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The physical preparation and chemical analysis of NIST SRMs 2786 and 2787, lead paint in soil

by

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INTRODUCTION

Under a cooperative agreement between the U. S. Geological Survey (USGS), the National Institute for Standards and Technology (NIST), and the Environmental Protection Agency (EPA) a project was undertaken to prepare two soil reference materials contaminated with different concentrations of lead. The standard reference materials (SRMs) were requested by EPA for use in their lead abatement program and will compliment NIST dust and paint SRMs currently under development. This cooperative study will provide EPA with the standards necessary to properly monitor its lead abatement program which is designed to quantify the presence of lead in the residential environment. Reliable information on the quantity and type of lead is important in developing appropriate remediation plans.

At the beginning of the study it was determined that the material used in the preparation of these soil standards should be from an urban or suburban environment, where the principal source of lead is believed to be old house paint. This form of lead is different from lead in existing NIST soil SRMs, (2709-2711) which is derived primarily from geologic sources. Lead concentrations in the proposed SRMs were targeted at 400 µg/g and 3000 µg/g in order to compliment the existing NIST soil SRMs.

SRM COLLECTION

SRM 2586

Soil material used in the preparation of SRM 2586 was collected from the Baltimore, Maryland area. Selection of the material used in this project was coordinated through Ms. Rebecca Fahy of the Maryland Department of Public Health (MD-DPH). Soil material for the SRM was derived from approximately 20,000 samples collected as part of the EPA three cities study (TCS) conducted in 1990 (EPA report 1993). The 20,000 samples were transported directly to the USGS and then transferred to a series of 10 five-gallon plastic containers. The combined sample weight was approximately 280 kg. Soil samples were classified by the MD-DPH into two particle sizes (<60 and <100 mesh) and used in this project without additional drying or particle separation. The soil samples varied in color, but could be separated into four major groups; white, brown, red, and black.

SRM 2587

Material used in the preparation of SRM 2587 was obtained from the Hartford, Connecticut area. A suburban home site previously identified by EPA as having elevated lead concentrations in garden soils was selected as the collection

site. Soil material was obtained from two locations immediately adjacent to the front of the house. The soil material was collected within the top 25 cm after surficial organic material had been removed. Visual inspection of the soil material revealed that it was dark brown to black in color and contained moderate quantities of partially decomposed organic matter as well as minor amount of masonry debris. A total of 14 five-gallon plastic containers were obtained for an estimated total weight of 350 kg. Field measurements using a portable energy dispersive X-ray fluorescence instrument indicated that the lead concentration in the soil material ranged between 2000 and 20000 μg/g. Soil material (80 kg) with background lead concentration of <100 μg/g, was later collected from the Boston, Massachusetts area to serve as a diluent.

Soil material used in the preparation of SRM 2587 was initially dried at room temperature for 5 days in a series of 5 x 30 x 50 cm cardboard trays. After drying, the soil was lightly ground using a mechanical disaggregator equipped with a ceramic auger and mortar. The disaggregated soil material was sieved and material with a particle size <2 mm was collected in 5 gallon plastic containers for further processing.

SRM PREPARATION

In the SRM preparation procedure, soil from each of the five gallon containers was initially analyzed to determine the proper material to use in the final preparation step. The contents of each five gallon container was blended for 1 hour in a 3 ft^3 V-blender and then a cross-sectional sample was obtained. Ten samples from SRM 2586 and 14 samples from SRM 2587 were collected in this manner and submitted to the lab for chemical analysis. Results from these analyses were then used to mathematically select those containers that when combined would approximate the target lead concentrations of 400 μg/g (SRM 2586) and 3000 μg/g (SRM 2587). The material finally selected for each SRM was ground to <200 mesh (75μ) using a Hardinger ball mill equipped with an air separator system. The 250 Kg of final product was then mixed for 20 hours using a 10ft^3 cross-flow V-blender. After blending, a series of ten samples were collected and analyzed for their total element concentration. In a separate study to determine the effect of blending time, samples of SRM 2586 were collected after 5, 10, 15, and 20 hours of mixing.

STERILIZATION AND BOTTLING

Prior to soil sterilization the SRM material was split into 8 kg aliquots and transferred to cardboard boxes lined with plastic bags. The material was taken to the radiation sterilization facility located at COBE laboratory, Lakewood, Colorado. The material was sterilized using Co-60 irradiation and received a radiation dosage between 3.9 and 6.7 Mrad. After sterilization, the material was returned to the USGS facility and reblended for 3 hours in the $10ft^3$ blender. Aliquots (55±2 g) of the final product were then transferred to glass bottles supplied by NIST. When filled, the bottles were returned to their shipping boxes, with the first and last bottle in each of the two layers labeled "start" and "end" respectively. When the bottling was complete the boxes were mailed back to NIST for further testing and distribution. During the bottling process every 50th bottle was set aside in a special USGS sample subset (E50B) to be used in future testing.

CHEMICAL ANALYSIS

Samples analyzed for between-bottle homogeneity tests were dried at 110°C for two hours prior to analysis. Procedures used in the analysis of SRM 2586 and 2587 have been previously described (Arbogast, B.F., 1990) and will be summarized below

Inductively Coupled Plasma - Atomic Emission Spectroscopy (ICP-AES)

In the ICP-AES procedure, a 0.200-g sample is transferred to a 30-ml teflon beaker and 50 μg of lutetium (Lu_2O_3 in 5% HCl solution) added as an internal standard. The sample is digested to dryness overnight using concentrated HCl (3mL), HNO_3 (2 mL), $HClO_4$ (1 mL), and HF (2 mL). The residue is redigested using additional $HClO_4$ (1 mL), and water at 150°C and taken to dryness again. One milliliter of aqua regia is added to the container, and the sample is diluted to 10.00 g with 1% HNO_3. Solutions were analyzed for 40 elements simultaneously using a Jarrell Ash model 1160 spectrometer.

Wavelength Dispersive X-ray Fluoresence (WDXRF)

In the WDXRF procedure, a 0.8000 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% solution of lithium bromide, which serves as a nonwetting agent. The crucible is then placed in a muffle furnace on an "automatic fluxer", and the sample is fused and homogenized for 45 minutes at 1120°C. After the fusion is complete, the molten mixture is poured in a specially designed two-piece platinum mold. When cooled, the resulting glass disc is analyzed for its major 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.

Cold Vapor Atomic Absorption spectrophotometry (CV-AAS)

In the CV-AAS procedure a 0.100 g sample is transferred to a 16x100 mm disposable borosilicate test tube and 2.0 ml of 16M HNO_3 added along with 0.50 ml of a 25% (w/v) sodium dichromate solution to initiate the sample decomposition. The test tube is place in a specially designed aluminum hot block for three hours at 110 °C. After heating, the sample is removed from the heating block, allowed to cool overnight and then diluted to 12 ml with deionized water. The tube is capped and the contents shaken. The solution is finally analyzed in a continuous flow procedure using a Perkin Elmer model 3030B atomic absorption spectrophotometer.

