Collection, Preparation and Testing of NIST Hard Rock Mine Waste Reference Material SRM 2780

by S.A. Wilson[1], P.H. Briggs[1], Z.A. Brown[1], J.E. Taggart[1] and R. Knight[1]

Open File Report 99-370

1999

U.S. DEPARTMENT OF THE INTERIOR
U.S. GEOLOGICAL SURVEY

[1] Denver, Colorado

Table of Contents

Introduction

In the western United States there are an estimated five hundred thousand abandoned mines located on public and private lands. These sites commonly contain significant quantities of mine waste material which was discarded without environmental concern during historic mining operations. Typically this waste is responsible for the generation of acidic (pH 2-4) drainage, containing elevated concentrations of heavy metals. This toxic combination contaminates adjacent waterways leaving them void of biological activity for several kilometers downstream. Remediating historic mine sites often requires millions of public and private dollars and thousands of man hours to return the site to its premining condition.

Accurately evaluating the environmental impact of mine waste is hampered by the variability in laboratory methodology used to assess its hazardous nature. This problem is compounded by the difficulty encountered in comparing data from different labs even though they use the same techniques. These problems have developed in part due to the lack of appropriate geochemical reference materials (GRMs) which mimic mine waste composition. In response to this need, the United States Geological Survey (USGS), the National Institute for Standards and Technology (NIST) and the United States Environmental Protection Agency (U.S. EPA) cooperated in the development of a reference material dedicated to the study of hard rock mine waste. The goal was to produce a GRM which could be used in the development of appropriate analytical procedures, assist in intra-laboratory data evaluation, and help create reliable quality assurance programs for the industry. Due to the complex nature of mine waste drainage, the project was designed to examine both the total element composition and water extractable constituents of the proposed GRM. Determining the total element composition of this material was the responsibility of NIST and the USGS. Quantifying water extractable constituents was coordinated through the Acid Drainage Technology Initiative (ADTI) group. ADTI is a working group of scientists from the public and private sectors dedicated to a better geochemical understanding of coal and hard rock mine waste in the United States.

The USGS was charged with identifying a collection site, as well as collecting and physically preparing two "identical" lots of material. The area selected for sample collection was the Silverton mining district, located in San Juan county (392 mi^2), Colorado. The district, located near the town of Silverton, Colorado, sits at the southwestern end of the Colorado mineral belt (Cunningham et al., 1995; Ransome, F.L., 1901). The area was extensively mined in the late 1800's to mid 1900's, with limited mining occurring as late as the 1980's. At the height of its mining activity the district officially employed up to 500 people at 31 mines and mills. State production records indicate that between 1873 and 1980, 1.4 billion pounds of lead, copper, and zinc were produced, as well as 51 million ounces of gold and silver (Colorado Bureau of Mines, 1873-1980). The estimated net worth of this activity is placed at 270 billion dollars.

Evidence of this intensive mining activity is still apparent today, in the several abandoned mills located along the Animas River and in the thousands of abandoned mine sites located throughout the valley. Abandoned mine sites are normally characterized by extensive piles of crushed rock adjacent to the mine entrance. These piles range in color from pale yellow to dark red, and typically contain significant quantities of pyrite, galena, arsenopyrite, chalcopyrite, sphalerite and other sulfide minerals. These metal sulfides undergo gradual oxidation producing significant quantities of sulfuric acid. An example of this oxidation process is presented in equations 1-4 for the mineral pyrite (FeS_2).

1. $FeS_2 + 3.5O_2 + H_2O \rightleftharpoons Fe^{2+} + 2H^+ + 2SO_4^{2-}$

2. $2Fe^{2+} + 0.5O_2 + 2H^+ \rightleftharpoons 2Fe^{3+} + H_2O$

3. $Fe^{3+} + 3H_2O \rightleftharpoons Fe(OH)_3 + 3H^+$

4. $FeS_2 + 14Fe^{3+} + 8H_2O \rightleftharpoons 15Fe^{2+} + 2SO_4^{2-} + 16H^+$

The acid generated during pyrite oxidation leaches the surrounding waste material of metals which are then deposited on the surface of adjacent particles as water soluble salts. During high water flow periods such as the spring runoff and summer thundershowers these acidic salts are mobilized from the waste pile, eventually draining into local streams. The combination of elevated metal concentrations and high acidity makes it difficult for indigenous animal species to survive in affected waterways. Extensive federal and private reclamation projects are currently underway in the Silverton area to remediate several of the most important mine waste locations.

The site selected for sample collection was located next to the Mayday mine, which is situated on the northeastern side of the Silverton caldera field, at latitude 37° 50' 52" and longitude 107° 40' 45". The mine sits atop intra-ash flows of andesitic lava, primarily of the tertiary age (Ransome, F.L., 1901; Tweto, O., 1979). Mines located along this side of the Silverton caldera field produced significant quantities of lead, copper and zinc as well as important amounts of tungsten (hubnerite).

