FINAL REPORT

TASK ORDER 1026

IN VITRO BIOACCESSIBILITY REFERENCE MATERIALS FOR LEAD and ARSENIC ROUND ROBIN STUDY REPORT

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Statistical Analysis of Round Robin Results

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TABLE OF CONTENTS

LIST OF APPENDICES

ACRONYMS AND ABBREVIATIONS

ANOVA	Analysis of Variance
CI	Confidence Interval
CLP	USEPA Superfund Contract Laboratory Program
COA	Certificate of Analysis
df	Degrees of Freedom
F	Value Calculated
F-Crit	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
LCS	Laboratory Control Sample
MS	Mean Square
NERL	National Exposure Research Laboratory
NIST	National Institute of Standards and Technology
PI	Prediction Interval
PT	Performance Testing
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
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

FINAL REPORT
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IN VITRO BIOACCESSIBILITY REFERENCE MATERIALS FOR ARSENIC ROUND ROBIN STUDY REPORT

I. SUMMARY

The Bioavailability Committee of the USEPA Technical Review Workgroup for Metals and Asbestos (http://epa.gov/superfund/bioavailability/trw.htm) "TRW Bioavailability Committee" conducted a round robin Study (herein referred to as the Study) of EPA *In Vitro* Bioaccessibility Assay (IVBA) for arsenic and lead (EPA Method 1340), and EPA Methods 3050B/3051A for lead and arsenic, in the Flat Creek Soil Reference Material (FCRM) and in the NIST SRM 2710a.

Objectives:

- To derive a mean consensus IVBA value for arsenic in the NIST SRM 2710a and the FCRM < 250 µm and < 74 µm, and a consensus lead IVBA value in the FCRM < 74 µm.

- To report the total amount of arsenic in the FCRM < 250 µm and < 74 µm, and total lead in the FCRM < 74 µm, as measured by EPA Methods 3050B or 3051A.

This Study included the participation of seven (7) laboratories, each reporting five (5) replicate analysis results for arsenic in the NIST SRM 2710a and the FCRM < 250 µm and < 74 µm, and for arsenic and lead in the FCRM < 74 µm, which yielded a total of thirty-five (35) results for each analysis on each reference material. Note: NIST SRM 2710a was also used as the Control Soil for the IVBA arsenic extractions, which were added to the replicate analysis results for each laboratory. Therefore, there are a total of six (6) IVBA arsenic extraction results (42 total results before processing) from each laboratory for this RM.

The EPA "Standard Operating Procedure for an *In Vitro* Bioaccessibility Assay" (Method 1340, US EPA, 2017), and EPA Methods 3050B/3051A, as well as the Scope of Work to be performed, were provided to the participating laboratories. The results were statistically evaluated for IVBA of arsenic and lead, and total arsenic and lead, to derive the final consensus values provided in Table 1. Table 2 shows the FCRM (<250 µm) results from a previous study (US EPA, 2012).

II. INTRODUCTION

Utilization of IVBA assays, as estimators of the bioaccessibility and bioavailability of lead in soil, have 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 (US EPA, 2007). A similar comparison was done for arsenic in 2017 and showed a high correlation between the two assays (US EPA, 2017). 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 three (3) SRMs; FCRM < 74 µm, FCRM < 250 µm, and NIST SRM 2710a. The objective of this Study is twofold:

(1) Derive a mean consensus IVBA value for arsenic in the NIST SRM 2710a and the FCRM < 250 µm and < 74 µm, and a consensus lead IVBA value in the FCRM < 74 µm, using EPA Method 1340 (OLEM 9200.2-164, updated to include arsenic).

(2) Determine total amount of arsenic in the FCRM < 250 µm and FCRM < 74 µm, and total amount of lead in the FCRM < 74 µm, as measured by EPA Methods 3050B or 3051A.

Table 1. FCRM and NIST SRM 2710a Summary of Results (Rounded Values)				
Arsenic NIST SRM 2710a[1]	**Low 99% PI**	**Mean**	**High 99% PI**	**RSD**
IVBA Extracted (mg/Kg)	379	552	725	11.2%
IVBA (%)	24.4	38.4	52.5	13.4%
Arsenic FCRM < 250 µm[2]	**Low 99% PI**	**Mean**	**High 99% PI**	**RSD**
IVBA Extracted (mg/Kg)	113	154	195	9.0%
3050B/3051A (mg/Kg)	606	704	803	5.0%
IVBA (%)	15.8	21.9	28.0	10.3%
Arsenic FCRM < 74 µm[3]	**Low 99% PI**	**Mean**	**High 99% PI**	**RSD**
IVBA Extracted (mg/Kg)	92.8	143	193	12.5%
3050B/3051A (mg/Kg)	642	710	778	3.5%
IVBA (%)	13.1	20.1	27.1	13.0%
Lead FCRM < 74 µm[4]	**Low 99% PI**	**Mean**	**High 99% PI**	**RSD**
IVBA Extracted (mg/Kg)	4290	4770	5240	3.5%
3050B/3051A (mg/Kg)	5620	6370	7120	4.3%
IVBA (%)	63.8	74.8	85.9	5.5%

Table 2. FCRM <250 µm Summary of Lead Results, previous study (Rounded Values)				
Arsenic FCRM < 250 µm	**Low 99% PI**	**Mean**	**High 99% PI**	**RSD**
IVBA Extracted (mg/Kg)	3990	4620	5250	4.9%
3050B/3051A (mg/Kg)	5490	6440	7400	5.4%
IVBA (%)	57.8	71.7	85.6	7.3%

This report provides the data and statistical analysis of the arsenic and lead results from the Study, conducted by the United States Environmental Protection Agency (US EPA), which validates their use as additional soil reference materials for EPA Method 1340 and EPA Methods 3050B/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 TRW Bioavailability Committee initiated the task of verification of the arsenic IVBA values for the NIST SRM 2710a and the new FCRM in August, 2016. This Study was coordinated, evaluated, and reported by APTIM Federal Services, LLC under its USEPA Quality Assurance Technical Support (QATS) Contract. 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 seven (7) laboratories participating in the Study was requested to analyze each of the three (3) reference materials in five (5) replicate analyses, along with the EPA Method 1340-required QC samples, including blanks, spike samples, and control soils.

[1] Results from two laboratories are excluded. See table 7 and discussion in section V.C.1.1.
[2] Results from three laboratories are excluded. See table 9 and discussion in section V.C.1.2.
[3] Results from one laboratory are excluded. See table 11 and discussion in section V.C.1.3.
[4] Results from two laboratories are excluded. See table 13 and discussion in section V.C.2.1.

III. BACKGROUND

Bioavailability can be a critical factor in determining the potential uptake of contaminants by receptors and an important consideration in determining potential threats to human health that may be posed by contaminated sites. The utilization of IVBA methods as estimators 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, and has a relatively quick turnaround time. A comparison of the *in vivo* and *in vitro* methods is presented in USEPA OSWER 9285.7-77 (US EPA, 2007). The TRW recently updated the EPA Method 1340 (lead only) by including the new analyte, arsenic (US EPA, 2017).

Three (3) methods were employed in the performance of this Study: 1) EPA Method 1340 (IVBA) for arsenic and lead determination, 2) EPA Method 3050B for the determination of total arsenic and lead using heated block digesters, and 3) EPA Method 3051A for microwave digestion for the determination of total arsenic and total lead. These methods are summarized below.

III. A. EPA Method 1340 (SOP 9200.2-164)

EPA Method 1340 (SOP 9200.2-164) was the method used for the determination of arsenic and lead IVBA results for the Study samples. Throughout this report, the term "IVBA" is used synonymously with EPA Method 1340. 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 soil particles that are less than 150 µm in diameter[5]. 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, are added. The sample bottle(s), and associated quality control sample bottles, are then placed on a rotary extractor (30 RPM ± 2 RPM) for one (1) hour while being heated at a constant temperature of 37º C ± 2º C. 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 is removed using an in-line filter and a 20-mL syringe. This filtered, 20-mL volume of supernatant is then analyzed for lead and arsenic (or other analytes) by ICP-AES or ICP-MS.

The IVBA value for the RMs is expressed as the ratio of the IVBA result divided by the EPA Method 3050B or 3051A total analyte result, multiplied by 100.

$$IVBA\ (\%) = \frac{Method\ 1340\ Result}{Method\ 3050B\ or\ 3051A\ Result} \times 100$$

III. B. EPA Methods 3050B/3051A

EPA Method 3050B was used as the sample preparation method for the determination of total lead and total arsenic concentrations in the Study samples by some of the laboratories. Using EPA Method 3050B, solid samples are digested in 1:1 nitric acid using a heated block digester as the heat source. The sample is covered and heated to 95º ± 5º C for ten (10) to fifteen (15) minutes, without boiling, and the sample is allowed to cool. Concentrated nitric acid is added to the sample, the beaker is covered with a watch glass, and the sample is refluxed at 95º ± 5º C for thirty (30) minutes. If brown fumes are observed, which is an indication of oxidation of the sample by nitric acid, repeated addition of concentrated nitric acid and refluxing at 95º ± 5º C is required. When no additional brown fumes are observed, the sample is evaporated to the prescribed volume.

[5] This is the recommended sieve size for soil samples. The grain size of the reference materials are independent of the method.

Additional heating should not continue for more than two (2) hours, and the sample should not be allowed to go to dryness. Water and 30% hydrogen peroxide are then added to the sample, the sample is heated until effervescence subsides, and the sample is cooled. Aliquots of 30% hydrogen peroxide are added to the sample, with warming, until sample change is minimal, being careful not to add more than 10 mL of hydrogen peroxide. The sample is then heated at 95º ± 5º C until the prescribed reduced volume is achieved. For ICP-MS analysis, after cooling, the sample is diluted to the prescribed volume with water, and the particulates are removed by filtration, centrifugation, or settling. The sample is now ready for ICP-MS analysis by EPA SW-846 Method 6020A. For ICP-AES analysis, prior to the dilution step above, concentrated hydrochloric acid is added to the digest, and the sample is covered and refluxed at 95º ± 5º C for fifteen (15) minutes. After cooling, the sample is filtered and diluted to the prescribed volume. The sample is now ready for ICP-AES analysis by EPA SW-846 Method 6010C.

EPA Method 3050B typically specifies the use of a one (1) gram sample, calculated on a dry weight basis, and prescribes the specific volumes of reagents and final dilution volume used to process the sample. The laboratories in this Study were instructed to use a 0.5 ± 0.0001 gram sample, and adjust to one-half the prescribed volumes of reagents and final dilution volume used to process the sample.

EPA Method 3051A was used for the determination of total lead and total arsenic concentrations in the Study samples by some of the laboratories. Using EPA Method 3051A, solid samples are digested in concentrated nitric acid or, alternatively, concentrated nitric acid and concentrated hydrochloric acid using microwave heating within a suitable laboratory microwave unit. The addition of concentrated hydrochloric acid to concentrated nitric acid is expected to enhance the solubility and/or stabilization of certain analytes in solution; however, the addition of hydrochloric acid is not expected to improve the digestion of lead or arsenic in the Study samples. In this Study, 0.5 ± 0.0001 grams of sample, and either 10 ± 0.1 mL of concentrated nitric acid, or 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 a power setting(s) that causes the mixture within the vessel to rise to a temperature of 175º ± 5º C in approximately 5.5 minutes ± 0.25 minutes, and remain at 175º ± 5º C for 4.5 minutes, or for the remainder of the ten (10) minute digestion period. After cooling, the vessel contents were 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).

The EPA Method 1340 and EPA Methods 3050B/3051A and can be accessed using the following USEPA website hyperlinks:

https://semspub.epa.gov/src/document/HQ/100000153.pdf

https://www.epa.gov/homeland-security-research/epa-Method-3050b-acid-digestion-sediments-sludges-and-soils

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

IV. TECHNICAL APPROACH

APTIM Federal Services, LLC 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 for EPA IVBA Method 1340 and EPA Method 3050B/3051A, if they had not already done so in a previous Lead IVBA Round Robin Study;

- TRW Bioavailability Committee review of the completed IDP forms and selection of laboratories to participate in the Study;

- Round Robin Study design;

- Development of a Statement of Work (SOW), including IVBA extraction and Method 3050B/3051A 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, QATS Program personnel first contacted laboratories with previous IVBA experience. Five (5) of the nine (9) laboratories initially contacted for potential participation in this Study were participants in a previous Lead IVBA Round Robin Study conducted by the TRW Bioavailability Committee and coordinated by QATS Program personnel, which was completed in 2011. The laboratories were required 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. From previous participation, and the IDP form response, the TRW Bioavailability Committee selected a total of seven (7) laboratories for participation in the Study, which are presented in Table 3. In order to maintain the anonymity of the Study participants, the IDP forms provided by the laboratories are presented in Appendix K in redacted format, with a letter assigned to each laboratory as an identifier, in an order inconsistent with the order presented in Table 3. The IDP forms, without redaction, are available to the USEPA Headquarters (HQ) Co-Chair for the Technical Review Workgroup (TRW), https://www.epa.gov/superfund/soil-bioavailability-superfund-sites-technical-assistance.

IV.B. Study Design

IV.B.1. FCRM and Number of Replicates

The FCRM used in this Study was provided to the QATS Laboratory for sub-aliquoting and shipment in April 2017 by USEPA National Exposure Research Laboratory (NERL) personnel, who had previously received this material from the USGS Associate Project Chief. The FCRM used in this Study consists of two (2) batches of two (2) particle sizes, less than 250 µm, and less than 74 µm.

Sufficient FCRM of both the < 250 µm and < 74 µm materials were mixed in separate containers 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, in part, by the NIST Analytical Chemistry Division, from a previous Study conducted at the QATS Laboratory in 2008, and was supplemented with more NIST SRM 2710a purchased from NIST for this Study. A total of 260 grams of NIST SRM 2710a was combined and mixed before sub-aliquots were bottled for distribution to the laboratories.

Table 3. Laboratories Selected to Participate in the Study			
Laboratory		**Address**	**Contact Name and e-mail Address**
1	USEPA National Exposure Research Laboratory (NERL), Research Triangle Park (RTP)	109 T.W. Alexander Drive Research Triangle Park, NC 27709	Dr. Karen Bradham (bradham.karen@epa.gov)
2	USEPA Region 10, Manchester Environmental Laboratory	7411 Beach Drive East Port Orchard, WA 98366	Ms. Katie Adams (adams.katie@epa.gov)
3	USEPA Region 9 Laboratory	1337 South 46th Street Building 201 Richmond, CA 94804	Mr. Richard Bauer (bauer.richard@epa.gov)
4	USEPA Region 7 Laboratory	300 Minnesota Avenue Kansas City, KS 66101	Margie St. Germain (stgermain.margie@epa.gov) Anthony Tata (tata.anthony@epa.gov)
5	ACZ Laboratories	2773 Downhill Drive Steamboat Springs, CO 80487	Mr. Tim Van Wyngarden (timv@acz.com) Ms. Sue Webber (suew@acz.com)
6	PRIMA Environmental	5070 Robert J Mathews Pkwy, Suite 300 El Dorado Hills, CA 95762	Dr. Cindy Schreier (cschreier@primaenvironmental.com)
7	Future Industries Institute, University of South Australia	Future Industries Institute, University of South Australia, Building X, X1-17, Mawson Lakes Campus, Mawson Lakes, SA 5095, ipc MLK-40, p GPO Box 2471, Adelaide, SA 5001	Dr. Albert Juhasz (albert.juhasz@unisa.edu.au)

The moisture content of the FCRM was < 0.5%, and was determined by heating a five (5)-gram sample in a drying 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 J. Table 4 provides the lead and arsenic concentrations, particle size, and moisture content for NIST SRM 2710a, derived from the Certificate of Analysis.

Table 4. 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 TRW Bioavailability Committee determined that five (5) replicate analyses of each of the NIST SRM 2710a and FCRM < 250 µm and < 74 µm 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 IVBA mean value and control limits for the Study RMs.

IV.B.2. QC Samples

In this Study, the laboratories were instructed to analyze the samples in strict accordance with the EPA Method 1340 and IVBA Study SOW, including all of the associated quality control samples, with noted exceptions. Table 5 below provides the EPA Method 1340-required and IVBA Study SOW QC samples and associated control limits used in this Study. The Study QC sample results, associated with the various IVBA extractions or Methods 3050B/3051A digestions, are presented in the appendices in Tables A-2, B-2, C-2, D-2, E-2, F-2, and G-2. The QC results evaluated in this Study included blanks, blank spike recoveries, the individual RM matrix spike recoveries, and the NIST SRM 2710a control sample recoveries. The acceptance range criteria are listed in the tables as percent recovery, concentration range, or IVBA value range, as appropriate.

NIST SRM 2710a was used as the control soil for both the arsenic and lead IVBA and EPA Method 3050B/3051A portions of the Study, followed by analysis. Both of the arsenic and lead leachable mean values and ranges 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 RMs 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, nor was the duplicate sample precision requirement evaluated as part of this Study. The laboratories were instructed to perform the analysis of one (1) set of QC samples with each RM batch for IVBA 9200.2-164 and EPA Methods 3050B (heated block digester) or 3051A (microwave digestion). The QC sample results presented in the appendices are presented “as received” from the laboratories, using the values as rounded by the laboratories.

IV.C. Statement of Work for the Study

An SOW was developed by QATS personnel and the TRW Bioavailability Committee 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 IVBA batch, a recommended autosampler analytical sequence for the instrumental analysis of the IVBA samples, as well as reporting forms for submitting the RM sample analyses and QC sample results. The SOW also provided a list of the required associated QC sample analyses and QC sample control limits derived from the EPA Method 1340. The first laboratory reporting for this Study in Appendix L has been coded as Laboratory A, and the entire SOW is presented for this first laboratory only.

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

The Study samples were shipped to the seven (7) participating laboratories in April, 2017. The laboratories were provided a sixty (60)-day turnaround time for submitting the sample results. The Study sample shipments also included a hardcopy listing of the shipment box contents. Laboratory participants were e-mailed the SOW, the Methods, and the SOPs. A Group teleconference call between EPA TRW committee members, participating laboratory personnel, and QATS Program personnel was held the week prior to the initiation of the Round Robin Study.

Table 5. EPA Method 1340-Required and IVBA Study SOW QC Samples and Control Limits	
QC Sample	**Control Limits**
Reagent Blank	< 25 µg/L Pb and As
Method Blank	< 50 µg/L Pb and As
LCS Method Blank Spike (10 mg/L)	85% -115% Recovery Pb and As
Matrix Spike (10 mg/L)	75% -125% Recovery Pb and As
Duplicate Sample	± 20% RPD Pb and As
Control Soil (NIST SRM 2710a and 2711a; FCRM)	Pb NIST 2710a: IVBA Mean = 67.5% (Acceptable IVBA Range 60.7% - 74.2%)
	Pb NIST 2711a: IVBA Mean = 85.7% (Acceptable IVBA Range: 75.2% - 96.2%)
	Pb FCRM: IVBA Mean = 71.7% (Acceptable IVBA Range: 57.8% - 85.6%)
	As NIST 2710a: IVBA Mean = 40.8% (Acceptable IVBA Range: 33.1% - 48.4%)
	As NIST 2710a: Leachable Mean = 1400 mg/Kg (3050B/3051A Concentration Acceptable Range: 90% - 110%; 1260 – 1540 mg/Kg)
	Pb NIST 2710a: Leachable Mean = 5100 mg/Kg) (3050B/3051A Concentration Acceptable Range: 90% - 110%; 4590 – 5610 mg/Kg)

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.

Outlier testing was performed using the Grubbs' test for outliers, which was applied to each full data set, and any outliers identified were removed prior to subsequent statistical calculations.

The statistical tool ANOVA, single factor (either arsenic or lead), was used to discern the intralaboratory versus the interlaboratory sources of variance of each RM data set derived from the Study. ANOVA, single factor performs a simple analysis of variance on data for two (2) or more sample sets. The analysis provides a test of the hypothesis that each sample is drawn from the same underlying probability distribution against the alternative hypothesis that underlying probability distributions are not the same for all samples. The variance uses the null hypothesis in which 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 (2)-tail test, which means that both the upper and the lower ends of the data distribution are tested. The ANOVA algorithm calculates (or selects from an algorithm table) the f-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 variances of individual laboratory results, is greater than the f-critical value, then the null hypothesis is rejected.

For each set of laboratory data, the ANOVA results tables in the report appendices present the number of sample replicates (n), as well as the sum, mean, and variance (square of the data set standard deviation) values. The tables also provide the various statistical calculation values that are used by the ANOVA algorithm to test the variance of all of the results 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 (F-Crit), and probability value (P-value).

The QC samples, including the reagent blank, method bank, LCS (method blank spike), matrix spike, and control soil NIST SRM 2710a, were also processed with the arsenic and lead IVBA extraction/EPA Method 3050B/3051A digested RM 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 RM statistical values and control limits.

The statistical *t*-test was used to analyze the data from the different laboratories to determine, for example, if one (1) set of data was statistically different from the others. The *t*-test function in Microsoft® Excel was used, which is the two (2)-sample *t*-test (assuming unequal variances, alpha 0.05, 95th percentile). The *t*-test was performed on RM data sets subsequent to the Grubbs' test for outliers, ANOVA, and QC sample results evaluations to determine if any data points or individual laboratory data sets should be removed prior to calculating the prediction intervals (PIs) and confidence intervals (CIs). Generally, the individual laboratory data sets for each RM that exhibited a mean value greater than a ± 15% difference from the mean of the remaining composite data were evaluated using the statistical *t*-test. The *t*-test results were included in the report appendices if it resulted in the removal of an individual laboratory's data set from the composite data set prior to calculating the PIs and CIs.

The data processing scheme for each RM data set included:

- Identifying and removing outliers from further analysis using the Grubbs' test for outliers;
- Calculating the PIs and CIs for the preliminary data set, subsequent to the removal of outliers;
- Evaluating each preliminary data set using ANOVA;
- Removing laboratory results from the composite data set based on QC or control sample results;
- Performing a *t*-test comparing individual laboratory results which were identified to be significantly different from the composite data set, to determine if they should be excluded from further processing; and,
- Calculating the PIs and CIs for the composite data set, subsequent to the removal of laboratory results based on QC flags or *t*-test results.

V. RESULTS AND DISCUSSION

V.A. Initial Demonstration of Proficiency

The IDP forms provided by the laboratories are presented in Appendix K. 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 nine (9) candidate laboratories, seven (7) laboratories were selected to be participants in the Study.

V.B. Study Results

Each of the seven (7) laboratories participating in the Study analyzed the RMs using five (5) replicate aliquots, providing a total of thirty-five (35) results for the IVBA extractions, and thirty-five (35) results for the EPA Method 3050B or 3051A procedure for each analyte in each RM, with one (1) exception. As previously noted and discussed below, NIST SRM 2710a was also used as the Control Soil for the IVBA arsenic extractions, thus yielding a total of six (6) replicate analysis results (42 total results before processing) from each laboratory for this RM. 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 L in redacted form. The original, unredacted SOW forms completed by the laboratories are available from the USEPA HQ Co-Chair for the TRW. The detection limits are provided in the Appendix L individual laboratory results, but are not provided in the text tables for brevity.

As mentioned above, the analytical results for the RMs submitted by the laboratories are located in Appendix L. These analytical results, which are also included in the Appendices A through G tables, are presented using the number of significant figures as submitted by the laboratories. The statistical processing of the laboratories' results was performed using the number of significant figures in the results "as received" from the laboratories. The results of the statistical processing were rounded to the appropriate number of significant figures, and presented in the report discussion and Appendix tables as such. The final Study results, as presented in Tables 1, 17, 18, and 19 of this report, have been rounded to the generally-accepted number of significant figures appropriate for the methods.

V.C. RM Results and Statistical Analysis

V.C.1. Arsenic Results, EPA Method 1340

V.C.1.1. The results of EPA Method 1340 IVBA extraction of arsenic for the **NIST SRM 2710a** are presented in Appendix A, Table A-1, and the derived statistics are summarized in Table 6 below. As previously noted, five (5) replicates of this RM were analyzed as samples, and each of the laboratories analyzed a single QC control soil that was also NIST SRM 2710a, which provided an additional seven (7) results, for a total of forty-two (42) results prior to statistical processing. The formulas used for the PI and CI of the mean are provided in Exhibit 1, using the example where the number (n) of sample results is 42. Laboratory G extracted these samples on May 18, 2017 and analyzed the extracts on June 14, 2017. The SOW specifies that the extracts should be analyzed within (1) week; however, this delay is not expected to have an effect on the analytical results.

Appendix A, Table A-2, presents the EPA Method 1340 IVBA extraction arsenic results for the associated QC samples that were determined from the five (5) replicate NIST SRM 2710a IVBA extractions. These results include the LCS (method blank spike) recovery, matrix spike recovery, and the control soil NIST SRM 2710a results and percent recovery. All reported detection limit results were less than the limits provided in the EPA Method 1340, and in Table 5 above, across all laboratories and all sample sets. The detection limits are provided in the Appendix K individual laboratory results, but are not provided in the text tables for brevity. All results are within the control limits presented in the EPA Method 1340, and in Table 5 above, with the exceptions of the EPA Method 1340 NIST SRM 2710a arsenic control soil results for Laboratories F and G, and the matrix spike recovery result for Laboratory F. The control soil results from Laboratories F and G

were both slightly lower than the arsenic NIST SRM 2710a control limits established prior to initiating the Study. The NIST SRM 2710a control limits were established using historical data from one (1) laboratory, which is expected to exhibit less variability than interlaboratory data. The EPA Method 1340 NIST SRM 2710a control soil mean arsenic result for all seven (7) laboratories is 91.2%, which indicates good overall accuracy. The Laboratory F result for the NIST SRM 2710a matrix spike recovery is 73.9%, which is outside the 75% to 125% matrix spike recovery range; however, because the spiking ratio was less than a 1:4 spike-to-sample concentration, this spike result is not a reliable predictor of accuracy.

Table 6. NIST SRM 2710a IVBA Arsenic Results – All Labs				
Mean: 523.7 mg/Kg	SD: 69.6 mg/Kg	RSD: 13.3%	No. of Labs: 7	n: 42
Low 99% PI	Mean		High 99% PI	
333.4 mg/Kg	523.7 mg/Kg		714.0 mg/Kg	
± 99% PI = 36.3% of the Mean Value				
Low 99% CI	Mean		High 99% CI	
494.7 mg/Kg	523.7 mg/Kg		552.7 mg/Kg	
± 99th Percentile of the CI of the Mean = 5.5% of the Mean Value				
SD of the Mean: 10.7 mg/Kg		RSD of the Mean: 2.1%		

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*-test; for n = 42, df = 41, t = 2.701, for 99th percentile

sm = standard deviation of the mean

The mean IVBA value is the (mean extraction result) / (mean EPA Method 3050B/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 (2) independent variables with unequal means . Please note that the standard deviations are normalized to percentiles before squaring.

$$sd\ IVBA\ ratio = \left(IVBA\ ratio * \left(\sqrt{((sd / mean\ Extraction)\text{^}2 + (sd / mean\ Digestion)\text{^}2} \right) \right)$$

Note: The square root of the sum of the variances squared Method was used as an estimator of the combined variance for the final 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 digestion results are not subsets of the same population and, therefore, their respective variances are additive, even during the division operation.

Appendix A, Table A-3, presents the ANOVA for the NIST SRM 2710a EPA Method 1340 IVBA extraction arsenic results. The results of the ANOVA assessment, 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 (32675) compared to the low intralaboratory group results MS value (79). As previously stated in Section IV.E, if the calculated f-value is greater than the f-critical value, then the null hypothesis is rejected, which is the case for the arsenic extraction data sets. The ANOVA results presented in Table A-3 indicate that the

variances in interlaboratory data were 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 sample preparation and analysis, 2) different samples were being analyzed, or 3) the intralaboratory variances were small compared to what might be expected. The third choice is most likely correct, given that the RSDs for the NIST SRM 2710a for the intralaboratory (n=5) results are all quite low (equal to or less than 2.3% RSD for each of the seven (7) data sets – See Appendix A, Table A-1), and care was taken to ensure the uniformity of the method used and the samples analyzed.

The NIST SRM 2710a IVBA extraction arsenic control soil recovery results for Laboratories F and G were outside the control limits established prior to the Study initiation. Therefore, the NIST SRM 2710a IVBA extraction arsenic PIs and CIs were recalculated after omitting the data set results from these laboratories. The results of EPA Method 1340 IVBA extraction of arsenic for the NIST SRM 2710a, omitting Laboratories F and G results, are presented in Appendix A, Table A-4, and the derived statistics are summarized in Table 7 below. Again, five (5) replicates of this RM were analyzed as samples, and each of the laboratories analyzed a single QC control soil, which was also NIST SRM 2710a, providing a total of thirty (30) results. Excluding the results from Laboratories F and G resulted in a PI +/- range of 31.3%, which is lower than the initial PI +/- range of 36.3%, when results from all seven (7) laboratories were included.

Table 7. NIST SRM 2710a IVBA Arsenic Results – Excluding Laboratories F and G				
Mean: 552.4 mg/Kg	SD: 61.7 mg/Kg	RSD: 11.2%	No. of Labs: 5	n: 30
Low 99% PI	Mean		High 99% PI	
379.4 mg/Kg	552.4 mg/Kg		725.4 mg/Kg	
± 99% PI = 31.3% of the Mean Value				
Low 99% CI	Mean		High 99% CI	
521.4 mg/Kg	552.4 mg/Kg		583.5 mg/Kg	
± 99th Percentile of the CI of the Mean = 5.6% of the Mean Value				
SD of the Mean: 11.3 mg/Kg		RSD of the Mean: 2.1%		

V.C.1.2. The results of EPA Method 1340 IVBA extraction of arsenic for the **FCRM < 250 µm** are presented in Appendix B, Table B-1, and the derived statistics are summarized in Table 8 below. Five (5) replicates of this RM were analyzed by all seven (7) laboratories, for a total of thirty-five (35) results.

Appendix B, Table B-2, presents the EPA Method 1340 IVBA extraction arsenic results for the associated QC samples that were determined with the five (5) FCRM < 250 µm replicate IVBA extractions. These results include the LCS (method blank spike) recovery, matrix spike recovery, and the control soil NIST SRM 2710a results and percent recovery. All results were within the control limits presented in the EPA Method 1340 and in Table 5 above, with the exceptions of the EPA Method 1340 NIST SRM 2710a arsenic control soil results for Laboratories F and G. The control soil results from Laboratories F and G were both lower than the arsenic NIST SRM 2710a control limits established prior to initiating the Study. The EPA Method 9200.2-164 NIST SRM 2710a control soil mean arsenic result for all seven (7) laboratories is 93.9%, which indicates good overall accuracy.

Table 8. FCRM < 250 µm IVBA Arsenic Results – All Laboratories				
Mean: 134.1 mg/Kg	SD: 27.3 mg/Kg	RSD: 20.4%	No. of Labs: 7	n: 35
Low 99% PI	Mean		High 99% PI	
58.6 mg/Kg	134.1 mg/Kg		209.7 mg/Kg	

± 99% PI = 56.3% of the Mean Value		
Low 99% CI	Mean	High 99% CI
121.5 mg/Kg	134.1 mg/Kg	146.7 mg/Kg
± 99th Percentile of the CI of the Mean = 9.4% of the Mean Value		
SD of the Mean: 4.6 mg/Kg	RSD of the Mean: 3.4%	

Appendix B, Table B-3, presents the ANOVA for the FCRM < 250 µm EPA Method 1340 IVBA extraction arsenic results. The results of the ANOVA assessment, which are presented in Table B-3, indicate that the intralaboratory variance is low compared to interlaboratory variance. This is reflected by the larger MS value for the interlaboratory group results (4122) compared to the low intralaboratory group results MS value (22). As previously stated in Section IV.E, if the calculated f-value is greater than the f-critical value, then the null hypothesis is rejected, which is the case for the arsenic extraction data sets. The ANOVA results presented in Table B-3 indicate that the variances in interlaboratory data were large relative to the intralaboratory data variances; therefore, the null hypothesis is rejected with a high degree of confidence (low P-value). The intralaboratory variances were small. The RSDs for the FCRM < 250 µm for the intralaboratory (n=5) results are all quite low, equal to or less than 3.7% RSD for six (6) of the seven (7) data sets, and one (1) data set, Laboratory G, exhibited a 7.8% RSD (See Appendix B, Table B-1). However, the RM results of the Laboratory E data set are quite low in comparison to the other laboratory results.
As discussed above, the NIST SRM 2710a IVBA extraction arsenic control soil recovery results for Laboratories F and G were outside the control limits established prior to the Study initiation, and the associated RM results were omitted from further statistical calculations.

Appendix B, Table B-4, provides the statistical *t*-test comparison of the Laboratory E Method 9200.2-164 IVBA extraction arsenic results with the Laboratories A – D arsenic results. This *t*-test was performed because the arsenic results from Laboratory E, as shown in Table B-1, were much lower than the other laboratory results (laboratory mean result >15% difference from the remaining composite mean). The *t*-test was employed to evaluate if there was a statistical difference between the results from Laboratory E versus the other reported results. The *t*-test results, presented in Table B-4, show that there is a significant difference between the data from Laboratory E compared to the results derived from the other laboratories collectively, as indicated by a P (T ≤ t) value that is less than 0.05 for the *t*-tests performed on the data set. A *t*-Stat value that is greater than the *t*-critical value also indicates a significant difference between the Laboratory E data and the remaining data sets. The *t*-test comparison results, which are presented in Table B-4, indicate that the extraction results for Laboratory E can reasonably be excluded with a less than 5% chance of being incorrect.

The FCRM < 250 µm EPA Method 1340 IVBA extraction arsenic results for Laboratory E were omitted, and arsenic PIs and CIs were re-calculated, after omitting the results from the Laboratories E – G. The results of EPA Method 1340 IVBA extraction of arsenic for the FCRM < 250 µm, for the remaining four (4) laboratories, are presented in Appendix B, Table B-5, and the derived statistics are summarized in Table 9 below. Excluding the results from Laboratories E through G resulted in a PI +/- range of 26.4%, which is lower than the PI +/- range of 56.3%, when results from all seven (7) laboratories were included.

Table 9. FCRM < 250 µm IVBA Arsenic Results – Excluding Laboratories E – G				
Mean: 154.1 mg/Kg	SD: 13.9 mg/Kg	RSD: 9.0%	No. of Labs: 4	n: 20
Low 99% PI	Mean		High 99% PI	
113.4 mg/Kg	154.1 mg/Kg		194.7 mg/Kg	
± 99% PI = 26.4% of the Mean Value				

Low 99% CI	Mean	High 99% CI
145.2 mg/Kg	154.1 mg/Kg	162.9 mg/Kg
± 99th Percentile of the CI of the Mean = 5.8% of the Mean Value		
SD of the Mean: 3.1 mg/Kg	RSD of the Mean: 2.0%	

V.C.1.3. The results of EPA Method 1340 IVBA extraction of arsenic for the **FCRM < 74 µm** are presented in Appendix C, Table C-1, and the derived statistics are summarized in Table 10 below. Five (5) replicates of this RM were analyzed by all seven (7) laboratories, for a total of thirty-five (35) results.

Table 10. FCRM < 74 um IVBA Arsenic Results – All Laboratories				
Mean: 134.1 mg/Kg	SD: 27.2 mg/Kg	RSD: 20.3%	No. of Labs: 7	n: 35
Low 99% PI	Mean	High 99% PI		
58.7 mg/Kg	134.1 mg/Kg	209.4 mg/Kg		
± 99% PI = 56.2% of the Mean Value				
Low 99% CI	Mean	High 99% CI		
121.5 mg/Kg	134.1 mg/Kg	146.6 mg/Kg		
± 99th Percentile of the CI of the Mean = 9.4% of the Mean Value				
SD of the Mean: 4.6 mg/Kg	RSD of the Mean: 3.4%			

Appendix C, Table C-2, presents the EPA Method 1340 IVBA extraction arsenic results for the associated QC samples that were determined with the five (5) FCRM < 74 µm replicate IVBA extractions. These results include the LCS (method blank spike) recovery, matrix spike recovery, and the control soil NIST SRM 2710a results and percent recovery. All results were within the control limits presented in the EPA Method 1340 and in Table 5 above. The EPA Method 1340 NIST SRM 2710a control soil mean arsenic result for all seven (7) laboratories is 94.7%, which indicates good overall accuracy.

Appendix C, Table C-3, presents the ANOVA for the FCRM < 74 µm EPA Method 1340 IVBA extraction arsenic results. The results of the ANOVA assessment which are presented in Table C-3 indicate that the intralaboratory variance is low compared to interlaboratory variance. This is reflected by the larger MS value for the interlaboratory group results (4090) compared to the low intralaboratory group results MS value (24). As previously stated in Section IV.E, if the calculated f-value is greater than the f-critical value, then the null hypothesis is rejected, which is the case for the arsenic extraction data sets. The ANOVA results presented in Table C-3 indicate that the variances in interlaboratory data were large relative to the intralaboratory data variances; therefore the null hypothesis is rejected with a high degree of confidence (low P-value). The intralaboratory variances were small. The RSDs for the FCRM < 74 µm for the intralaboratory (n=5) results are all quite low, equal to or less than 2.9% RSD for five (5) of the seven (7) data sets, and equal to or less than 8.4% for Laboratories E and G (See Appendix C, Table C-1). However, the RM results of the Laboratory E data set are quite low in comparison to the other laboratory results.

Appendix C, Table C-4, provides the statistical *t*-test comparison of the Laboratory E Method 9200.2-164 IVBA extraction arsenic results with the Laboratories A, B, C, D, F, and G composite arsenic results. This *t*-test was performed because the arsenic results from Laboratory E, as shown in Table C-1, were much lower than the other laboratory results (laboratory mean result >15% difference from the remaining composite mean). The *t*-test was employed to evaluate if there was a statistical difference between the results from Laboratory E versus the other reported results. The *t*-test results presented in Table C-4 show that there is a significant difference between the data from Laboratory E compared to the results derived from the other laboratories

collectively, as indicated by a P (T ≤ t) value that is less than 0.05 for the *t*-tests performed on the data set. A *t*-Stat value that is greater than the *t*-critical value also indicates a significant difference between the Laboratory E data and the remaining data sets. The *t*-test comparison results, which are presented in Table C-4, indicate that the extraction results for Laboratory E can reasonably be excluded with a less than 5% chance of being incorrect. As discussed in Section V.C.1.2 above, the Laboratory E Method 9200.2-164 IVBA extraction arsenic results for the FCRM < 250 µm were also excluded from the respective composite data set based on *t*-test results. As with the Laboratories F and G Method 9200.2-164 IVBA extraction arsenic results for the FCRM < 250 µm, the Laboratories F and G Method 9200.2-164 IVBA extraction arsenic results for the FCRM < 74 µm were lower (10.0 %D and 7.5 %D, respectively) than the composite results. Laboratories F and G Method 9200.2-164 IVBA extraction arsenic results for the FCRM < 250 µm were excluded from the respective composite data set based on the outlier NIST SRM 2710a batch QC sample results. However, the NIST SRM 2710a batch QC sample results associated with the Laboratories F and G Method 9200.2-164 IVBA extraction arsenic results for the FCRM < 74 µm were within the control limits, and the RM results were retained in the respective composite data set.

The FCRM < 74 µm EPA Method 1340 IVBA extraction arsenic results for Laboratory E were omitted, and arsenic PIs and CIs were re-calculated, using the results from the remaining laboratories. The results of EPA Method 1340 IVBA extraction of arsenic for the FCRM < 74 µm for the remaining laboratories are presented in Appendix C, Table C-5, and the derived statistics are summarized in Table 11 below. Excluding the results from Laboratory E resulted in a PI +/- range of 35.0%, which is lower than the initial PI +/- range of 56.2%, when results from all seven (7) laboratories were included.

Table 11. FCRM < 74 um IVBA Arsenic Results – Excluding Laboratory E				
Mean: 142.8 mg/Kg	SD: 17.8 mg/Kg	RSD: 12.5%	No. of Labs: 6	n: 30
Low 99% PI	Mean		High 99% PI	
92.8 mg/Kg	142.8 mg/Kg		192.7 mg/Kg	
± 99% PI = 35.0% of the Mean Value				
Low 99% CI	Mean		High 99% CI	
133.8 mg/Kg	142.8 mg/Kg		151.8 mg/Kg	
± 99th Percentile of the CI of the Mean = 6.3% of the Mean Value				
SD of the Mean: 3.3 mg/Kg		RSD of the Mean: 2.3%		

V.C.2. Lead Results, EPA Method 9200.2-164

V.C.2.1. The results of EPA Method 1340 IVBA extraction of lead for the **FCRM < 74 µm** are presented in Appendix D, Table D-1, and the derived statistics are summarized in Table 12 below. Five (5) replicates of this RM were analyzed by each of the seven (7) laboratories, for a total of thirty-five (35) results. Grubb's test for outliers was performed and indicated that the first replicate value of the Laboratory G results was an outlier, and this value was removed prior to performing subsequent statistical calculations, leaving a total of thirty-four (34) results for processing. The outlier is marked with an asterisk in Table D-1.

Table 12. FCRM < 74 µm IVBA Lead Results – All Laboratories				
Mean: 4644 mg/Kg	SD: 271.6 mg/Kg	RSD: 5.8%	No. of Labs: 7	n: 34
Low 99% PI	Mean		High 99% PI	
3891 mg/Kg	4644 mg/Kg		5397 mg/Kg	
± 99% PI = 16.2% of the Mean Value				
Low 99% CI	Mean		High 99% CI	

4517 mg/Kg	4644 mg/Kg	4771 mg/Kg
± 99th Percentile of the CI of the Mean = 2.7% of the Mean Value		
SD of the Mean: 46.6 mg/Kg	RSD of the Mean: 1.0%	

Appendix D, Table D-2, presents the EPA Method 1340 IVBA extraction lead results for the associated QC samples that were determined with the five (5) FCRM < 74 µm replicate IVBA extractions. These results include the LCS (method blank spike) recovery, matrix spike recovery, and the control soil NIST SRM 2710a results and percent recovery. All results are within the control limits presented in the EPA Method 1340, and in Table 5 above, with the exception of the EPA Method 1340 NIST SRM 2710a lead control soil results for Laboratories C and F. The control soil results from Laboratories C and F were both slightly lower than the lead NIST SRM 2710a control limits established prior to initiating the Study. The EPA Method 1340 NIST SRM 2710a control soil mean lead result for all seven (7) laboratories is 93.3%, which indicates good overall accuracy.

Appendix D, Table D-3, presents the ANOVA for the FCRM < 74 µm EPA Method 1340 IVBA extraction lead results. The results of the ANOVA assessment, presented in Table D-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 (369947) compared to the low intralaboratory group results MS value (7980). As previously stated in Section IV.E, if the calculated f-value 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 D-3 indicate that the variances in the interlaboratory data were large relative to the intralaboratory data variances; therefore the null hypothesis is rejected with a high degree of confidence (low P-value). The intralaboratory variances were small. The RSDs for the FCRM < 74 µm for the intralaboratory (n=5) results are all quite low, equal to or less than 2.6% RSD for each of the seven (7) data sets (See Appendix D, Table D-1).