Hydride Generation-Atomic Absorption spectroscopy

In the HG-AAS procedure a 0.25 g sample is transferred to a 30-ml teflon bomb and decomposed overnight at 105 °C, using a combination of HNO_3, H_2SO_4, $HClO_4$, and HF. The next day, 25 ml of 6M HCl is added to the digestion solution (2 ml) and the contents allowed to stand for half an hour. The solution is then transferred to a tared 60-ml polyethylene bottle and brought to a final mass of 55 g with deionized water. An 8 ml aliquot of the final solution is transferred to a 13x80 mm disposable test tube. The solution is then analyzed by flow injection procedure using a Perkin Elmer model 2100 atomic absorption spectrophotometer.

Inductively coupled plasma-mass spectroscopy (ICP-MS)

In this procedure a sample (0.100 g) is first decomposed using a sodium peroxide (0.500 g) sinter in a 5 ml zirconium crucible at 445 °C for 30 minutes. After sintering is complete and the crucible has cooled, the sinter cake is transferred to a 60 ml polyethylene bottle and 20 ml of deionized water are added. The container is capped, the contents mixed, and the sample

stored until the day of analysis. On the day of analysis, 20 ml of 15% (v/v) nitric acid is added to the polyethylene container and the contents thoroughly mixed. A 10 g sample is transferred to a 17x100 mm polyethylene test tube and 100 μL of a rare earth internal standard added. The test tube is then capped and the contents vigorously shaken. Samples are finally analyzed for rare earth elements on a Sciex Elan model 250 ICP/MS.

RESULTS AND DISCUSSION

Element concentration ranges for the original soil samples collected from the Baltimore, and Hartford areas are presented in table 1, along with results for the Boston area soil which served as a diluent in the preparation of SRM 2587. Comparison of element concentrations in the Baltimore and Hartford samples with values for eastern United States soils (Boegeron and Schaklette, 1984) reveal that most element concentrations are within the 95% confidence interval for eastern United States soils. Exceptions are noted for the maximum concentrations of chromium (Cr), nickel (Ni), and scandium (Sc) in SRM 2586 and all copper (Cu) concentrations in SRM 2587. In both SRMs even the minimum concentrations of lead (Pb) and zinc (Zn) exceed the 95% confidence interval for eastern United States soils. Element concentrations in the Boston metropolitan soil samples are typically within the 95% confidence interval for eastern United States soils except for Pb and neodymium (Nd) which are greater and less than that range respectively.

Analytical results for the blending homogeneity tests (n=10) are summarized in tables 2 and 3 for SRM 2586 and SRM 2587 respectively. Major element analysis for both SRMs by WDXRF indicate the SRMS were homogeneous, based on between-sample precision (percent relative standard deviation) of <3% which is within the limits of the method. ICP-AES results generally confirm the SRM homogeneity with %RSD for most elements of less than 5%. Higher %RSD are noted for elements near their respective determination limits (Cd, Th, and Yb in SRM 2586 and As, Th, and Yb in SRM 2587). Based on the excellent analytical precision observed for the major and trace elements in both SRMs, the standards were approved for bottling.

Analytical results for the between-bottle homogeneity tests for SRM 2586 and SRM 2587 are presented in appendices A and B, with summarized results presented in tables 4 and 5. For each SRM, results are based on the analysis of 20 randomly selected bottles obtained from the USGS sample subset E50B. Trace and

major element results for SRM 2586 by the ICP-AES and WDXRF procedures reveal a %RSD of less than 3% for most elements. Slightly higher %RSDs are noted for Be, Ga, Nb, and Th which is attributed to the poorer precision of the ICP procedure near element determination limits. Graphical examination of the lead concentration results versus bottle number in figures 1 and 2 showed no apparent trends in the data, suggesting that no significant partitioning of the standards had occurred over the seven day bottling process. Similar distributions are noted for all the other trace and major elements.

Because both reference materials were designed to have lead concentrations which complimented existing NIST SRMs, the final concentrations of lead in SRM 2586 and 2587 were contractually targeted at 400 and 3000 μg/g ±15% respectively. Examination of the average lead concentrations in tables 4 and 5 reveals that based on USGS analyses the final concentrations of 420 and 3250 μg/g are within 9% of the target values.

In addition to the anomalous concentrations of lead and zinc in the two SRMs, concentrations of tin are also believed to be elevated compared to the mean value (0.86 μg/g) for eastern United States soils. Tin concentrations ranged between <5 and 10 μg/g for both SRMs. Average tin concentrations are not reported because of the potential for an incomplete decomposition of tin minerals using acid decompositions procedures similar to the USGS method (Dolezal J., 1966, Johnson, W., and Maxwell, J., 1981). Decompositions by a sodium peroxide fusion or sinter are recommended if reliable tin concentrations by ICP-AES are required.

To determine if blending time played a role in element homogeneity a separate study was carried out during the blending of SRM 2586 which was based on the analysis of samples collected after 5, 10, 15, and 20 hours of blending. Statistical analysis of the major and trace element data indicate the one can accept the hypothesis that there is no significant difference in element concentrations with increased blending time at the 95% confidence interval. Based on these limited observations it is apparent that materials of similar composition (soils) ground initially to <75 μm can be effectively homogenized in as little as 5 hours of blending with equipment currently in use at the USGS.

To test the accuracy of USGS between-bottle results for SRM 2586, a sample of SRM 2711 was included in the sample set analyzed. SRM 2711 was also analyzed in the between-bottle study for SRM 2587, but only by ICP-AES. The comparison of USGS results for SRM 2711 with NIST certified and recommended values are reported in table 6. Examination of table 6 reveals that all of the USGS major element results for SRM 2711 by both ICP-AES

and WDXRF are within ± 3σ of the NIST recommended value. Exceptions are noted for Na and K in SRM 2586 and 2587 respectively. In the case of trace elements, USGS values are in general agreement with NIST certified and information values, but only Ba, Mn, Pb, and V are consistently within the ± 3σ boundary.

During the bottling process, the presence of black fibers were occasionally observed protruding from the cap's thread area. These fibers were similar in composition to the cap material and appeared to be the result of an incomplete threading process. The fibers ranged in length from 1 to 3 cm and were typically 0.05 mm in diameter. When observed, the fibers were removed by hand or blown off the cap using compressed air. To determine if these fibers were consistently present in the bottled SRMs a test was performed on both SRMs using four randomly selected samples from the USGS sample subsets E50B. The contents of each bottle were sieved for twenty minutes through an 80 mesh screen. After all the soil material had passed through the sieve, the screen surface was carefully inspected. Fibers similar to that seen on the bottle caps were not detected in any of the samples tested.

BIBLIOGRAPHY

Arbogast, B.F. 1990, Quality assurance manual for the Branch of Geochemistry, U.S. Geological Survey, USGS Open File Report 90-668, 177 p.

Dolezal J., Povondra, P., and Sulcek Z., 1966, Decomposition techniques in inorganic analysis, London Iliffe Books LTD., New York American Elsevier Publishing Co. Inc. 225 p.

EPA/600/AP-93/00/c, 1993, Urban soil lead abatement demonstration project, vol 3: part 1, Baltimore report.

Johnson, W., and Maxwell, J., 1981, Rock and Mineral Analysis, John Wiley and Sons, New York, 489 p.

Shacklette, H.T., and Boerngen, J.G., 1984, Element Concentrations in soils and other surficial materials of the conterminous United States, USGS Professional Paper 1270, 105 p.