Sample Collection and Preparation

The Mayday mine site is located on Bureau of Land Management (BLM) land and covers a total area of approximately 1 acre. The waste pile is adjacent to the mine opening and is arranged in a poorly defined two-bench structure. The mine dump is generally devoid of vegetation, despite the fact that the mine site is situated in a lush evergreen forest. Measuring from the adjacent county road, the waste pile is approximately 100 meters high, 200 meters in width, with a sixty degree angle of repose. The mine waste surface material has a gray to pale yellow appearance, with isolated streaks of dark red, brown, or white material. The dump's surface material is stable to foot traffic, and is composed of crushed rock ranging in size from 0.5 to 5 cm in length. Large clods of agglomerated waste material are evident, but are easily disaggregated to particles 1 cm or less in diameter. Erosional effects on the pile are apparent, but areas of deep erosional scaring are limited.

The upper bench of the waste pile was selected for sample collection. It was divided into fifteen sections approximately 10 m in width and 30 m in length (top to the bottom of bench). Sample collection was performed by rapelling down the center of each section in 3 meter increments, and then collecting material within five meters of the rope. Samples were obtained from the top 20 cm of surface material, using a common garden spade. The sample was transferred to five gallon plastic buckets lined with a polyethylene bag. Using this procedure approximately 30 kg of material was collected from each section resulting in a total collected sample weight of over 450 kg.

Upon arrival at the USGS, material from each bucket was transferred to six plastic lined cardboard trays, and dried in a forced air oven (room temperature) for 24 hours. After drying material was mixed for 20 minutes in a 3 ft^3 V-blender, and then transferred back into its original container. A sub-sample from each bucket was obtained using a plastic sample thief (30 cm x 2 cm) and then analyzed for its total element content by the USGS. The contents of selected buckets were eventually combined into one batch and blended for 1 hour using a 10 ft^3 cross flow V-blender. After blending, the sample was transferred back into five gallon containers.

As part of the cooperative arrangement between NIST, USGS, and ADTI, the blended mine waste material was to be divided in two equal size lots. The sample splitting was accomplished using a USGS designed spinning riffler. In this procedure, material from each five gallon container was transferred to a specially designed funnel apparatus which allowed material to flow onto a rotating table (2 m diameter) spinning at approximately six rpms. On the table were positioned a set of 12 stainless steel sample splitters which transferred the material into a set (12) of 5 gallon plastic buckets. After splitting was complete, the odd and even numbered containers were set aside for ADTI and NIST studies respectively. Material for NIST was ground to pass 200 mesh (67μ) and then blended in a 10 ft^3 cube blender for 24 hours. The ground material was transferred to 5 kg polyethylene bags, boxed, and radiation sterilized at COBE Laboratories, Lakewood, Colorado. The radiation dosage during sterilization ranged between 1.5 and 3.2 Mrad.

In the final preparation step, the ground/sterilized material was reblended and then split using the spinning riffler to produce ~4 kg aliquots. The spinning riffler was refitted with a 72 position sample ring, and each 4 kg aliquot was used to fill containers(55-60g) with material. A total of 2200 units were prepared in this manner.

Results and Discussion

Prior to shipping the bottled material to NIST, the USGS analyzed samples for major and trace elements to determine between-bottle homogeneity. Analytical methods used included Wavelength Dispersive X-ray Fluorescence (WD-XRF), Inductively Coupled Plasma Atomic Emission Spectroscopy (ICP-AES), Instrumental Neutron Activation Analysis (INAA), Hydride Generation Atomic Absorption Spectroscopy (HYG-AAS), Cold Vapor Atomic Absorption Spectroscopy (CV-AAS), Combustion Infrared detection (C-ID), Coulometric Titration (CT), Fire Assay (FA), Graphite Furnace Atomic Absorption Spectroscopy (GF-AAS),and Potentiometric Titration (PT). Analyses were performed at the USGS laboratory facilities, Lakewood, Colorado and at the USGS contract lab in Ottawa, Canada. Contract lab results are designed to provide information on those elements not analyzed at the USGS. Samples used in the USGS homogeneity test represented every 100^{th} bottle (22), as well as six samples selected at random. A sample of NIST SRM 2710 was included in the sample set for quality control purposes. All samples were analyzed on an as received basis. A brief description of USGS techniques used and their acceptable average element variation is presented in Table 1. More detailed information on USGS analytical procedures is available (Arbogast 1996). Homogeneity testing performed at the USGS contract lab used six samples selected at random. Analytical procedures used at the USGS contract lab are similar to USGS procedures with only minor modifications.

Results from the between-bottle analysis of SRM 2780 are presented in appendices A through E and summarized in table 2 for both the USGS and contract lab. Results are presented by individual techniques except for CV-AAS, HY-AAS, and C-ID which are grouped under the general heading LATA. In this study the estimate of total analytical uncertainty is represented by the percent relative standard deviation (%RSD) as determined using equation 5.

$$5. \quad \%RSD = \left[\frac{\text{element standard deviation}}{\text{average element conc.}}\right] \times 100$$

The %RSD represents a combination of two error components. The first reflects the between-bottle variation in element concentration (Vconc) and the second reflects the error associated with the actual analytical measurement (Em). The USGS value of Em for any technique is estimated through the USGS method validation process, which relies on the analysis of selected geochemical reference materials over three nonconsecutive days. Table 1 presents information on the average %RSD for each USGS technique used.

