**	**5596000**	**5600**
New RM (Sample 2)	**325.3**	**20000**	**6506000**	**6500**
New RM (Sample 3)	**317.5**	**20000**	**6350000**	**6350**
New RM (Sample 4)	**343.7**	**20000**	**6870000**	**6870**
New RM (Sample 5)	**310.2**	**20000**	**6200000**	**6200**
Control Soil NIST SRM 2710a	**268.6**	**20000**	**5372000**	**5370**
Blank Spike	**13.98**	**2000**	**27960**	**27.96**
New RM Matrix Spike	**342.4**	**20000**	**6848000**	**6850**

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Laboratory Submitted Study Results

Table 4. 3051A Digestion Spiked Blank and Spiked Sample Results for New RM: Results for Lead

Laboratory Performing Extraction	Lab B
Laboratory Performing Analysis	Lab B
3051A Digestion Spiked Blank and Spiked Sample Results for New RM: Lead	
Blank Spike Result (mg/L)	**27.96**
Blank Spike Percent Recovery	111.8
Average (5) Result New RM (mg/L)	6300
New RM Matrix Spike Result (mg/L)	6850
New RM Matrix Spike Percent Recovery	220 (250 MG/KG SPK)

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Laboratory Submitted Study Results

Table 5. Laboratory, Instrument, Instrumental MDL, and 3051A Digestion Data Reporting Form for New RM: Results for Arsenic

Laboratory Performing Extraction	**Lab B**			
Laboratory Performing Analysis	**Lab B**			
Digestion Batch Results New RM: Arsenic				
Instrument Type? (ICP-AES or ICP-MS)	**ICP-MS**	**Instrument Method Detection Limit (MDL) (ug/L)**	**333**	
Digestion Date	**05/14/2012**			
Digestion Arsenic Standard Manufacturer and Lot #	**CPI INT'L**	**#11L036**		
Analysis Date(s)	**5/16/2012**			
Analysis Arsenic Standard Manufacturer and Lot #	**Inorganic Ventures**		**E2-MEB373122**	
Initial Calibration Verification Standard Source and Lot #	**ACCUSTANDARD**		**211055033**	
Interference Check Sample Source and Lot #	**Inorganic Ventures**		**E2-MEB348034**	
Sample Name	**Instrument result for the analytical solution (ug/L)**	**Dilution Factor**	**Final Instrumental result analytical solution (corrected for dilution) (ug/L)**	**Result in mg/Kg (corrected for 0.5g/100mL extraction)(i.e ug/L times 200/1000 = mg/kg)**
EXAMPLE SOIL	**35**	**10**	**350**	**70**
Reagent Blank	**0.38**	**2000**	**760**	**ND @ 1.0**
New RM (Sample 1)	**358**	**2000**	**716000**	**716**
New RM (Sample 2)	**380**	**2000**	**760000**	**760**
New RM (Sample 3)	**396**	**2000**	**792000**	**792**
New RM (Sample 4)	**416**	**2000**	**832000**	**830**
New RM (Sample 5)	**411**	**2000**	**822000**	**822**
Control Soil NIST SRM 2710a	**826**	**2000**	**1652000**	**1650**
Blank Spike	**14.33**	**2000**	**28660**	**28.7**
New RM Matrix Spike	**358.3**	**2000**	**716600**	**717**

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Laboratory Submitted Study Results

Table 6. 3051A Digestion Spiked Blank and Spiked Sample Results for New RM: Results for Arsenic

Laboratory Performing Extraction	**Lab B**
Laboratory Performing Analysis	**Lab B**
3051A Digestion Spiked Blank and Spiked Sample Results for New RM: Arsenic	
Blank Spike Result (mg/L)	**28.7**
Blank Spike Percent Recovery	114.8
Average (5) Result New RM (mg/L)	784
New RM Matrix Spike Result (mg/L)	717
New RM Matrix Spike Percent Recovery	-268 (25 MG/KG SPK)

Laboratory C

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(1) EPA SOP EPA 9200.2-86 (Lead IVBA)

Table 3. Laboratory, Instrument, Instrumental MDL, and IVBA Extraction Batch Data Reporting Form for New RM

Laboratory Performing Extraction	**Lab C**			
Laboratory Performing Analysis	**Lab C**			
IVBA Extraction Batch Results New RM: Lead				
Instrument Type? (ICP-AES or ICP-MS)	**ICP**	**Instrument Method Detection Limit (MDL) (ug/L)**	**40 ug/L**	
Extraction Date	**04/23/12**			
Extraction Lead Standard Manufacturer and Lot #	**CPI International**	**Lot# 11G022**		
Analysis Date(s)	**04/26/12**			
Analysis Lead Standard Manufacturer and Lot #	**CPI International**	**Lot#**	**11G022**	
Initial Calibration Verification Standard Source and Lot #	**SPEX**	**Lot#**	**6-171CR**	
Interference Check Sample Source and Lot #	**SPEX**	**Lot#**	**3-50YP**	
Sample Name	**Instrument result for the analytical solution (ug/L)**	**Dilution Factor**	**Final Instrumental result analytical solution (corrected for dilution) (ug/L)**	**Result in mg/Kg (corrected for 1g/100mL extraction) (i.e ug/L times 100/1000 = mg/kg)**
EXAMPLE SOIL	**70**	**10**	**700**	**70**
Reagent Blank	**<40**	**1**	**<40**	**<4**
Bottle Blank	**<40**	**1**	**<40**	**<4**
New RM (Extractor Position 1)	**9734**	**5**	**48700**	**4870**
New RM (Extractor Position 2)	**9994**	**5**	**50000**	**5000**
New RM (Extractor Position 3)	**10112**	**5**	**50600**	**5060**
New RM (Extractor Position 4)	**10261**	**5**	**51300**	**5130**
New RM (Extractor Position 5)	**9497**	**5**	**47500**	**4750**
Control Soil NIST SRM 2710a	**7886**	**5**	**39430**	**3943**
Blank Spike	**2166**	**5**	**10830**	**1083**
New RM Matrix Spike	**12248**	**5**	**61240**	**6124**

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Table 4. Lead Extraction Batch Spiked Blank and Spiked Sample Results for New RM

Laboratory Performing Extraction	**Lab C**
Laboratory Performing Analysis	**Lab C**
IVBA Extraction Batch Spiked Blank and Spiked Sample Results for New RM: Lead	
Bottle Blank Result (mg/L)	<0.04
Blank Spike Result (mg/L)	10.83
Blank Spike Percent Recovery	108.3%
Average (5) Result New RM (mg/L)	49.62
New RM Matrix Spike Result (mg/L)	61.24
New RM Matrix Spike Percent Recovery	116.2%*

- The spike recovery value is unusable since the analyte concentration is disproportionate to the spike level. The recovery of the associated control sample (LCS or LFB) was acceptable.