Table 1. Element concentration ranges in soils from the Baltimore Maryland, Hartford Conneticut, and Boston Massachusetts locations

	SRM 2586		SRM 2587		
	BALTIMORE		HARTFORD		BOSTON
Element	min[1]	max[2]	min[1]	max[2]	Obs[3]
Al, %	6.3	7.3	6.3	7.1	4.2
Ca, %	1.8	3.0	0.7	0.99	0.74
Fe, %	4.4	5.7	3.0	3.1	1.2
K , %	0.72	1.3	1.6	1.7	1.6
Mg, %	1.3	2.5	0.71	0.84	0.21
Na, %	0.38	0.68	1.2	1.3	1.4
P, %	0.08	0.13	0.12	0.16	0.068
Ti, %	0.48	0.61	0.43	0.55	0.18
Ag, ppm	<2	<2	<2	<2	<2
As, ppm	<10	11	<10	28	<10
Au, ppm	<8	<8	<8	<8	<8
Ba, ppm	320	460	690	1300	350
Be, ppm	1	2	2	2	2
Bi, ppm	<10	<10	<10	<10	<10
Cd, ppm	<2	2	<2	<2	<2
Ce, ppm	49	69	54	73	46
Co, ppm	26	49	13	16	3
Cr, ppm	140	420	66	78	20
Cu, ppm	61	83	200	410	13
Eu, ppm	<2	<2	<2	<2	<2
Ga, ppm	15	18	14	20	9
La, ppm	26	36	30	40	24
Li, ppm	23	27	46	58	12
Mn, ppm	820	1200	750	900	340
Mo, ppm	<2	<2	<2	<2	<2
Nb, ppm	12	16	13	17	8
Nd, ppm	23	33	27	39	17
Ni, ppm	46	120	29	35	8
Pb, ppm	260	610	4200	21000	76
Sc, ppm	20	27	12	14	4
Sn, ppm	8	23	<5	59	<5
Sr, ppm	80	110	120	140	140
Ta, ppm	<40	<40	<40	<40	<40
Th, ppm	6	8	<4	11	7
U, ppm	<100	<100	<100	<100	<100
V, ppm	140	170	110	130	24
Y, ppm	17	26	20	24	9
Yb, ppm	2	3	<1	2	<2
Zn, ppm	280	440	410	1700	34

[1] Minimim
[2] Maximum
[3] Observed, single analysis

Table 2. Blending homogeniety results for SRM 2586

	ICP-AES			WDXRF		
Element	Avg[1]	STDS[2]	%RSD[3]	Avg[1]	STDS[2]	%RSD[3]
Al, %	6.58	0.042	0.64	6.52	0.023	0.36
Ca, %	2.1	<0.05	-	2.15	0.005	0.22
Fe, %	4.99	0.032	0.63	5.06	0.008	0.16
K, %	0.959	0.003	0.33	0.963	0.006	0.61
Mg, %	1.65	0.053	3.2	1.65	0.012	0.73
Na, %	0.513	0.005	0.94	0.42	0.009	2.18
P, %	0.092	0.004	4.6	0.105	0.002	2.08
Ti, %	0.548	0.004	0.77	0.623	0.003	0.48
Si, %	-	-	-			
Ag, ppm	<2	-	-			
As, ppm	<10	-	-			
Ba, ppm	404	5.2	1.3			
Be, ppm	3	<0.5	-			
Bi, ppm	<10	-	-			
Cd, ppm	2	0.3	15			
Ce, ppm	55	1.2	2.2			
Co, ppm	36	0.5	1.3			
Cr, ppm	300	8.2	2.7			
Cu, ppm	75	3.6	4.8			
Eu, ppm	<2	-	-			
Ga, ppm	16	1.0	6.2			
La, ppm	31	0.4	1.4			
Li, ppm	25	0.3	1.3			
Mn, ppm	966	7.0	0.7			
Mo, ppm	<2	-	-			
Nb, ppm	16	0.5	3.1			
Nd, ppm	28	1.3	4.7			
Ni, ppm	70	1.1	1.5			
Pb, ppm	410	6.3	1.6			
Sc, ppm	23	<0.5	-			
Sn, ppm	14	4.	28			
Sr, ppm	85	0.5	0.57			
Th, ppm	8	1.1	14			
V, ppm	160	3.	1.9			
Y, ppm	21	<0.5	-			
Yb, ppm	2	0.3	15.			
Zn, ppm	320	3.2	1.0			

[1] Average
[2] Standard Deviation
[3] Percent Relative Standard Deviation

Table 3. Blending homogeniety results for SRM 2587

	ICP-AES			WDXRF		
Element	Avg[1]	STDS[2]	%RSD[3]	Avg[1]	STDS[2]	%RSD[3]
Al, %	5.7	0.052	0.9	5.87	0.02	0.29
Ca, %	0.9	0.005	0.6	0.91	<0.005	-
Fe, %	2.8	<0.05	-	2.78	0.01	0.30
K, %	1.5	0.042	2.8	1.59	0.01	0.33
Mg, %	0.7	0.005	0.8	0.66	0.01	0.77
Na, %	1.1	<0.05	-	1.03	0.01	0.99
P, %	0.1	0.003	3.5	0.10	<0.005	-
Ti, %	0.4	0.005	1.4	0.42	<0.005	-
Si, %	-	-	-	32.7	0.05	0.15
Ag, ppm	<2	-	-			
As, ppm	13	1.4	11			
Au, ppm	<8	-	-			
Ba, ppm	560	6.7	1.2			
Be, ppm	11	<0.5	-			
Bi, ppm	<10	-	-			
Cd, ppm	<2	-	-			
Ce, ppm	61	1.8	3.0			
Co, ppm	14	0.5	3.9			
Cr, ppm	94	1.5	1.6			
Cu, ppm	160	7.0	4.5			
Eu, ppm	<2	-	-			
Ga, ppm	14	0.5	3.9			
Ho, ppm	<4	-	-			
La, ppm	33	0.9	2.9			
Li, ppm	33	0.5	1.5			
Mn, ppm	650	7.0	1.1			
Mo, ppm	<2	-	-			
Nb, ppm	14	0.5	3.4			
Nd, ppm	28	0.9	3.3			
Ni, ppm	35	0.9	2.7			
Pb, ppm	3090	32	1.0			
Sc, ppm	11	<0.5	-			
Sr, ppm	130	<0.5	-			
Ta, ppm	<40	-	-			
Th, ppm	10	1.0	10			
U, ppm	<100	-	-			
V, ppm	82	0.5	0.6			
Y, ppm	16	0.5	3.1			
Yb, ppm	2	0.3	17			
Zn, ppm	330	5.2	1.6			

[1] arithmetic average
[2] standard deviation
[3] percent relative standard deviation

Table 4. Between-bottle homogeniety results for SRM 2586 (n=20)