For the majority of elements quantified in SRM 2780, the observed %RSD is equal to or less than the technique Em. Under USGS guidelines, the material is considered homogeneous for that element within the limits established by the method. Selected elements display %RSD in excess of the Em values and should be carefully evaluated before they are considered homogeneously distributed. These elements include Na, P (WDXRF), Au, Ni, W (INAA), Hg (CV-AAS), and Co(ICP-AES).

Examination of results in table 2 reveal that several elements are present at elevated concentrations. In the case of Ag, As, Cd, Pb, Sb, and Zn, element concentrations are at least two orders of magnitude greater than average crustal material(Fortescue, J., 1992; Schacklette, H. and Boerngen, J., 1984). Concentrations of other elements such as Cu, S, and W, are 2 to 10 times greater than average abundances. USGS X-ray diffraction studies indicate that the major mineral phases in the sample include, quartz, muscovite, feldspar, chlorite, a series of jarosite minerals as well as a significant fraction of amorphous alumino silicates. Also present are minor amounts of galena, sphalerite, and pyrite.

An evaluation of analytical accuracy was performed by comparing USGS results for the analysis of SRM 2710 with NIST certificate values (table 3). NIST SRM 2710 was selected as a QC sample because its element concentrations and mineralized composition are similar to SRM 2780. Prior to sample analysis a confidence interval was established for each element based on certificate information and USGS quality control guidelines. In the case of NIST certified elements, the acceptable element concentration range (AECR) was established as the certified value ± twice the NIST 95% confidence interval. Elements with NIST information values were assigned an AECR equal to the recommended value ± 20% of the mean value. Element concentrations in table 3 that fall outside their calculated AECR are contained within parentheses. All values were corrected for their percent moisture values (1.6%), which was previously determined by the USGS.

Examination of major element results in table 3 reveal that the majority (82%) of major elements concentrations fall within their designated AECR. Exceptions are noted for Na, Si (WDXRF), Ti (ICP-AES), and Ca (INAA). The bias observed for Na and Si using WDXRF reflects a consistent trend in USGS analysis of this SRM (Wilson et al., 1994). Comparison of USGS results with NIST certificate values for minor and trace elements show that 88% of elements have observed concentrations within their calculated AECR. Exceptions to this trend include Co, Cr, Ga,(ICP-AES), and Ag, Cr, Ni, Zn, (INAA).

Conclusion

The NIST hard rock mine waste reference material SRM 2780 was evaluated for element homogeneity by the USGS using a variety of analytical procedures. Results indicate that for the majority of elements tested the total element concentrations show no significant between-bottles variation. Concentrations of selected metals are several orders of magnitude above normal crustal abundances and major mineralogical phases are consistent with the mineralized nature of the area.

Bibliography

Arbogast, B.F., 1996, Analytical Methods Manual for the Mineral Resources Surveys Program, U.S. Geological Survey, Open File report 96-525, p 248

Colorado Bureau of Mines, 1898-1985, A Summary of Mineral Industry Activities in Colorado, State of Colorado, p 100.

Cunningham, C.G., Naeser, C.W., Marvin, R.F., Luedke, R.G., Wallace, A.R., 1994, Ages of Selected Intrusive Rocks and Associated Ore Deposits in the Colorado Mineral Belt, U.S. Geological Survey Bulletin, 2109, 26 p.

Fortescue, J., 1992, Landscape geochemistry: retrospect and prospect — 1990: Applied Geochemistry, v 7, p. 1-53.

Ransome, F.L., 1901, A Report on the Economic Geology of the Silverton Quadrangle, Colorado, U.S. Geological Survey Bulletin, p262.

Schacklette, H.T., and Boerngen, J. , 1984, Element concentrations in soils and other surficial materials of the conterminous United States, U.S. Geological Survey Professional Paper 1270, p 105.

Tweto, O., 1979, Geologic Map of Colorado 1:500,000, U.S. Geological Survey Map GSG-6-2.

Table 1. USGS analytical procedures

Method	Decomposition Procedure	Sample wt g	Precision %RSD
ICP-AES	multi acid	0.200	5 - 10
WD-XRF	Lithium Tetaborate Fusion	0.800	1 - 2
INAA	none	0.7	1 - 10
CV-AAS	Dichromate/ nitric acid	0.25	2 - 5
HYG-AAS	multi acid	0.25	5 - 10
C-ID	High temp. combustion	0.25	5 - 10