The NIST SRM 2710a IVBA extraction lead control soil recovery results for Laboratories C and F were outside the control limits established prior to the Study initiation. Therefore, the FCRM <74 µm IVBA extraction lead PIs and CIs were re-calculated after omitting the data set results from these laboratories. The results of EPA Method 1340 IVBA extraction of lead for the FCRM <74 µm, omitting Laboratories C and F results, are presented in Appendix D, Table D-4, and the derived statistics are summarized in Table 13 below. Excluding the results from Laboratories C and F resulted in a PI +/- range of 10.0%, which is lower than the initial PI +/- range of 16.2%, when results from all seven (7) laboratories were included (minus the one (1) outlier).

V.C.3. Arsenic Results, EPA Method 3050B or 3051A

V.C.3.1. The results of EPA Method 3050B or 3051A digestion for arsenic for the **FCRM < 250 µm** are presented in Appendix E, Table E-1, and the derived statistics are summarized in Table 14 below. Five (5) replicates of this RM were analyzed as samples by each of the laboratories for a total of thirty-five (35) results.

Table 13. FCRM < 74 µm IVBA Lead Results – Excluding Laboratories C and F				
Mean: 4768 mg/Kg	SD: 165.8 mg/Kg	RSD: 3.5%	No. of Labs: 5	n: 24
Low 99% PI	Mean		High 99% PI	
4293 mg/Kg	4768 mg/Kg		5243 mg/Kg	
± 99% PI = 10.0% of the Mean Value				
Low 99% CI	Mean		High 99% CI	

4673 mg/Kg	4768 mg/Kg	4863 mg/Kg
± 99th Percentile of the CI of the Mean = 2.0% of the Mean Value		
SD of the Mean: 33.8 mg/Kg	RSD of the Mean: 0.71%	

Table 14. FCRM < 250 µm Total Arsenic Results – All Laboratories				
Mean: 709.0 mg/Kg	SD: 35.1 mg/Kg	RSD: 5.0%	No. of Labs: 7	n: 35
Low 99% PI	Mean		High 99% PI	
611.8 mg/Kg	709.0 mg/Kg		806.1 mg/Kg	
± 99% PI = 13.7% of the Mean Value				
Low 99% CI	Mean		High 99% CI	
692.8 mg/Kg	709.0 mg/Kg		725.2 mg/Kg	
± 99th Percentile of the CI of the Mean = 2.3% of the Mean Value				
SD of the Mean: 5.9 mg/Kg	RSD of the Mean: 0.84%			

Appendix E, Table E-2, presents the EPA Method 3050B or 3051A digestion arsenic results for the associated QC samples that were determined from the five (5) FCRM < 250 µm replicate arsenic digestions. These results include the LCS (method blank spike) recovery, matrix spike recovery, and the control soil NIST SRM 2710a results and percent recovery. All results are within the control limits presented in the EPA Method 1340, and in Table 5 above, with the exception of the EPA Method 3050B or 3051A NIST SRM 2710a arsenic control soil result from Laboratory E, and the matrix spike result from Laboratory G. The EPA Method 3050B/3051A NIST SRM 2710a control soil mean arsenic result, based on the nominal value provided in the addendum to the NIST SRM 2710a Certificate of Analysis for leachable arsenic of 1400 mg/Kg, for all seven (7) laboratories, is 106.2%, which indicates good overall accuracy. The Laboratory G FCRM < 250 µm digestion results for arsenic were not excluded from the composite data set based on the outlier matrix spike results, since the spike concentration was less than one-fourth of the indigenous sample concentration for arsenic, which can result in unreliable recoveries.

Appendix E, Table E-3, presents the ANOVA for the FCRM < 250 µm EPA Method 3050B or 3051A digestion arsenic results. The results of the ANOVA assessment, presented in Table E-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 (4660) compared to the low intralaboratory group results MS value (498). As previously stated in Section IV.E, if the calculated f-value is greater than the f-critical value, then the null hypothesis is rejected, which is the case for the arsenic digestion data sets. The ANOVA results presented in Table E-3 indicate that the variances in the interlaboratory data were large relative to the intralaboratory data variances; therefore the null hypothesis is rejected with a high degree of confidence (low P-value). The intralaboratory variances were small. The RSDs for the FCRM < 250 µm for the intralaboratory (n=5) results are all quite low, equal to or less than 3.8% RSD for each of the seven (7) data sets (See Appendix E, Table E-1).

The EPA Method 3050B or 3051A digestion arsenic control soil recovery results for Laboratory E are outside the control limits. Therefore, the FCRM < 250 µm EPA Method 3050B or 3051A digestion arsenic PIs and CIs were re-calculated after omitting the data set results from this laboratory. The results of EPA Method 3050B or 3051A digestion of arsenic for the FCRM < 250 µm, omitting Laboratory E, are presented in Appendix E, Table E-4, and the derived statistics are summarized in Table 15 below. Excluding the results from Laboratory E allowed for the calculation of a PI +/- range of 13.9%, which is slightly higher than the initial PI +/- range of 13.7%, when results from all seven (7) laboratories were included. As previously stated, the Laboratory G matrix

spike result associated with the FCRM < 250 µm digestion results for arsenic exceeded the matrix spike recovery criteria. The Laboratory G FCRM < 250 µm digestion results for arsenic were not excluded from the composite data set based on the outlier matrix spike results, since the matrix spike concentration was less than one-fourth of the indigenous sample concentration for arsenic, leading to an unreliable recovery result.

Table 15. FCRM < 250 µm Total Arsenic Results – Excluding Laboratory E				
Mean: 704.5 mg/Kg	SD: 35.0 mg/Kg	RSD: 5.0%	No. of Labs: 6	n: 30
Low 99% PI	Mean		High 99% PI	
606.4 mg/Kg	704.5 mg/Kg		802.7 mg/Kg	
± 99% PI = 13.9% of the Mean Value				
Low 99% CI	Mean		High 99% CI	
686.9 mg/Kg	704.5 mg/Kg		722.2 mg/Kg	
± 99th Percentile of the CI of the Mean = 2.3% of the Mean Value				
SD of the Mean: 6.4 mg/Kg		RSD of the Mean: 0.91%		

V.C.3.2. The results of EPA Method 3050B or 3051A digestion of arsenic for the **FCRM < 74 µm** are presented in Appendix F, Table F-1, and the derived statistics are summarized in Table 16 below. Five (5) replicates of this RM were analyzed by each of the laboratories, for a total of thirty-five (35) results. However, one (1) result was removed from subsequent calculations after the Grubbs' test showed it to be an outlier. The outlier is marked with an asterisk in Table F-1.

Table 16. FCRM < 74 µm Total Arsenic Results – All Laboratories				
Mean: 710.0 mg/Kg	SD: 24.5 mg/Kg	RSD: 3.5%	No. of Labs: 7	n: 34
Low 99% PI	Mean		High 99% PI	
642.0 mg/Kg	710.0 mg/Kg		778.0 mg/Kg	
± 99% PI = 9.6% of the Mean Value				
Low 99% CI	Mean		High 99% CI	
698.5 mg/Kg	710.0 mg/Kg		721.5 mg/Kg	
± 99th Percentile of the CI of the Mean = 1.6% of the Mean Value				
SD of the Mean: 4.2 mg/Kg		RSD of the Mean: 0.59%		

Appendix F, Table F-2, presents the EPA Method 3050B or 3051A digestion arsenic results for the associated QC samples that were determined from the five (5) FCRM < 74 µm replicate arsenic digestions. These results include the LCS (method blank spike) recovery, matrix spike recovery, and the control soil NIST SRM 2710a results and percent recovery. All results are within the control limits presented in the EPA Method 1340, and in Table 5 above, with the exception of the EPA Method 3050B or 3051A NIST SRM 2710a arsenic matrix spike recovery for Laboratory G. The Laboratory G result for the NIST SRM 2710a matrix spike recovery is 215%, which is outside the 75% to 125% matrix spike recovery range; however, because the spiking ratio was less than a 1:4 spike-to-sample concentration, this spike result is not a reliable predictor of accuracy, therefore, the Laboratory G results were retained. The EPA Method 3050B/3051A NIST SRM 2710a control soil mean arsenic result, based on the nominal value provided in the NIST SRM 2710a Certificate of Analysis for leachable arsenic of 1400 mg/Kg, for all seven (7) laboratories, is 106.3%, which indicates good overall accuracy.

Appendix F, Table F-3, presents the ANOVA for the FCRM < 74 µm EPA Method 3050B or 3051A digestion arsenic results. The results of the ANOVA assessment, which are presented in Table F-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 (2100) compared to the low intralaboratory group results MS value (268). As previously stated in Section IV.E, if the calculated f-value is greater than the f-critical value, then the null hypothesis is rejected, which is the case for the arsenic extraction data sets. The ANOVA results presented in Table F-3 indicate that the variances in interlaboratory data were large relative to the intralaboratory data variances; therefore, the null hypothesis is rejected with a high degree of confidence (low P-value). The intralaboratory variances were small. The RSDs for the FCRM < 74 µm for the intralaboratory (n=5) results are all quite low, equal to or less than 3.2% RSD for each of the seven (7) data sets (See Appendix F, Table F-1). The rejected null hypothesis is likely due to the low intralaboratory variance relative to the interlaboratory variance, however; there are no RM outlier results or QC results that would indicate RM results from any single laboratory should be eliminated. Therefore, all of the results from this data set were retained for statistical processing.

V.C.4. Lead Results, EPA Method 3050B or 3051A

V.C.4.1. The results of EPA Method 3050B or 3051A digestion of lead for the **FCRM < 74 µm** are presented in Appendix G, Table G-1, and the derived statistics are summarized in Table 17 below. Five (5) replicates of this RM were analyzed as samples by each of the laboratories for a total of thirty-five (35) results.

Table 17. FCRM < 74 µm Total Lead Results – All Laboratories				
Mean: 6371 mg/Kg	SD: 271.4 mg/Kg	RSD: 4.3%	No. of Labs: 7	n: 35
Low 99% PI	Mean		High 99% PI	
5620 mg/Kg	6371 mg/Kg		7123 mg/Kg	
± 99% PI = 11.8% of the Mean Value				
Low 99% CI	Mean		High 99% CI	
6246 mg/Kg	6371 mg/Kg		6497 mg/Kg	
± 99th Percentile of the CI of the Mean = 2.0% of the Mean Value				
SD of the Mean: 45.9 mg/Kg		RSD of the Mean: 0.72%		

Appendix G, Table G-2, presents the EPA Method 3050B or 3051A digestion lead results for the associated QC samples that were determined from the five (5) FCRM < 74 µm replicate lead digestions. These results include the LCS (method blank spike) recovery, matrix spike recovery, and the control soil NIST SRM 2710a results and percent recovery. All results are within the control limits presented in the EPA Method 1340, and in Table 5 above, with the exception of the EPA Method 3050B or 3051A NIST SRM 2710a lead matrix spike recovery for Laboratory G. The Laboratory G result for the NIST SRM 2710a matrix spike recovery is 1900%, which is outside the 75% to 125% matrix spike recovery range; however, because the spiking ratio was less than a 1:4 spike-to-sample concentration, this spike result is not a reliable predictor of accuracy, therefore, the Laboratory G results were retained. The EPA Method 3050B/3051A NIST SRM 2710a control soil mean lead result, based on the nominal value provided in the NIST SRM 2710a Certificate of Analysis leachable lead of 5100 mg/Kg, for all seven (7) laboratories, is 98.3%, which indicates good overall accuracy.

Appendix G, Table G-3, presents the ANOVA for the FCRM < 74 µm EPA Method 3050B or 3051A digestion lead results. The results of the ANOVA assessment, which are presented in Table G-3, indicate that the intralaboratory variance is low compared to the interlaboratory variance. This is reflected by the large MS value for the interlaboratory group results (356639) compared to the low intralaboratory group results MS value (13048). As previously stated in Section IV.E, if the calculated f-value 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 G-3 indicate that

the variances in interlaboratory data were large relative to the intralaboratory data variances; therefore the null hypothesis is rejected with a high degree of confidence (low P-value). The intralaboratory variances were small. The RSDs for the FCRM < 74 µm for the intralaboratory (n=5) results are all quite low, equal to or less than 2.6% RSD for each of the seven (7) data sets (See Appendix G, Table G-1). As with the EPA Method 3050B or 3051A digestion arsenic results for the FCRM < 74 µm discussed in the previous section, the rejected null hypothesis is likely due to the low intralaboratory variance relative to the interlaboratory variance, however; there are no RM outlier results or QC results that would indicate RM results from any single laboratory should be eliminated. Therefore, all of the results from this data set were retained for statistical processing.

V.D. Summary of NIST SRM 2710a and FCRM Study Results and IVBA (%) Prediction and Confidence Intervals

V.D.1. NIST SRM 2710a IVBA (%)

Appendix H, Table H-1, presents the NIST SRM 2710a Method 3050B arsenic results (n=12) derived from a six (6)-laboratory Contract Laboratory Program (CLP) Proficiency Testing (PT) event in the year 2008. The mean and range calculated from this data were used by NIST to set the NIST SRM 2710a arsenic leachable value of 1400 mg/Kg presented in the Addendum to the NIST SRM 2710a Certificate of Analysis. These results, along with the NIST SRM 2710a IVBA extraction results from this Study, were used to calculate the NIST SRM 2710a IVBA (%) statistics and 99th percentile PIs.

Appendix I, Table I-1, presents the NIST SRM 2710a arsenic IVBA PIs and CIs. The intervals were calculated using the IVBA (n=30) extraction results for arsenic in Appendix A, after the omission of the results from Laboratories F and G, and using the Method 3050B/3051A digestion (n=12) results derived from the CLP PT event described above.

The mean arsenic IVBA (%) result is 38.4%, with a pooled RSD value of 13.4%. The calculated arsenic 99th percentile PIs based on the EPA Method 1340 IVBA (%) extraction is ± 36.6%. As shown in Table I-1, the calculated relative standard deviation of the mean is 2.06%, and the calculated 99th percentile CI of the mean is ± 5.6%.

V.D.2. FCRM < 250 µm Arsenic IVBA (%)

Appendix I, Table I-2, presents the FCRM < 250 µm arsenic IVBA (%) PIs and CIs. The intervals were calculated using the IVBA (n=20) extraction results for arsenic in Appendix B, after the omission of the results from Laboratories E, F, and G, and using the Method 3050B/3051A (n=30) digestion results presented in Appendix E.

The mean FCRM < 250 µm arsenic IVBA (%) result is 21.9%, with a pooled RSD value of 10.3%. The calculated arsenic 99th percentile PIs based on the EPA Method 1340 IVBA (%) is ± 27.8%. As shown in Table I-2, the calculated relative standard deviation of the mean is 1.45%, and the calculated 99th percentile CI of the mean is ± 3.9%.

V.D.3. FCRM < 74 µm Arsenic IVBA (%)

Appendix I, Table I-3, presents the FCRM < 74 µm arsenic IVBA (%) PIs and CIs. The intervals were calculated using the IVBA (n=30) extraction results for arsenic presented in Appendix C, after the omission of the results from Laboratory E, and using the Method 3050B/ 3051A (n=34) (one (1) outlier removed) digestion results presented in Appendix F.

The mean FCRM < 74 µm arsenic IVBA (%) result is 20.1%, with a pooled RSD value of 13.0%. The calculated arsenic 99th percentile PIs based on the EPA Method 1340 IVBA (%) is ± 34.7%. As shown in Table I-3, the calculated relative standard deviation of the mean is 1.62%, and the calculated 99th percentile CI of the mean is ± 4.3%.

V.D.4. FCRM < 74 µm Lead IVBA (%)

Appendix I, Table I-4, presents the FCRM < 74 µm lead IVBA PIs and CIs. The intervals were calculated using the IVBA (n=24) extraction results for lead in Appendix D, after the omission of the results from Laboratories C and F, and one (1) outlier from Laboratory G, and using the Method 3050B/3051A (n=35) digestion results presented in Appendix G.

The mean FCRM < 74 µm lead IVBA (%) result is 74.8%, with a pooled RSD value of 5.5%. The calculated arsenic 99th percentile PIs based on the EPA Method 1340 IVBA (%) is ± 14.8%. As shown in Table I-4, the calculated relative standard deviation of the mean is 0.72%, and the calculated 99th percentile CI of the mean is ± 1.9%.

V.D.5. Summary of FCRM Study Results and Prediction Intervals

Table 18 below provides the EPA Method 3050B/3051A lead and arsenic results, statistics, and 99th percentile PIs for the FCRM.

Table 18. FCRM Summary of EPA 3050B or 3051A Results (Rounded Values)

RM Type	Low 99% PI	Mean	High 99% PI	RSD	N
Arsenic FCRM < 250 (mg/Kg)	606	705	803	5.0%	30
Arsenic FCRM < 74 (mg/Kg)	642	710	778	3.5%	34
Lead FCRM < 74 (mg/Kg)	5620	6370	7120	4.3%	35

Table 19 below provides the NIST SRM 2710a and FCRM lead and arsenic results, IVBA (%) and IVBA (mg/kg), statistics, and 99th percentile PIs.

Table 19. NIST SRM 2710a and FCRM: IVBA (%) and IVBA (mg/Kg) Results and Statistics (Rounded Values)

RM Type	Mean Result IVBA (%)	IVBA (%) (± 99% PI)	RSD*	Mean Result IVBA (mg/Kg)	IVBA (mg/Kg) (± 99% PI)	RSD*	N
Arsenic NIST SRM 2710a	38.4	(24.4 - 52.5)	13.4%	552	(379 - 725)	11.2%	30
Arsenic FCRM < 250 µm	21.9	(15.8 - 28.0)	10.3%	154	(113 - 195)	9.0%	20
Arsenic FCRM < 74 µm	20.1	(13.1 - 27.1)	13.0%	143	(92.8 - 193)	12.5%	30
Lead FCRM < 74 µm	74.8	(63.8 - 85.9)	5.5%	4770	(4290 - 5240)	3.5%	24

* RSD was derived from the replicate IVBA results.

VI. CONCLUSIONS AND RECOMMENDATIONS

The main objectives of this Study were to derive mean consensus arsenic values for the NIST SRM 2710a and FCRM < 250 µm, and arsenic and lead values for the FCRM <74 µm, using EPA Method 1340. Another objective was to derive mean total leachable values, with known confidence, for the lead and arsenic concentrations of the FCRM, based on the EPA Method 3050B/3051A results from this Study. Some of the Study results from the seven (7) participating

laboratories were determined to be unacceptable using conventional statistics and Grubbs' test for outliers. The *t*-test was used for the exclusion of laboratory results, which then allowed for the establishment of IVBA values for the RMs with known and acceptable precision. The associated QC results provided by the participating laboratories were generally within control limits defined in EPA Method 1340, with a few noted exceptions.

Table 20 below presents the final rounded values for the mean result and 99th percentile PIs for the NIST SRM 2710a arsenic results, FCRM < 250 µm IVBA arsenic results, FCRM < 74 µm arsenic and lead results, as well as the EPA Method 3050B/3051A lead and arsenic values and PIs based on the pooled Study results. The PIs for the EPA Method 3050B/3051A lead and arsenic values are presented in mg/Kg, while the IVBA PIs are presented in both mg/Kg and as IVBA (%) values.

Table 20. NIST SRM 2710a and FCRM Summary of Results (Rounded Values)

Arsenic NIST SRM 2710a	Low 99% PI	Mean	High 99% PI	RSD
IVBA Extracted (mg/Kg)	379	552	725	11.2%
IVBA (%)	24.4	38.4	52.5	13.4%
Arsenic FCRM < 250	**Low 99% PI**	**Mean**	**High 99% PI**	**RSD**
IVBA Extracted (mg/Kg)	113	154	195	9.0%
3050B/3051A (mg/Kg)	606	704	803	5.0%
IVBA (%)	15.8	21.9	28.0	10.3%
Arsenic FCRM < 74	**Low 99% PI**	**Mean**	**High 99% PI**	**RSD**
IVBA Extracted (mg/Kg)	92.8	143	193	12.5%
3050B/3051A (mg/Kg)	642	710	778	3.5%
IVBA (%)	13.1	20.1	27.1	13.0%
Lead FCRM < 74	**Low 99% PI**	**Mean**	**High 99% PI**	**RSD**
IVBA Extracted (mg/Kg)	4290	4770	5240	3.5%
3050B/3051A (mg/Kg)	5620	6370	7120	4.3%
IVBA (%)	63.8	74.8	85.9	5.5%

VII. REFERENCES

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APPENDIX A

NIST SRM 2710a
Arsenic IVBA Results and Statistics

Appendix A
NIST SRM 2710a EPA Method 1340 IVBA Arsenic Results and Statistics

Table A-1. NIST SRM 2710a EPA Method 1340 IVBA Arsenic Results with Prediction Intervals and Confidence Intervals – All Labs

NIST SRM 2710a EPA Method 1340 IVBA Arsenic Results (mg/Kg)							
Laboratory >	**A**	**B**	**C**	**D**	**E**	**F**	**G**
Replicate 1	564	591.503	470.1	643.3	479.5	426.2	445
Replicate 2	564	599.921	485.3	640.7	482.5	444.5	463
Replicate 3	567	611.050	481.2	624.3	476.6	446.9	460
Replicate 4	568	600.735	487.3	646.9	484.7	444.7	467
Replicate 5	574	597.696	483.2	615.8	492.2	444.5	465
Replicate 6	567	577.420	486.0	621.8	489.4	458.4	457
Mean	567.3	596.4	482.2	632.1	484.2	444.2	459.5
SD	3.7	11.2	6.3	13.0	5.9	10.3	7.9
RSD	0.65%	1.9%	1.3%	2.1%	1.2%	2.3%	1.7%

Pooled Results (n-1) n=42	
Mean	523.7 mg/Kg
SD	69.6 mg/Kg
RSD	13.3%

NIST 2710a EPA Method 1340 IVBA – 99th Percentile Prediction Interval (mg/Kg)		
Low 99% PI	**Mean**	**High 99% PI**
333.4	523.7	714.0
± 99% Prediction Interval = 36.3% of the Mean Value		

The range above should be used to determine if a Laboratory EPA Method 1340 IVBA extracted arsenic result is acceptable.

NIST 2710a EPA Method 1340 IVBA – 99th Percentile Confidence Interval of the Mean (mg/Kg)		
523.7 = Mean	10.7 = SD of the Mean	2.1% = RSD of the Mean
Low 99% CI	**Mean**	**High 99% CI**
494.7	523.7	552.7
± 99th Percentile of the Confidence Interval of the Mean = 5.5% 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 A
NIST SRM 2710a EPA Method 1340 IVBA Arsenic Results and Statistics

Table A-2. NIST SRM 2710a EPA Method 1340 Batch QC Sample Arsenic Results

Laboratory>	A	B	C	D	E	F	G	Mean
Blank Spike Recovery (Nominal: 10 mg/L) (Range: 85% to 115%)	105.0%	101.7%	114.0%	103.1%	91.3%	92.0%	90.0%	99.6%
NIST 2710a Matrix Spike Recovery (Nominal: 10 mg/L) (Range: 75% to 125%)	101.0%	103.0%	85.3%	107.1%	87.9%	(73.9%)	92.0%	92.9%
Control Soil NIST SRM 2710a IVBA Extract Acceptance Range (Nominal: 573 mg/Kg) (Range: 464 – 681 mg/Kg)	567.0	577.4	486.0	621.8	489.4	(458.4)	(457.0)	522.4
Arsenic IVBA NIST SRM 2710a Recovery Based on Nominal Value of 573 mg/Kg	99.0%	100.8%	84.8%	108.5%	85.4%	80.0%	80.1%	91.2%
Control Soil NIST SRM 2710a IVBA Value Based on Leachable Value of 1400 mg/Kg (Acceptance Range 33.1% - 48.4%)	40.5%	41.2%	34.7%	44.4%	35.0%	(32.7%)	(32.6%)	37.3%

Values in parentheses are outside the associated control limits.

Table A-3. NIST SRM 2710a EPA Method 1340 IVBA Analysis of Variance

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

SUMMARY

Groups	*Count*	*Sum*	*Mean*	*Variance*
Laboratory A	6	3404	567	13
Laboratory B	6	3578	596	126
Laboratory C	6	2893	482	40
Laboratory D	6	3793	632	170
Laboratory E	6	2905	484	35
Laboratory F	6	2665	444	107
Laboratory G	6	2757	460	63

ANOVA

Source of Variation	*SS*	*df*	*MS*	*F*	*P-value*	*F-Crit*
Interlaboratory	196049	6	32675	413	5.87388E-31	2.4
Intralaboratory	2771	35	79			
Total	198821	41				

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 A
NIST SRM 2710a EPA Method 1340 IVBA Arsenic Results and Statistics

Table A-4. NIST SRM 2710a EPA Method 1340 IVBA Arsenic Results with Prediction Intervals and Confidence Intervals – Minus Labs F and G

NIST 2710a EPA Method 1340 IVBA Arsenic Results (mg/Kg)							
Laboratory >	**A**	**B**	**C**	**D**	**E**	**F**	**G**
Replicate 1	564	591.503	470.1	643.3	479.5		
Replicate 2	564	599.921	485.3	640.7	482.5		
Replicate 3	567	611.050	481.2	624.3	476.6		
Replicate 4	568	600.735	487.3	646.9	484.7		
Replicate 5	574	597.696	483.2	615.8	492.2		
Replicate 6	567	577.420	486.0	621.8	489.4		
Mean	567.3	596.4	482.2	632.1	484.2		
SD	3.7	11.2	6.3	13.0	5.9		
RSD	0.65%	1.9%	1.3%	2.1%	1.2%		

Pooled Results (n-1) n=30	
Mean	552.4 mg/Kg
SD	61.7 mg/Kg
RSD	11.2%

NIST 2710a EPA Method 1340 IVBA – 99th Percentile Prediction Interval (mg/Kg)		
Low 99% PI	**Mean**	**High 99% PI**
379.4	552.4	725.4
± 99% Prediction Interval = 31.3% of the Mean Value		
The range above should be used to determine if a Laboratory EPA Method 1340 IVBA extracted arsenic result is acceptable.		

NIST 2710a EPA Method 1340 IVBA – 99th Percentile Confidence Interval of the Mean (mg/Kg)		
552.4 = Mean	11.3 = SD of the Mean	2.1% = RSD of the Mean
Low 99% CI	**Mean**	**High 99% CI**
521.4	552.4	583.5
± 99th Percentile of the Confidence Interval of the Mean = 5.6% 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 B

FCRM < 250 µm

Arsenic IVBA Results and Statistics

Appendix B
FCRM < 250 um EPA Method 1340 IVBA Arsenic Results and Statistics

Table B-1. FCRM < 250 µm EPA Method 1340 IVBA Arsenic Results with Prediction Intervals and Confidence Intervals – All Labs

FCRM < 250 µm EPA Method 1340 IVBA Arsenic Results (mg/Kg)							
Laboratory >	**A**	**B**	**C**	**D**	**E**	**F**	**G**
Replicate 1	165	167.033	130.2	158.3	90.1	106.6	139
Replicate 2	158	170.819	131.6	163.3	89.4	107.7	119
Replicate 3	154	164.706	133.8	160.3	89.5	116.5	118
Replicate 4	154	166.269	132.5	157.0	95.2	107.6	122
Replicate 5	160	165.244	131.1	158.1	86.4	111.3	115
Mean	158.2	166.8	131.8	159.4	90.1	109.9	122.6
SD	4.6	2.4	1.4	2.5	3.2	4.1	9.5
RSD	2.9%	1.4%	1.0%	1.6%	3.5%	3.7%	7.8%

Pooled Results (n-1) n=35	
Mean	134.1 mg/Kg
SD	27.3 mg/Kg
RSD	20.4%

FCRM < 250 µm EPA Method 1340 IVBA – 99th Percentile Prediction Interval (mg/Kg)		
Low 99% PI	**Mean**	**High 99% PI**
58.6	134.1	209.7
± 99% Prediction Interval = 56.3% of the Mean Value		

The range above should be used to determine if a Laboratory EPA Method 1340 IVBA extracted arsenic result is acceptable.

FCRM < 250 µm EPA Method 1340 IVBA – 99th Percentile Confidence Interval of the Mean (mg/Kg)		
134.1 = Mean	4.6 = SD of the Mean	3.4% = RSD of the Mean
Low 99% CI	**Mean**	**High 99% CI**
121.5	134.1	146.7
± 99th Percentile of the Confidence Interval of the Mean = 9.4% 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 B
FCRM < 250 um EPA Method 1340 IVBA Arsenic Results and Statistics

Table B-2. FCRM < 250 µm EPA Method 1340 Batch QC Sample Arsenic Results

Laboratory >	A	B	C	D	E	F	G	Mean
Blank Spike Recovery (Nominal: 10 mg/L) (Range: 85% to 115%)	108.0%	103.1%	112.0%	104.5%	85.9%	91.2%	93.0%	99.7%
FCRM <250 µm Matrix Spike Recovery (Nominal: 10 mg/L) (Range: 75% to 125%)	105.0%	97.8%	95.4%	96.8%	88.4%	78.1%	100.0%	94.5%
Control Soil NIST SRM 2710a IVBA Extract Acceptance Range (Nominal: 573 mg/Kg) (Range: 464 – 681 mg/Kg)	604.0	659.1	520.1	621.9	466.6	(439.5)	(461.0)	538.9
Arsenic IVBA NIST SRM 2710a Recovery Based on Nominal Value of 573 mg/Kg	105.0%	115.0%	90.8%	108.5%	81.4%	76.7%	80.0%	93.9%
Control Soil NIST SRM 2710a IVBA Value Based on Leachable Value of 1400 mg/Kg (Acceptance Range 33.1% - 48.4%)	43.1%	47.1%	37.2%	44.4%	33.3%	31.4%	32.9%	38.5%

Values in parentheses are outside the associated control limit.

Table B-3. FCRM < 250 µm EPA Method 1340 IVBA Analysis of Variance All Seven Laboratories

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

SUMMARY

Groups	*Count*	*Sum*	*Mean*	*Variance*
Laboratory A	5	791	158	21
Laboratory B	5	834	167	6
Laboratory C	5	659	132	2
Laboratory D	5	797	159	6
Laboratory E	5	451	90	10
Laboratory F	5	550	110	17
Laboratory G	5	613	123	90

ANOVA

Source of Variation	*SS*	*df*	*MS*	*F*	*P-value*	*F-Crit*
Interlaboratory	24732	6	4122	189.6	2.43742E-21	2.45
Intralaboratory	609	28	22			
Total	25341	34				

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 < 250 um EPA Method 1340 IVBA Arsenic Results and Statistics

Table B-4. FCRM < 250 µm Arsenic Results and *t*-Test for Laboratory E Extraction Data

FCRM < 250 µm EPA Method 9200.2-164 Arsenic Results *t*-Test (mg/Kg)								
Laboratory>	**A**	**B**	**C**	**D**	**F**	**G**	**Laboratory >**	**E**
Replicate 1	165	167.0	130.2	158.3			**Replicate 1**	90.1
Replicate 2	158	170.8	131.6	163.3			**Replicate 2**	89.4
Replicate 3	154	164.7	133.8	160.3			**Replicate 3**	89.5
Replicate 4	154	166.3	132.5	157.0			**Replicate 4**	95.2
Replicate 5	160	165.2	131.1	158.1			**Replicate 5**	86.4
Mean	158.2	166.8	131.8	159.4			**Mean**	90.1
SD	4.6	2.4	1.4	2.5			**SD**	3.2
RSD	2.9%	1.4%	1.0%	1.6%			**RSD**	3.5%

Labs A-G		**Percent Difference**	**Lab E**	
	n=20			**n=5**
Mean	154.1	52.4%	**Mean**	90.1
SD	13.9		**SD**	3.2
RSD	9.0%		**RSD**	3.5%

Excel *t*-Test: Two-Sample Assuming Unequal Variances
alpha = 0.05 (1 in 20)

	Lab A-D	**Lab E**
Mean	154.1	90.1
Variance	192.0	10.1
Observations	20	5
Hypothesized Mean Diff.	0	
df	23	
t-Stat	18.75	
P (T ≤ t) (two-tail)	1.9656E-15	
t-Critical (two-tail)	2.07	

The t-Stat value of 18.75 is greater than t-Critical two-tail value of 2.07; 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 0.05 indicates a greater than 95% 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 E can be excluded.

Appendix B
FCRM < 250 um EPA Method 1340 IVBA Arsenic Results and Statistics

Table B-5. FCRM < 250 µm EPA Method 1340 Arsenic Results With Prediction Intervals and Confidence Intervals – Minus Laboratories E-G

FCRM < 250 µm EPA Method 1340 Arsenic Results (mg/Kg)							
Laboratory >	**A**	**B**	**C**	**D**	**E**	**F**	**G**
Replicate 1	165	167.033	130.2	158.3			
Replicate 2	158	170.819	131.6	163.3			
Replicate 3	154	164.706	133.8	160.3			
Replicate 4	154	166.269	132.5	157.0			
Replicate 5	160	165.244	131.1	158.1			
Mean	158.2	166.8	131.8	159.4			
SD	4.6	2.4	1.4	2.5			
RSD	2.9%	1.4%	1.0%	1.6%			

Pooled Results (n-1) n=20	
Mean	154.1 mg/Kg
SD	13.9 mg/Kg
RSD	9.0%

FCRM < 250 µm Method 9200.2-164 Arsenic Results – 99th Percentile Prediction Interval (mg/Kg)		
Low 99% PI	**Mean**	**High 99% PI**
113.4	154.1	194.7
± 99% Prediction Interval = 26.4% of the Mean Value		

The range above should be used to determine if a Laboratory EPA Method 1340 IVBA extracted arsenic result is acceptable.

FCRM < 250 µm Method 9200.2-164 Arsenic Results – 99th Percentile Confidence Interval of the Mean (mg/Kg)		
154.1 = Mean	3.1 = SD of the Mean	2.0% = RSD of the Mean
Low 99% CI	**Mean**	**High 99% CI**
145.2	154.1	162.9
± 99th Percentile of the Confidence Interval of the Mean = 5.8% 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 < 74 µm

Arsenic IVBA Results and Statistics

Appendix C
FCRM < 74 um EPA Method 1340 IVBA Arsenic Results and Statistics

Table C-1. FCRM < 74 µm Laboratory IVBA Arsenic Results with Prediction Intervals and Confidence Intervals – All Labs

FCRM < 74 EPA Method 1340 Arsenic Results (mg/Kg)							
Laboratory >	**A**	**B**	**C**	**D**	**E**	**F**	**G**
Replicate 1	158	157.822	130.4	161.7	85.6	122.9	143
Replicate 2	155	162.211	134.6	164.8	87.6	120.8	119
Replicate 3	155	165.000	133.1	162.0	79.2	123.2	120
Replicate 4	156	160.274	134.9	160.5	77.8	120.6	120
Replicate 5	158	160.884	132.7	152.4	79.1	118.8	120
Mean	156.4	161.2	133.1	160.3	81.9	121.3	124.4
SD	1.5	2.6	1.8	4.7	4.4	1.8	10.4
RSD	0.97%	1.6%	1.4%	2.9%	5.4%	1.5%	8.4%

Pooled Results (n-1) n=35	
Mean	134.1 mg/Kg
SD	27.2 mg/Kg
RSD	20.3%

FCRM < 74 µm EPA Method 1340 Arsenic – 99th Percentile Prediction Interval (mg/Kg)		
Low 99% PI	**Mean**	**High 99% PI**
58.7	134.1	209.4
± 99% Prediction Interval = 56.2% of the Mean Value		

The range above should be used to determine if a Laboratory EPA Method 1340 IVBA extracted arsenic result is acceptable.

FCRM < 74 EPA Method 1340 Arsenic – 99th Percentile Confidence Interval of the Mean (mg/Kg)		
134.1 = Mean	4.6 = SD of the Mean	3.4% = RSD of the Mean
Low 99% CI	**Mean**	**High 99% CI**
121.5	134.1	146.6
± 99th Percentile of the Confidence Interval of the Mean = 9.4% 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 < 74 um EPA Method 1340 IVBA Arsenic Results and Statistics

Table C-2. FCRM < 74 µm EPA Method 1340 Batch QC Sample Arsenic Results

Laboratory >	A	B	C	D	E	F	G	Mean
Blank Spike Recovery (Range: 85% to 115%)	106.0%	101.2%	108.4%	98.0%	86.4%	90.9%	93.0%	97.7%
FCRM <74 µm Matrix Spike Recovery (Range: 75% to 125%)	99.0%	81.2%	91.9%	98.9%	92.9%	78.7%	98.0%	91.5%
NIST SRM 2710a Arsenic (Mean: 573mg/Kg) (Range: 464 – 681 mg/Kg)	599.0	620.9	533.4	618.4	481.6	467.5	474.0	542.1
Arsenic IVBA NIST SRM 2710a Recovery Based on Nominal Value of 573 mg/Kg	105.0%	108.4%	93.1%	107.9%	84.0%	81.6%	83.0%	94.7%
Control Soil NIST SRM 2710a IVBA Value Based on Leachable Value of 1400 mg/Kg (Acceptance Range 33.1% - 48.4%)	42.8%	44.4%	38.1%	44.2%	34.4%	33.4%	33.9%	38.7%

Values in parentheses are outside the associated control limits.

Table C-3. FCRM EPA Method 1340 Arsenic Results Analysis of Variance

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

SUMMARY

Groups	*Count*	*Sum*	*Mean*	*Variance*
Laboratory A	5	782	156	2
Laboratory B	5	806	161	7
Laboratory C	5	666	133	3
Laboratory D	5	801	160	22
Laboratory E	5	409	82	20
Laboratory F	5	606	121	3
Laboratory G	5	622	124	108

ANOVA

Source of Variation	*SS*	*df*	*MS*	*F*	*P-value*	*F-Crit*
Interlaboratory	24540	6	4090	173	8.49536E-21	2.45
Intralaboratory	662	28	24			
Total	25202	34				

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 C
FCRM < 74 um EPA Method 1340 IVBA Arsenic Results and Statistics

Table C-4. FCRM < 74 µm Arsenic Results and *t*-Test for Laboratory E Extraction Data

FCRM < 74 µm EPA Method 1340 Arsenic Results *t*-Test (mg/Kg)								
Laboratory>	**A**	**B**	**C**	**D**	**F**	**G**	**Laboratory >**	**E**
Replicate 1	158.0	157.8	130.4	161.7	122.9	143	**Replicate 1**	85.6
Replicate 2	155.0	162.2	134.6	164.8	120.8	119	**Replicate 2**	87.6
Replicate 3	155.0	165.0	133.1	162.0	123.2	120	**Replicate 3**	79.2
Replicate 4	156.0	160.3	134.9	160.5	120.6	120	**Replicate 4**	77.8
Replicate 5	158.0	160.9	132.7	152.4	118.8	120	**Replicate 5**	79.1
Mean	156.4	161.2	133.1	160.3	121.3	124.4	**Mean**	81.9
SD	1.5	2.6	1.8	4.7	1.8	10.4	**SD**	4.4
RSD	1.0%	1.6%	1.4%	2.9%	1.5%	8.4%	**RSD**	5.4%

Labs A, B, C, D, F, & G		Percent Difference	Lab E	
	n=30			**n=5**
Mean	142.8	54.2%	**Mean**	81.9
SD	17.8		**SD**	4.4
RSD	12.5%		**RSD**	5.4%

Excel *t*-Test: Two-Sample Assuming Unequal Variances

alpha = 0.05 (1 in 20)

	Labs A, B, C, D, F, & G	**Lab E**
Mean	142.8	81.9
Variance	317.8	19.5
Observations	30	5
Hypothesized Mean Diff.	0	
df	27	
t-Stat	16.0	
P (T ≤ t) (two-tail)	2.66691E-15	
t-Critical (two-tail)	2.05	

The t-Stat value of 16.0 is greater than t-Critical two-tail value of 2.05; 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 0.05 indicates a greater than 95% 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 E can be excluded.

Appendix C
FCRM < 74 um EPA Method 1340 IVBA Arsenic Results and Statistics

Table C-5. FCRM < 74 µm Laboratory IVBA Arsenic Results with Prediction Intervals and Confidence Intervals – Minus Lab E

FCRM < 74 µm EPA Method 1340 Arsenic Results (mg/Kg)							
Laboratory >	**A**	**B**	**C**	**D**	**E**	**F**	**G**
Replicate 1	158	157.822	130.4	161.7		122.9	143
Replicate 2	155	162.211	134.6	164.8		120.8	119
Replicate 3	155	165.000	133.1	162.0		123.2	120
Replicate 4	156	160.274	134.9	160.5		120.6	120
Replicate 5	158	160.884	132.7	152.4		118.8	120
Mean	156.4	161.2	133.1	160.3		121.3	124.4
SD	1.5	2.6	1.8	4.7		1.8	10.4
RSD	0.97%	1.6%	1.4%	2.9%		1.5%	8.4%

Pooled Results (n-1) n=30	
Mean	142.8 mg/Kg
SD	17.8 mg/Kg
RSD	12.5%

FCRM < 74 µm EPA Method 1340 Arsenic – 99th Percentile Prediction Interval (mg/Kg)		
Low 99% PI	**Mean**	**High 99% PI**
92.8	142.8	192.7
± 99% Prediction Interval = 35.0% of the Mean Value		

The range above should be used to determine if a Laboratory EPA Method 9200.2-164 IVBA extracted arsenic result is acceptable.

FCRM < 74 µm EPA Method 1340 Arsenic – 99th Percentile Confidence Interval of the Mean (mg/Kg)		
142.8 = Mean	3.3 = SD of the Mean	2.3% = RSD of the Mean
Low 99% CI	**Mean**	**High 99% CI**
133.8	142.8	151.8
± 99th Percentile of the Confidence Interval of the Mean = 6.3% 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

APPENDIX D

FCRM < 74 µm

Lead IVBA Results and Statistics

Appendix D
FCRM < 74 um EPA Method 1340 IVBA Lead Results and Statistics

Table D-1. FCRM < 74 µm Laboratory IVBA Lead Results with Prediction Intervals and Confidence Intervals – All Labs

FCRM < 74 µm Lead IVBA Results (mg/Kg)							
Laboratory >	**A**	**B**	**C**	**D**	**E**	**F**	**G**
Replicate 1	4840	4644.630	4512	4846.1	4647.4	4155.3	5700*
Replicate 2	4780	4703.114	4541	4827.0	4711.2	4083.1	4854
Replicate 3	4800	4845.000	4687	4890.5	4548.2	4102.3	4981
Replicate 4	4830	4654.213	4556	4877.1	4416.2	4074.8	5128
Replicate 5	4790	4610.539	4580	4584.5	4572.9	4172.5	5047
Mean	4808	4691	4575	4805	4579	4118	5003
SD	25.9	92.0	67.2	125.8	111.4	43.9	115.8
RSD	0.54%	2.0%	1.5%	2.6%	2.4%	1.1%	2.3%

Pooled Results (n-1) n=34	
Mean	4644 mg/Kg
SD	271.6 mg/Kg
RSD	5.8%

FCRM < 74 µm Lead IVBA – 99th Percentile Prediction Interval (mg/Kg)		
Low 99% PI	**Mean**	**High 99% PI**
3891	4644	5397
± 99% Prediction Interval = 16.2% of the Mean Value		
The range above should be used to determine if a Laboratory EPA Method 1340 IVBA extracted lead result is acceptable.		

