

U. S. DEPARTMENT OF THE INTERIOR  
GEOLOGICAL SURVEY

ASSESSMENT OF CHEMICAL VARIABILITY IN THREE  
INDEPENDENTLY PREPARED BATCHES OF NATIONAL  
INSTITUTE FOR STANDARDS AND TECHNOLOGY SRM 2704,  
BUFFALO RIVER SEDIMENT

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## INTRODUCTION

Under a cooperative agreement between the National Institute for Standards and Technology (NIST) and the United States Geological Survey (USGS), a study was conducted to determine the total element concentrations in three batches of standard reference material SRM 2704, also known as Buffalo River Sediment (National Institute for Standards and Technology, 1990). All three batches of SRM 2704 discussed in this report were derived from the same supply of the standard. The different SRM designations used in this report are designed to identify the different treatment procedures.

The first batch, identified as SRM 2704-original represents the currently distributed material that is certified for 25 major and trace elements. This supply of the standard was radiation sterilized immediately prior to bottling to stabilize the inorganic constituents against potential bacterial/microbial degradation. The second batch identified as SRM 2704-organic represents a supply of the original material that was destined to be certified for organic contaminants. SRM 2704-organic was not sterilized due to concerns over possible organic compound degradation by radiation sterilization. Comparisons of total element concentrations for these two batches of SRM 2704 was requested in order to determine if bottles of SRM 2704-organic could be added to the reserves of SRM 2704-original and help extend the lifetime of this valuable international reference material. If between batch comparisons of total element concentrations showed significant differences, then valuable information could be obtained regarding the long term stability of elements in a nonsterilized material.

The third batch of SRM 2704 used in this study was identified as 2704-organic/sterilized. This batch represents a separate split of 2704-organic that had undergone radiation sterilization just prior to shipment of the samples. In this phase of the study a comparison was made between 2704-organic and 2704-organic/sterilized to determine if radiation sterilization has an effect on total element concentrations. Of particular interest was the effect radiation sterilization would have on potentially volatile elements such as, arsenic, cadmium, mercury, and selenium.

In addition to the between batch comparison studies cited above, analytical results from the analysis of SRM 2704-original were also examined to evaluate the accuracy of USGS procedures. Comparison of USGS results with NIST certified values would be used to determine if a statistically significant bias existed for specific elements using USGS analytical procedures.

## EXPERIMENTAL

In phase one of the study, samples from SRM 2704-original and 2704-organic were randomly analyzed (in single job) to evaluate the between batch differences. Analytical results for SRM 2704-organic are based on the analysis of 25 individual bottles and 3 bottle duplicates (n=28). Results for SRM 2704-original are based on the analysis of 5 individual bottles and 2 bottle duplicates (n=7). Approximately 1 to 7 g of material from each bottle of SRM was transferred to 2-ounce glass bottles and dried for two hours at 110°C. Following the two hour drying time the bottles were immediately transferred to a desiccator to cool. After cooling the samples were removed from the desiccator, capped, and forwarded to the lab for analysis.

In phase two of the study, comparisons were made between 2704-organic and 2704-organic/sterilized. For this comparison, 5 bottles of SRM 2704-organic/sterilized were sampled along with 2 bottle duplicates (n=7). Results for 2704/organic were based on samples from 3 individual bottles (n=3). Samples were dried prior to analysis as described above. All total element concentrations are reported on a dry-weight basis.

All samples were analyzed using a combination of inductively coupled plasma-atomic emission spectrometry (ICP-AES), wavelength dispersive X-ray fluorescence spectroscopy (WDXRF), hydride generation-atomic absorption spectrophotometry (HG-AAS), and cold vapor-atomic absorption spectrophotometry (CV-AAS). The procedures used have been described in detail elsewhere (Arbogast, 1990; Baedeker, 1987; Crock, and others 1983) and will only be summarized here.

The ICP-AES procedure utilizes a 0.200-g sample which is transferred to a 30-mL teflon beaker and 50 µg of lutetium ( $\text{Lu}_2\text{O}_3$  in 5 percent HCl solution) added as an internal standard. The sample is digested to dryness overnight using concentrated HCl (3 mL),  $\text{HNO}_3$  (2 mL),  $\text{HClO}_4$  (1 mL), and HF (2 mL). The residue is redigested using  $\text{HClO}_4$  (1 mL), and water at 150°C, and taken to dryness again. One milliliter of aqua regia is finally added to the container, and the sample is diluted to 10.00 g with 1 percent  $\text{HNO}_3$ . The solution is then analyzed for 40 elements simultaneously using a Jarrell Ash model 1160 spectrometer.

In WDXRF analyses, a  $0.8000 \pm 0.004$  g sample is ignited in a tared platinum crucible at 925°C for 45 minutes. The weight loss is reported as percent loss on ignition (% LOI). An 8.00 g charge of lithium tetraborate is then added to the crucible along with a 0.250 mL aliquot of a 50 percent solution of lithium bromide, which serves as a nonwetting agent. Up to 7 crucibles are then placed on an "automatic fluxer," and the entire unit inserted into a muffle furnace, where the sample is fused for 40

minutes at 1120°C. After the fusion is complete the fluxing unit is removed from the muffle furnace, and the molten samples poured into specially designed two-piece platinum molds. When cooled, the individual glass disc is analyzed for its major element constituents using a Phillips model 1606 X-ray spectrometer. Major element concentrations are determined using calibration curves compiled from approximately 70 international geologic reference materials.

In the analysis of arsenic and selenium, 0.25 g of material are transferred to a 30-mL teflon bomb and moistened with 1 mL of 1 percent  $\text{HNO}_3$ . The sample is digested overnight at 105°C using concentrated  $\text{HNO}_3$  (9 mL),  $\text{HClO}_4$  (6 mL), and  $\text{HF}$  (10 mL). Following overnight digestion, 25 mL of 50 percent (v/v)  $\text{HCl}$  is added to the container and the solution allowed to stand for 30 minutes. The solution is then transferred to a 60-mL polyethylene bottle and the mass adjusted to 54 g with deionized water. Samples are analyzed for arsenic and selenium using a Perkin Elmer 4100 and 303 atomic absorption spectrophotometers respectively. Both instruments are equipped with specially designed continuous-flow systems and the instruments are calibrated with commercially available aqueous standards. The procedure has a percent relative standard deviation (%RSD) of approximately 10 percent (Crock, J.G., and Lichte, F.E., 1982; Sanzolone and Chao, 1987).

Total mercury concentration in the three batches of SRM 2704 was determined using a cold vapor-atomic absorption spectrophotometric procedure (CV-AAS). In this procedure, (Kennedy and Crock, 1987) 0.100 g of sample is transferred to a 16 x 100-mm glass test tube and 0.5 mL of sodium dichromate (25 percent w/v) and 2 mL of concentrated nitric acid added. The tube is then transferred to an aluminum heating block and the sample digested for 3 hours at 110°C. Following digestion, the sample is cooled and the volume adjusted to 12 mL with deionized water. Samples are then analyzed on a Perkin Elmer model 303 atomic absorption spectrophotometer equipped with a USGS designed continuous-flow sample delivery system. Routine analysis of reference materials reveals that the method has a percent relative standard deviation of 10 percent.