2) SW-846 METHOD 3051A MICROWAVE ASSISTED ACID DIGESTION OF SEDIMENTS, SLUDGES, SOILS, AND OILS

Table 3. Laboratory, Instrument, Instrumental MDL, and 3051A Digestion Data Reporting Form for New RM: Results for Lead

Laboratory Performing Extraction	**Lab C**			
Laboratory Performing Analysis	**Lab C**			
Digestion Batch Results New RM: Lead				
Instrument Type? (ICP-AES or ICP-MS)	**ICP0**	**Instrument Method Detection Limit (MDL) (ug/L)**		
Digestion Date	**05/02/12**	**40**		
Digestion Lead Standard Manufacturer and Lot #	**CPI**	**Lot# 11G022**		
Analysis Date(s)	**05/03/12**			
Analysis Lead Standard Manufacturer and Lot #	**CPI**	**Lot#**	**11G022**	
Initial Calibration Verification Standard Source and Lot #	**SPEX**	**Lot#**	**6-171CR**	
Interference Check Sample Source and Lot #	**SPEX**	**Lot#**	**3-50YP**	
Sample Name	**Instrument result for the analytical solution (ug/L)**	**Dilution Factor**	**Final Instrumental result analytical solution (corrected for dilution) (ug/L)**	**Result in mg/Kg (corrected for 0.5g/100mL extraction)(i.e ug/L times 200/1000 = mg/kg)**
EXAMPLE SOIL	**35**	**10**	**350**	**70**
Reagent Blank	**<40**	**100**	**<4000**	**<400**
New RM (Sample 1)	**57615**	**100**	**5761500**	**576150**
New RM (Sample 2)	**60189**	**100**	**6018900**	**601890**
New RM (Sample 3)	**61556**	**100**	**6155600**	**615560**
New RM (Sample 4)	**58450**	**100**	**5845000**	**584500**
New RM (Sample 5)	**56994**	**100**	**5699400**	**569940**
Control Soil NIST SRM 2710a	**24410**	**200**	**4882000**	**488200**
Blank Spike	**9634**	**1**	**9634**	**963**
New RM Matrix Spike	**42683**	**200**	**8536600**	**853660**

Table 4. 3051A Digestion Spiked Blank and Spiked Sample Results for New RM: Results for Lead

Laboratory Performing Extraction	**Lab C**
Laboratory Performing Analysis	**Lab C**
3051A Digestion Spiked Blank and Spiked Sample Results for New RM: Lead	
Blank Spike Result (mg/L)	9.63
Blank Spike Percent Recovery	96.3%
Average (5) Result New RM (mg/L)	6872.5
New RM Matrix Spike Result (mg/L)	8536.6
New RM Matrix Spike Percent Recovery	83.2%*

- The spike recovery value is unusable since the analyte concentration is disproportionate to the spike level. The recovery of the associated control sample (LCS or LFB) was acceptable.

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Laboratory Submitted Study Results

Table 5. Laboratory, Instrument, Instrumental MDL, and 3051A Digestion Data Reporting Form for New RM: Results for Arsenic

Laboratory Performing Extraction	**Lab C**			
Laboratory Performing Analysis	**Lab C**			
Digestion Batch Results New RM: Arsenic				
Instrument Type? (ICP-AES or ICP-MS)	**ICP**	**Instrument Method Detection Limit (MDL) (ug/L)**		
Digestion Date	**05/02/12**	**60**		
Digestion Arsenic Standard Manufacturer and Lot #	**CPI**	**Lot# 11B077**		
Analysis Date(s)	**05/03/12**			
Analysis Arsenic Standard Manufacturer and Lot #	**CPI**	**Lot#**	**11B077**	
Initial Calibration Verification Standard Source and Lot #	**SPEX**	**Lot#**	**9-61-CR**	
Interference Check Sample Source and Lot #	**SPEX**	**Lot#**	**3-50YP**	
Sample Name	**Instrument result for the analytical solution (ug/L)**	**Dilution Factor**	**Final Instrumental result analytical solution (corrected for dilution) (ug/L)**	**Result in mg/Kg (corrected for 0.5g/100mL extraction)(i.e ug/L times 200/1000 = mg/kg)**
EXAMPLE SOIL	**35**	**10**	**350**	**70**
Reagent Blank	**<60**	**100**	**<6000**	**<600**
New RM (Sample 1)	**6206**	**100**	**620600**	**62060**
New RM (Sample 2)	**6734**	**100**	**673400**	**67340**
New RM (Sample 3)	**6550**	**100**	**655000**	**65500**
New RM (Sample 4)	**6393**	**100**	**639300**	**63930**
New RM (Sample 5)	**6312**	**100**	**631200**	**63120**
Control Soil NIST SRM 2710a	**7300**	**200**	**1460000**	**146000**
Blank Spike	**9054**	**1**	**9054**	**905**
New RM Matrix Spike	**13897**	**200**	**2779400**	**277940**

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Laboratory Submitted Study Results

Table 6. 3051A Digestion Spiked Blank and Spiked Sample Results for New RM: Results for Arsenic

Laboratory Performing Extraction	**Lab C**
Laboratory Performing Analysis	**Lab C**
3051A Digestion Spiked Blank and Spiked Sample Results for New RM: Arsenic	
Blank Spike Result (mg/L)	9.05
Blank Spike Percent Recovery	90.5%
Average (5) Result New RM (mg/L)	643.9
New RM Matrix Spike Result (mg/L)	2779.4
New RM Matrix Spike Percent Recovery	106.7%

Laboratory D

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Laboratory Submitted Study Results

(1) EPA SOP EPA 9200.2-86 (Lead IVBA)

Table 3. Laboratory, Instrument, Instrumental MDL, and IVBA Extraction Batch Data Reporting Form for New RM

