USGS
science for a changing world

Chapter J

The determination of forty-two elements in geological materials by inductively coupled plasma- mass spectrometry for NAWQA

By Paul H. Briggs and Allen L. Meier

U.S. Geological Survey Open File Report 02-223-J

Analytical methods for chemical analysis of geologic and other materials, U.S. Geological Survey

Contents

Tables

The Determination of Forty-Two Elements in Geological Materials by Inductively Coupled Plasma – Mass Spectrometry for NAWQA

By Paul H. Briggs and Allen L. Meier

Code: T22 Accepted: 03/07/02

Principle

In geological materials, 42 major, minor and trace elements are determined using ICP-MS. A multi-acid decomposition (a mixture of hydrochloric, nitric, perchloric, and hydrofluoric acids) is used to digest the powdered geological material (Crock and others, 1983). The ICP-MS is calibrated on a 1% nitric acid blank solution and two multi-element standard solutions to cover the mass range and generate the mass response curve.

Interferences

ICP-MS interferences come from matrix effects, instrumental drift, and isobaric overlap of some elemental isotopes (Perkin Elmer, 1997). Molecular ions formed in the plasma can also result in enhancement of measured ion intensity due to isobaric interferences. Internal standards are added to compensate for matrix effects and instrumental drift (Lichte and others, 1987). The isotopes measured are selected to minimize isobaric overlap from other elements and molecular species that might be present. For a few isotopes, isobaric overlap corrections are computed based on relative isotopic abundances of the elements involved, and oxide or double-charged ion intensities.

Sample decomposition using this multi-acid digestion technique is suited to dissolve certain rock types, soils, and sediments. As with any technique there are going to be exceptions. The method does not fully dissolve refractory or resistant minerals and some secondary minerals. Examples of incomplete digestion are as follows: Ba in barite, Cr in chromite, Ti in rutile, Sn in cassiterite, Al in corundum, and rear earth elements in monazite. Samples that contain elements in high concentrations where normally the element is a trace constituent will have to be diluted (ie, Mg in dolomite, Pb in galena, Zn in sphalerite, Cu in chalcopyrite). This dilution increases the lower reporting limits (Briggs, 1996).

Scope

Analysis by ICP-MS for major, minor, and trace elements is useful for a variety of geochemical investigations. The elements analyzed and the reporting limits are shown in Table 1.

Table 1. Elements analyzed and lower reporting limits (LRL) for elemental analysis of geological materials by ICP-MS.

Element	LRL	Element	LRL	Element	LRL
Aluminum, Al	.005 %	Cadmium, Cd	0.1 ppm	Nickel, Ni	2.0 ppm
Calcium, Ca	.005 %	Cerium, Ce	1.0 ppm	Lead, Pb	1.0 ppm
Iron, Fe	.005 %	Cobalt, Co	1.0 ppm	Antimony, Sb	0.1 ppm
Potassium, K	.005 %	Chromium, Cr	1.0 ppm	Scandium, Sc	2.0 ppm
Magnesium, Mg	.005 %	Copper, Cu	1.0 ppm	Tin, Sn	1.0 ppm
Sodium, Na	.005 %	Europium, Eu	1.0 ppm	Strontium, Sr	2.0 ppm
Phosphorus, P	.005 %	Gallium, Ga	1.0 ppm	Tantalum, Ta	1.0 ppm
Titanium, Ti	.005 %	Holmium, Ho	1.0 ppm	Thorium, Th	1.0 ppm
Silver, Ag	0.5 ppm	Lanthanum, La	1.0 ppm	Thallium, Tl	1.0 ppm
Arsenic, As	1.0 ppm	Lithium, Li	1.0 ppm	Uranium, U	0.1 ppm
Gold, Au	1.0 ppm	Manganese, Mn	4.0 ppm	Vanadium, V	2.0 ppm
Barium, Ba	1.0 ppm	Molybdenum, Mo	0.5 ppm	Yttrium, Y	1.0 ppm
Beryllium, Be	0.1 ppm	Niobium, Nb	4.0 ppm	Ytterbium, Yb	1.0 ppm
Bismuth, Bi	1.0 ppm	Neodymium, Nd	1.0 ppm	Zinc, Zn	2.0 ppm