## RESULTS AND DISCUSSION

Prior to the comparison of between batch data, results from the USGS analysis of SRM 2704-original were compared with NIST certified values to evaluate the accuracy of USGS procedures. Results for 2704-original reported on a dry weight basis are presented in Appendix A. A summary of SRM 2704-original results along with NIST certified and noncertified values are reported in table 1. Using statistical guidelines published by NIST (Becker,

D., and others, 1992), USGS results were compared to certified values to test the hypothesis that the mean total element concentrations are equal at the 95 percent confidence level. Comparison of WDXRF results for 9 major elements with the corresponding NIST values reveals that we can accept the hypothesis that the mean concentrations are the same at the 95 percent confidence level. A similar conclusion is reached for major elements quantified by the ICP-AES procedure, except for titanium which shows a significant bias (low) compared to the NIST value.

Comparison of ICP-AES trace element results with the NIST certified values reveals that only in the cases of arsenic, chromium, and cobalt is the hypothesis that the means are equal rejected at the 95 percent confidence level. Arsenic is also found to be biased (low) by the HY-AAS technique, though this observed concentration compares favorably with the ICP-AES value. A comparison of USGS mercury results using the CV-AAS procedure with the NIST certified value reveals the mean values are equal at the 95 percent confidence level.

One explanation for the lower concentrations of arsenic by the ICP-AES and HG-AAS procedures is that the three batches of SRM 2704 used in this study were oven dried according to the NIST protocol prior to analysis. It is possible that at the drying temperature of 110°C, a portion of the arsenic was volatilized, leading to the lower concentrations observed in this study. Analysis of undried SRM 2704 was not possible under the guidelines of this study.

Comparison of NIST noncertified values with ICP-AES values shows general agreement except for cerium, where the USGS value appears to be significantly lower. Based on the good agreement between USGS and NIST total element concentrations, it is reasonable to assume that USGS analytical procedures provide an accurate estimation of total element concentrations in SRM 2704.

In phase one of the study, data from SRM 2704-original were compared against results for SRM 2704-organic to determine if significant differences in total element concentrations existed between the two batches of standards. Because it was unclear at the start of this study if SRM 2704-organic had ever been examined for homogeneity with respect to its inorganic constituents, a test was performed to evaluate the between- and within-bottle variability. The homogeneity evaluation for SRM 2704-organic was based on the analysis of 25 individual bottles and 3 bottle replicates (n=28). Individual results corrected to dry weight are reported in Appendix B. Results for ICP-AES and WDXRF analyses were evaluated using an analysis of variance (ANOVA). The ANOVA revealed no significant difference for between- or within-bottle total element concentrations at the 95 percent confidence level. Two exceptions were ICP-AES results for

aluminum and nickel. In the case of aluminum, conclusions based on ICP-AES results are not substantiated by the WDXRF data, which show no statistical difference in the data sets. In the case of nickel, even though the means are statistically different, the fact that both means fall within the NIST 95 percent confidence interval and that the relative difference in mean concentrations is less than 3 percent suggests that from a practical standpoint the values should be considered the same.

After establishing that no significant between-bottle differences existed for SRM 2704-organic, a comparison of total element concentrations was made between 2704-original and 2704-organic. Differences in mean total element concentrations were evaluated using a two sample t-test (Walpole, R.E. and Myers, R.H., 1989). A comparison of average total element concentrations is summarized in table 2 along with an indication if there was (Y) or was not (N) a statistical difference in mean total element concentrations at the 95 percent confidence level. Of the 26 trace elements evaluated, a total of 11 (42 percent) show a statistically significant difference in mean total element concentrations for the two batches of SRM 2704. Most notable are the difference observed for chromium, lead, and zinc by ICP-AES, arsenic by CV-AAS, and mercury by CV-AAS. Conclusions regarding the between batch differences for arsenic and mercury should be viewed with caution, because of the drying step used in the sample preparation and the possibility of element volatilization. While element volatilization is a distinct possibility, it should also be pointed out that the two batches were randomly combined and dried simultaneously under identical conditions. If volatilization did occur, it is reasonable to assume that all samples would be affected equally.

In the case of the major elements, where both ICP-AES and WDXRF results are available, conclusions regarding between batch differences are less definitive. For magnesium, phosphorous, and titanium, both ICP-AES and WDXRF results indicate that there is no statistical difference in the mean concentrations at the 95 percent confidence level. In case of aluminum, calcium, potassium, and sodium, conclusions regarding between batch differences in mean total element concentrations are unclear because of the conflicting statistical results. Only in the case of iron do both techniques indicate that the two batches are statistically different.

In phase two of the study samples of SRM-organic and SRM-organic/sterilized were compared to determine if radiation sterilization affects total element concentrations. In a separate experiment, samples from five bottles of SRM 2704-organic/sterilized supplied by NIST were randomly analyzed along with samples from three bottles of SRM 2704-organic. A statistical summary of the data is presented in table 3 and individual values corrected to dry weight are presented in Appendices C and D.

Average concentrations and standard deviations for 2704-organic\sterilized represent a combination of between and within bottle results (n=7). Average concentrations and standard deviations for 2704-organic are based solely on the analysis of between bottle determinations (n=3). Comparison of mean total element concentrations for 2704-organic and 2704-organic/sterilized using a pooled t-test reveals that for the majority (89 percent) of elements mean total element concentrations are considered equal at the 95 percent confidence level. Exceptions include sodium, magnesium, and calcium by ICP-AES, and potassium by WDXRF.

## CONCLUSIONS

Comparison of analytical results for SRM 2704-original and SRM 2704-organic indicates that for the majority of elements there is no significant difference in mean total element concentration at the 95 percent confidence level. Where statistical differences do exist, the lower concentrations are normally associated with SRM 2704-organic, suggesting that unsterilized material can change over time. It is also apparent from the excellent agreement between USGS and NIST certified values for SRM 2704-original that once the material has been radiation sterilized it is stable over time. The effect of radiation sterilization on total element concentration on SRM 2704 is found to be negligible as evidenced by the similarity in analytical results for 2704-organic and 2704-organic/sterilized. This is especially significant for volatile elements such as mercury, arsenic, selenium, and cadmium. Before a final conclusion is reached regarding the between batch differences observed in this study, NIST scientists need to evaluate the magnitude of these difference with respect to the recommended confidence interval for each element.

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## BIBLIOGRAPHY

- Arbogast , B.F., (ed.), 1990, Quality Assurance Manual for the Branch of Geochemistry, U.S. Geological Survey, 184 p.
- Baedecker, P.A., (ed.), 1987, Methods of Geochemical Analysis, U.S. Geological Survey Bulletin 1770, 180 p.
- Becker, D., Christensen, R., Currie, L., Diamondstone, B., Eberhardt, K., Gills, T., Hertz, H., Klouda, G., Moody, J., Parris, R., Schaffer, R., Steel, E., Taylor, J., Watters, R., Zeisler, R., 1992, Use of NIST Standard Reference Materials for Decisions on Performance of Analytical Chemical Methods and Laboratories, NIST Special Publication 829, 27p.
- Crock, J.G., and Lichte, F.E., (1982), An improved method for the determination of trace levels of arsenic and antimony in geologic materials by automated hydride generation-atomic absorption spectroscopy, *Analytica Chimica Acta*, 144, 223-33
- Crock, J.G., Lichte, F.E., and Briggs, P.H., 1983  
Determination of elements in National Bureau of Standards' geologic reference materials SRM 278 Obsidian and SRM 688 Basalt by Inductively Coupled Argon Plasma-Atomic Emission Spectroscopy, *Geostandards Newsletter*, 7, 335-340
- Kennedy, K.R., and Crock, J.G., 1987, Determination of mercury in geological material by continuous flow, cold-vapor, atomic absorption spectrophotometry, *Analytical Letters*, 20, 899-908
- National Institute for Standards and Technology, 1990  
Certificate, Standard Reference Material 2704, Buffalo River Sediment.
- Sanzolone R.F. and Chao, T.T., 1987, Determination of selenium in thirty-two geochemical reference materials by continuous-flow hydride generation atomic absorption spectrophotometry, *Geostandards Newsletter*, 11, 81-85.
- Walpole R.E., and Myers, R.H., 1989, Probability and Statistics for Engineers and Scientists, 4th ed. MacMillan Publishing Company, New York, 765 p.