FCRM < 74 µm Lead IVBA – 99th Percentile Confidence Interval of the Mean (mg/Kg)		
4644 = Mean	46.6 = SD of the Mean	1.0% = RSD of the Mean
Low 99% PI	**Mean**	**High 99% PI**
4517	4644	4771
± 99th Percentile of the Confidence Interval of the Mean = 2.7% of the Mean Value		
The range above can be used to statistically assess the confidence in the accuracy of the mean result.		

"*" = Grubb's Outlier
SD = Standard Deviation
RSD = Relative Standard Deviation
CI = Confidence Interval
PI = Prediction

Appendix D
FCRM < 74 um EPA Method 1340 IVBA Lead Results and Statistics

Table D-2. FCRM < 74 µm EPA Method 1340 Batch QC Sample Lead Results

Laboratory >	A	B	C	D	E	F	G	Mean
Blank Spike Recovery (Nominal: 10 mg/L) (Range: 85% to 115%)	100.0%	99.5%	98.5%	97.6%	107.0%	86.4%	100.0%	98.4%
FCRM <74 µm Matrix Spike Recovery (Nominal: 10 mg/L) (Range: 75% to 125%)	97.0%	85.2%	91.6%	96.7%	105.8%	85.5%	88.0%	92.8%
Control Soil NIST SRM 2710a IVBA Lead Extract Acceptance Range (Nominal: 3440 mg/Kg) (Range: (3096 – 3785 mg/Kg)	3210	3216	(3060)	3350	3381	(2794)	3489	3214
Lead IVBA NIST SRM 2710a Recovery Based on Nominal Value of 3440 mg/Kg	93.0%	93.5%	89.0%	97.4%	98.3%	81.2%	101.0%	93.3%
Control Soil NIST SRM 2710a IVBA Value Based on Leachable Value of 5100 mg/Kg (Acceptance Range 60.7% - 74.2%)	62.9%	63.1%	(60.0%)	65.7%	66.3%	(54.8%)	68.4%	63.0%

Values in parentheses are outside the associated control limit.

Table D-3. FCRM < 74 µm Lead IVBA - Analysis of Variance Results

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

SUMMARY

Groups	*Count*	*Sum*	*Mean*	*Variance*
Laboratory A	5	24040	4808	670
Laboratory B	5	23457	4691	8460
Laboratory C	5	22876	4575	4514
Laboratory D	5	24025	4805	15826
Laboratory E	5	22896	4579	12411
Laboratory F	5	20588	4118	1923
Laboratory G	4	20010	5003	13415

ANOVA

Source of Variation	*SS*	*df*	*MS*	*F*	*P-value*	*F-Crit*
Interlaboratory	2219679	6	369947	46	5.73227E-13	2.46
Intralaboratory	215457	27	7980			
Total	2435136	33				

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 < 74 um EPA Method 1340 IVBA Lead Results and Statistics

Table D-4. FCRM < 74 µm Laboratory IVBA Lead Results with Prediction Intervals and Confidence Intervals – Minus Labs C and F

FCRM < 74 µm Lead IVBA Results (mg/Kg)							
Laboratory >	**A**	**B**	**C**	**D**	**E**	**F**	**G**
Replicate 1	4840	4644.630		4846.1	4647.4		
Replicate 2	4780	4703.114		4827.0	4711.2		4854
Replicate 3	4800	4845.000		4890.5	4548.2		4981
Replicate 4	4830	4654.213		4877.1	4416.2		5128
Replicate 5	4790	4610.539		4584.5	4572.9		5047
Mean	4808	4691		4805	4579		5003
SD	25.9	92.0		125.8	111.4		115.8
RSD	0.54%	2.0%		2.6%	2.4%		2.3%

Pooled Results (n-1) n=24	
Mean	4768 mg/Kg
SD	165.8 mg/Kg
RSD	3.5%

FCRM < 74 µm Lead IVBA – 99th Percentile Prediction Interval (mg/Kg)		
Low 99% PI	**Mean**	**High 99% PI**
4293	4768	5243
± 99% Prediction Interval = 10.0% of the Mean Value		
The range above should be used to determine if a Laboratory EPA Method 1340 IVBA extracted lead result is acceptable.		

FCRM < 74 µm Lead IVBA – 99th Percentile Confidence Interval of the Mean (mg/Kg)		
4768 = Mean	33.8 = SD of the Mean	0.71% = RSD of the Mean
Low 99% PI	**Mean**	**High 99% PI**
4673	4768	4863
± 99th Percentile of the Confidence Interval of the Mean = 2.0% 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

APPENDIX E

FCRM < 250 µm

Method 3050B/3051A Arsenic Results and Statistics

Appendix E
FCRM < 250 um EPA Method 3050B or 3051A Arsenic Results and Statistics

Table E-1. FCRM < 250 µm Laboratory Method 3050B or 3051A Arsenic Results with Prediction Intervals and Confidence Intervals – All Labs

FCRM <250 µm Arsenic 3050B or 3051A Results (mg/Kg)							
Laboratory >	**A**	**B**	**C**	**D**	**E**	**F**	**G**
Replicate 1	750	712.959	680.5	642.3	734.0	694	718
Replicate 2	713	705.167	699.4	665.6	743.5	691	732
Replicate 3	704	691.109	654.6	697.8	757.0	679	779
Replicate 4	727	688.011	656.5	668.8	696.2	727	743
Replicate 5	772	736.663	664.9	693.0	747.3	688	762
Mean	733.2	706.8	671.2	673.5	735.6	695.8	746.8
SD	27.8	19.6	18.8	22.5	23.5	18.3	24.1
RSD	3.8%	2.8%	2.8%	3.3%	3.2%	2.6%	3.2%

Pooled Results (n-1) n=35	
Mean	709.0 mg/Kg
SD	35.1 mg/Kg
RSD	5.0%

FCRM < 250 µm Arsenic – 99th Percentile Prediction Interval (mg/Kg)		
Low 99% PI	**Mean**	**High 99% PI**
611.8	709.0	806.1
± 99% Prediction Interval = 13.7% of the Mean Value		
The range above should be used to determine if a Laboratory EPA Method 3050B or 3051A digested arsenic result is acceptable.		

FCRM < 250 µm Arsenic – 99th Percentile Confidence Interval of the Mean (mg/Kg)		
709.0 = Mean	5.9 = SD of the Mean	0.84% = RSD of the Mean
Low 99% PI	**Mean**	**High 99% PI**
692.8	709.0	725.2
± 99th 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

Appendix E
FCRM < 250 um EPA Method 3050B or 3051A Arsenic Results and Statistics

Table E-2. FCRM < 250 µm EPA Method 3050B or 3051A – Batch QC Sample Arsenic Results

Laboratory >	A	B	C	D	E	F	G	Mean
Blank Spike Recovery (Range: 85% to 115%)	92.0%	93.4%	86.3%	96.0%	103.0%	96.3%	101.0%	95.4%
FCRM <250 µm Matrix Spike % Recovery (Range: 75.0% to 125.0%)	98.0%	96.7%	84.6%	100.4%	107.2%	96.0%	(175.0%)	108.3% *97.2%
NIST SRM 2710a Arsenic (Nominal: 1400 mg/Kg) (Range: 1260 – 1540 mg/Kg)	1490	1431	1374	1508	(1614)	1470	1524	1487
Arsenic NIST SRM 2710a % Recovery Based on Nominal Value of 1400 mg/Kg (Range: 90% - 110%)	106.0%	102.2%	98.1%	107.7%	(115.3%)	105.0%	109.0%	106.2%

Values in parentheses are outside the associated control limits.
*Mean value excluding Laboratory G result.

Table E-3. FCRM < 250 µm Arsenic Method 3050B or 3051A – Analysis of Variance Results

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

SUMMARY

Groups	*Count*	*Sum*	*Mean*	*Variance*
Laboratory A	5	3666	733	772
Laboratory B	5	3534	707	383
Laboratory C	5	3356	671	353
Laboratory D	5	3368	674	507
Laboratory E	5	3678	736	553
Laboratory F	5	3479	696	336
Laboratory G	5	3734	747	583

ANOVA

Source of Variation	*SS*	*df*	*MS*	*F*	*P-value*	*F-Crit*
Interlaboratory	27959	6	4660	9	1.16698E-05	2.45
Intralaboratory	13946	28	498			
Total	41905	34				

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 E
FCRM < 250 um EPA Method 3050B or 3051A Arsenic Results and Statistics

Table E-4. FCRM < 250 µm Laboratory Method 3050B or 3051A Arsenic Results with Prediction Intervals and Confidence Intervals – Minus Lab E

FCRM < 250 µm Arsenic 3050B or 3051A Results (mg/Kg)							
Laboratory >	**A**	**B**	**C**	**D**	**E**	**F**	**G**
Replicate 1	750	712.959	680.5	642.3		694	718
Replicate 2	713	705.167	699.4	665.6		691	732
Replicate 3	704	691.109	654.6	697.8		679	779
Replicate 4	727	688.011	656.5	668.8		727	743
Replicate 5	772	736.663	664.9	693.0		688	762
Mean	733.2	706.8	671.2	673.5		695.8	746.8
SD	27.8	19.6	18.8	22.5		18.3	24.1
RSD	3.8%	2.8%	2.8%	3.3%		2.6%	3.2%

Pooled Results (n-1) n=30	
Mean	704.5 mg/Kg
SD	35.0 mg/Kg
RSD	5.0%

FCRM < 250 µm Arsenic – 99th Percentile Prediction Interval (mg/Kg)		
Low 99% PI	**Mean**	**High 99% PI**
606.4	704.5	802.7
± 99% Prediction Interval = 13.9% of the Mean Value		
The range above should be used to determine if a Laboratory EPA Method 3050B or 3051A digested arsenic result is acceptable.		

FCRM < 250 µm Arsenic – 99th Percentile Confidence Interval of the Mean (mg/Kg)		
704.5 = Mean	6.4 = SD of the Mean	0.91% = RSD of the Mean
Low 99% PI	**Mean**	**High 99% PI**
686.9	704.5	722.2
± 99th 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 F

FCRM < 74 µm

Method 3050B/3051A Arsenic Results and Statistics

Appendix F
FCRM < 74 um EPA Method 3050B or 3051A Arsenic Results and Statistics

Table F-1. FCRM < 74 µm Laboratory Method 3050B or 3051A Arsenic Results with Prediction Intervals and Confidence Intervals – All Labs

FCRM < 74 µm Arsenic 3050B or 3051A Results (mg/Kg)							
Laboratory >	**A**	**B**	**C**	**D**	**E**	**F**	**G**
Replicate 1	753	672.445	689.5	687.0	737.8	708	710
Replicate 2	743	732.328	693.3	722.7	805.0*	676	692
Replicate 3	762	720.315	695.1	679.4	713.1	670	712
Replicate 4	750	710.253	701.4	662.9	723.4	700	723
Replicate 5	736	699.262	713.8	696.3	729.9	716	710
Mean	748.8	706.9	698.6	689.6	726.1	694.0	709.4
SD	9.9	22.8	9.5	22.2	10.5	20.1	11.1
RSD	1.3%	3.2%	1.4%	3.2%	1.4%	2.9%	1.6%

Pooled Results (n-1) n=34	
Mean	710.0 mg/Kg
SD	24.5 mg/Kg
RSD	3.5%

FCRM < 74 µm Arsenic – 99th Percentile Prediction Interval (mg/Kg)		
Low 99% PI	**Mean**	**High 99% PI**
642.0	710.0	778.0
± 99% Prediction Interval = 9.6% of the Mean Value		
The range above should be used to determine if a Laboratory EPA Method 3050B or 3051A digested arsenic result is acceptable.		

FCRM < 74 µm Arsenic – 99th Percentile Confidence Interval of the Mean (mg/Kg)		
710.0 = Mean	4.2 = SD of the Mean	0.59% = RSD of the Mean
Low 99% PI	**Mean**	**High 99% PI**
698.5	710.0	721.5
± 99th Percentile of the Confidence Interval of the Mean = 1.6% of the Mean Value		
The range above can be used to statistically assess the confidence in the accuracy of the mean result.		

"*" = Grubb's Outlier
SD = Standard Deviation
RSD = Relative Standard Deviation
CI = Confidence Interval
PI = Prediction Interval

Appendix F
FCRM < 74 um EPA Method 3050B or 3051A Arsenic Results and Statistics

Table F-2. FCRM < 74 µm EPA Method 3050B or 3051A – Batch QC Sample Arsenic Results

Laboratory >	A	B	C	D	E	F	G	Mean
Blank Spike % Recovery (Range: 85.0% to 115.0%)	93.0%	95.9%	93.4%	95.5%	99.4%	96.7%	99.0%	96.1%
FCRM <74 µm Matrix Spike % Recovery (Range: 75.0% to 125.0%)	94.0%	89.6%	93.1%	98.7%	112.0%	91.0%	(215.0%)	113.3% *96.4%
NIST SRM 2710a Arsenic (Nominal: 1400 mg/Kg) (Range: 1260 – 1540 mg/Kg)	1530	1482	1454	1471	1513	1460	1509	1488
Arsenic NIST SRM 2710a % Recovery Based on Nominal Value of 1400 mg/Kg (Range: 90% - 110%)	109.0%	105.9%	104.0%	105.1%	108.0%	104.3%	108.0%	106.3%

Values in parentheses are outside the associated control limits.
*Mean value excluding Laboratory G result.

Table F-3. FCRM < 74 µm Arsenic Method 3050B or 3051A – Analysis of Variance Results

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

SUMMARY

Groups	*Count*	*Sum*	*Mean*	*Variance*
Laboratory A	5	3744	749	98
Laboratory B	5	3535	707	521
Laboratory C	5	3493	699	91
Laboratory D	5	3448	690	491
Laboratory E	4	2904	726	109
Laboratory F	5	3470	694	404
Laboratory G	5	3547	709	124

ANOVA

Source of Variation	*SS*	*df*	*MS*	*F*	*P-value*	*F-Crit*
Interlaboratory	12603	6	2100	7.83	6.01267E-05	2.46
Intralaboratory	7239	27	268			
Total	19842	33				

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 G

FCRM < 74 µm

Method 3050B/3051A Lead Results and Statistics

Appendix G
FCRM < 74 um EPA Method 3050B or 3051A Lead Results and Statistics

Table G-1. FCRM < 74 µm Laboratory Method 3050B or 3051A Lead Results with Prediction Intervals and Confidence Intervals – All Labs

FCRM < 74 µm Lead 3050B or 3051A Results (mg/Kg)							
Laboratory >	**A**	**B**	**C**	**D**	**E**	**F**	**G**
Replicate 1	6360	6098.824	5936	6384.8	6886.0	6220	6439
Replicate 2	6310	6025.359	5941	6630.1	7017.8	6350	6253
Replicate 3	6500	6250.249	6107	6632.4	6600.3	6320	6503
Replicate 4	6610	6089.168	6138	6597.0	6682.1	6240	6322
Replicate 5	6390	6123.528	6114	6576.8	6934.0	6180	6239
Mean	6434	6117	6047	6564	6824	6262	6351
SD	120.5	82.6	99.9	103.0	175.8	70.9	116.0
RSD	1.9%	1.4%	1.7%	1.6%	2.6%	1.1%	1.8%

Pooled Results (n-1) n=35	
Mean	6371 mg/Kg
SD	271.4 mg/Kg
RSD	4.3%

FCRM < 74 µm Lead – 99th Percentile Prediction Interval (mg/Kg)		
Low 99% PI	**Mean**	**High 99% PI**
5620	6371	7123
± 99% Prediction Interval = 11.8% of the Mean Value		
The range above should be used to determine if a Laboratory EPA Method 3050B or 3051A digested lead result is acceptable.		

FCRM < 74 µm Lead – 99th Percentile Confidence Interval of the Mean (mg/Kg)		
6371 = Mean	45.9 = SD of the Mean	0.72% = RSD of the Mean
Low 99% PI	**Mean**	**High 99% PI**
6246	6371	6497
± 99th Percentile of the Confidence Interval of the Mean = 2.0% 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 G
FCRM < 74 um EPA Method 3050B or 3051A Lead Results and Statistics

Table G-2. FCRM <74 µm EPA Method 3050B or 3051A – Batch QC Sample Lead Results

Laboratory>	A	B	C	D	E	F	G	Mean
Blank Spike % Recovery (Range: 85% to 115%)	97.0%	100.3%	94.4%	98.3%	99.8%	100.1%	101.0%	98.7%
FCRM <74 µm Matrix Spike % Recovery (Range: 75.0% to 125.0%)	88.0%	88.2%	90.1%	110.5%	106.0%	92.3%	(1900.0%)	353.6% *95.9%
NIST SRM 2710a Lead (Nominal: 5100 mg/Kg) (Range: 4590 – 5610 mg/Kg)	4920	4938	4698	5220	5151	5090	5075	5013
Lead NIST SRM 2710a % Recovery Based on Nominal Value of 5100 mg/Kg (Range: 90% - 110%)	96.0%	96.8%	92.1%	102.3%	101.0%	99.8%	100.0%	98.3%

Values in parentheses are outside the associated control limits.
*Mean value excluding Laboratory G result.

Table G-3. FCRM < 74 µm Lead Method 3050B or 3051A – 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	32170	6434	14530
Laboratory B	5	30587	6117	6825
Laboratory C	5	30236	6047	9982
Laboratory D	5	32821	6564	10603
Laboratory E	5	34120	6824	30920
Laboratory F	5	31310	6262	5020
Laboratory G	5	31756	6351	13459

ANOVA

Source of Variation	*SS*	*df*	*MS*	*F*	*P-value*	*F-Crit*
Interlaboratory	2139832	6	356639	27.3	1.76364E-10	2.45
Intralaboratory	365356	28	13048			
Total	2505188	34				

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 H

NIST SRM 2710a

Method 3050B/3051A CLP PT Results and Statistics

Appendix H
NIST SRM 2710a Method 3050B or 3051A CLP PT Results and Statistics

Table H-1. NIST SRM 2710a Arsenic Results Derived from a CLP Event in 2008 using EPA Method 3050B (mg/Kg)

NIST SRM 2710a	Replicate 1	Replicate 2
Lab 1	1422	1426
Lab 2	1364	1359
Lab 3	1554	1297
Lab 4	1585	1616
Lab 5	1490	1467
Lab 6	1335	1341
Mean = 1438 SD = 105.5 RSD = 7.3 n = 12		

APPENDIX I

Summary IVBA (%) Statistics for

NIST SRM 2710a and FCRM

Appendix I
Summary IVBA (%) Statistics for NIST SRM 2710a and FCRM

Table I-1. NIST SRM 2710a Arsenic IVBA Prediction and Confidence Intervals Arsenic IVBA minus Lab F and G, and using EPA Method 3050B Arsenic Results from CLP Performance Testing (PT) Event

NIST SRM 2710a Arsenic IVBA – 99th Percentile Prediction Interval (%)		
Low 99% PI	**Mean**	**High 99% PI**
24.4	38.4	52.5
± 99th Prediction Interval = 36.6% of the Mean Value		
The pooled extraction results n = 30 been divided by the pooled digestion results n = 12 to derive an arsenic IVBA result that includes the variance of both extraction and digestion results.		
IVBA = 38.4 or 38.4%	SD = 5.1	RSD = 13.4%
The range above should be used to determine if a Laboratory arsenic IVBA result is acceptable.		
NIST SRM 2710a Arsenic IVBA – 99th Percentile Confidence Interval of the Mean (%)		
38.4 = Mean	0.8 = SD of the Mean	2.06% = RSD of the Mean
Low 99% CI	**Mean**	**High 99% CI**
36.3	38.4	40.6
± 99th Percentile of the Confidence Interval of the Mean = 5.6% 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 I
Summary IVBA (%) Statistics for NIST SRM 2710a and FCRM

Table I-2. FCRM < 250 µm Arsenic IVBA Prediction and Confidence Intervals Arsenic IVBA minus Labs E, F, and G IVBA Extractions, and Lab E Digestions

FCRM < 250 µm Arsenic IVBA – 99th Percentile Prediction Interval (%)		
Low 99% PI	**Mean**	**High 99% PI**
15.8	21.9	28.0
± 99% Prediction Interval = 27.8% of the Mean Value		
The pooled extraction results n = 20 been divided by the pooled digestion results n = 30 to derive a arsenic IVBA result that includes the variance of both extraction and digestion results.		
IVBA = 21.9 or 21.9%	SD = 2.2	RSD = 10.3%
The range above should be used to determine if a Laboratory arsenic IVBA result is acceptable.		
FCRM < 250 µm Arsenic IVBA – 99th Percentile Confidence Interval of the Mean (%)		
21.9 = Mean	0.3 = SD of the Mean	1.45% = RSD of the Mean
Low 99% CI	**Mean**	**High 99% CI**
21.0	21.9	22.7
± 99th 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 I
Summary IVBA (%) Statistics for NIST SRM 2710a and FCRM

Table I-3. FCRM < 74 µm Arsenic IVBA Prediction and Confidence Intervals
Arsenic IVBA minus Lab E IVBA Extractions

FCRM < 74 µm Arsenic IVBA – 99th Percentile Prediction Interval (%)		
Low 99% PI	**Mean**	**High 99% PI**
13.1	20.1	27.1
± 99% Prediction Interval = 34.7% of the Mean Value		
The pooled extraction results n = 30 been divided by the pooled digestion results n = 35 to derive a arsenic IVBA result that includes the variance of both extraction and digestion results.		
IVBA = 20.1 or 20.1%	SD = 2.6	RSD = 13.0%
The range above should be used to determine if a Laboratory arsenic IVBA result is acceptable.		
FCRM < 74 µm Arsenic IVBA – 99th Percentile Confidence Interval of the Mean (%)		
20.1 = Mean	0.3 = SD of the Mean	1.62% = RSD of the Mean
Low 99% CI	**Mean**	**High 99% CI**
19.2	20.1	21.0
± 99th Percentile of the Confidence Interval of the Mean = 4.3% 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 I
Summary IVBA (%) Statistics for NIST SRM 2710a and FCRM

Table I-4. FCRM < 74 µm Lead IVBA Prediction and Confidence Intervals Lead IVBA minus Lab C and Lab F IVBA Extractions and One (1) Lab G Outlier

FCRM < 74 µm Lead IVBA – 99th Percentile Prediction Interval (%)		
Low 99% PI	**Mean**	**High 99% PI**
63.8	74.8	85.9
± 99% Prediction Interval = 14.8% of the Mean Value		
The pooled extraction results n = 24 been divided by the pooled digestion results n = 35 to derive a lead IVBA result that includes the variance of both extraction and digestion results.		
IVBA = 74.8 or 74.8%	SD = 4.1	RSD = 5.5%
The range above should be used to determine if a Laboratory lead IVBA result is acceptable.		
FCRM < 74 µm Lead IVBA – 99th Percentile Confidence Interval of the Mean (%)		
74.8 = Mean	0.5 = SD of the Mean	0.72% = RSD of the Mean
Low 99% CI	**Mean**	**High 99% CI**
73.4	74.8	76.3
± 99th Percentile of the Confidence Interval of the Mean = 1.9% 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 J

NIST SRM 2710a

Certificate of Analysis

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 J
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.

---

[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 J
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 J
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 J
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 J
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 Branch of Geochemistry Denver, CO, USA	M.G. Adams Z.A. Brown P.L. Lamothe J.E. Taggart S.A. Wilson

REFERENCES

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[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 J
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 K

Laboratory Initial Demonstration of Proficiency (IDP) Forms

Laboratory A

Appendix K
Laboratory Submitted IDP Forms

LAB A Initial Demonstration of Proficiency (IDP) Form For Lead and Arsenic IVBA Round Robin Year 2017, with Digestion of RM for Lead and Arsenic using either EPA Method 3051A or 3050B

(version 01-04-17)

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, Please send completed from to Clifton Jones (Quality Assurance Technical Support Laboratory) Thank You! US (702 895-8713) clifton.jones@cbifederalservices.com

General and Facility Questions

	IVBA	
1	Number of IVBA analyses your facility has performed for lead using the attached IVBA SOP EPA 9200.2-164?	250+
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 9200.2-164 as written? (air controlled temperature is OK) (Yes, or Provide comment Below in 7)	No (See 7)
7	If the answer the question 6 is no, please provide the deviation from the EPA 9200.2-164 Method in the field provided here. Comment- 1. Our typical reporting limits for arsenic and lead by 6010 are 40 and 25 ug/L, respectively. Note that the arsenic reporting limit is above the limit requested for the reagent blank verification analysis (25 ug/L). Our preference is not to perform a separate analysis of the reagent blank by ICPMS. We will attempt to demonstrate arsenic proficiency on ICPAES at 25 ug/L on the days of analysis, but our usual criterion of +/-30% might not be consistently achievable. If necessary, reagent blank results reported below 40 ug/L As may be qualified as estimates. 2. Our citation for 6010 is version D, not C. Specifically, we invoke the option to calibrate with two points, followed by multiple levels of calibration verification standards. This approach allows us to minimize the effects of spectral interferences. 3. We will attempt to use a hotblock digestion system to perform Method 3050B. However, it has been our experience that our digestion tubes do not stand up well to Method 3050B, given its aggressive acid composition and heating protocol. Tubes have sometimes degraded to the point of sample loss through breaches in the tube. If this becomes a frequent occurrence, we will consult the project regarding acceptable alternatives.	

Appendix K
Laboratory Submitted IDP Forms

	Microwave Digestion using 3051A	
1	Total number of analyses your facility has performed for lead and arsenic using the attached EPA Method 3051A.	
2	Will your facility conduct the digestion? (Yes/No)	
3	If the answer to question 2 is no, please provide the name of the Laboratory that will be conducting the digestion. (Lab Name)	
4	Will your facility conduct the digest analysis? (Yes/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)	
6	Will your facility be able to conduct the attached EPA Method 3051A or as written? (Yes, or provide comment Below in 7)	
7	If the answer the question 6 is no, please provide the deviation from EPA Method 3051A in the field provided here. Comment-	

	Block Digestion using Method 3050B	
1	Total number of analyses your facility has performed for lead and arsenic using the attached EPA Method 3050B.	1000+
2	Will your facility conduct the digestion? (Yes/No)	Y
3	If the answer to question 2 is no, please provide the name of the Laboratory that will be conducting the Digestion. (Lab Name)	
4	Will your facility conduct the digest analysis? (Yes/No)	Y
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 EPA Method 3050B as written? (Yes, or provide comment Below in 7)	Y, see comment
7	If the answer the question 6 is no, please provide the deviation from EPA Method 3050B in the field provided here. Comment- As noted above, 3050B using hotblock vessels is not always successful. 3050B as written would allow the use of hotplates/beakers, which would eliminate the problems we have experienced.	

Appendix K
Laboratory Submitted IDP Forms

Apparatus IVBA

1	Does the IVBA apparatus your facility has use air or water as the 37^{O}C thermal conducting/controlling medium. (Air, Water)	water
2	How many bottle positions does your apparatus have?	10
3	Does your usual protocol allow for the pre-incubation of the extraction solution to 37°C before initiation of the IVBA extraction.	yes
4	How do you measure temperature of the controlling apparatus?	With a thermometer checked vs. a NIST thermometer annually
5	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.	
6	If your lab uses air control, what type of temperature control device is being used (i.e., benchtop, upright, or walk-in incubator)?	
7	Does your lab use a pH probe which compensates for temperature (i.e., Automatic Temperature Control (ATC probe))?	yes
8	If not, how does your lab control for temperature when measuring the pH?	

Analytical IVBA

1	Type of analytical instrument typically used for the final Determination (ICP-AES) (ICP-MS) or specify other instrument type.	ICP-AES
2	Please provide the aqueous Method detection limit for the procedure that you currently use for the IVBA Method for both Lead and Arsenic. (ug/L)	We typically analyze samples at 10X to maintain instrument stability. RLs adjusted for dilution: Pb 250 ug/L, As 400 ug/L
3	Name of Control Soil - Reference Material typically used by your facility for the IVBA extraction. (e.g., NIST 2710a or 2711a, or other)	2710A, 2711A
4	Blank spike amount (mg/L) used in your procedure.	Will use 10 mg/L as described in SOW
5	Matrix spike amount (mg/L) used in your procedure.	Will use 10 mg/L as described in SOW

Apparatus Microwave

1	Does the Microwave apparatus at your facility have temperature or pressure control.	
2	How many vessel positions does your apparatus have?	
3	Please provide the manufacturer and model of your microwave apparatus.	
4	What procedure do you use for the microwave power calibration?	
5	When was your microwave apparatus last power calibrated?	

Appendix K
Laboratory Submitted IDP Forms

Apparatus Block Digester

1	What size digestion vessel do you use in mL.	70 mL
2	Does your digestion procedure call for 0.5 g to 50 mL, or 1 g to 100 mL.	0.5 g to 50 mL
3	How many vessel positions does your Block Digester apparatus have?	48
4	Please provide the manufacturer and model of your Block Digester.	CPI Modblock
5	What procedure do you use for the Block Digester temperature verification?	With a thermometer checked vs. a NIST thermometer annually. A Digestion vessel filled with DI water and covered with a ribbed watchglass (with hole for temperature probe) is randomly placed for measurement.

Appendix K
Laboratory Submitted IDP Forms

Table of Batch Lead 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	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 2711a	9.23 mg/L	7.1%	82.9%	
1	02/27/2013	<250 (10X)	<250 (10X)	10.57	106%	101%	4.5%	NIST 2710a (0.5g)	23.1	1.7%RPD	82%	ICP-AES
2	02/27/2013		<250 (10X)	10.57	106%	100%	0.34%	NIST 2710a (0.5g)	22.7		83%	ICP-AES
3	02/27/2013						6.5%					ICP-AES
4	02/27/2013						3.7%					ICP-AES
5	02/27/2013						3.1%					ICP-AES
6	02/27/2013						1.7%					ICP-AES
7												
8	See also attached PDF											
9												
10												

Note Row A presents the quality control acceptance criteria from the USEPA IVBA Method EPA 9200, and Row B provides an example.

Appendix K
Laboratory Submitted IDP Forms

Table of Batch Arsenic 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	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 2710a	4.75 mg/L	7.1%	82.9%	
1	02/27/2013	<450ug/L (10X)	<450ug/L (10X)	2.04	102%	102%	8.0%	NIST 2710a	1.892	3.5%RPD	60%	ICP-AES
2	02/27/2013		<450ug/L (10X)	2.05	103%	101%	0.3%	NIST 2710a	1.960		62%	ICP-AES
3	02/27/2013						9.4%					ICP-AES
4	02/27/2013						4.9%					ICP-AES
5	02/27/2013						1.8%					ICP-AES
6	02/27/2013						2.3%					ICP-AES
7												
8	See also attached PDF											
9												
10												

Note Row A presents the quality control acceptance criteria from the USEPA IVBA Method EPA 9200, and Row B provides an example.

Appendix K
Laboratory Submitted IDP Forms

Table of EPA Block Digester Method 3050B 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 2711a	1300 mg/kg	1078 mg/L	82.9%	
1	02/08/2017	<25 ug/L	94%	2% RPD	LCS	1 mg/L	1.046 mg/L	105%	ICPAES
2	02/08/2017		96%		LCS	1 mg/L	1.034 mg/L	103%	ICPAES
3	02/08/2017				ERA metals in soil 540	102 mg/Kg	102 mg/Kg	100%	ICPAES
4	02/09/2017	<25 ug/L	Native sample too high to calculate spike rec	1% RPD	LCS	1 mg/L	0.913 mg/L	91%	ICPAES
5	02/09/2017				LCS	1 mg/L	0.920 mg/L	92%	ICPAES
6	02/09/2017				ERA metals in soil 540	102 mg/Kg	91.4 mg/Kg	90%	ICPAES
7	02/13/2017	<25 ug/L	108%	4.5% RPD	LCS	1 mg/L	1.058 mg/L	106%	ICPAES
8	02/13/2017		113%		LCS	1 mg/L	1.049 mg/L	105%	ICPAES
9	02/13/2017				ERA metals in soil 540	102 mg/Kg	1.175	117%	ICPAES
10	** NOTE these are hotplate/ beaker results								

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

Appendix K
Laboratory Submitted IDP Forms

Table of EPA Block Digester Method 3050B 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 2710a	1400 mg/kg	1260 mg/kg	90.0%	ICP-MS
1	02/14/2017	<2 ug/L	106%	1% RPD	LCS	500 ug/L	543 ug/L	109%	ICP-MS
2	02/14/2017		107%		LCS	500 ug/L	557 ug/L	111%	ICP-MS
3	02/14/2017				ERA metals in soil 540	114 mg/Kg	126 mg/Kg	111%	ICP-MS
4	02/10/2017	<2 ug/L	106%	2.3% RPD	LCS	500 ug/L	524 ug/L	105%	ICP-MS
5	02/10/2017		105%		LCS	500 ug/L	527 ug/L	105%	ICP-MS
6	02/10/2017				ERA metals in soil 540	114 mg/Kg	122 mg/Kg	107%	ICP-MS
7	02/08/2017	<2 ug/L	106%	3.7%RPD	LCS	500 ug/L	550 ug/L	110%	ICP-MS
8	02/08/2017		110%		LCS	500 ug/L	560 ug/L	112%	ICP-MS
9	02/08/2017				ERA metals in soil 540	114 mg/Kg	128 mg/Kg	112%	ICP-MS
10	** NOTE these are hotplate/ beaker results								

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

Appendix K
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 2711a	1300 mg/kg	1078 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 K
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 2710a	1400 mg/kg	1260 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.

Laboratory B

Appendix K
Laboratory Submitted IDP Forms

LAB B 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 9200.2-164?	* 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 9200.2-164 as written? (air controlled temperature is OK) (Yes, or Provide comment Below in 7)	Yes, see LAB B Lab SOP 256.
7	If the answer the question 6 is no, please provide the deviation from the EPA 9200.2-164 Method in the field provided here. Comment-	

*** The LAB B has performed 9200.2-164 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.**

Appendix K
Laboratory Submitted IDP Forms

	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 B 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. (μg/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 K
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 9200.2-164, and Row B provides an example.

Appendix K
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 K
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.

Laboratory C

Appendix K
Laboratory Submitted IDP Forms

LAB C 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 9200.2-164 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 9200.2-164 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. (μg/L)	50 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)	2711
13	Blank spike amount (mg/L) used in your procedure.	100 μg/L
14	Matrix spike amount (mg/L) used in your procedure.	100 μg/L

Appendix K
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 9200.2-164, 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 D

Appendix K
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)

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^{o}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 K
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 E

Appendix K
Laboratory Submitted IDP Forms

LAB E Initial Demonstration of Proficiency (IDP) Form For Lead and Arsenic IVBA Round Robin Year 2017, with Digestion of RM for Lead and Arsenic using either EPA Method 3051A or 3050B

(version 01-04-17)

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, Please send completed from to Clifton Jones (Quality Assurance Technical Support Laboratory) Thank You! US (702 895-8713) clifton.jones@cbifederalservices.com

General and Facility Questions

	IVBA	
1	Number of IVBA analyses your facility has performed for lead using the attached IVBA SOP EPA 9200.2-164?	> 500
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 9200.2-164 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 9200.2-164 Method in the field provided here. Comment-	

	Microwave Digestion using 3051A	
1	Total number of analyses your facility has performed for lead and arsenic using the attached EPA Method 3051A.	> 1000
2	Will your facility conduct the digestion? (Yes/No)	yes
3	If the answer to question 2 is no, please provide the name of the Laboratory that will be conducting the digestion. (Lab Name)	
4	Will your facility conduct the digest 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 EPA Method 3051A or as written? (Yes, or provide comment Below in 7)	yes
7	If the answer the question 6 is no, please provide the deviation from EPA Method 3051A in the field provided here. Comment-	

Appendix K
Laboratory Submitted IDP Forms

Apparatus IVBA

1	Does the IVBA apparatus your facility has use air or water as the 37^{O}C thermal conducting/controlling medium. (Air, Water)	Air
2	How many bottle positions does your apparatus have?	2 x apparatus; 10 positions each
3	Does your usual protocol allow for the pre-incubation of the extraction solution to 37°C before initiation of the IVBA extraction.	Yes
4	How do you measure temperature of the controlling apparatus?	Apparatus in constant Temp. room
5	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.	Ratek suspension mixer (#RSM6)
6	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 constant temp. room
7	Does your lab use a pH probe which compensates for temperature (i.e., Automatic Temperature Control (ATC probe))?	yes
8	If not, how does your lab control for temperature when measuring the pH?	

Analytical IVBA

1	Type of analytical instrument typically used for the final Determination (ICP-AES) (ICP-MS) or specify other instrument type.	ICP-MS
2	Please provide the aqueous Method detection limit for the procedure that you currently use for the IVBA Method for both Lead and Arsenic. (μg/L)	1 μg/L
3	Name of Control Soil - Reference Material typically used by your facility for the IVBA extraction. (e.g., NIST 2710a or 2711a, or other)	In-house As/Pb soil but have 2711a available
4	Blank spike amount (mg/L) used in your procedure.	1-10
5	Matrix spike amount (mg/L) used in your procedure.	1-10

Apparatus Microwave

1	Does the Microwave apparatus at your facility have temperature or pressure control.	Temp and pressure control
2	How many vessel positions does your apparatus have?	40
3	Please provide the manufacturer and model of your microwave apparatus.	CEM Mars 6
4	What procedure do you use for the microwave power calibration?	Heating of water
5	When was your microwave apparatus last power calibrated?	Every 3 months

Appendix K
Laboratory Submitted IDP Forms

Table of Batch Lead 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	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 2711a	9.23 mg/L	7.1%	82.9%	
1		<10	<10	10.3	103.2	98.9	1.1	In-house (mid)	4.2	3.4	58.3	ICP-MS
2		<10	<10	9.9	98.7	99.4	3.2	In-house (mid)	4.6	2.6	64.1	ICP-MS
3		<10	<10	9.8	97.8	95.6	5.3	In-house (mid)	4.5	4.2	62.8	ICP-MS
4		<10	<10	9.8	98.2	96.9	4.5	In-house (mid)	4.4	0.3	60.9	ICP-MS
5		<10	<10	9.6	95.6	93.8	7.9	In-house (mid)	4.3	2.9	59.2	ICP-MS
6		<10	<10	9.9	99.0	96.0	1.6	In-house (high)	5.5	6.3	97.4	ICP-MS
7		<10	<10	10.3	102.8	95.2	2.4	In-house (high)	5.4	3.0	94.5	ICP-MS
8		<10	<10	10.2	101.9	98.6	9.7	In-house (high)	5.3	1.5	93.3	ICP-MS
9		<10	<10	9.7	97.4	94.7	0.5	In-house (high)	5.3	2.8	94.3	ICP-MS
10		<10	<10	9.8	97.7	92.7	1.3	In-house (high)	5.5	0.9	96.3	ICP-MS

Note Row A presents the quality control acceptance criteria from the USEPA IVBA Method EPA 9200, and Row B provides an example.

Appendix K
Laboratory Submitted IDP Forms

Table of Batch Arsenic 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	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 2710a	4.75 mg/L	7.1%	82.9%	
1		<10	<10	5.1	101.0	89.6	2.3	In-house (low)	0.30	1.6	5.3	ICP-MS
2		<10	<10	4.8	96.4	94.3	4.3	In-house (low)	0.33	0.6	5.9	ICP-MS
3		<10	<10	4.8	95.8	96.2	4.5	In-house (low)	0.27	2.7	4.6	ICP-MS
4		<10	<10	5.0	100.6	93.5	1.2	In-house (low)	0.33	2.9	5.7	ICP-MS
5		<10	<10	5.0	99.2	98.1	1.8	In-house (low)	0.32	4.6	5.6	ICP-MS
6		<10	<10	4.9	98.2	97.8	2.9	In-house (high)	4.95	0.9	82.0	ICP-MS
7		<10	<10	4.9	97.0	95.5	3.4	In-house (high)	5.02	6.7	83.1	ICP-MS
8		<10	<10	4.9	97.8	95.1	5.5	In-house (high)	4.85	5.2	80.3	ICP-MS
9		<10	<10	4.9	98.4	96.2	4.2	In-house (high)	4.89	2.9	81.0	ICP-MS
10		<10	<10	4.9	97.2	92.2	6.0	In-house (high)	5.10	6.2	84.4	ICP-MS

Note Row A presents the quality control acceptance criteria from the USEPA IVBA Method EPA 9200, and Row B provides an example.

Appendix K
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 2711a	1300 mg/kg	1078 mg/L	82.9%	
1		<10	92.3	3.4	NIST 2711	1162	1092	94.0	ICP-MS
2		<10	94.5	5.4	NIST 2711	1162	1188	102.2	ICP-MS
3		<10	96.1	1.8	NIST 2711	1162	1050	90.4	ICP-MS
4		<10	95.0	6.3	NIST 2711	1162	1089	93.7	ICP-MS
5		<10	102.8	3.2	NIST 2711	1162	1090	93.8	ICP-MS
6		<10	98.2	2.7	NIST 2711	1162	1062	91.4	ICP-MS
7		<10	97.1	2.4	NIST 2711	1162	1100	94.7	ICP-MS
8		<10	95.5	3.1	NIST 2711	1162	1155	99.4	ICP-MS
9		<10	93.1	1.6	NIST 2711	1162	1086	93.5	ICP-MS
10		<10	92.8	5.0	NIST 2711	1162	1096	94.3	ICP-MS

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

Appendix K
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 2710a	1400 mg/kg	1260 mg/kg	90.0%	
1		<10	91.8	2.1	NIST 2711	105	101	96.2	ICP-MS
2		<10	95.5	3.8	NIST 2711	105	97	92.4	ICP-MS
3		<10	98.6	3.4	NIST 2711	105	99	94.3	ICP-MS
4		<10	98.7	3.3	NIST 2711	105	102	97.1	ICP-MS
5		<10	98.9	2.5	NIST 2711	105	96	91.1	ICP-MS
6		<10	100.5	0.8	NIST 2711	105	98	93.3	ICP-MS
7		<10	104.0	1.4	NIST 2711	105	103	98.3	ICP-MS
8		<10	95.8	1.2	NIST 2711	105	101	96.0	ICP-MS
9		<10	95.9	1.6	NIST 2711	105	97	92.5	ICP-MS
10		<10	95.8	3.9	NIST 2711	105	96	91.4	ICP-MS

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

Laboratory F

Appendix K
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 7-26-2010)

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 9200.2-164 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 9200.2-164 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. (μg/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 K
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 F-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 F-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 9200.2-164, and Row B provides an example.

