FINAL REPORT

TASK ORDER 1003
DISTRIBUTION AND MAINTENANCE OF THE SUPERFUND PERFORMANCE EVALUATION SAMPLE (PES/RM) INVENTORY

LEAD IVBA ROUND ROBIN ANALYSIS OF NIST SRM 2710A AND SRM 2711A

Prepared by:

QATS Analytical Group

Quality Assurance Technical Support Laboratory
Shaw Environmental, Inc.
2700 Chandler Ave.
Las Vegas, Nevada 89120

April 19, 2011

Contract Number: EP-W-10-033

Task: 07
NIST Bioaccessibility Study

Prepared for Phil Cocuzza

Analytical Services Branch
U.S. Environmental Protection Agency
Washington, D.C. 20460

OFFICE OF SUPERFUND REMEDIATION AND TECHNICAL INNOVATION U.S.
ENVIRONMENTAL PROTECTION AGENCY
WASHINGTON, D.C. 20460

TABLE OF CONTENTS

PAGE

LIST OF APPENDICES

FINAL REPORT
TASK ORDER 1003
DISTRIBUTION AND MAINTENANCE OF THE SUPERFUND PERFORMANCE EVALUATION SAMPLES (PES/RM) INVENTORY

TASK 7 – NIST BIOACCESSABILITY STUDY
LEAD IVBA ROUND ROBIN ANALYSIS OF NIST SRM 2710A AND SRM 2711A

INTRODUCTION

This report provides the data and statistical analysis of the lead results from the In-Vitro Bioaccessibility (IVBA) Round Robin Study conducted by the USEPA to provided data for the two (2) replacement National Institute of Standards and Technology (NIST) Standard Reference Materials (SRMs) 2710a “Montana Soil” and 2711a “Montana Soil II”. These two (2) SRMs became available for purchase in April 2009. The previous lots of these materials, which have the same SRM number without an “a” suffix, became unavailable for purchase from NIST in late 2008. Since the previous lots of these “Montana Soils”, SRM 2710 and 2711, were used as Control Soils for the lead IVBA extraction and analysis in the EPA IVBA SOP 9200.1-86, and other methods used by the bioaccessibility community, it became necessary to develop new lead IVBA means and acceptance ranges for these recently released replacement SRMs (USEPA, 2008). The Round Robin sample analyses were conducted in late 2010 using seven (7) participating laboratories. Each laboratory was requested to analyze each of the SRMs in five (5) replicate analyses, along with the EPA IVBA SOP-required Quality Control (QC) samples, including blank, matrix spike, and Control Soil.

The EPA Technical Review Workgroup for Metals and Asbestos (TRW) Bioavailability Committee initiated the task of verification of the lead IVBA values for these two (2) SRMs. The (TRW) Bioavailability Committee contacted the EPA Analytical Services Branch (ASB) which oversees the EPA Quality Assurance Technical Support (QATS) contract operated by Shaw Environmental, Inc., for support in the coordination of the NIST SRM Round Robin Study. The QATS Laboratory support included the SRM bottling and shipping, the development of the Round Robin instructions in the form of a Statement of Work, laboratory coordination, statistical analysis of results, and report preparation.

BACKGROUND

The utilization of IVBA methods as an estimator of the bioavailability of lead in soil matrices has been studied and generally adopted by the bioavailability community in various forms. 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. Prior to the development of IVBA methods, the method of choice employed juvenile swine assays. A comparison of the in-vivo and in-vitro methods, as well as the initial setting of the IVBA values for the former NIST SRM lots 2710 and 2711, are presented in EPA OSWER 9285.7-77 (USEPA, 2007).

Briefly, the IVBA method is performed by first retrieving soil to be assessed for bioaccessibility. The soil is processed by drying at less than 40ºC, and passing the dried material through a sieve to obtain the soil particles that are less than 250µm. One (1) gram of the soil material is placed in a plastic bottle, and 100 mL of 0.4 M glycine, at a pH of 1.5, is added. The sample bottle(s), and associated quality control sample bottles, are then placed on a rotary extractor (30 ± 2 RPM) for one (1) hour while being heated at a constant temperature of 37± 2ºC.

The heating of the bottles and rotary extraction apparatus can be 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). The bottles are removed from the extraction apparatus and the supernatant is removed using an in-line filter and a 20 mL syringe. The filtered supernatant is then analyzed for lead (or other analytes) by inductively coupled plasma - atomic emission spectroscopy (ICP-AES) or by ICP-mass spectroscopy (ICP-MS). The IVBA method also requires the digestion of a separate one (1) gram aliquot of the soil material using EPA Method 3050, which is a strong leach acid digestion using nitric acid and hydrogen peroxide. The resulting digest is analyzed for lead (or other analytes) by either ICP-AES or ICP-MS. The IVBA value for the soil sample is the ratio of the extracted amount divided by the digested amount times 100. The full IVBA extraction procedure used in this Round Robin Study can be found in EPA IVBA SOP 9200.1-86. This SOP version specifies the use of a water bath as a temperature controlling medium for the heating of the sample bottles, although for this study, the air heating of the sample bottles was allowed.

SUMMARY

This Round Robin Study consisted of seven (7) laboratories, each reporting five (5) replicate analysis results for each SRM 2710a and 2711a, resulting in total of 35 results for each SRM. The IVBA SOP 9200-1-86 was the method used for the Round Robin Study. The statistical analysis of the Round Robin sample results provided a mean and relative standard deviation (RSD) for the each of the two (2) NIST SRMs that are consistent with previous studies. No outlying sample results were indentified using the Grubb's test either within each laboratory (n=5), or collectively for the n=35 data set for the individual SRMs, based on conventional statistical analysis. The associated Quality Control (QC) sample results provided by the laboratories for the reagent blank, bottle blank, spiked blank, matrix spike, and Control Soil were all within the acceptance criteria presented in the EPA IVBA SOP 9200.1-86. A statistical comparison (t-test) was made between the SRM data derived from IVBA extractions that were performed by laboratories employing air (incubator type) as the temperature controlling (37± 2ºC) medium, versus water (aquarium type). The comparison showed that, for this set of results, there was no statistical difference between the two (2) techniques of controlling the temperature of sample bottles during the extraction.

TECHNICAL APPROACH

The subtasks involved in this Round Robin Study included:

- Contacting candidate laboratories with previous IVBA experience;
- Requesting laboratories to complete an Initial Demonstration of Proficiency (IDP) form;
- 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, including IVBA data reporting forms;
- Shipment of the IVBA samples and associated QC samples; and
- Statistical analysis of the Round Robin Study results and report preparation.

Contacting Laboratories, IDP form, and Participating Laboratory Selection

The first subtask of this Round Robin Study included contacting laboratories with previous IVBA experience, and soliciting their participation in the study. The laboratories were asked to complete an Initial Demonstration of Proficiency (IDP) form, developed by QATS personnel and the Bioavailability Committee, which included several questions relating to the level of experience their facility has 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. After careful review of the IDP forms by the TRW Bioavailability Committee, seven (7) laboratories were selected that were deemed suitable for participation in the NIST SRM Round Robin Study. The seven (7) laboratories/institutions selected to participate in the IVBA NIST Round Robin Study are presented in Table 1. The IDP forms provided by the laboratories are presented in Appendix A in redacted form, with an alphanumeric letter used as an identifier, in an order inconsistent with the order present in Table 1. The IDP forms, without redaction, are available from USEPA Office of Superfund Remediation and Technology Innovation (OSRTI) Committee Chair Mr. James Konz.

Table 1. Laboratories Selected to Participate in the Round Robin Study

	Laboratory	Address	Contact Name and e-mail Address
1	ACZ Laboratories	2773 Downhill Drive Steamboat Springs, CO; 80487	Mr. Tim VanWyngarden timv@acz.com Ms. Sue Webber; suew@acz.com
2	EPA National Exposure Research Laboratory (NERL) Research Triangle Park (RTP)	US EPA 109 T.W. Alexander Dr. Research Triangle Park, NC. 27709	Dr. Karen Bradham Bradham.Karen@epamail.epa.gov
3	Ohio State University	410 C Kottman Hall School of Environment and Natural Resources 2021 Coffey Rd. The Ohio State University Columbus, OH 43210-1043	Dr. Nicholas Basta basta.4@osu.edu
4	PRIMA Environmental	5070 Robert J Mathews Pkwy, Ste 300 El Dorado Hills, CA 95762	Dr. Cindy Schreier cschreier@primaenvironmental.com
5	Royal Military College of Canada	Environmental Sciences Group Royal Military College PO Box 17000, Station Forces Kingston, Ontario CANADA K7K 7B4	Dr. Reimer reimer-k@rmc.ca Dr. Iris Koch koch-i@rmc.ca Ms. Tamara Van Dyck tamara.vandyck@rmc.ca
6	Royal Roads University (Canada	Royal Roads University 2005 Sooke Road Victoria, BC, Canada;V9B 5Y2	Dr. Matt Dodd Matt.Dodd@RoyalRoads.ca
7	University of Colorado	2200 Colorado Av. Benson Earth Science University of Colorado Boulder, CO 80309	Dr. John Drexler Drexlerj@Colorado.EDU

Round Robin Study Design

NIST SRM Sample Material and Number of Replicates

The NIST SRM Materials used in this Round Robin Study were provided by Dr. Elizabeth Mackey of NIST from a previous study conducted at the QATS Laboratory in 2009. The SRM materials were provided in 50 gram glass bottles, and sufficient SRM material was combined and mixed before aliquots were bottled for Round Robin Sample analysis. Since the NIST SRM material was of limited quantity, the participating laboratories were instructed not to dry the material at the temperature of less than 40ºC or sieve the material through a 250µm screen, as specified in Section 3.0 of the EPA IVBA SOP 9200.1-86. The particle size reduction by sieving the SRM material through a 250µm screen would be unnecessary as the SRM material was processed by sieving through a 74µm screen during preparation at NIST. From the NIST SRM 2710a and 2711a certificate of analysis, the moisture content of the materials is approximately 2%. The 2% moisture content for the NIST SRM 2711a was determined by oven heating for two (2) hours at 110ºC, which may not be comparable to the EPA IVBA SOP 9200.1-86 procedure of drying at a temperature of less than 40ºC. The lower temperature of heating should, of course, result in more moisture retained by the sample. It is apparent from the IDP forms provided by the laboratories in this Round Robin Study that it is common practice in the Bioavailability community to use these relatively expensive SRMs “as is” without correction for the rather low moisture content, without sieving of the material. Although the SOP indicates the use of NIST SRM 2711 as a Control Soil, it does not specify that the SRM material must be processed as a sample by drying and sieving.

The certificate of analysis for the NIST SRMs 2710, 2711, 2710a, and 2711a are presented in Appendix B. Table 2 provides the particle size and moisture content for these SRMs derived from the NIST certificates of analysis.

Table 2. Round Robin Study SRMs Lead Concentration, Particle Size and Moisture Content

NIST SRM	Lead total (mg/Kg)	Lead Leachable EPA Method 3050 (mg/Kg)	Particle Size	Moisture Content
2710	5532	5100	<74 µm	1.7 - 2.3%
2711	1162	1100	<74 µm	1.5 - 2.2%
2710a	5520	5100	<74 µm	~2%
2711a	1400	1300	<74 µm	~2%

The TRW Committee concluded that five (5) replicate analyses of each SRM would be conducted by each laboratory participating in the Round Robin Study, in order to ensure that a sufficient number of results were available for setting a statistically sound IVBA mean and acceptance range to the new lots of NIST SRMs 2710a and 2711a.

QC Samples

To provide continuity for the new NIST SRM IVBA results, the previous lot of NIST SRM 2711 (no “a” suffix) was used as the Control Soil for the IVBA NIST SRM Round Robin Study batch extraction and analysis. The previous lot of SRM 2711 was kindly provided by several of the participating laboratories. The aliquots of NIST SRM 2711 material provided by the laboratories were each analyzed separately by the QATS Laboratory using EPA Method 3050 digestion, followed by ICP-AES analyses in order to verify that the materials were indeed the previous lot of SRM 2711, before mixing the sub-aliquots together for use in the Round Robin Study.

The laboratories were instructed to analyze the samples in strict accordance with the EPA IVBA SOP 9200.1-86 including all of the associated quality control samples, with the noted exceptions. Table 3 below provides the EPA IVBA SOP required quality control samples and associated acceptance criteria used in this study. Since the SRM materials were extracted and analyzed in five (5) replicates, there was no requirement for a duplicate sample analysis in this Round Robin Study.

To avoid the possibility of cross contamination, the laboratories were instructed to perform the extraction for the two (2) SRMs in separate extraction batches. The separate extraction batches allowed for the association of one set of QC samples with one SRM batch of samples.

Table 3. IVBA SOW Required QC Samples and Control Limits

QC Sample	Control Limits
Reagent blank	<25 µg/L lead
Bottle blank	<50 µg/L lead
Blank spike (10 mg/L)	85-115% recovery
Matrix spike (10 mg/L)	75-125% recovery
Duplicate sample	±20% RPD
Control soil (NIST 2710 or 2711)	±10% RPD

Extraction Apparatus and Air vs. Water Temperature Controlling Medium

During the review of the IDP forms provided by the candidate laboratories, it was noted that most of the laboratories had only one type of extraction apparatus, employing either air or water as the temperature controlling medium (37 ± 2ºC). The TRW Bioavailability Committee, realizing that there may not be a formal comparison of the two techniques of heat controlling mediums available, requested a statistical comparison of the two (2) temperature control mechanisms.

Statement of Work for the Round Robin Study

A statement of work (SOW) was developed by QATS personnel and the TRW Bioavailability Committee which provided instructions on the analysis and reporting of the IVBA Round Robin samples. The SOW provided a list of samples for each extraction batch and a recommended sequence of instrumental analysis of the extracted samples. The SOW also provided a list of the required associated QC sample analysis and QC sample acceptance ranges derived from the EPA IVBA SOP 9200.1-86. The SOW indicted that a separate digestion of the SRM materials using EPA Method 3050 was not required, as this data is available from the NIST SRM certificates of analysis.

Shipment of the IVBA Samples and Associated QC Samples

The Round Robin Study samples were shipped to seven (7) participating laboratories in October 2010. The laboratories were provided a 30 day turnaround time for submitting the sample results. The Round Robin sample shipments also included hardcopies of the SOW and the EPA IVBA SOP 9200.1-86.

Statistical Analysis of the Round Robin Study Results

Conventional statistical analysis techniques were used to analyze the data collected from the Round Robin Study. In general, the statistical analysis was performed in Microsoft Excel, using statistical analysis techniques such as Analysis of Variance (ANOVA) and t-test, which were performed using the Excel Analysis Tool Pac add-in package. Microsoft Excel 2003 (version 11.8328.8329) SP3; with statistical add-in package (Analysis Tool Pac, version 2003.110.8161) were used.

The main objective of this Round Robin Study was to derive a reasonably accurate estimate of the mean IVBA value for lead with known confidence for the two (2) NIST SRMs 2710a and 2711a, as well as to estimate the 99 percentile prediction interval. The mean and prediction interval for these SRMs allow the use of these NIST SRMs as Control Soils for various IVBA methods including the EPA IVBA SOP 9200.1-86. The prediction interval for the SRM generated by this study can also be used as guidance for setting, or verifying the acceptability, of the current IVBA acceptance criteria for the Control Soils in various IVBA methods including the EPA IVBA SOP 9200.1-86.

The statistical tool ANOVA, single factor (lead), was used to discern the intra-laboratory versus the inter-laboratory sources of variance of each SRM data set derived from the Round Robin Study.

A secondary objective was to investigate whether there is a statistically significant difference between air versus water as a temperature controlling medium of the extraction process. The t-test was used to statistically analyze the data derived from laboratories using an extraction apparatus employing air versus water data derived from those laboratories employing water as the temperature controlling medium. Specifically, the t-test employed was two (2) sample, assuming equal variances t-test.

The QC samples including the reagent blank, bottle bank, spiked blank, matrix, spike, and Control Soil SRM 2711 were co-extracted with the SRM samples and evaluated to determine if there were any anomalous data that might exclude the results submitted by a participating laboratory.

RESULTS AND DISCUSSION

Initial Demonstration of Proficiency

The initial demonstration of proficiency forms provided by the candidate laboratories are presented in Appendix A. As discussed in a previous section, these forms have been redacted to preserve anonymity. The original un-redacted forms are available from OSRTI Committee Chair Mr. James Konz. Out of the ten (10) candidate laboratories submitting IDP forms, seven (7) laboratories were selected to be participants in the study.

Round Robin Study Results

The seven (7) laboratories participating in the Round Robin Study analyzed each SRM in five (5) replicates, providing a total of 35 results for each of the two NIST SRMs 2710a and 2711a. The SOW provided to the laboratories contained several tables that allowed the laboratory to fill in the Round Robin sample analysis results using the Microsoft WORD application. The participating laboratories were asked to email the results to QATS, followed by hard copy results that could not be converted to electronic files. The results provided by the laboratories in the SOW Tables are presented in Appendix C, again, in redacted form. The original un-redacted SOW forms completed by the laboratories are available from the EPA TRW Bioavailability Committee chair, Mr. Michael Beringer of USEPA Region 7.

NIST SRM 2710a and SRM 2711a Results and Statistical Analysis

Tables 4 and 8 present the Round Robin Study results for the NIST SRM samples 2710a and 2711a, respectively, along with the mean, standard deviation (n-1 weighting), and the relative standard deviation (RSD) for each sample set. Please note that the values presented in these Tables are not rounded, and the pertinent rounded values will be presented later in this section. The Tables also present the type of extraction temperature controlling medium used by the laboratory in the header for each column of laboratory results. Tables 4 and 8 also present the 99 percentile prediction interval for the extracted lead in mg/Kg. The extracted lead prediction interval was converted to the IVBA prediction interval by dividing by the strong leach digestion value presented in the respective SRM certificates of analysis. The lead values for the EPA Method 3050 strong leach digestion of the SRMs 2710a and 2711a, are 5100 mg/Kg and 1300 mg/Kg, respectively. Tables 4 and 8 also provide the confidence interval of the mean for the two SRMs. The formulas used for the prediction interval and confidence interval of the mean are provided below.

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

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

where sd = standard deviation
t = Student's t; for n = 35, t = 2.728, for 99 percentile
sm = standard deviation of the mean

The average value (n=35) of extracted lead from SRM 2710a was 3440 mg/Kg, which is 67.5% (IVBA 67.5%) of the NIST certificate of analysis lead value of 5100 mg/Kg determined using the strong leach digestion EPA Method 3050. The average value (n=35) of the IVBA extracted lead from SRM 2711a was 1114 mg/Kg, which is 85.7 % (IVBA 85.7%) of the NIST certificate of analysis lead concentration of 1300 mg/Kg value determined using a strong leach digestion of 1300 mg/Kg. The NIST SRM 2710a collective results (n=35) exhibited an 11.4% range (lowest to highest value), and the NIST SRM 2711a collective results (n=35) exhibited a 14.7% range.

The calculated Relative Standard Deviations (RSDs) of the SRMs 2710a and SRM 2711a were 3.6 % (coefficient of variation (CV) = 0.0036) and 4.4% (CV = 0.0044), respectively. The calculated 99 percentile prediction interval for lead for SRM 2710a and 2711a, both as extracted lead in mg/Kg and as an IVBA value, is ± 10.0% and 12.1%, respectively. The calculated standard deviation of the mean (Sm) in percent for SRMs 2710a and 2711a is 0.61% and 0.75%, respectively. The calculated 99 percentile confidence interval of the mean for SRMs 2710a and 2711a, are ± 1.7% and ± 2.0%, respectively.

Tables 5 and 9 present results for the associated QC samples that were co-extracted with the SRMs 2710a and 2711a, respectively. These results include the reagent blank, bottled blank, blank spike, matrix spike, and the Control Soil SRM 2711. All results are within the acceptance ranges presented in the EPA IVBA SOP 9200.1-86, with the exception of the submitted blank values from laboratories A and C, at <30 and <40 ug/L, which are greater than the IVBA SOP stipulated detection limit of <25 ug/L. These minors errors in reporting do not have an impact on the sample results, as the samples analyzed were at least 100 fold more concentrated than these detection limits. Laboratory F did not report a bottled blank result, indicating that it was unnecessary because they were performing the extraction using air as the temperature controlling medium. However, they did report a reagent blank which was within the QC acceptance limit of < 25 ug/L.