Apparatus

- Inductively Coupled Plasma Mass Spectrometer, Perkin Elmer Elan 6000
- Hotplate with aluminum heating block
- 30 mL Teflon vessels with caps (Savillex)
- 13x100 mm disposable polypropylene test tubes with caps

Reagents

- Deionized (DI) water
- Hydrochloric acid (HCl), conc. reagent grade (37%)
- Nitric acid (HNO_3), conc. reagent grade (70%)
- Perchloric acid ($HClO_4$), conc. reagent grade (70%)
- Hydrofluoric acid (HF), conc. reagent grade (48%)
- 1% HNO_3 **:** Dilute 10 mL conc. HNO_3 to 1000 mL with DI water
- Hydrogen peroxide (H_2O_2), solution (30%)

Internal Standards

- Prepare 2 L of a solution containing 500 µg/L ^{6}Li, 20 µg/L Rh, and 10 µg/L Ir by performing serial dilutions of commercial aqueous standards using 1% HNO_3. This solution is mixed in a 1:1 ratio with the sample to be analyzed using a dual channel peristaltic pump equipped with a mixing manifold and coil.

Calibration Standards

- Standard 1 contains 40 µg/L of the following elements: Ag, As, Au, Ba, Be, Bi, Cd, Ce, Co, Eu, Ga, Ho, La, Li,, Mn, Mo, Nb, Nd, Ni, P, Pb, Sb, Sc, Sn, Sr, Ta, Th, Ti, Tl, U, V, Y, Yb; 80 µg/L Cu; 100 µg/L Mg; 150 µg/L Cr; 200 µg/L Al and Na; 400 µg/L K and Zn; 2000 µg/L Fe; 20000 µg/L Ca all in 1% HNO_3
- Standard 2 contains 100 µg/L Pb; 500 µg/L Ba; 1000 µg/L Mn; 20000 µg/L Fe and Na; 50000 µg/L Al and K; 100000 µg/L Mg; 200000 µg/L Ca all in 1% HNO_3

Safety Precautions

All laboratory personnel must wear safety glasses, a lab coat or apron, and gloves when working in the laboratory. All personnel must read the laboratory Chemical Hygiene Plan (CHP) and the Material Safety Data Sheets (MSDS) for each procedure.

Procedure

Prior to analyzing samples, a dual detector calibration and auto-lens adjustment are performed according to manufacturer's recommendations. The instrument is then calibrated using the calibration standards listed above, and the operating parameters shown in table 2.

Table 2. Operating conditions for Perkin Elmer Elan 6000 ICP-MS

Method Timing	
Sweeps/Reading	35
Readings/Replicate	1
Number of Replicates	1
Tuning File	Default.tun
Optimization File	Default.dac
QO Enabled:	Yes
Settling Time	Normal