Table 1. Comparison of USGS values for SRM 2704-original  
with NIST certified or recommended concentrations

<u>Element</u>	<u>USGS Method</u>	<b>USGS</b>		<b>NIST</b>	
		<u>Average conc, %<sup>1</sup></u>	<u>STD<sup>2</sup></u>	<u>Certified<sup>3</sup> conc, %</u>	<u>STD<sup>4</sup></u>
Al	ICP	6.14	0.03	6.11	0.16
Al	WDXRF	6.19	0.12		
Ca	ICP	2.65	0.02	2.60	0.03
Ca	WDXRF	2.62	0.01		
Fe	ICP	4.08	0.03	4.11	0.10
Fe	WDXRF	4.14	0.01		
K,	ICP	1.95	0.01	2.00	0.04
K,	WDXRF	2.01	0.01		
Mg	ICP	1.23	0.02	1.20	0.02
Mg	WDXRF	1.20	0.01		
Na	ICP	0.60	0.01	0.547	0.014
Na	WDXRF	0.56	0.01		
P,	ICP	0.10	<0.005	0.0998	0.0028
P,	WDXRF	0.10	<0.005		
Si	WDXRF	29.15	0.12	29.08	0.13
Ti	ICP	0.40	0.02	0.457	0.018
Ti	WDXRF	0.46	<0.005		

<u>Element</u>	<u>Method</u>	<u>µg/g</u>	<u>STD</u>	<u>µg/g</u>	<u>STD</u>
As	ICP	20	1.4	23.4	0.8
As	HY-AAS	20	0.5		
Ba	ICP	412	4	414	12
Cd	ICP	3	0.5	3.45	0.22
Cr	ICP	143	2	135	5
Co	ICP	16	0.5	14	0.6
Cu	ICP	97	1	98.6	5
Hg	CV-AAS	1.4	0.05	1.44	0.07
Mn	ICP	575	8	555	19
Ni	ICP	44	1	44.1	3.0
Pb	ICP	157	6	161	17
V,	ICP	90	1	95	4
Zn	ICP	429	9	438	12

<u>Element</u>	<u>USGS Method</u>	<u>µg/g</u>	<u>STD</u>	<b>Recommended<sup>3</sup></b>	
				<u>conc.</u>	<u>µg/g</u>
Ce	ICP	57	2		72
La	ICP	32	0.5		29
Li	ICP	49	0.5		50
Sc	ICP	12	<0.5		12
Se	HY-AAS	1.0	<0.05		1.1
Sr	ICP	136	1		130
Th	ICP	9	1		9.2
Yb	ICP	2	<0.5		2.8

<sup>1</sup> Average based on seven determinations

<sup>2</sup> One standard deviation

<sup>3</sup> See certificate for definition

<sup>4</sup> NIST's calculated 95 percent confidence interval

Table 2. Summary results for 2704-original and SRM 2704-organic

Element	Method	SRM 2704 original		SRM 2704 organic		Signif. Diff. Y/N <sup>4</sup>
		Avg. <sup>1</sup>	STD. <sup>2</sup>	Avg. <sup>3</sup>	STD. <sup>2</sup>	
Al, %	ICP	6.14	0.03	6.09	0.03	N
Al, %	WDXRF	6.19	0.03	6.16	0.03	Y
Ca, %	ICP	2.65	0.02	2.66	0.02	N
Ca, %	WDXRF	2.62	0.01	2.64	0.01	Y
Fe, %	ICP	4.08	0.03	3.99	0.02	Y
Fe, %	WDXRF	4.14	0.01	4.06	0.02	Y
K, %	ICP	1.95	0.01	1.94	0.01	Y
K, %	WDXRF	2.01	0.01	2.00	0.01	N
Mg, %	ICP	1.23	0.02	1.22	0.01	N
Mg, %	WDXRF	1.20	0.01	1.20	0.01	N
Na, %	ICP	0.60	0.01	0.61	0.01	N
Na, %	WDXRF	0.56	0.01	0.58	0.01	Y
P, %	ICP	0.10	<0.005	0.10	<0.005	N
P, %	WDXRF	0.10	<0.005	0.10	<0.005	N
Si, %	WDXRF	29.14	0.12	29.23	0.10	N
Ti, %	ICP	0.40	0.02	0.40	0.02	N
Ti, %	WDXRF	0.46	<0.005	0.46	<0.005	N
LOI, %	WDXRF	9.23	0.08	9.16	0.08	Y
As, µg/g	ICP	20	1.4	15	1.2	Y
As, µg/g	HG-AAS	20.	1.0	16	0.6	Y
Ba, µg/g	ICP	412	4.2	412	5.2	N
Be, µg/g	ICP	2	<0.5	2	<0.5	N
Cd, µg/g	ICP	3.3	0.5	3	<0.5	Y
Ce, µg/g	ICP	57	1.7	56	1.9	N
Co, µg/g	ICP	16	0.5	15	0.6	N
Cr, µg/g	ICP	143	2.0	128	2.5	Y
Cu, µg/g	ICP	97	1.2	92	5.0	Y
Ga, µg/g	ICP	15	0.5	15	0.5	N
Hg, µg/g	CV-AAS	1.4	0.05	1.0	0.1	Y
La, µg/g	ICP	32	0.5	32	1.1	N
Li, µg/g	ICP	49	0.5	48	0.4	Y
Mn, µg/g	ICP	575	7.5	563	5.1	Y
Nb, µg/g	ICP	9	1.3	8	0.7	N
Nd, µg/g	ICP	29	1.2	30	1.2	N
Ni, µg/g	ICP	44	1.1	43	1.9	N
Pb, µg/g	ICP	157	5.8	146	7.5	Y
Sc, µg/g	ICP	12	<0.5	12	<0.5	N
Se, µg/g	HG-AAS	1	<0.05	1	<0.05	N
Sr, µg/g	ICP	136	1.1	137	1.1	N
Th, µg/g	ICP	9	1.2	10	0.8	N
V, µg/g	ICP	90	0.8	89	0.8	Y
Y, µg/g	ICP	31	0.8	31	0.5	N
Yb, µg/g	ICP	2	<0.5	2	0.31	N
Zn, µg/g	ICP	429	8.6	395	8.7	Y