Tables 5 and 9 also present the percent recovery of the Control Soil SRM 2711 as a percent of the expected value of 1100 mg/Kg, based on the leachable lead results from the NIST SRM 2711 certificate of analysis. All of the SRM 2711 Control Soil recoveries for both the SRM 2710a and 2711a extraction batches are within the acceptable range of ±10%, and the mean recovery of the Control Soil across all laboratories is 101.1% and 101.3%, respectively. It was noted in the IDP forms and the Round Robin Study results that some of the laboratories are using the total digested lead value of 1162 mg/Kg as the divisor for the calculation of the IVBA results, which is incorrect since the IVBA methods generally stipulate the use of EPA Method 3050 which is a strong acid leach digestion, not a total digestion (NFESC 2003). However, the difference in this case is small, as the total lead digested value for SRM 2711 is 1162 mg/Kg, and the leachable value is 1100 mg/Kg. As stated in the previous section, the SRM certificates of analysis are presented in Appendix B.

Tables 6 and 10 present the ANOVA results for SRM 2710a and SRM 2711a, respectively. For both sets of SRM results, the ANOVA results indicate that the intra-laboratory variance is low compared to inter-laboratory variance. This is indicated by the large value of the mean squared deviation about the mean (MS) for the inter-laboratory group compared to the lower intra-laboratory group MS value. The variance f-test (alpha 0.050, actually 0.0025 two tailed, 95 percentile) uses the null hypothesis that the data sets provided by the laboratories represent the same samples performed by the same method. The ANOVA algorithm calculates (or looks up in an algorithm table) the f-critical value based on the assumption of normal distributions of the intra-laboratory results and the entire group of samples results from all the laboratories. If the calculated f-value, which is based on the ratio of variances displayed by the between laboratory results to the variance of individual laboratory results, is greater than the f-critical value, then the null hypothesis is rejected, which is the case with both the NIST SRMs 2710a and 2711a data sets. These ANOVA results indicate that the difference in inter-laboratory data is large relative to the intra-laboratory data variances, and therefore the null hypothesis is rejected with a high confidence (low p-value). The rejection of the null hypothesis could indicate: 1) different methods were used in the analysis, 2) different samples were being analyzed, or 3) the intra-laboratory variance is small compared to what might be expected. The latter choice must be accepted as correct, considering the RSDs for the two (2) SRMs for the intra-laboratory (n=5) results all quite low, all less than 2%, and for nine (9) of the fourteen (14) sets of laboratory results, the RSD is less than 1%. The possibility that the rejection of the null hypothesis was because there were really two (2) types of

IVBA extractions performed, one using air and the other water as the temperature controlling medium, was evaluated using the t-test, which is discussed in the following section.

Air Versus Water Temperature Controlling Medium t-test

The t-test, specifically the two (2) sample - assuming equal variances t-test, (alpha 0.050, 95 percentile) was used to evaluate if there was a statistical difference between the results derived from the laboratories using air versus water as the temperature control medium for both SRMs. The Excel t-test output for both SRMs 2710a and 2711a are presented in Tables 7 and 11, respectively. The t-test results indicated that there is no difference between the data derived from the laboratories performing the extraction using air versus water as the medium for controlling the extraction temperature, as indicated by a $P(T \leq t)$ value that is greater than 0.05 for both t-tests performed on the data sets from the two SRMs analyses. Also, for both t-tests performed on the two (2) SRMs data sets, the t-Stat does not exceed the t Critical value for either. The percent difference between the mean of the data sets for air versus water as a temperature controlling medium was evaluated for both SRMs 2710a and 2711a and determined to be 2.4% and 1.1%, respectively. It can be seen that the individual result values for the air and water data sets overlap, for both the SRM 2710a and 2711a sets of data.

Comparison to Previous SRM IVBA values.

The previous NIST SRM 2710 and 2711 Control Soil accepted nominal values were determined by Dr. John Drexler, using 68 and 66 intra-laboratory results, respectively. The previous NIST SRM IVBA values were derived from the EPA report OSWER 9285.7-77, May 2007. Table 12 below presents the mean, standard deviation and coefficient of variation for the previous NIST materials, as well as the value of the replacement NIST SRMs 2710a and 2711a determined by the Round Robin Study. The NIST SRM 2711 Control Soil used in this study, for both batches of analyses, for all seven (7) laboratories (n=14), exhibited a mean IVBA value of 85.4%, which translates to 101.2% of the accepted nominal value.

Table 12 presents the NIST SRM 2710a IVBA pooled value at 67.5%, which is lower than the accepted nominal value for the previous lot of SRM 2710 at 75.5%. This difference in IVBA value between the current and previous batches of SRM may be due to different material(s) used for spiking the lead during the SRM preparation, or perhaps due to a difference in mean particle size. Table 12 also presents the SRM 2711a pooled IVBA value of 85.7%, which is slightly higher than the IVBA of 84.4% for previous lot of this SRM. Table 12 presents the pooled RSDs of the IVBA results for the two new lots of SRMs 2710a and 2711a, used in this Round Robin Study. The RSDs are less than those derived from the previous lots of these NIST SRMs, which were derived from intra-laboratory analysis (Fig 3-3 EPA, 2007). This indicates that the Round Robin Study results for the new lots of SRMs were performed with overall good precision.

Rounded Values for NIST SRMs 2710a and 2711a

Table 13 presents the 99 percentile acceptance range rounded values for NIST SRMs 2710a and 2711a based on the pooled Round Robin results. The acceptance ranges are presented both in mg/Kg and as IVBA values.

Table 4. Laboratory Results and the Prediction and Confidence Intervals for NIST 2710a

NIST 2710a	**Analyte: Lead**	**Units: mg/Kg**					
Laboratory >	**A**	**B**	**C**	**D**	**E**	**F**	**G**
Extraction Type >	**Water**	**Water**	**Water**	**Water**	**Water**	**Air**	**Air**
Rep 1	3290	3520	3320	3567.5	3652.5	3372	3430
Rep 2	3270	3470	3300	3592.6	3623.4	3314	3370
Rep 3	3290	3483	3360	3495.6	3663.2	3321	3420
Rep 4	3300	3479	3330	3536.2	3632.6	3347	3430
Rep 5	3290	3538	3370	3617.0	3605.6	3348	3460
AVG	3288.0	3498.0	3336.0	3561.8	3635.5	3340.4	3422.0
Std Dev	10.95	29.39	28.81	47.61	22.94	23.31	32.71
RSD	0.33	0.84	0.86	1.34	0.63	0.70	0.96

Pooled	**n=35**
Average	3440.23
Std Dev	124.58
RSD	3.62

Extracted Pb 99 - Percentile Prediction Interval (mg/Kg)		
99 low	**Average**	**99 high**
3095.56	3440.23	3784.91
10.02% = **± 99 prediction interval in percent**		

The range above should be used to determine if a laboratory extracted lead result is acceptable.

Lead IVBA 99-Percentile Prediction Interval		
99 low	**Average**	**99 high**
60.70	67.46	74.21
NIST 2710a Digestion EPA Method 3050 median result from the NIST certificate is 5100 mg/Kg		
IVBA = 67.46 or 67.5%	**SD =** 2.44	**RSD =** 3.62

The range above should be used to determine if a laboratory lead IVBA result is acceptable.

Confidence Interval of the Mean		
3440.23 = **Mean**	21.05798 = **SD of the Mean**	0.61 = **RSD of the Mean**
99 low	**Average**	**99 high**
3382.79	3440.23	3497.68
1.67 % = ± 99 percentile of the confidence interval of the mean		

Std Dev = Standard Deviation
RSD = Relative Standard Deviation
CI = Confidence Interval

Table 5. SRM 2710a Batch QC Sample Results, Lead

Laboratory>	A	B	C	D	E	F	G	Mean
Extraction Type>	**Water**	**Water**	**Water**	**Water**	**Water**	**Air**	**Air**	
Reagent Blank <25 ug/L	<30	<5	<40	<0.95	1.98	2.67	9.6	**na**
Bottle Blank ug/L <50 ug/L	<30	<5	<40	<0.95	1.86	NA	5.1	**na**
Blank Spike Percent Recovery (85-115%)	96.1	98.6	96.3	99.0	100.0	97.0	98.0	**97.9**
SRM 2710a Matrix Spike Percent Recovery (75 -125%)	96.0	89.2	96.9	99.0	83.6	79.0	83.0	**89.5**
Control Soil SRM 2711 mg/Kg (nominal =928.4 mg/Kg)	865	953	910	977.8	1007.2	906.6	953	**938.9**
IVBA Control Soil SRM 2711 mg/Kg IVBA = 84.4 (%)	78.6%	86.6%	82.7%	88.9%	91.6%	82.4%	86.6%	**85.4%**
IVBA Control Soil SRM 2711 Percent Recovery (%)	93.2%	102.6%	98.0%	105.3%	108.5%	97.7%	102.6%	**101.1%**

na = not applicable

Table 6. NIST 2710a Round Robin Results Analysis of Variance

Excel ANOVA: Single Factor (Lead)

note alpha at 0.05 (95 percentile)

SUMMARY

Groups	***Count***	***Sum***	***Average***	***Variance***
Laboratory A	5	16440	3288	120
Laboratory B	5	17490	3498	863.5
Laboratory C	5	16680	3336	830
Laboratory D	5	17808.9	3561.78	2266.492
Laboratory E	5	18177.3	3635.46	526.278
Laboratory F	5	16702	3340.4	543.3
Laboratory G	5	17110	3422	1070

ANOVA

Source of Variation	***SS***	***df***	***MS***	***F***	***P-value***	***F crit***
Inter-laboratory	502813.7789	6	83802.29648	94.31778649	2.93938E-17	2.445259395
Intra-laboratory	24878.28	28	888.51			
Total	527692.0589	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

Table 7. NIST SRM 2710a Results, Air versus Water Temperature Control Medium, T-Test

NIST 2710a	**Analyte: Lead**		**Units: mg/Kg**						
Extraction Type>	**WATER**							**AIR**	
Laboratory>	**A**	**B**	**C**	**D**	**E**			**F**	**G**
Rep 1	3290	3520	3320	3567.5	3652.5		**Rep 1**	3372	3430
Rep 2	3270	3470	3300	3592.6	3623.4		**Rep 2**	3314	3370
Rep 3	3290	3483	3360	3495.6	3663.2		**Rep 3**	3321	3420
Rep 4	3300	3479	3330	3536.2	3632.6		**Rep 4**	3347	3430
Rep 5	3290	3538	3370	3617	3605.6		**Rep 5**	3348	3460
AVG	3288.0	3498.0	3336.0	3561.8	3635.5		**AVG**	3340.4	3422.0
Std Dev	10.95	29.39	28.81	47.61	22.94		**Std Dev**	23.31	32.71
RSD	0.33	0.84	0.86	1.34	0.63		**RSD**	0.70	0.96

	WATER	**PERCENT DIFFERENCE**		**AIR**
	n=25	**2.41%**		**n=10**
AVG	3463.9		**AVG**	3381.2
Std Dev	137.8		**Std Dev**	50.7
RSD	4.0		**RSD**	1.5

Excel t-Test: Two-Sample Assuming Equal Variances

		alpha = 0.05
	WATER	**AIR**
Mean	3463.85	3381.2
Variance	18991.74177	2566.622222
Observations	25	10
Pooled Variance	14512.16371	
Hypothesized Mean Difference	0	
df	33	
t Stat	1.833590061	
P(T ≤ t) two-tail	0.075747815	
t Critical two-tail	2.034515287	

t-Stat = t-statistic
t crit = t critical value

P(T ≤ t) two tail = if the value is less than 0.05 indicates a 95% probability that the means of the two groups do not come from the same population

Table 8. Laboratory Results and the Prediction and Confidence Intervals for NIST 2711a

NIST SRM 2711a Analyte: Lead Units: mg/Kg							
Laboratory>	**A**	**B**	**C**	**D**	**E**	**F**	**G**
Extraction Type>	**Water**	**Water**	**Water**	**Water**	**Water**	**Air**	**Air**
Rep 1	1040	1145	1080	1138.3	1181.7	1099	1130
Rep 2	1030	1147	1100	1121.3	1194.2	1057	1130
Rep 3	1040	1122	1080	1155.1	1177.6	1089	1130
Rep 4	1030	1157	1080	1150.8	1182.2	1086	1120
Rep 5	1030	1165	1060	1151.1	1190.8	1082	1130
AVG	1034.0	1147.2	1080.0	1143.3	1185.3	1082.6	1128.0
Std Dev	5.48	16.22	14.14	13.83	6.92	15.63	4.47
RSD	0.53	1.41	1.31	1.21	0.58	1.44	0.40

Pooled	**n=35**
Average	1114.4
Std Dev	49.4
RSD	4.4

Extracted Pb 99 – Percentile Prediction Interval (mg/Kg)		
99low	**Average**	**99 high**
979.64	1114.35	1249.05
12.09 **= ± 99 percentile prediction interval in percent**		

The range above should be used to determine if a laboratory extracted lead result is acceptable

IVBA 99-Percentile Prediction Interval		
99 low	**Average**	**99 high**
75.21	85.72	96.23
NIST 2711a Digestion EPA Method 3050 the median result from the NIST certificate of analysis is 1300 mg/Kg		
so IVBA =85.72 or 85.7%	**SD=** 3.80	**RSD =** 4.43

The range above should be used to determine if a laboratory lead IVBA result is acceptable

Confidence Interval of the Mean at 99 percentile		
1114.35 = **Mean**	8.346 = **SD of the Mean**	0.749 = **RSD of the Mean**
99 low	**Average**	**99 high**
1091.58	1114.35	1137.11
2.04% = **± 99 percentile confidence interval of the mean**		

Std Dev = Standard Deviation
RSD = Relative Standard Deviation
CI = Confidence Interval

Table 9. SRM 2711a Batch QC Sample Results, Lead

Laboratory>	A	B	C	D	E	F	G	Mean
Extraction Type>	**Water**	**Water**	**Water**	**Water**	**Water**	**Air**	**Air**	
Reagent Blank <25 ug/L	<30	<5	<40	<0.95	1.7	0.55	11.4	**na**
Bottle Blank ug/L <50 ug/L	<30	<5	<40	<0.95	1.42	nr	4.6	**na**
Blank Spike Percent Recovery (85-115%)	95.7%	96.6%	95.7%	95%	98.6%	98%	98%	**96.8%**
SRM 2711a Matrix Spike Percent Recovery (75 -125%)	93.6%	95.4%	82.7%	93%	108.8%	75%	93%	**91.6%**
Control Soil SRM 2711 mg/Kg (nominal =928.4 mg/Kg)	861.1	967	900	958.8	1014	921.7	958	**940.1**
IVBA Control Soil SRM 2711 mg/Kg IVBA = 84.4 (%)	78.3%	87.9%	81.8%	87.2%	92.2%	83.8%	87.1%	**85.5%**
IVBA Control Soil SRM 2711 Percent Recovery (%)	92.8%	104.2%	96.9%	103.3%	109.2%	99.3%	103.2%	**101.3%**

nr = not reported
na = not applicable

Table 10. NIST 2711a Round Robin Results Analysis of Variance

Anova: Single Factor (Lead)

note alpha at 0.05 (95 percentile)

SUMMARY

Groups	*Count*	*Sum*	*Average*	*Variance*
Laboratory A	5	5170	1034	30
Laboratory B	5	5736	1147.2	263.2
Laboratory C	5	5400	1080	200
Laboratory D	5	5716.6	1143.32	191.332
Laboratory E	5	5926.5	1185.3	47.83
Laboratory F	5	5413	1082.6	244.3
Laboratory G	5	5640	1128	20

ANOVA

Source of Variation	*SS*	*df*	*MS*	*F*	*P-value*	*F crit*
Inter-laboratory	78913.57886	6	13152.26314	92.37418704	3.87283E-17	2.445259395
Intra-laboratory	3986.648	28	142.3802857			
Total	82900.22686	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

Table 11. NIST SRM 2711a Results, Air versus Water Temperature Control Medium, T-Test

NIST 2711a	**Analyte: Lead**		**Units: mg/Kg**					
Extraction Type>	**WATER**						**AIR**	
Laboratory>	A	B	C	D	E		**F**	**G**
Rep 1	1040	1145	1080	1138.3	1181.7		1099	1130
Rep 2	1030	1147	1100	1121.3	1194.2		1057	1130
Rep 3	1040	1122	1080	1155.1	1177.6		1089	1130
Rep 4	1030	1157	1080	1150.8	1182.2		1086	1120
Rep 5	1030	1165	1060	1151.1	1190.8		1082	1130
AVG	1034.0	1147.2	1080.0	1143.3	1185.3		1082.6	1128.0
Std Dev	5.48	16.22	14.14	13.83	6.92		15.63	4.47
RSD	0.53	1.41	1.31	1.21	0.58		1.44	0.40

	WATER	**PERCENT DIFFERENCE**		**AIR**
	n=25	**1.14**		**n=10**
AVG	1117.96		**AVG**	1105.30
Std Dev	56.10		**Std Dev**	26.27
RSD	5.02		**RSD**	2.38

Excel t-Test: Two-Sample Assuming Equal Variances

		alpha = 0.05
	Water	**Air**
Mean	1117.964	1105.3
Variance	3147.690733	690.0111111
Observations	25	10
Pooled Variance	2477.414473	
Hypothesized Mean Difference	0	
df	33	
t Stat	0.679997865	
P(T ≤ t) two-tail	0.501248691	
t Critical two-tail	2.034515287	

t-Stat = t-statistic
t crit = t critical value

P(T ≤ t) two tail = if the value is less than 0.05 indicates a 95% probability that the means of the two groups do not come from the same population

Table 12. Round Robin Study SRM IVBA Results Compared to Previous IVBA Results

SRM	Mean IVBA	Standard Deviation	RSD	CV	N
2710 Previous Lot	75.5%	4.7	6.2	0.062	68
2711 Previous Lot	84.4%	4.7	5.5	0.055	66
2711 This Study	85.4%	4.3	5.0	0.050	14
2710a	67.5%	2.4	3.6	0.036	35
2711a	85.7%	3.8	4.4	0.044	35

Table 13. NIST SRMs 2710a and 2711a 99 Percentile Rounded Values

SRM	Low 99	Average	High 99
SRM 2710a (mg/Kg)	3100	3440	3780
SRM 2710a IVBA	60.7	67.5	74.2
SRM 2711a (mg/Kg)	980	1110	1250
SRM 2711a IVBA	75.2	85.7	96.2

CONCLUSIONS AND RECOMENDATIONS

The primary objective this Round Robin Study was the determination of new lead IVBA values with known confidence, along with suitable acceptance ranges for the two (2) new NIST SRMs 2710a and 2711a. The Round Robin Study results from the seven (7) participating laboratories were all determined to be acceptable and allowed the establishment of the IVBA values for the new SRM 2710a and 2711a with a known and acceptable precision. This study also provided for the determination of IVBA lead result (mg/Kg) with known and acceptable precision for the two new SRMs. The associated QC results provided by the participating laboratories were all within the EPA SOP 9200.1-86 defined acceptable ranges, with a few noted exceptions.

A secondary objective of this study was to evaluate if the use of air or water as a temperature controlling medium resulted in any statistically significant difference in the resulting data. It appears, from this Round Robin Study data set, that the two methods of controlling the IVBA extraction temperature are not statistically different.