Analyte	Mass	Scan Mode	MCA Ch	Dwell Time ms	Integration Time ms
Li	7.016	Peak Hopping	1	50	1750
Be	9.012	Peak Hopping	1	10	350
Na	22.990	Peak Hopping	1	10	350
Mg	24.986	Peak Hopping	1	10	350
Al	26.982	Peak Hopping	1	10	350
P	30.994	Peak Hopping	1	10	350
K	38.964	Peak Hopping	1	10	350
Ca	42.959	Peak Hopping	1	20	700
Sc	44.956	Peak Hopping	1	20	700
Ti	49.000	Peak Hopping	1	20	700
V	50.944	Peak Hopping	1	10	350
Cr	51.941	Peak Hopping	1	10	350
Mn	54.938	Peak Hopping	1	10	350
Fe	56.935	Peak Hopping	1	10	350
Co	58.933	Peak Hopping	1	10	350
Ni	59.933	Peak Hopping	1	20	700
Cu	64.928	Peak Hopping	1	20	700
Zn	65.926	Peak Hopping	1	30	1050
Ga	70.925	Peak Hopping	1	10	350
As	74.922	Peak Hopping	1	3	105
Sr	87.906	Peak Hopping	1	10	350
Y	88.905	Peak Hopping	1	20	700
Nb	92.906	Peak Hopping	1	20	700
Mo	97.906	Peak Hopping	1	20	700
Ag	108.905	Peak Hopping	1	10	350
Cd	113.904	Peak Hopping	1	10	350
Sn	117.902	Peak Hopping	1	20	700
Sb	120.904	Peak Hopping	1	10	350
Ba	137.905	Peak Hopping	1	10	350
La	138.906	Peak Hopping	1	10	350
Ce	139.905	Peak Hopping	1	10	350
Nd	145.913	Peak Hopping	1	10	350
Eu	150.920	Peak Hopping	1	20	700
Ho	164.930	Peak Hopping	1	20	700
Yb	171.937	Peak Hopping	1	10	350
Ta	180.948	Peak Hopping	1	40	1400
Au	196.967	Peak Hopping	1	10	350
Tl	204.975	Peak Hopping	1	3	105
Pb	207.977	Peak Hopping	1	20	700
Bi	208.980	Peak Hopping	1	10	350
Th	232.038	Peak Hopping	1	10	350
U	238.050	Peak Hopping	1	10	350

Signal Processing

Detector Mode	Dual
Measurement Units	Counts/sec
Auto Lens	On
Spectral Processing	Average
Signal Processing	Average
Blank Subtraction	After Internal Standard
Baseline Reading	0
Smoothing	Yes, factor 5

Multi-acid digestion:

1. Weigh 0.200 g sample into a Teflon vessel. Standard reference materials, duplicates, and three reagent blanks are taken through the digestion procedure.
2. Rinse sample from side walls of Teflon vessel with a minimum of DI water.
3. Slowly add 3 mL conc. HCl.
4. Add 2 mL conc. HNO_3 and allow any reaction to subside.
5. Add 1 mL conc. $HClO_4$ and 2 mL conc. HF.
6. Place Teflon vessels on aluminum heating block preset at 110°C and heat to incipient dryness. Raise the temperature to 160°C and bring sample to complete dryness. Remove Teflon vessels from hotplate.
7. Add 1 mL conc. $HClO_4$ and take to complete dryness at 160°C.
8. Remove Teflon vessels from hotplate and allow to cool.
9. Add 1 mL conc. HNO_3 and two drops of H_2O_2 and let reaction subside.
10. Add 19 mL 1% HNO_3, Cap vessels and heat in drying oven preset at 100°C for 30 minutes.
11. For ICP-MS analysis, make a 1:10 dilution of sample solution by taking 0.5 mL of sample solution and diluting with 4.5 mL 1% HNO_3 .Place diluted sample in polypropylene test tube and cap until ready for analysis.
12. Wash Teflon vessels and caps with soap and water, rinse with DI water and dry on drying rack.

Calculations

$$\text{Concentration (ppm or \%)} = \frac{\text{Sample volume (mL) x ICP-MS reading (ppm or \%)}}{\text{Sample weight (g)}}$$

Assignment of Uncertainty

The analytical results for selected reference materials are summarized in table 3.