	ICP-AES			WDXRF		
Element	Avg[1]	STDS[2]	%RSD[3]	Avg[1]	STDS[2]	%RSD[3]
Al, %	6.70	0.12	1.73	6.63	0.018	0.27
Ca, %	2.26	0.02	1.08	2.19	0.004	0.18
Fe, %	5.24	0.03	0.55	5.16	0.015	0.29
K, %	0.96	0.02	1.65	0.98	0.003	0.33
Mg, %	1.77	0.03	1.48	1.69	0.007	0.41
Na, %	0.52	0.01	1.73	0.41	0.011	2.56
P, %	0.10	0.00	1.04	0.11	0.002	2.09
Ti, %	0.54	0.01	1.71	0.64	0.004	0.57
Si, %	-	-	-	29.11	0.057	0.19
Ag, ppm	<2	-	-			
As, ppm	<10	-	-			
Ba, ppm	404	0.0	1.2			
Be, ppm	1.4	0.2	14			
Cd, ppm	<2	-	-			
Ce, ppm	52	0.71	1.4			
Co, ppm	35	0.78	2.2			
Cr, ppm	329	6.7	2.0			
Cu, ppm	81	1.3	1.6			
Ga, ppm	14	0.83	5.9			
La, ppm	26	0.56	2.1			
Li, ppm	25	0.32	1.3			
Mn, ppm	1004	9.5	0.95			
Nb, ppm	6	0.61	10			
Nd, ppm	25	0.47	1.9			
Ni, ppm	75	1.1	1.5			
Pb, ppm	420	7.8	1.8			
Sc, ppm	24	0.43	1.8			
Sr, ppm	85	1.4	1.6			
Th, ppm	7	0.45	6.8			
V, ppm	160	2.7	1.7			
Y, ppm	21	0.31	1.5			
Yb, ppm	2	0.03	1.5			
Zn, ppm	360	4.3	1.2			

ICP-MS			
Element	Avg	STDS	%RSD
Ce, ppm	64	4.4	6.9
Dy, ppm	5.4	0.2	4.5
Er, ppm	3.3	0.15	4.5
Eu, ppm	1.5	0.09	6.1
Gd, ppm	5.8	0.31	5.4
Ho, ppm	1.1	0.05	4.8
La, ppm	33	2.4	7.2
Nd, ppm	28	1.9	6.9
Pr, ppm	7.3	0.5	6.7
Sm, ppm	6.1	0.36	6.0
Tb, ppm	0.9	0.06	6.2
Tm, ppm	0.5	0.02	4.5
Yb, ppm	3.1	0.14	4.4

MISC.			
Element	Avg[1]	STDS[2]	%RSD[3]
As, ppm	8.5	0.12	1.5
Se, ppm	0.6	<0.01	-
Hg, ppm	0.31	0.01	2.8

[1] arithmetic average
[2] standard deviation
[3] percent relative standard deviation

Table 5. Between-bottle homogeniety results for SRM 2587 (n=20)

	ICP-AES			WDXRF			MISC.			
Element	Avg	STD	%RSD	Avg	STD	%RSD	Element	Avg	STD	%RSD
Al, %	5.72	0.02	0.37	5.75	0.016	0.27	Tot C, %	2.61	0.01	0.55
Ca, %	0.92	0.01	0.58	0.92	0.004	0.45	Hg, ppm	0.24	0.02	6.4
Fe, %	2.81	0.01	0.49	2.82	0.027	0.95				
K, %	1.54	0.01	0.58	1.59	0.007	0.43				
Mg, %	0.67	0.00	0.27	0.67	0.004	0.54				
Na, %	1.15	0.00	0.39	1.10	0.014	1.3				
P, %	0.10	0.00	0.89	0.11	0.001	1.2				
Ti, %	0.33	0.01	4.51	0.42	0.003	0.61				
Si, %	-	-	-	32.95	0.046	0.14				
Ag, ppm	<2	-	-							
As, ppm	13	1.27	9.							
Ba, ppm	570	4.76	0.8							
Be, ppm	9.2	1.75	19.							
Cd, ppm	<2	-	-							
Ce, ppm	57	1.74	3.1							
Co, ppm	14	0.33	2.4							
Cr, ppm	98	2.14	2.2							
Cu, ppm	160	17	10.							
Ga, ppm	13	0.60	4.6							
La, ppm	29	0.89	3.0							
Li, ppm	32	0.29	0.9							
Mn, ppm	644	3.57	0.5							
Nb, ppm	14	0.73	5.4							
Nd, ppm	25	1.39	5.6							
Ni, ppm	36	0.71	2.0							
Pb, ppm	3250	31	0.9							
Sc, ppm	11	0.17	1.5							
Sr, ppm	129	0.59	0.4							
Th, ppm	7.5	0.83	11.							
V, ppm	78	0.52	0.6							
Y, ppm	15	0.20	1.3							
Yb, ppm	1.6	0.31	19.4							
Zn, ppm	337	2.06	0.6							

[1] arithmetic average
[2] standard deviation
[3] percent relative standard deviation

Table 6. Laboratory results for the analysis of SRM 2711 during the between-bottle homogeniety tests for SRM 2586 and 2587

Element	USGS ICP.[1]	USGS ICP.[2]	USGS WDXRF[1]	USGS ICP-MS[1]	USGS MISC.[1]	NIST Mean		NIST STDS
Al, %	6.76	6.45	6.51			6.53	±	0.09
Ca, %	3.01	2.87	2.87			2.88	±	0.08
Fe, %	3.06	2.89	2.92			2.89	±	0.06
K, %	2.50	2.91	2.47			2.45	±	0.08
Mg, %	1.11	1.06	1.04			1.05	±	0.03
Na, %	1.26	1.19	1.09			1.14	±	0.03
P, %	0.089	0.085	0.09			0.086	±	0.007
Ti, %	0.28	0.26	0.31			0.306	±	0.023
Si, %			30.19			30.44	±	0.19
Ag, ppm	7	3				4.63	±	0.39
As, ppm	100	80				105	±	8
Ba, ppm	730	720				726	±	38
Be, ppm	1.2	2.1						
Cd, ppm	35	36				41.7	±	0.25
Ce, ppm	67	66		75		69		
Co, ppm	12	11				10		
Cr, ppm	48	47				47		
Cu, ppm	140	110				114	±	2
Ga, ppm	16	16				15		
Hg, ppm					5.6	6.25	±	0.19
La, ppm	34	37		41		40		
Li, ppm	30	28						
Mn, ppm	670	640				638	±	28
Mo, ppm	<2	<2				1.6		
Nb, ppm	11	18						
Nd, ppm	28	28		29		31		
Ni, ppm	21	20				20.6		
Pb, ppm	1160	1170				1162	±	31
Sc, ppm	10	9				9		
Se, ppm					1.1	1.52	±	0.14
Sr, ppm	260	250				245.3	±	0.7
Th, ppm	14	11				14		
V, ppm	87	80				81.6	±	2.9
Y, ppm	25	24				25		
Yb, ppm	2.5	2.7		3.3		2.7		
Zn, ppm	390	350				350.4	±	4.8

[1] SRM 2711 analyzed with samples of SRM 2586, [2] SRM 2711 analyzed with samples of SRM 2587

Appendix A Analytical results for the between-bottle analysis of SRM 2586

ICP-AES[1]