Laboratory Performing Extraction	**Lab D**			
Laboratory Performing Analysis	**Lab D**			
IVBA Extraction Batch Results New RM: Lead				
Instrument Type? (ICP-AES or ICP-MS)	**ICP-MS**	**Instrument Method Detection Limit (MDL) (ug/L)**	**0.031**	
Extraction Date	**5/7/12**			
Extraction Lead Standard Manufacturer and Lot #	**SPEX 11-116PB**			
Analysis Date(s)	**5/8/12**			
Analysis Lead Standard Manufacturer and Lot #	**SPEX 11-116PB**			
Initial Calibration Verification Standard Source and Lot #	**SPEX 20-140JB**			
Interference Check Sample Source and Lot #	**Environmental Express 0929914 + 1119513**			
Sample Name	**Instrument result for the analytical solution (ug/L)**	**Dilution Factor**	**Final Instrumental result analytical solution (corrected for dilution) (ug/L)**	**Result in mg/Kg (corrected for 1g/100mL extraction) (i.e ug/L times 100/1000 = mg/kg)**
EXAMPLE SOIL		**50**		
Reagent Blank	**0**		**0**	**NA**
Bottle Blank	**0**		**-2.3**	**NA**
New RM (Extractor Position 1)	**952**	**50**	**47624**	**4762**
New RM (Extractor Position 2)	**927**	**50**	**46389**	**4639**
New RM (Extractor Position 3)	**924**	**50**	**46221**	**4622**
New RM (Extractor Position 4)	**915**	**50**	**45759**	**4576**
New RM (Extractor Position 5)	**943**	**50**	**47199**	**4720**
Control Soil NIST SRM 2710a	**718**	**50**	**35947**	**3595**
Blank Spike	**200**	**50**	**10041**	**NA**
New RM Matrix Spike	**1056**	**50**	**52836**	**5284**

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Table 4. Lead Extraction Batch Spiked Blank and Spiked Sample Results for New RM

Laboratory Performing Extraction	Lab D
Laboratory Performing Analysis	Lab D
IVBA Extraction Batch Spiked Blank and Spiked Sample Results for New RM: Lead	
Bottle Blank Result (mg/L)	0.0
Blank Spike Result (mg/L)	10
Blank Spike Percent Recovery	100
Average (5) Result New RM (mg/L)	46639
New RM Matrix Spike Result (mg/L)	52836
New RM Matrix Spike Percent Recovery	62

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Laboratory Submitted Study Results

(2) SW-846 METHOD 3051A MICROWAVE ASSISTED ACID DIGESTION OF SEDIMENTS, SLUDGES, SOILS, AND OILS

Table 3. Laboratory, Instrument, Instrumental MDL, and 3051A Digestion Data Reporting Form for New RM: Results for Lead

Laboratory Performing Extraction	**Lab D**			
Laboratory Performing Analysis	**Lab D**			
Digestion Batch Results New RM: Lead				
Instrument Type? (ICP-AES or ICP-MS)	**ICP-MS**	**Instrument Method Detection Limit (MDL) (ug/L)**		**0.031**
Digestion Date	**5/7/12**			
Digestion Lead Standard Manufacturer and Lot #	**SPEX 11-116PB**			
Analysis Date(s)	**5/8/12**			
Analysis Lead Standard Manufacturer and Lot #	**SPEX 11-116PB**			
Initial Calibration Verification Standard Source and Lot #	**SPEX 20-140JB**			
Interference Check Sample Source and Lot #	**Environmental Express 0929914 + 1119513**			
Sample Name	**Instrument result for the analytical solution (ug/L)**	**Dilution Factor**	**Final Instrumental result analytical solution (corrected for dilution) (ug/L)**	**Result in mg/Kg (corrected for 0.5g/50mL extraction)(i.e ug/L times 100/1000 = mg/kg)**
Reagent Blank	**0**		**0**	**NA**
New RM (Sample 1)	**1566**	**50**	**78303**	**7812**
New RM (Sample 2)	**1724**	**50**	**86215**	**8141**
New RM (Sample 3)	**1664**	**50**	**83192**	**8087**
New RM (Sample 4)	**1631**	**50**	**81542**	**7878**
New RM (Sample 5)	**1625**	**50**	**81256**	**7898**
Control Soil NIST SRM 2710a	**989**	**50**	**49429**	**4912**
Blank Spike	**215**	**50**	**10761**	**NA**
New RM Matrix Spike	**1742**	**50**	**87100**	**8534**

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Table 4. 3051A Digestion Spiked Blank and Spiked Sample Results for New RM: Results for Lead

Laboratory Performing Extraction	**Lab D**
Laboratory Performing Analysis	**Lab D**
3051A Digestion Spiked Blank and Spiked Sample Results for New RM: Lead	
Blank Spike Result (mg/L)	10.7
Blank Spike Percent Recovery	108
Average (5) Result New RM (mg/kg)	7963
New RM Matrix Spike Result (mg/kg)	8534
New RM Matrix Spike Percent Recovery	57

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Laboratory Submitted Study Results

Table 5. Laboratory, Instrument, Instrumental MDL, and 3051A Digestion Data Reporting Form for New RM: Results for Arsenic

Laboratory Performing Extraction	**Lab D**			
Laboratory Performing Analysis	**Lab D**			
Digestion Batch Results New RM: Arsenic				
Instrument Type? (ICP-AES or ICP-MS)	**ICP-MS**	**Instrument Method Detection Limit (MDL) (ug/L)**		**0.015**
Digestion Date	**5/7/12**			
Digestion Arsenic Standard Manufacturer and Lot #	**SPEX 11-116PB**			
Analysis Date(s)	**5/8/12**			
Analysis Arsenic Standard Manufacturer and Lot #	**SPEX 11-116PB**			
Initial Calibration Verification Standard Source and Lot #	**SPEX 20-140JB**			
Interference Check Sample Source and Lot #	**Environmental Express 0929914 + 1119513**			
Sample Name	**Instrument result for the analytical solution (ug/L)**	**Dilution Factor**	**Final Instrumental result analytical solution (corrected for dilution) (ug/L)**	**Result in mg/Kg (corrected for 0.5g/50mL extraction)(i.e ug/L times 100/1000 = mg/kg)**
Reagent Blank	**0**		**0**	**NA**
New RM (Sample 1)	**160**	**50**	**8025**	**800**
New RM (Sample 2)	**178**	**50**	**8913**	**841**
New RM (Sample 3)	**167**	**50**	**8399**	**816**
New RM (Sample 4)	**165**	**50**	**8276**	**799**
New RM (Sample 5)	**166**	**50**	**8302**	**806**
Control Soil NIST SRM 2710a	**311**	**50**	**15561**	**1546**
Blank Spike	**219**	**50**	**10963**	**NA**
New RM Matrix Spike	**381**	**50**	**19082**	**1869**

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Laboratory Submitted Study Results

Table 6. 3051A Digestion Spiked Blank and Spiked Sample Results for New RM: Results for Arsenic