Table 2. Summary results for USGS and contract lab analysis of NIST SRM 2780

Element	USGS WDXRF			USGS ICP-AES			USGS INAA			USGS LATA			Contract lab		
	AVG	STDS	%RSD	AVG	STDS	%RSD	AVG	STDS	%RSD	AVG	STDS	%RSD	AVG	STDS	%RSD
Al %	8.79	0.03	0.30	8.50	0.23	2.7									
Ctot										0.17	0.01	6.0	0.17	0.02	12
Cinorg													0.010	0.004	10
Corg													0.16	0.02	12
Ca	0.20	0.004	2.1	0.19	0.01	4.5	0.29	0.01	3.4						
Fe	2.73	0.02	0.6	2.7	0.02	0.8	2.77	0.03	1.2						
FeO													0.68	0.04	6
K	3.43	0.03	0.74	3.2	0.09	2.9	4.08	0.17	4.1						
Mg	0.54	0.01	1.2	0.51	0.01	1.0									
Na	(0.32)	0.01	3.1	0.21	0.01	3.3	0.22	0.002	0.81						
P	(0.062)	0.003	5.3	0.04	0.00	6.9									
Stot										1.23	0.02	1.2	1.29	0.03	2
Si	34.0	0.06	0.2												
Ti	0.698	0.004	0.6	0.64	0.04	6.3									
Ag ppm				27	1	2.6	22.6	0.6	2.7						
As				72	6	7.9	48.2	0.8	1.7	44	3	7.8			
Au				<8	-	-	(0.184)	26	14				0.20	0.04	18
Ba				900	40	4.5	964	17	1.8						
Cd				12	1	4.1	12.0	1.0	8.5						
Co				61	5	7.9	67.1	0.9	1.3						
Co				(2.1)	0.5	24	2.21	0.04	1.8						
Cr				48	2	3.7	40.6	1.4	3.5						
Cs							13.1	0.1	1.0						
Cu				220	11	5.2									
Ga				26.0	0.5	1.8									
Hf							4.41	0.07	1.5						
Hg										(0.73)	0.10	14	0.7	0.1	14
Ho				<4	-	-	0.84	0.03	3.9						
La				38	3	9.0	37.6	0.4	0.9						
Li				18	0.2	1.1									
Mn	(450)	30	7.2	450	10	2.3									
Mo				(13)	2.6	20	10.6	0.9	8.9						
Nb				18	1	5.3									
Nd				26	2	7.4	30.5	0.6	2.1						
Ni				12	0.5	4.0	(19.5)	3.2	16						
Pb				5700	110	1.9									
Rb							173	2	1.2						
Sb							159	2	1.3				125	10	8
Sc				23	2	7.5	23.7	0.3	1.1						
Se							3.4	0.2	5.2	5.15	0.13	2.5	4.4	0.4	9
Sr				220	6	2.7	294	6	2.1						
Tb							0.58	0.01	1.8						
Te													5.3	0.7	13
Th				(8)	2	32	11.5	0.1	1.3				9.5	0.5	6
Tl													4.9	0.4	7
Tm							0.40	0.02	3.9						
U				<100	-	-	3.95	0.08	2.0						
V				260	5	2.0									
W							(20)	3	16				23.8	2.1	9
Zn				2800	130	4.7	2380	53	2.2						
Zr							162	16	9.9						

Element concentration in paraentheses have observed %RSD in excess of method %RSD

Table 3. USGS summary results for the analysis of NIST SRM 2710*

Element	WDXRF	ICP-AES	INAA	LATA	value	+/-	limit	limit
Al %	6.45	6.50			6.44	0.08	6.6	6.28
Ctot				2.99	3		3.6	2.4
Ca	1.26	1.22	(1.82)		1.25	0.03	1.31	1.19
Fe	3.31	3.35	3.47		3.38	0.1	3.58	3.18
K	2.07	2.03	2.08		2.11	0.11	2.33	1.89
Mg	0.86	0.86			0.853	0.042	0.937	0.769
Na	(1.32)	1.12	1.11		1.14	0.06	1.26	1.02
P	0.12	0.11			0.106	0.015	0.136	0.076
Stot				0.02				
Si	(28.31)				28.97	0.18	29.33	28.61
Ti	0.27	0.13			0.283	0.01	0.303	0.263
Ag ppm		36	(28)		35.3	1.5	38.3	32.3
As		650	668	599	626	38	702	550
Au		<8	0.49		0.6		0.7	0.5
Ba		691	740		707	51	809	605
Cd		21	21		21.8	0.2	22.2	21.4
Ce		58	57.3		57		68	46
Co		(13)	9.11		10		12	8
Cr		(30)	(30.3)		39		47	31
Cs			109					
Cu		3048			2950	130	3210	2690
Ga		(16)			34		41	27
Hf			3.58		3.2		3.8	2.6
Hg				30	32.6	1.8	36.2	29
Ho			0.89		0.6		0.7	0.5
La		35.6	30.7		34		41	27
Li		37.6						
Mn	9956.80	9550			10100	400	10900	9300
Mo		20.3	24.2		19		23	15
Nb		16.3						
Nd		21.3	25.9		23		28	18
Ni		16	(10.1)		14.3	1	16.3	12.3
Pb		5588			5532	80	5692	5372
Rb			129		120		144	96
Sb			36		38.4	3	44.4	32.4
Sc		10	9.1		8.7		10.4	7.0
Se			0.28	0.55				
Sr		345	358		330		396	264
Tb			0.68					
Th			13		13		16	10
Tm			0.37					
U			27		25		30	20
V		77			76.6	2.3	81.2	72
W			91		93		112	74
Zn		6909	(6532)		6952	91	7134	6770
Zr			111					