<sup>1</sup> Average based on 7 determinations<sup>2</sup> One Standard Deviation<sup>3</sup> Average based on 28 determinations<sup>4</sup> Average values are (Y), or are not (N), significantly different at the 95 percent confidence interval

Table 3. Summary results for SRM 2704-organic and 2704-organic/sterilized

Element	Method	SRM 2704 organic		SRM 2704 organic/sterilized		Signif. Different (Y/N) <sup>4</sup>
		Avg. <sup>1</sup>	STD. <sup>2</sup>	Avg. <sup>3</sup>	STD. <sup>2</sup>	
Al, %	ICP	6.10	0.05	6.08	0.05	N
Al, %	WDXRF	6.10	0.03	6.12	0.03	N
Ca, %	ICP	2.63	0.01	2.59	0.02	Y
Ca, %	WDXRF	2.63	0.01	2.63	0.01	N
Fe, %	ICP	4.15	<0.005	4.11	0.04	N
Fe, %	WDXRF	4.07	0.01	4.08	0.04	N
K, %	ICP	1.99	0.09	2.02	0.03	N
K, %	WDXRF	1.99	<0.005	2.00	0.01	Y
Mg, %	ICP	1.26	0.01	1.25	0.01	Y
Mg, %	WDXRF	1.18	0.02	1.19	0.01	N
Na, %	ICP	0.62	<0.015	0.61	0.01	Y
Na, %	WDXRF	0.57	<0.005	0.56	0.01	N
P, %	ICP	0.11	<0.005	0.11	<0.005	N
P, %	WDXRF	0.10	<0.005	0.10	<0.005	N
Si, %	WDXRF	29.07	0.09	29.08	0.03	N
Ti, %	ICP	0.34	0.02	0.32	0.01	N
Ti, %	WDXRF	0.45	<0.005	0.46	<0.005	N
LOI, %	WDXRF	9.12	<0.005	9.08	0.06	N
As, µg/g	ICP	22	1.2	20	1.8	N
As, µg/g	HG-AAS	17	1.1	17	0.5	N
Ba, µg/g	ICP	414	2.6	409	4.3	N
Be, µg/g	ICP	2	0.6	2	<0.5	N
Cd, µg/g	ICP	3	0.07	3	<0.5	N
Ce, µg/g	ICP	60	2.2	58	1.	N
Co, µg/g	ICP	15	0.3	15	0.4	N
Cr, µg/g	ICP	126	2.0	123	1.4	N
Cu, µg/g	ICP	92	2.9	92	4	N
Ga, µg/g	ICP	15	0.2	15	0.7	N
Hg, µg/g	CV-AAS	1.0	0.08	1.1	0.17	N
La, µg/g	ICP	29	0.7	29	0.5	N
Li, µg/g	ICP	49	0.2	49	0.2	N
Mn, µg/g	ICP	585	3	577	4.7	N
Nb, µg/g	ICP	9	0.5	9	0.5	N
Nd, µg/g	ICP	29	0.9	29	0.8	N
Ni, µg/g	ICP	42	0.3	42	1.8	N
Pb, µg/g	ICP	148	8.7	144	4.7	N
Sc, µg/g	ICP	12	<0.5	12	<0.5	N
Se, µg/g	HG-AAS	1.0	0.06	1.1	0.04	N
Sr, µg/g	ICP	139	1.2	136	1.3	N
Th, µg/g	ICP	10	0.7	10	0.9	N
V, µg/g	ICP	95	0.4	94	0.8	N
Y, µg/g	ICP	24	0.1	24	0.2	N
Yb, µg/g	ICP	2	<0.5	2	0.2	N
Zn, µg/g	ICP	408	2.3	404	8.1	N

<sup>1</sup> Average value based on 3 determinations

<sup>2</sup> One Standard Deviation

<sup>3</sup> Average value based on 5 determinations

<sup>4</sup> Values are (Y), or are not (N) significantly different at the 95 percent confidence interval

# Appendix A Results for SRM-2704 original on dry weight basis, by ICP-AES, WDXRF, HG-AAS, and CV-AAS

## ICP-AES

Sample ID	Al, %	Ca, %	Fe, %	K, %	Mg, %	Na, %	P, %	Ti, %
Orig-1A	6.12	2.64	4.07	1.94	1.23	0.60	0.10	0.42
Orig-1B	6.13	2.64	4.07	1.94	1.22	0.61	0.10	0.40
Orig-2	6.13	2.64	4.05	1.95	1.23	0.60	0.10	0.41
Orig-3	6.20	2.70	4.14	1.98	1.25	0.61	0.10	0.40
Orig-4	6.10	2.63	4.07	1.95	1.20	0.60	0.10	0.37
Orig-5A	6.15	2.65	4.10	1.95	1.23	0.60	0.10	0.41
Orig-5B	6.15	2.67	4.09	1.96	1.24	0.61	0.11	0.40

Sample ID	Ag ppm	As ppm	Au ppm	Ba ppm	Be ppm	Bi ppm	Cd ppm	Ce ppm
Orig-1A	<2	18	<8	408	2	<10	3	59
Orig-1B	<2	19	<8	419	2	<10	3	58
Orig-2	<2	19	<8	411	2	<10	3	56
Orig-3	<2	21	<8	416	2	<10	4	58
Orig-4	<2	21	<8	408	2	<10	3	54
Orig-5A	<2	22	<8	409	2	<10	3	58
Orig-5B	<2	20	<8	413	2	<10	4	56

Sample ID	Co ppm	Cr ppm	Cu ppm	Eu ppm	Ga ppm	Ho ppm	La ppm	Li ppm
Orig-1A	16	143	97	<2	16	<4	32	49
Orig-1B	15	142	96	<2	15	<4	32	49
Orig-2	15	141	96	<2	15	<4	32	48
Orig-3	15	144	99	<2	15	<4	32	49
Orig-4	16	140	98	<2	16	<4	31	48
Orig-5A	16	146	96	<2	15	<4	32	49
Orig-5B	16	144	98	<2	16	<4	31	48

Sample ID	Mn ppm	Mo ppm	Nb ppm	Nd ppm	Ni ppm	Pb ppm	Sc ppm	Sn ppm
Orig-1A	567	<2	9	30	43	161	12	<5
Orig-1B	574	2	9	30	44	149	12	<5
Orig-2	572	2	8	29	42	156	12	<5
Orig-3	591	<2	8	29	45	158	12	<5
Orig-4	573	<2	11	29	44	153	12	<5
Orig-5A	574	2	8	31	43	155	12	<5
Orig-5B	575	<2	7	27	44	167	12	<5

ICP-AES									
Sample	Sr	Ta	Th	U	V	Y	Yb	Zn	
ID	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm	
Orig-1A	135	<40	9	<100	90	31	2	428	
Orig-1B	136	<40	10	<100	90	30	2	425	
Orig-2	135	<40	10	<100	91	32	2	427	
Orig-3	138	<40	8	<100	91	32	2	443	
Orig-4	136	<40	8	<100	89	31	2	419	
Orig-5A	136	<40	11	<100	90	31	2	421	
Orig-5B	135	<40	8	<100	91	32	2	437	

  