M3 Flag on Lab F'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 G

Appendix K
Laboratory Submitted IDP Forms

Lab G 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 9200.2-164 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 9200.2-164 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. (μg/L)**	**0.106 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.**	**10.0 mg/L**
14	**Matrix spike amount (mg/L) used in your procedure.**	**n.a.**

Appendix K
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 9200.2-164, and Row B provides an example.

APPENDIX L

Laboratory Round Robin Study Results

Appendix L
Laboratory Submitted Study Results

Statement of Work for IVBA Round Robin Analysis of Arsenic in NIST SRM 2710a and FCRM <250 µm, and Arsenic and Lead in FCRM <74 µm

(Version 1, February 2017)

SUPPLIED TO LABS A through G

Introduction: The purpose of this Statement of Work (SOW) is to provide specific information and procedures for the analysis and reporting for the (A) EPA Method 1340 for arsenic in Flat Creek Reference Material <250 µm (FCRM <250 µm), arsenic in NIST SRM 2710a, and arsenic and lead in Flat Creek Reference Material <74 µm (FCRM <74 µm), and (B) SW 846 Method 3051A Microwave or Method 3050B Digestion Block for arsenic and lead in FCRM <250 µm and <74 µm. Please read carefully. Analysis of the reference materials must be performed in strict accordance with the EPA SOP EPA 9200.2-164 (updated to include arsenic), which is attached. Any exceptions to the SOP procedures are provided in this Statement of Work.

Analyses Required: The table below presents a summary of analytical requirements, not including QC samples. **The sample turn-around-time is sixty (60) days, including reporting.**

Reference Material	Arsenic IVBA	Arsenic 3050B or 3051A	Lead IVBA	Lead 3050B or 3051A
NIST SRM 2710a	5 Replicates	Previous Study	Previous Study	Previous Study
FCRM <250 µm	5 Replicates	5 Replicates	Previous Study	Previous Study
FCRM <74 µm	5 Replicates	5 Replicates	5 Replicates	5 Replicates
Total Number of Results	15 replicates	10 Replicates	5 Replicates	5 Replicates

Required Reporting Tables: The table below presents the Reporting Tables that are required to be submitted for the Study.

Reference Material	Arsenic IVBA Reporting Tables	Arsenic 3050B or 3051A Reporting Tables	Lead IVBA Reporting Tables	Lead 3050B or 3051A Reporting Tables
NIST SRM 2710a	A3 - A4	Previous Study	Previous Study	Previous Study
FCRM <250 µm	A5 - A6	B3 - B4	Previous Study	Previous Study
FCRM <74 µm	A7 - A8	B5 - B6	A9 - A10	B7 - B8

Sample Receipt: Four small Nalgene (polyethylene) wide-mouth bottles will be provided to each Laboratory in the Round Robin Study. One 30 mL bottle will contain approximately fifteen (15) grams of **Sample** - NIST SRM 2710a. Each Laboratory will be given two (2) 60 mL bottles, each containing approximately 30 grams of sample FCRM <250 µm and FCRM <74 µm, respectively. An additional 30 mL bottle be provided containing approximately 15 grams of **QC Sample** - NIST SRM 2710a to be used as an IVBA/Digestion batch control soil. The bottles should be logged into your usual sample receipt log-in system. These soil materials do not require refrigeration.

Instructions and Reporting: This SOW is divided into two sections: **Section A,** IVBA extraction and reporting using EPA SOP EPA 9200.2-164 (updated to include arsenic IVBA), and **Section B**, Soil Digestion and Reporting using SW-846 Methods 3050B Acid Digestion of Sediments, Sludges, and Soils, or Method 3051A Microwave Assisted Acid Digestion of Sediments, Sludges, Soils, and Oils.

Appendix L
Laboratory Submitted Study Results

Section A: IVBA Extraction and Reporting using EPA SOP EPA 9200.2-164
(updated to include arsenic 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 9200.2-164. **It is imperative for this Study that all of the required quality control samples are prepared and analyzed as specified in the SOP EPA 9200.2-164, which has been updated to include arsenic.** It was also noted during the review of the IDP Forms that some Laboratories uses non-specified acceptance criteria for the quality control parameters. **It is a requirement for this Study** that the acceptance criteria presented in the SOP EPA 9200.2-164, updated to include arsenic, be used for quality control sample results. Below is a table of the required quality control samples and respective control limits, which were derived from Section 9 of the SOP EPA 9200.2-164 (updated to include arsenic). Please note that a designated duplicate sample is **not** required for these analyses.

QC Sample	Control Limits
Reagent Blank	<25 ug/L Pb, As
Method Blank	<50 µg/L Pb, As
Method Blank Spike (10 mg/L) As, Pb	85-115% recovery
Matrix Spike (10 mg/L) As, Pb	75-125% recovery
Duplicate Sample	±20% RPD
Control Soil (NIST SRM 2710a)	±10% RPD

All Quality Control Samples must be run with every batch extraction of the NIST SRM 2710a and FCRM materials. **Each of the RMs (NIST SRM 2710a, FCRM <250 µm, and FCRM <74 µm) must be extracted separately, with 5 replicates in a batch and a complete set of QC samples.**

Sample Preparation: The provided NIST SRM 2710a and FCRM sample materials should be used **as is**. The oven drying and the sieving to less than 150 µm **should not** be performed. Also, riffle splitting **should not** be performed on these SRM materials. The NIST SRM 2710a, FCRM <250 µm, and FCRM <74 µm, must be extracted in separate extraction batches, with five (5) replicate samples for each batch, along with a complete set of associated QC samples for each batch. To insure homogeneity, the SRM bottles **must** be rotated along the x, y, and z axes for at least one minute before sub-sampling for extraction. **Note: All the SRM materials used for the IVBA extraction must be weighted out to 1.00 +/- 0.05 g to the nearest 0.0001and record the exact weight.** The exact weights of each sample should be recorded on the reporting table (second column of Table A3, for example). 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 the 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 A1 below. Again, please note that a designated duplicate sample is not required. The sample extraction should proceed as presented in the SOP EPA 9200.2-164.

Appendix L
Laboratory Submitted Study Results

Table A1. Arsenic IVBA Extraction Batch for Round Robin Analysis		
Extractor Position	**Sample Name**	**Comment**
NONE	Reagent Blank	Do not IVBA-extract
1	Method Blank	
2	RM (Sample 1)	
3	RM (Sample 2)	
4	RM (Sample 3)	
5	RM (Sample 4)	
6	RM (Sample 5)	
7	Control Soil NIST SRM 2710a	
8	LCS (Method Blank Spike)	10 mg/L Pb and As
9	RM Matrix Spike	10 mg/L Pb and As

Sample Filtering and Analysis: Sample filtering and analysis should proceed as indicated in the SOP in Sections 11.10 and 11.13, respectively. The analysis must 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 A2.

Reporting: Tables A3 through A10 **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.

Required IVBA Results Reporting Tables: The table below presents the required Reporting Tables that are to be submitted for the Study. The tables are presented at the end of this section.

Reference Material	Arsenic IVBA Reporting Tables	Lead IVBA Reporting Tables
NIST SRM 2710a	A3 - A4	Previous Study
FCRM <250 µm	A5 - A6	Previous Study
FCRM <74 µm	A7 - A8	A9 - A10

Please complete Results Tables A3 through A10 and transmit via electronic mail to clifton.jones@cbifederalservices.com, followed by 2nd day FedEx shipment of Results Tables A3 through A10, 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.

Thank You!

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

Table A2. Analytical Sequence for a Lead or Arsenic IVBA Extraction Batch		
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	(From Lab Bench)
11	Method Blank	(From Extractor Position 1)
12	RM (Extractor Position 1)	
13	RM (Extractor Position 2)	
14	RM (Extractor Position 3)	
15	RM (Extractor Position 4)	
16	RM (Extractor Position 5)	
17	Control Soil NIST SRM 2710a	(From Extractor Position 7)
18	LCS (Method Blank Spike)	10 mg/L (From Extractor Position 8)
19	RM Matrix Spike	10 mg/L (From Extractor Position 9)
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.	

Section B: Soil Digestion and Reporting using SW-846 Method 3050B Acid Digestion of Sediment, Sludges, and Soils, and Method 3051A Microwave Assisted Acid Digestion of Sediments, Sludges, Soils, and Oils (for complete details see EPA Methods 3050B and 3051A attached).

Summary of Digestion Methods

Soil/Sediment EPA Method 3050B using a Block Digestor: In this Method, the RM samples are digested using acids and hydrogen peroxide employing a heated block digestor as the heat source for the digestion vessels. The soil/sediment digest is allowed to cool and is then allowed to either settle overnight or it is filtered to remove particles. The digest is then brought to volume and analyzed by either inductively coupled plasma-atomic emission spectrometry (ICP-AES) Method 6010C or inductively coupled plasma-mass spectrometry (ICP-MS) EPA Method 6020A. The digestion procedure presented in the EPA Method 3050B was originally written using a hot plate heat source; however, **the Method 3050B alternative heat source block digestor must be used exclusively in this Round Robin Study**. Most, but not all, Laboratories using EPA Method 3050B are using a reduced volume heated block digestion procedure, where the weight of the soil/sediment samples and the final volume of digestion reagents have been reduced to one-half of the amounts stated in the procedure. **When using the block digestor, use either a full sample (1 gram) to final volume 100 mL, or a one-half reduced sample (0.5 gram) to 50 mL final volume digestion procedure.**

Microwave Digestion EPA Method 3051A: The RM samples are digested and/or dissolved in concentrated nitric acid, or alternatively, concentrated nitric acid and concentrated hydrochloric

acid using microwave heating with a suitable Laboratory microwave unit. The sample and acid(s) are placed in a fluorocarbon polymer (PFA or TFM) or quartz microwave vessel or vessel liner. The vessel is sealed and heated in the microwave unit for a specified period of time. After cooling, the vessel contents are filtered, centrifuged, or allowed to settle and then diluted to volume and analyzed by the appropriate determinative Method.

Method 3051A is provided as an alternative to EPA Method 3050B. This Method provides options for improving the performance for certain analytes, such as antimony, iron, aluminum, and silver by the addition of hydrochloric acid, when necessary. It is intended to provide a rapid multi-element acid digestion or dissolution prior to analysis so that decisions can be made about materials and site cleanup levels, the need for TCLP testing of a waste (see Method 1311), and whether a BDAT process is providing acceptable performance. Digests produced by the Method are suitable for analysis by inductively coupled plasma-atomic emission spectrometry (ICP-AES) and inductively coupled plasma-mass spectrometry (ICP-MS) according to EPA Method 6010C and 6020A, respectively. However, the addition of HCl may limit the quantitation Methods, or increase the difficulties of quantitation with some techniques. Due to the rapid advances in microwave technology, consult your manufacturer's recommended instructions for guidance on their microwave digestion system.

Required Quality Assurance/Quality Control: Using either EPA Method 3050B or 3051A (attached) as directed, the RM samples (5 replicates) shall be digested in separate batches. QA/QC procedures listed in EPA Method 3050B or 3051A shall be strictly followed, and any deviations from this Method should be reported prior to digestion of the RMs, and written confirmation of acceptance of these changes shall be required by contacting Clifton Jones at clifton.jones@cbifederalservices.com .

It is imperative for this Study that all of the required quality control samples are prepared and analyzed as specified in the SOP EPA 9200.2-164, which has been updated to include arsenic. Below is a table of the required quality control samples and the control limits for this Round Robin Study.

QC Sample	Control Limits
Reagent Blank	<25 ug/L As, Pb
Blank Spike (10 mg/L) As, Pb	85-115% recovery
Matrix Spike (10 mg/L) As, Pb	75-125% recovery
Control Soil (NIST SRM 2710a)	±10% RPD

All quality Control Samples must be run with every 3050B or 3051A digestion batch of the RM. **Each of the RMs (SRM 2710a, FCRM <250 µm, and FCRM <74 µm) must be digested separately, in 5 replicate batches with a complete set of QC samples.**

EPA Method 3050B or 3051A Sample Preparation: The provided NIST SRM 2710a and FCRM sample materials should be used **as is**. The oven drying and the sieving to less than 150 µm **should not** be performed. Also, riffle splitting **should not** be performed on these SRM materials. Each of the RMs must be digested in a single digestion batch, with five (5) replicate RM samples, along with a complete associated QC samples for each batch. To insure homogeneity, the NIST SRM 2710a and FCRM bottles **must** be rotated along the x, y, and z axes for at least one minute before sub-sampling for digestion.
For the EPA Method 3050B block digestion, reduced to one-half volume, the NIST SRM 2710a and FCRM materials used in this Study must be weighed out to 0.50 +/- 0.025 and recorded to nearest 0.0001 g , and a final volume of 50 mL must be used. For a full

volume block digestion, weigh out to 1.00 grams +/- 0.05 g and record to the nearest 0.0001 g, and a final volume of 100 mL must be used.

For the EPA Microwave Method 3051A, the NIST SRM 27010a and FCRM materials used in this Study must be weighed out to 0.50 +/- 0.025 and recorded to nearest 0.0001 g.

For the EPA Method 3051A microwave digestion, Section 11.3.2, use the addition of 9 ± 0.1 mL concentrated nitric acid and 3 ± 0.1 mL concentrated hydrochloric acid to the microwave digestion vessel. For the EPA 3051A, the final volume should be 100 mL.

The list of samples for the EPA 3050B block digestion, or EPA 3051A microwave digestion batch, that **must** be used for this Study are provided in Table B1 below. The sample digestion should proceed as presented in either EPA Method 3050B block digestion, or EPA Method 3051A microwave digestion.

Table B1. Method 3050B or 3051A Microwave Digestion Batch of RM

Sample No.	Sample Name	Comment
1	Method Blank	
2	RM (Sample 1)	
3	RM (Sample 2)	
4	RM (Sample 3)	
5	RM (Sample 4)	
6	RM (Sample 5)	
7	Control Soil NIST SRM 2710a	
8	LCS (Method Blank Spike)	10 mg/L Pb, As
9	RM Matrix Spike	10 mg/L Pb, As

Sample Filtering and Analysis:

EPA Method 3050B: Sample filtering and analysis should proceed as indicated in the Section 7.2.4 for ICP-MS analysis, or as described in Section 7.4 for ICP-AES analysis.

EPA Method 3051A: Sample filtering and analysis should proceed as indicated in the Section 11.3.7.

The analysis must 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 B2 below.

Reporting: Tables B3 through B8 **must** be used for reporting the 3050B or 3051A analysis results for the NIST SRM 2710a and FCRM, 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.

Required Reporting Tables: The Table below presents the required Digestion Reporting Tables that **must** be submitted for the Study. The Tables are presented at the end of this section.

Appendix L
Laboratory Submitted Study Results

Reference Material	Arsenic 3050B or 3051A Reporting Tables	Lead 3050B or 3051A Reporting Tables
NIST SRM 2710a	Previous Study	Previous Study
FCRM <250 µm	B3 - B4	Previous Study
FCRM <74 µm	B5 - B6	B7 - B8

Please complete the Results Tables B3 through B8 and transmit them to Clifton Jones at clifton.jones@cbifederalservices.com, followed by a 2nd day FedEx shipment of the Results Tables B3 - B10, 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 digestion and analysis with the data submission.

Thank You!

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

Table B2. Analytical Sequence for 3050B or 3051A Digestion Batch for the 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.	
11 <Proxy Position no.	Method Blank	
12	RM (Sample 1)	
13	RM (Sample 2)	
14	RM (Sample 3)	
15	RM (Sample 4)	
16	RM (Sample 5)	
17	Control Soil NIST SRM 2710a	
18	LCS (Method Blank Spike)	10 mg/L (Pb, As)
19	RM Matrix Spike	10 mg/L (Pb, As)
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.	

Laboratory A

Appendix L
Laboratory Submitted Study Results

LABORATORY A, Table A3. IVBA Extraction Batch Results, (NIST SRM 2710a): Arsenic (As)			
Lab Performing Extraction	Laboratory A	Analyst Performing Extraction	[redacted]
Lab Performing Analysis	Laboratory A	Analyst Performing Analysis	[redacted]
Instrument Type: ICP-MS or ICP-AES	ICP-AES	Instrument Method Detection Limit (MDL) (ug/L)	5.3
Extraction Date	5/18/2017	Extraction As Standard Manufacturer and Lot #	CPI; #1106923
Analysis Date(s)	5/19/2017	Analysis As Standard Manufacturer and Lot #	CPI; # 16C023
Initial Calibration Verification Standard Source and Lot #	High Purity lot# 1629316	Interference Check Sample Source and Lot #	INFCS-4 High Purity Lot# 1534806; Na-10,000mg/L High Purity lot# 1609122

Sample Name	Mass of Sample Material (g)[1]	Volume of Extraction Fluid (mL)[2]	Dilution Factor	Instrument As Result for the Analytical Solution (mg/L)	Results for IVBA extractable As in sample material (mg/kg)
Reagent Blank		100	10	0.0032	<25
Method Blank		100	10	0.0025	<25
NIST SRM 2710a (Extraction 1)	1.0005	100	10	0.5641	564
NIST SRM 2710a (Extraction 2)	1.0002	100	10	0.5637	564
NIST SRM 2710a (Extraction 3)	1.0006	100	10	0.5671	567
NIST SRM 2710a (Extraction 4)	1.0009	100	10	0.5690	568
NIST SRM 2710a (Extraction 5)	1.0009	100	10	0.5746	574
Control Soil – NIST SRM 2710a	1.0002	100	10	0.5667	567
LCS (Method Blank Spike)		102	10	1.029	1050
NIST SRM 2710a Matrix Spike	1.0004	100	10	10.66	107

1 – Measure 1.00 +/- 0.05 g to the nearest 0.0001g, enter exact mass in table. Extraction mass should not be reduced. If your lab requires more reference material, please contact Clifton Jones at clifton.jones@cbifederalservices.com

2 – Add 100 +/- 0.5 mL of 0.4M glycine, pH 1.5, delivered via graduated cylinder or automated dispenser.
Results of IVBA Extractable As in sample material (mg/kg) = [Instrument As Results for Analytical Solution (mg/L) x Dilution Factor x 100 mL extraction fluid x (1 L/1000 mL)]/[mass of sample material (g) x (1 kg/1000 g)].

Appendix L
Laboratory Submitted Study Results

LABORATORY A, Table A4. IVBA Extraction Batch Spiked Blank, Spiked Sample, and Control Soil Results for RM Batch (NIST SRM 2710a): Arsenic (As)	
Laboratory Performing Extraction	Laboratory A
Laboratory Performing Analysis	Laboratory A
Method Blank Result (mg/L)	<0.25
LCS (Method Blank Spike) Result (mg/L)	10.50
LCS (Method Blank Spike) Percent Recovery	105
Average (5) Result RM NIST SRM 2710a (mg/L)	5.68
RM NIST SRM 2710a Matrix Spike Result (mg/L)	106.6
RM NIST SRM 2710a Matrix Spike Percent Recovery	101
Control Soil NIST SRM 2710a Result (mg/Kg)	567
Control Soil NIST SRM 2710a IVBA Extract Acceptance Range - Arsenic (mg/Kg)	(As) 464 – 681 mg/Kg
Control Soil NIST SRM 2710a IVBA Arsenic Nominal Value (mg/Kg)	(As) 573 mg/Kg
Control Soil NIST SRM 2710a Percent Recovery (%)	99

Appendix L
Laboratory Submitted Study Results

LABORATORY A, Table A5. IVBA Extraction Batch Results, (FCRM <250 µm): Arsenic (As)			
Lab Performing Extraction	Laboratory A	Analyst Performing Extraction	[redacted]
Lab Performing Analysis	Laboratory A	Analyst Performing Analysis	[redacted]
Instrument Type: ICP-MS or ICP-AES	ICP-AES	Instrument Method Detection Limit (MDL) (ug/L)	5.3
Extraction Date	5/18/2017	Extraction As Standard Manufacturer and Lot #	CPI; #1106923
Analysis Date(s)	5/19/2017	Analysis As Standard Manufacturer and Lot #	CPI; # 16C023
Initial Calibration Verification Standard Source and Lot #	High Purity lot# 1629316	Interference Check Sample Source and Lot #	INFCS-4 High Purity Lot# 1534806; Na-10,000mg/L High Purity lot# 1609122

Sample Name	Mass of Sample Material (g)[1]	Volume of Extraction Fluid (mL)[2]	Dilution Factor	Instrument As Result for the Analytical Solution (mg/L)	Results for IVBA extractable As in sample material (mg/kg)
Reagent Blank		100	10	0.0013	<25
Method Blank		100	10	0.0018	<25
FCRM <250 µm (Extraction 1)	1.0002	100	10	0.1646	165
FCRM <250 µm (Extraction 2)	1.0006	100	10	0.1578	158
FCRM <250 µm (Extraction 3)	1.0003	100	10	0.1545	154
FCRM <250 µm (Extraction 4)	1.0006	100	10	0.1542	154
FCRM <250 µm (Extraction 5)	1.0003	100	10	0.1596	160
Control Soil – NIST SRM 2710A	1.0004	100	10	0.6041	604
LCS (Method Blank Spike)		102	10	1.061	1080
FCRM <250 µm Matrix Spike	1.0006	100	10	10.69	107

1 – Measure 1.00 +/- 0.05 g to the nearest 0.0001g, enter exact mass in table. Extraction mass should not be reduced. If your lab requires more reference material, please contact Clifton Jones at clifton.jones@cbifederalservices.com.

2 – Add 100 +/- 0.5 mL of 0.4M glycine, pH 1.5, delivered via graduated cylinder or automated dispenser.
Results of IVBA Extractable As in sample material (mg/kg) = [Instrument As Results for Analytical Solution (mg/L) x Dilution Factor x 100 mL extraction fluid x (1 L/1000 mL)]/[mass of sample material (g) x (1 kg/1000 g)].

Appendix L
Laboratory Submitted Study Results

LABORATORY A, Table A6. IVBA Extraction Batch Spiked Blank, Spiked Sample, and Control Soil Results for RM Batch (FCRM <250 µm): Arsenic (As)	
Laboratory Performing Extraction	Laboratory A
Laboratory Performing Analysis	Laboratory A
Method Blank Result (mg/L)	<0.25
LCS (Method Blank Spike) Result (mg/L)	10.80
LCS (Method Blank Spike) Percent Recovery	108
Average (5) Result FCRM <250 µm (mg/L)	1.58
FCRM <250 µm Matrix Spike Result (mg/L)	106.9
FCRM <250 µm Matrix Spike Percent Recovery	105
Control Soil NIST SRM 2710a Result (mg/Kg)	604
Control Soil NIST SRM 2710a IVBA Extract Acceptance Range - Arsenic (mg/Kg)	(As) 464 – 681 mg/Kg
Control Soil NIST SRM 2710a IVBA Arsenic Nominal Value (mg/Kg)	(As) 573 mg/Kg
Control Soil NIST SRM 2710a Percent Recovery (%)	105

Appendix L
Laboratory Submitted Study Results

LABORATORY A, Table A7. IVBA Extraction Batch Results, (FCRM <74 µm): Arsenic (As)					
Lab Performing Extraction	Laboratory A		Analyst Performing Extraction	[redacted]	
Lab Performing Analysis	Laboratory A		Analyst Performing Analysis	[redacted]	
Instrument Type: ICP-MS or ICP-AES	ICP-AES		Instrument Method Detection Limit (MDL) (ug/L)	5.3	
Extraction Date	5/18/2017		Extraction As Standard Manufacturer and Lot #	CPI; #1106923	
Analysis Date(s)	5/19/2017		Analysis As Standard Manufacturer and Lot #	CPI; # 16C023	
Initial Calibration Verification Standard Source and Lot #	High Purity lot# 1629316		Interference Check Sample Source and Lot #	INFCS-4 High Purity Lot# 1534806; Na-10,000mg/L High Purity lot# 1609122	
Sample Name	Mass of Sample Material (g)[1]	Volume of Extraction Fluid (mL)[2]	Dilution Factor	Instrument As Result for the Analytical Solution (mg/L)	Results for IVBA extractable As in sample material (mg/kg)
Reagent Blank		100	10	0.0021	<25
Method Blank		100	10	0.0026	<25
FCRM <74 µm (Extraction 1)	1.0004	100	10	0.1578	158
FCRM <74 µm (Extraction 2)	1.0004	100	10	0.1547	155
FCRM <74 µm (Extraction 3)	1.0004	100	10	0.1554	155
FCRM <74 µm (Extraction 4)	1.0003	100	10	0.1565	156
FCRM <74 µm (Extraction 5)	1.0001	100	10	0.1578	158
Control Soil – NIST SRM 2710A	1.0004	100	10	0.5995	599
LCS (Method Blank Spike)		102	10	1.037	1060
FCRM <74 µm Matrix Spike	1.0007	100	10	10.08	101

1 – Measure 1.00 +/- 0.05 g to the nearest 0.0001g, enter exact mass in table. Extraction mass should not be reduced. If your lab requires more reference material, please contact Clifton Jones at clifton.jones@cbifederalservices.com.

2 – Add 100 +/- 0.5 mL of 0.4M glycine, pH 1.5, delivered via graduated cylinder or automated dispenser.
Results of IVBA Extractable As in sample material (mg/kg) = [Instrument As Results for Analytical Solution (mg/L) x Dilution Factor x 100 mL extraction fluid x (1 L/1000 mL)]/[mass of sample material (g) x (1 kg/1000 g)].

Appendix L
Laboratory Submitted Study Results

LABORATORY A, Table A8. IVBA Extraction Batch Spiked Blank, Spiked Sample, and Control Soil Results for RM Batch (FCRM <74 µm): Arsenic (As)	
Laboratory Performing Extraction	Laboratory A
Laboratory Performing Analysis	Laboratory A
Method Blank Result (mg/L)	<0.25
LCS (Method Blank Spike) Result (mg/L)	10.60
LCS (Method Blank Spike) Percent Recovery	106
Average (5) Result FCRM <250 µm (mg/L)	1.56
FCRM <74 µm Matrix Spike Result (mg/L)	100.8
FCRM <74 µm Matrix Spike Percent Recovery	99
Control Soil NIST SRM 2710a Result (mg/Kg)	599
Control Soil NIST SRM 2710a IVBA Extract Acceptance Range - Arsenic (mg/Kg)	(As) 464 – 681 mg/Kg
Control Soil NIST SRM 2710a IVBA Arsenic Nominal Value (mg/Kg)	(As) 573 mg/Kg
Control Soil NIST SRM 2710a Percent Recovery (%)	105

Appendix L
Laboratory Submitted Study Results

LABORATORY A, Table A9. IVBA Extraction Batch Results, (FCRM <74 µm): Lead (Pb)					
Lab Performing Extraction	Laboratory A		Analyst Performing Extraction	[redacted]	
Lab Performing Analysis	Laboratory A		Analyst Performing Analysis	[redacted]	
Instrument Type: ICP-MS or ICP-AES	ICP-AES		Instrument Method Detection Limit (MDL) (ug/L)	1.1	
Extraction Date	5/18/2017		Extraction Pb Standard Manufacturer and Lot #	CPI; # 16E068	
Analysis Date(s)	5/19/2017		Analysis Pb Standard Manufacturer and Lot #	CPI; # 16F068	
Initial Calibration Verification Standard Source and Lot #	High Purity Lot# 1629316		Interference Check Sample Source and Lot #	INFCS-4 High Purity Lot# 1534806; Na-10,000mg/L High Purity lot# 1609122	
Sample Name	Mass of Sample Material (g)[1]	Volume of Extraction Fluid (mL)[2]	Dilution Factor	Instrument Pb Result for the Analytical Solution (mg/L)	Results for IVBA extractable Pb in sample material (mg/kg)
Reagent Blank		100	10	0.0006	<25
Method Blank		100	10	0.0001	<25
FCRM <74 µm (Extraction 1)	1.0004	100	10	4.842	4840
FCRM <74 µm (Extraction 2)	1.0004	100	10	4.780	4780
FCRM <74 µm (Extraction 3)	1.0004	100	10	4.799	4800
FCRM <74 µm (Extraction 4)	1.0003	100	10	4.833	4830
FCRM <74 µm (Extraction 5)	1.0001	100	10	4.788	4790
Control Soil – NIST SRM 2710A	1.0004	100	10	3.208	3210
LCS (Method Blank Spike)		102	10	0.9837	1000
FCRM <74 µm Matrix Spike	1.0007	100	10	14.47	14500

1 – Measure 1.00 +/- 0.05 g to the nearest 0.0001g, enter exact mass in table. Extraction mass should not be reduced. If your lab requires more reference material, please contact Clifton Jones at clifton.jones@cbifederalservices.com.

2 – Add 100 +/- 0.5 mL of 0.4M glycine, pH 1.5, delivered via graduated cylinder or automated dispenser.
Results of IVBA Extractable Pb in sample material (mg/kg) = [Instrument Pb Results for Analytical Solution (mg/L) x Dilution Factor x 100 mL extraction fluid x (1 L/1000 mL)]/[mass of sample material (g) x (1 kg/1000 g)].

Appendix L
Laboratory Submitted Study Results

LABORATORY A, Table A10. IVBA Extraction Batch Spiked Blank, Spiked Sample, and Control Soil Results for RM Batch (FCRM <74 µm): Lead (Pb)	
Laboratory Performing Extraction	Laboratory A
Laboratory Performing Analysis	Laboratory A
Method Blank Result (mg/L)	<0.25
LCS (Method Blank Spike) Result (mg/L)	10.03
LCS (Method Blank Spike) Percent Recovery	100
Average (5) Result FCRM <250 µm (mg/L)	48.1
FCRM <74 µm Matrix Spike Result (mg/L)	145.0
FCRM <74 µm Matrix Spike Percent Recovery	97
Control Soil NIST SRM 2710a Result (mg/Kg)	3210
Control Soil NIST SRM 2710a IVBA Extract Acceptance Range – Lead (mg/Kg)	(Pb) 3096 – 3785 mg/Kg
Control Soil NIST SRM 2710a IVBA Lead Nominal Value (mg/Kg)	(Pb) 3440 mg/Kg
Control Soil NIST SRM 2710a Percent Recovery (%)	93

Appendix L
Laboratory Submitted Study Results

LABORATORY A, Table B3. Method 3050B or 3051A Digestion Batch Results, (FCRM <250 µm): Arsenic (As)					
Lab Performing Digestion	Laboratory A			Analyst Performing Digestion	[redacted]
Lab Performing Analysis	Laboratory A			Analyst Performing Analysis	[redacted]
Instrument Type: ICP-MS or ICP-AES	ICP-AES			Instrument Method Detection Limit (MDL) (ug/L)	5.3
Digestion Date	04/28/2017			Digestion As Standard Manufacturer and Lot #	CPI lot# 14L093
Analysis Date(s)	05/12/2017			Analysis As Standard Manufacturer and Lot #	CPI lot# 16C023
Initial Calibration Verification Standard Source and Lot #	High Purity Lot# 1629316			Interference Check Sample Source and Lot #	High Purity (INFCS-4) lot#1534806; High Purity (10,000 mg/L Na) lot # 1609122
Sample Name	Mass of Sample Material (g)[1]	Volume of Digestion Fluid (mL)	Dilution Factor	Instrument As Result for the Analytical Solution (mg/L)	Results from digested Arsenic in sample material (mg/kg)[2]
Method Blank	-	50	1	-.0025	<2.5
FCRM <250 µm (Digestion 1)	0.5034	50	5	1.510	750
FCRM <250 µm (Digestion 2)	0.5058	50	5	1.443	713
FCRM <250 µm (Digestion 3)	0.5044	50	5	1.421	704
FCRM <250 µm (Digestion 4)	0.5019	50	5	1.459	727
FCRM <250 µm (Digestion 5)	0.5056	50	5	1.562	772
Control Soil – NIST SRM 2710a	0.4992	50	5	2.978	1490
LCS (Method Blank Spike)	-	50	1	9.237	924
FCRM <250 µm Matrix Spike	0.5023	50	5	3.418	1700

1 – Measure 1.00 +/- 0.05 g to the nearest 0.0001g, enter exact mass in table, or if one-half reduced volume digestion is performed, measure 0.50 +/- 0.05 g to the nearest 0.0001g. If your lab requires more reference material, please contact Clifton Jones at clifton.jones@cbifederalservices.com.

2 – Arsenic in sample material (mg/kg) = [Instrument As Results for Analytical Solution (mg/L) x Dilution Factor x 100 mL Digest Final Volume x (1 L/1000 mL)]/[mass of sample material (g) x (1 kg/1000 g)].

Appendix L
Laboratory Submitted Study Results

LABORATORY A, Table B4. Method 3050B or 3051A Digestion Spiked Blank, and Spiked Sample Results for (FCRM <250 µm): Arsenic (As)	
Laboratory Performing Digestion	Laboratory A
Laboratory Performing Analysis	Laboratory A
Blank Spike Result (mg/L)	9.24
Blank Spike Percent Recovery	92%
Average (5) Result RM (mg/L)	7.33
RM Matrix Spike Result (mg/L)	17.1
RM Matrix Spike Percent Recovery	98%
Control Soil NIST SRM 2710a Result (mg/Kg)	1490
Control Soil NIST SRM 2710a Digestion Acceptance Range - Arsenic (mg/Kg)	(As) 1260 -1540 mg/kg
Control Soil NIST SRM 2710a Arsenic Nominal Value (mg/Kg)	(As) 1400 mg/kg
Control Soil NIST SRM 2710a Percent Recovery (%)	106%

Appendix L
Laboratory Submitted Study Results

LABORATORY A, Table B5. Method 3050B or 3051A Digestion Batch Results, (FCRM <74 µm): Arsenic (As)					
Lab Performing Digestion	Laboratory A		Analyst Performing Digestion	[redacted]	
Lab Performing Analysis	Laboratory A		Analyst Performing Analysis	[redacted]	
Instrument Type: ICP-MS or ICP-AES	ICP-AES		Instrument Method Detection Limit (MDL) (ug/L)	5.3	
Digestion Date	04/28/2017		Digestion As Standard Manufacturer and Lot #	CPI lot# 14L093	
Analysis Date(s)	05/15/2017		Analysis As Standard Manufacturer and Lot #	CPI lot# 16C023	
Initial Calibration Verification Standard Source and Lot #	High Purity Lot# 1629316		Interference Check Sample Source and Lot #	High Purity (INFCS-4) lot#1534806; High Purity (10,000 mg/L Na) lot # 1609122	
Sample Name	Mass of Sample Material (g)[1]	Volume of Digestion Fluid (mL)	Dilution Factor	Instrument As Result for the Analytical Solution (mg/L)	Results from digested Arsenic in sample material (mg/kg)[2]
Method Blank	-	50.9	1	-.0038	<2.5
FCRM <74 µm (Digestion 1)	0.5003	50	5	1.506	753
FCRM <74 µm (Digestion 2)	0.4999	50	5	1.485	743
FCRM <74 µm (Digestion 3)	0.5004	50	5	1.525	762
FCRM <74 µm (Digestion 4)	0.4992	50	5	1.497	750
FCRM <74 µm (Digestion 5)	0.5047	50	5	1.485	736
Control Soil – NIST SRM 2710a	0.5140	50	5	3.155	1530
LCS (Method Blank Spike)	-	50	1	9.338	934
FCRM <74 µm Matrix Spike	0.5058	50	5	3.385	1670

1 – Measure 1.00 +/- 0.05 g to the nearest 0.0001g, enter exact mass in table, or if one-half reduced volume digestion is performed, measure 0.50 +/- 0.05 g to the nearest 0.0001g. If your lab requires more reference material, please contact Clifton Jones at clifton.jones@cbifederalservices.com.

2 – Arsenic in sample material (mg/kg) = [Instrument As Results for Analytical Solution (mg/L) x Dilution Factor x 100 mL Digest Final Volume x (1 L/1000 mL)]/[mass of sample material (g) x (1 kg/1000 g)].

Appendix L
Laboratory Submitted Study Results

LABORATORY A, Table B6. Method 3050B or 3051A Digestion Spiked Blank, and Spiked Sample Results for (FCRM <74 µm): Arsenic (As)	
Laboratory Performing Digestion	Laboratory A
Laboratory Performing Analysis	Laboratory A
Blank Spike Result (mg/L)	9.34
Blank Spike Percent Recovery	93%
Average (5) Result RM (mg/L)	7.49
RM Matrix Spike Result (mg/L)	16.9
RM Matrix Spike Percent Recovery	94%
Control Soil NIST SRM 2710a Result (mg/Kg)	1530
Control Soil NIST SRM 2710a Digestion Acceptance Range - Arsenic (mg/Kg)	(As) 1260 -1540 mg/kg
Control Soil NIST SRM 2710a Arsenic Nominal Value (mg/Kg)	(As) 1400 mg/kg
Control Soil NIST SRM 2710a Percent Recovery (%)	109%

Appendix L
Laboratory Submitted Study Results

LABORATORY A, Table B7. Method 3050B or 3051A Digestion Batch Results, (FCRM <74 µm): Lead (Pb)					
Lab Performing Digestion	Laboratory A		Analyst Performing Digestion	[redacted]	
Lab Performing Analysis	Laboratory A		Analyst Performing Analysis	[redacted]	
Instrument Type: ICP-MS or ICP-AES	ICP-AES		Instrument Method Detection Limit (MDL) (ug/L)	1.1	
Digestion Date	04/28/2017		Digestion Pb Standard Manufacturer and Lot #	CPI lot# 16E068	
Analysis Date(s)	05/15/2017		Analysis Pb Standard Manufacturer and Lot #	CPI lot# 16F068	
Initial Calibration Verification Standard Source and Lot #	High Purity lot# 1629316		Interference Check Sample Source and Lot #	High Purity (INFCS-4) lot#1534806; High Purity (10,000 mg/L Na) lot # 1609122	
Sample Name	Mass of Sample Material (g)[1]	Volume of Digestion Fluid (mL)	Dilution Factor	Instrument Pb Result for the Analytical Solution (mg/L)	Results from digested Lead in sample material (mg/kg)[2]
Method Blank	-	50.9	1	-.0005	<2.5
FCRM <74 µm (Digestion 1)	0.5003	50	5	12.73	6360
FCRM <74 µm (Digestion 2)	0.4999	50	5	12.62	6310
FCRM <74 µm (Digestion 3)	0.5004	50	5	13.01	6500
FCRM <74 µm (Digestion 4)	0.4992	50	5	13.19	6610
FCRM <74 µm (Digestion 5)	0.5047	50	5	12.91	6390
Control Soil – NIST SRM 2710a	0.5140	50	5	10.12	4920
LCS (Method Blank Spike)	-	50	1	9.657	966
FCRM <74 µm Matrix Spike	0.5058	50	5	14.63	7230

1 – Measure 1.00 +/- 0.05 g to the nearest 0.0001g, enter exact mass in table, or if one-half reduced volume digestion is performed, measure 0.50 +/- 0.05 g to the nearest 0.0001g. If your lab requires more reference material, please contact Clifton Jones at clifton.jones@cbifederalservices.com.

2 – Lead in sample material (mg/kg) = [Instrument Pb Results for Analytical Solution (mg/L) x Dilution Factor x 100 mL Digest Final Volume x (1 L/1000 mL)]/[mass of sample material (g) x (1 kg/1000 g)].

Appendix L
Laboratory Submitted Study Results

LABORATORY A, Table B8. Method 3050B or 3051A Digestion Spiked Blank, and Spiked Sample Results for (FCRM <74 µm): Lead (Pb)	
Laboratory Performing Digestion	Laboratory A
Laboratory Performing Analysis	Laboratory A
Blank Spike Result (mg/L)	9.66
Blank Spike Percent Recovery	97%
Average (5) Result RM (mg/L)	64.3
RM Matrix Spike Result (mg/L)	73.15
RM Matrix Spike Percent Recovery	88%
Control Soil NIST SRM 2710a Result (mg/Kg)	4920
Control Soil NIST SRM 2710a Digestion Acceptance Range - Lead (mg/Kg)	(Pb) 4590 -5610 mg/kg
Control Soil NIST SRM 2710a Lead Nominal Value (mg/Kg)	(Pb) 5100 mg/kg
Control Soil NIST SRM 2710a Percent Recovery (%)	96%

Laboratory B

Appendix L
Laboratory Submitted Study Results

LABORATORY B, Table A3. IVBA Extraction Batch Results, (NIST SRM 2710a): Arsenic (As)			
Lab Performing Extraction	Laboratory B	Analyst Performing Extraction	[redacted]
Lab Performing Analysis	Laboratory B	Analyst Performing Analysis	[redacted]
Instrument Type: ICP-MS or ICP-AES	ICP-AES	Instrument Method Detection Limit (MDL) (ug/L)	4.0 April 2016
Extraction Date	06/07/17	Extraction As Standard Manufacturer and Lot #	Inorganic Ventures, Lot # K2-AS650402
Analysis Date(s)	06/12/17	Analysis As Standard Manufacturer and Lot #	Inorganic Ventures, Lot # K2-AS650402
Initial Calibration Verification Standard Source and Lot #	Spex CertiPrep #2-111MKBXA	Interference Check Sample Source and Lot #	Inorganic Ventures: Al Lot # J2-AL05009, Ultra Scientific: Cr # CM-6110, Fe # R00990, Mo #Cr-1020

Sample Name	Mass of Sample Material (g)[1]	Volume of Extraction Fluid (mL)[2]	Dilution Factor	Instrument As Result for the Analytical Solution (mg/L)	Results for IVBA extractable As in sample material (mg/kg)
Reagent Blank	N/A	100	1	0.001	N/A
Method Blank	N/A	100	1	0.001	N/A
NIST SRM 2710a (Ex573traction 1)	0.9956	100	1	5.889	591.503
NIST SRM 2710a (Extraction 2)	1.0098	100	1	6.058	599.921
NIST SRM 2710a (Extraction 3)	1.0027	100	1	6.127	611.050
NIST SRM 2710a (Extraction 4)	1.0066	100	1	6.047	600.735
NIST SRM 2710a (Extraction 5)	1.0044	100	1	6.006	597.696
Control Soil – NIST SRM 2710a	1.0062	100	1	5.810	577.420
LCS (Method Blank Spike)	N/A	100	1	10.17	N/A
NIST SRM 2710a Matrix Spike	1.0134	100	1	16.330	1611.407

1 – Measure 1.00 +/- 0.05 g to the nearest 0.0001g, enter exact mass in table. Extraction mass should not be reduced. If your lab requires more reference material, please contact Clifton Jones at clifton.jones@cbifederalservices.com.

2 – Add 100 +/- 0.5 mL of 0.4M glycine, pH 1.5, delivered via graduated cylinder or automated dispenser.
Results of IVBA Extractable As in sample material (mg/kg) = [Instrument As Results for Analytical Solution (mg/L) x Dilution Factor x 100 mL extraction fluid x (1 L/1000 mL)]/[mass of sample material (g) x (1 kg/1000 g)].