REFERENCES

US EPA (2008). *Standard Operating Procedure for an In Vitro Bioaccessibility Assay for Lead in Soil*, USEPA OSWER 9200.1-86, November 2008

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

NFESC (2003). *Guide for Incorporating Bioavailability Adjustments into Human Health and Ecological Risk Assessments at U. S. Department of Defense Facilities*, Naval Facilities Engineering Service Center (NFESC), Air Force Center for Environmental Excellence (AFCEE), Army Environmental Center (AEC), Part 2, Appendix C, Ruby, M., Drexler, J., *"In Vitro Method for Determination of Lead Bioaccessibility Standard Operating Procedure for Stomach Phase Extraction"*, June 2003. Retrieved: March14, 2011, from: http://web.ead.anl.gov/ecorisk/related/documents/Bioavail_Part_2-_Appendices-final.pdf

APPENDIX A

Initial Demonstration of Proficiency Forms

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

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

General and Facility Questions

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

Apparatus

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

Analytical

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

Analytical (continued)

Table of Batch IVBA Results

No	Batch Date	Reagent Blank µg/L	Bottle Blank µg/L	Spiked Blank Result	Spike Blank Percent Recovery	Matrix Spike Percent Recovery	Duplicate Relative Percent Difference	Reference Material Name	Control Soil Result (mg/L) (analytical solution)	Control Soil RPD	Control Soil IVBA
A	Date	<25 µg/L	<50 µg/L	(mg/L)	85-115%	75-125%	<20%RPD			<10%RPD	IVBA%
B	mm/dd/yyyy	<25 µg/L	<50 µg/L	9.2	92.4%	87.3%	7.4%	NIST 2711	9.12 mg/L	7.1%	75.5%
1	01/15/2007	<50	<50	9.5	95.0	N/A	11.9	NIST 2711	10.1	9.54	84.6
2	10/02/2007	<50	<50	9.5	95.0		0.0	NIST 2711	9.42	2.17	81.4
3	11/26/2007	<50	<50	9.5	94.7		9.5	NIST 2711	9.85	6.83	84.8
4	11/28/2007	<50	<50	9.3	93.3		2.4	NIST 2711	9.59	4.01	82.5
5	12/03/2007	<50	<50	9.4	94.1		27.0	NIST 2711	9.44	2.39	81.2
6	12/04/2007	<50	<50	9.5	94.7		6.9	NIST 2711	9.75	5.75	83.9
7	12/05/2007	<50	<50	9.5	95.0		31.6	NIST 2711	9.85	6.83	84.8
8	12/05/2007	<50	<50	9.5	95.2		14	NIST 2711	10.0	8.46	86.1
9	12/05/2007	<50	<50	9.5	95.2		5.1	NIST 2711	10.1	9.54	86.9
10	12/09/2007	<50	<50	9.8	97.8		10.0	NIST 2711	9.42	2.17	81.1
11	03/25/2008	<0.1	1.1	9.3	93.0		6.2	NIST 2711	10.2	10.6	89.2
12	03/28/2008	<0.1	0.7	10.5	105		2.3	NIST 2711	10.7	16.1	90.6
13	05/08/2008	<0.1	0.7	9.8	98		0.4	NIST 2711	10.9	18.2	91.5
14	05/08/2008	<0.1	0.8	10.2	102		2.7	NIST 2711	10.1	9.54	86.8
15	11/26/2008	<0.1	4.2	9.8	98		2.8	NIST 2711	9.79	6.18	84.3
16	11/27/2008	<0.1	1.0	10.5	105		4.2	NIST 2711	10.4	12.8	89.5
17	02/03/2009	<0.1	3.5	9.9	99		3.6	NIST 2711	9.50	3.04	80.6
18	02/04/2009	<0.1	0.9	9.9	99		7.7	NIST 2711	9.77	5.97	82.6
19	02/05/2009	<0.1	0.5	10.5	105		2.3	NIST 2711	8.97	-2.71	75.7
20	02/05/2009	<0.1	0.5	9.9	99		1.5	NIST 2711	9.34	1.30	79.3
21	02/05/2009	<0.1	0.4	9.9	99		1.5	NIST 2711	8.97	-2.71	75.7
22	02/10/2009	<0.1	12.5	11.1	111		14.5	NIST 2711	10.0	8.46	84.4
23	02/11/2009	<0.1	0.2	10.3	103		1.6	NIST 2711	10.1	9.54	86.2
24	02/12/2009	<0.1	0.2	10.2	102		2.2	NIST 2711	10.2	10.6	87.1
25	02/12/2009	<50	<50	10.3	103		2.9	NIST 2711	9.34	1.30	78.4
26	10/05/2009	<50	<50	9.5	95.0		4.5	NIST 2711	10.6	15.0	91.6
27	11/23/2009	<0.1	<0.5	10.3	103		11.9	NIST 2711	10.4	12.8	88.6

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

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

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

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

General and Facility Questions

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

Apparatus

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

Analytical

10	**Type of analytical instrument use for the final Determination (ICP-AES) (ICP-MS) (GFAA) or specify other instrument type**	**ICP-MS**
11	**Please provide the instrumental detection limit for the procedure that you currently use for the IVBA method. (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.**

Analytical (continued)

Table of Batch IVBA Results

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

* Spiked to 1.0 mg/L Pb.

** NIST soil extracted 4 times during this data set.

^ Assumes concentration of lead in NIST 2711 soil is 1162 mg/kg, per certificate of analysis.

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

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

(Submitted 7-26-2010)

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

General and Facility Questions

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

Apparatus

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

Analytical

10	**Type of analytical instrument use for the final Determination (ICP-AES) (ICP-MS) (GFAA) or specify other instrument type.**	**ICP-AES or ICP-MS (We have both)**
11	**Please provide the aqueous method detection limit for the procedure that you currently use for the IVBA method. (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**

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	ACZ 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	ACZ M3 FLAG* SEE Below	2.5	NIST 2711	8.92	2.7	77

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

M3 Flag on Lab -C'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)

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

(Submitted 7-21-2010)

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

General and Facility Questions

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

Apparatus

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

Analytical

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

Analytical (continued)

Table of Batch IVBA Results

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

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

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

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

General and Facility Questions

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

Apparatus

8	**Does the IVBA apparatus your facility has use air or water as the 37^{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**

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.

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

(Submitted 7-13-2010)

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

General and Facility Questions

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

Apparatus

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

Analytical

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

Analytical (continued)

Table of Batch IVBA Results

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

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

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

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

(Received 7-28-2010)

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?**	**105**
2	**Will your facility conduct the extraction? (Yes/No)**	**Yes**
3	**If the answer to question 2 is no, please provide the name of the laboratory that will be conducting the extraction. (Lab Name)**	
4	**Will your facility conduct the extract analysis? (Yes/No)**	**No**
5	**If the answer to question 4 is no, please provide the name of the laboratory that will be conducting the analysis. (Lab Name)**	**Other Lab Name was here.**
6	**Will your facility be able to conduct the attached IVBA Method EPA 9200.1-86 as written? (air controlled temperature is OK) (Yes, or Provide comment Below in 7)**	**Yes, by air controlled temperature.**
7	**If the answer the question 6 is no, please provide the deviation from the EPA 9200.1-86 method in the field provided here. Comment-**	

Apparatus

8	**Does the IVBA apparatus your facility has use air or water as the 37^{O}C thermal conducting/controlling medium. (Air, Water)**	**Air**
9	**How many bottle positions does your apparatus have?**	**~20 samples**

Analytical

10	**Type of analytical instrument use for the final Determination (ICP-AES) (ICP-MS) (GFAA) or specify other instrument type.**	**ICP-AES (we have also used ICP-MS in past measurements)**
11	**Please provide the aqueous method detection limit for the procedure that you currently use for the IVBA method. (µg/L)**	**5 µg/L (0.5 µg/L for ICP-MS)**
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 and 2711**
13	**Blank spike amount (mg/L) used in your procedure.**	**50 µg/L with ICP-MS analysis**
14	**Matrix spike amount (mg/L) used in your procedure.**	**120 µg/L with ICP-MS analysis**

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	06/17/2009	no	200 μg/L	n/a	100%	no	no	2711	9.29 mg/L	no	75%
2	01/20/2006	no	<40 μg/L	n/a	no	no	4.8%	2710	41.6 mg/L	no	75.2%
3	06/16/2009	no	100 μg/L	n/a	111%	90%	no	2711	8.84 mg/L	no	76%
4	06/17/2009	no	200 μg/L	n/a	111%	82%	no	2711	8.88 mg/L	no	76%
5	02/17/2009	no	0.69 μg/L	n/a	no	no	no	2711	8.73 mg/L	no	75%
6	02/17/2009	no	0.69 μg/L	n/a	no	no	no	2711	8.69 mg/L	no	75%
7	02/17/2009	no	0.69 μg/L	n/a	no	no	no	2711	8.57 mg/L	no	74%
8	02/17/2009	no	0.69 μg/L	n/a	no	no	no	2711	8.54 mg/L	no	74%
9	02/17/2009	no	0.69 μg/L	n/a	no	no	no	2711	8.43 mg/L	no	73%
10	02/17/2009	no	0.69 μg/L	n/a	no	no	no	2711	8.44 mg/L	no	73%
11	01/20/2006	no	<40 μg/L	n/a	no	no	3.9%	2710	39.9 mg/L	no	72.1%
12	01/20/2006	no	<30 μg/L	n/a	no	no	4.9%	2710	41.2 mg/L	no	74.4%
13	01/20/2006	no	<30 μg/L	n/a	no	no	3.6%	2710	43.2 mg/L	no	78.1%

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

APPENDIX B

NIST SRM Certificates of Analysis

National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 2710

Montana 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. SRM 2710 is a highly contaminated soil that was oven-dried, sieved, radiation sterilized, and blended to achieve a high degree of homogeneity. A unit of SRM 2710 consists of 50 g of the dried material.

The certified elements for SRM 2710 are given in Table 1. The values are based on measurements using one definitive method or two or more independent and reliable analytical methods. Noncertified values for a number of elements are given in Table 2 as additional information on the composition. The noncertified values should **NOT** be used for calibration or quality control. Analytical methods used for the characterization of this SRM are given in Table 3 along with analysts and cooperating laboratories. All values (except for carbon) are based on measurements using a sample weight of at least 250 mg. Carbon measurements are based on 100 mg samples.

NOTICE AND WARNINGS TO USERS

Expiration of Certification: This certification of SRM 2710 is valid, within the measurement uncertainties specified, until **31 December 2011**, provided the SRM is handled in accordance with 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. Return of the attached registration card will facilitate notification.

The overall direction and coordination of the analyses were under the chairmanship of M.S. Epstein and R.L. Watters, Jr. of the NIST Analytical Chemistry Division of the NIST Measurement Services Division.

Statistical consultation was provided by S.B. Schiller of the NIST Statistical Engineering Division.

The technical and support aspects involved in the original preparation, certification, and issuance of this SRM were coordinated through the NIST Standard Reference Materials Program by T.E. Gills and J.S. Kane. Revision of this certificate was coordinated through the NIST Standard Reference Materials Program by B.S. MacDonald of the NIST Measurement Services Division.

Willie E. May, Chief
Analytical Chemistry Division

Gaithersburg, MD 20899
Certificate Issue Date: 18 July 2003
See Certificate Revision History on Page 6

John Rumble, Jr., Chief
Measurement Services Division

INSTRUCTIONS FOR USE

Use: A minimum sample weight of 250 mg (dry weight - see Instructions for Drying) should be used for analytical determinations to be related to the certified values on this Certificate of Analysis.

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

Instructions for Drying: When nonvolatile elements are to be determined, samples should be dried for 2 h at 110 °C. Volatile elements (i.e., Hg, As, Se) 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 is to 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 weight loss on drying has been found to be in the range of 1.7 % to 2.3 %.

PREPARATION AND ANALYSIS

Source and Preparation of Material: The U.S. Geological Survey (USGS), under contract to NIST, collected and processed the material for SRM 2710. The soil was collected from the top 10 cm (4 in) of pasture land located at Longitude 112° 47' and Latitude 46° 01' along Silver Bow Creek in the Butte, Montana area. The site is approximately nine miles east of the local Anaconda plant and 6.5 miles south of settling ponds that feed the creek. The creek periodically floods, depositing sediment with high concentrations of copper, manganese, and zinc at the collection site. The material was shoveled from a 6.1 m × 6.1 m (20 ft × 20 ft) area into polyethylene bags in cardboard cartons for shipment to the USGS laboratory for processing.

The material was spread on 30.5 cm × 61 cm (1 ft × 2 ft) polyethylene-lined drying trays in an air drying oven and dried for three days at room temperature. The material was then passed over a vibrating 2 mm screen to remove plant material, rocks, and large chunks of aggregated soil. Material remaining on the screen was deaggregated and rescreened. The combined material passing the screen was ground in a ball mill to pass a 74 µm screen and blended for 24 h. Twenty grab samples were taken and measured for the major oxides using X-ray fluorescence spectrometry and for several trace elements using inductively coupled plasma atomic emission analysis to provide preliminary assessment of the homogeneity of the material prior to bottling. The material was bottled into 50 g units and randomly selected bottles were taken for the final homogeneity testing.

Analysis: The homogeneity, using selected elements in the bottled material as indicators, was assessed using X-ray fluorescence spectrometry and neutron activation analysis. In a few cases, statistically significant differences were observed, and the variance due to material inhomogeneity is included in the overall uncertainties of the certified values. The estimated relative standard deviation for material inhomogeneity is less than 2 % for those elements for which homogeneity was assessed.

Certified Values and Uncertainties: The certified values are weighted means of results from two or more independent analytical methods, or the mean of results from a single definitive method, except for mercury. Mercury certification is based on cold vapor atomic absorption spectrometry used by two different laboratories employing different methods of sample preparation prior to measurement. The weights for the weighted means were computed according to the iterative procedure of Paule and Mandel [1]. The stated uncertainties include allowances for measurement imprecision, material variability, and differences among analytical methods. Each uncertainty is the sum of the half-width of a 95 % prediction interval and includes an allowance for systematic error among the methods used. In the absence of systematic error, a 95 % prediction interval predicts where the true concentrations of 95 % of the samples of this SRM lie. The certified values were corroborated by analyses from nine Polish laboratories cooperating on the certification under the direction of T. Plebanski and J. Lipinski, Polish Committee for Standardization, Measures, and Quality Control. The Polish laboratory work was supported by the Maria Sklodowska-Curie Joint Fund.

Table 1. Certified Values

Element	Mass Fraction (%)			Element	Mass Fraction (mg/kg)		
Aluminum	6.44	±	0.08	Antimony	38.4	±	3
Calcium	1.25	±	0.03	Arsenic	626	±	38
Iron	3.38	±	0.10	Barium	707	±	51
Magnesium	0.853	±	0.042	Cadmium	21.8	±	0.2
Manganese	1.01	±	0.04	Copper	2950	±	130
Phosphorus	0.106	±	0.015	Lead	5532	±	80
Potassium	2.11	±	0.11	Mercury	32.6	±	1.8
Silicon	28.97	±	0.18	Nickel	14.3	±	1.0
Sodium	1.14	±	0.06	Silver	35.3	±	1.5
Sulfur	0.240	±	0.006	Vanadium	76.6	±	2.3
Titanium	0.283	±	0.010	Zinc	6952	±	91

Noncertified Values: Noncertified values shown below are provided for information only. An element concentration value is not certified if a bias is suspected in one or more of the methods used for certification, or if two independent methods are not available.

Table 2. Noncertified Values

Element	Mass Fraction (%)	Element	Mass Fraction (mg/kg)
Carbon	3	Bromine	6
		Cerium	57
		Cesium	107
		Chromium	39
		Cobalt	10
		Dysprosium	5.4
		Europium	1
		Gallium	34
		Gold	0.6
		Hafnium	3.2
		Holmium	0.6
		Indium	5.1
		Lanthanum	34
		Molybdenum	19
		Neodymium	23
		Rubidium	120
		Samarium	7.8
		Scandium	8.7
		Strontium	330
		Thallium	1.3
		Thorium	13
		Tungsten	93
		Uranium	25
		Ytterbium	1.3
		Yttrium	23

Table 3. Analytical Methods Used for the Analysis of SRM 2710

Element	Certification Methods*	Element	Certification Methods*
Ag	ID ICPMS, RNAA, **INAA**	Mg	XRF1, ICP
Al	XRF1, XRF2, DCP, **ICP**	Mn	INAA, DCP, XRF2
As	RNAA, HYD AAS, **ICP, INAA**	Mo	**ID ICPMS**
Au	**INAA**, FAAS	Na	INAA, FAES
Ba	XRF2, FAES	Nd	**ICP**
Br	**INAA**	Ni	ID ICPMS, ETAAS, **INAA**
C	**COUL**	P	DCP, COLOR, XRF1, XRF2
Ca	XRF1, XRF2, DCP	Pb	ID TIMS, **POLAR**, **ICP**
Cd	ID ICPMS, RNAA	Rb	**INAA**
Ce	**INAA**, **ICP**	S	ID TIMS
Co	**INAA**, **ETAAS**, **ICP**	Sb	RNAA, ETAAS
Cr	**INAA**, **DCP**, **ICP**	Sc	**INAA**, **ICP**
Cs	**INAA**	Si	XRF1, XRF2, GRAV
Cu	RNAA, FAES, **ICP**	Sm	**INAA**
Dy	**INAA**	Sr	ID TIMS, **INAA**, **ICP**
Eu	**INAA**	Th	**ID TIMS, INAA**, **ICP**
Fe	XRF1, XRF2, DCP, **INAA**, **ICP**	Ti	XRF1, XRF2, DCP
Ga	**INAA**, **ICP**	Tl	**ID TIMS**, **LEAFS**
Hf	**INAA**	U	**ID TIMS**, **INAA**
Hg	CVAAS	V	INAA, ICP
Ho	**INAA**	W	**INAA**
In	**INAA**	Y	**ICP**
K	XRF1, XRF2, FAES, **ICP**	Yb	**INAA**
La	**INAA**, **ICP**	Zn	ID TIMS, **ICP**, **INAA**, **POLAR**

*Methods in **bold** were used to corroborate certification methods or to provide information values.

COLOR	Colorimetry; lithium metaborate fusion
COUL	Combustion coulometry
CVAAS	Cold vapor atomic absorption spectrometry
DCP	Direct current plasma atomic emission spectrometry; lithium metaborate fusion
ETAAS	Electrothermal atomic absorption spectrometry; mixed acid digestion
FAAS	Flame atomic absorption spectrometry; mixed acid digestion except for Au, leached with $HBr-Br_2$
FAES	Flame atomic emission spectrometry; mixed acid digestion
GRAV	Gravimetry; sodium carbonate fusion
HYD AAS	Hydride generation atomic absorption spectrometry
ICP	Inductively coupled plasma atomic emission spectrometry; mixed acid digestion
ID ICPMS	Isotope dilution inductively coupled plasma mass spectrometry; mixed acid digestion
ID TIMS	Isotope dilution thermal ionization mass spectrometry; mixed acid digestion
INAA	Instrumental neutron activation analysis
LEAFS	Laser enhanced atomic fluorescence spectrometry; mixed acid digestion
POLAR	Polarography
RNAA	Radiochemical neutron activation analysis; mixed acid digestion
XRF1	Wavelength dispersive X-ray fluorescence spectrometry on fused borate discs
XRF2	Wavelength dispersive X-ray fluorescence spectrometry on pressed powder

Participating NIST Analysts:

M. Adriaens	A. Marlow
E.S. Beary	J.R. Moody
C.A. Beck II	P.J. Paulsen
D.S. Braverman	P. Pella
M.S. Epstein	T.A. Rush
J.D. Fassett	J.M. Smeller
K.M. Garrity	G.C. Turk
R.R. Greenberg	T.W. Vetter
W.R. Kelly	R.D. Vocke
R.M. Lindstrom	L.J. Wood
E.A. Mackey	R.L. Watters, Jr.

Participating Laboratories:

P. Briggs, D. Siems, J. Taggart, S. Wilson
U.S. Geological Survey
Branch of Geochemistry
Denver, CO, USA

J.B. Bodkin
College of Earth and Mineral Sciences
The Pennsylvania State University
University Park, PA, USA

S.E. Landsberger, V.G. Vermette
Department of Nuclear Engineering
University of Illinois
Urbana, IL, USA

J. Lipinski, T. Plebanski
Polish Committee for Standardization,
Measures and Quality Control
Warsaw, Poland

M. Bielawska, B. Galczynska,
J. Galczynska, K. Galczynski,
K. Wiacek
Institute of Soil Science and Plant
Cultivation
Pulawy, Poland

I. Matuszczyk
Forest Research Institute
Division in Katowice,
Warsaw, Poland

Z. Jonca
Institute of Environmental Protection
Warsaw, Poland

P. Bienkowski
Institute of Ecology
Dziekanow Lesny, Poland

H. Matusiewicz
Technical University
Poznan, Poland

B. Ksiazek
Geological Enterprise
Warsaw, Poland

G. Szoltyk
Forest Research Institute
Division in Sekocin,
Warsaw, Poland

J. Rojek
District Chemical Agricultural
Station
Bydgoszcz, Poland

E. Gorecka
Polish Geological Institute
Warsaw, Poland

REFERENCE

[1] Paule, R.C.; Mandel, J.; *NBS Journal of Research*; Vol. 87, pp. 377-385 (1982).