Lab ID	Bottle #	Al %	Ca %	Fe %	K %	Mg %	Na %	P %	Ti %
D-573897	400	6.64	2.25	5.27	0.961	1.78	0.522	0.100	0.544
D-573898	800	6.92	2.29	5.20	0.966	1.78	0.521	0.100	0.544
D-573899	1050	6.70	2.24	5.23	0.963	1.74	0.52	0.098	0.543
D-573900	150	6.66	2.28	5.22	0.968	1.75	0.522	0.100	0.552
D-573901	2200	6.76	2.26	5.25	0.962	1.79	0.522	0.100	0.545
D-573902	1400	6.64	2.23	5.25	0.956	1.75	0.515	0.099	0.537
D-573903	1600	6.73	2.25	5.21	0.957	1.71	0.516	0.099	0.540
D-573904	650	6.87	2.28	5.23	0.976	1.79	0.528	0.101	0.547
D-573905	2400	6.61	2.23	5.20	0.935	1.73	0.507	0.099	0.532
D-573906	200	6.78	2.28	5.27	0.987	1.80	0.535	0.100	0.551
D-573907	1900	6.72	2.29	5.28	0.967	1.80	0.524	0.102	0.542
D-573908	750	6.47	2.22	5.22	0.946	1.76	0.508	0.098	0.523
D-573909	2100	6.59	2.25	5.22	0.951	1.75	0.517	0.099	0.543
D-573910	2300	6.67	2.24	5.20	0.939	1.77	0.527	0.101	0.538
D-573911	2550	6.61	2.25	5.19	0.956	1.73	0.520	0.099	0.528
D-573912	1950	6.66	2.27	5.24	0.963	1.76	0.523	0.100	0.544
D-573913	1000	6.59	2.28	5.28	0.972	1.76	0.518	0.100	0.541
D-573914	550	6.9	2.30	5.24	1.000	1.79	0.542	0.101	0.564
D-573915	2500	6.68	2.28	5.25	0.960	1.76	0.525	0.100	0.551
D-573916	100	6.85	2.30	5.27	0.988	1.80	0.541	0.101	0.555

Lab ID	Bottle No	Ag ppm	As ppm	Au ppm	Ba ppm	Be ppm	Cd ppm	Ce ppm	Co ppm
D-573897	400	<2	<10	<8	402	1.7	<2	51.7	35.8
D-573898	800	<2	<10	<8	408	1.3	<2	52.4	35.2
D-573899	1050	<2	<10	<8	397	1.4	<2	50.9	35.7
D-573900	150	<2	<10	<8	407	1.4	<2	51.9	35.5
D-573901	2200	<2	<10	<8	400	1.4	<2	52.0	34.6
D-573902	1400	<2	<10	<8	400	1.5	<2	51.6	35.4
D-573903	1600	<2	<10	<8	402	1.3	<2	53.0	35.4
D-573904	650	<2	<10	<8	410	1.5	<2	52.2	35.5
D-573905	2400	<2	<10	<8	395	1.4	<2	53.5	33.9
D-573906	200	<2	<10	<8	406	1.2	<2	51.4	37.0
D-573907	1900	<2	<10	<8	409	1.3	<2	51.1	34.4
D-573908	750	<2	<10	<8	397	2.0	<2	51.4	34.6
D-573909	2100	<2	<10	<8	399	1.5	<2	50.7	35.6
D-573910	2300	<2	<10	<8	401	1.1	<2	52.4	34.8
D-573911	2550	<2	<10	<8	402	1.3	<2	52.5	34.3
D-573912	1950	<2	<10	<8	404	1.2	<2	51.9	35.4
D-573913	1000	<2	<10	<8	412	1.2	<2	51.2	34.3
D-573914	550	<2	<10	<8	409	1.2	<2	52.1	35.3
D-573915	2500	<2	<10	<8	405	1.5	<2	52.0	33.6
D-573916	100	<2	<10	<8	410	1.3	<2	51.1	35.3

Appendix A cont.

Lab ID	Bottle #	Cr ppm	Cu ppm	Eu ppm	Ga ppm	Ho ppm	La ppm	Li ppm	Mn ppm
D-573897	400	331	82.1	<2	13.2	<4	26.3	25.6	1010
D-573898	800	334	81.0	<2	13.4	<4	27.3	25.1	988
D-573899	1050	324	80.8	<2	13.9	<4	25.9	25.3	999
D-573900	150	328	81.0	<2	13.5	<4	25.6	25.4	1010
D-573901	2200	337	81.7	<2	13.8	<4	27.1	25.7	1010
D-573902	1400	325	80.8	<2	14.9	<4	25.9	25.0	994
D-573903	1600	314	80.1	<2	12.1	<4	25.6	25.1	1000
D-573904	650	336	80.6	<2	14.9	<4	26.8	25.3	1010
D-573905	2400	322	81.3	<2	13.4	<4	26.1	24.6	995
D-573906	200	341	80.7	<2	14.1	<4	26.8	25.7	1010
D-573907	1900	335	81.2	<2	15.0	<4	26.4	25.4	1020
D-573908	750	328	80.9	<2	13.5	<4	26.1	25.1	994
D-573909	2100	322	81.0	<2	12.7	<4	25.5	25.1	989
D-573910	2300	329	82.4	<2	14.5	<4	26.4	25.2	997
D-573911	2550	318	80.7	<2	13.5	<4	25.8	25.2	1000
D-573912	1950	327	86.2	<2	14.7	<4	26.4	25.4	1010
D-573913	1000	328	82.2	<2	14.2	<4	25.3	25.5	1020
D-573914	550	332	81.9	<2	14.7	<4	26.8	25.6	1010
D-573915	2500	328	81.9	<2	14.2	<4	25.7	24.9	997
D-573916	100	334	81.4	<2	15.3	<4	26.2	26.0	1010

Lab ID	Bottle #	Mo ppm	Nb ppm	Nd ppm	Ni ppm	Pb ppm	Sc ppm	Sn ppm	Sr ppm
D-573897	400	<2	5.43	24.3	76.1	424	24.2	<5	84.6
D-573898	800	<2	6.38	25.3	74.7	418	24.8	<5	86.8
D-573899	1050	<2	5.22	24.4	74.5	420	24.0	<5	84.3
D-573900	150	<2	5.27	24.8	75.1	430	24.0	<5	85.0
D-573901	2200	<2	7.08	25.3	75.9	424	24.8	<5	85.3
D-573902	1400	<2	6.01	23.9	74.1	425	23.9	<5	83.8
D-573903	1600	<2	5.83	24.7	73.9	407	23.7	<5	84.7
D-573904	650	<2	6.66	24.5	76.2	429	24.7	<5	86.8
D-573905	2400	<2	7.01	24.4	77.5	412	23.9	<5	83.2
D-573906	200	<2	5.92	24.7	76.1	435	24.6	<5	86.7
D-573907	1900	<2	6.48	24.4	75.0	434	25.4	<5	86.3
D-573908	750	<2	5.99	24.5	73.7	415	24.2	<5	83.2
D-573909	2100	<2	5.70	24.3	74.6	422	23.9	<5	84.2
D-573910	2300	<2	6.70	25.2	74.2	425	24.5	<5	85.9
D-573911	2550	<2	5.35	23.8	73.2	417	23.9	<5	84.1
D-573912	1950	<2	6.57	24.5	75.6	427	24.4	<5	85.6
D-573913	1000	<2	5.50	24.1	74.7	429	24.3	<5	85.4
D-573914	550	<2	5.52	25.4	75.7	430	24.7	<5	87.6
D-573915	2500	<2	6.58	25.3	74.0	427	24.2	<5	86.5
D-573916	100	2	5.28	24.6	76.8	437	24.7	<5	88.0

Appendix A cont.