Appendix L
Laboratory Submitted Study Results

LABORATORY B, Table A4. IVBA Extraction Batch Spiked Blank, Spiked Sample, and Control Soil Results for RM Batch (NIST SRM 2710a): Arsenic (As)	
Laboratory Performing Extraction	Laboratory B
Laboratory Performing Analysis	Laboratory B
Method Blank Result (mg/L)	0.001
LCS (Method Blank Spike) Result (mg/L)	10.17
LCS (Method Blank Spike) Percent Recovery	101.7%
Average (5) Result RM NIST SRM 2710a (mg/L)	6.025
RM NIST SRM 2710a Matrix Spike Result (mg/L)	16.33
RM NIST SRM 2710a Matrix Spike Percent Recovery	103%
Control Soil NIST SRM 2710a Result (mg/Kg)	577.420
Control Soil NIST SRM 2710a IVBA Extract Acceptance Range - Arsenic (mg/Kg)	(As) 464 – 681 mg/Kg
Control Soil NIST SRM 2710a IVBA Arsenic Nominal Value (mg/Kg)	(As) 573 mg/Kg
Control Soil NIST SRM 2710a Percent Recovery (%)	100.8%

Appendix L
Laboratory Submitted Study Results

LABORATORY B, Table A5. IVBA Extraction Batch Results, (FCRM <250 µm): Arsenic (As)			
Lab Performing Extraction	Laboratory B	Analyst Performing Extraction	[redacted]
Lab Performing Analysis	Laboratory B	Analyst Performing Analysis	[redacted]
Instrument Type: ICP-MS or ICP-AES	ICP-AES	Instrument Method Detection Limit (MDL) (ug/L)	4.0 April 2016
Extraction Date	06/09/17	Extraction As Standard Manufacturer and Lot #	Inorganic Ventures, Lot # K2-AS650402
Analysis Date(s)	06/15/17	Analysis As Standard Manufacturer and Lot #	Inorganic Ventures, Lot # K2-AS650402
Initial Calibration Verification Standard Source and Lot #	Spex CertiPrep #2-111MKBXA	Interference Check Sample Source and Lot #	Inorganic Ventures: Al # J2-AL05009, Ultra Scientific: Cr # CM-6110, Fe # R00990, Mo #Cr-1020

Sample Name	Mass of Sample Material (g)[1]	Volume of Extraction Fluid (mL)[2]	Dilution Factor	Instrument As Result for the Analytical Solution (mg/L)	Results for IVBA extractable As in sample material (mg/kg)
Reagent Blank	N/A	100	1	-0.009	N/A
Method Blank	N/A	100	1	-0.015	N/A
FCRM <250 µm (Extraction 1)	0.9998	100	1	1.670	167.033
FCRM <250 µm (Extraction 2)	1.0075	100	1	1.721	170.819
FCRM <250 µm (Extraction 3)	1.0115	100	1	1.666	164.706
FCRM <250 µm (Extraction 4)	1.0068	100	1	1.674	166.269
FCRM <250 µm (Extraction 5)	0.9961	100	1	1.646	165.244
Control Soil – NIST SRM 2710A	0.9963	100	1	6.567	659.139
LCS (Method Blank Spike)	N/A	100	1	10.31	N/A
FCRM <250 µm Matrix Spike	1.0269	100	1	11.45	1115.0063

1 – Measure 1.00 +/- 0.05 g to the nearest 0.0001g, enter exact mass in table. Extraction mass should not be reduced. If your lab requires more reference material, please contact Clifton Jones at clifton.jones@cbifederalservices.com.

2 – Add 100 +/- 0.5 mL of 0.4M glycine, pH 1.5, delivered via graduated cylinder or automated dispenser.
Results of IVBA Extractable As in sample material (mg/kg) = [Instrument As Results for Analytical Solution (mg/L) x Dilution Factor x 100 mL extraction fluid x (1 L/1000 mL)]/[mass of sample material (g) x (1 kg/1000 g)].

Appendix L
Laboratory Submitted Study Results

LABORATORY B, Table A6. IVBA Extraction Batch Spiked Blank, Spiked Sample, and Control Soil Results for RM Batch (FCRM <250 µm): Arsenic (As)	
Laboratory Performing Extraction	Laboratory B
Laboratory Performing Analysis	Laboratory B
Method Blank Result (mg/L)	-0.0149
LCS (Method Blank Spike) Result (mg/L)	10.31
LCS (Method Blank Spike) Percent Recovery	103.1%
Average (5) Result FCRM <250 µm (mg/L)	1.675
FCRM <250 µm Matrix Spike Result (mg/L)	11.45
FCRM <250 µm Matrix Spike Percent Recovery	97.75%
Control Soil NIST SRM 2710a Result (mg/Kg)	659.139
Control Soil NIST SRM 2710a IVBA Extract Acceptance Range - Arsenic (mg/Kg)	(As) 464 – 681 mg/Kg
Control Soil NIST SRM 2710a IVBA Arsenic Nominal Value (mg/Kg)	(As) 573 mg/Kg
Control Soil NIST SRM 2710a Percent Recovery (%)	115.03%

Appendix L
Laboratory Submitted Study Results

LABORATORY B, Table A7. IVBA Extraction Batch Results, (FCRM <74 µm): Arsenic (As)			
Lab Performing Extraction	Laboratory B	Analyst Performing Extraction	[redacted]
Lab Performing Analysis	Laboratory B	Analyst Performing Analysis	[redacted]
Instrument Type: ICP-MS or ICP-AES	ICP-AES	Instrument Method Detection Limit (MDL) (ug/L)	4.0 April 2016
Extraction Date	06/09/17	Extraction As Standard Manufacturer and Lot #	Inorganic Ventures, Lot # K2-AS650402
Analysis Date(s)	06/13/17	Analysis As Standard Manufacturer and Lot #	Inorganic Ventures, Lot # K2-AS650402
Initial Calibration Verification Standard Source and Lot #	Spex CertiPrep # 2-111MKBXA	Interference Check Sample Source and Lot #	Inorganic Ventures: Al Lot # J2-AL05009, Ultra Scientific: Cr # CM-6110, Fe # R00990, Mo #Cr-1020

Sample Name	Mass of Sample Material (g)[1]	Volume of Extraction Fluid (mL)[2]	Dilution Factor	Instrument As Result for the Analytical Solution (mg/L)	Results for IVBA extractable As in sample material (mg/kg)
Reagent Blank	N/A	100	1	-0.005	N/A
Method Blank	N/A	100	1	-0.014	N/A
FCRM <74 µm (Extraction 1)	1.0074	100	1	1.590	157.822
FCRM <74 µm (Extraction 2)	0.9987	100	1	1.620	162.211
FCRM <74 µm (Extraction 3)	1.0000	100	1	1.650	165.000
FCRM <74 µm (Extraction 4)	1.0064	100	1	1.613	160.274
FCRM <74 µm (Extraction 5)	1.0001	100	1	1.609	160.884
Control Soil – NIST SRM 2710A	1.0145	100	1	6.299	620.897
LCS (Method Blank Spike)	N/A	100	1	10.115	N/A
FCRM <74 µm Matrix Spike	1.0137	100	1	9.733	960.146

1 – Measure 1.00 +/- 0.05 g to the nearest 0.0001g, enter exact mass in table. Extraction mass should not be reduced. If your lab requires more reference material, please contact Clifton Jones at clifton.jones@cbifederalservices.com.

2 – Add 100 +/- 0.5 mL of 0.4M glycine, pH 1.5, delivered via graduated cylinder or automated dispenser.
Results of IVBA Extractable As in sample material (mg/kg) = [Instrument As Results for Analytical Solution (mg/L) x Dilution Factor x 100 mL extraction fluid x (1 L/1000 mL)]/[mass of sample material (g) x (1 kg/1000 g)].

Appendix L
Laboratory Submitted Study Results

LABORATORY B, Table A8. IVBA Extraction Batch Spiked Blank, Spiked Sample, and Control Soil Results for RM Batch (FCRM <74 µm): Arsenic (As)	
Laboratory Performing Extraction	Laboratory B
Laboratory Performing Analysis	Laboratory B
Method Blank Result (mg/L)	-0.014
LCS (Method Blank Spike) Result (mg/L)	10.12
LCS (Method Blank Spike) Percent Recovery	101.2%
Average (5) Result FCRM <250 µm (mg/L)	1.616
FCRM <74 µm Matrix Spike Result (mg/L)	9.733
FCRM <74 µm Matrix Spike Percent Recovery	81.2%
Control Soil NIST SRM 2710a Result (mg/Kg)	620.897
Control Soil NIST SRM 2710a IVBA Extract Acceptance Range - Arsenic (mg/Kg)	(As) 464 – 681 mg/Kg
Control Soil NIST SRM 2710a IVBA Arsenic Nominal Value (mg/Kg)	(As) 573 mg/Kg
Control Soil NIST SRM 2710a Percent Recovery (%)	108.4%

Appendix L
Laboratory Submitted Study Results

LABORATORY B, Table A9. IVBA Extraction Batch Results, (FCRM <74 µm): Lead (Pb)					
Lab Performing Extraction	Laboratory B		Analyst Performing Extraction	[redacted]	
Lab Performing Analysis	Laboratory B		Analyst Performing Analysis	[redacted]	
Instrument Type: ICP-MS or ICP-AES	ICP-AES		Instrument Method Detection Limit (MDL) (ug/L)	2.0 April 2016	
Extraction Date	06/09/17		Extraction Pb Standard Manufacturer and Lot #	Inorganic Ventures, Lot # K2-PB03074	
Analysis Date(s)	06/13/17		Analysis Pb Standard Manufacturer and Lot #	Inorganic Ventures, Lot # K2-PB03074	
Initial Calibration Verification Standard Source and Lot #	Spex CertiPrep #2-111MKBXA		Interference Check Sample Source and Lot #	Inorganic Ventures: Al Lot # J2-AL05009, Ultra Scientific: Cr # CM-6110, Fe # R00990, Mo #Cr-1020	
Sample Name	Mass of Sample Material (g)[1]	Volume of Extraction Fluid (mL)[2]	Dilution Factor	Instrument Pb Result for the Analytical Solution (mg/L)	Results for IVBA extractable Pb in sample material (mg/kg)
Reagent Blank	N/A	100	1	-0.0021	N/A
Method Blank	N/A	100	1	-0.0020	N/A
FCRM <74 µm (Extraction 1)	1.0074	100	1	46.790	4644.630
FCRM <74 µm (Extraction 2)	0.9987	100	1	46.970	4703.114
FCRM <74 µm (Extraction 3)	1.0000	100	1	48.450	4845.000
FCRM <74 µm (Extraction 4)	1.0064	100	1	46.840	4654.213
FCRM <74 µm (Extraction 5)	1.0001	100	1	46.110	4610.539
Control Soil – NIST SRM 2710A	1.0145	100	1	32.630	3216.363
LCS (Method Blank Spike)	N/A	100	1	9.9490	N/A
FCRM <74 µm Matrix Spike	1.0137	100	1	55.550	5479.925

1 – Measure 1.00 +/- 0.05 g to the nearest 0.0001g, enter exact mass in table. Extraction mass should not be reduced. If your lab requires more reference material, please contact Clifton Jones at clifton.jones@cbifederalservices.com.

2 – Add 100 +/- 0.5 mL of 0.4M glycine, pH 1.5, delivered via graduated cylinder or automated dispenser.
Results of IVBA Extractable Pb in sample material (mg/kg) = [Instrument Pb Results for Analytical Solution (mg/L) x Dilution Factor x 100 mL extraction fluid x (1 L/1000 mL)]/[mass of sample material (g) x (1 kg/1000 g)].

Appendix L
Laboratory Submitted Study Results

LABORATORY B, Table A10. IVBA Extraction Batch Spiked Blank, Spiked Sample, and Control Soil Results for RM Batch (FCRM <74 µm): Lead (Pb)	
Laboratory Performing Extraction	Laboratory B
Laboratory Performing Analysis	Laboratory B
Method Blank Result (mg/L)	-0.002
LCS (Method Blank Spike) Result (mg/L)	9.949
LCS (Method Blank Spike) Percent Recovery	99.5%
Average (5) Result FCRM <250 µm (mg/L)	47.032
FCRM <74 µm Matrix Spike Result (mg/L)	55.55
FCRM <74 µm Matrix Spike Percent Recovery	85.2%
Control Soil NIST SRM 2710a Result (mg/Kg)	3216.363
Control Soil NIST SRM 2710a IVBA Extract Acceptance Range – Lead (mg/Kg)	(Pb) 3096 – 3785 mg/Kg
Control Soil NIST SRM 2710a IVBA Lead Nominal Value (mg/Kg)	(Pb) 3440 mg/Kg
Control Soil NIST SRM 2710a Percent Recovery (%)	93.5%

Appendix L
Laboratory Submitted Study Results

LABORATORY B, Table B3. Method 3050B or 3051A Digestion Batch Results, (FCRM <250 µm): Arsenic (As)					
Lab Performing Digestion	Laboratory B		Analyst Performing Digestion	[redacted]	
Lab Performing Analysis	Laboratory B		Analyst Performing Analysis	[redacted]	
Instrument Type: ICP-MS or ICP-AES	ICP-AES		Instrument Method Detection Limit (MDL) (ug/L)	4.0 April 2016	
Digestion Date	06/13/17		Digestion As Standard Manufacturer and Lot #	Inorganic Ventures # K2-AS650402	
Analysis Date(s)	06/14/17		Analysis As Standard Manufacturer and Lot #	Inorganic Ventures # K2-AS650402	
Initial Calibration Verification Standard Source and Lot #	Spex CertiPrep #2-111MKBXA		Interference Check Sample Source and Lot #	Inorganic Ventures: Al Lot # J2-AL05009, Ultra Scientific: Cr # CM-6110, Fe # R00990, Mo #Cr-1020	
Sample Name	Mass of Sample Material (g)[1]	Volume of Digestion Fluid (mL)	Dilution Factor	Instrument As Result for the Analytical Solution (mg/L)	Results from digested Arsenic in sample material (mg/kg)[2]
Method Blank	N/A	50	1	-0.0016	N/A
FCRM <250 µm (Digestion 1)	0.5008	50	1	7.141	712.959
FCRM <250 µm (Digestion 2)	0.5013	50	1	7.070	705.167
FCRM <250 µm (Digestion 3)	0.5005	50	1	6.918	691.109
FCRM <250 µm (Digestion 4)	0.5013	50	1	6.898	688.011
FCRM <250 µm (Digestion 5)	0.5005	50	1	7.374	736.663
Control Soil – NIST SRM 2710a	0.5009	50	1	14.340	1431.423
LCS (Method Blank Spike)	N/A	50	1	9.340	N/A
FCRM <250 µm Matrix Spike	0.5013	50	1	16.750	1670.656

1 – Measure 1.00 +/- 0.05 g to the nearest 0.0001g, enter exact mass in table, or if one-half reduced volume digestion is performed, measure 0.50 +/- 0.05 g to the nearest 0.0001g. If your lab requires more reference material, please contact Clifton Jones at clifton.jones@cbifederalservices.com.

2 – Arsenic in sample material (mg/kg) = [Instrument As Results for Analytical Solution (mg/L) x Dilution Factor x 100 mL Digest Final Volume x (1 L/1000 mL)]/[mass of sample material (g) x (1 kg/1000 g)].

Appendix L
Laboratory Submitted Study Results

LABORATORY B, Table B4. Method 3050B or 3051A Digestion Spiked Blank, and Spiked Sample Results for (FCRM <250 µm): Arsenic (As)	
Laboratory Performing Digestion	Laboratory B
Laboratory Performing Analysis	Laboratory B
Blank Spike Result (mg/L)	9.340
Blank Spike Percent Recovery	93.4%
Average (5) Result RM (mg/L)	7.0802
RM Matrix Spike Result (mg/L)	16.75
RM Matrix Spike Percent Recovery	96.7%
Control Soil NIST SRM 2710a Result (mg/Kg)	1431.423
Control Soil NIST SRM 2710a Digestion Acceptance Range - Arsenic (mg/Kg)	(As) 1260 -1540 mg/kg
Control Soil NIST SRM 2710a Arsenic Nominal Value (mg/Kg)	(As) 1400 mg/kg
Control Soil NIST SRM 2710a Percent Recovery (%)	102.2%

Appendix L
Laboratory Submitted Study Results

LABORATORY B, Table B5. Method 3050B or 3051A Digestion Batch Results, (FCRM <74 µm): Arsenic (As)			
Lab Performing Digestion	Laboratory B	Analyst Performing Digestion	[redacted]
Lab Performing Analysis	Laboratory B	Analyst Performing Analysis	[redacted]
Instrument Type: ICP-MS or ICP-AES	ICP-AES	Instrument Method Detection Limit (MDL) (ug/L)	4.0 April 2016
Digestion Date	06/13/17	Digestion As Standard Manufacturer and Lot #	Inorganic Ventures # K2-AS650402
Analysis Date(s)	06/14/17	Analysis As Standard Manufacturer and Lot #	Inorganic Ventures # K2-AS650402
Initial Calibration Verification Standard Source and Lot #	Spex CertiPrep #2-111MKBXA	Interference Check Sample Source and Lot #	Inorganic Ventures: Al Lot # J2-AL05009, Ultra Scientific: Cr # CM-6110, Fe # R00990, Mo #Cr-1020

Sample Name	Mass of Sample Material (g)[1]	Volume of Digestion Fluid (mL)	Dilution Factor	Instrument As Result for the Analytical Solution (mg/L)	Results from digested Arsenic in sample material (mg/kg)[2]
Method Blank	N/A	50	1	0.00002	N/A
FCRM <74 µm (Digestion 1)	0.5019	50	1	6.750	672.445
FCRM <74 µm (Digestion 2)	0.5008	50	1	7.335	732.328
FCRM <74 µm (Digestion 3)	0.5011	50	1	7.219	720.315
FCRM <74 µm (Digestion 4)	0.5013	50	1	7.121	710.253
FCRM <74 µm (Digestion 5)	0.5011	50	1	7.008	699.262
Control Soil – NIST SRM 2710a	0.5012	50	1	14.860	1482.442
LCS (Method Blank Spike)	N/A	50	1	9.586	N/A
FCRM <74 µm Matrix Spike	0.5005	50	1	16.050	1603.397

1 – Measure 1.00 +/- 0.05 g to the nearest 0.0001g, enter exact mass in table, or if one-half reduced volume digestion is performed, measure 0.50 +/- 0.05 g to the nearest 0.0001g. If your lab requires more reference material, please contact Clifton Jones at clifton.jones@cbifederalservices.com.

2 – Arsenic in sample material (mg/kg) = [Instrument As Results for Analytical Solution (mg/L) x Dilution Factor x 100 mL Digest Final Volume x (1 L/1000 mL)]/[mass of sample material (g) x (1 kg/1000 g)].

Appendix L
Laboratory Submitted Study Results

LABORATORY B, Table B6. Method 3050B or 3051A Digestion Spiked Blank, and Spiked Sample Results for (FCRM <74 µm): Arsenic (As)	
Laboratory Performing Digestion	Laboratory B
Laboratory Performing Analysis	Laboratory B
Blank Spike Result (mg/L)	9.586
Blank Spike Percent Recovery	95.9%
Average (5) Result RM (mg/L)	7.087
RM Matrix Spike Result (mg/L)	16.050
RM Matrix Spike Percent Recovery	89.6%
Control Soil NIST SRM 2710a Result (mg/Kg)	1482.442
Control Soil NIST SRM 2710a Digestion Acceptance Range - Arsenic (mg/Kg)	(As) 1260 -1540 mg/kg
Control Soil NIST SRM 2710a Arsenic Nominal Value (mg/Kg)	(As) 1400 mg/kg
Control Soil NIST SRM 2710a Percent Recovery (%)	105.9%

Appendix L
Laboratory Submitted Study Results

LABORATORY B, Table B7. Method 3050B or 3051A Digestion Batch Results, (FCRM <74 µm): Lead (Pb)					
Lab Performing Digestion	Laboratory B		Analyst Performing Digestion	[redacted]	
Lab Performing Analysis	Laboratory B		Analyst Performing Analysis	[redacted]	
Instrument Type: ICP-MS or ICP-AES	ICP-AES		Instrument Method Detection Limit (MDL) (ug/L)	2.0 April 2016	
Digestion Date	06/13/17		Digestion Pb Standard Manufacturer and Lot #	Inorganic Ventures # K2-PB03074	
Analysis Date(s)	06/14/17		Analysis Pb Standard Manufacturer and Lot #	Inorganic Ventures # K2-PB03074	
Initial Calibration Verification Standard Source and Lot #	Spex CertiPrep #2-111MKBXA		Interference Check Sample Source and Lot #	Inorganic Ventures: Al Lot # J2-AL05009, Ultra Scientific: Cr # CM-6110, Fe # R00990, Mo #Cr-1020	
Sample Name	Mass of Sample Material (g)[1]	Volume of Digestion Fluid (mL)	Dilution Factor	Instrument Pb Result for the Analytical Solution (mg/L)	Results from digested Lead in sample material (mg/kg)[2]
Method Blank	N/A	50	1	-0.00159	N/A
FCRM <74 µm (Digestion 1)	0.5019	50	1	61.22	6098.824
FCRM <74 µm (Digestion 2)	0.5008	50	1	60.35	6025.359
FCRM <74 µm (Digestion 3)	0.5011	50	1	62.64	6250.249
FCRM <74 µm (Digestion 4)	0.5013	50	1	61.05	6089.168
FCRM <74 µm (Digestion 5)	0.5011	50	1	61.37	6123.528
Control Soil – NIST SRM 2710a	0.5012	50	1	49.50	4938.148
LCS (Method Blank Spike)	N/A	50	1	10.03	N/A
FCRM <74 µm Matrix Spike	0.5005	50	1	70.15	7007.992

1 – Measure 1.00 +/- 0.05 g to the nearest 0.0001g, enter exact mass in table, or if one-half reduced volume digestion is performed, measure 0.50 +/- 0.05 g to the nearest 0.0001g. If your lab requires more reference material, please contact Clifton Jones at clifton.jones@cbifederalservices.com.

2 – Lead in sample material (mg/kg) = [Instrument Pb Results for Analytical Solution (mg/L) x Dilution Factor x 100 mL Digest Final Volume x (1 L/1000 mL)]/[mass of sample material (g) x (1 kg/1000 g)].

Appendix L
Laboratory Submitted Study Results

LABORATORY B, Table B8. Method 3050B or 3051A Digestion Spiked Blank, and Spiked Sample Results for (FCRM <74 µm): Lead (Pb)	
Laboratory Performing Digestion	Laboratory B
Laboratory Performing Analysis	Laboratory B
Blank Spike Result (mg/L)	10.03
Blank Spike Percent Recovery	100.3%
Average (5) Result RM (mg/L)	61.326
RM Matrix Spike Result (mg/L)	70.15
RM Matrix Spike Percent Recovery	88.2%
Control Soil NIST SRM 2710a Result (mg/Kg)	4938.148
Control Soil NIST SRM 2710a Digestion Acceptance Range - Lead (mg/Kg)	(Pb) 4590 -5610 mg/kg
Control Soil NIST SRM 2710a Lead Nominal Value (mg/Kg)	(Pb) 5100 mg/kg
Control Soil NIST SRM 2710a Percent Recovery (%)	96.8%

Laboratory C

Appendix L
Laboratory Submitted Study Results

LABORATORY C, Table A3. IVBA Extraction Batch Results, (NIST SRM 2710a): Arsenic (As)					
Lab Performing Extraction	Laboratory C		Analyst Performing Extraction	[redacted]	
Lab Performing Analysis	Laboratory C		Analyst Performing Analysis	[redacted]	
Instrument Type: ICP-MS or ICP-AES	ICP-AES		Instrument Method Detection Limit (MDL) (ug/L)	11.0	
Extraction Date	5/9/17		Extraction As Standard Manufacturer and Lot #	SPEX CertiPrep. Lot #: 22 - 122ASY	
Analysis Date(s)	5/10/17		Analysis As Standard Manufacturer and Lot #	Absolute Standards. QC Std. 1 Lot#: 113016	
Initial Calibration Verification Standard Source and Lot #	Absolute Standards. QC Std. 1 Lot#: 113016		Interference Check Sample Source and Lot #	ICSAB: ICS Part A CBI QATS Lot #: 1211; ICS Part B CBI QATS Lot #: 0710.	
Sample Name	Mass of Sample Material (g)[1]	Volume of Extraction Fluid (mL)[2]	Dilution Factor	Instrument As Result for the Analytical Solution (mg/L)	Results for IVBA extractable As in sample material (mg/kg)
Reagent Blank	1.0000	100	1	0	0
Method Blank	1.0000	100	1	0	0
NIST SRM 2710a (Extraction 1)	1.0036	100	1	4.718	470.1
NIST SRM 2710a (Extraction 2)	1.0090	100	1	4.897	485.3
NIST SRM 2710a (Extraction 3)	1.0068	100	1	4.844	481.2
NIST SRM 2710a (Extraction 4)	1.0066	100	1	4.905	487.3
NIST SRM 2710a (Extraction 5)	1.0062	100	1	4.862	483.2
Control Soil – NIST SRM 2710a	1.0011	100	1	4.866	486.0
LCS (Method Blank Spike)	1.0000	100	1	11.440	1144
NIST SRM 2710a Matrix Spike	1.0097	100	1	13.380	1326

1 – Measure 1.00 +/- 0.05 g to the nearest 0.0001g, enter exact mass in table. Extraction mass should not be reduced. If your lab requires more reference material, please contact Clifton Jones at clifton.jones@cbifederalservices.com.

2 – Add 100 +/- 0.5 mL of 0.4M glycine, pH 1.5, delivered via graduated cylinder or automated dispenser.
Results of IVBA Extractable As in sample material (mg/kg) = [Instrument As Results for Analytical Solution (mg/L) x Dilution Factor x 100 mL extraction fluid x (1 L/1000 mL)]/[mass of sample material (g) x (1 kg/1000 g)].

Appendix L
Laboratory Submitted Study Results

LABORATORY C, Table A4. IVBA Extraction Batch Spiked Blank, Spiked Sample, and Control Soil Results for RM Batch (NIST SRM 2710a): Arsenic (As)	
Laboratory Performing Extraction	Laboratory C
Laboratory Performing Analysis	Laboratory C
Method Blank Result (mg/L)	0
LCS (Method Blank Spike) Result (mg/L)	11.440
LCS (Method Blank Spike) Percent Recovery	114
Average (5) Result RM NIST SRM 2710a (mg/L)	4.8452
RM NIST SRM 2710a Matrix Spike Result (mg/L)	13.380
RM NIST SRM 2710a Matrix Spike Percent Recovery	85.3
Control Soil NIST SRM 2710a Result (mg/Kg)	486
Control Soil NIST SRM 2710a IVBA Extract Acceptance Range - Arsenic (mg/Kg)	(As) 464 – 681 mg/Kg
Control Soil NIST SRM 2710a IVBA Arsenic Nominal Value (mg/Kg)	(As) 573 mg/Kg
Control Soil NIST SRM 2710a Percent Recovery (%)	84.8

Appendix L
Laboratory Submitted Study Results

LABORATORY C, Table A5. IVBA Extraction Batch Results, (FCRM <250 µm): Arsenic (As)					
Lab Performing Extraction	Laboratory C		Analyst Performing Extraction	[redacted]	
Lab Performing Analysis	Laboratory C		Analyst Performing Analysis	[redacted]	
Instrument Type: ICP-MS or ICP-AES	ICP-AES		Instrument Method Detection Limit (MDL) (ug/L)	11.0	
Extraction Date	5/10/17		Extraction As Standard Manufacturer and Lot #	SPEX CertiPrep. Lot #: 22-122ASY	
Analysis Date(s)	5/10/17		Analysis As Standard Manufacturer and Lot #	Absolute Standards. QC Std. 1 Lot#: 113016	
Initial Calibration Verification Standard Source and Lot #	Absolute Standards. QC Std. 1 Lot#: 113016		Interference Check Sample Source and Lot #	ICSAB: ICS Part A CBI QATS Lot #: 1211; ICS Part B CBI QATS Lot #: 0710.	
Sample Name	Mass of Sample Material (g)[1]	Volume of Extraction Fluid (mL)[2]	Dilution Factor	Instrument As Result for the Analytical Solution (mg/L)	Results for IVBA extractable As in sample material (mg/kg)
Reagent Blank	1.0000	100	1	0.004211	0.421
Method Blank	1.0000	100	1	0	0
FCRM <250 µm (Extraction 1)	1.0046	100	1	1.308	130.2
FCRM <250 µm (Extraction 2)	1.0050	100	1	1.322	131.6
FCRM <250 µm (Extraction 3)	1.0044	100	1	1.343	133.8
FCRM <250 µm (Extraction 4)	1.0012	100	1	1.326	132.5
FCRM <250 µm (Extraction 5)	1.0093	100	1	1.323	131.1
Control Soil – NIST SRM 2710A	1.0025	100	1	5.214	520.1
LCS (Method Blank Spike)	1.0000	100	1	11.170	1117
FCRM <250 µm Matrix Spike	1.0085	100	1	10.860	1077

1 – Measure 1.00 +/- 0.05 g to the nearest 0.0001g, enter exact mass in table. Extraction mass should not be reduced. If your lab requires more reference material, please contact Clifton Jones at clifton.jones@cbifederalservices.com.

2 – Add 100 +/- 0.5 mL of 0.4M glycine, pH 1.5, delivered via graduated cylinder or automated dispenser.
Results of IVBA Extractable As in sample material (mg/kg) = [Instrument As Results for Analytical Solution (mg/L) x Dilution Factor x 100 mL extraction fluid x (1 L/1000 mL)]/[mass of sample material (g) x (1 kg/1000 g)].

Appendix L
Laboratory Submitted Study Results

LABORATORY C, Table A6. IVBA Extraction Batch Spiked Blank, Spiked Sample, and Control Soil Results for RM Batch (FCRM <250 µm): Arsenic (As)	
Laboratory Performing Extraction	Laboratory C
Laboratory Performing Analysis	Laboratory C
Method Blank Result (mg/L)	0
LCS (Method Blank Spike) Result (mg/L)	11.170
LCS (Method Blank Spike) Percent Recovery	112
Average (5) Result FCRM <250 µm (mg/L)	1.3244
FCRM <250 µm Matrix Spike Result (mg/L)	10.860
FCRM <250 µm Matrix Spike Percent Recovery	95.4
Control Soil NIST SRM 2710a Result (mg/Kg)	520.1
Control Soil NIST SRM 2710a IVBA Extract Acceptance Range - Arsenic (mg/Kg)	(As) 464 – 681 mg/Kg
Control Soil NIST SRM 2710a IVBA Arsenic Nominal Value (mg/Kg)	(As) 573 mg/Kg
Control Soil NIST SRM 2710a Percent Recovery (%)	90.8

Appendix L
Laboratory Submitted Study Results

LABORATORY C, Table A7. IVBA Extraction Batch Results, (FCRM <74 µm): Arsenic (As)					
Lab Performing Extraction	Laboratory C		Analyst Performing Extraction	[redacted]	
Lab Performing Analysis	Laboratory C		Analyst Performing Analysis	[redacted]	
Instrument Type: ICP-MS or ICP-AES	ICP-AES		Instrument Method Detection Limit (MDL) (ug/L)	11.0	
Extraction Date	5/10/17		Extraction As Standard Manufacturer and Lot #	SPEX CertiPrep. Lot #: 22-122ASY	
Analysis Date(s)	5/11/17		Analysis As Standard Manufacturer and Lot #	Absolute Standards. QC Std. 1 Lot#: 113016	
Initial Calibration Verification Standard Source and Lot #	Absolute Standards. QC Std. 1 Lot#: 113016		Interference Check Sample Source and Lot #	ICSAB: ICS Part A CBI QATS Lot #: 1211; ICS Part B CBI QATS Lot #: 0710.	
Sample Name	Mass of Sample Material (g)[1]	Volume of Extraction Fluid (mL)[2]	Dilution Factor	Instrument As Result for the Analytical Solution (mg/L)	Results for IVBA extractable As in sample material (mg/kg)
Reagent Blank	1.0000	100	1	0.002394	0.239
Method Blank	1.0000	100	1	0	0
FCRM <74 µm (Extraction 1)	1.0049	100	1	1.310	130.4
FCRM <74 µm (Extraction 2)	1.0030	100	1	1.350	134.6
FCRM <74 µm (Extraction 3)	1.0032	100	1	1.336	133.1
FCRM <74 µm (Extraction 4)	1.0032	100	1	1.354	134.9
FCRM <74 µm (Extraction 5)	1.0097	100	1	1.340	132.7
Control Soil – NIST SRM 2710A	1.0002	100	1	5.335	533.4
LCS (Method Blank Spike)	1.0000	100	1	10.840	1084
FCRM <74 µm Matrix Spike	1.0090	100	1	10.530	1043

1 – Measure 1.00 +/- 0.05 g to the nearest 0.0001g, enter exact mass in table. Extraction mass should not be reduced. If your lab requires more reference material, please contact Clifton Jones at clifton.jones@cbifederalservices.com.

2 – Add 100 +/- 0.5 mL of 0.4M glycine, pH 1.5, delivered via graduated cylinder or automated dispenser.
Results of IVBA Extractable As in sample material (mg/kg) = [Instrument As Results for Analytical Solution (mg/L) x Dilution Factor x 100 mL extraction fluid x (1 L/1000 mL)]/[mass of sample material (g) x (1 kg/1000 g)].

Appendix L
Laboratory Submitted Study Results

LABORATORY C, Table A8. IVBA Extraction Batch Spiked Blank, Spiked Sample, and Control Soil Results for RM Batch (FCRM <74 µm): Arsenic (As)	
Laboratory Performing Extraction	Laboratory C
Laboratory Performing Analysis	Laboratory C
Method Blank Result (mg/L)	0
LCS (Method Blank Spike) Result (mg/L)	10.840
LCS (Method Blank Spike) Percent Recovery	108
Average (5) Result FCRM <74µm (mg/L)	1.338
FCRM <74 µm Matrix Spike Result (mg/L)	10.530
FCRM <74 µm Matrix Spike Percent Recovery	91.9
Control Soil NIST SRM 2710a Result (mg/Kg)	533.4
Control Soil NIST SRM 2710a IVBA Extract Acceptance Range - Arsenic (mg/Kg)	(As) 464 – 681 mg/Kg
Control Soil NIST SRM 2710a IVBA Arsenic Nominal Value (mg/Kg)	(As) 573 mg/Kg
Control Soil NIST SRM 2710a Percent Recovery (%)	93.1

Appendix L
Laboratory Submitted Study Results

LABORATORY C, Table A9. IVBA Extraction Batch Results, (FCRM <74 µm): Lead (Pb)			
Lab Performing Extraction	Laboratory C	Analyst Performing Extraction	[redacted]
Lab Performing Analysis	Laboratory C	Analyst Performing Analysis	[redacted]
Instrument Type: ICP-MS or ICP-AES	ICP-AES	Instrument Method Detection Limit (MDL) (ug/L)	9.59
Extraction Date	5/11/17	Extraction Pb Standard Manufacturer and Lot #	SPEX CertiPrep. Lot #: 22-149BPY
Analysis Date(s)	5/12/17	Analysis Pb Standard Manufacturer and Lot #	Absolute Standards. QC Std. 1 Lot#: 113016
Initial Calibration Verification Standard Source and Lot #	Absolute Standards. QC Std. 1 Lot#: 113016	Interference Check Sample Source and Lot #	ICSAB: ICS Part A CBI QATS Lot #: 1211; ICS Part B CBI QATS Lot #: 0710.

Sample Name	Mass of Sample Material (g)[1]	Volume of Extraction Fluid (mL)[2]	Dilution Factor	Instrument Pb Result for the Analytical Solution (mg/L)	Results for IVBA extractable Pb in sample material (mg/kg)
Reagent Blank	1.0000	100	1	0	0
Method Blank	1.0000	100	1	0.007475	0.747
FCRM <74 µm (Extraction 1)	1.0022	100	1	45.220	4512
FCRM <74 µm (Extraction 2)	1.0038	100	1	45.580	4541
FCRM <74 µm (Extraction 3)	1.0051	100	1	47.110	4687
FCRM <74 µm (Extraction 4)	1.0007	100	1	45.590	4556
FCRM <74 µm (Extraction 5)	1.0091	100	1	46.210	4580
Control Soil – NIST SRM 2710A	1.0086	100	1	30.860	3060
LCS (Method Blank Spike)	1.0000	100	1	9.849	984.9
FCRM <74 µm Matrix Spike	1.0038	100	1	55.100	5489

1 – Measure 1.00 +/- 0.05 g to the nearest 0.0001g, enter exact mass in table. Extraction mass should not be reduced. If your lab requires more reference material, please contact Clifton Jones at clifton.jones@cbifederalservices.com.

2 – Add 100 +/- 0.5 mL of 0.4M glycine, pH 1.5, delivered via graduated cylinder or automated dispenser.
Results of IVBA Extractable Pb in sample material (mg/kg) = [Instrument Pb Results for Analytical Solution (mg/L) x Dilution Factor x 100 mL extraction fluid x (1 L/1000 mL)]/[mass of sample material (g) x (1 kg/1000 g)].

Appendix L
Laboratory Submitted Study Results

LABORATORY C, Table A10. IVBA Extraction Batch Spiked Blank, Spiked Sample, and Control Soil Results for RM Batch (FCRM <74 µm): Lead (Pb)	
Laboratory Performing Extraction	Laboratory C
Laboratory Performing Analysis	Laboratory C
Method Blank Result (mg/L)	0.007475
LCS (Method Blank Spike) Result (mg/L)	9.849
LCS (Method Blank Spike) Percent Recovery	98.5
Average (5) Result FCRM <250 µm (mg/L)	45.942
FCRM <74 µm Matrix Spike Result (mg/L)	55.100
FCRM <74 µm Matrix Spike Percent Recovery	91.6
Control Soil NIST SRM 2710a Result (mg/Kg)	3060
Control Soil NIST SRM 2710a IVBA Extract Acceptance Range – Lead (mg/Kg)	(Pb) 3096 – 3785 mg/Kg
Control Soil NIST SRM 2710a IVBA Lead Nominal Value (mg/Kg)	(Pb) 3440 mg/Kg
Control Soil NIST SRM 2710a Percent Recovery (%)	89.0

Appendix L
Laboratory Submitted Study Results

LABORATORY C, Table B3. Method 3050B or 3051A Digestion Batch Results, (FCRM <250 µm): Arsenic (As)					
Lab Performing Digestion	Laboratory C		Analyst Performing Digestion	[redacted]	
Lab Performing Analysis	Laboratory C		Analyst Performing Analysis	[redacted]	
Instrument Type: ICP-MS or ICP-AES	ICP-AES		Instrument Method Detection Limit (MDL) (ug/L)	11.0	
Digestion Date	6/8/17		Digestion As Standard Manufacturer and Lot #	SPEX CertiPrep. Lot #: 22-122ASY	
Analysis Date(s)	6/9/17		Analysis As Standard Manufacturer and Lot #	Absolute Standards. QC Std. 1 Lot#: 113016	
Initial Calibration Verification Standard Source and Lot #	Absolute Standards. QC Std. 1 Lot#: 113016		Interference Check Sample Source and Lot #	ICSAB: ICS Part A CBI QATS Lot #: 1211; ICS Part B CBI QATS Lot #: 0710.	
Sample Name	Mass of Sample Material (g)[1]	Volume of Digestion Fluid (mL)	Dilution Factor	Instrument As Result for the Analytical Solution (mg/L)	Results from digested Arsenic in sample material (mg/kg)[2]
Method Blank	0.5000	50	1	0.0	0.0
FCRM <250 µm (Digestion 1)	0.5051	50	1	6.875	680.5
FCRM <250 µm (Digestion 2)	0.5083	50	1	7.110	699.4
FCRM <250 µm (Digestion 3)	0.5078	50	1	6.648	654.6
FCRM <250 µm (Digestion 4)	0.5062	50	1	6.646	656.5
FCRM <250 µm (Digestion 5)	0.5086	50	1	6.764	664.9
Control Soil – NIST SRM 2710a	0.5065	50	1	13.920	1374
LCS (Method Blank Spike)	0.5000	50	1	8.629	826.9
FCRM <250 µm Matrix Spike	0.5071	50	1	15.270	1506

1 – Measure 1.00 +/- 0.05 g to the nearest 0.0001g, enter exact mass in table, or if one-half reduced volume digestion is performed, measure 0.50 +/- 0.05 g to the nearest 0.0001g. If your lab requires more reference material, please contact Clifton Jones at clifton.jones@cbifederalservices.com.

2 – Arsenic in sample material (mg/kg) = [Instrument As Results for Analytical Solution (mg/L) x Dilution Factor x 100 mL Digest Final Volume x (1 L/1000 mL)]/[mass of sample material (g) x (1 kg/1000 g)].

Appendix L
Laboratory Submitted Study Results

LABORATORY C, Table B4. Method 3050B or 3051A Digestion Spiked Blank, and Spiked Sample Results for (FCRM <250 µm): Arsenic (As)	
Laboratory Performing Digestion	Laboratory C
Laboratory Performing Analysis	Laboratory C
Blank Spike Result (mg/L)	8.629
Blank Spike Percent Recovery	86.3
Average (5) Result RM (mg/L)	6.8086
RM Matrix Spike Result (mg/L)	15.270
RM Matrix Spike Percent Recovery	84.6
Control Soil NIST SRM 2710a Result (mg/Kg)	1374
Control Soil NIST SRM 2710a Digestion Acceptance Range - Arsenic (mg/Kg)	(As) 1260 -1540 mg/kg
Control Soil NIST SRM 2710a Arsenic Nominal Value (mg/Kg)	(As) 1400 mg/kg
Control Soil NIST SRM 2710a Percent Recovery (%)	98.1

Appendix L
Laboratory Submitted Study Results

LABORATORY C, Table B5. Method 3050B or 3051A Digestion Batch Results, (FCRM <74 µm): Arsenic (As)					
Lab Performing Digestion	Laboratory C		Analyst Performing Digestion	[redacted]	
Lab Performing Analysis	Laboratory C		Analyst Performing Analysis	[redacted]	
Instrument Type: ICP-MS or ICP-AES	ICP-AES		Instrument Method Detection Limit (MDL) (ug/L)	11.0	
Digestion Date	5/17/17		Digestion As Standard Manufacturer and Lot #	SPEX CertiPrep. Lot #: 22-122ASY	
Analysis Date(s)	5/31/17		Analysis As Standard Manufacturer and Lot #	Absolute Standards. QC Std. 1 Lot#: 113016	
Initial Calibration Verification Standard Source and Lot #	Absolute Standards. QC Std. 1 Lot#: 113016		Interference Check Sample Source and Lot #	ICSAB: ICS Part A CBI QATS Lot #: 1211; ICS Part B CBI QATS Lot #: 0710.	
Sample Name	Mass of Sample Material (g)[1]	Volume of Digestion Fluid (mL)	Dilution Factor	Instrument As Result for the Analytical Solution (mg/L)	Results from digested Arsenic in sample material (mg/kg)[2]
Method Blank	0.5000	50	1	0.003644	0.364
FCRM <74 µm (Digestion 1)	0.5045	50	1	6.957	689.5
FCRM <74 µm (Digestion 2)	0.5059	50	1	7.015	693.3
FCRM <74 µm (Digestion 3)	0.5027	50	1	6.989	695.1
FCRM <74 µm (Digestion 4)	0.5071	50	1	7.113	701.4
FCRM <74 µm (Digestion 5)	0.5087	50	1	7.262	713.8
Control Soil – NIST SRM 2710a	0.5085	50	1	14.790	1454
LCS (Method Blank Spike)	0.5000	50	1	9.337	933.7
FCRM <74 µm Matrix Spike	0.5088	50	1	16.380	1610

1 – Measure 1.00 +/- 0.05 g to the nearest 0.0001g, enter exact mass in table, or if one-half reduced volume digestion is performed, measure 0.50 +/- 0.05 g to the nearest 0.0001g. If your lab requires more reference material, please contact Clifton Jones at clifton.jones@cbifederalservices.com.