* values corrected for percent moisture
concentrations in parentheses are outside AECR for SRM 2710

Appendix A. WD-XRF results for NIST SRM 2780

Lab No	Al %	Ca%	Fe, %	K %	Mg %	Mn %	Na %	P %	Si %	Ti %
F-027719	8.79	0.20	2.75	3.45	0.53	0.05	0.31	0.061	30.0	0.695
F-027720	8.84	0.21	2.73	3.45	0.54	0.05	0.31	0.061	30.0	0.695
F-027721	8.79	0.20	2.71	3.45	0.53	0.04	0.34	0.061	30.0	0.689
F-027722	8.79	0.21	2.73	3.44	0.54	0.05	0.31	0.065	29.9	0.695
F-027723	8.84	0.20	2.72	3.45	0.54	0.04	0.33	0.065	30.0	0.701
F-027724	8.79	0.20	2.71	3.45	0.52	0.05	0.33	0.061	30.0	0.701
F-027725	8.73	0.21	2.75	3.41	0.54	0.05	0.33	0.061	30.0	0.695
F-027726	8.79	0.20	2.72	3.42	0.54	0.04	0.33	0.057	30.0	0.701
F-027727	8.79	0.20	2.71	3.40	0.52	0.05	0.33	0.061	30.0	0.695
F-027728	8.73	0.20	2.69	3.39	0.53	0.05	0.31	0.061	29.9	0.701
F-027729	8.79	0.21	2.72	3.40	0.55	0.05	0.31	0.061	30.0	0.701
F-027730	8.79	0.21	2.73	3.39	0.54	0.05	0.32	0.061	30.0	0.695
F-027731	8.79	0.21	2.72	3.40	0.53	0.05	0.33	0.061	30.1	0.707
F-027732	8.79	0.21	2.73	3.46	0.54	0.04	0.32	0.070	30.1	0.701
F-027733	8.79	0.21	2.71	3.45	0.54	0.05	0.32	0.061	30.0	0.701
F-027734	8.79	0.21	2.74	3.46	0.55	0.05	0.33	0.065	30.1	0.701
F-027735	8.79	0.20	2.73	3.45	0.54	0.04	0.33	0.065	30.0	0.695
F-027736	8.79	0.20	2.76	3.45	0.54	0.05	0.33	0.065	30.0	0.695
F-027737	8.79	0.20	2.73	3.45	0.53	0.05	0.32	0.057	30.0	0.695
F-027738	8.84	0.20	2.75	3.45	0.54	0.05	0.33	0.065	30.1	0.701
F-027739	8.79	0.21	2.72	3.42	0.54	0.05	0.33	0.057	30.0	0.695
F-027740	8.79	0.21	2.72	3.42	0.54	0.05	0.32	0.065	30.0	0.701
F-027741	8.79	0.20	2.74	3.40	0.54	0.05	0.34	0.065	30.0	0.695
F-027742	8.84	0.21	2.72	3.41	0.54	0.05	0.31	0.065	30.0	0.695

Appendix B. ICP-AES results for NIST SRM 2780

Lab No	Al %	Ca %	Fe %	K %	Mg %	Na %	P %	Ti %	Ag ppm	As ppm	Au ppm	Ba ppm	Be ppm	Cd ppm	Ce ppm	Co ppm	Cr ppm
F-027719	8.4	0.19	2.7	3.1	0.51	0.20	0.04	0.61	27	68	< 8	850	2	13	63	2	46
F-027720	8.5	0.19	2.6	3.2	0.51	0.20	0.04	0.60	27	73	< 8	820	2	12	64	3	49
F-027721	8.7	0.19	2.7	3.3	0.51	0.21	0.04	0.63	26	72	< 8	890	2	12	64	2	47
F-027722	8.6	0.20	2.7	3.2	0.51	0.20	0.04	0.69	26	62	< 8	910	2	12	66	1	47
F-027723	8.7	0.19	2.7	3.2	0.52	0.21	0.05	0.69	28	80	< 8	920	2	13	67	2	50
F-027724	8.5	0.20	2.7	3.2	0.52	0.20	0.04	0.68	27	68	< 8	900	2	12	62	3	47
F-027725	8.8	0.19	2.7	3.3	0.51	0.21	0.05	0.65	26	71	< 8	920	2	12	67	2	53
F-027726	8.9	0.19	2.7	3.3	0.51	0.21	0.04	0.60	26	70	< 8	940	2	12	68	2	48
F-027727	8.9	0.19	2.7	3.3	0.52	0.22	0.04	0.60	27	79	< 8	910	2	13	65	2	47
F-027728	8.3	0.19	2.7	3.0	0.51	0.20	0.04	0.68	27	76	< 8	900	2	13	67	2	48
F-027729	8.4	0.17	2.7	3.2	0.51	0.21	0.04	0.62	28	75	< 8	830	2	12	50	2	46
F-027730	8.5	0.19	2.7	3.3	0.52	0.21	0.04	0.60	26	64	< 8	900	2	12	59	2	47
F-027731	8.4	0.18	2.7	3.2	0.51	0.21	0.04	0.60	26	74	< 8	820	2	13	57	2	46
F-027732	8.1	0.17	2.7	3.2	0.51	0.20	0.04	0.69	26	85	< 8	910	2	12	56	2	48
F-027733	8.3	0.17	2.7	3.2	0.50	0.21	0.04	0.68	26	68	< 8	890	2	13	57	1	46
F-027734	8.3	0.18	2.7	3.3	0.51	0.21	0.04	0.71	27	73	< 8	950	2	13	53	2	49
F-027735	8.4	0.18	2.7	3.2	0.51	0.21	0.04	0.65	27	68	< 8	960	2	13	59	2	50
F-027736	8.6	0.19	2.7	3.2	0.52	0.21	0.04	0.56	28	70	< 8	840	2	12	56	3	45
F-027737	8.4	0.19	2.7	3.1	0.51	0.20	0.04	0.63	27	65	< 8	850	2	12	59	3	46
F-027738	8.0	0.18	2.7	3.0	0.51	0.19	0.04	0.67	26	78	< 8	900	2	12	59	2	48
F-027739	8.5	0.18	2.7	3.3	0.51	0.21	0.04	0.67	27	78	< 8	930	2	12	57	2	48
F-027740	8.6	0.18	2.7	3.3	0.51	0.21	0.04	0.62	27	68	< 8	930	2	13	59	2	47
F-027741	8.8	0.19	2.7	3.3	0.51	0.22	0.04	0.62	27	65	< 8	900	2	13	61	2	46
F-027742	8.5	0.18	2.7	3.3	0.50	0.21	0.04	0.68	26	72	< 8	920	2	13	58	2	46