Lab ID	Bottle #	Ta ppm	Th ppm	U ppm	V ppm	Y ppm	Yb ppm	Zn ppm
D-573897	400	<40	6.2	<100	162	21.3	2.22	364
D-573898	800	<40	7.0	<100	162	21.6	2.21	361
D-573899	1050	<40	6.4	<100	159	20.8	2.18	363
D-573900	150	<40	6.4	<100	159	20.9	2.19	368
D-573901	2200	<40	7.0	<100	164	21.6	2.20	368
D-573902	1400	<40	6.6	<100	160	21.0	2.15	364
D-573903	1600	<40	6.1	<100	155	20.7	2.16	360
D-573904	650	<40	6.9	<100	164	21.6	2.23	364
D-573905	2400	<40	6.4	<100	157	21.1	2.15	359
D-573906	200	<40	7.3	<100	165	21.6	2.24	371
D-573907	1900	<40	7.1	<100	164	21.8	2.22	363
D-573908	750	<40	6.5	<100	160	21.2	2.14	360
D-573909	2100	<40	6.3	<100	159	21.0	2.18	354
D-573910	2300	<40	6.4	<100	160	21.3	2.20	363
D-573911	2550	<40	6.5	<100	157	20.9	2.17	358
D-573912	1950	<40	6.9	<100	161	21.3	2.20	367
D-573913	1000	<40	6.0	<100	159	20.9	2.20	363
D-573914	550	<40	7.8	<100	163	21.5	2.23	363
D-573915	2500	<40	6.2	<100	160	21.2	2.22	365
D-573916	100	<40	6.4	<100	162	21.2	2.26	372

Appendix A (cont.)

WDXRF[2]

Lab #	Bottle #	SiO_2 %	Al_2O_3 %	Fe_2O_3 %	MgO %	CaO %	Na_2O %	K_2O %	TiO_2 %	P_2O_5 %	MnO %	LOI %	Total %
D-577356	400	70.44	11.14	3.99	1.11	1.28	1.47	1.92	0.695	0.241	0.082	6.35	98.83
D-577357	800	70.66	11.22	4.00	1.12	1.29	1.49	1.91	0.704	0.244	0.083	6.23	99.08
D-577358	1050	70.50	11.19	4.00	1.12	1.28	1.51	1.92	0.703	0.240	0.083	6.21	98.89
D-577359	150	70.51	11.18	4.09	1.12	1.28	1.52	1.92	0.706	0.244	0.086	6.19	99.04
D-577360	2200	70.33	11.15	4.07	1.11	1.29	1.48	1.92	0.702	0.241	0.086	6.21	98.75
D-577361	1400	70.37	11.17	4.07	1.11	1.28	1.52	1.91	0.708	0.238	0.085	6.09	98.75
D-577362	1600	70.58	11.2	4.11	1.12	1.27	1.48	1.92	0.697	0.247	0.086	6.17	99.08
D-577363	650	70.42	11.13	4.07	1.12	1.28	1.48	1.92	0.695	0.247	0.086	6.16	98.78
D-577364	2400	70.59	11.21	4.04	1.12	1.29	1.49	1.92	0.708	0.245	0.084	6.20	99.06
D-577365	200	70.46	11.16	3.99	1.11	1.29	1.52	1.93	0.702	0.244	0.084	6.35	99.00
D-577366	1900	70.54	11.16	4.01	1.11	1.28	1.47	1.92	0.709	0.239	0.083	6.27	98.95
D-577367	750	70.57	11.24	4.02	1.12	1.29	1.50	1.93	0.709	0.242	0.085	6.23	99.07
D-577368	2100	70.65	11.19	4.03	1.10	1.29	1.48	1.92	0.699	0.241	0.084	6.16	98.98
D-577369	2300	70.33	11.13	4.04	1.11	1.28	1.48	1.91	0.704	0.239	0.084	6.20	98.62
D-577370	2550	70.56	11.21	4.04	1.11	1.28	1.48	1.92	0.698	0.248	0.084	6.14	98.93
D-577371	1950	70.60	11.19	4.02	1.12	1.28	1.48	1.93	0.703	0.248	0.084	6.24	99.07
D-577372	1000	70.49	11.20	4.03	1.12	1.28	1.47	1.93	0.701	0.242	0.084	6.20	98.92
D-577373	550	70.54	11.18	4.04	1.11	1.29	1.45	1.93	0.702	0.242	0.085	6.20	98.92
D-577374	2500	70.40	11.16	3.97	1.11	1.28	1.51	1.91	0.703	0.242	0.083	6.11	98.61
D-577375	100	70.43	11.15	3.97	1.11	1.28	1.48	1.90	0.699	0.243	0.083	6.13	98.60

Appendix A (cont.)

ICP/MS[3]

Lab ID	Bottle #	Ce ppm	Dy ppm	Er ppm	Eu ppm	Gd ppm	Ho ppm
D-573897	400	58	5.2	3.3	1.4	5.4	1.1
D-573898	800	59	5.4	3.3	1.4	5.6	1.1
D-573899	1050	67	5.6	3.5	1.6	6.5	1.1
D-573900	150	70	5.6	3.4	1.6	5.9	1.2
D-573901	2200	69	5.4	3.3	1.6	5.9	1.1
D-573902	1400	64	5.2	3.1	1.4	5.4	1.0
D-573903	1600	70	5.5	3.4	1.6	6.2	1.1
D-573904	650	68	5.7	3.4	1.5	5.9	1.1
D-573905	2400	68	5.8	3.6	1.5	6.0	1.1
D-573906	200	70	5.6	3.6	1.6	6.2	1.1
D-573907	1900	65	5.3	3.3	1.5	6.1	1.1
D-573908	750	64	5.6	3.3	1.5	5.5	1.0
D-573909	2100	65	5.5	3.3	1.6	5.7	1.0
D-573910	2300	66	5.4	3.2	1.5	5.7	1.0
D-573911	2550	67	5.6	3.5	1.5	6.0	1.1
D-573912	1950	59	5.2	3.2	1.5	5.8	1.1
D-573913	1000	60	5.2	3.1	1.4	5.6	1.1
D-573914	550	59	5.2	3.3	1.4	5.5	1.1
D-573915	2500	58	4.8	3.1	1.3	5.3	1.0
D-573916	100	59	5.1	3.2	1.4	5.8	1.1

Lab ID	Bottle #	La ppm	Nd ppm	Pr ppm	Sm ppm	Tb ppm	Tm ppm	Yb ppm
D-573897	400	30	27	6.8	5.8	0.8	0.5	3.1
D-573898	800	30	26	6.7	5.6	0.9	0.5	3.1
D-573899	1050	34	30	7.5	6.3	0.9	0.5	3.1
D-573900	150	36	30	7.8	6.6	0.9	0.5	3.1
D-573901	2200	34	30	7.8	6.3	1.0	0.5	3.2
D-573902	1400	33	29	7.1	6.1	0.8	0.5	2.9
D-573903	1600	36	31	7.9	6.5	0.9	0.5	3.2
D-573904	650	35	30	7.7	6.1	0.9	0.5	3.1
D-573905	2400	35	30	7.7	6.3	0.9	0.5	3.1
D-573906	200	37	31	7.9	6.5	1.0	0.5	3.2
D-573907	1900	34	28	7.4	6.1	0.9	0.5	3.2
D-573908	750	34	29	7.5	6.3	0.9	0.5	3.1
D-573909	2100	34	28	7.3	6.3	0.9	0.5	3.0
D-573910	2300	34	28	7.3	6.1	0.9	0.5	3.2
D-573911	2550	35	29	7.7	6.3	0.9	0.5	3.2
D-573912	1950	30	25	6.7	5.5	0.8	0.5	2.9
D-573913	1000	32	26	7.1	5.6	0.8	0.5	2.9
D-573914	550	31	26	6.6	5.7	0.9	0.5	3.3
D-573915	2500	29	25	6.3	5.5	0.9	0.4	2.9
D-573916	100	30	27	6.8	5.6	0.9	0.5	2.8

Appendix A (cont.)