Laboratory Performing Extraction	**Lab D**
Laboratory Performing Analysis	**Lab D**
3051A Digestion Spiked Blank and Spiked Sample Results for New RM: Arsenic	
Blank Spike Result (mg/L)	10.9
Blank Spike Percent Recovery	110
Average (5) Result New RM (mg/L)	813
New RM Matrix Spike Result (mg/L)	1869
New RM Matrix Spike Percent Recovery	106

Laboratory E

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(1) EPA SOP EPA 9200.2-86 (Lead IVBA)

Table 3. Laboratory, Instrument, Instrumental MDL, and IVBA Extraction Batch Data Reporting Form for New RM

Laboratory Performing Extraction	LAB E			
Laboratory Performing Analysis	LAB E			
IVBA Extraction Batch Results New RM: Lead				
Instrument Type? (ICP-AES or ICP-MS)	ICP-MS	**Instrument Method Detection Limit (MDL) (ug/L)**	0.03	
Extraction Date	05/29/12			
Extraction Lead Standard Manufacturer and Lot #	VHG – lot # 101-0037			
Analysis Date(s)	06/11/12			
Analysis Lead Standard Manufacturer and Lot #	VHG – lot # 102-0115			
Initial Calibration Verification Standard Source and Lot #	VHG – lot # 011-0103			
Interference Check Sample Source and Lot #	VHG – lot #'s 102-0114 & 104-0052			
Sample Name	**Instrument result for the analytical solution (ug/L)**	**Dilution Factor**	**Final Instrumental result analytical solution (corrected for dilution) (ug/L)**	**Result in mg/Kg (corrected for 1g/100mL extraction) (i.e ug/L times 100/1000 = mg/kg)**
EXAMPLE SOIL	**70**	**10**	**700**	**70**
Reagent Blank	0.2	9.8	1.8	NA
Bottle Blank	0.2	9.9	1.6	NA
New RM (Extractor Position 1)	506.3	97.2	49209.2	4920.9
New RM (Extractor Position 2)	497.9	97.2	48394.6	4839.5
New RM (Extractor Position 3)	490.3	98.9	48487.8	4848.8
New RM (Extractor Position 4)	500.6	97.0	48565.9	4856.6
New RM (Extractor Position 5)	494.4	97.4	48159.8	4816.0
Control Soil NIST SRM 2710a	374.5	96.5	36149.8	3615.0
Blank Spike	19.9	98.1	1956.1	NA
New RM Matrix Spike	535.4	97.3	52085.4	5208.5

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Laboratory Submitted Study Results

Table 4. Lead Extraction Batch Spiked Blank and Spiked Sample Results for New RM

Laboratory Performing Extraction	LAB E
Laboratory Performing Analysis	LAB E
IVBA Extraction Batch Spiked Blank and Spiked Sample Results for New RM: Lead	
Bottle Blank Result (mg/L)	.002
Blank Spike Result (mg/L)	1.96 (adjusted to 10 in Table) CLJ
Blank Spike Percent Recovery	98.0 %
Average (5) Result New RM (mg/L)	48.6
New RM Matrix Spike Result (mg/L)	52.1
New RM Matrix Spike Percent Recovery	103.1 %

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Laboratory Submitted Study Results

(2) SW-846 METHOD 3051A MICROWAVE ASSISTED ACID DIGESTION OF SEDIMENTS, SLUDGES, SOILS, AND OILS

Table 3. Laboratory, Instrument, Instrumental MDL, and 3051A Digestion Data Reporting Form for New RM: Results for Lead

Laboratory Performing Extraction	LAB E			
Laboratory Performing Analysis	LAB E			
Digestion Batch Results New RM: Lead				
Instrument Type? (ICP-AES or ICP-MS)	ICP-MS	**Instrument Method Detection Limit (MDL) (ug/L)**	0.03	
Digestion Date	06/07/12			
Digestion Lead Standard Manufacturer and Lot #	VHG – lot # 101-0037			
Analysis Date(s)	06/11/12			
Analysis Lead Standard Manufacturer and Lot #	VHG – lot # 102-0115			
Initial Calibration Verification Standard Source and Lot #	VHG – lot # 011-0103			
Interference Check Sample Source and Lot #	VHG – lot #'s 102-0114 & 104-0052			
Sample Name	**Instrument result for the analytical solution (ug/L)**	**Dilution Factor**	**Final Instrumental result analytical solution (corrected for dilution) (ug/L)**	**Result in mg/Kg (corrected for 0.5g/100mL extraction)(i.e ug/L times 200/1000 = mg/kg)**
EXAMPLE SOIL	**35**	**10**	**350**	**70**
Reagent Blank	0.5	9.2	4.6	NA
New RM (Sample 1)	729.9	93.0	67876.7	6787.7
New RM (Sample 2)	687.0	95.2	65428.5	6542.8
New RM (Sample 3)	715.9	93.4	66867.2	6686.7
New RM (Sample 4)	701.9	93.6	65663.4	6566.3
New RM (Sample 5)	698.0	93.6	65334.0	6533.4
Control Soil NIST SRM 2710a	496.8	91.3	45372.8	4537.3
Blank Spike	5.07	91.7	464.8	NA
New RM Matrix Spike	711.6	91.4	65066.9	6506.7

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Table 4. 3051A Digestion Spiked Blank and Spiked Sample Results for New RM: Results for Lead

Laboratory Performing Extraction	LAB E
Laboratory Performing Analysis	LAB E
3051A Digestion Spiked Blank and Spiked Sample Results for New RM: Lead	
Blank Spike Result (mg/L)	0.465
Blank Spike Percent Recovery	96.9 %
Average (5) Result New RM (mg/L)	66.2
New RM Matrix Spike Result (mg/L)	65.1
New RM Matrix Spike Percent Recovery	98.1 %

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Table 5. Laboratory, Instrument, Instrumental MDL, and 3051A Digestion Data Reporting Form for New RM: Results for Arsenic