2 – Arsenic in sample material (mg/kg) = [Instrument As Results for Analytical Solution (mg/L) x Dilution Factor x 100 mL Digest Final Volume x (1 L/1000 mL)]/[mass of sample material (g) x (1 kg/1000 g)].

Appendix L
Laboratory Submitted Study Results

LABORATORY C, Table B6. Method 3050B or 3051A Digestion Spiked Blank, and Spiked Sample Results for (FCRM <74 µm): Arsenic (As)	
Laboratory Performing Digestion	Laboratory C
Laboratory Performing Analysis	Laboratory C
Blank Spike Result (mg/L)	9.337
Blank Spike Percent Recovery	93.4
Average (5) Result RM (mg/L)	7.0672
RM Matrix Spike Result (mg/L)	16.380
RM Matrix Spike Percent Recovery	93.1
Control Soil NIST SRM 2710a Result (mg/Kg)	1454
Control Soil NIST SRM 2710a Digestion Acceptance Range - Arsenic (mg/Kg)	(As) 1260 -1540 mg/kg
Control Soil NIST SRM 2710a Arsenic Nominal Value (mg/Kg)	(As) 1400 mg/kg
Control Soil NIST SRM 2710a Percent Recovery (%)	104

Appendix L
Laboratory Submitted Study Results

LABORATORY C, Table B7. Method 3050B or 3051A Digestion Batch Results, (FCRM <74 µm): Lead (Pb)			
Lab Performing Digestion	Laboratory C	Analyst Performing Digestion	[redacted]
Lab Performing Analysis	Laboratory C	Analyst Performing Analysis	[redacted]
Instrument Type: ICP-MS or ICP-AES	ICP-AES	Instrument Method Detection Limit (MDL) (ug/L)	9.59
Digestion Date	5/31/17	Digestion Pb Standard Manufacturer and Lot #	SPEX CertiPrep. Lot #: 22 – 149BPY
Analysis Date(s)	6/8/17	Analysis Pb Standard Manufacturer and Lot #	Absolute Standards. QC Std. 1 Lot#: 113016
Initial Calibration Verification Standard Source and Lot #	Absolute Standards. QC Std. 1 Lot#: 113016	Interference Check Sample Source and Lot #	ICSAB: ICS Part A CBI QATS Lot #: 1211; ICS Part B CBI QATS Lot #: 0710.

Sample Name	Mass of Sample Material (g)[1]	Volume of Digestion Fluid (mL)	Dilution Factor	Instrument Pb Result for the Analytical Solution (mg/L)	Results from digested Lead in sample material (mg/kg)[2]
Method Blank	0.5000	50	1	0.004704	0.470
FCRM <74 µm (Digestion 1)	0.5095	50	1	60.480	5936
FCRM <74 µm (Digestion 2)	0.5048	50	1	59.980	5941
FCRM <74 µm (Digestion 3)	0.5056	50	1	61.760	6107
FCRM <74 µm (Digestion 4)	0.5064	50	1	62.160	6138
FCRM <74 µm (Digestion 5)	0.5043	50	1	61.670	6114
Control Soil – NIST SRM 2710a	0.5037	50	1	47.330	4698
LCS (Method Blank Spike)	0.5000	50	1	9.444	944.4
FCRM <74 µm Matrix Spike	0.5074	50	1	70.220	6920

1 – Measure 1.00 +/- 0.05 g to the nearest 0.0001g, enter exact mass in table, or if one-ha1lf reduced volume digestion is performed, measure 0.50 +/- 0.05 g to the nearest 0.0001g. If your lab requires more reference material, please contact Clifton Jones at clifton.jones@cbifederalservices.com.

2 – Lead in sample material (mg/kg) = [Instrument Pb Results for Analytical Solution (mg/L) x Dilution Factor x 100 mL Digest Final Volume x (1 L/1000 mL)]/[mass of sample material (g) x (1 kg/1000 g)].

Appendix L
Laboratory Submitted Study Results

LABORATORY C, Table B8. Method 3050B or 3051A Digestion Spiked Blank, and Spiked Sample Results for (FCRM <74 µm): Lead (Pb)	
Laboratory Performing Digestion	Laboratory C
Laboratory Performing Analysis	Laboratory C
Blank Spike Result (mg/L)	9.444
Blank Spike Percent Recovery	94.4
Average (5) Result RM (mg/L)	61.210
RM Matrix Spike Result (mg/L)	70.220
RM Matrix Spike Percent Recovery	90.1
Control Soil NIST SRM 2710a Result (mg/Kg)	4698
Control Soil NIST SRM 2710a Digestion Acceptance Range - Lead (mg/Kg)	(Pb) 4590 -5610 mg/kg
Control Soil NIST SRM 2710a Lead Nominal Value (mg/Kg)	(Pb) 5100 mg/kg
Control Soil NIST SRM 2710a Percent Recovery (%)	92.1

Laboratory D

Appendix L
Laboratory Submitted Study Results

LABORATORY D, Table A3. IVBA Extraction Batch Results, (NIST SRM 2710a): Arsenic (As)			
Lab Performing Extraction	Laboratory D	Analyst Performing Extraction	[redacted]
Lab Performing Analysis	Laboratory D	Analyst Performing Analysis	[redacted]
Instrument Type: ICP-MS or ICP-AES	ICP-MS	Instrument Method Detection Limit (MDL) (ug/L)	MDL = 1.7 ug/L (*3 x IDL*) LLOQ = 10 ug/L (*lowest calibration standard*)
Extraction Date	06/15/2017	Extraction As Standard Manufacturer and Lot #	SCP Science: Lot S160912001
Analysis Date(s)	06/22/2017	Analysis As Standard Manufacturer and Lot #	SCP Science Custom Std: Lot S160715008
Initial Calibration Verification Standard Source and Lot #	SCP Science Custom Std: Lot S160715009	Interference Check Sample Source and Lot #	SCP Science Custom Std: Lot S160718008 & SCP Science Custom Std: Lot S160715005

Sample Name	Mass of Sample Material (g)[1]	Volume of Extraction Fluid (mL)[2]	Dilution Factor	Instrument As Result for the Analytical Solution (mg/L)	Results for IVBA extractable As in sample material (mg/kg)
Reagent Blank	NA	100	10	-0.00084	NA
Method Blank	NA	100	10	0.00024	NA
NIST SRM 2710a (Extraction 1)	1.0070	100	100	0.06478	643.3
NIST SRM 2710a (Extraction 2)	1.0051	100	100	0.06440	640.7
NIST SRM 2710a (Extraction 3)	1.0071	100	100	0.06287	624.3
NIST SRM 2710a (Extraction 4)	1.0030	100	100	0.06488	646.9
NIST SRM 2710a (Extraction 5)	1.0082	100	100	0.06208	615.8
Control Soil – NIST SRM 2710a	1.0009	100	100	0.06224	621.8
LCS (Method Blank Spike)	NA	100	100	0.10310	NA
NIST SRM 2710a Matrix Spike	1.0070	100	100	0.17190	NA

1 – Measure 1.00 +/- 0.05 g to the nearest 0.0001g, enter exact mass in table. Extraction mass should not be reduced. If your lab requires more reference material, please contact Clifton Jones at clifton.jones@cbifederalservices.com.

2 – Add 100 +/- 0.5 mL of 0.4M glycine, pH 1.5, delivered via graduated cylinder or automated dispenser.
Results of IVBA Extractable As in sample material (mg/kg) = [Instrument As Results for Analytical Solution (mg/L) x Dilution Factor x 100 mL extraction fluid x (1 L/1000 mL)]/[mass of sample material (g) x (1 kg/1000 g)].

Appendix L
Laboratory Submitted Study Results

LABORATORY D, Table A4. IVBA Extraction Batch Spiked Blank, Spiked Sample, and Control Soil Results for RM Batch (NIST SRM 2710a): Arsenic (As)	
Laboratory Performing Extraction	Laboratory D
Laboratory Performing Analysis	Laboratory D
Method Blank Result (mg/L)	0.00024
LCS (Method Blank Spike) Result (mg/L)	10.31
LCS (Method Blank Spike) Percent Recovery	103.1 %
Average (5) Result RM NIST SRM 2710a (mg/L)	6.48 *
RM NIST SRM 2710a Matrix Spike Result (mg/L)	17.19
RM NIST SRM 2710a Matrix Spike Percent Recovery	107.1 %
Control Soil NIST SRM 2710a Result (mg/Kg)	621.8 mg/Kg
Control Soil NIST SRM 2710a IVBA Extract Acceptance Range - Arsenic (mg/Kg)	(As) 464 – 681 mg/Kg
Control Soil NIST SRM 2710a IVBA Arsenic Nominal Value (mg/Kg)	(As) 573 mg/Kg
Control Soil NIST SRM 2710a Percent Recovery (%)	108.5 %

* Aliquot of NIST SRM 2710a (Extraction 1) extract was spiked post extraction so reported value for that sample only rather than the average of 5

Appendix L
Laboratory Submitted Study Results

LABORATORY D, Table A5. IVBA Extraction Batch Results, (FCRM <250 µm): Arsenic (As)					
Lab Performing Extraction	Laboratory D		Analyst Performing Extraction	[redacted]	
Lab Performing Analysis	Laboratory D		Analyst Performing Analysis	[redacted]	
Instrument Type: ICP-MS or ICP-AES	ICP-MS		Instrument Method Detection Limit (MDL) (ug/L)	MDL = 1.7 ug/L (*3 x IDL*) LLOQ = 10 ug/L (*lowest calibration standard*)	
Extraction Date	06/15/2017		Extraction As Standard Manufacturer and Lot #	SCP Science: Lot S160912001	
Analysis Date(s)	06/22/2017		Analysis As Standard Manufacturer and Lot #	SCP Science Custom Std: Lot S160715008	
Initial Calibration Verification Standard Source and Lot #	SCP Science Custom Std: Lot S160715009		Interference Check Sample Source and Lot #	SCP Science Custom Std: Lot S160718008 & SCP Science Custom Std: Lot S160715005	
Sample Name	Mass of Sample Material (g)[1]	Volume of Extraction Fluid (mL)[2]	Dilution Factor	Instrument As Result for the Analytical Solution (mg/L)	Results for IVBA extractable As in sample material (mg/kg)
Reagent Blank	NA	100	10	0.00000	NA
Method Blank	NA	100	10	-0.00045	NA
FCRM <250 µm (Extraction 1)	1.0031	100	100	0.01588	158.3
FCRM <250 µm (Extraction 2)	1.0046	100	100	0.01641	163.3
FCRM <250 µm (Extraction 3)	1.0081	100	100	0.01616	160.3
FCRM <250 µm (Extraction 4)	1.0020	100	100	0.01573	157.0
FCRM <250 µm (Extraction 5)	1.0047	100	100	0.01588	158.1
Control Soil – NIST SRM 2710A	1.0062	100	100	0.06258	621.9
LCS (Method Blank Spike)	NA	100	100	0.10450	NA
FCRM <250 µm Matrix Spike	1.0031	100	100	0.11270	NA

1 – Measure 1.00 +/- 0.05 g to the nearest 0.0001g, enter exact mass in table. Extraction mass should not be reduced. If your lab requires more reference material, please contact Clifton Jones at clifton.jones@cbifederalservices.com.

2 – Add 100 +/- 0.5 mL of 0.4M glycine, pH 1.5, delivered via graduated cylinder or automated dispenser.
Results of IVBA Extractable As in sample material (mg/kg) = [Instrument As Results for Analytical Solution (mg/L) x Dilution Factor x 100 mL extraction fluid x (1 L/1000 mL)]/[mass of sample material (g) x (1 kg/1000 g)].

Appendix L
Laboratory Submitted Study Results

LABORATORY D, Table A6. IVBA Extraction Batch Spiked Blank, Spiked Sample, and Control Soil Results for RM Batch (FCRM <250 µm): Arsenic (As)	
Laboratory Performing Extraction	Laboratory D
Laboratory Performing Analysis	Laboratory D
Method Blank Result (mg/L)	-0.0045
LCS (Method Blank Spike) Result (mg/L)	10.45
LCS (Method Blank Spike) Percent Recovery	104.5 %
Average (5) Result FCRM <250 µm (mg/L)	1.59 *
FCRM <250 µm Matrix Spike Result (mg/L)	11.27
FCRM <250 µm Matrix Spike Percent Recovery	96.8 %
Control Soil NIST SRM 2710a Result (mg/Kg)	621.9 mg/Kg
Control Soil NIST SRM 2710a IVBA Extract Acceptance Range - Arsenic (mg/Kg)	(As) 464 – 681 mg/Kg
Control Soil NIST SRM 2710a IVBA Arsenic Nominal Value (mg/Kg)	(As) 573 mg/Kg
Control Soil NIST SRM 2710a Percent Recovery (%)	108.5 %

* Aliquot of FCRM <250 (Extraction 1) extract was spiked post extraction so reported value for that sample only rather than average of 5.

Appendix L
Laboratory Submitted Study Results

LABORATORY D, Table A7. IVBA Extraction Batch Results, (FCRM <74 µm): Arsenic (As)			
Lab Performing Extraction	Laboratory D	Analyst Performing Extraction	[redacted]
Lab Performing Analysis	Laboratory D	Analyst Performing Analysis	[redacted]
Instrument Type: ICP-MS or ICP-AES	ICP-MS	Instrument Method Detection Limit (MDL) (ug/L)	MDL = 1.7 ug/L (*3 x IDL*) LLOQ = 10 ug/L (*lowest calibration standard*)
Extraction Date	06/15/2017	Extraction As Standard Manufacturer and Lot #	SCP Science: Lot S160912001
Analysis Date(s)	06/22/2017	Analysis As Standard Manufacturer and Lot #	SCP Science Custom Std: Lot S160715008
Initial Calibration Verification Standard Source and Lot #	SCP Science Custom Std: Lot S160715009	Interference Check Sample Source and Lot #	SCP Science Custom Std: Lot S160718008 & SCP Science Custom Std: Lot S160715005

Sample Name	Mass of Sample Material (g)[1]	Volume of Extraction Fluid (mL)[2]	Dilution Factor	Instrument As Result for the Analytical Solution (mg/L)	Results for IVBA extractable As in sample material (mg/kg)
Reagent Blank	NA	100	10	-0.00021	NA
Method Blank	NA	100	10	-0.00043	NA
FCRM <74 µm (Extraction 1)	1.0041	100	100	0.01624	161.7
FCRM <74 µm (Extraction 2)	1.0029	100	100	0.01653	164.8
FCRM <74 µm (Extraction 3)	1.0048	100	100	0.01628	162.0
FCRM <74 µm (Extraction 4)	1.0051	100	100	0.01613	160.5
FCRM <74 µm (Extraction 5)	1.0036	100	100	0.01529	152.4
Control Soil – NIST SRM 2710A	1.0055	100	100	0.06218	618.4
LCS (Method Blank Spike)	NA	100	100	0.09799	NA
FCRM <74 µm Matrix Spike	1.0041	100	100	0.11510	NA

1 – Measure 1.00 +/- 0.05 g to the nearest 0.0001g, enter exact mass in table. Extraction mass should not be reduced. If your lab requires more reference material, please contact Clifton Jones at clifton.jones@cbifederalservices.com.

2 – Add 100 +/- 0.5 mL of 0.4M glycine, pH 1.5, delivered via graduated cylinder or automated dispenser.
Results of IVBA Extractable As in sample material (mg/kg) = [Instrument As Results for Analytical Solution (mg/L) x Dilution Factor x 100 mL extraction fluid x (1 L/1000 mL)]/[mass of sample material (g) x (1 kg/1000 g)].

Appendix L
Laboratory Submitted Study Results

LABORATORY D, Table A8. IVBA Extraction Batch Spiked Blank, Spiked Sample, and Control Soil Results for RM Batch (FCRM <74 µm): Arsenic (As)	
Laboratory Performing Extraction	Laboratory D
Laboratory Performing Analysis	Laboratory D
Method Blank Result (mg/L)	-0.0043
LCS (Method Blank Spike) Result (mg/L)	9.80
LCS (Method Blank Spike) Percent Recovery	98.0%
Average (5) Result FCRM <250 µm (mg/L)	1.624 *
FCRM <74 µm Matrix Spike Result (mg/L)	11.51
FCRM <74 µm Matrix Spike Percent Recovery	98.9 %
Control Soil NIST SRM 2710a Result (mg/Kg)	618.4 mg/Kg
Control Soil NIST SRM 2710a IVBA Extract Acceptance Range - Arsenic (mg/Kg)	(As) 464 – 681 mg/Kg
Control Soil NIST SRM 2710a IVBA Arsenic Nominal Value (mg/Kg)	(As) 573 mg/Kg
Control Soil NIST SRM 2710a Percent Recovery (%)	107.9 %

* Aliquot of FCRM <74 (Extraction 1) extract was spiked post extraction so reported value for that sample only rather than average of 5.

Appendix L
Laboratory Submitted Study Results

LABORATORY D, Table A9. IVBA Extraction Batch Results, (FCRM <74 µm): Lead (Pb)					
Lab Performing Extraction	Laboratory D		Analyst Performing Extraction	[redacted]	
Lab Performing Analysis	Laboratory D		Analyst Performing Analysis	[redacted]	
Instrument Type: ICP-MS or ICP-AES	ICP-MS		Instrument Method Detection Limit (MDL) (ug/L)	MDL = 0.2 ug/L (*3 x IDL*) LLOQ = 10 ug/L (*lowest calibration standard*)	
Extraction Date	06/15/2017		Extraction Pb Standard Manufacturer and Lot #	SCP Science: Lot S160512028	
Analysis Date(s)	06/22/2017		Analysis Pb Standard Manufacturer and Lot #	SCP Science Custom Std: Lot S160715008	
Initial Calibration Verification Standard Source and Lot #	SCP Science Custom Std: Lot S160715009		Interference Check Sample Source and Lot #	SCP Science Custom Std: Lot S160718008 & SCP Science Custom Std: Lot S160715005	
Sample Name	Mass of Sample Material (g)[1]	Volume of Extraction Fluid (mL)[2]	Dilution Factor	Instrument Pb Result for the Analytical Solution (mg/L)	Results for IVBA extractable Pb in sample material (mg/kg)
Reagent Blank	NA	100	10	0.000348	NA
Method Blank	NA	100	10	0.000276	NA
FCRM <74 µm (Extraction 1)	1.0041	100	100	0.4866	4846.1
FCRM <74 µm (Extraction 2)	1.0029	100	100	0.4841	4827.0
FCRM <74 µm (Extraction 3)	1.0048	100	100	0.4914	4890.5
FCRM <74 µm (Extraction 4)	1.0051	100	100	0.4902	4877.1
FCRM <74 µm (Extraction 5)	1.0036	100	100	0.4601	4584.5
Control Soil – NIST SRM 2710A	1.0055	100	100	0.3368	3349.6
LCS (Method Blank Spike)	NA	100	100	0.09762	NA
FCRM <74 µm Matrix Spike	1.0041	100	100	0.5833	NA

1 – Measure 1.00 +/- 0.05 g to the nearest 0.0001g, enter exact mass in table. Extraction mass should not be reduced. If your lab requires more reference material, please contact Clifton Jones at clifton.jones@cbifederalservices.com.

2 – Add 100 +/- 0.5 mL of 0.4M glycine, pH 1.5, delivered via graduated cylinder or automated dispenser.
Results of IVBA Extractable Pb in sample material (mg/kg) = [Instrument Pb Results for Analytical Solution (mg/L) x Dilution Factor x 100 mL extraction fluid x (1 L/1000 mL)]/[mass of sample material (g) x (1 kg/1000 g)].

Appendix L
Laboratory Submitted Study Results

LABORATORY D, Table A10. IVBA Extraction Batch Spiked Blank, Spiked Sample, and Control Soil Results for RM Batch (FCRM <74 µm): Lead (Pb)	
Laboratory Performing Extraction	Laboratory D
Laboratory Performing Analysis	Laboratory D
Method Blank Result (mg/L)	0.00028
LCS (Method Blank Spike) Result (mg/L)	9.76
LCS (Method Blank Spike) Percent Recovery	97.6 %
Average (5) Result FCRM <250 µm (mg/L)	48.66 *
FCRM <74 µm Matrix Spike Result (mg/L)	58.33
FCRM <74 µm Matrix Spike Percent Recovery	96.7 %
Control Soil NIST SRM 2710a Result (mg/Kg)	3349.6 mg/Kg
Control Soil NIST SRM 2710a IVBA Extract Acceptance Range - Lead (mg/Kg)	(Pb) 3096 – 3785 mg/Kg
Control Soil NIST SRM 2710a IVBA Lead Nominal Value (mg/Kg)	(Pb) 3440 mg/Kg
Control Soil NIST SRM 2710a Percent Recovery (%)	97.4 %

* Aliquot of FCRM <74 (Extraction 1) extract was spiked post extraction so reported value for that sample only rather than average of 5.

Appendix L
Laboratory Submitted Study Results

LABORATORY D, Table B3. Method 3050B or 3051A Digestion Batch Results, (FCRM <250 µm): Arsenic (As)					
Lab Performing Digestion	Laboratory D		Analyst Performing Digestion	[redacted]	
Lab Performing Analysis	Laboratory D		Analyst Performing Analysis	[redacted]	
Instrument Type: ICP-MS or ICP-AES	ICP-AES		Instrument Method Detection Limit (MDL) (ug/L)	MDL = 1.9 ug/L (*3 x IDL*) LLOQ = 10 ug/L (*low level calibration verification standard*)	
Digestion Date	05/10/2017		Digestion As Standard Manufacturer and Lot #	SCP Science: Lot S160912001	
Analysis Date(s)	05/15/2017		Analysis As Standard Manufacturer and Lot #	SCP Science Custom Std: Lot S160715008	
Initial Calibration Verification Standard Source and Lot #	SCP Science Custom Std: Lot S160715009		Interference Check Sample Source and Lot #	SCP Science Custom Std: Lot S160718008 & SCP Science Custom Std: Lot S160715005	
Sample Name	Mass of Sample Material (g)[1]	Volume of Digestion Fluid (mL)[3]	Dilution Factor	Instrument As Result for the Analytical Solution (mg/L)	Results from digested Arsenic in sample material (mg/kg)[2]
Method Blank	NA	10	5	-.0003	NA
FCRM <250 µm (Digestion 1)	0.4998	10	10	.3210	642.3
FCRM <250 µm (Digestion 2)	0.4976	10	10	.3312	665.6
FCRM <250 µm (Digestion 3)	0.4941	10	10	.3448	697.8
FCRM <250 µm (Digestion 4)	0.4991	10	10	.3338	668.8
FCRM <250 µm (Digestion 5)	0.4941	10	10	.3424	693.0
Control Soil – NIST SRM 2710a	0.5021	10	10	.7569	1507.5
LCS (Method Blank Spike)	NA	10	5	.1921	NA
FCRM <250 µm Matrix Spike	0.4998	10	(see table B4)	.5217	NA

1 – Measure 1.00 +/- 0.05 g to the nearest 0.0001g, enter exact mass in table, or if one-half reduced volume digestion is performed, measure 0.50 +/- 0.05 g to the nearest 0.0001g. If your lab requires more reference material, please contact Clifton Jones at clifton.jones@cbifederalservices.com.

2 – Arsenic in sample material (mg/kg) = [Instrument As Results for Analytical Solution (mg/L) x Dilution Factor x 100 mL Digest Final Volume x (1 L/1000 mL)]/[mass of sample material (g) x (1 kg/1000 g)].

3 – The volume entered by the laboratory should be 100 mL instead of 10 mL.

Appendix L
Laboratory Submitted Study Results

LABORATORY D, Table B4. Method 3050B or 3051A Digestion Spiked Blank, and Spiked Sample Results for (FCRM <250 µm): Arsenic (As)	
Laboratory Performing Digestion	Laboratory D
Laboratory Performing Analysis	Laboratory D
Blank Spike Result (mg/L)	9.60 *
Blank Spike Percent Recovery	96.0 %
Average (5) Result RM (mg/L)	0.3210 (raw value)**
RM Matrix Spike Result (mg/L)	0.5217 (raw value)
RM Matrix Spike Percent Recovery	100.4 % ***
Control Soil NIST SRM 2710a Result (mg/Kg)	1507.5
Control Soil NIST SRM 2710a Digestion Acceptance Range - Arsenic (mg/Kg)	(As) 1260 -1540 mg/kg
Control Soil NIST SRM 2710a Arsenic Nominal Value (mg/Kg)	(As) 1400 mg/kg
Control Soil NIST SRM 2710a Percent Recovery (%)	107.7 %

* Blank spike calculated by multiplying the ICP-AES raw value by 50 (10X dilution up to 100 mL multiplied by 5X dilution prior to analysis).

** Aliquot of FCRM <250 (Digestion 1) digest was spiked post digestion so reported raw value for that sample only rather than average of 5.

*** Matrix spike was spiked post digestion at a concentration of 0.2 mg/L, which is equivalent to the concentration of the blank spike (10 ppm) in 10 mL digestion fluid after accounting for dilution. Percent recovery, therefore, was calculated as (RM matrix spike result [mg/L] - FCRM <250 Digestion 1 [mg/L]) / 0.2 mg/L x 100

Appendix L
Laboratory Submitted Study Results

LABORATORY D, Table B5. Method 3050B or 3051A Digestion Batch Results, (FCRM <74 µm): Arsenic (As)			
Lab Performing Digestion	Laboratory D	Analyst Performing Digestion	[redacted]
Lab Performing Analysis	Laboratory D	Analyst Performing Analysis	[redacted]
Instrument Type: ICP-MS or ICP-AES	ICP-AES	Instrument Method Detection Limit (MDL) (ug/L)	MDL = 1.9 (*3 x IDL*) LLOQ = 10 (*low level calibration verification std*)
Digestion Date	05/10/2017	Digestion As Standard Manufacturer and Lot #	SCP Science : Lot S160912001
Analysis Date(s)	05/15/2017	Analysis As Standard Manufacturer and Lot #	SCP Science Custom Std: Lot S160715008
Initial Calibration Verification Standard Source and Lot #	SCP Science Custom Std: Lot S160715009	Interference Check Sample Source and Lot #	SCP Science Custom Std: Lot S160718008 & SCP Science Custom Std: Lot S160715005

Sample Name	Mass of Sample Material (g)[1]	Volume of Digestion Fluid (mL)[3]	Dilution Factor	Instrument As Result for the Analytical Solution (mg/L)	Results from digested Arsenic in sample material (mg/kg)[2]
Method Blank	NA	10	5	.0010	NA
FCRM <74 µm (Digestion 1)	0.4990	10	10	.3428	687.0
FCRM <74 µm (Digestion 2)	0.4926	10	10	.3560	722.7
FCRM <74 µm (Digestion 3)	0.4959	10	10	.3369	679.4
FCRM <74 µm (Digestion 4)	0.4975	10	10	.3298	662.9
FCRM <74 µm (Digestion 5)	0.4972	10	10	.3462	696.3
Control Soil – NIST SRM 2710a	0.4920	10	10	.7239	1471.3
LCS (Method Blank Spike)	NA	10	5	.1909	NA
FCRM <74 µm Matrix Spike	0.4990	10	(see table B6)	.5402	NA

1 – Measure 1.00 +/- 0.05 g to the nearest 0.0001g, enter exact mass in table, or if one-half reduced volume digestion is performed, measure 0.50 +/- 0.05 g to the nearest 0.0001g. If your lab requires more reference material, please contact Clifton Jones at clifton.jones@cbifederalservices.com.

2 – Arsenic in sample material (mg/kg) = [Instrument As Results for Analytical Solution (mg/L) x Dilution Factor x 100 mL Digest Final Volume x (1 L/1000 mL)]/[mass of sample material (g) x (1 kg/1000 g)].

3 – The volume entered by the laboratory should be 100 mL instead of 10 mL.

Appendix L
Laboratory Submitted Study Results

LABORATORY D, Table B6. Method 3050B or 3051A Digestion Spiked Blank, and Spiked Sample Results for (FCRM <74 µm): Arsenic (As)	
Laboratory Performing Digestion	Laboratory D
Laboratory Performing Analysis	Laboratory D
Blank Spike Result (mg/L)	9.55 *
Blank Spike Percent Recovery	95.5%
Average (5) Result RM (mg/L)	0.3428 (raw value) **
RM Matrix Spike Result (mg/L)	0.5402 (raw value)
RM Matrix Spike Percent Recovery	98.7 % ***
Control Soil NIST SRM 2710a Result (mg/Kg)	1471.3
Control Soil NIST SRM 2710a Digestion Acceptance Range - Arsenic (mg/Kg)	(As) 1260 -1540 mg/kg
Control Soil NIST SRM 2710a Arsenic Nominal Value (mg/Kg)	(As) 1400 mg/kg
Control Soil NIST SRM 2710a Percent Recovery (%)	105.1 %

* Blank spike calculated by multiplying the ICP-AES raw value by 50 (10X dilution up to 100 mL multiplied by 5X dilution prior to analysis).

** Aliquot of FCRM <250 (Digestion 1) digest was spiked post digestion so reported raw value for that sample only rather than average of 5.

*** Matrix spike was spiked post digestion at a concentration of 0.2 mg/L, which is equivalent to the concentration of the blank spike (10 ppm) in 10 mL digestion fluid after accounting for dilution. Percent recovery, therefore, was calculated as (RM matrix spike result [mg/L] - FCRM <74 Digestion 1 [mg/L]) / 0.2 mg/L x 100

Appendix L
Laboratory Submitted Study Results

LABORATORY D, Table B7. Method 3050B or 3051A Digestion Batch Results, (FCRM <74 µm): Lead (Pb)					
Lab Performing Digestion	Laboratory D		Analyst Performing Digestion	[redacted]	
Lab Performing Analysis	Laboratory D		Analyst Performing Analysis	[redacted]	
Instrument Type: ICP-MS or ICP-AES	ICP-AES		Instrument Method Detection Limit (MDL) (ug/L)	MDL = 1.5 (*3 x IDL*) LLOQ = 10 (*low level calibration verification std*)	
Digestion Date	05/10/2017		Digestion Pb Standard Manufacturer and Lot #	SCP Science: Lot S160512028	
Analysis Date(s)	05/15/2017		Analysis Pb Standard Manufacturer and Lot #	SCP Science Custom Std: Lot S160715008	
Initial Calibration Verification Standard Source and Lot #	SCP Science Custom Std: Lot S160715009		Interference Check Sample Source and Lot #	SCP Science Custom Std: Lot S160718008 & SCP Science Custom Std: Lot S160715005	
Sample Name	Mass of Sample Material (g)[1]	Volume of Digestion Fluid (mL)[3]	Dilution Factor	Instrument Pb Result for the Analytical Solution (mg/L)	Results from digested Lead in sample material (mg/kg)[2]
Method Blank	NA	10	5	-.0005	NA
FCRM <74 µm (Digestion 1)	0.4990	10	10	3.186	6384.8
FCRM <74 µm (Digestion 2)	0.4926	10	10	3.266	6630.1
FCRM <74 µm (Digestion 3)	0.4959	10	10	3.289	6632.4
FCRM <74 µm (Digestion 4)	0.4975	10	10	3.282	6597.0
FCRM <74 µm (Digestion 5)	0.4972	10	10	3.270	6576.8
Control Soil – NIST SRM 2710a	0.492	10	10	2.568	5219.5
LCS (Method Blank Spike)	NA	10	5	.1965	NA
FCRM <74 µm Matrix Spike	0.4990	10	(see table B8)	3.407	NA

1 – Measure 1.00 +/- 0.05 g to the nearest 0.0001g, enter exact mass in table, or if one-half reduced volume digestion is performed, measure 0.50 +/- 0.05 g to the nearest 0.0001g. If your lab requires more reference material, please contact Clifton Jones at clifton.jones@cbifederalservices.com.

2 – Lead in sample material (mg/kg) = [Instrument Pb Results for Analytical Solution (mg/L) x Dilution Factor x 100 mL Digest Final Volume x (1 L/1000 mL)]/[mass of sample material (g) x (1 kg/1000 g)].

3 – The volume entered by the laboratory should be 100 mL instead of 10 mL.

Appendix L
Laboratory Submitted Study Results

LABORATORY D, Table B8. Method 3050B or 3051A Digestion Spiked Blank, and Spiked Sample Results for (FCRM <74 µm): Lead (Pb)	
Laboratory Performing Digestion	Laboratory D
Laboratory Performing Analysis	Laboratory D
Blank Spike Result (mg/L)	9.83 *
Blank Spike Percent Recovery	98.3 %
Average (5) Result RM (mg/L)	3.186 (raw value) **
RM Matrix Spike Result (mg/L)	3.407 (raw value)
RM Matrix Spike Percent Recovery	110.5 % ***
Control Soil NIST SRM 2710a Result (mg/Kg)	5219.5
Control Soil NIST SRM 2710a Digestion Acceptance Range - Lead (mg/Kg)	(Pb) 4590 -5610 mg/kg
Control Soil NIST SRM 2710a Lead Nominal Value (mg/Kg)	(Pb) 5100 mg/kg
Control Soil NIST SRM 2710a Percent Recovery (%)	102.3%

* Blank spike calculated by multiplying the ICP-AES raw value by 50 (10X dilution up to 100 mL multiplied by 5X dilution prior to analysis).

** Aliquot of FCRM <74 (Digestion 1) digest was spiked post digestion so reported raw value for that sample only rather than average of 5.

*** Matrix spike was spiked post digestion at a concentration of 0.2 mg/L, which is equivalent to the concentration of the blank spike (10 ppm) in 10 mL digestion fluid after accounting for dilution. Percent recovery, therefore, was calculated as (RM matrix spike result [mg/L] - FCRM <74 Digestion 1 [mg/L]) / 0.2 mg/L x 100

Laboratory E

Appendix L
Laboratory Submitted Study Results

LABORATORY E, Table A3. IVBA Extraction Batch Results (NIST SRM 2710a): Arsenic (As) LAB E					
Lab Performing Digestion	Laboratory E		**Analyst Performing Digestion**	[redacted]	
Lab Performing Analysis	Laboratory E		**Analyst Performing Analysis**	[redacted]	
Instrument Type	ICP-MS		**Instrument Method Detection Limit (MDL) (µg/L)**	0.1	
Digestion Date	7 July 2017		**Digestion As Standard Manufacturer and Lot #**	Agilent Technologies #0001062944	
Analysis Date	12 July 2017		**Analysis As Standard Manufacturer and Lot #**	Agilent Technologies #0001062944	
Initial Calibration Verification Standard Source and Lot #	High Purity Standards #1316825		**Interference Check Sample Source and Lot #**	High Purity Standards #1206027; #1311509	
Sample Name	**Mass of Sample Material (g)**	**Vol. of Digestion Fluid (mL)**	**Dilution Factor**	**Instrument As Result for the Analytical Solution (µg/L)**	**Results from IVBA Extractable Arsenic in Sample Material (mg/kg)**
IVBA1: Reagent Blank	-	-	-	<0.000	-
IVBA2: Method Blank	-	100	-	<0.000	-
IVBA3: NIST SRM 2710a (Extraction 1)	1.008	100	99.3 and 1:50	96.614	479.5
IVBA4: NIST SRM 2710a (Extraction 2)	1.008	100	99.3 and 1:50	97.221	482.5
IVBA5: NIST SRM 2710a (Extraction 3)	1.007	100	99.4 and 1:50	95.935	476.6
IVBA6: NIST SRM 2710a (Extraction 4)	1.002	100	99.8 and 1:50	97.116	484.7
IVBA7: NIST SRM 2710a (Extraction 5)	1.005	100	99.6 and 1:50	98.892	492.2
IVBA8: Control Soil – NIST SRM 2710a	1.003	100	99.8 and 1:50	98.116	489.4
IVBA9: LCS (Method Blank Spike)		100	1:50	182.551	9.1
IVBA10: NIST SRM 2710a Matrix Spike	1.003	100	99.8 and 1:50	273.009	1361.6

LABORATORY E, Table A4. IVBA Extraction Batch Spiked Blank, Spiked Sample and Control Soil Results for RM Batch (NIST SRM 2710a): Arsenic (As)	
Laboratory Performing Digestion	Laboratory E
Laboratory Performing Analysis	Laboratory E
Blank Spike Result (mg/L)	10.0
LCS (Method Blank Spike) Result (mg/L)	9.1
LCS (Method Blank Spike) Percent Recovery	91.3
Average (5) Result RM NIST SRM 2710a (µg/L)	483.1
RM NIST SRM 2710a Matrix Spike Result (µg/L)	1361.6
RM NIST SRM 2710a Matrix Spike Percent Recovery	87.9
Control Soil NIST SRM 2710a Result (mg/kg)	489.4
Control soil NIST SRM 2710a IVBA Acceptance Range – Arsenic (mg/kg)	464-681
Control soil NIST SRM 2710a IVBA Arsenic Nominal Value (mg/kg)	573
Control Soil NIST SRM 2710a Percent Recovery	85.4

Appendix L
Laboratory Submitted Study Results

LABORATORY E, Table A5. IVBA Extraction Batch Results (FCRM <250 µm): Arsenic (As)					
Lab Performing Digestion	Laboratory E		**Analyst Performing Digestion**	[redacted]	
Lab Performing Analysis	Laboratory E		**Analyst Performing Analysis**	[redacted]	
Instrument Type	ICP-MS		**Instrument Method Detection Limit (MDL) (µg/L)**	0.1	
Digestion Date	7 July 2017		**Digestion As Standard Manufacturer and Lot #**	Agilent Technologies #0001062944	
Analysis Date	12 July 2017		**Analysis As Standard Manufacturer and Lot #**	Agilent Technologies #0001062944	
Initial Calibration Verification Standard Source and Lot #	High Purity Standards #1316825		**Interference Check Sample Source and Lot #**	High Purity Standards #1206027; #1311509	
Sample Name	**Mass of Sample Material (g)**	**Vol. of Digestion Fluid (mL)**	**Dilution Factor**	**Instrument As Result for the Analytical Solution (µg/L)**	**Results from IVBA Extractable Arsenic in Sample Material (mg/kg)**
IVBA11: Reagent Blank	-	-		<0.000	
IVBA12: Method Blank	-	100		<0.000	
IVBA13: FCRM <250 µm (Extraction 1)	1.005	100	99.5 and 1:50	18.100	90.1
IVBA14: FCRM <250 µm (Extraction 2)	1.002	100	99.8 and 1:50	17.911	89.4
IVBA15: FCRM <250 µm (Extraction 3)	1.002	100	99.9 and 1:50	17.918	89.5
IVBA16: FCRM <250 µm (Extraction 4)	1.004	100	99.6 and 1:50	19.110	95.2
IVBA17: FCRM <250 µm (Extraction 5)	1.004	100	99.6 and 1:50	17.347	86.4
IVBA18: Control Soil – NIST SRM2710a	1.005	100	99.5 and 1:50	93.782	466.6
IVBA19: LCS (Method Blank Spike)		100	1:50	171.896	8.6
IVBA20: FCRM <250 µm Matrix Spike	1.001	100	99.9 and 1:50	195.100	974.5

LABORATORY E, Table A6. IVBA Extraction Batch Spiked Blank, Spiked Sample and Control Soil Results for RM Batch (FCRM <250 µm): Arsenic (As)	
Laboratory Performing Digestion	Laboratory E
Laboratory Performing Analysis	Laboratory E
Blank Spike Result (mg/L)	10.0
LCS (Method Blank Spike) Result (mg/L)	8.6
LCS (Method Blank Spike) Percent Recovery	85.9
Average (5) Result RM FCRM < 250 µm (µg/L)	90.1
FCRM < 250 µm Matrix Spike Result (µg/L)	974.5
FCRM < 250 µm Matrix Spike Percent Recovery	88.4
Control Soil NIST SRM 2710a Result (mg/kg)	466.6
Control soil NIST SRM 2710a IVBA Acceptance Range – Arsenic (mg/kg)	464-681
Control soil NIST SRM 2710a IVBA Arsenic Nominal Value (mg/kg)	573
Control Soil NIST SRM 2710a Percent Recovery	81.4

Appendix L
Laboratory Submitted Study Results

LABORATORY E, Table A7. IVBA Extraction Batch Results (FCRM <74 µm): Arsenic (As)					
Lab Performing Digestion	Laboratory E		**Analyst Performing Digestion**	[redacted]	
Lab Performing Analysis	Laboratory E		**Analyst Performing Analysis**	[redacted]	
Instrument Type	ICP-MS		**Instrument Method Detection Limit (MDL) (µg/L)**	0.1	
Digestion Date	7 July 2017		**Digestion As Standard Manufacturer and Lot #**	Agilent Technologies #0001062944	
Analysis Date	12 July 2017		**Analysis As Standard Manufacturer and Lot #**	Agilent Technologies #0001062944	
Initial Calibration Verification Standard Source and Lot #	High Purity Standards #1316825		**Interference Check Sample Source and Lot #**	High Purity Standards #1206027; #1311509	
Sample Name	**Mass of Sample Material (g)**	**Vol. of Digestion Fluid (mL)**	**Dilution Factor**	**Instrument As Result for the Analytical Solution (µg/L)**	**Results from IVBA Extractable Arsenic in Sample Material (mg/kg)**
IVBA21: Reagent Blank	-	-	-	<0.000	
IVBA22: Method Blank	-	100	-	<0.000	
IVBA23: FCRM <74 µm (Extraction 1)	1.001	100	99.9 and 1:50	17.144	85.6
IVBA24: FCRM <74 µm (Extraction 2)	1.004	100	99.7 and 1:50	17.587	87.6
IVBA25: FCRM <74 µm (Extraction 3)	1.002	100	99.8 and 1:50	15.876	79.2
IVBA26: FCRM <74 µm (Extraction 4)	1.002	100	99.8 and 1:50	15.581	77.8
IVBA27: FCRM <74 µm (Extraction 5)	1.003	100	99.7 and 1:50	15.876	79.1
IVBA28: Control Soil – NIST SRM2710a	1.004	100	99.6 and 1:50	96.699	481.6
IVBA29: LCS (Method Blank Spike)		100	1:50	172.871	8.6
IVBA30: FCRM <74 µm Matrix Spike	1.002	100	99.8 and 1:50	202.532	1010.9