Lab No	Cu ppm	Ga ppm	Ho ppm	La ppm	Li ppm	Mn ppm	Mo ppm	Nb ppm	Nd ppm	Ni ppm	Pb ppm	Sc ppm	Sr ppm	Th ppm	U ppm	V ppm	Zn ppm
F-027719	200	26	< 4	39	18	440	12	18	28	12	5700	23	220	10	< 100	260	2900
F-027720	240	26	< 4	40	18	440	12	19	28	13	5600	24	220	11	< 100	250	2600
F-027721	220	25	< 4	41	18	450	12	18	28	12	5500	25	220	11	< 100	260	2700
F-027722	230	26	< 4	41	18	470	20	21	28	12	5500	25	230	10	< 100	260	2800
F-027723	210	27	< 4	42	18	440	12	18	29	13	5700	25	220	11	< 100	260	2800
F-027724	220	26	< 4	39	18	460	12	18	26	12	5800	24	220	9	< 100	260	2600
F-027725	200	26	< 4	42	18	440	12	18	29	13	5600	25	220	10	< 100	250	2700
F-027726	220	25	< 4	42	18	440	12	18	29	13	5600	26	220	11	< 100	250	2700
F-027727	240	26	< 4	41	18	450	12	20	29	12	5800	26	220	10	< 100	250	2900
F-027728	230	26	< 4	42	19	450	12	19	29	13	5700	23	220	11	< 100	260	2700
F-027729	220	26	< 4	31	18	440	12	18	22	12	5600	22	210	5	< 100	250	2600
F-027730	220	27	< 4	36	18	460	12	18	26	12	5700	22	210	6	< 100	260	2700
F-027731	230	26	< 4	36	18	450	12	17	25	12	5700	22	210	6	< 100	250	2800
F-027732	210	26	< 4	33	18	460	14	19	24	13	5600	21	210	6	< 100	260	2700
F-027733	210	26	< 4	34	18	450	22	17	24	12	5500	21	210	5	< 100	250	2800
F-027734	230	27	< 4	32	18	480	12	17	24	13	5800	21	210	5	< 100	260	3000
F-027735	210	26	< 4	36	18	450	12	18	26	13	5800	21	210	5	< 100	260	3000
F-027736	220	26	< 4	35	18	450	12	17	25	12	5700	23	220	6	< 100	250	2700
F-027737	200	26	< 4	37	18	460	12	18	26	12	5600	22	210	6	< 100	250	2600
F-027738	220	26	< 4	36	18	440	13	17	26	13	5700	20	210	6	< 100	260	2500
F-027739	220	26	< 4	35	18	450	12	18	25	12	5600	22	210	6	< 100	260	2700
F-027740	210	26	< 4	36	18	450	12	18	26	12	5800	23	220	6	< 100	250	2900
F-027741	210	26	< 4	38	18	460	14	18	26	12	5800	24	210	6	< 100	250	2700
F-027742	210	26	< 4	36	18	460	13	19	26	12	5500	22	220	6	< 100	260	2800