Laboratory Performing Extraction	LAB E			
Laboratory Performing Analysis	LAB E			
Digestion Batch Results New RM: Arsenic				
Instrument Type? (ICP-AES or ICP-MS)	ICP-MS	**Instrument Method Detection Limit (MDL) (ug/L)**	0.02	
Digestion Date	06/07/12			
Digestion Arsenic Standard Manufacturer and Lot #	VHG – lot # 101-0037			
Analysis Date(s)	06/11/12			
Analysis Arsenic Standard Manufacturer and Lot #	VHG – lot # 102-0115			
Initial Calibration Verification Standard Source and Lot #	VHG – lot # 011-0103			
Interference Check Sample Source and Lot #	VHG – lot #'s 102-0114 & 104-0052			
Sample Name	**Instrument result for the analytical solution (ug/L)**	**Dilution Factor**	**Final Instrumental result analytical solution (corrected for dilution) (ug/L)**	**Result in mg/Kg (corrected for 0.5g/100mL extraction)(i.e ug/L times 200/1000 = mg/kg)**
EXAMPLE SOIL	**35**	**10**	**350**	**70**
Reagent Blank	0.035	9.2	0.3	NA
New RM (Sample 1)	69.76	93.0	6487.3	648.7
New RM (Sample 2)	68.87	95.2	6559.0	655.9
New RM (Sample 3)	69.47	93.4	6488.7	648.9
New RM (Sample 4)	69.88	93.6	6537.3	653.7
New RM (Sample 5)	71.27	93.6	6671.0	667.1
Control Soil NIST SRM 2710a	144.7	91.3	13215.4	1321.5
Blank Spike	24.98	91.7	2290.2	NA
New RM Matrix Spike	92.99	91.4	8502.8	850.3

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Table 6. 3051A Digestion Spiked Blank and Spiked Sample Results for New RM: Results for Arsenic

Laboratory Performing Extraction	LAB E
Laboratory Performing Analysis	LAB E
3051A Digestion Spiked Blank and Spiked Sample Results for New RM: Arsenic	
Blank Spike Result (mg/L)	2.3 (adjusted to 10 in table) CLJ
Blank Spike Percent Recovery	95.8 %
Average (5) Result New RM (mg/L)	6.5
New RM Matrix Spike Result (mg/L)	8.5
New RM Matrix Spike Percent Recovery	120.7%

Laboratory F

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Laboratory Submitted Study Results

(1) EPA SOP EPA 9200.2-86 (Lead IVBA)

Table 3. Laboratory, Instrument, Instrumental MDL, and IVBA Extraction Batch Data Reporting Form for New RM

Laboratory Performing Extraction	**LAB F**			
Laboratory Performing Analysis	**LAB F**			
IVBA Extraction Batch Results New RM: Lead				
Instrument Type? (ICP-AES or ICP-MS)	**AES**	**Instrument Method Detection Limit (MDL) (ug/L)**	**20**	
Extraction Date	**5/4/2012**			
Extraction Lead Standard Manufacturer and Lot #	**Lot# 17-94PB**	**SPEX Certiprep 1000 mg/L Pb Std.**		
Analysis Date(s)	**5/9/2012**			
Analysis Lead Standard Manufacturer and Lot #	**Lot# 17-94PB**		**SPEX Certiprep 1000 mg/L Pb Std.**	
Initial Calibration Verification Standard Source and Lot #	**Lot# 24-134JB**		**SPEX Certiprep LPC Std1, 20 mg/L Pb. ICV, CCV prepared by diluting Std into 0.4M glycine to match matrix**	
Interference Check Sample Source and Lot #	**Lot# 37-29AS**		**SPEX Certiprep 5000 mg/L Al, Ca, Mg; 2000 mg/L Fe prepared by x10 dilution into 0.4M glycine and spiked with 5mg/L Pb**	
Sample Name	**Instrument result for the analytical solution~~(ug/L)~~mg/L**	**Dilution Factor**	**Final Instrumental result analytical solution(corrected for dilution) ~~(ug/L)~~mg/L**	**Result in mg/Kg (corrected for 1g/100mL extraction) (i.e ug/L times 100/1000 = mg/kg)**
EXAMPLE SOIL	**70**	**10**	**700**	**70**
Reagent Blank	**- 0.00387**	**1**	**- 0.00387**	
Bottle Blank	**- 0.00249**	**1**	**- 0.00249**	
New RM (Extractor Position 1)	**46.09**	**1**	**46.09**	**4609**
New RM (Extractor Position 2)	**46.04**	**1**	**46.04**	**4604**
New RM (Extractor Position 3)	**45.49**	**1**	**45.49**	**4549**
New RM (Extractor Position 4)	**45.63**	**1**	**45.63**	**4563**
New RM (Extractor Position 5)	**45.05**	**1**	**45.05**	**4505**
Control Soil NIST SRM 2710a	**34.00**	**1**	**34.00**	**3400**
Blank Spike	**11.31**	**1**	**11.31**	**1131**
New RM Matrix Spike	**55.66**	**1**	**55.66**	**5566**

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Table 4. Lead Extraction Batch Spiked Blank and Spiked Sample Results for New RM

Laboratory Performing Extraction	LAB F
Laboratory Performing Analysis	LAB F
IVBA Extraction Batch Spiked Blank and Spiked Sample Results for New RM: Lead	
Bottle Blank Result (mg/L)	0.00249
Blank Spike Result (mg/L)	11.31
Blank Spike Percent Recovery	113%
Average (5) Result New RM (mg/L)	45.66
New RM Matrix Spike Result (mg/L)	55.66
New RM Matrix Spike Percent Recovery	100%

(2) SW-846 METHOD 3051A MICROWAVE ASSISTED ACID DIGESTION OF SEDIMENTS, SLUDGES, SOILS, AND OILS

Table 3. Laboratory, Instrument, Instrumental MDL, and 3051A Digestion Data Reporting Form for New RM: Results for Lead

Laboratory Performing Extraction	**LAB F**			
Laboratory Performing Analysis	**LAB F**			
Digestion Batch Results New RM: Lead				
Instrument Type? (ICP-AES or ICP-MS)	**AES**	**Instrument Method Detection Limit (MDL) (ug/L)**	**6.1**	
Digestion Date	**5/18/2012**			
Digestion Lead Standard Manufacturer and Lot #	**Lot# 17-94PB**	**SPEX Certiprep 1000 mg/L Pb Std.**		
Analysis Date(s)	**5/22/2012**			
Analysis Lead Standard Manufacturer and Lot #	**Lot# 17-94PB**		**SPEX Certiprep 1000 mg/L Pb Std.**	
Initial Calibration Verification Standard Source and Lot #	**Lot# 24-134JB**		**SPEX Certiprep LPC Std1, 20 mg/L Pb. ICV, CCV prepared by diluting Std to match sample matrix**	
Interference Check Sample Source and Lot #	**Lot#37-29AS**		**SPEX Certiprep 5000 mg/L Al, Ca, Mg; 2000 mg/L Fe prepared by x10 dilution into sample matrix and spike with 5 mg/L Pb (B)**	
Sample Name	**Instrument result for the analytical solution~~(ug/L)~~mg/L**	**Dilution Factor**	**Final Instrumental result analytical solution(corrected for dilution)~~(ug/L)~~mg/L**	**Result in mg/Kg (corrected for 0.5g/100mL extraction)(i.e ug/L times 200/1000 = mg/kg)**
EXAMPLE SOIL	**35**	**10**	**350**	**70**
Reagent Blank	**- 0.008072**	**10**	**- 0.08072**	
New RM (Sample 1)	**6.838**	**10**	**68.38**	**6838**
New RM (Sample 2)	**6.742**	**10**	**67.42**	**6742**
New RM (Sample 3)	**6.815**	**10**	**68.15**	**6815**
New RM (Sample 4)	**6.739**	**10**	**67.39**	**6739**
New RM (Sample 5)	**6.844**	**10**	**68.44**	**6844**
Control Soil NIST SRM 2710a	**5.491**	**10**	**54.91**	**5491**
Blank Spike	**1.041**	**10**	**10.41**	
New RM Matrix Spike	**7.561**	**10**	**75.61**	