CV-AAS[4], HG-AAS[5]

Lab ID	Bottle #	As ppm	Se ppm	Hg ppm
D-573897	400	8.7	0.6	0.32
D-573898	800	8.4	0.6	0.30
D-573899	1050	8.5	0.6	0.31
D-573900	150	8.4	0.6	0.32
D-573901	2200	8.3	0.6	0.30
D-573902	1400	8.4	0.6	0.31
D-573903	1600	8.5	0.6	0.30
D-573904	650	8.4	0.6	0.31
D-573905	2400	8.4	0.6	0.32
D-573906	200	8.4	0.6	0.31
D-573907	1900	8.4	0.6	0.31
D-573908	750	8.5	0.6	0.33
D-573909	2100	8.4	0.6	0.31
D-573910	2300	8.2	0.6	0.32
D-573911	2550	8.7	0.6	0.30
D-573912	1950	8.6	0.6	0.30
D-573913	1000	8.4	0.6	0.31
D-573914	550	8.5	0.6	0.31
D-573915	2500	8.5	0.6	0.31
D-573916	100	8.6	0.6	0.30

[1] ICP-AES, Inductively coupled plasma-atomic emission spectroscopy
[2] WDXRF, Wavelength dispersive X-ray fluoresence
[3] ICP-MS, Inductively Couple Plasma-Mass Spectrometry
[4] CV-AAS, Cold vapor - atomic absorption spectrophotometry
[5] HY-AAS, Hydride generation atomic-atomic absorption spectrophotometry

Appendix B, Analytical results for the between-bottle analysis of SRM 2587

ICP-AES[1]

Lab ID	Bottle #	Al %	Ca %	Fe %	K %	Mg %	Na %	P %	Ti %
D-577356	1750	5.69	0.920	2.79	1.52	0.664	1.15	0.094	0.359
D-577357	2150	5.72	0.931	2.84	1.54	0.667	1.15	0.094	0.353
D-577358	1050	5.68	0.919	2.81	1.54	0.662	1.15	0.096	0.338
D-577359	200	5.72	0.920	2.80	1.55	0.668	1.15	0.096	0.330
D-577360	650	5.70	0.920	2.81	1.55	0.666	1.15	0.097	0.326
D-577361	550	5.72	0.913	2.81	1.56	0.667	1.16	0.095	0.316
D-577362	2350	5.69	0.925	2.81	1.54	0.667	1.15	0.096	0.315
D-577363	1250	5.72	0.925	2.81	1.55	0.668	1.15	0.097	0.320
D-577364	2450	5.72	0.915	2.78	1.54	0.665	1.15	0.096	0.327
D-577365	450	5.74	0.922	2.80	1.55	0.666	1.16	0.096	0.323
D-577366	750	5.77	0.913	2.84	1.54	0.667	1.15	0.095	0.367
D-577367	1700	5.75	0.928	2.82	1.54	0.668	1.16	0.096	0.356
D-577368	1500	5.72	0.925	2.81	1.54	0.668	1.15	0.096	0.328
D-577369	1300	5.71	0.916	2.80	1.53	0.665	1.15	0.095	0.322
D-577370	50	5.72	0.921	2.81	1.54	0.667	1.15	0.095	0.319
D-577371	350	5.73	0.927	2.81	1.54	0.669	1.15	0.097	0.332
D-577372	300	5.72	0.920	2.80	1.54	0.663	1.15	0.096	0.329
D-577373	2200	5.74	0.929	2.81	1.54	0.667	1.15	0.096	0.328
D-577374	700	5.72	0.922	2.81	1.53	0.666	1.14	0.096	0.329
D-577375	1600	5.73	0.929	2.81	1.53	0.668	1.15	0.096	0.322

Lab ID	Bottle #	Ag ppm	As ppm	Ba ppm	Be ppm	Bi ppm	Cd ppm	Ce ppm	Co ppm	Cr ppm	Cu ppm
D-577356	1750	<2	14	572	13	<10	<2	57	14.2	98.7	155
D-577357	2150	<2	14	564	11	<10	<2	59	13.6	100	222
D-577358	1050	<2	12	568	8.8	<10	<2	58	13.8	97.4	156
D-577359	200	<2	14	577	8.7	<10	<2	55	13.8	98.5	151
D-577360	650	<2	12	576	8.2	<10	<2	57	13.7	100	191
D-577361	550	<2	14	569	7.6	<10	<2	52	13.6	93.4	161
D-577362	2350	<2	16	564	7.4	<10	<2	58	14.1	97.8	150
D-577363	1250	<2	14	570	8.3	<10	<2	57	14.0	96.8	156
D-577364	2450	<2	12	578	8.3	<10	<2	55	13.7	100	154
D-577365	450	<2	13	561	8.2	<10	<2	55	14.0	97.7	158
D-577366	750	<2	13	569	14	<10	<2	59	14.1	96.9	152
D-577367	1700	<2	14	565	11	<10	<2	58	14.2	98.2	154
D-577368	1500	<2	12	567	8.7	<10	<2	56	13.6	95.5	155
D-577369	1300	<2	11	566	8.2	<10	<2	56	13.1	101	152
D-577370	50	<2	12	564	7.9	<10	<2	57	14.3	95.6	156
D-577371	350	<2	12	569	9.8	<10	<2	58	14.2	97.4	155
D-577372	300	<2	12	563	9.0	<10	<2	57	14.0	96.1	154
D-577373	2200	<2	13	565	8.6	<10	<2	55	13.8	97.6	156
D-577374	700	<2	14	565	8.8	<10	<2	59	14.2	102	158
D-577375	1600	<2	15	565	8.4	<10	<2	57	14.6	94.8	166

Appendix B cont.