Certificate Revision History: 18 July 2003 (The description of the SRM has been updated to include that this SRM was radiation sterilized, which was previously omitted); 18 January 2002 (This revision reflects a change in the certification expiration date); 23 August 1993 (Addendum added); 30 October 1992 (Original certificate date).

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) 975-6776; fax (301) 926-4751; e-mail srminfo@nist.gov; or via the Internet http://www.nist.gov/srm.

Addendum to Certificates

SRM 2709 San Joaquin Soil
SRM 2710 Montana Soil
SRM 2711 Montana Soil

Leachable Concentrations Using U.S. EPA Method 3050 for Flame Atomic Absorption Spectrometry (FAAS) and Inductively Coupled Plasma Atomic Emission Spectrometry (ICP-AES)

The certified concentrations of constituent elements in essentially all National Institute of Standards and Technology (NIST) chemical composition Standard Reference Materials (SRMs) are given as total concentrations. The certified concentrations are based on measurements obtained by two or more independent methods or techniques. The measurement methods require complete sample decomposition, or the sample may be analyzed nondestructively. 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.

For a number of environmental monitoring purposes, the concentrations of labile or extractable fractions of elements are more useful than total concentrations. Concentrations of labile or extractable fractions are generally determined using relatively mild leach conditions, which are unlikely to totally decompose the sample. It should be noted that results obtained using the mild leach conditions are often erroneously depicted in reports as total concentrations. However, reported concentrations of labile or extractable fractions of elements are generally lower than total concentrations; recovery can be total if an element in a given sample is completely labile. Results are often presented as measured concentration in the leachate in comparison to the total or certified concentration. The recovery of an element as a percent of total concentration is a function of several factors such as the mode of occurrence in the sample, leach medium, leach time and temperature conditions, and pH of the sample-leach medium mixture. References [1] through [27] may be consulted for detailed discussions of these factors and their effect on leach results. Some of these references provide leach data for one or more reference materials.

In its monitoring programs, the U.S. Environmental Protection Agency (EPA) has established a number of leach methods for the determination of labile or extractable elements. They include Methods 3015, 3050, and 3051. A number of cooperating laboratories using the variation to U.S. EPA Method 3050 for Flame Atomic Absorption Spectrometry (FAAS) and Inductively Coupled Plasma-Atomic Emission Spectrometry (ICP-AES) measurements, have reported data for SRMs 2709, 2710, and 2711. This variation of the method uses hydrochloric acid in its final step, which is different from Method 3050 for ICP-MS and Hydride Generation-Atomic Absorption Spectrometry (HG-AAS) measurements. The data obtained are presented in Tables 1, 2, and 3 of this addendum. The names of the cooperating laboratories are listed in Table 4. Several laboratories provided replicate (3 to 6) analyses for each of the three soil SRMs. The number of results for a given element varied from only one to as many as nine, as indicated in the data presented in Tables 1 through 3. Because of the wide range of interlaboratory results for most elements, only the data range and median of the individual laboratory means are given. Ranges differ somewhat from those in reference [26], since this addendum is based on a larger data set than had been available previously.

For SRMs 2710 and 2711, 17 laboratories provided data as part of contract work for the U.S. EPA. Each SRM was treated as a blind sample in one quarter of 1992. Since there was no within-laboratory replication of analysis in the design of the exercise, the 17-laboratory means of results were treated as single laboratory results from laboratories using replication, in establishing the median of the full data set. In a few cases, however, the contract laboratories mean was the only result available for a particular element (e.g., Antimony in SRM 2710). In others, the contract laboratories mean is also the median for the full leach data set (e.g., Arsenic in SRM 2710). An asterisk identifies those cases where the contract laboratories' means are given as the median value.

Please note none of the values in Tables 1 through 3 are certified, but are given as information on the performance of the three soils when used to evaluate, or to provide quality control for Method 3050 followed by FAAS and ICP-AES measurements only. The data should not be used for any other purpose. **The certified values, provided as total concentrations, are the best estimate of the true concentrations.**

Gaithersburg, MD 20899
Addendum Issue Date: 18 July 2003

John Rumble, Jr., Chief
Measurement Services Division

Table 1. Leach Data from Cooperating Laboratories for Soil SRM 2709

Element	Range	Median	N	% Leach Recovery†
	Wt %			
Aluminum	2.0 - 3.1	2.6	5	35
Calcium	1.4 - 1.7	1.5	5	79
Iron	2.5 - 3.3	3.0	8	86
Magnesium	1.2 - 1.5	1.4	5	93
Phosphorus	0.05 - 0.07	0.07	3	100
Potassium	0.26 - 0.37	0.32	5	16
Silicon	--- ---	< 0.01	1	< 1
Sodium	0.063 - 0.11	0.068	4	6
Titanium	0.03 - 0.04	0.038	3	11
	mg/kg			
Antimony	--- ---	< 10	1	···
Arsenic	--- ---	< 20	2	···
Barium	392 - 400	398	2	41
Cadmium	--- ---	< 1	5	···
Chromium	60 - 115	79	5	61
Cobalt	10 - 15	12	5	90
Copper	26 - 40	32	7	92
Lead	12 - 18	13	5	69
Manganese	360 - 600	470	7	87
Molybdenum	--- ---	< 2	2	···
Nickel	65 - 90	78	7	89
Selenium	nr - nr	0.014	1	< 1
Strontium	100 - 112	101	3	44
Vanadium	51 - 70	62	3	55
Zinc	87 - 120	100	7	94

$$\dagger\ \%\ \text{Leach Recovery} = 100 \times \left[\frac{\text{Median Value}}{\text{Certified/Information Value}}\right]$$

--- at or below the detection limit
··· no % Leach Recovery calculated
nr no range reported by the laboratory

Table 2. Leach Data from Cooperating Laboratories for Soil SRM 2710

Element	Range	Median	N	% Leach Recovery†
	Wt %			
Aluminum	1.2 - 2.6	1.8	6	28
Calcium	0.38 - 0.48	0.41	7	33
Iron	2.2 - 3.2	2.7	9	80
Magnesium	0.43 - 0.60	0.57	6	67
Phosphorus	0.106 - 0.11	0.11	2	100
Potassium	0.37 - 0.50	0.45	6	21
Silicon	--- ---	< 0.01	1	< 1
Sodium	0.049 - 0.062	0.054	5	5
Titanium	0.092 - 0.11	0.10	3	35
	mg/kg			
Antimony	3.4 - 12	7.9*	1*	21
Arsenic	490 - 600	590	3	94
Barium	300 - 400	360	3	51
Cadmium	13 - 26	20	8	92
Chromium	15 - 23	19	6	(49)
Cobalt	6.3 - 12	8.2	7	(82)
Copper	2400 - 3400	2700	8	92
Lead	4300 - 7000	5100	8	92
Manganese	6200 - 9000	7700	8	76
Mercury	27 - 37	32*	1*	98
Molybdenum	13 - 27	20	2	(100)
Nickel	8.8 - 15	10.1	8	71
Silver	24 - 30	28	3	79
Selenium	nr - nr	0.002	1	···
Strontium	94 - 110	100	3	(42)
Thallium	0.50 - 0.76	0.63*	1*	(48)
Vanadium	37 - 50	43	4	56
Zinc	5200 - 6900	5900	9	85

$$\dagger\ \%\ \text{Leach Recovery} = 100 \times \left[\frac{\text{Median Value}}{\text{Certified/Information Value}}\right]$$

( ) indicates that information value was used
--- at or below the detection limit
··· no % Leach Recovery could be calculated
nr no range reported by the laboratory
* U.S. EPA contact laboratories mean; treated as one laboratory since no within-laboratory replication; see text

Table 3. Leach Data from Cooperating Laboratories for Soil SRM 2711

Element	Range			Median	N	% Leach Recovery†
		Wt %				
Aluminum	1.2	-	2.3	1.8	5	28
Calcium	2.0	-	2.5	2.1	5	73
Iron	1.7	-	2.6	2.2	7	76
Magnesium	0.72	-	0.89	0.81	5	77
Phosphorus	0.06	-	0.09	0.088	3	100
Potassium	0.26	-	0.53	0.38	5	16
Silicon	---		---	< 0.01	1	< 1
Sodium	0.020	-	0.029	0.026	4	2.3
Titanium	0.039	-	0.048	0.042	2	14
		mg/kg				
Antimony	···		···	< 10	1	···
Arsenic	88	-	110	90	3	86
Barium	170	-	260	200	2	28
Cadmium	32	-	46	40	6	96
Chromium	15	-	25	20	4	(43)
Cobalt	7	-	12	8.2	5	(82)
Copper	91	-	110	100	6	88
Lead	930	-	1500	1100	7	95
Manganese	400	-	620	490*	7	77
Molybdenum	---		---	< 2	2	···
Nickel	14	-	20	16	7	78
Silver	2.5	-	5.5	4.0	1	86
Selenium	nr	-	nr	0.009	1	< 1
Strontium	48	-	55	50	3	20
Vanadium	34	-	50	42	3	51
Zinc	290	-	340	310	7	89

$$\dagger\ \%\ \text{Leach Recovery} = 100 \times \left[\frac{\text{Median Value}}{\text{Certified/Information Value}}\right]$$

( ) indicates that information value was used
--- at or below the detection limit
··· no % Leach Recovery could be calculated
nr no range reported by the laboratory
* U.S. EPA contact laboratories mean; treated as one laboratory since no within-laboratory replication; see text

Table 4. Leach Study for Cooperating Laboratories

SRMs 2709, 2710, and 2711

S.A. Wilson: U.S. Geological Survey; Lakewood, CO, USA

J. Lipinski and T. Plebanski: Polish Committee for Standardization, Measures and Quality Control; Warsaw, Poland

E. Gorecka: Polish Geological Institute; Warsaw, Poland

M. Paul: Research Institute of Vegetable Crops; Skierniewice, Poland

I. Matuszczyk: Forest Research Institute; Warsaw, Poland

Z. Jonca: Institute of Environmental Protection; Warsaw, Poland

B. Ksiazek: Geological Enterprise; Warsaw, Poland

I. Twardowska: Polish Academy of Sciences, Institute of Environmental Engineering; Zabrze, Poland

SRMs 2710 and 2711

L. Butler and D. Hillman; U.S. Environmental Protection Agency, Las Vegas, NV, and 17 contract laboratories

REFERENCES

[1] Gallagher, P.H.; Walsh, T.; *Proceedings of the Royal Irish Academy*, Vol. 49B, pp. 1-17 (1943).
[2] McKeague, J.A.; Day, J.A.; *Canadian Journal of Soil Science*, Vol. 46, pp. 13-22 (1966).
[3] Arshad, M.A.; St. Arnaud, R.J.; Huang, P.M.; *Canadian Journal of Soil Science*, Vol. 52, pp. 19-26 (1972).
[4] Chao, T.T.; Zhou, L.; *Soil Science Society of America Journal*, Vol. 47, pp. 225-232 (1983).
[5] Olade, M.E.; Fletcher, K.; *Journal of Geochemical Exploration*, Vol. 3, pp. 337-344 (1974).
[6] Chao, T.T.; *Journal of Geochemical Exploration*, Vol. 20, pp. 101-135 (1984).
[7] Lepp, N.W.; Ed.; *Effect of Heavy Metal Pollution on Plants,*Vol. 1, *Effects of Trace Metals on Plant Function*; Applied Science Publishers, London (1981).
[8] Sondag, F.; *Journal of Geochemical Exploration*; Vol. 15, pp. 645-652 (1981).
[9] Federal Register 40 CFR Part 136.
[10] Binstock, D.A.; Grohse, P.M.; Gaskill, A. Jr.; Sellers, C.; Kingston, H.M.; Jassie, L.B.; J*ournal of Association of Official Analytical Chemists*; Vol. 74 (#2), pp. 360-366 (1991). (Data: SRMs 2704, 4355, 1086, 1634b on 2704).
[11] Kingston, H.M.; Walter, P.J.; *Spectroscopy*; Vol. 7, pp. 20-27 (1992).
[12] Martens, D.C.; Chesters, G.; Peterson, L.A.; *Soil Science Society of American Proceedings*; Vol. 29, pp. 411-413 (1966).
[13] Trefry, J.H.; Metz, S.; *Analytical Chemistry*, Vol. 56, pp. 745-749 (1984).
[14] Rendell, P.S.; Batley, G.E.; Cameron, J.A.; *Environmental Science and Technology*, Vol. 14, pp. 314-318 (1980).
[15] Wilson, S.A.; *Unpublished Report to NIST Standard Reference Materials Program*; (1992). (Data: SRMs 2709, 2710, 2711, and 2704).
[16] Lipinski, J.; *Unpublished Report to NIST Standard Reference Materials Program*; (1992). (Data: SRMs 2709, 2710, 2711, and 2704).
[17] Butler, L.; Hillman, D.; *Unpublished Report to NIST Standard Reference Materials Program*; Quarterly Blind Summary from CLP, (1992). (Data: SRM 2710, 17 laboratories).
[18] Kanm-iin, W.R.; Brandt, M.J.; *Spectroscopy*; Vol. 4 (#3), pp. 49-52 (1989). (Data: EPA solid QC sample).
[19] Harper, S.L.; Walling, J.F.; Holland, D.M.; Pranger, L.J.; *Analytical Chemistry*; Vol. 55 (#9), pp. 1553-1557 (1983). (Data: SRM 1648).
[20] Hewitt, A.D.; Reynolds, C.M.; *Atomic Spectroscopy*, Vol. 11 (#5), pp. 187-192 (1990). (Data: SRM 2704, RMA Soil Standard).
[21] Nieuwenliuize, J.; Poley-Vos, C.H.; van den Akker, A.H.; van DeIft, W.; *Analyst*; Vol. 116, pp. 347-351 (1991). (Data: JAFA and BCR CRMs).
[22] Hewitt, A.D.; Cragin, J.H.; *Environmental Science and Technology*; Vol. 25, pp. 985-986 (1991).
[23] Hewitt, A.D.; Cragin, J.H.; *Environmental Science and Technology*; Vol. 26, p. 1848 (1992).
[24] Hewitt, A.D.; Cragin, J.H.; *U.S. Army Toxic and Hazardous Materials Agency Report CETHATS-CR-92061* (1992).
[25] Hewitt, A.D.; Reynolds, C.M.; *U.S. Army Toxic and Hazardous Materials Agency Report CETHATS-CR-90052* (1990).
[26] Rasberry, S.D.; Kane, J.S.; *American Environmental Laboratory*; Vol. 2/93, pp. 34-35 (1993). (Data: SRMs 2709, 2710, 2711; a subset of the data reported in this addendum).
[27] Kane, J.S.; Wilson, S.D.; Lipinski, J.; Butler, L.; *American Environmental Laboratory*; Vol. 6/93, pp. 14-15 (1993).

DEPARTMENT OF COMMERCE UNITED STATES OF AMERICA

National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 2711

Montana Soil

Moderately 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. SRM 2711 is a moderately contaminated soil that was oven-dried, sieved, radiation sterilized, and blended to achieve a high degree of homogeneity. A unit of SRM 2711 consists of 50 g of the dried material.

The certified elements for SRM 2711 are given in Table 1. The values are based on measurements using one definitive method or two or more independent and reliable analytical methods. Noncertified values for a number of elements are given in Table 2 as additional information on the composition. The noncertified values should **NOT** be used for calibration or quality control. Analytical methods used for the characterization of this SRM are given in Table 3 along with analysts and cooperating laboratories. All values (except for carbon) are based on measurements using a sample weight of at least 250 mg. Carbon measurements are based on 100 mg samples.

NOTICE AND WARNINGS TO USERS

Expiration of Certification: This certification of SRM 2711 is valid, within the measurement uncertainties specified, until **31 December 2011**, provided the SRM is handled in accordance with 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. Return of the attached registration card will facilitate notification.

The overall direction and coordination of the analyses were under the chairmanship of M.S. Epstein and R.L. Watters, Jr. of the NIST Inorganic Analytical Research Division.

Statistical consultation was provided by S.B. Schiller of the NIST Statistical Engineering Division.

The technical and support aspects involved in the preparation, certification, and issuance of this SRM were coordinated through the NIST Standard Reference Materials Program by T.E. Gills and J.S. Kane. Revision of this certificate was coordinated through the NIST Standard Reference Materials Program by B.S. MacDonald of the NIST Measurement Services Division.

Willie E. May, Chief
Analytical Chemistry Division

John Rumble, Jr., Chief
Measurement Services Division

Gaithersburg, MD 20899
Certificate Issue Date: 18 July 2003
See Certificate Revision History on Page 6

INSTRUCTIONS FOR USE

Use: A minimum sample weight of 250 mg (dry weight - see *Instructions for Drying*) should be used for analytical determinations to be related to the certified values on this Certificate of Analysis.

To obtain the certified values, sample preparation procedures should be designed to effect complete dissolution. If volatile elements (i.e., mercury (Hg), arsenic (As), selenium (Se)) are to be determined, precautions should be taken in the dissolution of SRM 2711 to avoid volatilization losses.

Instructions for Drying: When nonvolatile elements are to be determined, samples should be dried for 2 h at 110 °C. Volatile elements (i.e., Hg, As, Se) 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 is to 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 approximate weight loss on drying has been found to be in the range of 1.5 % to 2.2 %.

Source and Preparation of Material: The U.S. Geological Survey (USGS), under contract to the NIST, collected and processed the material for SRM 2711. The material is an agricultural soil collected in the till layer (upper 15.2 cm (6 in)) of a wheat field. The soil from a 3.05 m × 3.05 m (10 ft × 10 ft) area was shoveled into 0.114 m^3 (3 gal) plastic pails for shipment to the USGS laboratory for processing.

The material was spread on 30.5 cm × 61 cm (1 ft × 2 ft) polyethylene-lined drying trays in an air drying oven and dried for three days at room temperature. The material was then passed over a vibrating 2 mm screen to remove plant material, rocks, and large chunks of aggregated soil. Material remaining on the screen was deaggregated and rescreened. The combined material passing the screen was ground in a ball mill to pass a 74 µm screen and blended for 24 h. Twenty grab samples were taken and measured for the major oxides using X-ray fluorescence spectrometry and for several trace elements by using inductively coupled plasma atomic emission analysis to provide preliminary assessment of the homogeneity prior to bottling. The material was bottled into 50 g units and randomly selected bottles were taken for the final homogeneity testing.

Analysis: The homogeneity, using selected elements in the bottled material as indicators, was assessed using X-ray fluorescence spectrometry and neutron activation analysis. In a few cases, statistically significant differences were observed, and the variance due to material inhomogeneity is included in the overall uncertainty of the certified values. The estimated relative standard deviation is less than 3 % for those elements for which homogeneity was assessed.

Certified Values and Uncertainties: The certified values are weighted means of results from two or more analytical methods, or the mean of results from a single definitive method, except for mercury. Mercury certification is based on cold vapor atomic absorption spectrometry used by two different laboratories employing different methods of sample preparation prior to measurement. The weights for the weighted means were computed according to the iterative procedures of Paule and Mandel [1]. The stated uncertainty includes allowances for measurement imprecision, material variability, and differences among analytical methods. Each uncertainty is the sum of the half-width of a 95 % prediction interval and includes an allowance for systematic error among the methods used. In the absence of systematic error, a 95 % prediction interval predicts where the true concentrations of 95 % of the samples of this SRM lie. The certified values were corroborated by analyses from nine Polish laboratories cooperating on the certification under the direction of T. Plebanski and J. Lipinski, Polish Committee for Standardization Measures, and Quality Control. The Polish laboratory work was supported by the Maria Sklodowska-Curie Joint Fund.