WDXRF											
Sample	SiO <sub>2</sub> %	Al <sub>2</sub> O <sub>3</sub> %	Fe <sub>2</sub> O <sub>3</sub> %	MgO %	CaO %	Na <sub>2</sub> O %	K <sub>2</sub> O %	TiO <sub>2</sub> %	P <sub>2</sub> O <sub>5</sub> %	MnO %	LOI %
orig-1A	62.3	11.7	5.94	1.98	3.68	0.78	2.41	0.77	0.23	0.08	9.10
orig-1B	62.3	11.7	5.93	1.96	3.66	0.74	2.43	0.77	0.23	0.08	9.34
orig-2	62.3	11.8	5.93	2.02	3.68	0.76	2.41	0.77	0.24	0.08	9.21
orig-3	62.7	11.7	5.94	2.01	3.67	0.76	2.42	0.77	0.24	0.08	9.23
orig-4	62.2	11.7	5.91	1.99	3.67	0.76	2.42	0.77	0.24	0.08	9.33
orig-5A	62.0	11.6	5.89	2.01	3.66	0.77	2.41	0.76	0.24	0.08	9.19
orig-5B	62.7	11.7	5.93	1.98	3.67	0.76	2.42	0.77	0.23	0.08	9.24

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# Appendix B Results for SRM 2704-organic on dry weight basis, by ICP-AES, WDXRF, HG-AAS, and CV-AAS

## ICP-AES

### Sample

<u>ID</u>	<u>Al, %</u>	<u>Ca, %</u>	<u>Fe, %</u>	<u>K, %</u>	<u>Mg, %</u>	<u>Na, %</u>	<u>P, %</u>	<u>Ti, %</u>
ORG-06	6.05	2.62	3.99	1.93	1.20	0.61	0.10	0.38
ORG-07	6.09	2.65	4.01	1.93	1.21	0.60	0.10	0.39
ORG-08	6.06	2.69	3.97	1.92	1.22	0.60	0.10	0.39
ORG-09	6.09	2.66	3.99	1.93	1.22	0.61	0.10	0.40
ORG-10	6.13	2.70	4.02	1.96	1.24	0.60	0.10	0.41
ORG-10B	6.12	2.67	4.00	1.95	1.22	0.60	0.10	0.39
ORG-11	6.06	2.68	3.97	1.93	1.21	0.60	0.10	0.36
ORG-12	6.08	2.65	3.98	1.93	1.22	0.60	0.10	0.42
ORG-13	6.09	2.67	3.99	1.94	1.22	0.61	0.10	0.40
ORG-14	6.12	2.69	4.02	1.93	1.23	0.62	0.10	0.42
ORG-15	6.08	2.66	3.98	1.94	1.21	0.61	0.10	0.35
ORG-16	6.11	2.67	3.98	1.94	1.23	0.61	0.10	0.41
ORG-17	6.04	2.65	3.98	1.93	1.22	0.61	0.10	0.42
ORG-18	6.06	2.63	3.97	1.93	1.21	0.61	0.10	0.38
ORG-19	6.16	2.66	3.98	1.95	1.23	0.60	0.10	0.41
ORG-20	6.12	2.68	4.02	1.94	1.23	0.62	0.10	0.40
ORG-20B	6.12	2.66	4.02	1.95	1.3	0.61	0.10	0.40
ORG-21	6.07	2.65	4.00	1.94	1.22	0.61	0.10	0.40
ORG-22	6.03	2.65	3.97	1.92	1.21	0.61	0.10	0.40
ORG-23	6.13	2.70	3.98	1.95	1.24	0.62	0.10	0.39
ORG-24	6.10	2.67	4.03	1.95	1.23	0.61	0.10	0.41
ORG-25	6.11	2.71	4.02	1.95	1.24	0.61	0.10	0.40
ORG-26	6.08	2.65	3.95	1.92	1.21	0.62	0.10	0.40
ORG-27	6.07	2.65	3.98	1.91	1.21	0.62	0.10	0.40
ORG-28	6.12	2.68	4.01	1.95	1.23	0.61	0.10	0.40
ORG-29	6.05	2.65	3.96	1.93	1.21	0.61	0.10	0.37
ORG-30	6.08	2.66	3.97	1.93	1.22	0.60	0.10	0.41
ORG-30B	6.07	2.65	3.98	1.93	1.21	0.62	0.10	0.37

Appendix B cont.

ICP-AES Sample ID	Ag $\mu\text{g/g}$	As $\mu\text{g/g}$	Au $\mu\text{g/g}$	Ba $\mu\text{g/g}$	Be $\mu\text{g/g}$	Bi $\mu\text{g/g}$	Cd $\mu\text{g/g}$	Ce $\mu\text{g/g}$
ORG-06	<2	14	<8	411	2	<10	3	55
ORG-07	<2	16	<8	414	2	<10	3	56
ORG-08	<2	15	<8	411	2	<10	3	56
ORG-09	<2	16	<8	413	2	<10	3	56
ORG-10	<2	14	<8	408	2	<10	3	54
ORG-10B	<2	15	<8	425	2	<10	3	58
ORG-11	<2	14	<8	409	2	<10	3	56
ORG-12	<2	16	<8	412	2	<10	3	60
ORG-13	<2	16	<8	414	2	<10	3	55
ORG-14	<2	14	<8	421	2	<10	3	58
ORG-15	<2	14	<8	404	2	<10	3	55
ORG-16	<2	14	<8	410	2	<10	3	54
ORG-17	<2	16	<8	408	2	<10	3	56
ORG-18	<2	14	<8	415	2	<10	3	56
ORG-19	<2	11	<8	409	2	<10	3	58
ORG-20	<2	14	<8	414	2	<10	3	63
ORG-20B	<2	15	<8	405	2	<10	3	55
ORG-21	<2	15	<8	415	2	<10	3	56
ORG-22	<2	15	<8	410	2	<10	3	56
ORG-23	<2	14	<8	419	2	<10	3	54
ORG-24	<2	15	<8	415	2	<10	3	56
ORG-25	<2	14	<8	419	2	<10	3	55
ORG-26	<2	13	<8	405	2	<10	3	55
ORG-27	<2	14	<8	411	2	<10	3	57
ORG-28	<2	13	<8	414	2	<10	3	57
ORG-29	<2	15	<8	406	2	<10	3	57
ORG-30	<2	16	<8	406	2	<10	3	57
ORG-30B	<2	16	<8	405	2	<10	3	54



Appendix B cont.

ICP-AES Sample ID	Co $\mu\text{g/g}$	Cr $\mu\text{g/g}$	Cu $\mu\text{g/g}$	Eu $\mu\text{g/g}$	Ga $\mu\text{g/g}$	Ho $\mu\text{g/g}$	La $\mu\text{g/g}$	Li $\mu\text{g/g}$
ORG-06	15	130	92	<2	16	<4	31	47
ORG-07	16	128	95	<2	15	<4	31	48
ORG-08	15	127	88	<2	15	<4	32	48
ORG-09	15	125	89	<2	15	<4	32	48
ORG-10	16	130	93	<2	16	<4	30	48
ORG-10B	15	128	91	<2	15	<4	32	48
ORG-11	16	131	89	2	15	<4	31	48
ORG-12	16	129	91	<2	15	<4	33	48
ORG-13	16	129	91	<2	15	<4	32	48
ORG-14	16	131	91	<2	15	<4	32	48
ORG-15	15	128	91	<2	15	<4	31	48
ORG-16	15	129	100	<2	15	<4	31	48
ORG-17	15	130	97	<2	14	<4	31	47
ORG-18	15	129	89	<2	15	<4	32	48
ORG-19	16	131	92	<2	15	<4	32	48
ORG-20	16	125	87	<2	16	<4	36	49
ORG-20B	15	126	87	<2	14	<4	31	48
ORG-21	15	125	92	<2	15	<4	31	47
ORG-22	16	127	97	<2	14	<4	32	48
ORG-23	15	134	96	<2	15	<4	31	48
ORG-24	14	132	92	<2	16	<4	31	48
ORG-25	16	130	98	<2	15	<4	31	48
ORG-26	15	123	90	<2	14	<4	32	48
ORG-27	15	128	110	<2	15	<4	32	47
ORG-28	15	127	88	<2	15	<4	31	48
ORG-29	15	126	95	<2	15	<4	31	47
ORG-30	16	127	90	<2	15	<4	32	48
ORG-30B	15	125	86	<2	15	<4	30	48

Appendix B cont.