Appendix C. INAA results for NIST SRM 2780

lab no	Fe%	Ca %	Na %	K %	Ag ppm	As ppm	Au ppb	Ba ppm	Cd ppm	Ce ppm	Co ppm	Cr ppm	Cs ppm	Gd ppm	Hf ppm
F-027719	2.75	0.26	0.22	3.74	22.0	49.2	135	957	12.1	65.6	2.22	39.3	12.9	3.59	4.40
F-027720	2.78	0.25	0.22	3.77	23.1	49.0	155	952	13.8	66.9	2.26	40.1	13.0	3.58	4.40
F-027721	2.80	0.27	0.23	4.06	22.4	48.5	212	983	11.9	66.7	2.24	42.0	13.2	3.66	4.47
F-027722	2.79	0.27	0.22	4.06	22.3	48.6	165	998	12.8	67.5	2.19	41.4	13.1	3.62	4.41
F-027723	2.78	0.27	0.22	3.78	23.3	47.3	172	963	13.0	67.9	2.18	39.4	13.3	3.56	4.29
F-027724	2.82	0.26	0.22	4.05	23.0	48.7	168	957	10.9	67.8	2.33	40.5	13.3	3.59	4.45
F-027725	2.83	0.27	0.22	3.87	22.4	47.7	144	967	9.9	68.1	2.22	43.0	13.3	3.63	4.51
F-027726	2.73	0.27	0.22	3.94	21.6	47.8	143	936	11.5	65.9	2.16	40.0	13.0	3.51	4.33
F-027727	2.77	0.25	0.23	3.97	22.0	48.2	171	977	12.7	66.9	2.23	40.7	13.2	3.50	4.42
F-027728	2.81	0.26	0.22	3.92	23.4	48.7	140	973	12.0	67.2	2.21	42.5	13.2	3.57	4.47
F-027729	2.73	0.26	0.22	3.94	23.6	49.8	206	958	13.3	65.9	2.23	39.8	13.1	3.58	4.37
F-027730	2.79	0.27	0.22	3.96	23.4	48.6	204	961	11.4	68.8	2.21	39.2	13.2	3.60	4.40
F-027731	2.71	0.26	0.22	4.09	22.2	49.0	143	933	11.1	66.3	2.18	37.4	12.9	3.55	4.27
F-027732	2.76	0.29	0.22	4.44	22.8	46.4	138	943	11.3	66.9	2.22	40.4	13.2	3.64	4.45
F-027733	2.76	0.28	0.22	4.52	22.2	49.7	119	948	10.1	67.2	2.18	40.7	13.1	3.57	4.41
F-027734	2.74	0.31	0.23	4.35	22.2	47.4	181	966	10.1	68.5	2.14	38.5	12.8	3.53	4.34
F-027735	2.76	0.35	0.22	4.32	22.8	47.9	133	950	12.8	66.7	2.17	44.2	13.0	3.54	4.45
F-027736	2.76	0.30	0.22	3.77	22.4	45.9	199	991	12.8	66.1	2.18	39.2	13.0	3.66	4.39
F-027737	2.77	0.31	0.22	4.52	23.0	47.7	557	965	11.9	67.2	2.17	39.5	13.1	3.57	4.41
F-027738	2.78	0.36	0.22	4.25	23.3	50.8	203	1000	13.7	68.0	2.21	41.5	13.2	3.61	4.56
F-027739	2.80	0.32	0.21	4.65	21.9	48.0	201	965	13.7	67.1	2.26	42.8	13.2	3.55	4.41
F-027740	2.79	0.34	0.22	4.00	22.9	48.4	209	963	10.7	66.8	2.17	41.6	13.2	3.55	4.46
F-027741	2.77	0.37	0.22	4.08	22.7	46.0	135	970	12.5	66.2	2.25	41.0	12.9	3.63	4.46
F-027742	2.77	0.32	0.22	3.89	20.8	46.6	176	955	12.5	67.1	2.12	40.7	13.0	3.60	4.39