Table 1. Certified Values

Element	Mass Fraction (%)	Element	Mass Fraction (µg/g)
Aluminum	6.53 ± 0.09	Antimony	19.4 ± 1.8
Calcium	2.88 ± 0.08	Arsenic	105 ± 8
Iron	2.89 ± 0.06	Barium	726 ± 38
Magnesium	1.05 ± 0.03	Cadmium	41.70 ± 0.25
Phosphorus	0.086 ± 0.007	Copper	114 ± 2
Potassium	2.45 ± 0.08	Lead	1162 ± 31
Silicon	30.44 ± 0.19	Manganese	638 ± 28
Sodium	1.14 ± 0.03	Mercury	6.25 ± 0.19
Sulfur	0.042 ± 0.001	Nickel	20.6 ± 1.1
Titanium	0.306 ± 0.023	Selenium	1.52 ± 0.14
		Silver	4.63 ± 0.39
		Strontium	245.3 ± 0.7
		Thallium	2.47 ± 0.15
		Vanadium	81.6 ± 2.9
		Zinc	350.4 ± 4.8

Noncertified Values: Noncertified values, shown below, are provided for information only. An element concentration value may not be certified, if a bias is suspected in one or more of the methods used for certification, or if two independent methods are not available.

Table 2. Noncertified Values

Element	Mass Fraction (%)	Element	Mass Fraction (µg/g)
Carbon	2	Bromine	5
		Cerium	69
		Cesium	6.1
		Chromium	47
		Cobalt	10
		Dysprosium	5.6
		Europium	1.1
		Gallium	15
		Gold	.03
		Hafnium	7.3
		Holmium	1
		Indium	1.1
		Iodine	3
		Lanthanum	40
		Molybdenum	1.6
		Neodymium	31
		Rubidium	110
		Samarium	5.9
		Scandium	9
		Thorium	14
		Tungsten	3
		Uranium	2.6
		Ytterbium	2.7
		Yttrium	25
		Zirconium	230

Table 3. Analytical Methods Used for the Analysis of SRM 2711

Element	Certification Methods*	Element	Certification Methods*
Ag	ID ICPMS; RNAA; **INAA**	Mo	**ID ICPMS**
Al	XRF1; XRF2; INAA; DCP; **ICP**	Na	INAA; FAES
As	RNAA; HYD AAS; **INAA**	Nd	**ICP**
Au	**INAA; FAAS**	Ni	ID ICPMS; ETAAS; **INAA**
Ba	XRF2; FAES; **ICP; INAA**	P	DCP; COLOR; XRF2; **ICP**
Br	**INAA**	Pb	ID TIMS; **POLAR; ICP**
C	**COUL**	Rb	**INAA**
Ca	XRF1; XRF2; DCP; INAA; **ICP**	S	ID TIMS
Cd	ID ICPMS; RNAA	Sb	INAA; ETAAS
Ce	**INAA; ICP**	Sc	**INAA; ICP**
Co	**INAA; ETAAS; ICP**	Se	RNAA; HYD AAS; **INAA**
Cr	**INAA; DCP; ICP**	Si	XRF1; XRF2; GRAV
Cs	**INAA**	Sm	**INAA**
Cu	RNAA; FAES; **ICP**	Sr	ID TIMS; **INAA; ICP**
Dy	**INAA**	Th	**ID TIMS; INAA; ICP**
Eu	**INAA**	Ti	INAA; XRF1; XRF2; DCP
Fe	XRF1; XRF2; DCP; **INAA**	Tl	ID TIMS; LEAFS
Ga	**INAA; ICP**	U	**ID TIMS**
Hf	**INAA**	V	INAA; ICP
Hg	CVAAS	W	**INAA**
Ho	**INAA**	Y	**ICP**
I	**INAA**	Yb	**INAA; ICP**
In	**INAA**	Zn	ID TIMS; **ICP; INAA; POLAR**
K	XRF1; XRF2; FAES; **ICP; INAA**	Zr	**INAA**
La	**INAA; ICP**		
Mg	XRF1; ICP		
Mn	INAA; ICP; XRF2; **XRF1**		

*Methods in **bold** were used to corroborate certification methods or to provide information values.

COLOR	Colorimetry; lithium metaborate fusion.
COUL	Combustion coulometry.
CVAAS	Cold vapor atomic absorption spectrometry.
DCP	Direct current plasma atomic emission spectrometry; lithium metaborate fusion.
ETAAS	Electrothermal atomic absorption spectrometry; mixed acid digestion.
FAAS	Flame atomic absorption spectrometry; mixed acid digestion, except for Au, leached with $HBr-Br_2$.
FAES	Flame atomic emission spectrometry; mixed acid digestion.
GRAV	Gravimetry; sodium carbonate fusion.
HYD AAS	Hydride generation atomic absorption spectrometry.
ICP	Inductively coupled plasma atomic emission spectrometry; mixed acid digestion.
ID ICPMS	Isotope dilution inductively coupled plasma mass spectrometry; mixed acid digestion.
ID TIMS	Isotope dilution thermal ionization mass spectrometry; mixed acid digestion.
INAA	Instrumental neutron activation analysis.
LEAFS	Laser enhanced atomic fluorescence spectrometry; mixed acid digestion.
POLAR	Polarography.
RNAA	Radiochemical neutron activation analysis; mixed acid digestion.
XRF1	Wavelength dispersive X-ray fluorescence on fused borate discs.
XRF2	Wavelength dispersive X-ray fluorescence spectrometry on pressed powder.

Participating NIST Analysts:

M. Adriaens
E.S. Beary
C.A. Beck
D.S. Braverman
M.S. Epstein
J.D. Fassett
K.M. Garrity
R.R. Greenberg
W.R. Kelly
R.M. Lindstrom
E.A. Mackey
A. Marlow
J.R. Moody
P.J. Paulsen
P. Pella
T.A. Rush
J.M. Smeller
G.C. Turk
T.W. Vetter
R.D. Vocke
L.J. Wood
R.L. Watters, Jr.

Participating Laboratories:

P. Briggs, D. Siems, J. Taggart, S. Wilson
U.S. Geological Survey
Branch of Geochemistry
Denver, CO, USA

J.B. Bodkin
College of Earth and Mineral Sciences
The Pennsylvania State University
University Park, PA, USA

S.E. Landsberger, V.G. Vermette
Department of Nuclear Engineering
University of Illinois
Urbana, IL, USA

J. Lipinski, T. Plebanski
Polish Committee for Standardization,
Measures and Quality Control
Warsaw, Poland

M. Bielawska, B. Galczynska,
J. Galczynska, K. Galczynski,
K. Wiacek
Institute of Soil Science and Plant
Cultivation
Pulawy, Poland

I. Matuszczyk
Forest Research Institute
Division in Katowice,
Warsaw, Poland

Z. Jonca
Institute of Environmental
Protection
Warsaw, Poland

P. Bienkowski
Institute of Ecology
Dziekanow Lesny, Poland

H. Matusiewicz
Technical University
Poznan, Poland

B. Ksiazek
Geological Enterprise
Warsaw, Poland

G. Szoltyk
Forest Research Institute
Division in Sekocin,
Warsaw, Poland

J. Rojek
District Chemical Agricultural
Station
Bydgoszcz, Poland

E. Gorecka
Polish Geological Institute
Warsaw, Poland

REFERENCE

[1] Paule, R.C.; Mandel, J.; *NBS Journal of Research*; Vol. 87, pp. 377-385, (1982).

Certificate Revision History: 18 July 2003 (The description of the SRM has been updated to include that this SRM was radiation sterilized, which was previously omitted); 18 January 2002 (This revision reflects a change in the certification expiration date); 23 August 1993 (Addendum added); 30 October 1992 (Original certificate date).

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) 975-6776; fax (301) 926-4751; e-mail srminfo@nist.gov; or via the Internet http://www.nist.gov/srm.

Addendum to Certificates

SRM 2709 San Joaquin Soil
SRM 2710 Montana Soil
SRM 2711 Montana Soil

Leachable Concentrations Using U.S. EPA Method 3050 for Flame Atomic Absorption Spectrometry (FAAS) and Inductively Coupled Plasma Atomic Emission Spectrometry (ICP-AES)

The certified concentrations of constituent elements in essentially all National Institute of Standards and Technology (NIST) chemical composition Standard Reference Materials (SRMs) are given as total concentrations. The certified concentrations are based on measurements obtained by two or more independent methods or techniques. The measurement methods require complete sample decomposition, or the sample may be analyzed nondestructively. 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.

For a number of environmental monitoring purposes, the concentrations of labile or extractable fractions of elements are more useful than total concentrations. Concentrations of labile or extractable fractions are generally determined using relatively mild leach conditions, which are unlikely to totally decompose the sample. It should be noted that results obtained using the mild leach conditions are often erroneously depicted in reports as total concentrations. However, reported concentrations of labile or extractable fractions of elements are generally lower than total concentrations; recovery can be total if an element in a given sample is completely labile. Results are often presented as measured concentration in the leachate in comparison to the total or certified concentration. The recovery of an element as a percent of total concentration is a function of several factors such as the mode of occurrence in the sample, leach medium, leach time and temperature conditions, and pH of the sample-leach medium mixture. References [1] through [27] may be consulted for detailed discussions of these factors and their effect on leach results. Some of these references provide leach data for one or more reference materials.

In its monitoring programs, the U.S. Environmental Protection Agency (EPA) has established a number of leach methods for the determination of labile or extractable elements. They include Methods 3015, 3050, and 3051. A number of cooperating laboratories using the variation to U.S. EPA Method 3050 for Flame Atomic Absorption Spectrometry (FAAS) and Inductively Coupled Plasma-Atomic Emission Spectrometry (ICP-AES) measurements, have reported data for SRMs 2709, 2710, and 2711. This variation of the method uses hydrochloric acid in its final step, which is different from Method 3050 for ICP-MS and Hydride Generation-Atomic Absorption Spectrometry (HG-AAS) measurements. The data obtained are presented in Tables 1, 2, and 3 of this addendum. The names of the cooperating laboratories are listed in Table 4. Several laboratories provided replicate (3 to 6) analyses for each of the three soil SRMs. The number of results for a given element varied from only one to as many as nine, as indicated in the data presented in Tables 1 through 3. Because of the wide range of interlaboratory results for most elements, only the data range and median of the individual laboratory means are given. Ranges differ somewhat from those in reference [26], since this addendum is based on a larger data set than had been available previously.

For SRMs 2710 and 2711, 17 laboratories provided data as part of contract work for the U.S. EPA. Each SRM was treated as a blind sample in one quarter of 1992. Since there was no within-laboratory replication of analysis in the design of the exercise, the 17-laboratory means of results were treated as single laboratory results from laboratories using replication, in establishing the median of the full data set. In a few cases, however, the contract laboratories mean was the only result available for a particular element (e.g., Antimony in SRM 2710). In others, the contract laboratories mean is also the median for the full leach data set (e.g., Arsenic in SRM 2710). An asterisk identifies those cases where the contract laboratories' means are given as the median value.

Please note none of the values in Tables 1 through 3 are certified, but are given as information on the performance of the three soils when used to evaluate, or to provide quality control for Method 3050 followed by FAAS and ICP-AES measurements only. The data should not be used for any other purpose. **The certified values, provided as total concentrations, are the best estimate of the true concentrations.**

Gaithersburg, MD 20899 John Rumble, Jr., Chief

Table 1. Leach Data from Cooperating Laboratories for Soil SRM 2709

Element	Range	Median	N	% Leach Recovery†
	Wt %			
Aluminum	2.0 - 3.1	2.6	5	35
Calcium	1.4 - 1.7	1.5	5	79
Iron	2.5 - 3.3	3.0	8	86
Magnesium	1.2 - 1.5	1.4	5	93
Phosphorus	0.05 - 0.07	0.07	3	100
Potassium	0.26 - 0.37	0.32	5	16
Silicon	--- ---	< 0.01	1	< 1
Sodium	0.063 - 0.11	0.068	4	6
Titanium	0.03 - 0.04	0.038	3	11
	mg/kg			
Antimony	--- ---	< 10	1	...
Arsenic	--- ---	< 20	2	...
Barium	392 - 400	398	2	41
Cadmium	--- ---	< 1	5	...
Chromium	60 - 115	79	5	61
Cobalt	10 - 15	12	5	90
Copper	26 - 40	32	7	92
Lead	12 - 18	13	5	69
Manganese	360 - 600	470	7	87
Molybdenum	--- ---	< 2	2	...
Nickel	65 - 90	78	7	89
Selenium	nr - nr	0.014	1	< 1
Strontium	100 - 112	101	3	44
Vanadium	51 - 70	62	3	55
Zinc	87 - 120	100	7	94

$$\dagger\ \%\ \text{Leach Recovery} = 100 \times \left[\frac{\text{Median Value}}{\text{Certified/Information Value}}\right]$$

--- at or below the detection limit
... no % Leach Recovery calculated
nr no range reported by the laboratory

Table 2. Leach Data from Cooperating Laboratories for Soil SRM 2710

Element	Range	Median	N	% Leach Recovery†
	Wt %			
Aluminum	1.2 - 2.6	1.8	6	28
Calcium	0.38 - 0.48	0.41	7	33
Iron	2.2 - 3.2	2.7	9	80
Magnesium	0.43 - 0.60	0.57	6	67
Phosphorus	0.106 - 0.11	0.11	2	100
Potassium	0.37 - 0.50	0.45	6	21
Silicon	--- ---	< 0.01	1	< 1
Sodium	0.049 - 0.062	0.054	5	5
Titanium	0.092 - 0.11	0.10	3	35
	mg/kg			
Antimony	3.4 - 12	7.9*	1*	21
Arsenic	490 - 600	590	3	94
Barium	300 - 400	360	3	51
Cadmium	13 - 26	20	8	92
Chromium	15 - 23	19	6	(49)
Cobalt	6.3 - 12	8.2	7	(82)
Copper	2400 - 3400	2700	8	92
Lead	4300 - 7000	5100	8	92
Manganese	6200 - 9000	7700	8	76
Mercury	27 - 37	32*	1*	98
Molybdenum	13 - 27	20	2	(100)
Nickel	8.8 - 15	10.1	8	71
Silver	24 - 30	28	3	79
Selenium	nr - nr	0.002	1	...
Strontium	94 - 110	100	3	(42)
Thallium	0.50 - 0.76	0.63*	1*	(48)
Vanadium	37 - 50	43	4	56
Zinc	5200 - 6900	5900	9	85

$$\dagger\ \%\ \text{Leach Recovery} = 100 \times \left[\frac{\text{Median Value}}{\text{Certified/Information Value}}\right]$$

( ) indicates that information value was used
--- at or below the detection limit
... no % Leach Recovery could be calculated
nr no range reported by the laboratory
* U.S. EPA contact laboratories mean; treated as one laboratory since no within-laboratory replication; see text

Table 3. Leach Data from Cooperating Laboratories for Soil SRM 2711

Element	Range			Median	N	% Leach Recovery†
	Wt %					
Aluminum	1.2	-	2.3	1.8	5	28
Calcium	2.0	-	2.5	2.1	5	73
Iron	1.7	-	2.6	2.2	7	76
Magnesium	0.72	-	0.89	0.81	5	77
Phosphorus	0.06	-	0.09	0.088	3	100
Potassium	0.26	-	0.53	0.38	5	16
Silicon	---		---	< 0.01	1	< 1
Sodium	0.020	-	0.029	0.026	4	2.3
Titanium	0.039	-	0.048	0.042	2	14
	mg/kg					
Antimony	...		...	< 10	1	...
Arsenic	88	- -	110	90	3	86
Barium	170	- -	260	200	2	28
Cadmium	32	-	46	40	6	96
Chromium	15	-	25	20	4	(43)
Cobalt	7	-	12	8.2	5	(82)
Copper	91	-	110	100	6	88
Lead	930	- -	1500	1100	7	95
Manganese	400	-	620	490*	7	77
Molybdenum	---		---	< 2	2	...
Nickel	14	-	20	16	7	78
Silver	2.5	-	5.5	4.0	1	86
Selenium	nr	-	nr	0.009	1	< 1
Strontium	48	-	55	50	3	20
Vanadium	34	- -	50	42	3	51
Zinc	290	- -	340	310	7	89

$$\dagger\ \%\ \text{Leach Recovery} = 100 \times \left[\frac{\text{Median Value}}{\text{Certified/Information Value}}\right]$$

( ) indicates that information value was used
--- at or below the detection limit
... no % Leach Recovery could be calculated
nr no range reported by the laboratory
* U.S. EPA contact laboratories mean; treated as one laboratory since no within-laboratory replication; see text

Table 4. Leach Study for Cooperating Laboratories

SRMs 2709, 2710, and 2711

S.A. Wilson: U.S. Geological Survey; Lakewood, CO, USA

J. Lipinski and T. Plebanski: Polish Committee for Standardization, Measures and Quality Control; Warsaw, Poland

E. Gorecka: Polish Geological Institute; Warsaw, Poland

M. Paul: Research Institute of Vegetable Crops; Skierniewice, Poland

I. Matuszczyk: Forest Research Institute; Warsaw, Poland

Z. Jonca: Institute of Environmental Protection; Warsaw, Poland

B. Ksiazek: Geological Enterprise; Warsaw, Poland

I. Twardowska: Polish Academy of Sciences, Institute of Environmental Engineering; Zabrze, Poland

SRMs 2710 and 2711

L. Butler and D. Hillman; U.S. Environmental Protection Agency, Las Vegas, NV, and 17 contract laboratories

REFERENCES

[1] Gallagher, P.H.; Walsh, T.; *Proceedings of the Royal Irish Academy*; Vol. 49B, pp. 1-17 (1943).

[2] McKeague, J.A.; Day, J.A.; *Canadian Journal of Soil Science*; Vol. 46, pp. 13-22 (1966).

[3] Arshad, M.A.; St. Arnaud, R.J.; Huang, P.M.; *Canadian Journal of Soil Science*; Vol. 52, pp. 19-26 (1972).

[4] Chao, T.T.; Zhou, L.; *Soil Science Society of America Journal*; Vol. 47, pp. 225-232 (1983).

[5] Olade, M.E.; Fletcher, K.; *Journal of Geochemical Exploration*; Vol. 3, pp. 337-344 (1974).

[6] Chao, T.T.; *Journal of Geochemical Exploration*; Vol. 20, pp. 101-135 (1984).

[7] Lepp, N.W.; Ed.; *Effect of Heavy Metal Pollution on Plants*; Vol. 1, *Effects of Trace Metals on Plant Function*; Applied Science Publishers: London (1981).

[8] Sondag, F.; *Journal of Geochemical Exploration*; Vol. 15, pp. 645-652 (1981).

[9] Federal Register 40 CFR Part 136.

[10] Binstock, D.A.; Grohse, P.M.; Gaskill, A. Jr.; Sellers, C.; Kingston, H.M.; Jassie, L.B.; J*ournal of Association of Official Analytical Chemists*; Vol. 74 (#2), pp. 360-366 (1991). (Data: SRMs 2704, 4355, 1086, 1634b on 2704).

[11] Kingston, H.M.; Walter, P.J.; *Spectroscopy*; Vol. 7, pp. 20-27 (1992).

[12] Martens, D.C.; Chesters, G.; Peterson, L.A.; *Soil Science Society of American Proceedings*; Vol. 29, pp. 411-413 (1966).

[13] Trefry, J.H.; Metz, S.; *Analytical Chemistry*, Vol. 56, pp. 745-749 (1984).

[14] Rendell, P.S.; Batley, G.E.; Cameron, J.A.; *Environmental Science and Technology*, Vol. 14, pp. 314-318 (1980).

[15] Wilson, S.A.; *Unpublished Report to NIST Standard Reference Materials Program*; (1992). (Data: SRMs 2709, 2710, 2711, and 2704).

[16] Lipinski, J.; *Unpublished Report to NIST Standard Reference Materials Program*; (1992). (Data: SRMs 2709, 2710, 2711, and 2704).

[17] Butler, L.; Hillman, D.; *Unpublished Report to NIST Standard Reference Materials Program*; Quarterly Blind Summary from CLP, (1992). (Data: SRM 2710, 17 laboratories).

[18] Kanm-iin, W.R.; Brandt, M.J.; *Spectroscopy*; Vol. 4 (#3), pp. 49-52 (1989). (Data: EPA solid QC sample).