LABORATORY E, Table A8. IVBA Extraction Batch Spiked Blank, Spiked Sample and Control Soil Results for RM Batch (FCRM <74 µm): Arsenic (As)	
Laboratory Performing Digestion	Laboratory E
Laboratory Performing Analysis	Laboratory E
Blank Spike Result (mg/L)	10.0
LCS (Method Blank Spike) Result (mg/L)	8.6
LCS (Method Blank Spike) Percent Recovery	86.4
Average (5) Result FCRM < 74 µm (µg/L)	81.9
FCRM < 74 µm Matrix Spike Result (µg/L)	1010.9
FCRM < 74 µm Matrix Spike Percent Recovery	92.9
Control Soil NIST SRM 2710a Result (mg/kg)	481.6
Control soil NIST SRM 2710a IVBA Acceptance Range – Arsenic (mg/kg)	464-681
Control soil NIST SRM 2710a IVBA Arsenic Nominal Value (mg/kg)	573
Control Soil NIST SRM 2710a Percent Recovery	84.0

Appendix L
Laboratory Submitted Study Results

LABORATORY E, Table A9. IVBA Extraction Batch Results (FCRM <74 µm): Lead (Pb)					
Lab Performing Digestion	Laboratory E		**Analyst Performing Digestion**	[redacted]	
Lab Performing Analysis	Laboratory E		**Analyst Performing Analysis**	[redacted]	
Instrument Type	ICP-MS		**Instrument Method Detection Limit (MDL) (µg/L)**		
Digestion Date	7 July 2017		**Digestion Pb Standard Manufacturer and Lot #**	High Purity Standards #1105409	
Analysis Date	12 July 2017		**Analysis Pb Standard Manufacturer and Lot #**	High Purity Standards #1105409	
Initial Calibration Verification Standard Source and Lot #	High Purity Standards #1316825		**Interference Check Sample Source and Lot #**		

Sample Name	Mass of Sample Material (g)	Vol. of Digestion Fluid (mL)	Dilution Factor	Instrument Pb Result for the Analytical Solution (µg/L)	Results from IVBA Extractable Lead in Sample Material (mg/kg)
IVBA21: Reagent Blank	-	-	-	<0.000	-
IVBA22: Method Blank	-	100	-	<0.000	-
IVBA23: FCRM <74 µm (Extraction 1)	1.001	100	99.9 and 1:50	930.418	4647.4
IVBA24: FCRM <74 µm (Extraction 2)	1.004	100	99.7 and 1:50	945.533	4711.2
IVBA25: FCRM <74 µm (Extraction 3)	1.002	100	99.8 and 1:50	911.693	4548.2
IVBA26: FCRM <74 µm (Extraction 4)	1.002	100	99.8 and 1:50	884.780	4416.2
IVBA27: FCRM <74 µm (Extraction 5)	1.003	100	99.7 and 1:50	917.559	4572.9
IVBA28: Control Soil – NIST SRM2710a	1.004	100	99.6 and 1:50	678.794	3380.5
IVBA29: LCS (Method Blank Spike)		100	1:50	213.982	10.7
IVBA30: FCRM <74 µm Matrix Spike	1.002	100	99.8 and 1:50	1129.403	5637.1

LABORATORY E, Table A10. IVBA Extraction Batch Spiked Blank, Spiked Sample and Control Soil Results for RM Batch (FCRM <74 µm): Lead (Pb)	
Laboratory Performing Digestion	Laboratory E
Laboratory Performing Analysis	Laboratory E
Blank Spike Result (mg/L)	10.0
LCS (Method Blank Spike) Result (mg/L)	10.7
LCS (Method Blank Spike) Percent Recovery	107.0
Average (5) Result FCRM < 74 µm (µg/L)	4579.2
FCRM < 74 µm Matrix Spike Result (µg/L)	5637.1
FCRM < 74 µm Matrix Spike Percent Recovery	105.8
Control Soil NIST SRM 2710a Result (mg/kg)	3380.5
Control soil NIST SRM 2710a IVBA Acceptance Range – Lead (mg/kg)	3096-3785
Control soil NIST SRM 2710a IVBA Lead Nominal Value (mg/kg)	3440
Control Soil NIST SRM 2710a Percent Recovery	98.3

Appendix L
Laboratory Submitted Study Results

LABORATORY E, Table B3. Method 3051A Digestion Batch Results (FCRM < 250 µm): Arsenic (As)					
Lab Performing Digestion	Laboratory E		**Analyst Performing Digestion**	[redacted]	
Lab Performing Analysis	Laboratory E		**Analyst Performing Analysis**	[redacted]	
Instrument Type	ICP-MS		**Instrument Method Detection Limit (MDL) (µg/L)**	0.1 ppb	
Digestion Date	14 June 2017		**Digestion As Standard Manufacturer and Lot #**	Agilent Technologies #0001062944	
Analysis Date	20 June 2017		**Analysis As Standard Manufacturer and Lot #**	Agilent Technologies #0001062944	
Initial Calibration Verification Standard Source and Lot #	High Purity Standards #1316825		**Interference Check Sample Source and Lot #**	High Purity Standards #1206027; #1311509	
Sample Name	**Mass of Sample Material (g)**	**Vol. of Digestion Fluid (mL)**	**Dilution Factor**	**Instrument As Result for the Analytical Solution (µg/L)**	**Results from Digested Arsenic in Sample Material (mg/kg)**
US1: Method Blank	-	-	-	4.364	
US2: FCRM <250 µm (digestion 1)	0.5014	100	199.4 and 1:10	368.009	734.0
US3: FCRM <250 µm (digestion 2)	0.5009	100	199.6 and 1:10	372.438	743.5
US4: FCRM <250 µm (digestion 3)	0.5024	100	199.0 and 1:10	380.311	757.0
US5: FCRM <250 µm (digestion 4)	0.5022	100	199.1 and 1:10	349.631	696.2
US6: FCRM <250 µm (digestion 5)	0.5012	100	199.5 and 1:10	374.555	747.3
US7: Control Soil – NIST SRM 2710a	0.5030	100	198.8 and 1:10	811.889	1614.1
US8: LCS (Method Blank Spike)	-	-	1:10	1030.310	10.30
US9: FCRM <250 µm Matrix Spike	0.5022	100	199.1 and 1:10	1440.524	

LABORATORY E, Table B4. Method 3051A Digestion Spiked Blank and Spiked Sample Results for FCRM <250 µm: Arsenic (As)	
Laboratory Performing Digestion	Laboratory E
Laboratory Performing Analysis	Laboratory E
Blank Spike Result (mg/L)	10.30
Blank Spike Percent Recovery	103.03
Average (5) Result RM (µg/L)	368.989
RM Matrix Spike Result (µg/L)	1440.524
RM Matrix Spike Percent Recovery	107.15
Control Soil NIST SRM 2710a Result (mg/kg)	1614.1
Control soil NIST SRM 2710a Digestion Acceptance Range – Arsenic (mg/kg)	1260-1540
Control soil NIST SRM 2710a Arsenic Nominal Value (mg/kg)	1400
Control Soil NIST SRM 2710a Percent Recovery	115.29

Appendix L
Laboratory Submitted Study Results

LABORATORY E, Table B5. Method 3051A Digestion Batch Results (FCRM < 74 µm): Arsenic (As)					
Lab Performing Digestion	Laboratory E		**Analyst Performing Digestion**	[redacted]	
Lab Performing Analysis	Laboratory E		**Analyst Performing Analysis**	[redacted]	
Instrument Type	ICP-MS		**Instrument Method Detection Limit (MDL) (µg/L)**	0.1 ppb	
Digestion Date	14 June 2017		**Digestion As Standard Manufacturer and Lot #**	Agilent Technologies #0001062944	
Analysis Date	20 June 2017		**Analysis As Standard Manufacturer and Lot #**	Agilent Technologies #0001062944	
Initial Calibration Verification Standard Source and Lot #	High Purity Standards #1316825		**Interference Check Sample Source and Lot #**	High Purity Standards #1206027; #1311509	
Sample Name	**Mass of Sample Material (g)**	**Vol. of Digestion Fluid (mL)**	**Dilution Factor**	**Instrument As Result for the Analytical Solution (µg/L)**	**Results from Digested Arsenic in Sample Material (mg/kg)**
US10: Method Blank	-	-	-	2.320	
US11: FCRM <74 µm (digestion 1)	0.5028	100	198.9 and 1:10	370.958	737.8
US12: FCRM <74 µm (digestion 2)	0.5016	100	199.4 and 1:10	403.781	805.0
US13: FCRM <74 µm (digestion 3)	0.5013	100	199.5 and 1:10	357.473	713.1
US14: FCRM <74 µm (digestion 4)	0.5027	100	198.9 and 1:10	363.650	723.4
US15: FCRM <74 µm (digestion 5)	0.5023	100	199.1 and 1:10	366.623	729.9
US16: Control Soil – NIST SRM 2710a	0.5030	100	198.8 and 1:10	760.812	1512.5
US17: LCS (Method Blank Spike)	-	-	1:10	993.861	9.94
US18: FCRM <74 µm Matrix Spike	0.5027	100	198.9 and 1:10	1492.793	

LABORATORY E, Table B6. Method 3051A Digestion Spiked Blank and Spiked Sample Results for FCRM <74 µm: Arsenic (As)	
Laboratory Performing Digestion	Laboratory E
Laboratory Performing Analysis	Laboratory E
Blank Spike Result (mg/L)	9.93
Blank Spike Percent Recovery	99.39
Average (5) Result RM (µg/L)	372.497
RM Matrix Spike Result (µg/L)	1492.793
RM Matrix Spike Percent Recovery	112.03
Control Soil NIST SRM 2710a Result (mg/kg)	1512.5
Control soil NIST SRM 2710a Digestion Acceptance Range – Arsenic (mg/kg)	1260-1540
Control soil NIST SRM 2710a Arsenic Nominal Value (mg/kg)	1400
Control Soil NIST SRM 2710a Percent Recovery	108.04

Appendix L
Laboratory Submitted Study Results

LABORATORY E, Table B7. Method 3051A Digestion Batch Results, (FCRM <74 µm): Lead (Pb)					
Lab Performing Digestion	Laboratory E		Analyst Performing Digestion	[redacted]	
Lab Performing Analysis	Laboratory E		Analyst Performing Analysis	[redacted]	
Instrument Type: ICP-MS or ICP-AES	ICP-MS		Instrument Method Detection Limit (MDL) (ug/L)	0.1 ppb	
Digestion Date	14 June 2017		Digestion Pb Standard Manufacturer and Lot #	High Purity Standards #1105409	
Analysis Date(s)	20 June 2017		Analysis Pb Standard Manufacturer and Lot #	High Purity Standards #1105409	
Initial Calibration Verification Standard Source and Lot #	High Purity Standards #1316825		Interference Check Sample Source and Lot #		
Sample Name	Mass of Sample Material (g)[1]	Volume of Digestion Fluid (mL)	Dilution Factor	Instrument Pb Result for the Analytical Solution (mg/L)	Results from digested Lead in sample material (mg/kg)[2]
US10: Method Blank	-	-	-	0.466	
US11: FCRM <74 µm (digestion 1)	0.5028	100	198.9 and 1:10	3462.261	6886.0
US12: FCRM <74 µm (digestion 2)	0.5016	100	199.4 and 1:10	3520.129	7017.8
US13: FCRM <74 µm (digestion 3)	0.5013	100	199.5 and 1:10	3308.712	6600.3
US14: FCRM <74 µm (digestion 4)	0.5027	100	198.9 and 1:10	3359.087	6682.1
US15: FCRM <74 µm (digestion 5)	0.5023	100	199.1 and 1:10	3482.955	6934.0
US16: Control Soil – NIST SRM 2710a	0.5030	100	198.8 and 1:10	2590.905	5150.9
US17: LCS (Method Blank Spike)	-	-	1:10	998.309	9.98
US18: FCRM <74 µm Matrix Spike	0.5027	100	198.9 and 1:10	4486.973	

Appendix J
Laboratory Submitted Study Results

LABORATORY E, Table B8. Method 3051A Digestion Spiked Blank, and Spiked Sample Results for (FCRM <74 µm): Lead (Pb)	
Laboratory Performing Digestion	Laboratory E
Laboratory Performing Analysis	Laboratory E
Blank Spike Result (mg/L)	9.98
Blank Spike Percent Recovery	99.83
Average (5) Result RM (mg/L)	3426.629
RM Matrix Spike Result (mg/L)	4486.973
RM Matrix Spike Percent Recovery	106.03
Control Soil NIST SRM 2710a Result (mg/Kg)	5150.9
Control Soil NIST SRM 2710a Digestion Acceptance Range - Lead (mg/Kg)	4590-5610
Control Soil NIST SRM 2710a Lead Nominal Value (mg/Kg)	5100
Control Soil NIST SRM 2710a Percent Recovery (%)	101.0

Laboratory F

Appendix L
Laboratory Submitted Study Results

LABORATORY F, Table A3. IVBA Extraction Batch Results, (NIST SRM 2710a): Arsenic (As)					
Lab Performing Extraction	Laboratory F		Analyst Performing Extraction	[redacted]	
Lab Performing Analysis	Laboratory F		Analyst Performing Analysis	[redacted]	
Instrument Type: ICP-MS or ICP-AES	ICP-AES		Instrument Method Detection Limit (MDL) (ug/L)	40	
Extraction Date	6/1/17		Extraction As Standard Manufacturer and Lot #	SCP Science – S150804001	
Analysis Date(s)	6/5/17		Analysis As Standard Manufacturer and Lot #	Ultra Scientific – CM2913	
Initial Calibration Verification Standard Source and Lot #	SPEX 38-010CR		Interference Check Sample Source and Lot #	Ultra Scientific – CM2913	
Sample Name	Mass of Sample Material (g)[1]	Volume of Extraction Fluid (mL)[2]	Dilution Factor	Instrument As Result for the Analytical Solution (mg/L)	Results for IVBA extractable As in sample material (mg/kg)
Reagent Blank	N/A – 0 g	100	1	<0.025 *	<0.025
Method Blank	0 g	100	10	<0.04	<0.4
NIST SRM 2710a (Extraction 1)	1.0006	100	10	0.4265	426.2
NIST SRM 2710a (Extraction 2)	1.0011	100	10	0.4450	444.5
NIST SRM 2710a (Extraction 3)	1.0001	100	10	0.4469	446.9
NIST SRM 2710a (Extraction 4)	1.0002	100	10	0.4448	444.7
NIST SRM 2710a (Extraction 5)	1.0009	100	10	0.4449	444.5
Control Soil – NIST SRM 2710a	1.0008	100	10	0.4588	458.4
LCS (Method Blank Spike)	0	100	10	0.9197	9.197
NIST SRM 2710a Matrix Spike	1.0000	100	10	1.181	1181

1 – Measure 1.00 +/- 0.05 g to the nearest 0.0001g, enter exact mass in table. Extraction mass should not be reduced. If your lab requires more reference material, please contact Clifton Jones at clifton.jones@cbifederalservices.com.

2 – Add 100 +/- 0.5 mL of 0.4M glycine, pH 1.5, delivered via graduated cylinder or automated dispenser.
Results of IVBA Extractable As in sample material (mg/kg) = [Instrument As Results for Analytical Solution (mg/L) x Dilution Factor x 100 mL extraction fluid x (1 L/1000 mL)]/[mass of sample material (g) x (1 kg/1000 g)].

*Run by ICPMS prior to extraction.

Appendix L
Laboratory Submitted Study Results

LABORATORY F, Table A4. IVBA Extraction Batch Spiked Blank, Spiked Sample, and Control Soil Results for RM Batch (NIST SRM 2710a): Arsenic (As)	
Laboratory Performing Extraction	Laboratory F
Laboratory Performing Analysis	Laboratory F
Method Blank Result (mg/L)	<0.4
LCS (Method Blank Spike) Result (mg/L)	9.197
LCS (Method Blank Spike) Percent Recovery	92%
Average (5) Result RM NIST SRM 2710a (mg/L)	4.42
RM NIST SRM 2710a Matrix Spike Result (mg/L)	11.81
RM NIST SRM 2710a Matrix Spike Percent Recovery	73.9%*
Control Soil NIST SRM 2710a Result (mg/Kg)	458.4
Control Soil NIST SRM 2710a IVBA Extract Acceptance Range - Arsenic (mg/Kg)	(As) 464 – 681 mg/Kg
Control Soil NIST SRM 2710a IVBA Arsenic Nominal Value (mg/Kg)	(As) 573 mg/Kg
Control Soil NIST SRM 2710a Percent Recovery (%)	80%

***Run and extracted multiple times. Last spike recovery = 73.9%**

Appendix L
Laboratory Submitted Study Results

LABORATORY F, Table A5. IVBA Extraction Batch Results, (FCRM <250 µm): Arsenic (As)					
Lab Performing Extraction	Laboratory F		Analyst Performing Extraction	[redacted]	
Lab Performing Analysis	Laboratory F		Analyst Performing Analysis	[redacted]	
Instrument Type: ICP-MS or ICP-AES	ICP-AES		Instrument Method Detection Limit (MDL) (ug/L)	40	
Extraction Date	5/24/17		Extraction As Standard Manufacturer and Lot #	SCP Science – S150804001	
Analysis Date(s)	5/31/17		Analysis As Standard Manufacturer and Lot #	Ultra Scientific – CM2913	
Initial Calibration Verification Standard Source and Lot #	SPEX 38-010CR		Interference Check Sample Source and Lot #	Ultra Scientific – CM2913	
Sample Name	Mass of Sample Material (g)[1]	Volume of Extraction Fluid (mL)[2]	Dilution Factor	Instrument As Result for the Analytical Solution (mg/L)	Results for IVBA extractable As in sample material (mg/kg)
Reagent Blank	N/A – 0 g	100	1	<0.025 *	<0.025
Method Blank	0 g	100	5	<0.04	<0.2
FCRM <250 µm (Extraction 1)	1.0004	100	5	0.2133	106.6
FCRM <250 µm (Extraction 2)	1.0003	100	5	0.2155	107.7
FCRM <250 µm (Extraction 3)	1.0006	100	5	0.2332	116.5
FCRM <250 µm (Extraction 4)	1.0000	100	5	0.2151	107.6
FCRM <250 µm (Extraction 5)	1.0000	100	5	0.2225	111.3
Control Soil – NIST SRM 2710A	1.0001	100	5	0.8791	439.5
LCS (Method Blank Spike)	0	100	5	1.824	9.12
FCRM <250 µm Matrix Spike	1.0000	100	5	8.91	891

1 – Measure 1.00 +/- 0.05 g to the nearest 0.0001g, enter exact mass in table. Extraction mass should not be reduced. If your lab requires more reference material, please contact Clifton Jones at clifton.jones@cbifederalservices.com.

2 – Add 100 +/- 0.5 mL of 0.4M glycine, pH 1.5, delivered via graduated cylinder or automated dispenser.
Results of IVBA Extractable As in sample material (mg/kg) = [Instrument As Results for Analytical Solution (mg/L) x Dilution Factor x 100 mL extraction fluid x (1 L/1000 mL)]/[mass of sample material (g) x (1 kg/1000 g)].

* Run by ICPMS prior to extraction.

Appendix L
Laboratory Submitted Study Results

LABORATORY F, Table A6. IVBA Extraction Batch Spiked Blank, Spiked Sample, and Control Soil Results for RM Batch (FCRM <250 µm): Arsenic (As)	
Laboratory Performing Extraction	Laboratory F
Laboratory Performing Analysis	Laboratory F
Method Blank Result (mg/L)	<0.03
LCS (Method Blank Spike) Result (mg/L)	9.12
LCS (Method Blank Spike) Percent Recovery	91.2%
Average (5) Result FCRM <250 µm (mg/L)	1.10
FCRM <250 µm Matrix Spike Result (mg/L)	8.91
FCRM <250 µm Matrix Spike Percent Recovery	78.1%
Control Soil NIST SRM 2710a Result (mg/Kg)	439.5
Control Soil NIST SRM 2710a IVBA Extract Acceptance Range – Arsenic (mg/Kg)	(As) 464 – 681 mg/Kg
Control Soil NIST SRM 2710a IVBA Arsenic Nominal Value (mg/Kg)	(As) 573 mg/Kg
Control Soil NIST SRM 2710a Percent Recovery (%)	76.7%

***Run and extracted multiple times. Last spike recovery = 73.9%**

Appendix L
Laboratory Submitted Study Results

LABORATORY F, Table A7. IVBA Extraction Batch Results, (FCRM <74 µm): Arsenic (As)					
Lab Performing Extraction	Laboratory F		Analyst Performing Extraction	[redacted]	
Lab Performing Analysis	Laboratory F		Analyst Performing Analysis	[redacted]	
Instrument Type: ICP-MS or ICP-AES	ICP-AES		Instrument Method Detection Limit (MDL) (ug/L)	40	
Extraction Date	5/24/17		Extraction As Standard Manufacturer and Lot #	SCP Science – S150804001	
Analysis Date(s)	5/31/17		Analysis As Standard Manufacturer and Lot #	Ultra Scientific – CM2913	
Initial Calibration Verification Standard Source and Lot #	SPEX 38-010CR		Interference Check Sample Source and Lot #	Ultra Scientific – CM2913	
Sample Name	Mass of Sample Material (g)[1]	Volume of Extraction Fluid (mL)[2]	Dilution Factor	Instrument As Result for the Analytical Solution (mg/L)	Results for IVBA extractable As in sample material (mg/kg)
Reagent Blank	0	100	1	<0.025*	<0.025*
Method Blank	0	100	5	<0.04	<0.04
FCRM <74 µm (Extraction 1)	1.0010	100	5	0.2461	122.9
FCRM <74 µm (Extraction 2)	1.0007	100	5	0.2417	120.8
FCRM <74 µm (Extraction 3)	1.0003	100	5	0.2465	123.2
FCRM <74 µm (Extraction 4)	1.0003	100	5	0.2413	120.6
FCRM <74 µm (Extraction 5)	1.0000	100	5	0.2376	118.8
Control Soil – NIST SRM 2710A	1.0008	100	5	0.9357	467.5
LCS (Method Blank Spike)	0	100	5	1.817	9.085
FCRM <74 µm Matrix Spike	1.0003	100	5	1.817	908.2

1 – Measure 1.00 +/- 0.05 g to the nearest 0.0001g, enter exact mass in table. Extraction mass should not be reduced. If your lab requires more reference material, please contact Clifton Jones at clifton.jones@cbifederalservices.com.

2 – Add 100 +/- 0.5 mL of 0.4M glycine, pH 1.5, delivered via graduated cylinder or automated dispenser.
Results of IVBA Extractable As in sample material (mg/kg) = [Instrument As Results for Analytical Solution (mg/L) x Dilution Factor x 100 mL extraction fluid x (1 L/1000 mL)]/[mass of sample material (g) x (1 kg/1000 g)].

*Run by ICPMS prior to extraction.

Appendix L
Laboratory Submitted Study Results

LABORATORY F, Table A8. IVBA Extraction Batch Spiked Blank, Spiked Sample, and Control Soil Results for RM Batch (FCRM <74 µm): Arsenic (As)	
Laboratory Performing Extraction	Laboratory F
Laboratory Performing Analysis	Laboratory F
Method Blank Result (mg/L)	<0.04
LCS (Method Blank Spike) Result (mg/L)	9.085
LCS (Method Blank Spike) Percent Recovery	90.9%
Average (5) Result FCRM <250 µm (mg/L)	1.2132
FCRM <74 µm Matrix Spike Result (mg/L)	9.085
FCRM <74 µm Matrix Spike Percent Recovery	78.7%
Control Soil NIST SRM 2710a Result (mg/Kg)	467.5
Control Soil NIST SRM 2710a IVBA Extract Acceptance Range - Arsenic (mg/Kg)	(As) 464 – 681 mg/Kg
Control Soil NIST SRM 2710a IVBA Arsenic Nominal Value (mg/Kg)	(As) 573 mg/Kg
Control Soil NIST SRM 2710a Percent Recovery (%)	81.6%

Appendix L
Laboratory Submitted Study Results

LABORATORY F, Table A9. IVBA Extraction Batch Results, (FCRM <74 µm): Lead (Pb)					
Lab Performing Extraction	Laboratory F		Analyst Performing Extraction	[redacted]	
Lab Performing Analysis	Laboratory F		Analyst Performing Analysis	[redacted]	
Instrument Type: ICP-MS or ICP-AES	ICP-AES		Instrument Method Detection Limit (MDL) (ug/L)	30	
Extraction Date	5/24/17		Extraction Pb Standard Manufacturer and Lot #	Ultra Scientific – CM3389	
Analysis Date(s)	5/31/17		Analysis Pb Standard Manufacturer and Lot #	Ultra Scientific – CM3300	
Initial Calibration Verification Standard Source and Lot #	SPEX 38-010CR		Interference Check Sample Source and Lot #	Ultra Scientific – CM3389	
Sample Name	Mass of Sample Material (g)[1]	Volume of Extraction Fluid (mL)[2]	Dilution Factor	Instrument Pb Result for the Analytical Solution (mg/L)	Results for IVBA extractable Pb in sample material (mg/kg)
Reagent Blank	0	100	1	<0.03	<0.03*
Method Blank	0	100	5	0.0035	0.0175
FCRM <74 µm (Extraction 1)	1.0010	100	5	8.319	4155.3
FCRM <74 µm (Extraction 2)	1.0007	100	5	8.172	4083.1
FCRM <74 µm (Extraction 3)	1.0003	100	5	8.207	4102.3
FCRM <74 µm (Extraction 4)	1.0003	100	5	8.152	4074.8
FCRM <74 µm (Extraction 5)	1.0000	100	5	8.345	4172.5
Control Soil – NIST SRM 2710A	1.0008	100	5	5.593	2794.3
LCS (Method Blank Spike)	0	100	5	1.731	8.66
FCRM <74 µm Matrix Spike	1.0003	100	5	9.950	4973.5

1 – Measure 1.00 +/- 0.05 g to the nearest 0.0001g, enter exact mass in table. Extraction mass should not be reduced. If your lab requires more reference material, please contact Clifton Jones at clifton.jones@cbifederalservices.com.

2 – Add 100 +/- 0.5 mL of 0.4M glycine, pH 1.5, delivered via graduated cylinder or automated dispenser.
Results of IVBA Extractable Pb in sample material (mg/kg) = [Instrument Pb Results for Analytical Solution (mg/L) x Dilution Factor x 100 mL extraction fluid x (1 L/1000 mL)]/[mass of sample material (g) x (1 kg/1000 g)].

*Run by ICPMS prior to extraction.

Appendix L
Laboratory Submitted Study Results

LABORATORY F, Table A10. IVBA Extraction Batch Spiked Blank, Spiked Sample, and Control Soil Results for RM Batch (FCRM <74 µm): Lead (Pb)	
Laboratory Performing Extraction	Laboratory F
Laboratory Performing Analysis	Laboratory F
Method Blank Result (mg/L)	0.0175
LCS (Method Blank Spike) Result (mg/L)	8.66
LCS (Method Blank Spike) Percent Recovery	86.4%
Average (5) Result FCRM <250 µm (mg/L)	41.2
FCRM <74 µm Matrix Spike Result (mg/L)	49.75
FCRM <74 µm Matrix Spike Percent Recovery	85.5%
Control Soil NIST SRM 2710a Result (mg/Kg)	2794.3
Control Soil NIST SRM 2710a IVBA Extract Acceptance Range – Lead (mg/Kg)	(Pb) 3096 – 3785 mg/Kg
Control Soil NIST SRM 2710a IVBA Lead Nominal Value (mg/Kg)	(Pb) 3440 mg/Kg
Control Soil NIST SRM 2710a Percent Recovery (%)	81.2%

Appendix L
Laboratory Submitted Study Results

LABORATORY F, Table B3. Method 3050B or 3051A Digestion Batch Results, (FCRM <250 µm): Arsenic (As)					
Lab Performing Digestion	Laboratory F		Analyst Performing Digestion	[redacted]	
Lab Performing Analysis	Laboratory F		Analyst Performing Analysis	[redacted]	
Instrument Type: ICP-MS or ICP-AES	ICP-AES		Instrument Method Detection Limit (MDL) (ug/L)	40	
Digestion Date	5/23/17		Digestion As Standard Manufacturer and Lot #	SCP Science – S150804001	
Analysis Date(s)	5/31/17		Analysis As Standard Manufacturer and Lot #	Ultra Scientific – CM2913	
Initial Calibration Verification Standard Source and Lot #	SPEX 38-010CR		Interference Check Sample Source and Lot #	Ultra Scientific – CM2913	
Sample Name	Mass of Sample Material (g)[1]	Volume of Digestion Fluid (mL)	Dilution Factor	Instrument As Result for the Analytical Solution (mg/L)	Results from digested Arsenic in sample material (mg/kg)[2]
Method Blank	0	50	100	0.0092	<4
FCRM <250 µm (Digestion 1)	0.5003	50	100	6.938	694
FCRM <250 µm (Digestion 2)	0.5004	50	100	6.910	691
FCRM <250 µm (Digestion 3)	0.5001	50	100	6.794	679
FCRM <250 µm (Digestion 4)	0.5000	50	100	7.265	727
FCRM <250 µm (Digestion 5)	0.5002	50	100	6.879	688
Control Soil – NIST SRM 2710a	0.5002	50	100	14.70	1470
LCS (Method Blank Spike)	0.5000	50	100	9.625	962.5
FCRM <250 µm Matrix Spike	0.5002	50	100	16.56	1656

1 – Measure 1.00 +/- 0.05 g to the nearest 0.0001g, enter exact mass in table, or if one-half reduced volume digestion is performed, measure 0.50 +/- 0.05 g to the nearest 0.0001g. If your lab requires more reference material, please contact Clifton Jones at clifton.jones@cbifederalservices.com.

2 – Arsenic in sample material (mg/kg) = [Instrument As Results for Analytical Solution (mg/L) x Dilution Factor x 100 mL Digest Final Volume x (1 L/1000 mL)]/[mass of sample material (g) x (1 kg/1000 g)].

Appendix L
Laboratory Submitted Study Results

LABORATORY F, Table B4. Method 3050B or 3051A Digestion Spiked Blank, and Spiked Sample Results for (FCRM <250 µm): Arsenic (As)	
Laboratory Performing Digestion	Laboratory F
Laboratory Performing Analysis	Laboratory F
Blank Spike Result (mg/L)	9.625
Blank Spike Percent Recovery	96%
Average (5) Result RM (mg/L)	6.957
RM Matrix Spike Result (mg/L)	16.56
RM Matrix Spike Percent Recovery	96%
Control Soil NIST SRM 2710a Result (mg/Kg)	1470
Control Soil NIST SRM 2710a Digestion Acceptance Range - Arsenic (mg/Kg)	(As) 1260 -1540 mg/kg
Control Soil NIST SRM 2710a Arsenic Nominal Value (mg/Kg)	(As) 1400 mg/kg
Control Soil NIST SRM 2710a Percent Recovery (%)	105

Appendix L
Laboratory Submitted Study Results

LABORATORY F, Table B5. Method 3050B or 3051A Digestion Batch Results, (FCRM <74 µm): Arsenic (As)					
Lab Performing Digestion	Laboratory F		Analyst Performing Digestion	[redacted]	
Lab Performing Analysis	Laboratory F		Analyst Performing Analysis	[redacted]	
Instrument Type: ICP-MS or ICP-AES	ICP-AES		Instrument Method Detection Limit (MDL) (ug/L)	40	
Digestion Date	5/25/17		Digestion As Standard Manufacturer and Lot #	SCP Science – S150804001	
Analysis Date(s)	5/26/17		Analysis As Standard Manufacturer and Lot #	Ultra Scientific – CM2913	
Initial Calibration Verification Standard Source and Lot #	SPEX 38-010CR		Interference Check Sample Source and Lot #	Ultra Scientific – CM2913	
Sample Name	Mass of Sample Material (g)[1]	Volume of Digestion Fluid (mL)	Dilution Factor	Instrument As Result for the Analytical Solution (mg/L)	Results from digested Arsenic in sample material (mg/kg)[2]
Method Blank	0	50	100	<0.04	<40
FCRM <74 µm (Digestion 1)	0.5001	50	100	7.080	708
FCRM <74 µm (Digestion 2)	0.5002	50	100	6.761	676
FCRM <74 µm (Digestion 3)	0.5001	50	100	6.704	670
FCRM <74 µm (Digestion 4)	0.5000	50	100	7.002	700
FCRM <74 µm (Digestion 5)	0.5004	50	100	7.163	716
Control Soil – NIST SRM 2710a	0.5003	50	100	14.56	1460
LCS (Method Blank Spike)	0.5000	50	100	9.672	967
FCRM <74 µm Matrix Spike	0.5000	50	100	16.04	1604

1 – Measure 1.00 +/- 0.05 g to the nearest 0.0001g, enter exact mass in table, or if one-half reduced volume digestion is performed, measure 0.50 +/- 0.05 g to the nearest 0.0001g. If your lab requires more reference material, please contact Clifton Jones at clifton.jones@cbifederalservices.com.

2 – Arsenic in sample material (mg/kg) = [Instrument As Results for Analytical Solution (mg/L) x Dilution Factor x 100 mL Digest Final Volume x (1 L/1000 mL)]/[mass of sample material (g) x (1 kg/1000 g)].

Appendix L
Laboratory Submitted Study Results

LABORATORY F, Table B6. Method 3050B or 3051A Digestion Spiked Blank, and Spiked Sample Results for (FCRM <74 µm): Arsenic (As)	
Laboratory Performing Digestion	Laboratory F
Laboratory Performing Analysis	Laboratory F
Blank Spike Result (mg/L)	967
Blank Spike Percent Recovery	96.7%
Average (5) Result RM (mg/L)	6.942
RM Matrix Spike Result (mg/L)	16.04
RM Matrix Spike Percent Recovery	91%
Control Soil NIST SRM 2710a Result (mg/Kg)	1460
Control Soil NIST SRM 2710a Digestion Acceptance Range - Arsenic (mg/Kg)	(As) 1260 -1540 mg/kg
Control Soil NIST SRM 2710a Arsenic Nominal Value (mg/Kg)	(As) 1400 mg/kg
Control Soil NIST SRM 2710a Percent Recovery (%)	104.3

Appendix L
Laboratory Submitted Study Results

LABORATORY F, Table B7. Method 3050B or 3051A Digestion Batch Results, (FCRM <74 µm): Lead (Pb)					
Lab Performing Digestion	Laboratory F		Analyst Performing Digestion	[redacted]	
Lab Performing Analysis	Laboratory F		Analyst Performing Analysis	[redacted]	
Instrument Type: ICP-MS or ICP-AES	ICP-AES		Instrument Method Detection Limit (MDL) (ug/L)	30	
Digestion Date	5/25/17		Digestion Pb Standard Manufacturer and Lot #	Ultra Scientific – CM3389	
Analysis Date(s)	5/26/17		Analysis Pb Standard Manufacturer and Lot #	Ultra Scientific – CM3300	
Initial Calibration Verification Standard Source and Lot #	SPEX 38-010CR		Interference Check Sample Source and Lot #	Ultra Scientific – CM3389	
Sample Name	Mass of Sample Material (g)[1]	Volume of Digestion Fluid (mL)	Dilution Factor	Instrument Pb Result for the Analytical Solution (mg/L)	Results from digested Lead in sample material (mg/kg)[2]
Method Blank	0	50	100	<0.03	<3
FCRM <74 µm (Digestion 1)	0.5001	50	100	62.24	6220
FCRM <74 µm (Digestion 2)	0.5002	50	100	63.54	6350
FCRM <74 µm (Digestion 3)	0.5001	50	100	63.22	6320
FCRM <74 µm (Digestion 4)	0.5000	50	100	62.40	6240
FCRM <74 µm (Digestion 5)	0.5004	50	100	61.75	6180
Control Soil – NIST SRM 2710a	0.5003	50	100	50.85	5090
LCS (Method Blank Spike)	0.5000	50	100	10.01	1001
FCRM <74 µm Matrix Spike	0.5000	50	100	71.86	7186

1 – Measure 1.00 +/- 0.05 g to the nearest 0.0001g, enter exact mass in table, or if one-half reduced volume digestion is performed, measure 0.50 +/- 0.05 g to the nearest 0.0001g. If your lab requires more reference material, please contact Clifton Jones at clifton.jones@cbifederalservices.com.

2 – Lead in sample material (mg/kg) = [Instrument Pb Results for Analytical Solution (mg/L) x Dilution Factor x 100 mL Digest Final Volume x (1 L/1000 mL)]/[mass of sample material (g) x (1 kg/1000 g)].

Appendix J
Laboratory Submitted Study Results

LABORATORY F, Table B8. Method 3050B or 3051A Digestion Spiked Blank, and Spiked Sample Results for (FCRM <74 µm): Lead (Pb)	
Laboratory Performing Digestion	Laboratory F
Laboratory Performing Analysis	Laboratory F
Blank Spike Result (mg/L)	10.01
Blank Spike Percent Recovery	100.1%
Average (5) Result RM (mg/L)	62.63
RM Matrix Spike Result (mg/L)	71.86
RM Matrix Spike Percent Recovery	92.3%
Control Soil NIST SRM 2710a Result (mg/Kg)	5090
Control Soil NIST SRM 2710a Digestion Acceptance Range - Lead (mg/Kg)	(Pb) 4590 -5610 mg/kg
Control Soil NIST SRM 2710a Lead Nominal Value (mg/Kg)	(Pb) 5100 mg/kg
Control Soil NIST SRM 2710a Percent Recovery (%)	99.8

Laboratory G

Appendix L
Laboratory Submitted Study Results

LABORATORY G, Table A3. IVBA Extraction Batch Results, (NIST SRM 2710a): Arsenic (As) – Control Soil LOW; all other QC met			
Lab Performing Extraction	Laboratory G	Analyst Performing Extraction	[redacted]
Lab Performing Analysis	Laboratory G	Analyst Performing Analysis	[redacted]
Instrument Type: ICP-MS or ICP-AES	ICP-AES	Instrument Method Detection Limit (MDL) (ug/L)	4
Extraction Date	May 18, 2017	Extraction As Standard Manufacturer and Lot #	Sigma Lot BCBP4276V
Analysis Date(s)	Jun 14, 2017	Analysis As Standard Manufacturer and Lot #	Inorganic Ventures Lot K2-MEB651894
Initial Calibration Verification Standard Source and Lot #	Inorganic Ventures Lot K2-MEB651896	Interference Check Sample Source and Lot #	Ti – Inorganic Ventures Lot J2-TI02101R V – Inorganic Ventures Low J2-V02101 Mn – Inorganic Ventures Lot K2-MN02127 Ni – Inorganic Ventures Lot K2NI02106 Cr – Inorganic Ventures Lot K2-CR03122 Cu – Inorganic Ventures Lot J2-CU03024 Al, Ca, Fe, Mg – Inorganic Ventures Lot K2-MEB643109

Sample Name	Mass of Sample Material (g)[1]	Volume of Extraction Fluid (mL)[2]	Dilution Factor	Instrument As Result for the Analytical Solution (mg/L)	Results for IVBA extractable As in sample material (mg/kg)
Reagent Blank		100	1.000	-0.00768994	
Method Blank		100	1.000	-0.00823989	
NIST SRM 2710a (Extraction 1)	0.9708	100	10.00	0.431606	445
NIST SRM 2710a (Extraction 2)	0.9575	100	10.00	0.443194	463
NIST SRM 2710a (Extraction 3)	0.9902	100	10.00	0.455354	460
NIST SRM 2710a (Extraction 4)	1.0109	100	10.00	0.472325	467
NIST SRM 2710a (Extraction 5)	1.0141	100	10.00	0.471219	465
Control Soil – NIST SRM 2710a	1.0345	100	10.00	0.472832	**457**
LCS (Method Blank Spike)		100	10.00	0.898596	
NIST SRM 2710a Matrix Spike	1.0010	100	10.00	1.37078	

1 – Measure 1.00 +/- 0.05 g to the nearest 0.0001g, enter exact mass in table. Extraction mass should not be reduced. If your lab requires more reference material, please contact Clifton Jones at clifton.jones@cbifederalservices.com.

2 – Add 100 +/- 0.5 mL of 0.4M glycine, pH 1.5, delivered via graduated cylinder or automated dispenser.
Results of IVBA Extractable As in sample material (mg/kg) = [Instrument As Results for Analytical Solution (mg/L) x Dilution Factor x 100 mL extraction fluid x (1 L/1000 mL)]/[mass of sample material (g) x (1 kg/1000 g)].

Appendix L
Laboratory Submitted Study Results

LABORATORY G, Table A4. IVBA Extraction Batch Spiked Blank, Spiked Sample, and Control Soil Results for RM Batch (NIST SRM 2710a): Arsenic (As)	
Laboratory Performing Extraction	Laboratory G
Laboratory Performing Analysis	Laboratory G
Method Blank Result (mg/L)	< 0.010
LCS (Method Blank Spike) Result (mg/L)	8.98596
LCS (Method Blank Spike) Percent Recovery	90
Average (5) Result RM NIST SRM 2710a (mg/L)	4.547396
RM NIST SRM 2710a Matrix Spike Result (mg/L)	13.7078
RM NIST SRM 2710a Matrix Spike Percent Recovery	92
Control Soil NIST SRM 2710a Result (mg/Kg)	457
Control Soil NIST SRM 2710a IVBA Extract Acceptance Range - Arsenic (mg/Kg)	(As) 464 – 681 mg/Kg
Control Soil NIST SRM 2710a IVBA Arsenic Nominal Value (mg/Kg)	(As) 573 mg/Kg
Control Soil NIST SRM 2710a Percent Recovery (%)	80

Appendix L
Laboratory Submitted Study Results

LABORATORY G, Table A3- RE-EXTRACTION. IVBA Extraction Batch Results, (NIST SRM 2710a): Arsenic (As) – Control Soil LOW; all other QC met			
Lab Performing Extraction	Laboratory G	Analyst Performing Extraction	[redacted]
Lab Performing Analysis	Laboratory G	Analyst Performing Analysis	[redacted]
Instrument Type: ICP-MS or ICP-AES	ICP-AES	Instrument Method Detection Limit (MDL) (ug/L)	4
Extraction Date	June 19, 2017	Extraction As Standard Manufacturer and Lot #	Sigma Lot BCBP4276V
Analysis Date(s)	Jun 30, 2017	Analysis As Standard Manufacturer and Lot #	Inorganic Ventures Lot K2-MEB651894
Initial Calibration Verification Standard Source and Lot #	Inorganic Ventures Lot K2-MEB651896	Interference Check Sample Source and Lot #	Ti – Inorganic Ventures Lot J2-TI02102R V – Inorganic Ventures Low J2-V02101 Mn – Inorganic Ventures Lot K2-MN02127 Ni – Inorganic Ventures Lot K2NI02106 Cr – Inorganic Ventures Lot K2-CR03122 Cu – Inorganic Ventures Lot J2-CU03024 Al, Ca, Fe, Mg – Inorganic Ventures Lot K2-MEB643109

Sample Name	Mass of Sample Material (g)[1]	Volume of Extraction Fluid (mL)[2]	Dilution Factor	Instrument As Result for the Analytical Solution (mg/L)	Results for IVBA extractable As in sample material (mg/kg)
Reagent Blank		100	1	-0.0109807	
Method Blank		100	1	-0.0113881	
NIST SRM 2710a (Extraction 1)	1.0113	100	100	0.0418168	413
NIST SRM 2710a (Extraction 2)	0.9623	100	100	0.0404696	421
NIST SRM 2710a (Extraction 3)	0.9883	100	100	0.0406799	412
NIST SRM 2710a (Extraction 4)	1.0413	100	100	0.0415812	399
NIST SRM 2710a (Extraction 5)	0.9867	100	100	0.0404025	409
Control Soil – NIST SRM 2710a	0.9954	100	100	0.0404631	**407**
LCS (Method Blank Spike)		100	100	0.0849563	
NIST SRM 2710a Matrix Spike	1.0236	100	100	0.129847	

1 – Measure 1.00 +/- 0.05 g to the nearest 0.0001g, enter exact mass in table. Extraction mass should not be reduced. If your lab requires more reference material, please contact Clifton Jones at clifton.jones@cbifederalservices.com.