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Table 4. 3051A Digestion Spiked Blank and Spiked Sample Results for New RM: Results for Lead

Laboratory Performing Extraction	LAB F
Laboratory Performing Analysis	LAB F
3051A Digestion Spiked Blank and Spiked Sample Results for New RM: Lead	
Blank Spike Result (mg/L)	10.41
Blank Spike Percent Recovery	104
Average (5) Result New RM (mg/L)	67.96
New RM Matrix Spike Result (mg/L)	75.61
New RM Matrix Spike Percent Recovery	76.5

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Laboratory Submitted Study Results

Table 5. Laboratory, Instrument, Instrumental MDL, and 3051A Digestion Data Reporting Form for New RM: Results for Arsenic

Laboratory Performing Extraction	**LAB F**			
Laboratory Performing Analysis	**LAB F**			
Digestion Batch Results New RM: Arsenic				
Instrument Type? (ICP-AES or ICP-MS)	**AES**	**Instrument Method Detection Limit (MDL) (ug/L)**	**1.3**	
Digestion Date	**5/18/2012**			
Digestion Arsenic Standard Manufacturer and Lot #	**Lot# 16-97AS**	**SPEX Certiprep 1000 mg/L Pb Std.**		
Analysis Date(s)	**5/22/2012**			
Analysis Arsenic Standard Manufacturer and Lot #	**Lot# 16-97AS**		**SPEX Certiprep 1000 mg/L As Std.**	
Initial Calibration Verification Standard Source and Lot #	**Lot# 24-134JB**		**SPEX Certiprep LPC Std1, 20 mg/L As. ICV, CCV prepared by diluting Std to match sample matrix**	
Interference Check Sample Source and Lot #	**Lot#37-29AS**		**SPEX Certiprep 5000 mg/L Al, Ca, Mg; 2000 mg/L Fe prepared by x10 dilution into sample matrix (A) and spike with 5 mg/L As (B)**	
Sample Name	**Instrument result for the analytical solution~~(ug/L)~~mg/L**	**Dilution Factor**	**Final Instrumental result analytical solution(corrected for dilution) ~~(ug/L)~~ mg/L**	**Result in mg/Kg (corrected for 0.5g/100mL extraction)(i.e ug/L times 200/1000 = mg/kg)**
EXAMPLE SOIL	**35**	**10**	**350**	**70**
Reagent Blank	**- 0.00009013**	**10**	**- 0.0009013**	
New RM (Sample 1)	**0.7760**	**10**	**7.760**	**776.0**
New RM (Sample 2)	**0.7890**	**10**	**7.890**	**789.0**
New RM (Sample 3)	**0.7695**	**10**	**7.695**	**769.5**
New RM (Sample 4)	**0.7648**	**10**	**7.648**	**764.8**
New RM (Sample 5)	**0.7977**	**10**	**7.977**	**797.7**
Control Soil NIST SRM 2710a	**1.684**	**10**	**16.84**	**168.4**
Blank Spike	**1.019**	**10**	**10.19**	
New RM Matrix Spike	**1.758**	**10**	**17.58**	

Table 6. 3051A Digestion Spiked Blank and Spiked Sample Results for New RM: Results for Arsenic

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Laboratory Performing Extraction	LAB F
Laboratory Performing Analysis	LAB F
3051A Digestion Spiked Blank and Spiked Sample Results for New RM: Arsenic	
Blank Spike Result (mg/L)	10.19
Blank Spike Percent Recovery	102
Average (5) Result New RM (mg/L)	7.794
New RM Matrix Spike Result (mg/L)	17.58
New RM Matrix Spike Percent Recovery	97.9

Laboratory G

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Laboratory Submitted Study Results

(1) EPA SOP EPA 9200.2-86 (Lead IVBA)

Table 3. Laboratory, Instrument, Instrumental MDL, and IVBA Extraction Batch Data Reporting Form for New RM

Laboratory Performing Extraction	Lab G			
Laboratory Performing Analysis	Lab G			
IVBA Extraction Batch Results New RM: Lead				
Instrument Type? (ICP-AES or ICP-MS)	ICP - AES	**Instrument Method Detection Limit (MDL) (ug/L)**	10.64 ug/L	
Extraction Date	4/23/2012			
Extraction Lead Standard Manufacturer and Lot #	Claritas Lot # 9-145CR			
Analysis Date(s)	4/24/2012			
Analysis Lead Standard Manufacturer and Lot #	CLaritas Lot # 9-145CR			
Initial Calibration Verification Standard Source and Lot #	Absolute Lot # 101110			
Interference Check Sample Source and Lot #	QATS Lot # 0503 & 0203			
Sample Name	**Instrument result for the analytical solution (ug/L)**	**Dilution Factor**	**Final Instrumental result analytical solution (corrected for dilution) (ug/L)**	**Result in mg/Kg (corrected for 1g/100mL extraction) (i.e ug/L times 100/1000 = mg/kg)**
EXAMPLE SOIL	**70**	**10**	**700**	**70**
Reagent Blank	<0			<0
Bottle Blank	<0			<0
New RM (Extractor Position 1)	45380			4538
New RM (Extractor Position 2)	44340			4434
New RM (Extractor Position 3)	45840			4584
New RM (Extractor Position 4)	45890			4589
New RM (Extractor Position 5)	46260			4626
Control Soil NIST SRM 2710a	33930			3393
Blank Spike	10180			1018
New RM Matrix Spike	57670			5767

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Table 4. Lead Extraction Batch Spiked Blank and Spiked Sample Results for New RM

Laboratory Performing Extraction	Lab G
Laboratory Performing Analysis	Lab G
IVBA Extraction Batch Spiked Blank and Spiked Sample Results for New RM: Lead	
Bottle Blank Result (mg/L)	<0
Blank Spike Result (mg/L)	10.2 mg/L
Blank Spike Percent Recovery	102%
Average (5) Result New RM (mg/L)	45.5 mg/L
New RM Matrix Spike Result (mg/L)	57.7 mg/L
New RM Matrix Spike Percent Recovery	122%