[19] Harper, S.L.; Walling, J.F.; Holland, D.M.; Pranger, L.J.; *Analytical Chemistry*; Vol. 55 (#9), pp. 1553-1557 (1983). (Data: SRM 1648).

[20] Hewitt, A.D.; Reynolds, C.M.; *Atomic Spectroscopy*, Vol. 11 (#5), pp. 187-192 (1990). (Data: SRM 2704, RMA Soil Standard).

[21] Nieuwenliuize, J.; Poley-Vos, C.H.; van den Akker, A.H.; van DeIft, W.; *Analyst*; Vol. 116, pp. 347-351 (1991). (Data: JAFA and BCR CRMs).

[22] Hewitt, A.D.; Cragin, J.H.; *Environmental Science and Technology*; Vol. 25, pp. 985-986 (1991).

[23] Hewitt, A.D.; Cragin, J.H.; *Environmental Science and Technology*; Vol. 26, p. 1848 (1992).

[24] Hewitt, A.D.; Cragin, J.H.; *U.S. Army Toxic and Hazardous Materials Agency Report CETHATS-CR-92061* (1992).

[25] Hewitt, A.D.; Reynolds, C.M.; *U.S. Army Toxic and Hazardous Materials Agency Report CETHATS-CR-90052* (1990).

[26] Rasberry, S.D.; Kane, J.S.; *American Environmental Laboratory*; Vol. 2/93, pp. 34-35 (1993). (Data: SRMs 2709, 2710, 2711; a subset of the data reported in this addendum).

[27] Kane, J.S.; Wilson, S.D.; Lipinski, J.; Butler, L.; *American Environmental Laboratory*; Vol. 6/93, pp. 14-15 (1993).

DEPARTMENT OF COMMERCE UNITED STATES OF AMERICA

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

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.

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].

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

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.

Table 5. Participating NIST Analysts:

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

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

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

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

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

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

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

Addendum to Certificate

Standard Reference Material® 2710a

Montana I Soil

Highly Elevated Trace Element Concentrations

Leachable Concentrations Determined Using USEPA Methods 200.7 and 3050B

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

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

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

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.

DEPARTMENT OF COMMERCE • UNITED STATES OF AMERICA

National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 2711a

Montana II Soil

Moderately 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 2711a consists of 50 g of the dried, powdered soil.

Certified Values: The certified concentrations for 25 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 eight constituents, expressed as mass fractions on a dry-mass basis, are provided in Table 2. The reference values are based on results obtained from a single NIST analytical method. 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 12 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 2711a 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: 22 May 2009

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

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 2711a 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 2711a. Soil was collected from the top 10 cm to 12 cm of an agricultural field located near a site formerly used by a smelting plant, in east Helena, Montana. Collection was performed using a common garden spade, and the material was stored in 20 plastic-lined five-gallon buckets with snap-on lids. At the USGS, the SRM 2711a 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. 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 assessment and sieving tests performed at NIST 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 analyses at NIST indicated that material homogeneity was acceptable (see below).

Analysis: The homogeneity was assessed for selected elements in the bottled material using instrumental neutron activation analysis (INAA). The estimated relative standard deviation for material inhomogeneity is ≤1 % for most elements evaluated. For antimony, magnesium, and zinc, a component for material heterogeneity (of 1 %, relative, at the 1s level) was included in the expanded uncertainties of the certified 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.

Table 1. Certified Values[a,b] (Dry-Mass Basis) for Selected Elements in SRM 2711a

Element	Mass Fraction (%)	Element	Mass Fraction (mg/kg)
Aluminum	6.72 ± 0.06	Antimony	23.8 ± 1.4
Calcium	2.42 ± 0.06	Arsenic	107 ± 5
Iron	2.82 ± 0.04	Barium	730 ± 15
Lead	0.140 ± 0.001	Cadmium	54.1 ± 0.5
Magnesium	1.07 ± 0.06	Chromium	52.3 ± 2.9
Potassium	2.53 ± 0.10	Cobalt	9.89 ± 0.18
Silicon	31.4 ± 0.7	Copper	140 ± 2
Sodium	1.20 ± 0.01	Manganese	675 ± 18
Titanium	0.317 ± 0.008	Mercury	7.42 ± 0.18
		Nickel	21.7 ± 0.7
		Phosphorous	842 ± 11
		Samarium	5.93 ± 0.28
		Strontium	242 ± 10
		Uranium	3.01 ± 0.12
		Vanadium	80.7 ± 5.7
		Zinc	414 ± 11

[a] Certified values for all elements except cadmium, 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]. A component for material heterogeneity is incorporated into the uncertainties for antimony, manganese, and zinc. 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 cadmium, 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].

Table 2. Reference Values[a] (Dry-Mass Basis) for Selected Elements in SRM 2711a

Element	Mass Fraction (mg/kg)
Cesium	6.7 ± 0.2
Europium	1.1 ± 0.2
Hafnium	9.2 ± 0.2
Lanthanum	38 ± 1
Neodymium	29 ± 2
Rubidium	120 ± 3
Scandium	8.5 ± 0.1
Thorium	15 ± 1

[a] Reference values 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].

Table 3. Information Values[(a)] (Dry-Mass Basis) for Selected Elements in SRM 2711a

Element	Mass Fraction (mg/kg)
Boron	50
Cerium	70
Dysprosium	5
Gadolinium	5
Indium	1
Lutetium	0.5
Selenium	2
Silver	6
Tantalum	1
Terbium	0.8
Thallium	3
Ytterbium	3

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

Table 4. NIST Methods Used for the Analysis of SRM 2711a

Element	Methods	Element	Methods
Ag	INAA	Mn	INAA; XRF
Al	INAA; XRF	Na	INAA; XRF
As	INAA; XRF	Nd	INAA
B	PGAA	Ni	ICP-MS; ICP-OES
Ba	ICP-OES; INAA: XRF	P	ICP-OES; XRF
Ca	INAA; XRF	Pb	ID-ICP-MS
Cd	ID-ICP-MS	Rb	INAA
Ce	INAA	Sb	ICP-MS; INAA
Co	INAA; ICP-OES	Sc	INAA
Cr	INAA; XRF	Se	CCT-ICP-MS
Cs	INAA	Si	PGAA; XRF
Cu	ICP-OES; ICP-MS	Sm	INAA(a); PGAA
Dy	INAA	Sr	ICP-OES; INAA; XRF
Eu	INAA	Ta	INAA
Fe	INAA; PGAA; XRF	Tb	INAA
Gd	PGAA	Th	INAA
Hf	INAA	Ti	INAA; PGAA; XRF
Hg	CV-ID-ICPMS	Tl	ICP-MS
In	INAA	U	ICP-MS; INAA
K	INAA; PGAA; XRF	V	INAA; XRF
La	INAA(a)	Yb	INAA
Lu	INAA	Zn	INAA; XRF
Mg	INAA; 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 samarium and lanthanum, respectively.
(b)USGS methods were used to confirm certified, reference, or information values.

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

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

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

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

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

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® 2711a

Montana II Soil

Moderately 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 2711a 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 soils 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 C. Jones, Quality Assurance and Technical Support Program (QATS), Shaw Environmental & Infrastructure Group, Las Vegas, NV, under the direction of J. Nebelsick, USEPA, Analytical Services Branch. The participating laboratories are listed in Table A2.

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

Element	n	Range (mg/kg)	Median (mg/kg)	Recovery (%)
Aluminum	6	9800 - 15000	13200	19
Antimony	6	2.8 - 7.2	4.9	21
Arsenic	6	81 - 110	89	85
Barium	6	170 - 200	190	25
Beryllium	6	0.73 - 1.1	0.93	--
Cadmium	6	43 - 56	47	90
Calcium	6	14000 - 17000	14000	61
Chromium	6	12 - 18	15	29
Cobalt	6	5.5 - 9.0	7.5	75
Copper	6	120 - 160	130	95
Iron	6	14000 - 18000	15000	54
Lead	6	1100 - 1400	1300	91
Magnesium	6	5000 - 6600	5700	54
Manganese	6	450 - 580	460	71
Mercury	6	6.3 - 8.3	7.4	100
Nickel	6	13 - 18	15	72
Potassium	6	3300 - 4600	3900	16
Selenium	5	1.4 - 1.9	1.7	85
Silver	6	4.0 - 6.1	5.5	89
Sodium	5	140 - 210	180	1.5
Thallium	5	0.71 - 3.1	2.1	68
Vanadium	6	24 - 34	28	36
Zinc	6	310 - 380	350	85

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 C

Laboratory Submitted IVBA Round Robin Sample Results

LAB A IVBA Results--Statement of Work for the Lead IVBA Round Robin Analysis of NIST SRMs 2710a and 2711a (version 4, September 30, 2010)

Introduction: The purpose of this Statement of Work (SOW) is to provide specific information and procedures for the analysis and reporting for the Lead IVBA Round Robin Analysis of the NIST SRM 2710a and 2711a. Please read carefully. The SRM analysis must be performed in strict accordance with the EPA SOP EPA 9200.1-86, which is attached. Any exceptions to the SOP procedures will be provided in this Statement of Work. **<u>We are requesting a 30 day turn-around –time for these analyses and reporting.</u>**

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 9200.1-86. **<u>It is imperative for this study that all of the required quality control samples are prepared and analyzed as specified in the SOP EPA 9200.1-86.</u>** It was also noted during the review of the IDP Forms that different laboratories use varying acceptance criteria for the quality control parameters. **<u>It is a requirement for this study</u>** that the acceptance criteria presented in the SOP EPA 9200.1-86 be used for quality control sample results. Below is a table of the required quality control samples and the control limits, which was derived from Section 9 of the SOP EPA 9200.1-86. Limits that are lower than those specified for the Reagent Blank and the Bottle Blank are acceptable. Please note that a designated duplicate sample is **<u>not</u>** required for these analyses.

QC Sample	Control Limits
Reagent blank	**<25 µg/L lead**
Bottle blank	**<50 µg/L lead**
Blank spike (10 mg/L)	**85-115% recovery**
Matrix spike (10 mg/L)	**75-125% recovery**
Duplicate sample	**±20% RPD**
Control soil (NIST 2710 or 2711)	**±10% RPD**

All quality Control Samples must be run on every batch extraction of the NIST materials. **<u>The NIST materials SRM 2710a and 2711a must each be extracted in separated batches with a complete set of quality control QC samples for each batch.</u>**

Sample Receipt: Two 30 mL Nalgene (polyethylene) wide mouth bottles will be provided to you. One bottle will contain approximately ten(10) grams of NIST SRM 2710a, and the second bottle will also contain approximately ten (10) grams of NIST SRM 2711a. A third 30 mL bottle will be provided containing approximately four (4) grams of the previous lot of NIST SRM 2711 to be used as an IVBA batch control soil. The bottles will be logged in to your usual sample receipt login system; however, these soil materials will not require refrigeration.

Sample preparation: The provided SRMs should be used <u>as is</u>. The oven drying and the sieving to less than 250µm should **<u>not</u>** be performed. Also, riffle splitting should **<u>not</u>** be performed on these SRM materials. The two (2) SRMs 2710a and 2711a, must be extracted in separate extraction batches, with five (5) replicate SRM samples for each batch, along with complete associated QC samples for each batch. To insure homogeneity, the SRM bottles **<u>must</u>** be rotated along the x, y, and z axes for at least one minute before sub-sampling for extraction. **<u>Note: All the SRM materials used in this study must be weighted out to 1.000 +/- 0.001 g, which is a more precise weighing than the SOP requirement.</u>** The extraction apparatus may have the extraction temperature controlled to 37 ± 2 °C by either air (incubator type) or water (aquarium type). For the either the incubator or aquarium type of extractor, after loading of the sample extraction bottles, wait until the apparatus medium (air or water) is within the 37 ± 2 °C temperature control range before proceeding with the 1 hour extraction time. For either incubator or aquarium type of extractor, the sample rotation speed must be 28 RPM as specified in the SOP. **Please note that the SRM samples <u>will not</u> require acid digestion and analysis by EPA Method 3050 and determination by ICP-AES.**

The batch sequences that <u>**must**</u> be used for this study for the two (2) SRMs 2710a and 2711a, are provided in Table 1 below. Again, please note that a designated duplicate sample is not required. The sample extraction will proceed as presented in the SOP.

Table 1. Extraction Batches for Round Robin Analysis of SRMs 2710a and 2711a.

Extraction Batch No. 1			Extraction Batch No. 2		
Extractor Position	**Sample Name**	**Comment**	**Extractor Position**	**Sample Name**	**Comment**
1	**SRM 2710a**		**1**	**SRM 2711a**	
2	**SRM 2710a**		**2**	**SRM 2711a**	
3	**SRM 2710a**		**3**	**SRM 2711a**	
4	**SRM 2710a**		**4**	**SRM 2711a**	
5	**SRM 2710a**		**5**	**SRM 2711a**	
6	**SRM 2710a Matrix Spike**	**10 mg/L Pb**	**6**	**SRM 2711a Matrix Spike**	**10 mg/L Pb**
7	**Bottle Blank**		**7**	**Bottle Blank**	
8	**Blank Spike**	**10 mg/L Pb**	**8**	**Blank Spike**	**10 mg/L Pb**
9	**Control Soil SRM 2711**	**(Previous Lot of SRM 2711)**	**9**	**Control Soil SRM 2711**	**(Previous Lot of SRM 2711)**

Sample Filtering and Analysis: Sample filtering and analysis should proceed as indicated in the SOP in Sections 7.7 and 7.8, respectively. The analysis will be performed using either EPA SW-846 method 6010 (ICP-AES) or 6020 (ICP-MS). However, the analytical sequence should be exactly as specified in Table 2. If one wishes, it is allowable to analyze the two (2) extraction batches in separate analytical runs; however, the QC samples associated with the extraction of a specific SRM <u>**must**</u> be analyzed in the same analytical sequence as the SRM itself.

Reporting: Tables 3 through 6 <u>**must**</u> be used for reporting the IVBA analysis results for the NIST SRMs 2710a and 2711a, and the associated QC sample results. The laboratory <u>**must**</u> provide copies of the calibration and the raw data print out from the instrumental analysis for both batches as part of the data submission.

Please complete the Results Tables 3 - 6 and e-mail to clifton.jones@shawgrp.com, followed by a 2nd day Fed-Ex mailing of the Results Tables 3-6, along with the copies of the calibration and the raw data print outs from the instrumental analysis, for both extraction batches, to the address provided below. Please provide any other pertinent information regarding the SRM extraction and analysis with the data submission.

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

Table 2. Analytical Sequence

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 6010 or 6020.	
10(<<proxy position no.)	Reagent Blank	
11	Bottle Blank	(from 2710a Extraction)
12	SRM 2710a (Replicate 1)	
13	SRM 2710a (Replicate 2)	
14	SRM 2710a (Replicate 3)	
15	SRM 2710a (Replicate 4)	
16	SRM 2710a (Replicate 5)	
17	Control Soil SRM 2711	(from 2710a Extraction)
18	Blank Spike	10 mg/L (from 2710a Extraction)
19	SRM 2710a Matrix Spike	10 mg/L (from 2710a Extraction
20	Continuing Calibration Verification Standard	
21	Continuing Calibration Verification Blank	
22	Reagent Blank	(from 2711a Extraction
23	Bottle Blank	(from 2711a Extraction)
24	SRM 2711a (Replicate 1)	
25	SRM 2711a (Replicate 2)	
26	SRM 2711a (Replicate 3)	
27	SRM 2711a (Replicate 4)	
28	SRM 2711a (Replicate 5)	
29	Control Soil SRM 2711	(from 2711a Extraction)
30	Blank Spike	10 mg/L (from 2711a Extraction)
31	SRM 2711a Matrix Spike	10 mg/L (from 2711a Extraction)
32	Continuing Calibration Verification Standard	
33	Continuing Calibration Verification Blank	
Analytical Run Closing QC Samples-	Interference Check Sample etc. as required by either EPA Methods 6010 or 6020.	

Table 3. Laboratory, Instrument, Instrumental MDL, and Extraction Batch 1 Data Reporting Form for NIST SRM 2710a

Laboratory Performing Extraction	**Lab A**			
Laboratory Performing Analysis	**Other Lab name was here.**			
Extraction Batch 1 Results NIST 2710a				
Instrument Type? (ICP-AES or ICP-MS)	ICP-AES	**Instrument Method Detection Limit (MDL) (ug/L)**	<30 µg/L	
Extraction Date	November 4, 2010			
Extraction Lead Standard Manufacturer and Lot #	Acros Organics 19607100 Lot# A0244027			
Analysis Date(s)	November 8, 2010			
Analysis Lead Standard Manufacturer and Lot #	Inorganic Ventures LOT# D2-MEB338091			
Initial Calibration Verification Standard Source and Lot #	High Purity Standards:1) CWW-TM-D LOT:0911303 2) CRM-TMDW LOT:101713			
Interference Check Sample Source and Lot #	N/A			
Sample Name	**Instrument result for the analytical solution (ug/L)**	**Dilution Factor**	**Final Instrumental result analytical solution (corrected for dilution) (ug/L)**	**Result in mg/Kg (corrected for 1g/100mL extraction)(i.e ug/L times 100/1000 = mg/kg)**
EXAMPLE SOIL (NIST 2710a)	**70**	**10**	**700**	**70**
Reagent Blank	<30	1	<30	
Bottle Blank	<30	1	<30	
SRM 2710a (Replicate 1)	33112	1	33100	3290
SRM 2710a (Replicate 2)	32821	1	32800	3270
SRM 2710a (Replicate 3)	32930	1	32900	3290
SRM 2710a (Replicate 4)	33064	1	33100	3300
SRM 2710a (Replicate 5)	32892	1	33000	3290
Control Soil SRM 2711	8645	1	8650	
Blank Spike	9512	1	9510	
SRM 2710a Matrix Spike	41279	1	41300	

Table 4. Extraction Batch 1 Spiked Blank and Spiked Sample Results for NIST SRM 2710a

Laboratory Performing Extraction	Lab A
Laboratory Performing Analysis	Other Lab name was here.
Extraction Batch 1 Spiked Blank and Spiked Sample Results for NIST SRM 2710a	
Bottle Blank Result (mg/L)	<0.03
Blank Spike Result (mg/L)	9.51
Blank Spike Percent Recovery	96.1%
Average (5) Result SRM 2710a (mg/L)	33.0
SRM 2710a Matrix Spike Result (mg/L)	41.3
SRM 2710a Matrix Spike Percent Recovery	96.0%

Table 5. Instrument, Instrumental MDL, and Extraction Batch 2 Data Reporting Form for NIST SRM 2711a

Laboratory Performing Extraction	**Lab A**			
Laboratory Performing Analysis	**Other Lab name was here.**			
Extraction Batch 2 Results NIST 2711a				
Instrument Type? (ICP-AES or ICP-MS)	ICP-AES	**Instrument Method Detection Limit (MDL) (ug/L)**	<30 µg/L	
Extraction Date	November 4, 2010			
Extraction Lead Standard Manufacturer and Lot #	Acros Organics 19607100 Lot# A0244027			
Analysis Date(s)	November 8, 2010			
Analysis Lead Standard Manufacturer and Lot #	Inorganic Ventures LOT# D2-MEB338091			
Initial Calibration Verification Standard Source and Lot #	High Purity Standards:1) CWW-TM-D LOT:0911303 2) CRM-TMDW LOT:101713			
Interference Check Sample Source and Lot #	N/A			
Sample Name	**Instrument result for the analytical solution (ug/L)**	Dilution Factor	**Final Instrumental result for analytical solution (corrected for dilution) (ug/L)**	**Result in mg/Kg (correct for 1 g/100ml extraction)(i.e. ug/L times 100/1000 = mg/kg)**
EXAMPLE SOIL (NIST 2711a)	**70**	**10**	**700**	**70**
Reagent Blank	<30	1	<30	
Bottle Blank	<30	1	<30	
SRM 2711a (Replicate 1)	10375	1	10400	1040
SRM 2711a (Replicate 2)	10373	1	10400	1030
SRM 2711a (Replicate 3)	10369	1	10400	1040
SRM 2711a (Replicate 4)	10286	1	10300	1030
SRM 2711a (Replicate 5)	10313	1	10300	1030
Control Soil SRM 2711	8610	1	8611	
Blank Spike	9479	1	9480	
SRM 2711a Matrix Spike	18883	1	18900	

Table 6. Extraction Batch 2 Spiked Blank and Spiked Sample Results for NIST SRM 2711a

Laboratory Performing Extraction	**Lab A**
Laboratory Performing Analysis	**Other Lab name was here.**
Extraction Batch 2 Spiked Blank and Spiked Sample Results for NIST SRM 2711a	
Bottle Blank Result (mg/L)	<0.3
Blank Spike Result (mg/L)	9.48
Blank Spike Percent Recovery	95.7%
Average (5) Result SRM 2711a (mg/L)	10.4
SRM 2711a Matrix Spike Result (mg/L)	18.9
SRM 2711a Matrix Spike Percent Recovery	93.6%

LAB B IVBA Results - Statement of Work for the Lead IVBA Round Robin Analysis of NIST SRMs 2710a and 2711a (version 4, September 30, 2010)

Introduction: The purpose of this Statement of Work (SOW) is to provide specific information and procedures for the analysis and reporting for the Lead IVBA Round Robin Analysis of the NIST SRM 2710a and 2711a. Please read carefully. The SRM analysis must be performed in strict accordance with the EPA SOP EPA 9200.1-86, which is attached. Any exceptions to the SOP procedures will be provided in this Statement of Work. **<u>We are requesting a 30 day turn-around –time for these analyses and reporting.</u>**

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 9200.1-86. **<u>It is imperative for this study that all of the required quality control samples are prepared and analyzed as specified in the SOP EPA 9200.1-86.</u>** It was also noted during the review of the IDP Forms that different laboratories use varying acceptance criteria for the quality control parameters. **<u>It is a requirement for this study</u>** that the acceptance criteria presented in the SOP EPA 9200.1-86 be used for quality control sample results. Below is a table of the required quality control samples and the control limits, which was derived from Section 9 of the SOP EPA 9200.1-86. Limits that are lower than those specified for the Reagent Blank and the Bottle Blank are acceptable. Please note that a designated duplicate sample is **<u>not</u>** required for these analyses.