Lab ID	Bottle #	Eu ppm	Ga ppm	La ppm	Li ppm	Mn ppm	Mo ppm	Nb ppm	Nd ppm	Ni ppm	Pb ppm
D-577356	1750	<2	12.2	29.1	31.7	640	<2	15.3	25.1	34.0	3250
D-577357	2150	<2	12.6	30.8	31.4	650	<2	12.8	24.6	35.1	3270
D-577358	1050	<2	13.0	29.5	31.8	640	<2	14.2	23.1	37.4	3210
D-577359	200	<2	13.1	28.9	32.4	643	<2	14.3	23.9	35.0	3280
D-577360	650	<2	12.4	29.4	31.6	648	<2	14.1	25.1	35.6	3210
D-577361	550	<2	12.8	27.0	31.8	647	<2	13.9	22.4	34.7	3250
D-577362	2350	<2	13.1	30.4	31.9	641	<2	14.0	25.8	35.4	3180
D-577363	1250	<2	13.4	29.8	31.9	645	<2	13.6	26.1	35.3	3310
D-577364	2450	<2	14.1	28.8	32.2	638	<2	14.8	25.4	34.5	3240
D-577365	450	<2	12.3	28.4	31.9	639	<2	13.6	23.1	35.2	3250
D-577366	750	<2	13.6	30.1	32.1	649	<2	13.2	26.5	35.8	3270
D-577367	1700	<2	14.2	30.1	31.9	646	<2	14.3	26.5	35.7	3260
D-577368	1500	<2	13.0	29.9	31.7	645	<2	12.9	23.5	35.5	3220
D-577369	1300	<2	13.0	29.8	32.1	641	<2	12.2	24.6	35.4	3260
D-577370	50	<2	13.8	29.4	32.1	644	<2	13.4	24.2	36.1	3280
D-577371	350	<2	13.3	29.5	31.7	644	<2	13.2	26.2	35.8	3280
D-577372	300	<2	13.8	30.1	31.8	645	<2	12.8	26.6	35.4	3220
D-577373	2200	<2	12.4	28.9	32.0	650	<2	13.7	25.4	36.1	3260
D-577374	700	<2	12.6	30.5	32.1	643	<2	13.7	27.5	35.9	3230
D-577375	1600	<2	12.6	28.3	31.1	645	<2	13.5	23.8	36.2	3260

Lab ID	Bottle #	Sc ppm	Sn ppm	Sr ppm	Ta ppm	Th ppm	V ppm	Y ppm	Yb ppm	Zn ppm
D-577356	1750	11.0	7.2	130	<40	6.9	77.9	15.0	1.2	336
D-577357	2150	11.0	11.0	130	<40	7.4	78.9	15.4	1.3	343
D-577358	1050	10.6	10.4	129	<40	7.2	77.9	15.1	1.3	335
D-577359	200	10.9	<5	130	<40	6.9	78.5	15.4	2.0	336
D-577360	650	10.9	<5	129	<40	6.9	77.9	15.2	1.2	336
D-577361	550	10.9	6.3	129	<40	5.3	78.2	14.8	1.8	335
D-577362	2350	10.7	5.9	129	<40	8.3	78.2	15.4	1.4	335
D-577363	1250	10.8	<5	129	<40	7.8	78.9	15.2	1.7	340
D-577364	2450	10.8	5.3	129	<40	7.9	77.8	15.3	1.7	338
D-577365	450	11.1	<5	130	<40	7.8	78.1	15.2	1.2	335
D-577366	750	11.1	6.2	129	<40	8.4	79.3	15.6	2.0	338
D-577367	1700	11.1	5.1	129	<40	8.7	79.5	15.4	1.7	338
D-577368	1500	10.8	<5	129	<40	7.5	78.4	15.2	1.5	337
D-577369	1300	10.6	<5	129	<40	6.6	78.0	15.3	2.2	336
D-577370	50	11.1	7.0	129	<40	8.6	78.0	15.3	1.7	338
D-577371	350	10.9	5.2	128	<40	7.7	78.0	15.4	1.6	340
D-577372	300	10.8	7.0	128	<40	8.2	77.8	15.3	1.3	336
D-577373	2200	10.9	6.6	130	<40	7.6	78.9	15.0	2.0	338
D-577374	700	11.1	6.4	129	<40	7.4	78.2	15.6	1.4	337
D-577375	1600	11.1	6.9	129	<40	6.4	77.8	15.1	1.4	336

Appendix B cont

WDXRF[2]

Lab ID	Bottle #	SiO_2 %	Al_2O_3 %	Fe_2O_3 %	MgO %	CaO %	Na_2O %	K_2O %	TiO_2 %	P_2O_5 %	MnO %
D-577356	1750	70.44	11.14	3.99	1.11	1.28	1.47	1.92	0.695	0.241	0.082
D-577357	2150	70.66	11.22	4.00	1.12	1.29	1.49	1.91	0.704	0.244	0.083
D-577358	1050	70.50	11.19	4.00	1.12	1.28	1.51	1.92	0.703	0.240	0.083
D-577359	200	70.51	11.18	4.09	1.12	1.28	1.52	1.92	0.706	0.244	0.086
D-577360	650	70.33	11.15	4.07	1.11	1.29	1.48	1.92	0.702	0.241	0.086
D-577361	550	70.37	11.17	4.07	1.11	1.28	1.52	1.91	0.708	0.238	0.085
D-577362	2350	70.58	11.20	4.11	1.12	1.27	1.48	1.92	0.697	0.247	0.086
D-577363	1250	70.42	11.13	4.07	1.12	1.28	1.48	1.92	0.695	0.247	0.086
D-577364	2450	70.59	11.21	4.04	1.12	1.29	1.49	1.92	0.708	0.245	0.084
D-577365	450	70.46	11.16	3.99	1.11	1.29	1.52	1.93	0.702	0.244	0.084
D-577366	750	70.54	11.16	4.01	1.11	1.28	1.47	1.92	0.709	0.239	0.083
D-577367	1700	70.57	11.24	4.02	1.12	1.29	1.50	1.93	0.709	0.242	0.085
D-577368	1500	70.65	11.19	4.03	1.10	1.29	1.48	1.92	0.699	0.241	0.084
D-577369	1300	70.33	11.13	4.04	1.11	1.28	1.48	1.91	0.704	0.239	0.084
D-577370	50	70.56	11.21	4.04	1.11	1.28	1.48	1.92	0.698	0.248	0.084
D-577371	350	70.60	11.19	4.02	1.12	1.28	1.48	1.93	0.703	0.248	0.084
D-577372	300	70.49	11.20	4.03	1.12	1.28	1.47	1.93	0.701	0.242	0.084
D-577373	2200	70.54	11.18	4.04	1.11	1.29	1.45	1.93	0.702	0.242	0.085
D-577374	700	70.40	11.16	3.97	1.11	1.28	1.51	1.91	0.703	0.242	0.083
D-577375	1600	70.43	11.15	3.97	1.11	1.28	1.48	1.90	0.699	0.243	0.083

Appendix B cont.

CV-AAS[3], Combustion[4]

Lab ID	Bottle #	Hg ppm	Ctot %
D-577356	1750	0.24	2.61
D-577357	2150	0.24	2.63
D-577358	1050	0.24	2.63
D-577359	200	0.24	2.63
D-577360	650	0.25	2.61
D-577361	550	0.24	2.61
D-577362	2350	0.34	2.62
D-577363	1250	0.25	2.61
D-577364	2450	0.24	2.58
D-577365	450	0.24	2.61
D-577366	750	0.24	2.62
D-577367	1700	0.24	2.59
D-577368	1500	0.25	2.62
D-577369	1300	0.25	2.60
D-577370	50	0.20	2.61
D-577371	350	0.27	2.60
D-577372	300	0.25	2.60
D-577373	2200	0.28	2.61
D-577374	700	0.25	2.63
D-577375	1600	0.23	2.59

[1] ICP-AES, Inductively coupled plasma-atomic emission spectroscopy
[2] WDXRF, Wavelength dispersive X-ray fluoresence
[3] CV-AAS, Cold Vapor-atomic absorption spectrophotometry
[4] CO_2-IR, Sample combustion and CO_2 determination by infra-red absorption