(2) SW-846 METHOD 3051A MICROWAVE ASSISTED ACID DIGESTION OF SEDIMENTS, SLUDGES, SOILS, AND OILS

Table 3. Laboratory, Instrument, Instrumental MDL, and 3051A Digestion Data Reporting Form for New RM: Results for Lead

Laboratory Performing Extraction	Lab G			
Laboratory Performing Analysis	Lab G			
Digestion Batch Results New RM: Lead				
Instrument Type? (ICP-AES or ICP-MS)	ICP-AES	**Instrument Method Detection Limit (MDL) (ug/L)**		10.64 ug/L
Digestion Date	4/23/2012			
Digestion Lead Standard Manufacturer and Lot #	Claritas Lot # 9-145CR			
Analysis Date(s)	4/24/2012			
Analysis Lead Standard Manufacturer and Lot #	CLaritas Lot # 9-145CR			
Initial Calibration Verification Standard Source and Lot #	Absolute Lot # 101110			
Interference Check Sample Source and Lot #	QATS Lot # 0503 & 0203			
Sample Name	**Instrument result for the analytical solution (ug/L)**	**Dilution Factor**	**Final Instrumental result analytical solution (corrected for dilution) (ug/L)**	**Result in mg/Kg (corrected for 1.0g/50mL extraction)(i.e ug/L times 50/1000 = mg/kg)**
EXAMPLE SOIL	**35**	**10**	**350**	**70**
Reagent Blank	<0			<0
New RM (Sample 1)	133400			6670
New RM (Sample 2)	129400			6470
New RM (Sample 3)	132100			6605
New RM (Sample 4)	133400			6670
New RM (Sample 5)	132600			6630
Control Soil NIST SRM 2710a	103900			5195
Blank Spike	10120			506
New RM Matrix Spike	140000			7000

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Laboratory Submitted Study Results

Table 4. 3051A Digestion Spiked Blank and Spiked Sample Results for New RM: Results for Lead

Laboratory Performing Extraction	Lab G
Laboratory Performing Analysis	Lab G
3051A Digestion Spiked Blank and Spiked Sample Results for New RM: Lead	
Blank Spike Result (mg/L)	10.1 mg/L
Blank Spike Percent Recovery	101%
Average (5) Result New RM (mg/L)	132 mg/L
New RM Matrix Spike Result (mg/L)	140 mg/L
New RM Matrix Spike Percent Recovery	80%

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Laboratory Submitted Study Results

Table 5. Laboratory, Instrument, Instrumental MDL, and 3051A Digestion Data Reporting Form for New RM: Results for Arsenic

Laboratory Performing Extraction	Lab G			
Laboratory Performing Analysis	Lab G			
Digestion Batch Results New RM: Arsenic				
Instrument Type? (ICP-AES or ICP-MS)	ICP - AES	**Instrument Method Detection Limit (MDL) (ug/L)**	9.09 ug/L	
Digestion Date	4/23/2012			
Digestion Arsenic Standard Manufacturer and Lot #	Claritas Lot # 9-145CR			
Analysis Date(s)	4/24/2012			
Analysis Arsenic Standard Manufacturer and Lot #	Claritas Lot # 9-145CR			
Initial Calibration Verification Standard Source and Lot #	Absolute Lot # 101110			
Interference Check Sample Source and Lot #	QATS Lot # 0503 & 0203			
Sample Name	**Instrument result for the analytical solution (ug/L)**	**Dilution Factor**	**Final Instrumental result analytical solution (corrected for dilution) (ug/L)**	**Result in mg/Kg (corrected for 1g/50mL extraction)(i.e ug/L times 50/1000 = mg/kg)**
EXAMPLE SOIL	**35**	**10**	**350**	**70**
Reagent Blank	<0			<0
New RM (Sample 1)	14290			714.5
New RM (Sample 2)	13990			699.5
New RM (Sample 3)	14240			712.0
New RM (Sample 4)	14110			705.5
New RM (Sample 5)	15040			752.0
Control Soil NIST SRM 2710a	30100			1505
Blank Spike	9990			499.5
New RM Matrix Spike	24280			1214

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Laboratory Submitted Study Results

Table 6. 3051A Digestion Spiked Blank and Spiked Sample Results for New RM: Results for Arsenic

Laboratory Performing Extraction	Lab G
Laboratory Performing Analysis	Lab G
3051A Digestion Spiked Blank and Spiked Sample Results for New RM: Arsenic	
Blank Spike Result (mg/L)	9.99 mg/L
Blank Spike Percent Recovery	99.9%
Average (5) Result New RM (mg/L)	14.3 mg/L
New RM Matrix Spike Result (mg/L)	24.3 mg/L
New RM Matrix Spike Percent Recovery	100%

Laboratory H

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Laboratory Submitted Study Results

(1) EPA SOP EPA 9200.2-86 (Lead IVBA)

Table 3. Laboratory, Instrument, Instrumental MDL, and IVBA Extraction Batch Data Reporting Form for New RM

Laboratory Performing Extraction	**Lab H**			
Laboratory Performing Analysis	**Lab H**			
IVBA Extraction Batch Results New RM: Lead				
Instrument Type? (ICP-AES or ICP-MS)	**ICP-AES**	**Instrument Method Detection Limit (MDL) (ug/L)**	**4.0**	
Extraction Date	**5/14/2012**			
Extraction Lead Standard Manufacturer and Lot #	**Inorganic Ventures CGPB1-1**			
Analysis Date(s)	**5/15/2012**			
Analysis Lead Standard Manufacturer and Lot #	**Inorganic Ventures CGPB1-1**			
Initial Calibration Verification Standard Source and Lot #	**Spex 43-47AS**			
Interference Check Sample Source and Lot #	**Inorganic Ventures E2-MEB348035 and E2-MEB399019**			
Sample Name	**Instrument result for the analytical solution (ug/L)**	**Dilution Factor**	**Final Instrumental result analytical solution (corrected for dilution) (ug/L)**	**Result in mg/Kg (corrected for 1g/100mL extraction) (i.e ug/L times 100/1000 = mg/kg)**
EXAMPLE SOIL	**70**	**10**	**700**	**70**
Reagent Blank	**4.51**	**1**	**4.51**	**0.451**
Bottle Blank	**4.13**	**1**	**4.13**	**0.413**
New RM (Extractor Position 1)	**44210**	**1**	**44210**	**4314**
New RM (Extractor Position 2)	**43580**	**1**	**43580**	**4285**
New RM (Extractor Position 3)	**43000**	**1**	**43000**	**4267**
New RM (Extractor Position 4)	**45000**	**1**	**45000**	**4393**
New RM (Extractor Position 5)	**43790**	**1**	**43790**	**4310**
Control Soil NIST SRM 2710a	**33860**	**1**	**33860**	**3332**
Blank Spike	**9835**	**1**	**9835**	**983.5**
New RM Matrix Spike	**53240**	**1**	**53240**	**5218**