Table 3. Analytical performance summary for elements in geological materials by ICP-MS

reference	*description*	*n*	*Mean*	*s*	*pv*	*% RSD*	*% R*
Al %							
NBS-2704	river sed	5	6.24	0.10	6.11	1.6	102
LKSD-3	lake sed	5	6.52	0.07	6.62	1.0	98
GSD-6	stream sed	5	7.32	0.23	7.49	3.2	98
TILL-4	till	5	7.58	0.24	7.62	3.2	99
GSD-2	stream sed	5	7.69	0.16	8.32	2.1	92
STSD-2	stream sed	5	8.32	0.04	8.52	0.5	98
Ca%							
GSD-2	stream sed	5	0.16	0.01	0.18	4.5	89
TILL-4	till	5	0.91	0.02	0.89	1.8	102
LKSD-3	lake sed	5	1.72	0.02	1.64	1.3	105
NBS-2704	river sed	5	2.69	0.04	2.60	1.4	103
GSD-6	stream sed	5	2.70	0.08	2.77	3.1	97
STSD-2	stream sed	5	2.97	0.04	2.86	1.3	104
Fe%							
GSD-2	stream sed	5	1.30	0.03	1.32	2.2	99
TILL-4	till	5	3.90	0.05	3.94	1.3	99
LKSD-3	lake sed	5	3.98	0.06	4.01	1.4	99
GSD-6	stream sed	5	3.92	0.12	4.11	2.9	95
NBS-2704	river sed	5	4.01	0.06	4.11	1.6	98
STSD-2	stream sed	5	5.23	0.11	5.25	2.1	100

Table 3. Analytical performance summary for elements in geological materials by ICP-MS ---Continued

reference	*description*	*n*	*Mean*	*s*	*pv*	*% RSD*	*% R*
K %							
STSD-2	stream sed	5	1.78	0.01	1.76	0.7	101
LKSD-3	lake sed	5	1.92	0.03	1.84	1.3	104
NBS-2704	river sed	5	2.05	0.02	2.0	1.0	103
GSD-6	stream sed	5	1.98	0.05	2.03	2.7	97
TILL-4	till	5	2.65	0.08	2.7	3.0	98
GSD-2	stream sed	5	4.14	0.10	4.31	2.5	96
Mg %							
GSD-2	stream sed	5	0.11	0.00	0.13	3.6	88
TILL-4	till	5	0.75	0.03	0.76	3.6	99
LKSD-3	lake sed	5	1.23	0.01	1.20	1.1	102
NBS-2704	river sed	5	1.23	0.03	1.20	2.4	103
GSD-6	stream sed	5	1.72	0.05	1.81	2.7	95
STSD-2	stream sed	5	1.85	0.02	1.88	0.8	99
Na %							
NBS-2704	river sed	5	0.60	0.01	0.55	1.8	109
STSD-2	stream sed	5	1.30	0.01	1.30	0.9	100
GSD-6	stream sed	5	1.64	0.05	1.71	3.2	96
LKSD-3	lake sed	5	1.76	0.02	1.72	1.1	102
TILL-4	till	5	1.89	0.05	1.82	2.5	104
GSD-2	stream sed	5	2.12	0.06	2.26	2.7	94
P %							
GSD-2	stream sed	5	0.02	0.000	0.02	1.5	81
TILL-4	till	5	0.10	0.002	0.09	2.2	106
GSD-6	stream sed	5	0.11	0.003	0.10	2.7	108
NBS-2704	river sed	5	0.10	0.003	0.10	2.8	104
LKSD-3	lake sed	5	0.11	0.001	0.11	0.8	104
STSD-2	stream sed	5	0.15	0.002	0.14	1.5	108
Ti %							
GSD-2	stream sed	5	0.11	0.00	0.14	4.4	76
LKSD-3	lake sed	5	0.32	0.00	0.31	0.9	102
NBS-2704	river sed	5	0.29	0.01	0.46	1.9	64
GSD-6	stream sed	5	0.40	0.01	0.47	2.8	86
STSD-2	stream sed	5	0.49	0.00	0.47	0.8	104
TILL-4	till	5	0.46	0.01	0.49	3.0	94
Ag ppm							
NBS-2704	river sed	5	0.39	0.13	—	33.3	—
TILL-4	till	5	0.23	0.10	—	42.4	—
GSD-2	stream sed	5	<.5	—	0.066	—	—
GSD-6	stream sed	5	0.10	0.14	0.36	137.6	28
STSD-2	stream sed	5	0.43	0.09	0.5	21.9	86
LKSD-3	lake sed	5	2.72	0.42	2.7	15.4	101