2 – Add 100 +/- 0.5 mL of 0.4M glycine, pH 1.5, delivered via graduated cylinder or automated dispenser.
Results of IVBA Extractable As in sample material (mg/kg) = [Instrument As Results for Analytical Solution (mg/L) x Dilution Factor x 100 mL extraction fluid x (1 L/1000 mL)]/[mass of sample material (g) x (1 kg/1000 g)].

Appendix L
Laboratory Submitted Study Results

LABORATORY G, Table A4 – RE-EXTRACTION. IVBA Extraction Batch Spiked Blank, Spiked Sample, and Control Soil Results for RM Batch (NIST SRM 2710a): Arsenic (As)	
Laboratory Performing Extraction	Laboratory G
Laboratory Performing Analysis	Laboratory G
Method Blank Result (mg/L)	< 0.010
LCS (Method Blank Spike) Result (mg/L)	8.49563
LCS (Method Blank Spike) Percent Recovery	85
Average (5) Result RM NIST SRM 2710a (mg/L)	4.099
RM NIST SRM 2710a Matrix Spike Result (mg/L)	12.9847
RM NIST SRM 2710a Matrix Spike Percent Recovery	89
Control Soil NIST SRM 2710a Result (mg/Kg)	**407**
Control Soil NIST SRM 2710a IVBA Extract Acceptance Range - Arsenic (mg/Kg)	(As) 464 – 681 mg/Kg
Control Soil NIST SRM 2710a IVBA Arsenic Nominal Value (mg/Kg)	(As) 573 mg/Kg
Control Soil NIST SRM 2710a Percent Recovery (%)	**71**

Appendix L
Laboratory Submitted Study Results

LABORATORY G, Table A5. IVBA Extraction Batch Results, (FCRM <250 µm): Arsenic (As) – Control Soil LOW; all other QC met			
Lab Performing Extraction	Laboratory G	Analyst Performing Extraction	[redacted]
Lab Performing Analysis	Laboratory G	Analyst Performing Analysis	[redacted]
Instrument Type: ICP-MS or ICP-AES	ICP-AES	Instrument Method Detection Limit (MDL) (ug/L)	4
Extraction Date	May 18, 2017	Extraction As Standard Manufacturer and Lot #	Sigma Lot BCBP4276V
Analysis Date(s)	Jun 14, 2017	Analysis As Standard Manufacturer and Lot #	Inorganic Ventures Lot K2-MEB651894
Initial Calibration Verification Standard Source and Lot #	Inorganic Ventures Lot K2-MEB651896	Interference Check Sample Source and Lot #	Ti – Inorganic Ventures Lot J2-TI02101R V – Inorganic Ventures Low J2-V02101 Mn – Inorganic Ventures Lot K2-MN02127 Ni – Inorganic Ventures Lot K2NI02106 Cr – Inorganic Ventures Lot K2-CR03122 Cu – Inorganic Ventures Lot J2-CU03024 Al, Ca, Fe, Mg – Inorganic Ventures Lot K2-MEB643109

Sample Name	Mass of Sample Material (g)[1]	Volume of Extraction Fluid (mL)[2]	Dilution Factor	Instrument As Result for the Analytical Solution (mg/L)	Results for IVBA extractable As in sample material (mg/kg)
Reagent Blank		100	1.000	0.0142658	
Method Blank		100	1.000	0.00317120	
FCRM <250 µm (Extraction 1)	0.9870	100	10.00	0.120821	139
FCRM <250 µm (Extraction 2)	1.0270	100	10.00	0.122509	119
FCRM <250 µm (Extraction 3)	1.0184	100	10.00	0.123462	118
FCRM <250 µm (Extraction 4)	0.9702	100	10.00	0.120243	122
FCRM <250 µm (Extraction 5)	1.0441	100	10.00	0.122158	115
Control Soil – NIST SRM 2710A	1.0128	100	10.00	0.467168	**461**
LCS (Method Blank Spike)		100	10.00	0.918028	
FCRM <250 µm Matrix Spike	1.0206	100	10.00	1.09746	

1 – Measure 1.00 +/- 0.05 g to the nearest 0.0001g, enter exact mass in table. Extraction mass should not be reduced. If your lab requires more reference material, please contact Clifton Jones at clifton.jones@cbifederalservices.com.

2 – Add 100 +/- 0.5 mL of 0.4M glycine, pH 1.5, delivered via graduated cylinder or automated dispenser.
Results of IVBA Extractable As in sample material (mg/kg) = [Instrument As Results for Analytical Solution (mg/L) x Dilution Factor x 100 mL extraction fluid x (1 L/1000 mL)]/[mass of sample material (g) x (1 kg/1000 g)].

Appendix L
Laboratory Submitted Study Results

LABORATORY G, Table A6. IVBA Extraction Batch Spiked Blank, Spiked Sample, and Control Soil Results for RM Batch (FCRM <250 µm): Arsenic (As)	
Laboratory Performing Extraction	Laboratory G
Laboratory Performing Analysis	Laboratory G
Method Blank Result (mg/L)	< 0.010
LCS (Method Blank Spike) Result (mg/L)	9.29908
LCS (Method Blank Spike) Percent Recovery	93
Average (5) Result FCRM <250 µm (mg/L)	1.23686
FCRM <250 µm Matrix Spike Result (mg/L)	11.2752
FCRM <250 µm Matrix Spike Percent Recovery	100
Control Soil NIST SRM 2710a Result (mg/Kg)	**461**
Control Soil NIST SRM 2710a IVBA Extract Acceptance Range - Arsenic (mg/Kg)	(As) 464 – 681 mg/Kg
Control Soil NIST SRM 2710a IVBA Arsenic Nominal Value (mg/Kg)	(As) 573 mg/Kg
Control Soil NIST SRM 2710a Percent Recovery (%)	80

Appendix L
Laboratory Submitted Study Results

LABORATORY G, Table A5- RE-EXTRACTION. IVBA Extraction Batch Results, (FCRM <250 µm): Arsenic (As) LCS Low, Control Soil LOW; all other QC met			
Lab Performing Extraction	Laboratory G	Analyst Performing Extraction	[redacted]
Lab Performing Analysis	Laboratory G	Analyst Performing Analysis	[redacted]
Instrument Type: ICP-MS or ICP-AES	ICP-AES	Instrument Method Detection Limit (MDL) (ug/L)	4
Extraction Date	June 19, 2017	Extraction As Standard Manufacturer and Lot #	Sigma Lot BCBP4276V
Analysis Date(s)	Jun 30, 2017	Analysis As Standard Manufacturer and Lot #	Inorganic Ventures Lot K2-MEB651894
Initial Calibration Verification Standard Source and Lot #	Inorganic Ventures Lot K2-MEB651896	Interference Check Sample Source and Lot #	Ti – Inorganic Ventures Lot J2-TI02102R V – Inorganic Ventures Low J2-V02101 Mn – Inorganic Ventures Lot K2-MN02127 Ni – Inorganic Ventures Lot K2NI02106 Cr – Inorganic Ventures Lot K2-CR03122 Cu – Inorganic Ventures Lot J2-CU03024 Al, Ca, Fe, Mg – Inorganic Ventures Lot K2-MEB643109

Sample Name	Mass of Sample Material (g)[1]	Volume of Extraction Fluid (mL)[2]	Dilution Factor	Instrument As Result for the Analytical Solution (mg/L)	Results for IVBA extractable As in sample material (mg/kg)
Reagent Blank		100	1	-0.0121107	
Method Blank		100	1	-0.0110099	
FCRM <250 µm (Extraction 1)	0.9788	100	100	0.00993815	101.5
FCRM <250 µm (Extraction 2)	0.9963	100	100	0.0108361	108.8
FCRM <250 µm (Extraction 3)	0.9919	100	100	0.0106728	107.6
FCRM <250 µm (Extraction 4)	1.0294	100	100	0.010591	102.9
FCRM <250 µm (Extraction 5)	0.9796	100	100	0.0104285	106.5
Control Soil – NIST SRM 2710A	1.0008	100	100	0.0406362	**406**
LCS (Method Blank Spike)		100	100	0.0839051	
FCRM <250 µm Matrix Spike	1.0496	100	100	0.0957816	

1 – Measure 1.00 +/- 0.05 g to the nearest 0.0001g, enter exact mass in table. Extraction mass should not be reduced. If your lab requires more reference material, please contact Clifton Jones at clifton.jones@cbifederalservices.com.

2 – Add 100 +/- 0.5 mL of 0.4M glycine, pH 1.5, delivered via graduated cylinder or automated dispenser.
Results of IVBA Extractable As in sample material (mg/kg) = [Instrument As Results for Analytical Solution (mg/L) x Dilution Factor x 100 mL extraction fluid x (1 L/1000 mL)]/[mass of sample material (g) x (1 kg/1000 g)].

Appendix L
Laboratory Submitted Study Results

LABORATORY G, Table A6 – RE-EXTRACTION. IVBA Extraction Batch Spiked Blank, Spiked Sample, and Control Soil Results for RM Batch (FCRM <250 µm): Arsenic (As)	
Laboratory Performing Extraction	Laboratory G
Laboratory Performing Analysis	Laboratory G
Method Blank Result (mg/L)	< 0.010
LCS (Method Blank Spike) Result (mg/L)	8.39051
LCS (Method Blank Spike) Percent Recovery	**84**
Average (5) Result FCRM <250 µm (mg/L)	1.049331
FCRM <250 µm Matrix Spike Result (mg/L)	9.57816
FCRM <250 µm Matrix Spike Percent Recovery	85
Control Soil NIST SRM 2710a Result (mg/Kg)	**406**
Control Soil NIST SRM 2710a IVBA Extract Acceptance Range - Arsenic (mg/Kg)	(As) 464 – 681 mg/Kg
Control Soil NIST SRM 2710a IVBA Arsenic Nominal Value (mg/Kg)	(As) 573 mg/Kg
Control Soil NIST SRM 2710a Percent Recovery (%)	71

Appendix L
Laboratory Submitted Study Results

LABORATORY G, Table A7. IVBA Extraction Batch Results, (FCRM <74 µm): Arsenic (As) – All QC Criteria Met			
Lab Performing Extraction	Laboratory G	Analyst Performing Extraction	[redacted]
Lab Performing Analysis	Laboratory G	Analyst Performing Analysis	[redacted]
Instrument Type: ICP-MS or ICP-AES	ICP-AES	Instrument Method Detection Limit (MDL) (ug/L)	4
Extraction Date	May 18, 2017	Extraction As Standard Manufacturer and Lot #	Sigma Lot BCBP4276V
Analysis Date(s)	Jun 14, 2017	Analysis As Standard Manufacturer and Lot #	Inorganic Ventures Lot K2-MEB651894
Initial Calibration Verification Standard Source and Lot #	Inorganic Ventures Lot K2-MEB651896	Interference Check Sample Source and Lot #	Ti – Inorganic Ventures Lot J2-TI02101R V – Inorganic Ventures Low J2-V02101 Mn – Inorganic Ventures Lot K2-MN02127 Ni – Inorganic Ventures Lot K2NI02106 Cr – Inorganic Ventures Lot K2-CR03122 Cu – Inorganic Ventures Lot J2-CU03024 Al, Ca, Fe, Mg – Inorganic Ventures Lot K2-MEB643109

Sample Name	Mass of Sample Material (g)[1]	Volume of Extraction Fluid (mL)[2]	Dilution Factor	Instrument As Result for the Analytical Solution (mg/L)	Results for IVBA extractable As in sample material (mg/kg)
Reagent Blank		100	1.000	0.0148699	
Method Blank		100	1.000	0.00315269	
FCRM <74 µm (Extraction 1)	0.9614	100	10.00	0.137629	143
FCRM <74 µm (Extraction 2)	1.0303	100	10.00	0.122109	119
FCRM <74 µm (Extraction 3)	0.9996	100	10.00	0.120345	120
FCRM <74 µm (Extraction 4)	0.9917	100	10.00	0.118533	120
FCRM <74 µm (Extraction 5)	1.0018	100	10.00	0.119814	120
Control Soil – NIST SRM 2710A	0.9850	100	10.00	0.466426	474
LCS (Method Blank Spike)		100	10.00	0.929908	
FCRM <74 µm Matrix Spike	1.0088	100	10.00	1.12752	

1 – Measure 1.00 +/- 0.05 g to the nearest 0.0001g, enter exact mass in table. Extraction mass should not be reduced. If your lab requires more reference material, please contact Clifton Jones at clifton.jones@cbifederalservices.com.

2 – Add 100 +/- 0.5 mL of 0.4M glycine, pH 1.5, delivered via graduated cylinder or automated dispenser.
Results of IVBA Extractable As in sample material (mg/kg) = [Instrument As Results for Analytical Solution (mg/L) x Dilution Factor x 100 mL extraction fluid x (1 L/1000 mL)]/[mass of sample material (g) x (1 kg/1000 g)].

Appendix L
Laboratory Submitted Study Results

LABORATORY G, Table A8. IVBA Extraction Batch Spiked Blank, Spiked Sample, and Control Soil Results for RM Batch (FCRM <74 µm): Arsenic (As)	
Laboratory Performing Extraction	Laboratory G
Laboratory Performing Analysis	Laboratory G
Method Blank Result (mg/L)	< 0.010
LCS (Method Blank Spike) Result (mg/L)	9.3
LCS (Method Blank Spike) Percent Recovery	93
Average (5) Result FCRM <74 µm (mg/L)	1.24
FCRM <74 µm Matrix Spike Result (mg/L)	11
FCRM <74 µm Matrix Spike Percent Recovery	98
Control Soil NIST SRM 2710a Result (mg/Kg)	474
Control Soil NIST SRM 2710a IVBA Extract Acceptance Range - Arsenic (mg/Kg)	(As) 464 – 681 mg/Kg
Control Soil NIST SRM 2710a IVBA Arsenic Nominal Value (mg/Kg)	(As) 573 mg/Kg
Control Soil NIST SRM 2710a Percent Recovery (%)	83

Appendix L
Laboratory Submitted Study Results

LABORATORY G, Table A9. IVBA Extraction Batch Results, (FCRM <74 µm): Lead (Pb) – All QC Criteria Met						
Lab Performing Extraction	Laboratory G		Analyst Performing Extraction	[redacted]		
Lab Performing Analysis	Laboratory G		Analyst Performing Analysis	[redacted]		
Instrument Type: ICP-MS or ICP-AES	ICP-AES		Instrument Method Detection Limit (MDL) (ug/L)	4		
Extraction Date	May 18, 2017		Extraction Pb Standard Manufacturer and Lot #	Fluka Lot BCBN3916V		
Analysis Date(s)	Jun 14, 2017		Analysis Pb Standard Manufacturer and Lot #	Inorganic Ventures Lot K2-MEB651894		
Initial Calibration Verification Standard Source and Lot #	Inorganic Ventures Lot K2-MEB651896		Interference Check Sample Source and Lot #	Ti – Inorganic Ventures Lot J2-TI02101R V – Inorganic Ventures Low J2-V02101 Mn – Inorganic Ventures Lot K2-MN02127 Ni – Inorganic Ventures Lot K2NI02106 Cr – Inorganic Ventures Lot K2-CR03122 Cu – Inorganic Ventures Lot J2-CU03024 Al, Ca, Fe, Mg – Inorganic Ventures Lot K2-MEB643109		
Sample Name	Mass of Sample Material (g)[1]	Volume of Extraction Fluid (mL)[2]	Dilution Factor	Instrument Pb Result for the Analytical Solution (mg/L)	Results for IVBA extractable Pb in sample material (mg/kg)	
Reagent Blank		100	1.000	0.0172521		
Method Blank		100	1.000	0.0127915		
FCRM <74 µm (Extraction 1)	0.9614	100	10.00	5.48000	5700	
FCRM <74 µm (Extraction 2)	1.0303	100	10.00	5.00129	4854	
FCRM <74 µm (Extraction 3)	0.9996	100	10.00	4.97907	4981	
FCRM <74 µm (Extraction 4)	0.9917	100	10.00	5.08525	5128	
FCRM <74 µm (Extraction 5)	1.0018	100	10.00	5.05622	5047	
Control Soil – NIST SRM 2710A	0.9850	100	10.00	3.43676	3489	
LCS (Method Blank Spike)		100	10.00	1.03964		
FCRM <74 µm Matrix Spike	1.0088	100	10.00	6.02668		

1 – Measure 1.00 +/- 0.05 g to the nearest 0.0001g, enter exact mass in table. Extraction mass should not be reduced. If your lab requires more reference material, please contact Clifton Jones at clifton.jones@cbifederalservices.com.

2 – Add 100 +/- 0.5 mL of 0.4M glycine, pH 1.5, delivered via graduated cylinder or automated dispenser.
Results of IVBA Extractable Pb in sample material (mg/kg) = [Instrument Pb Results for Analytical Solution (mg/L) x Dilution Factor x 100 mL extraction fluid x (1 L/1000 mL)]/[mass of sample material (g) x (1 kg/1000 g)].

Appendix L
Laboratory Submitted Study Results

LABORATORY G, Table A10. IVBA Extraction Batch Spiked Blank, Spiked Sample, and Control Soil Results for RM Batch (FCRM <74 µm): Lead (Pb)	
Laboratory Performing Extraction	Laboratory G
Laboratory Performing Analysis	Laboratory G
Method Blank Result (mg/L)	0.013
LCS (Method Blank Spike) Result (mg/L)	10
LCS (Method Blank Spike) Percent Recovery	100
Average (5) Result FCRM <74 µm (mg/L)	51.2
FCRM <74 µm Matrix Spike Result (mg/L)	60
FCRM <74 µm Matrix Spike Percent Recovery	88
Control Soil NIST SRM 2710a Result (mg/Kg)	3489
Control Soil NIST SRM 2710a IVBA Extract Acceptance Range – Lead (mg/Kg)	(Pb) 3096 – 3785 mg/Kg
Control Soil NIST SRM 2710a IVBA Lead Nominal Value (mg/Kg)	(Pb) 3440 mg/Kg
Control Soil NIST SRM 2710a Percent Recovery (%)	101

Appendix L
Laboratory Submitted Study Results

LABORATORY G, Table B3. Method 3050B or 3051A Digestion Batch Results, (FCRM <250 µm): Arsenic (As) Soil weighed to 0.01; MSS failed (spike too low)			
Lab Performing Digestion	Laboratory G	Analyst Performing Digestion	[redacted]
Lab Performing Analysis	Laboratory G	Analyst Performing Analysis	[redacted]
Instrument Type: ICP-MS or ICP-AES	ICP-AES	Instrument Method Detection Limit (MDL) (ug/L)	4.000
Digestion Date	Jun 9, 2017	Digestion As Standard Manufacturer and Lot #	High Purity Standards Lot # 1623617
Analysis Date(s)	Jun 14, 2017	Analysis As Standard Manufacturer and Lot #	Inorganic Ventures Lot K2-MEB651894
Initial Calibration Verification Standard Source and Lot #	Inorganic Ventures Lot K2-MEB651896	Interference Check Sample Source and Lot #	Ti – Inorganic Ventures Lot J2-TI02101R V – Inorganic Ventures Low J2-V02101 Mn – Inorganic Ventures Lot K2-MN02127 Ni – Inorganic Ventures Lot K2NI02106 Cr – Inorganic Ventures Lot K2-CR03122 Cu – Inorganic Ventures Lot J2-CU03024 Al, Ca, Fe, Mg – Inorganic Ventures Lot K2-MEB643109

Sample Name	Mass of Sample Material (g)[1]	Volume of Digestion Fluid (mL)	Dilution Factor	Instrument As Result for the Analytical Solution (mg/L)	Results from digested Arsenic in sample material (mg/kg)[2]
Method Blank	0.53	50	1	-0.00172662	-0.16289
FCRM <250 µm (Digestion 1)	0.50	50	10	0.742641	743
FCRM <250 µm (Digestion 2)	0.51	50	10	0.750642	736
FCRM <250 µm (Digestion 3)	0.49	50	10	0.73466	750
FCRM <250 µm (Digestion 4)	0.50	50	10	0.710426	710
FCRM <250 µm (Digestion 5)	0.53	50	10	0.755549	713
Control Soil – NIST SRM 2710a	0.51	50	100	0.147934	1450
LCS (Method Blank Spike)	0.50	50	1	0.494464	49.45
FCRM <250 µm Matrix Spike	0.52	50	10	0.805814	774.8

1 – Measure 1.00 +/- 0.05 g to the nearest 0.0001g, enter exact mass in table, or if one-half reduced volume digestion is performed, measure 0.50 +/- 0.05 g to the nearest 0.0001g. If your lab requires more reference material, please contact Clifton Jones at clifton.jones@cbifederalservices.com.

2 – Arsenic in sample material (mg/kg) = [Instrument As Results for Analytical Solution (mg/L) x Dilution Factor x 100 mL Digest Final Volume x (1 L/1000 mL)]/[mass of sample material (g) x (1 kg/1000 g)].

Appendix L
Laboratory Submitted Study Results

LABORATORY G, Table B4. Method 3050B or 3051A Digestion Spiked Blank, and Spiked Sample Results for (FCRM <250 µm): Arsenic (As)	
Laboratory Performing Digestion	Laboratory G
Laboratory Performing Analysis	Laboratory G
Blank Spike Result (mg/L)	0.494464 (spike = 0.5000 mg/L)
Blank Spike Percent Recovery	99
Average (5) Result RM (mg/L)	7.387836 (spike – 0.5000 mg/L)
RM Matrix Spike Result (mg/L)	8.05814
RM Matrix Spike Percent Recovery	134
Control Soil NIST SRM 2710a Result (mg/Kg)	1450
Control Soil NIST SRM 2710a Digestion Acceptance Range - Arsenic (mg/Kg)	(As) 1260 -1540 mg/kg
Control Soil NIST SRM 2710a Arsenic Nominal Value (mg/Kg)	(As) 1400 mg/kg
Control Soil NIST SRM 2710a Percent Recovery (%)	104

Appendix L
Laboratory Submitted Study Results

LABORATORY G, Table B3 RUN2. Method 3050B or 3051A Digestion Batch Results, (FCRM <250 µm): Arsenic (As) Soil weighed to 0.0001; MSS failed (spike too low)			
Lab Performing Digestion	Laboratory G	Analyst Performing Digestion	[redacted]
Lab Performing Analysis	Laboratory G	Analyst Performing Analysis	[redacted]
Instrument Type: ICP-MS or ICP-AES	ICP-AES	Instrument Method Detection Limit (MDL) (ug/L)	4.000
Digestion Date	Jun 19, 2017	Digestion As Standard Manufacturer and Lot #	High Purity Standards Lot # 1623617
Analysis Date(s)	Jun 20, 2017	Analysis As Standard Manufacturer and Lot #	Inorganic Ventures Lot K2-MEB651894
Initial Calibration Verification Standard Source and Lot #	Inorganic Ventures Lot K2-MEB651896	Interference Check Sample Source and Lot #	Ti – Inorganic Ventures Lot J2-TI02101R V – Inorganic Ventures Low J2-V02101 Mn – Inorganic Ventures Lot K2-MN02127 Ni – Inorganic Ventures Lot K2NI02106 Cr – Inorganic Ventures Lot K2-CR03122 Cu – Inorganic Ventures Lot J2-CU03024 Al, Ca, Fe, Mg – Inorganic Ventures Lot K2-MEB643109

Sample Name	Mass of Sample Material (g)[1]	Volume of Digestion Fluid (mL)	Dilution Factor	Instrument As Result for the Analytical Solution (mg/L)	Results from digested Arsenic in sample material (mg/kg)[2]
Method Blank	0.4979	50	1	0.00076033	0.076354
FCRM <250 µm (Digestion 1)	0.4997	50	10	0.717778	718
FCRM <250 µm (Digestion 2)	0.4946	50	10	0.724214	732
FCRM <250 µm (Digestion 3)	0.5036	50	10	0.784296	779
FCRM <250 µm (Digestion 4)	0.5183	50	10	0.77034	743
FCRM <250 µm (Digestion 5)	0.4952	50	10	0.754923	762
Control Soil – NIST SRM 2710a	0.5052	50	100	0.153974	1524
LCS (Method Blank Spike)	0.5186	50	1	0.20138	19.42
FCRM <250 µm Matrix Spike	0.5038	50	10	0.753807	748

1 – Measure 1.00 +/- 0.05 g to the nearest 0.0001g, enter exact mass in table, or if one-half reduced volume digestion is performed, measure 0.50 +/- 0.05 g to the nearest 0.0001g. If your lab requires more reference material, please contact Clifton Jones at clifton.jones@cbifederalservices.com.

2 – Arsenic in sample material (mg/kg) = [Instrument As Results for Analytical Solution (mg/L) x Dilution Factor x 100 mL Digest Final Volume x (1 L/1000 mL)]/[mass of sample material (g) x (1 kg/1000 g)].

Appendix L
Laboratory Submitted Study Results

LABORATORY G, Table B4 RUN #2. Method 3050B or 3051A Digestion Spiked Blank, and Spiked Sample Results for (FCRM <250 µm): Arsenic (As)	
Laboratory Performing Digestion	Laboratory G
Laboratory Performing Analysis	Laboratory G
Blank Spike Result (mg/L)	0.20138 (spike = 0.2000 mg/L)
Blank Spike Percent Recovery	101
Average (5) Result RM (mg/L)	7.503102 (spike – 0.2000 mg/L)
RM Matrix Spike Result (mg/L)	7.53807
RM Matrix Spike Percent Recovery	175
Control Soil NIST SRM 2710a Result (mg/Kg)	1524
Control Soil NIST SRM 2710a Digestion Acceptance Range - Arsenic (mg/Kg)	(As) 1260 -1540 mg/kg
Control Soil NIST SRM 2710a Arsenic Nominal Value (mg/Kg)	(As) 1400 mg/kg
Control Soil NIST SRM 2710a Percent Recovery (%)	109

Appendix L
Laboratory Submitted Study Results

LABORATORY G, Table B5. Method 3050B or 3051A Digestion Batch Results, (FCRM <74 µm): Arsenic (As) Soil weighed to 0.01; MSS failed (spike too low)			
Lab Performing Digestion	Laboratory G	Analyst Performing Digestion	[redacted]
Lab Performing Analysis	Laboratory G	Analyst Performing Analysis	[redacted]
Instrument Type: ICP-MS or ICP-AES	ICP-AES	Instrument Method Detection Limit (MDL) (ug/L)	4.000
Digestion Date	Jun 9, 2017	Digestion As Standard Manufacturer and Lot #	High Purity Standards Lot # 1623617
Analysis Date(s)	Jun 14, 2017	Analysis As Standard Manufacturer and Lot #	Inorganic Ventures Lot K2-MEB651894
Initial Calibration Verification Standard Source and Lot #	Inorganic Ventures Lot K2-MEB651896	Interference Check Sample Source and Lot #	Ti – Inorganic Ventures Lot J2-TI02101R V – Inorganic Ventures Low J2-V02101 Mn – Inorganic Ventures Lot K2-MN02127 Ni – Inorganic Ventures Lot K2NI02106 Cr – Inorganic Ventures Lot K2-CR03122 Cu – Inorganic Ventures Lot J2-CU03024 Al, Ca, Fe, Mg – Inorganic Ventures Lot K2-MEB643109

Sample Name	Mass of Sample Material (g)[1]	Volume of Digestion Fluid (mL)	Dilution Factor	Instrument As Result for the Analytical Solution (mg/L)	Results from digested Arsenic in sample material (mg/kg)[2]
Method Blank	0.52	50	1.000	-0.00011822	-0.00023
FCRM <74 µm (Digestion 1)	0.50	50	100.0	0.0680357	680
FCRM <74 µm (Digestion 2)	0.51	50	100.0	0.0665639	653
FCRM <74 µm (Digestion 3)	0.50	50	100.0	0.0683772	684
FCRM <74 µm (Digestion 4)	0.52	50	100.0	0.0708715	681
FCRM <74 µm (Digestion 5)	0.50	50	100.0	0.0702944	703
Control Soil – NIST SRM 2710a	0.53	50	100.0	0.143921	1358
LCS (Method Blank Spike)	0.53	50	1.000	0.468708	44.22
FCRM <74 µm Matrix Spike	0.51	50	10.00	0.759416	744.5

1 – Measure 1.00 +/- 0.05 g to the nearest 0.0001g, enter exact mass in table, or if one-half reduced volume digestion is performed, measure 0.50 +/- 0.05 g to the nearest 0.0001g. If your lab requires more reference material, please contact Clifton Jones at clifton.jones@cbifederalservices.com.

2 – Arsenic in sample material (mg/kg) = [Instrument As Results for Analytical Solution (mg/L) x Dilution Factor x 100 mL Digest Final Volume x (1 L/1000 mL)]/[mass of sample material (g) x (1 kg/1000 g)].

Appendix L
Laboratory Submitted Study Results

LABORATORY G, Table B6. Method 3050B or 3051A Digestion Spiked Blank, and Spiked Sample Results for (FCRM <74 µm): Arsenic (As)	
Laboratory Performing Digestion	Laboratory G
Laboratory Performing Analysis	Laboratory G
Blank Spike Result (mg/L)	0.468708 (spike = 0.5000)
Blank Spike Percent Recovery	94
Average (5) Result RM (mg/L)	6.882854 (spike = 0.5000)
RM Matrix Spike Result (mg/L)	7.59416
RM Matrix Spike Percent Recovery	142
Control Soil NIST SRM 2710a Result (mg/Kg)	1358
Control Soil NIST SRM 2710a Digestion Acceptance Range - Arsenic (mg/Kg)	(As) 1260 -1540 mg/kg
Control Soil NIST SRM 2710a Arsenic Nominal Value (mg/Kg)	(As) 1400 mg/kg
Control Soil NIST SRM 2710a Percent Recovery (%)	97

Appendix L
Laboratory Submitted Study Results

LABORATORY G, Table B5 – RUN #2. Method 3050B or 3051A Digestion Batch Results, (FCRM <74 µm): Arsenic (As) Soil weighed to 0.0001; MSS failed (spike too low)			
Lab Performing Digestion	Laboratory G	Analyst Performing Digestion	SL
Lab Performing Analysis	Laboratory G	Analyst Performing Analysis	TLO
Instrument Type: ICP-MS or ICP-AES	ICP-AES	Instrument Method Detection Limit (MDL) (ug/L)	4.000
Digestion Date	Jun 19, 2017	Digestion As Standard Manufacturer and Lot #	High Purity Standards Lot # 1623617
Analysis Date(s)	Jun 20, 2017	Analysis As Standard Manufacturer and Lot #	Inorganic Ventures Lot K2-MEB651894
Initial Calibration Verification Standard Source and Lot #	Inorganic Ventures Lot K2-MEB651896	Interference Check Sample Source and Lot #	Ti – Inorganic Ventures Lot J2-TI02101R V – Inorganic Ventures Low J2-V02101 Mn – Inorganic Ventures Lot K2-MN02127 Ni – Inorganic Ventures Lot K2NI02106 Cr – Inorganic Ventures Lot K2-CR03122 Cu – Inorganic Ventures Lot J2-CU03024 Al, Ca, Fe, Mg – Inorganic Ventures Lot K2-MEB643109

Sample Name	Mass of Sample Material (g)[1]	Volume of Digestion Fluid (mL)	Dilution Factor	Instrument As Result for the Analytical Solution (mg/L)	Results from digested Arsenic in sample material (mg/kg)[2]
Method Blank	0.5109	50	1	0.00142069	0.13904
FCRM <74 µm (Digestion 1)	0.4992	50	100	0.07089	710
FCRM <74 µm (Digestion 2)	0.4906	50	100	0.0678527	692
FCRM <74 µm (Digestion 3)	0.5136	50	100	0.0731522	712
FCRM <74 µm (Digestion 4)	0.5018	50	100	0.0725669	723
FCRM <74 µm (Digestion 5)	0.5023	50	100	0.0712764	710
Control Soil – NIST SRM 2710a	0.4928	50	100	0.148717	1509
LCS (Method Blank Spike)	0.5039	50	1	0.197089	19.56
FCRM <74 µm Matrix Spike	0.5042	50	10	0.75441	748

1 – Measure 1.00 +/- 0.05 g to the nearest 0.0001g, enter exact mass in table, or if one-half reduced volume digestion is performed, measure 0.50 +/- 0.05 g to the nearest 0.0001g. If your lab requires more reference material, please contact Clifton Jones at clifton.jones@cbifederalservices.com.

2 – Arsenic in sample material (mg/kg) = [Instrument As Results for Analytical Solution (mg/L) x Dilution Factor x 100 mL Digest Final Volume x (1 L/1000 mL)]/[mass of sample material (g) x (1 kg/1000 g)].

Appendix L
Laboratory Submitted Study Results

LABORATORY G, Table B6-RUN #2. Method 3050B or 3051A Digestion Spiked Blank, and Spiked Sample Results for (FCRM <74 µm): Arsenic (As)	
Laboratory Performing Digestion	Laboratory G
Laboratory Performing Analysis	Laboratory G
Blank Spike Result (mg/L)	0.197089 (spike = 0.2000)
Blank Spike Percent Recovery	99
Average (5) Result RM (mg/L)	7.114764 (spike = 0.2000)
RM Matrix Spike Result (mg/L)	7.5441
RM Matrix Spike Percent Recovery	215
Control Soil NIST SRM 2710a Result (mg/Kg)	1509
Control Soil NIST SRM 2710a Digestion Acceptance Range - Arsenic (mg/Kg)	(As) 1260 -1540 mg/kg
Control Soil NIST SRM 2710a Arsenic Nominal Value (mg/Kg)	(As) 1400 mg/kg
Control Soil NIST SRM 2710a Percent Recovery (%)	108

Appendix L
Laboratory Submitted Study Results

LABORATORY G, Table B7. Method 3050B or 3051A Digestion Batch Results, (FCRM <74 µm): Lead (Pb) Soil weighed to 0.01; MSS failed (spike too low)			
Lab Performing Digestion	Laboratory G	Analyst Performing Digestion	[redacted]
Lab Performing Analysis	Laboratory G	Analyst Performing Analysis	[redacted]
Instrument Type: ICP-MS or ICP-AES	ICP-AES	Instrument Method Detection Limit (MDL) (ug/L)	4.000
Digestion Date	Jun 9, 2017	Digestion Pb Standard Manufacturer and Lot #	High Purity Standards Lot # 1623617
Analysis Date(s)	Jun 14, 2017	Analysis Pb Standard Manufacturer and Lot #	Inorganic Ventures Lot K2-MEB651894
Initial Calibration Verification Standard Source and Lot #	Inorganic Ventures Lot K2-MEB651896	Interference Check Sample Source and Lot #	Ti – Inorganic Ventures Lot J2-TI02101R V – Inorganic Ventures Low J2-V02101 Mn – Inorganic Ventures Lot K2-MN02127 Ni – Inorganic Ventures Lot K2NI02106 Cr – Inorganic Ventures Lot K2-CR03122 Cu – Inorganic Ventures Lot J2-CU03024 Al, Ca, Fe, Mg – Inorganic Ventures Lot K2-MEB643109

Sample Name	Mass of Sample Material (g)[1]	Volume of Digestion Fluid (mL)	Dilution Factor	Instrument Pb Result for the Analytical Solution (mg/L)	Results from digested Lead in sample material (mg/kg)[2]
Method Blank	0.52	50	1.000	-0.00397195	< 1.0
FCRM <74 µm (Digestion 1)	0.50	50	100.0	0.618694	6200
FCRM <74 µm (Digestion 2)	0.51	50	100.0	0.605187	5900
FCRM <74 µm (Digestion 3)	0.50	50	100.0	0.641296	6400
FCRM <74 µm (Digestion 4)	0.52	50	100.0	0.640087	6200
FCRM <74 µm (Digestion 5)	0.50	50	100.0	0.652806	6500
Control Soil – NIST SRM 2710a	0.53	50	100.0	0.541843	5100
LCS (Method Blank Spike)	0.53	50	1.000	0.486275	45.88
FCRM <74 µm Matrix Spike	0.51	50	10.00	6.60252	6473

1 – Measure 1.00 +/- 0.05 g to the nearest 0.0001g, enter exact mass in table, or if one-half reduced volume digestion is performed, measure 0.50 +/- 0.05 g to the nearest 0.0001g. If your lab requires more reference material, please contact Clifton Jones at clifton.jones@cbifederalservices.com.

2 – Lead in sample material (mg/kg) = [Instrument Pb Results for Analytical Solution (mg/L) x Dilution Factor x 100 mL Digest Final Volume x (1 L/1000 mL)]/[mass of sample material (g) x (1 kg/1000 g)].

Appendix L
Laboratory Submitted Study Results

LABORATORY G, Table B8. Method 3050B or 3051A Digestion Spiked Blank, and Spiked Sample Results for (FCRM <74 µm): Lead (Pb)	
Laboratory Performing Digestion	Laboratory G
Laboratory Performing Analysis	Laboratory G
Blank Spike Result (mg/L)	0.486275 (spike = 0.5 mg/L)
Blank Spike Percent Recovery	97
Average (5) Result RM (mg/L)	63.1614 (spike = 0.5 mg/L)
RM Matrix Spike Result (mg/L)	66.0252
RM Matrix Spike Percent Recovery	573
Control Soil NIST SRM 2710a Result (mg/Kg)	5112
Control Soil NIST SRM 2710a Digestion Acceptance Range - Lead (mg/Kg)	(Pb) 4590 -5610 mg/kg
Control Soil NIST SRM 2710a Lead Nominal Value (mg/Kg)	(Pb) 5100 mg/kg
Control Soil NIST SRM 2710a Percent Recovery (%)	100

Appendix L
Laboratory Submitted Study Results

LABORATORY G, Table B7 RUN#2. Method 3050B or 3051A Digestion Batch Results, (FCRM <74 µm): Lead (Pb) Soil weighed to 0.0001; MSS failed (spike too low)			
Lab Performing Digestion	Laboratory G	Analyst Performing Digestion	[redacted]
Lab Performing Analysis	Laboratory G	Analyst Performing Analysis	[redacted]
Instrument Type: ICP-MS or ICP-AES	ICP-AES	Instrument Method Detection Limit (MDL) (ug/L)	4.000
Digestion Date	Jun 19, 2017	Digestion Pb Standard Manufacturer and Lot #	High Purity Standards Lot # 1623617
Analysis Date(s)	Jun 20, 2017	Analysis Pb Standard Manufacturer and Lot #	Inorganic Ventures Lot K2-MEB651894
Initial Calibration Verification Standard Source and Lot #	Inorganic Ventures Lot K2-MEB651896	Interference Check Sample Source and Lot #	Ti – Inorganic Ventures Lot J2-TI02101R V – Inorganic Ventures Low J2-V02101 Mn – Inorganic Ventures Lot K2-MN02127 Ni – Inorganic Ventures Lot K2NI02106 Cr – Inorganic Ventures Lot K2-CR03122 Cu – Inorganic Ventures Lot J2-CU03024 Al, Ca, Fe, Mg – Inorganic Ventures Lot K2-MEB643109

Sample Name	Mass of Sample Material (g)[1]	Volume of Digestion Fluid (mL)	Dilution Factor	Instrument Pb Result for the Analytical Solution (mg/L)	Results from digested Lead in sample material (mg/kg)[2]
Method Blank	0.5109	50	1	0.00299008	0.29263
FCRM <74 µm (Digestion 1)	0.4992	50	100	0.642881	6439
FCRM <74 µm (Digestion 2)	0.4906	50	100	0.613562	6253
FCRM <74 µm (Digestion 3)	0.5136	50	100	0.668025	6503
FCRM <74 µm (Digestion 4)	0.5018	50	100	0.634436	6322
FCRM <74 µm (Digestion 5)	0.5023	50	100	0.626786	6239
Control Soil – NIST SRM 2710a	0.4928	50	100	0.500149	5075
LCS (Method Blank Spike)	0.5039	50	1	0.201697	20.01
FCRM <74 µm Matrix Spike	0.5042	50	10	6.52237	6468

1 – Measure 1.00 +/- 0.05 g to the nearest 0.0001g, enter exact mass in table, or if one-half reduced volume digestion is performed, measure 0.50 +/- 0.05 g to the nearest 0.0001g. If your lab requires more reference material, please contact Clifton Jones at clifton.jones@cbifederalservices.com.

2 – Lead in sample material (mg/kg) = [Instrument Pb Results for Analytical Solution (mg/L) x Dilution Factor x 100 mL Digest Final Volume x (1 L/1000 mL)]/[mass of sample material (g) x (1 kg/1000 g)].

Appendix L
Laboratory Submitted Study Results

LABORATORY G, Table B8-RUN #2. Method 3050B or 3051A Digestion Spiked Blank, and Spiked Sample Results for (FCRM <74 µm): Lead (Pb)	
Laboratory Performing Digestion	Laboratory G
Laboratory Performing Analysis	Laboratory G
Blank Spike Result (mg/L)	0.201697 (spike = 0.2 mg/L)
Blank Spike Percent Recovery	101
Average (5) Result RM (mg/L)	61.43065 (spike = 0.2 mg/L)
RM Matrix Spike Result (mg/L)	65.2237
RM Matrix Spike Percent Recovery	1900
Control Soil NIST SRM 2710a Result (mg/Kg)	5075
Control Soil NIST SRM 2710a Digestion Acceptance Range - Lead (mg/Kg)	(Pb) 4590 -5610 mg/kg
Control Soil NIST SRM 2710a Lead Nominal Value (mg/Kg)	(Pb) 5100 mg/kg
Control Soil NIST SRM 2710a Percent Recovery (%)	100