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Table 4. Lead Extraction Batch Spiked Blank and Spiked Sample Results for New RM

Laboratory Performing Extraction	Lab H
Laboratory Performing Analysis	Lab H
IVBA Extraction Batch Spiked Blank and Spiked Sample Results for New RM: Lead	
Bottle Blank Result (mg/L)	0.00413
Blank Spike Result (mg/L)	9.835
Blank Spike Percent Recovery	98.35
Average (5) Result New RM (mg/L)	43.91
New RM Matrix Spike Result (mg/L)	53.24
New RM Matrix Spike Percent Recovery	92.2

(2) SW-846 METHOD 3051A MICROWAVE ASSISTED ACID DIGESTION OF SEDIMENTS, SLUDGES, SOILS, AND OILS

Table 3. Laboratory, Instrument, Instrumental MDL, and 3051A Digestion Data Reporting Form for New RM: Results for Lead

Laboratory Performing Extraction	**Lab H**			
Laboratory Performing Analysis	**Lab H**			
Digestion Batch Results New RM: Lead				
Instrument Type? (ICP-AES or ICP-MS)	**ICP-AES**	**Instrument Method Detection Limit (MDL) (ug/L)**		**1.5 ug/L (0.30 mg/kg)**
Digestion Date	**5/7/2012**			
Digestion Lead Standard Manufacturer and Lot #	**Inorganic Ventures CGPB1-1**			
Analysis Date(s)	**5/15/2012**			
Analysis Lead Standard Manufacturer and Lot #	**Inorganic Ventures CGPB1-1**			
Initial Calibration Verification Standard Source and Lot #	**Spex 43-47AS**			
Interference Check Sample Source and Lot #	**Inorganic Ventures E2-MEB348035 and E2-MEB399019**			
Sample Name	**Instrument result for the analytical solution (ug/L)**	**Dilution Factor**	**Final Instrumental result analytical solution (corrected for dilution) (ug/L)**	**Result in mg/Kg (corrected for 0.5g/100mL extraction)(i.e ug/L times 200/1000 = mg/kg)**
EXAMPLE SOIL	**35**	**10**	**350**	**70**
Reagent Blank	**-1.88**	**2**	**-3.76**	**-0.376**
New RM (Sample 1)	**31280**	**2**	**62560**	**6246**
New RM (Sample 2)	**32580**	**2**	**65160**	**6513**
New RM (Sample 3)	**32310**	**2**	**64620**	**6471**
New RM (Sample 4)	**32750**	**2**	**65500**	**6538**
New RM (Sample 5)	**33690**	**2**	**67380**	**6737**
Control Soil NIST SRM 2710a	**25910**	**2**	**51820**	**5181**
Blank Spike	**5237**	**2**	**10474**	**1047**
New RM Matrix Spike	**36600**	**2**	**73200**	**7313**

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Laboratory Submitted Study Results

Table 4. 3051A Digestion Spiked Blank and Spiked Sample Results for New RM: Results for Lead

Laboratory Performing Extraction	**Lab H**
Laboratory Performing Analysis	**Lab H**
3051A Digestion Spiked Blank and Spiked Sample Results for New RM: Lead	
Blank Spike Result (mg/L)	10.474
Blank Spike Percent Recovery	105
Average (5) Result New RM (mg/L)	65.044
New RM Matrix Spike Result (mg/L)	73.2
New RM Matrix Spike Percent Recovery	81.2

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Laboratory Submitted Study Results

Table 5. Laboratory, Instrument, Instrumental MDL, and 3051A Digestion Data Reporting Form for New RM: Results for Arsenic

Laboratory Performing Extraction	**Lab H**			
Laboratory Performing Analysis	**Lab H**			
Digestion Batch Results New RM: Arsenic				
Instrument Type? (ICP-AES or ICP-MS)		**Instrument Method Detection Limit (MDL) (ug/L)**		**2.35 (0.47 mg/kg)**
Digestion Date	**5/7/2012**			
Digestion Arsenic Standard Manufacturer and Lot #	**Inorganic Ventures CGAS1-1**			
Analysis Date(s)	**5/15/2012**			
Analysis Arsenic Standard Manufacturer and Lot #	**Inorganic Ventures CGAS1-1**			
Initial Calibration Verification Standard Source and Lot #	**Spex 43-47AS**			
Interference Check Sample Source and Lot #	**Inorganic Ventures E2-MEB348035 and E2-MEB399019**			
Sample Name	**Instrument result for the analytical solution (ug/L)**	**Dilution Factor**	**Final Instrumental result analytical solution (corrected for dilution) (ug/L)**	**Result in mg/Kg (corrected for 0.5g/100mL extraction)(i.e ug/L times 200/1000 = mg/kg)**
EXAMPLE SOIL	**35**	**10**	**350**	**70**
Reagent Blank	**-0.12**	**2**	**-0.24**	**-0.024**
New RM (Sample 1)	**3744**	**2**	**7488**	**747.6**
New RM (Sample 2)	**3654**	**2**	**7308**	**730.5**
New RM (Sample 3)	**3706**	**2**	**7412**	**742.2**
New RM (Sample 4)	**3957**	**2**	**7914**	**790.0**
New RM (Sample 5)	**3841**	**2**	**7682**	**768.0**
Control Soil NIST SRM 2710a	**7886**	**2**	**15772**	**1577**
Blank Spike	**5193**	**2**	**10386**	**1039**
New RM Matrix Spike	**8638**	**2**	**17276**	**1726**

Table 6. 3051A Digestion Spiked Blank and Spiked Sample Results for New RM: Results for Arsenic

Laboratory Performing Extraction	Lab H
Laboratory Performing Analysis	Lab H
3051A Digestion Spiked Blank and Spiked Sample Results for New RM: Arsenic	
Blank Spike Result (mg/L)	10.386
Blank Spike Percent Recovery	104
Average (5) Result New RM (mg/L)	7.561
New RM Matrix Spike Result (mg/L)	17.276
New RM Matrix Spike Percent Recovery	97.1