Table 3. Analytical performance summary for elements in geological materials by ICP-MS ---Continued

reference	*description*	*n*	*Mean*	*s*	*pv*	*% RSD*	*% R*
As ppm							
GSD-2	stream sed	5	8.56	0.96	6.2	11.2	138
GSD-6	stream sed	5	15.98	0.27	13.6	1.7	118
NBS-2704	river sed	5	24.90	1.20	23.4	4.8	106
LKSD-3	lake sed	5	28.46	2.18	27	7.6	105
STSD-2	stream sed	5	41.42	1.54	42	3.7	99
TILL-4	till	5	111.40	1.82	111	1.6	100
Au ppm							
LKSD-3	lake sed	5	<1	—	—	—	—
STSD-2	stream sed	5	<1	—	—	—	—
GSD-2	stream sed	5	<1	—	—	—	—
GSD-6	stream sed	5	<1	—	—	—	—
NBS-2704	river sed	5	<1	—	—	—	—
TILL-4	till	5	<1	—	—	—	—
Ba ppm							
GSD-2	stream sed	5	188.60	5.55	185	2.9	102
GSD-6	stream sed	5	330.80	7.26	330	2.2	100
TILL-4	till	5	396.40	5.41	395	1.4	100
NBS-2704	river sed	5	422.20	5.54	414	1.3	102
STSD-2	stream sed	5	518.40	4.83	540	0.9	96
LKSD-3	lake sed	5	684.80	11.95	680	1.7	101
Be ppm							
NBS-2704	river sed	5	2.09	0.10	—	5.0	—
GSD-6	stream sed	5	1.61	0.28	1.7	17.3	95
LKSD-3	lake sed	5	1.80	0.25	1.9	13.9	95
TILL-4	till	5	3.28	0.26	3.7	8.0	89
STSD-2	stream sed	5	4.91	0.62	5.2	12.7	94
GSD-2	stream sed	5	15.32	0.68	17.1	4.5	90
Bi ppm							
LKSD-3	lake sed	5	2.97	0.07	—	2.4	—
NBS-2704	river sed	5	<1	—	—	—	—
STSD-2	stream sed	5	3.88	0.14	—	3.7	—
GSD-2	stream sed	5	1.58	0.05	1.64	3.1	96
GSD-6	stream sed	5	4.87	0.24	5	4.9	97
TILL-4	till	5	43.86	1.05	40	2.4	110
Cd ppm							
TILL-4	till	5	0.50	0.08	—	15.5	—
LKSD-3	lake sed	5	0.53	0.06	—	11.8	—
STSD-2	stream sed	5	0.99	0.08	—	7.6	—
GSD-2	stream sed	5	0.36	0.06	0.065	16.3	557
GSD-6	stream sed	5	0.42	0.04	0.43	10.2	97
NBS-2704	river sed	5	3.85	0.26	3.45	6.9	111

Table 3. Analytical performance summary for elements in geological materials by ICP-MS ---Continued