QC Sample	Control Limits
Reagent blank	**<25 µg/L lead**
Bottle blank	**<50 µg/L lead**
Blank spike (10 mg/L)	**85-115% recovery**
Matrix spike (10 mg/L)	**75-125% recovery**
Duplicate sample	**±20% RPD**
Control soil (NIST 2710 or 2711)	**±10% RPD**

All quality Control Samples must be run on every batch extraction of the NIST materials. **<u>The NIST materials SRM 2710a and 2711a must each be extracted in separated batches with a complete set of quality control QC samples for each batch.</u>**

Sample Receipt: Two 30 mL Nalgene (polyethylene) wide mouth bottles will be provided to you. One bottle will contain approximately ten(10) grams of NIST SRM 2710a, and the second bottle will also contain approximately ten (10) grams of NIST SRM 2711a. A third 30 mL bottle will be provided containing approximately four (4) grams of the previous lot of NIST SRM 2711 to be used as an IVBA batch control soil. The bottles will be logged in to your usual sample receipt login system; however, these soil materials will not require refrigeration.

Sample preparation: The provided SRMs should be used <u>as is</u>. The oven drying and the sieving to less than 250µm should **<u>not</u>** be performed. Also, riffle splitting should **<u>not</u>** be performed on these SRM materials. The two (2) SRMs 2710a and 2711a, must be extracted in separate extraction batches, with five (5) replicate SRM samples for each batch, along with complete associated QC samples for each batch. To insure homogeneity, the SRM bottles **<u>must</u>** be rotated along the x, y, and z axes for at least one minute before sub-sampling for extraction. **<u>Note: All the SRM materials used in this study must be weighted out to 1.000 +/- 0.001 g, which is a more precise weighing than the SOP requirement.</u>** The extraction apparatus may have the extraction temperature controlled to 37 ± 2 ^{O}C by either air (incubator type) or water (aquarium type). For the either the incubator or aquarium type of extractor, after loading of the sample extraction bottles, wait until the apparatus medium (air or water) is within the 37 ± 2 ^{O}C temperature control range before proceeding with the 1 hour extraction time. For either incubator or aquarium type of extractor, the sample rotation speed must be 28 RPM as specified in the SOP. **Please note that the SRM samples <u>will not</u> require acid digestion and analysis by EPA Method 3050 and determination by ICP-AES.**

The batch sequences that **<u>must</u>** be used for this study for the two (2) SRMs 2710a and 2711a, are provided in Table 1 below. Again, please note that a designated duplicate sample is not required. The sample extraction will proceed as presented in the SOP.

Table 1. Extraction Batches for Round Robin Analysis of SRMs 2710a and 2711a.

Extraction Batch No. 1			Extraction Batch No. 2		
Extractor Position	**Sample Name**	**Comment**	**Extractor Position**	**Sample Name**	**Comment**
1	SRM 2710a		1	SRM 2711a	
2	SRM 2710a		2	SRM 2711a	
3	SRM 2710a		3	SRM 2711a	
4	SRM 2710a		4	SRM 2711a	
5	SRM 2710a		5	SRM 2711a	
6	SRM 2710a Matrix Spike	10 mg/L Pb	6	SRM 2711a Matrix Spike	10 mg/L Pb
7	Bottle Blank		7	Bottle Blank	
8	Blank Spike	10 mg/L Pb	8	Blank Spike	10 mg/L Pb
9	Control Soil SRM 2711	(Previous Lot of SRM 2711)	9	Control Soil SRM 2711	(Previous Lot of SRM 2711)

Sample Filtering and Analysis: Sample filtering and analysis should proceed as indicated in the SOP in Sections 7.7 and 7.8, respectively. The analysis will be performed using either EPA SW-846 method 6010 (ICP-AES) or 6020 (ICP-MS). However, the analytical sequence should be exactly as specified in Table 2. If one wishes, it is allowable to analyze the two (2) extraction batches in separate analytical runs; however, the QC samples associated with the extraction of a specific SRM **<u>must</u>** be analyzed in the same analytical sequence as the SRM itself.

Reporting: Tables 3 through 6 **<u>must</u>** be used for reporting the IVBA analysis results for the NIST SRMs 2710a and 2711a, and the associated QC sample results. The laboratory **<u>must</u>** provide copies of the calibration and the raw data print out from the instrumental analysis for both batches as part of the data submission.

Please complete the Results Tables 3 - 6 and e-mail to clifton.jones@shawgrp.com, followed by a 2nd day Fed-Ex mailing of the Results Tables 3-6, along with the copies of the calibration and the raw data print outs from the instrumental analysis, for both extraction batches, to the address provided below. Please provide any other pertinent information regarding the SRM extraction and analysis with the data submission.

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

Table 2. Analytical Sequence

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 6010 or 6020.	
10(<<proxy position no.)	Reagent Blank	
11	Bottle Blank	(from 2710a Extraction)
12	SRM 2710a (Replicate 1)	
13	SRM 2710a (Replicate 2)	
14	SRM 2710a (Replicate 3)	
15	SRM 2710a (Replicate 4)	
16	SRM 2710a (Replicate 5)	
17	Control Soil SRM 2711	(from 2710a Extraction)
18	Blank Spike	10 mg/L (from 2710a Extraction)
19	SRM 2710a Matrix Spike	10 mg/L (from 2710a Extraction
20	Continuing Calibration Verification Standard	
21	Continuing Calibration Verification Blank	
22	Reagent Blank	(from 2711a Extraction
23	Bottle Blank	(from 2711a Extraction)
24	SRM 2711a (Replicate 1)	
25	SRM 2711a (Replicate 2)	
26	SRM 2711a (Replicate 3)	
27	SRM 2711a (Replicate 4)	
28	SRM 2711a (Replicate 5)	
29	Control Soil SRM 2711	(from 2711a Extraction)
30	Blank Spike	10 mg/L (from 2711a Extraction)
31	SRM 2711a Matrix Spike	10 mg/L (from 2711a Extraction)
32	Continuing Calibration Verification Standard	
33	Continuing Calibration Verification Blank	
Analytical Run Closing QC Samples-	Interference Check Sample etc. as required by either EPA Methods 6010 or 6020.	

Table 3. Laboratory, Instrument, Instrumental MDL, and Extraction Batch 1 Data Reporting Form for NIST SRM 2710a

Laboratory Performing Extraction	Lab B			
Laboratory Performing Analysis	Other lab name was here.			
Extraction Batch 1 Results NIST 2710a				
Instrument Type? (ICP-AES or ICP-MS)	ICP-MS	**Instrument Method Detection Limit (MDL) (ug/L)**	209	
Extraction Date				
Extraction Lead Standard Manufacturer and Lot #				
Analysis Date(s)	11/02/2010			
Analysis Lead Standard Manufacturer and Lot #		Inorganic Ventures	Lot D2-MEB332136	
Initial Calibration Verification Standard Source and Lot #		Accustandard	Lot 210065064	
Interference Check Sample Source and Lot #		Inorganic Ventures CPI Int	Lot X-CICP15096 Lot 10J002	
Sample Name	**Instrument result for the analytical solution (ug/L)**	**Dilution Factor**	**Final Instrumental result analytical solution (corrected for dilution) (ug/L)**	**Result in mg/Kg (corrected for 1g/100mL extraction)(i.e ug/L times 100/1000 = mg/kg)**
EXAMPLE SOIL (NIST 2710a)	70	10	700	70
Reagent Blank			0	0
Bottle Blank			0	0
SRM 2710a (Replicate 1)			35,200	3,520
SRM 2710a (Replicate 2)			34,700	3,470
SRM 2710a (Replicate 3)			34,825	3,483
SRM 2710a (Replicate 4)			34,785	3,479
SRM 2710a (Replicate 5)			35,375	3,538
Control Soil SRM 2711			9,525	953
Blank Spike			9,865	987
SRM 2710a Matrix Spike			43,880	4,388

Table 4. Extraction Batch 1 Spiked Blank and Spiked Sample Results for NIST SRM 2710a

Laboratory Performing Extraction	**Lab B**
Laboratory Performing Analysis	**Other lab name was here**
Extraction Batch 1 Spiked Blank and Spiked Sample Results for NIST SRM 2710a	
Bottle Blank Result (mg/L)	0
Blank Spike Result (mg/L)	9.86
Blank Spike Percent Recovery	98.6
Average (5) Result SRM 2710a (mg/L)	35.0
SRM 2710a Matrix Spike Result (mg/L)	43.9
SRM 2710a Matrix Spike Percent Recovery	89.2

Table 5. Instrument, Instrumental MDL, and Extraction Batch 2 Data Reporting Form for NIST SRM 2711a

Laboratory Performing Extraction	LAB B			
Laboratory Performing Analysis	Other lab name was here			
Extraction Batch 2 Results NIST 2711a				
Instrument Type? (ICP-AES or ICP-MS)	ICP-MS	**Instrument Method Detection Limit (MDL) (ug/L)**	209	
Extraction Date				
Extraction Lead Standard Manufacturer and Lot #				
Analysis Date(s)	11/02/2010			
Analysis Lead Standard Manufacturer and Lot #		Inorganic Ventures CPI Int	Lot D2-MEB332136	
Initial Calibration Verification Standard Source and Lot #		Accustandard	Lot 210065064	
Interference Check Sample Source and Lot #		Inorganic Ventures CPI Int	Lot X-CICP15096 Lot 10J002	
Sample Name	**Instrument result for the analytical solution (ug/L)**	**Dilution Factor**	**Final Instrumental result for analytical solution (corrected for dilution) (ug/L)**	**Result in mg/Kg (correct for 1 g/100ml extraction)(i.e. ug/L times 100/1000 = mg/kg)**
EXAMPLE SOIL (NIST 2711a)	70	10	700	70
Reagent Blank			0	0
Bottle Blank			0	0
SRM 2711a (Replicate 1)			11,445	1,145
SRM 2711a (Replicate 2)			11,465	1,147
SRM 2711a (Replicate 3)			11,215	1,122
SRM 2711a (Replicate 4)			11,565	1,157
SRM 2711a (Replicate 5)			11,645	1,165
Control Soil SRM 2711			9,665	967
Blank Spike			9,660	966
SRM 2711a Matrix Spike			20,985	2,099

Table 6. Extraction Batch 2 Spiked Blank and Spiked Sample Results for NIST SRM 2711a

Laboratory Performing Extraction	**Lab B**
Laboratory Performing Analysis	**Other lab name was here**
Extraction Batch 2 Spiked Blank and Spiked Sample Results for NIST SRM 2711a	
Bottle Blank Result (mg/L)	0
Blank Spike Result (mg/L)	9.66
Blank Spike Percent Recovery	96.6
Average (5) Result SRM 2711a (mg/L)	11.5
SRM 2711a Matrix Spike Result (mg/L)	21.0
SRM 2711a Matrix Spike Percent Recovery	95.4

Table 3. Laboratory, Instrument, Instrumental MDL, and Extraction Batch 1 Data Reporting Form for NIST SRM 2710a

Laboratory Performing Extraction	LAB C			
Laboratory Performing Analysis	LAB C			
Extraction Batch 1 Results NIST 2710a				
Instrument Type? (ICP-AES or ICP-MS)	ICP	**Instrument Method Detection Limit (MDL) (ug/L)**	40 ug/L	
Extraction Date	11/02/10			
Extraction Lead Standard Manufacturer and Lot #	ULTRA	Lot# K00652		
Analysis Date(s)	11/04/10			
Analysis Lead Standard Manufacturer and Lot #	ULTRA	Lot# K00652		
Initial Calibration Verification Standard Source and Lot #	SPEX	Lot # 25-16JB		
Interference Check Sample Source and Lot #	CPI	Lot # 10C244		
Sample Name	**Instrument result for the analytical solution (ug/L)**	**Dilution Factor**	**Final Instrumental result analytical solution (corrected for dilution) (ug/L)**	**Result in mg/Kg (corrected for 1g/100mL extraction)(i.e ug/L times 100/1000 = mg/kg)**
EXAMPLE SOIL (NIST 2710a)	70	10	700	70
Reagent Blank	<40	1	<40	<4
Bottle Blank	<40	1	<40	<4
SRM 2710a (Replicate 1)	33,200	1	33,200	3320
SRM 2710a (Replicate 2)	33,000	1	33,000	3300
SRM 2710a (Replicate 3)	33,600	1	33,600	3360
SRM 2710a (Replicate 4)	33,300	1	33,300	3330
SRM 2710a (Replicate 5)	33,700	1	33,700	3370
Control Soil SRM 2711	9120	1	9120	910
Blank Spike	9584	1	9584	960
SRM 2710a Matrix Spike	42895	1	42895	4290

Table 4. Extraction Batch 1 Spiked Blank and Spiked Sample Results for NIST SRM 2710a

Laboratory Performing Extraction	**LAB C**
Laboratory Performing Analysis	**LAB C**
Extraction Batch 1 Spiked Blank and Spiked Sample Results for NIST SRM 2710a	
Bottle Blank Result (mg/L)	<0.04
Blank Spike Result (mg/L)	9.64
Blank Spike Percent Recovery	96.3%
Average (5) Result SRM 2710a (mg/L)	33.4
SRM 2710a Matrix Spike Result (mg/L)	42.9
SRM 2710a Matrix Spike Percent Recovery	96.9%

Table 5. Instrument, Instrumental MDL, and Extraction Batch 2 Data Reporting Form for NIST SRM 2711a

Laboratory Performing Extraction	**LAB C**			
Laboratory Performing Analysis	**LAB C**			
Extraction Batch 2 Results NIST 2711a				
Instrument Type? (ICP-AES or ICP-MS)	**ICP**	**Instrument Method Detection Limit (MDL) (ug/L)**	**40 ug/L**	
Extraction Date	**11/03/10**			
Extraction Lead Standard Manufacturer and Lot #	**ULTRA Lot #K00652**			
Analysis Date(s)	**11/04/10**			
Analysis Lead Standard Manufacturer and Lot #	**ULTRA**	**Lot# K00652**		
Initial Calibration Verification Standard Source and Lot #	**SPEX**	**Lot # 25-16JB**		
Interference Check Sample Source and Lot #	**CPI**	**Lot # 10C244**		
Sample Name	**Instrument result for the analytical solution (ug/L)**	**Dilution Factor**	**Final Instrumental result for analytical solution (corrected for dilution) (ug/L)**	**Result in mg/Kg (correct for 1 g/100ml extraction)(i.e. ug/L times 100/1000 = mg/kg)**
EXAMPLE SOIL (NIST 2711a)	**70**	**10**	**700**	**70**
Reagent Blank	**<40**	**1**	**<40**	**<4**
Bottle Blank	**<40**	**1**	**<40**	**<4**
SRM 2711a (Replicate 1)	**10,800**	**1**	**10,800**	**1080**
SRM 2711a (Replicate 2)	**11,000**	**1**	**11,000**	**1100**
SRM 2711a (Replicate 3)	**10,800**	**1**	**10,800**	**1080**
SRM 2711a (Replicate 4)	**10,800**	**1**	**10,800**	**1080**
SRM 2711a (Replicate 5)	**10,600**	**1**	**10,600**	**1060**
Control Soil SRM 2711	**8990**	**1**	**8990**	**900**
Blank Spike	**9644**	**1**	**9644**	**960**
SRM 2711a Matrix Spike	**19,076**	**1**	**19,076**	**1910**

Table 6. Extraction Batch 2 Spiked Blank and Spiked Sample Results for NIST SRM 2711a

Laboratory Performing Extraction	**LAB C**
Laboratory Performing Analysis	**LAB C**
Extraction Batch 2 Spiked Blank and Spiked Sample Results for NIST SRM 2711a	
Bottle Blank Result (mg/L)	<0.04
Blank Spike Result (mg/L)	9.58
Blank Spike Percent Recovery	95.7%
Average (5) Result SRM 2711a (mg/L)	10.8
SRM 2711a Matrix Spike Result (mg/L)	19.1
SRM 2711a Matrix Spike Percent Recovery	82.7%

Table 3. Laboratory, Instrument, Instrumental MDL, and Extraction Batch 1 Data Reporting Form for NIST SRM 2710a

Laboratory Performing Extraction	**LAB D**			
Laboratory Performing Analysis	**LAB D**			
Extraction Batch 1 Results NIST 2710a				
Instrument Type? (ICP-AES or ICP-MS)	**ICP-MS**	**Instrument Method Detection Limit (MDL) (ug/L)**	**.019**	
Extraction Date	**1-5-11**			
Extraction Lead Standard Manufacturer and Lot #	**SPEX CERTIPREP**	**11-116PB**		
Analysis Date(s)	**1-6-11**			
Analysis Lead Standard Manufacturer and Lot #	**SPEX CERTIPREP**	**11-116PB**		
Initial Calibration Verification Standard Source and Lot #	**SPEX CERTIPREP**	**15-120JB**		
Interference Check Sample Source and Lot #	**ENVIRONMENTAL EXPRESS**	**0929914**		
Sample Name	**Instrument result for the analytical solution (ug/L)**	**Dilution Factor**	**Final Instrumental result analytical solution (corrected for dilution) (ug/L)**	**Result in mg/Kg (corrected for 1g/100mL extraction)(i.e ug/L times 100/1000 = mg/kg)**
EXAMPLE SOIL (NIST 2710a)	**70**	**10**	**700**	**70**
Reagent Blank	DL	**50**	DL	**DL**
Bottle Blank	DL	**50**	DL	**DL**
SRM 2710a (Replicate 1)	713.503	**50**	35675	**3567.5**
SRM 2710a (Replicate 2)	718.5222	**50**	35926	**3592.6**
SRM 2710a (Replicate 3)	699.1193	**50**	34956	**3495.6**
SRM 2710a (Replicate 4)	707.2377	**50**	35362	**3536.2**
SRM 2710a (Replicate 5)	723.4018	**50**	36170	**3617.0**
Control Soil SRM 2711	195.5592	**50**	9778	**977.8**
Blank Spike	198.5753	**50**	9929	**992.9**
SRM 2710a Matrix Spike	909.96	**50**	45498	**4549.8**

Table 4. Extraction Batch 1 Spiked Blank and Spiked Sample Results for NIST SRM 2710a