ICP-AES Sample ID	Mn <u>µg/g</u>	Mo <u>µg/g</u>	Nb <u>µg/g</u>	Nd <u>µg/g</u>	Ni <u>µg/g</u>	Pb <u>µg/g</u>	Sc <u>µg/g</u>	Sn <u>µg/g</u>
ORG-06	563	<2	8	29	43	142	12	<5
ORG-07	561	<2	9	31	43	140	12	<5
ORG-08	558	<2	8	31	41	149	12	<5
ORG-09	559	<2	9	30	43	138	12	<5
ORG-10	571	<2	8	30	44	146	12	<5
ORG-10B	560	<2	8	30	44	152	12	<5
ORG-11	558	<2	7	30	43	150	12	<5
ORG-12	562	2	9	32	43	149	12	<5
ORG-13	568	<2	8	30	43	137	12	<5
ORG-14	563	<2	8	30	44	141	12	<5
ORG-15	567	<2	7	28	43	140	12	<5
ORG-16	560	2	9	28	44	155	12	<5
ORG-17	565	<2	8	28	44	166	12	<5
ORG-18	559	<2	8	30	43	146	12	<5
ORG-19	559	<2	8	30	44	156	12	<5
ORG-20	565	2	9	33	43	138	12	<5
ORG-20B	559	2	9	28	42	139	12	<5
ORG-21	564	<2	8	29	43	145	12	<5
ORG-22	562	3	9	30	42	138	12	<5
ORG-23	568	2	9	29	44	150	12	<5
ORG-24	562	<2	7	30	43	154	12	<5
ORG-25	565	<2	8	30	42	157	12	<5
ORG-26	560	<2	8	28	42	136	12	<5
ORG-27	558	<2	8	31	52	148	12	<5
ORG-28	568	<2	7	29	43	145	12	<5
ORG-29	567	<2	7	28	43	152	12	<5
ORG-30	562	<2	8	29	42	142	12	<5
ORG-30B	582	<2	7	30	42	138	12	<5

Appendix B cont.

ICP-AES Sample ID	Sr μg/g	Ta μg/g	Th μg/g	U μg/g	V μg/g	Y μg/g	Yb μg/g	Zn μg/g
ORG-06	136	<40	10	<100	89	31	2	394
ORG-07	137	<40	10	<100	89	31	2	383
ORG-08	137	<40	10	<100	89	31	2	388
ORG-09	136	<40	10	<100	90	31	2	402
ORG-10	136	<40	10	<100	90	31	2	403
ORG-10B	140	<40	9	<100	90	32	2	392
ORG-11	135	<40	10	<100	89	31	2	392
ORG-12	136	<40	11	<100	88	32	2	389
ORG-13	137	<40	10	<100	89	31	2	387
ORG-14	138	<40	9	<100	89	32	2	393
ORG-15	136	<40	10	<100	88	31	2	390
ORG-16	136	<40	9	<100	89	32	2	397
ORG-17	134	<40	8	<100	89	31	2	388
ORG-18	136	<40	10	<100	89	31	2	391
ORG-19	137	<40	10	<100	90	31	3	393
ORG-20	138	<40	10	<100	89	32	2	404
ORG-20B	137	<40	9	<100	90	32	2	393
ORG-21	137	<40	10	<100	89	31	2	393
ORG-22	136	<40	9	<100	89	31	2	386
ORG-23	136	<40	11	<100	89	31	2	406
ORG-24	136	<40	10	<100	90	31	3	405
ORG-25	137	<40	9	<100	90	32	3	415
ORG-26	138	<40	9	<100	88	31	2	381
ORG-27	136	<40	11	<100	89	31	2	400
ORG-28	137	<40	8	<100	90	31	2	409
ORG-29	136	<40	9	<100	87	30	2	395
ORG-30	136	<40	9	<100	88	31	2	387
ORG-30B	136	<40	10	<100	89	31	2	411

Appendix B cont.

HG-AAS and CV-AAS			
Sample	As	Se	Hg
ID	$\mu\text{g/g}$	$\mu\text{g/g}$	$\mu\text{g/g}$
ORG-06	17	1.0	1.0
ORG-07	15	1.0	0.98
ORG-08	15	1.0	0.92
ORG-09	15	1.0	0.97
ORG-10	15	1.0	1.0
ORG-10B	16	1.0	0.88
ORG-11	15	1.0	0.90
ORG-12	16	1.0	1.0
ORG-13	16	1.0	1.0
ORG-14	16	1.0	0.91
ORG-15	16	1.0	1.0
ORG-16	17	1.0	1.1
ORG-17	16	1.0	1.1
ORG-18	15	1.0	0.97
ORG-19	15	1.0	1.1
ORG-20	15	1.0	0.97
ORG-20B	16	1.0	0.94
ORG-21	15	1.0	0.96
ORG-22	15	1.0	1.0
ORG-23	16	1.0	1.0
ORG-24	15	1.0	1.0
ORG-25	15	1.0	0.95
ORG-26	16	1.0	0.88
ORG-27	16	1.0	1.4
ORG-28	16	1.0	0.94
ORG-29	16	1.0	1.1
ORG-30	16	1.0	0.92
ORG-30B	16	1.0	0.92

Appendix B cont.