lab no	La ppm	Mo ppm	Nd ppm	Ni ppm	Rb ppm	Sb ppm	Sc ppm	Sr ppm	Tb ppm	Th ppm	Tm ppm	U ppm	W ppm	Zn ppm	Zr ppm
F-027719	37.0	10.50	29.8	24.1	173	157	23.4	288	0.56	11.2	0.41	3.93	20.1	2430	155
F-027720	37.7	9.92	29.7	25.1	173	159	23.7	290	0.55	11.3	0.39	3.92	22.2	2410	172
F-027721	37.9	9.71	30.9	20.3	176	162	24.2	288	0.58	11.6	0.39	4.01	16.8	2330	195
F-027722	37.8	10.10	30.2	23.3	172	160	24.0	283	0.58	11.6	0.42	3.98	21.1	2370	183
F-027723	37.4	12.70	30.5	17.5	173	161	23.8	287	0.58	11.4	0.39	3.90	17.5	2350	152
F-027724	37.9	10.60	29.9	18.2	175	161	24.2	294	0.57	11.7	0.42	4.03	22.7	2430	137
F-027725	37.8	10.30	31.0	17.0	176	162	24.1	278	0.59	11.6	0.37	4.04	16.2	2430	165
F-027726	36.9	10.20	29.6	21.6	170	156	23.4	293	0.58	11.3	0.39	3.95	17.2	2310	165
F-027727	37.7	8.52	31.4	25.2	172	158	23.8	286	0.57	11.4	0.40	3.90	14.8	2460	143
F-027728	37.9	9.53	31.3	23.7	173	161	23.5	302	0.58	11.5	0.41	4.07	19.8	2450	159
F-027729	37.0	11.10	30.5	18.1	172	160	23.6	298	0.58	11.4	0.42	4.05	18.7	2340	173
F-027730	37.7	9.12	29.6	17.3	173	160	23.8	297	0.59	11.5	0.41	3.91	20.6	2390	138
F-027731	37.9	10.30	31.2	16.3	168	155	23.5	293	0.56	11.2	0.38	3.78	28.1	2410	167
F-027732	37.8	9.71	30.3	23.2	173	158	23.8	287	0.57	11.4	0.39	3.88	19.9	2500	172
F-027733	37.3	11.40	30.1	16.5	171	157	23.7	290	0.59	11.4	0.40	3.93	19.0	2340	159
F-027734	38.2	12.50	31.7	18.6	170	155	23.6	290	0.58	11.4	0.41	3.92	18.5	2390	177
F-027735	37.8	9.30	29.8	14.4	173	156	23.7	307	0.58	11.4	0.39	3.97	22.8	2390	159
F-027736	37.3	12.80	29.9	17.1	172	156	23.7	317	0.59	11.5	0.37	3.91	20.7	2290	149
F-027737	37.7	9.98	31.1	16.5	174	157	23.9	314	0.57	11.6	0.38	3.99	14.2	2270	149
F-027738	38.1	13.40	31.5	18.8	175	160	24.0	308	0.60	11.6	0.39	3.98	18.4	2380	171
F-027739	37.5	9.80	30.6	16.9	173	161	23.6	288	0.57	11.6	0.39	3.92	18.6	2400	153
F-027740	37.5	10.70	30.0	18.5	174	158	23.9	291	0.58	11.6	0.40	3.94	34.8	2380	170
F-027741	37.4	9.86	30.9	20.1	174	160	23.3	299	0.58	11.3	0.41	4.00	21.2	2320	164
F-027742	37.4	11.30	31.2	19.6	173	155	23.7	282	0.60	11.4	0.41	3.97	15.8	2380	170

Appendix D. USGS single element results for NIST SRM 2780

Lab No	As, ppm	Se, ppm	Hg, ppm	Ctot %	Stot %
F-027719	49	5.3	0.72	0.2	1.26
F-027720	41	5.3	0.63	0.17	1.26
F-027721	40	5.3	1.1	0.17	1.24
F-027722	45	5.1	0.69	0.17	1.24
F-027723	38	5.2	0.87	0.2	1.23
F-027724	42	5.2	0.90	0.17	1.23
F-027725	42	5.0	0.68	0.17	1.24
F-027726	38	5.2	0.69	0.17	1.23
F-027727	45	5.3	0.73	0.17	1.21
F-027728	41	5.0	0.65	0.16	1.22
F-027729	51	5.3	0.70	0.17	1.21
F-027730	42	5.2	0.84	0.16	1.21
F-027731	38	5.2	0.67	0.17	1.22
F-027732	43	5.2	0.79	0.17	1.21
F-027733	47	5.2	0.66	0.17	1.24
F-027734	46	5.1	0.72	0.17	1.21
F-027735	44	5.1	0.67	0.17	1.24
F-027736	44	5.1	0.72	0.17	1.21
F-027737	46	5.1	0.65	0.18	1.23
F-027738	42	5.1	0.72	0.17	1.23
F-027739	47	5.1	0.69	0.17	1.22
F-027740	47	5.2	0.70	0.16	1.23
F-027741	42	4.9	0.72	0.17	1.21
F-027742	44	4.8	0.65	0.19	1.24

Appendix E. Contract lab single element results for NIST SRM 2780

Lab No.	As ppm	Au ppm	Cco3	Corg %	Ctot %	FeO %	Hg ppm	Sb ppm	Se ppm	Stot %	Te ppm	Tl ppm	W ppm
Method	(5)	(4)	(2)		(1)	(2)	(3)	(5)	(5)	(1)	(6)	(6)	(7)
C-115714	48.8	0.261	0.01	0.18	0.19	0.6	0.73	129	4.8	1.32	5.9	4.6	25
C-115707	54.2	0.199	0.01	0.15	0.15	0.7	0.69	130	4.4	1.3	4.5	4.7	26
C-115703	55	0.217	0.01	0.15	0.16	0.7	0.76	129	4.0	1.31	5.6	5.3	24
C-115715	53.5	0.188	0.01	0.19	0.20	0.7	0.51	133	4.6	1.24	5.6	4.6	25
C-115710	48.1	0.152	0.01	0.16	0.17	0.7	0.66	106	4.5	1.28	5.9	4.6	23
C-115698	50.8	0.199	0.01	0.14	0.15	0.7	0.79	121	3.8	1.31	4.4	5.3	20

1 LECO Comb IR
2 Potentionmetric titration
3 Cold Vapor AAS
4 Fire Assay
5 Hydride generation AAS
6 Graphite furnace-AAS
7 INAA