Laboratory Performing Extraction	**LAB D**
Laboratory Performing Analysis	**LAB D**
Extraction Batch 1 Spiked Blank and Spiked Sample Results for NIST SRM 2710a	
Bottle Blank Result (mg/L)	0
Blank Spike Result (mg/L)	9.929 edit to mg/L CLJ
Blank Spike Percent Recovery	99
Average (5) Result SRM 2710a (mg/L)	35.618 edit to mg/L CLJ
SRM 2710a Matrix Spike Result (mg/L)	45.498 edit to mg/L CLJ
SRM 2710a Matrix Spike Percent Recovery	99

Table 5. Instrument, Instrumental MDL, and Extraction Batch 2 Data Reporting Form for NIST SRM 2711a

Laboratory Performing Extraction	**LAB D**			
Laboratory Performing Analysis	**LAB D**			
Extraction Batch 2 Results NIST 2711a				
Instrument Type? (ICP-AES or ICP-MS)	**ICP-MS**	**Instrument Method Detection Limit (MDL) (ug/L)**	**.019**	
Extraction Date	**1-5-11**			
Extraction Lead Standard Manufacturer and Lot #	**SPEX CERTIPREP**	**11-116PB**		
Analysis Date(s)	**1-6-11**			
Analysis Lead Standard Manufacturer and Lot #	**SPEX CERTIPREP**	**11-116PB**		
Initial Calibration Verification Standard Source and Lot #	**SPEX CERTIPREP**	**15-120JB**		
Interference Check Sample Source and Lot #	**ENVIRONMENTAL EXPRESS**	**0929914**		
Sample Name	**Instrument result for the analytical solution (ug/L)**	**Dilution Factor**	**Final Instrumental result for analytical solution (corrected for dilution) (ug/L)**	**Result in mg/Kg (correct for 1 g/100ml extraction)(i.e. ug/L times 100/1000 = mg/kg)**
EXAMPLE SOIL (NIST 2711a)	**70**	**10**	**700**	**70**
Reagent Blank	**DL**	**50**	DL	DL
Bottle Blank	**DL**	**50**	DL	DL
SRM 2711a (Replicate 1)	227.6558	**50**	11383	1138.3
SRM 2711a (Replicate 2)	224.2559	**50**	11213	1121.3
SRM 2711a (Replicate 3)	231.0153	**50**	11551	1155.1
SRM 2711a (Replicate 4)	230.1573	**50**	11508	1150.8
SRM 2711a (Replicate 5)	230.2234	**50**	11511	1151.1
Control Soil SRM 2711	191.7683	**50**	9588	958.8
Blank Spike	189.4788	**50**	9474	947.4
SRM 2711a Matrix Spike	414.0249	**50**	20701	2070.1

Table 6. Extraction Batch 2 Spiked Blank and Spiked Sample Results for NIST SRM 2711a

Laboratory Performing Extraction	**LAB D**
Laboratory Performing Analysis	**LAB D**
Extraction Batch 2 Spiked Blank and Spiked Sample Results for NIST SRM 2711a	
Bottle Blank Result (mg/L)	0
Blank Spike Result (mg/L)	9.474 edit to mg/L CLJ
Blank Spike Percent Recovery	95
Average (5) Result SRM 2711a (mg/L)	11.433 edit to mg/L CLJ
SRM 2711a Matrix Spike Result (mg/L)	20.701 edit to mg/L CLJ
SRM 2711a Matrix Spike Percent Recovery	93

Table 3. Laboratory, Instrument, Instrumental MDL, and Extraction Batch 1 Data Reporting Form for NIST SRM 2710a

Laboratory Performing Extraction	LAB E			
Laboratory Performing Analysis	LAB E			
Extraction Batch 1 Results NIST 2710a				
Instrument Type? (ICP-AES or ICP-MS)	ICP-MS	Instrument Method Detection Limit (MDL) (ug/L)	MDL calculated using 40 CRF Part 136, Appendix B (99% CL) = 0.055 ug/L in glycine matrix	
Extraction Date	11/4/10			
Extraction Lead Standard Manufacturer and Lot #	VHG 901-0099			
Analysis Date(s)	11/8/10			
Analysis Lead Standard Manufacturer and Lot #	VHG 00-0019			
Initial Calibration Verification Standard Source and Lot #	VHG 911-0014			
Interference Check Sample Source and Lot #	VHG 6020a ICS Stock Lot 001-0045 and VHG 6020a ICSAB Stock Lot 911-0015			
Sample Name	Instrument result for the analytical solution (ug/L)	Dilution Factor	Final Instrumental result analytical solution (corrected for dilution) (ug/L)	Result in mg/Kg (corrected for 1g/100mL extraction)(i.e ug/L times 100/1000 = mg/kg)
EXAMPLE SOIL (NIST 2710a)	70	10	700	70
Reagent Blank	0.198	9.81	1.94	NA
Bottle Blank	0.186	9.80	1.82	NA
SRM 2710a (Replicate 1)	183.9	198.8	36553.8	3652.5
SRM 2710a (Replicate 2)	182.2	198.9	36241.5	3623.4
SRM 2710a (Replicate 3)	184.6	198.4	36624.3	3663.2
SRM 2710a (Replicate 4)	182.2	199.4	36336.4	3632.6
SRM 2710a (Replicate 5)	182.2	198.0	36070.6	3605.6
Control Soil SRM 2711	50.9	197.9	10073.6	1007.2
Blank Spike	101.4	98.59	9997.0	NA
SRM 2710a Matrix Spike	204.3	197.7	40397.4	4038.9

Table 4. Extraction Batch 1 Spiked Blank and Spiked Sample Results for NIST SRM 2710a

Laboratory Performing Extraction	**LAB E**
Laboratory Performing Analysis	**LAB E**
Extraction Batch 1 Spiked Blank and Spiked Sample Results for NIST SRM 2710a	
Bottle Blank Result (mg/L)	0.0018
Blank Spike Result (mg/L)	10.0
Blank Spike Percent Recovery	100.0%
Average (5) Result SRM 2710a (mg/L)	36.35
SRM 2710a Matrix Spike Result (mg/L)	40.40
SRM 2710a Matrix Spike Percent Recovery	83.6%

Table 5. Instrument, Instrumental MDL, and Extraction Batch 2 Data Reporting Form for NIST SRM 2711a

Laboratory Performing Extraction	LAB E			
Laboratory Performing Analysis	LAB E			
Extraction Batch 2 Results NIST 2711a				
Instrument Type? (ICP-AES or ICP-MS)	ICP-MS	Instrument Method Detection Limit (MDL) (ug/L)	MDL calculated using 40 CRF Part 136, Appendix B (99% CL) = 0.055 ug/L in glycine matrix	
Extraction Date	11/4/10			
Extraction Lead Standard Manufacturer and Lot #	VHG 901-0099			
Analysis Date(s)	11/8/10			
Analysis Lead Standard Manufacturer and Lot #	VHG 00-0019			
Initial Calibration Verification Standard Source and Lot #	VHG 911-0014			
Interference Check Sample Source and Lot #	VHG 6020a ICS Stock Lot 001-0045 and VHG 6020a ICSAB Stock Lot 911-0015			
Sample Name	Instrument result for the analytical solution (ug/L)	Dilution Factor	Final Instrumental result for analytical solution (corrected for dilution) (ug/L)	Result in mg/Kg (correct for 1 g/100ml extraction)(i.e. ug/L times 100/1000 = mg/kg)
EXAMPLE SOIL (NIST 2711a)	70	10	700	70
Reagent Blank	0.173	9.80	1.70	NA
Bottle Blank	0.145	9.80	1.42	NA
SRM 2711a (Replicate 1)	59.80	197.7	11824.1	1181.7
SRM 2711a (Replicate 2)	60.45	197.6	11947.5	1194.2
SRM 2711a (Replicate 3)	59.71	197.3	11779.6	1177.6
SRM 2711a (Replicate 4)	60.26	196.2	11823.3	1182.2
SRM 2711a (Replicate 5)	60.12	198.1	11908.0	1190.8
Control Soil SRM 2711	104.1	97.46	10145.1	1014.0
Blank Spike	99.96	98.66	9861.9	NA
SRM 2711a Matrix Spike	116.3	196.9	22896.0	2290.1

Table 6. Extraction Batch 2 Spiked Blank and Spiked Sample Results for NIST SRM 2711a

Laboratory Performing Extraction	**LAB E**
Laboratory Performing Analysis	**LAB E**
Extraction Batch 2 Spiked Blank and Spiked Sample Results for NIST SRM 2711a	
Bottle Blank Result (mg/L)	0.0014
Blank Spike Result (mg/L)	9.86
Blank Spike Percent Recovery	98.6%
Average (5) Result SRM 2711a (mg/L)	11.86
SRM 2711a Matrix Spike Result (mg/L)	22.90
SRM 2711a Matrix Spike Percent Recovery	108.8%

Table 3. Laboratory, Instrument, Instrumental MDL, and Extraction Batch 1 Data Reporting Form for NIST SRM 2710a

Laboratory Performing Extraction	LAB F			
Laboratory Performing Analysis	LAB F			
Extraction Batch 1 Results NIST 2710a				
Instrument Type? (ICP-AES or ICP-MS)	AES		Instrument Method Detection Limit (MDL) (ug/L)	5.2
Extraction Date	11/08/2010			
Extraction Lead Standard Manufacturer and Lot #	Lot# 12-50PB	SPEX Certiprep 1,000 mg/L Pb Std used for Blk and Matrix Spike		
Analysis Date(s)	11/15/2010			
Analysis Lead Standard Manufacturer and Lot #	Lot# 12-50PB	Same SPEX Certiprep Std as extraction		
Initial Calibration Verification Standard Source and Lot #	Lot# 41-151AS	SPEX Certiprep LPC std 1.20mg/L Pb.	ICV, CCV prepared by diluting std into 0.4m Glycine to match calibration and sample matrix	
Interference Check Sample Source and Lot #	Lot# 37-29AS	SPEX Certiprep 5,000mg/L Al, Ca, Mg: 2,000mg/L Fe	Prepared by x10 dilution into 0.4m Glycine and spiked with 10mg/L Pb	
Sample Name	Instrument result for the analytical solution (mg/L)	Dilution Factor	Final Instrumental result analytical solution (corrected for dilution) (ug/L)	Result in mg/Kg (corrected for 1g/100mL extraction)(i.e ug/L times 100/1000 = mg/kg)
EXAMPLE SOIL (NIST 2710a)	70	10	700	70
Reagent Blank	0.002669	1	0.002669	
Bottle Blank	NA/Incubator			
SRM 2710a (Replicate 1)	33.72	1	33.72	3372
SRM 2710a (Replicate 2)	33.14	1	33.14	3314
SRM 2710a (Replicate 3)	33.21	1	33.21	3321
SRM 2710a (Replicate 4)	33.47	1	33.47	3347
SRM 2710a (Replicate 5)	33.48	1	33.48	3348
Control Soil SRM 2711	9.066	1	9.066	906.6
Blank Spike	9.748	1	9.748	974.8
SRM 2710a Matrix Spike	41.30	1	41.30	4130

Table 4. Extraction Batch 1 Spiked Blank and Spiked Sample Results for NIST SRM 2710a

Laboratory Performing Extraction	**LAB F**
Laboratory Performing Analysis	**LAB F**
Extraction Batch 1 Spiked Blank and Spiked Sample Results for NIST SRM 2710a	
Bottle Blank Result (mg/L)	NA
Blank Spike Result (mg/L)	9.748
Blank Spike Percent Recovery	97
Average (5) Result SRM 2710a (mg/L)	33.40
SRM 2710a Matrix Spike Result (mg/L)	41.30
SRM 2710a Matrix Spike Percent Recovery	79

Table 5. Instrument, Instrumental MDL, and Extraction Batch 2 Data Reporting Form for NIST SRM 2711a

Laboratory Performing Extraction	LAB F			
Laboratory Performing Analysis	LAB F			
Extraction Batch 2 Results NIST 2711a				
Instrument Type? (ICP-AES or ICP-MS)	AES	Instrument Method Detection Limit (MDL) (ug/L)	5.2	
Extraction Date	11/08/2010			
Extraction Lead Standard Manufacturer and Lot #	See Table 3			
Analysis Date(s)	11/15/2010			
Analysis Lead Standard Manufacturer and Lot #	See Table 3			
Initial Calibration Verification Standard Source and Lot #	See Table 3			
Interference Check Sample Source and Lot #	See Table 3			
Sample Name	Instrument result for the analytical solution (ug/L)	Dilution Factor	Final Instrumental result for analytical solution (corrected for dilution) (ug/L)	Result in mg/Kg (correct for 1 g/100ml extraction)(i.e. ug/L times 100/1000 = mg/kg)
EXAMPLE SOIL (NIST 2711a)	70	10	700	70
Reagent Blank	0.0005492	1	0.0005492	NA
Bottle Blank	NA/Incubator			
SRM 2711a (Replicate 1)	10.99	1	10.99	1099
SRM 2711a (Replicate 2)	10.57	1	10.57	1057
SRM 2711a (Replicate 3)	10.89	1	10.89	1089
SRM 2711a (Replicate 4)	10.86	1	10.86	1086
SRM 2711a (Replicate 5)	10.82	1	10.82	1082
Control Soil SRM 2711	9.217	1	9.217	921.7
Blank Spike	9.770	1	9.770	977.0
SRM 2711a Matrix Spike	18.31	1	18.31	1831

Table 6. Extraction Batch 2 Spiked Blank and Spiked Sample Results for NIST SRM 2711a

Laboratory Performing Extraction	**LAB F**
Laboratory Performing Analysis	**LAB F**
Extraction Batch 2 Spiked Blank and Spiked Sample Results for NIST SRM 2711a	
Bottle Blank Result (mg/L)	NA
Blank Spike Result (mg/L)	9.770
Blank Spike Percent Recovery	98
Average (5) Result SRM 2711a (mg/L)	10.83
SRM 2711a Matrix Spike Result (mg/L)	18.31
SRM 2711a Matrix Spike Percent Recovery	75

Table 3. Laboratory, Instrument, Instrumental MDL, and Extraction Batch 1 Data Reporting Form for NIST SRM 2710a

Laboratory Performing Extraction	**LAB G**			
Laboratory Performing Analysis	**Other Lab name was here.**			
Extraction Batch 1 Results NIST 2710a				
Instrument Type? (ICP-AES or ICP-MS)	ICP-AES	**Instrument Method Detection Limit (MDL) (ug/L)**	6 ug/L	
Extraction Date	30 Nov 2010			
Extraction Lead Standard Manufacturer and Lot #	Inorganic Ventures, A2-PB02138			
Analysis Date(s)	6 Dec 2010			
Analysis Lead Standard Manufacturer and Lot #	Low std = SCP Science, SC9118120 Medium std = custom blend from High Purity Standards, MES-0509-06 High std = custom blend from High Purity Standards, MES-0509-07			
Initial Calibration Verification Standard Source and Lot #	ICV = Inorganic Ventures, custom blend, 02-MEB 326072 CCV = Inorganic Ventures, custom blend, 02-MEB 326073			
Interference Check Sample Source and Lot #	SCP Science, SC0165933			
Sample Name	**Instrument result for the analytical solution (ug/L)**	**Dilution Factor**	**Final Instrumental result analytical solution (corrected for dilution) (ug/L)**	**Result in mg/Kg (corrected for 1g/100mL extraction)(i.e ug/L times 100/1000 = mg/kg)**
EXAMPLE SOIL (NIST 2710a)	**70**	**10**	**700**	**70**
Reagent Blank	9.6 (<25 ug/L)	1	9.6	--
Bottle Blank	5.1 (<50 ug/L)	1	5.1	--
SRM 2710a (Replicate 1)	34300	1	34300	3430
SRM 2710a (Replicate 2)	33700	1	33700	3370
SRM 2710a (Replicate 3)	34200	1	34200	3420
SRM 2710a (Replicate 4)	34300	1	34300	3430
SRM 2710a (Replicate 5)	34600	1	34600	3460
Control Soil SRM 2711	9530	1	9530	953 (953/1162 = 82.0%)
Blank Spike	10700	1	10700	1070
SRM 2710a Matrix Spike	43000	1	43000	4300

Table 4. Extraction Batch 1Spiked Blank and Spiked Sample Results for NIST SRM 2710a

Laboratory Performing Extraction	LAB G
Laboratory Performing Analysis	Other Lab name was here.
Extraction Batch 1 Spiked Blank and Spiked Sample Results for NIST SRM 2710a	
Bottle Blank Result (mg/L)	<25 ug/L
Blank Spike Result (mg/L)	10.7 mg/L
Blank Spike Percent Recovery	98%
Average (5) Results SRM 2710a (mg/L)	34.2 mg/L
SRM 2710a Matrix Spike Result (mg/L)	43.0 mg/L
SRM 2710a Matrix Spike Percent Recovery	83%

Blank spike concentration was 10,039 mg/L*0.1102 g/101.3429 g = 10.92 mg/L

Matrix spike concentration was 10,039 mg/L*0.1066g/101.6238 g = 10.53 mg/L

Table 5. Instrument, Instrumental MDL, and Extraction Batch 2 Data Reporting Form for NIST SRM 2711a

Laboratory Performing Extraction	**LAB G**			
Laboratory Performing Analysis	**Other Lab name was here.**			
Extraction Batch 1 Results NIST 2711a				
Instrument Type? (ICP-AES or ICP-MS)	ICP-AES		**Instrument Method Detection Limit (MDL) (ug/L)**	6 ug/L
Extraction Date	30 November 2010			
Extraction Lead Standard Manufacturer and Lot #	Inorganic Ventures, A2-PB02138			
Analysis Date(s)	6 December 2010			
Analysis Lead Standard Manufacturer and Lot #	Low std = SCP Science, SC9118120 Medium std = custom blend from High Purity Standards, MES-0509-06 High std = custom blend from High Purity Standards, MES-0509-07			
Initial Calibration Verification Standard Source and Lot #	ICV = Inorganic Ventures, custom blend, 02-MEB 326072 CCV = Inorganic Ventures, custom blend, 02-MEB 326073			
Interference Check Sample Source and Lot #	SCP Science, SC0165933			
Sample Name	**Instrument result for the analytical solution (ug/L)**	**Dilution Factor**	**Final Instrumental result analytical solution (corrected for dilution) (ug/L)**	**Result in mg/Kg (corrected for 1g/100mL extraction)(i.e ug/L times 100/1000 = mg/kg)**
EXAMPLE SOIL (NIST 2711a)	**70**	**10**	**700**	**70**
Reagent Blank	11.4 (<25 ug/L)	1	11.4 (<25 ug/L)	--
Bottle Blank	4.6 (<50 ug/L)	1	4.6 (<50 ug/L)	--
SRM 2711a (Replicate 1)	11300	1	11300	1130
SRM 2711a (Replicate 2)	11300	1	11300	1130
SRM 2711a (Replicate 3)	11300	1	11300	1130
SRM 2711a (Replicate 4)	11200	1	11200	1120
SRM 2711a (Replicate 5)	11300	1	11300	1130
Control Soil SRM 2711	9580	1	9580	958 (958/1162 = 82.4%)
Blank Spike	10100	1	10100	1010
SRM 2711a Matrix Spike	21100	1	21100	2110

Table 6. Extraction Batch 2Spiked Blank and Spiked Sample Results for NIST SRM 2711a

Laboratory Performing Extraction	**LAB G**
Laboratory Performing Analysis	**Other Lab Name was here.**
Extraction Batch 1 Spiked Blank and Spiked Sample Results for NIST SRM 2711a	
Bottle Blank Result (mg/L)	**<25 ug/L**
Blank Spike Result (mg/L)	**10.1 mg/L**
Blank Spike Percent Recovery	**98%**
Average (5) Results SRM 2711a (mg/L)	**11.3 mg/L**
SRM 2711a Matrix Spike Result (mg/L)	**21.1 mg/L**
SRM 2711a Matrix Spike Percent Recovery	**93%**

Blank spike concentration was 10,039 mg/L*0.1057 g/102.9097 g = 10.31 mg/L

Matrix spike concentration was 10,039 mg/L*0.1067g/101.721 g = 10.53 mg/L