WDXRF

Sample ID	SiO <sub>2</sub> , %	Al <sub>2</sub> O <sub>3</sub> , %	Fe <sub>2</sub> O <sub>3</sub> , %	MgO, %	CaO, %	Na <sub>2</sub> O, %	K <sub>2</sub> O, %	TiO <sub>2</sub> , %	P <sub>2</sub> O <sub>5</sub> , %	MnO, %	LOI, %
ORG-06	62.3	11.6	5.77	1.97	3.67	0.75	2.39	0.76	0.23	0.08	9.20
ORG-07	62.5	11.7	5.82	2.01	3.70	0.78	2.40	0.76	0.23	0.08	9.23
ORG-08	62.2	11.6	5.81	2.00	3.70	0.79	2.42	0.76	0.24	0.08	9.30
ORG-09	62.5	11.6	5.78	1.99	3.70	0.81	2.40	0.76	0.23	0.08	9.18
ORG-10	62.4	11.7	5.82	1.99	3.71	0.78	2.40	0.77	0.24	0.08	9.34
ORG-10B	62.4	11.7	5.80	2.01	3.69	0.79	2.41	0.77	0.24	0.08	9.23
ORG-11	62.6	11.7	5.81	2.01	3.70	0.79	2.40	0.76	0.24	0.08	9.19
ORG-12	62.5	11.6	5.81	1.98	3.69	0.78	2.40	0.76	0.24	0.08	9.20
ORG-13	62.6	11.6	5.81	1.98	3.70	0.79	2.40	0.76	0.23	0.08	9.08
ORG-14	62.5	11.6	5.81	1.99	3.68	0.80	2.41	0.76	0.23	0.08	9.11
ORG-15	62.6	11.6	5.79	1.98	3.71	0.77	2.40	0.76	0.24	0.08	9.20
ORG-16	63.0	11.6	5.85	1.98	3.69	0.79	2.40	0.77	0.23	0.08	9.14
ORG-17	62.6	11.6	5.78	1.97	3.68	0.78	2.41	0.77	0.23	0.08	9.24
ORG-18	62.3	11.6	5.82	1.97	3.69	0.78	2.41	0.76	0.23	0.08	9.12
ORG-19	62.8	11.7	5.81	1.98	3.71	0.78	2.41	0.77	0.23	0.08	9.04
ORG-20	62.5	11.6	5.78	2.02	3.68	0.77	2.41	0.76	0.23	0.08	9.05
ORG-20B	63.0	11.7	5.81	1.99	3.69	0.78	2.40	0.76	0.23	0.08	9.17
ORG-21	62.6	11.6	5.76	1.96	3.70	0.78	2.42	0.76	0.23	0.08	9.16
ORG-22	62.3	11.6	5.81	1.98	3.69	0.77	2.40	0.76	0.23	0.08	9.04
ORG-23	63.0	11.7	5.83	2.00	3.70	0.75	2.42	0.76	0.23	0.08	9.16
ORG-24	62.6	11.7	5.83	1.99	3.70	0.79	2.40	0.77	0.24	0.08	9.10
ORG-25	62.4	11.7	5.79	1.98	3.71	0.78	2.42	0.77	0.23	0.08	9.18
ORG-26	62.5	11.6	5.78	1.98	3.70	0.80	2.40	0.75	0.24	0.08	9.15
ORG-27	62.5	11.6	5.81	2.00	3.68	0.78	2.39	0.76	0.23	0.08	9.07
ORG-28	62.6	11.6	5.80	2.01	3.69	0.79	2.40	0.77	0.24	0.08	9.09
ORG-29	62.4	11.7	5.79	1.98	3.68	0.78	2.40	0.76	0.23	0.08	9.20
ORG-30	62.3	11.5	5.76	1.98	3.67	0.78	2.39	0.75	0.23	0.08	9.03
ORG-30B	62.5	11.6	5.76	1.99	3.70	0.80	2.39	0.76	0.23	0.08	9.20

Appendix C Results for SRM 2704-organic/sterilized on dry weight basis, by ICP-AES, WDXRF, HG-AAS and CV-AAS

ICP-AES													
Sample ID	Al, %	Ca, %	Fe, %	K, %	Mg, %	Na, %	P, %	Ti, %					
Ster-1	6.1	2.6	4.1	2.0	1.2	0.60	0.11	0.33					
Ster-2A	6.2	2.6	4.2	2.0	1.3	0.62	0.11	0.32					
Ster-2B	6.1	2.6	4.1	2.0	1.2	0.62	0.10	0.32					
Ster-3	6.0	2.6	4.1	2.0	1.2	0.61	0.10	0.32					
Ster-4	6.1	2.6	4.1	2.0	1.2	0.61	0.11	0.33					
Ster-5A	6.1	2.6	4.1	2.0	1.2	0.61	0.11	0.32					
Ster-5B	6.0	2.6	4.1	2.0	1.2	0.61	0.10	0.31					
Sample ID	Ag	As	Ba	Be	Bi	Cd	Ce	Co	Cr	Cu			
Ster-1	<2	19	410	1.7	<10	2.9	59	16	120	91			
Ster-2A	<2	21	420	1.8	<10	3.1	60	16	120	88			
Ster-2B	<2	22	410	1.7	<10	2.9	57	15	120	90			
Ster-3	<2	20	410	1.7	<10	3.0	57	15	120	89			
Ster-4	<2	23	410	1.8	<10	3.0	58	15	120	97			
Ster-5A	<2	18	410	1.7	<10	2.9	59	16	120	95			
Ster-5B	<2	19	400	1.7	<10	3.0	58	15	120	95			
Sample ID	Eu	Ga	La	Li	Mn	Mo	Nb	Nd	Ni	Pb			
Ster-1	<2	15	29	50	574	<2	8.5	28	41	140			
Ster-2A	<2	15	29	50	574	<2	8.2	29	46	140			
Ster-2B	<2	14	29	49	580	<2	8.8	29	41	150			
Ster-3	<2	14	28	49	576	<2	8.7	28	41	150			
Ster-4	<2	16	29	49	586	<2	9.2	29	42	150			
Ster-5A	<2	16	29	49	572	2.5	9.2	29	43	140			
Ster-5B	<2	16	28	49	578	<2	7.7	30	41	140			
Sample ID	Sc	Sn	Sr	Ta	Th	U	V	Y	Yb	Zn			
Ster-1	12	5	136	<40	9	<100	95	24	2.2	400			
Ster-2A	12	5	139	<40	10	<100	95	24	2.4	400			
Ster-2B	12	6	136	<40	10	<100	94	24	2.2	400			
Ster-3	12	6	136	<40	10	<100	94	24	2.3	420			
Ster-4	12	8	137	<40	9	<100	95	24	2.4	400			
Ster-5A	12	8	136	<40	11	<100	94	24	2.8	410			
Ster-5B	12	5	135	<40	11	<100	94	24	2.7	390			

Appendix C cont.

WDXRF Sample ID	SiO <sub>2</sub> %	Al <sub>2</sub> O <sub>3</sub> %	Fe <sub>2</sub> O <sub>3</sub> %	MgO %	CaO %	Na <sub>2</sub> O %	K <sub>2</sub> O %	TiO <sub>2</sub> %	P <sub>2</sub> O <sub>5</sub> %	MnO %	LOI %
Ster-1	62.2	11.5	5.80	1.98	3.69	0.76	2.40	0.76	0.24	0.08	9.04
Ster-2A	62.1	11.6	5.79	1.97	3.68	0.74	2.42	0.76	0.24	0.08	9.09
Ster-2B	62.3	11.6	5.87	1.99	3.70	0.76	2.41	0.76	0.24	0.08	9.10
Ster-3	62.2	11.6	5.79	1.97	3.69	0.76	2.41	0.76	0.24	0.08	9.19
Ster-4	62.2	11.5	5.80	1.96	3.68	0.76	2.41	0.75	0.24	0.08	9.09
Ster-5A	62.2	11.6	5.84	1.99	3.69	0.74	2.42	0.77	0.23	0.08	9.06
Ster-5B	62.3	11.6	5.93	1.99	3.67	0.77	2.41	0.76	0.24	0.08	9.00

HG-AAS Sample ID	As μg/g	Se μg/g
Ster-1	17	1.1
Ster-2A	16	1.1
Ster-2B	17	1.1
Ster-3	16	1.1
Ster-4	17	1.1
Ster-5A	17	1.0
Ster-5B	17	1.1

CV-AAS Sample ID	Hg μg/g
Ster-1	1.2
Ster-2A	0.93
Ster-2B	1.1
Ster-3	0.95
Ster-4	0.96
Ster-5A	1.4
Ster-5B	1.0

Appendix D Analytical results for SRM 2704-organic from second test group, analysis by ICP-AES, WDXRF, HG-AAS, and CV-AAS

ICP-AES												
Sample ID	Al %	Ca %	Fe %	K %	Mg %	Na %	P %	Ti %				
Organ-11	6.1	2.6	4.2	2.1	1.3	0.62	0.11	0.36				
Organ-19	6.2	2.6	4.2	1.9	1.3	0.62	0.11	0.33				
Organ-27	6.1	2.6	4.2	2.0	1.3	0.62	0.11	0.33				
Sample ID	Ag $\mu\text{g/g}$	As $\mu\text{g/g}$	Ba $\mu\text{g/g}$	Be $\mu\text{g/g}$	Bi $\mu\text{g/g}$	Cd $\mu\text{g/g}$	Ce $\mu\text{g/g}$	Co $\mu\text{g/g}$	Cr $\mu\text{g/g}$	Cu $\mu\text{g/g}$		
Organ-11	<2	22	416	1.8	<10	3.1	62	16	124	91		
Organ-19	<2	22	415	1.8	<10	3.2	58	15	126	89		
Organ-27	<2	20	411	1.7	<10	3.1	60	15	128	95		
Sample ID	Ga $\mu\text{g/g}$	La $\mu\text{g/g}$	Li $\mu\text{g/g}$	Mn $\mu\text{g/g}$	Mo $\mu\text{g/g}$	Nb $\mu\text{g/g}$	Nd $\mu\text{g/g}$	Ni $\mu\text{g/g}$	Pb $\mu\text{g/g}$	Sc $\mu\text{g/g}$		
Organ-11	15	30	49	584	<2	9	29	42	142	12		
Organ-19	15	28	49	588	<2	8	28	42	144	12		
Organ-27	15	29	49	582	2.0	9	30	42	158	11		
Sample ID	Sn $\mu\text{g/g}$	Sr $\mu\text{g/g}$	Ta $\mu\text{g/g}$	Th $\mu\text{g/g}$	U $\mu\text{g/g}$	V $\mu\text{g/g}$	Y $\mu\text{g/g}$	Yb $\mu\text{g/g}$	Zn $\mu\text{g/g}$			
Organ-11	4.6	138	<40	9	<100	96	24	2.2	409			
Organ-19	6.7	140	<40	10	<100	95	24	2.2	409			
Organ-27	6.2	138	<40	9	<100	95	24	2.6	405			
WDXRF												
Sample ID	SiO <sub>2</sub> %	Al <sub>2</sub> O <sub>3</sub> %	Fe <sub>2</sub> O <sub>3</sub> %	MgO %	CaO %	Na <sub>2</sub> O %	K <sub>2</sub> O %	TiO <sub>2</sub> %	P <sub>2</sub> O <sub>5</sub> %	MnO %	LOI %	
Organ-11	62.0	11.6	5.80	2.00	3.71	0.77	2.40	0.76	0.24	0.08	9.19	
Organ-19	62.4	11.5	5.83	1.93	3.68	0.77	2.39	0.75	0.24	0.08	8.98	
Organ-27	62.2	11.5	5.84	1.95	3.67	0.76	2.40	0.76	0.23	0.08	9.00	
HG-AAS and CV-AAS												
Sample ID	As $\mu\text{g/g}$	Se $\mu\text{g/g}$	Hg $\mu\text{g/g}$									
Organ-11	16	1.1	1.1									
Organ-19	17	1.0	0.94									
Organ-27	18	1.0	1.0									



Appendix D cont.

ICP-AES													
Sample ID	Al %	Ca %	Fe %	K %	Mg %	Na %	P %	Ti %					
Inorg-1	6.1	2.6	4.2	2.0	1.2	0.62	0.11	0.33					
Inorg-2	6.1	2.6	4.2	2.0	1.2	0.60	0.11	0.33					
Inorg-5	6.2	2.6	4.3	2.1	1.3	0.62	0.11	0.37					
Sample ID	Ag $\mu\text{g/g}$	As $\mu\text{g/g}$	Ba $\mu\text{g/g}$	Be $\mu\text{g/g}$	Bi $\mu\text{g/g}$	Cd $\mu\text{g/g}$	Ce $\mu\text{g/g}$	Co $\mu\text{g/g}$	Cr $\mu\text{g/g}$	Cu $\mu\text{g/g}$			
Inorg-5	<2	25	418	1.8	<10	3.6	62	16	142	96			
Inorg-1	<2	28	416	1.7	<10	3.4	58	16	137	99			
Inorg-2	<2	26	404	1.7	<10	3.7	58	16	138	99			
Sample ID	Eu $\mu\text{g/g}$	Ga $\mu\text{g/g}$	La $\mu\text{g/g}$	Li $\mu\text{g/g}$	Mn $\mu\text{g/g}$	Mo $\mu\text{g/g}$	Nb $\mu\text{g/g}$	Nd $\mu\text{g/g}$	Ni $\mu\text{g/g}$	Pb $\mu\text{g/g}$			
Inorg-1	<2	15	29	50	588	<2	7.9	28	42	150			
Inorg-2	<2	15	28	50	585	2.5	9.1	27	42	160			
Inorg-5	<2	16	30	51	598	2.0	9.4	30	44	158			
Sample ID	Sc $\mu\text{g/g}$	Sn $\mu\text{g/g}$	Sr $\mu\text{g/g}$	Ta $\mu\text{g/g}$	Th $\mu\text{g/g}$	U $\mu\text{g/g}$	V $\mu\text{g/g}$	Y $\mu\text{g/g}$	Yb $\mu\text{g/g}$	Zn $\mu\text{g/g}$			
Inorg-1	12	6	137	<40	9	<100	95	24	2.3	433			
Inorg-2	12	7	134	<40	10	<100	95	24	2.8	441			
Inorg-5	12	5	138	<40	10	<100	97	25	2.3	439			

WDXRF													
Sample ID	SiO <sub>2</sub> %	Al <sub>2</sub> O <sub>3</sub> %	Fe <sub>2</sub> O <sub>3</sub> %	MgO %	CaO %	Na <sub>2</sub> O %	K <sub>2</sub> O %	TiO <sub>2</sub> %	P <sub>2</sub> O <sub>5</sub> %	MnO %	LOI %		
Inorg-1	61.9	11.7	5.89	1.97	3.64	0.75	2.41	0.76	0.25	0.08	9.12		
Inorg-2	62.0	11.6	5.94	1.99	3.67	0.77	2.43	0.77	0.24	0.08	9.12		
Inorg-5	61.8	11.7	5.91	1.97	3.65	0.75	2.43	0.76	0.24	0.08	9.18		

HG-AAS and CV-AAS				
Sample ID	As $\mu\text{g/g}$	Se $\mu\text{g/g}$	Hg $\mu\text{g/g}$	
Inorg-1	23	1.1	1.5	
Inorg-2	22	1.1	1.5	
Inorg-5	23	1.1	1.4	