reference	*description*	*n*	*Mean*	*s*	*pv*	*% RSD*	*% R*
Ce ppm							
GSD-6	stream sed	5	67.56	3.92	68	5.8	99
NBS-2704	river sed	5	56.92	2.60	72	4.6	79
TILL-4	till	5	76.34	2.43	78	3.2	98
LKSD-3	lake sed	5	88.88	1.17	90	1.3	99
STSD-2	stream sed	5	87.36	1.35	93	1.5	94
GSD-2	stream sed	5	173.20	5.36	192	3.1	90
Co ppm							
GSD-2	stream sed	5	2.15	0.15	2.6	7.1	83
TILL-4	till	5	7.73	0.21	8	2.7	97
NBS-2704	river sed	5	13.82	0.25	14	1.8	99
STSD-2	stream sed	5	20.10	0.16	19	0.8	106
GSD-6	stream sed	5	25.16	0.43	24.4	1.7	103
LKSD-3	lake sed	5	31.64	0.59	30	1.9	105
Cr ppm							
GSD-2	stream sed	5	10.58	0.67	12.2	6.4	87
TILL-4	till	5	42.34	1.33	53	3.1	80
LKSD-3	lake sed	5	81.84	0.52	87	0.6	94
STSD-2	stream sed	5	97.26	1.70	116	1.7	84
NBS-2704	river sed	5	135.00	2.00	135	1.5	100
GSD-6	stream sed	5	185.20	4.21	190	2.3	97
Cu ppm							
GSD-2	stream sed	5	3.06	0.46	4.9	15.0	62
LKSD-3	lake sed	5	33.90	1.18	35	3.5	97
STSD-2	stream sed	5	45.36	1.97	47	4.3	97
NBS-2704	river sed	5	96.08	3.57	98.6	3.7	97
TILL-4	till	5	239.00	7.31	237	3.1	101
GSD-6	stream sed	5	361.00	8.28	383	2.3	94
Eu ppm							
TILL-4	till	5	1.32	0.02	<1	1.6	—
GSD-2	stream sed	5	<1	—	0.49	—	—
NBS-2704	river sed	5	1.35	0.03	1.3	1.9	104
GSD-6	stream sed	5	1.72	0.03	1.5	1.9	115
LKSD-3	lake sed	5	1.71	0.03	1.5	1.7	114
STSD-2	stream sed	5	2.36	0.04	2	1.8	118
Ga ppm							
TILL-4	till	5	16.16	0.51	—	3.2	—
STSD-2	stream sed	5	20.24	0.17	—	0.8	—
LKSD-3	lake sed	5	14.36	0.32	—	2.2	—
NBS-2704	river sed	5	14.44	0.25	15	1.7	96
GSD-6	stream sed	5	15.10	0.36	16.7	2.4	90
GSD-2	stream sed	5	25.64	0.75	27.4	2.9	94

Table 3. Analytical performance summary for elements in geological materials by ICP-MS ---Continued

reference	*description*	*n*	*Mean*	*s*	*pv*	*% RSD*	*% R*
Ho ppm							
TILL-4	till	5	1.10	0.07	__	6.3	__
NBS-2704	river sed	5	1.16	0.04	__	3.2	__
LKSD-3	lake sed	5	1.48	0.02	__	1.1	__
STSD-2	stream sed	5	1.80	0.04	__	2.1	__
GSD-6	stream sed	5	1.02	0.02	0.78	1.8	130
GSD-2	stream sed	5	2.56	0.04	2.9	1.4	88
La ppm							
NBS-2704	river sed	5	31.72	1.92	29	6.1	109
GSD-6	stream sed	5	37.70	0.81	39	2.1	97
TILL-4	till	5	47.10	1.52	41	3.2	115
LKSD-3	lake sed	5	56.06	0.69	52	1.2	108
STSD-2	stream sed	5	60.18	1.06	59	1.8	102
GSD-2	stream sed	5	93.84	3.13	90	3.3	104
Li ppm							
LKSD-3	lake sed	5	27.60	0.46	25	1.7	110
TILL-4	till	5	29.40	0.57	30	1.9	98
GSD-6	stream sed	5	40.32	1.01	40	2.5	101
NBS-2704	river sed	5	45.16	0.70	47.5	1.6	95
STSD-2	stream sed	5	69.88	1.15	65	1.7	108
GSD-2	stream sed	5	100.56	2.61	101	2.6	100
Mn ppm							
GSD-2	stream sed	5	246.20	4.92	232	2.0	106
TILL-4	till	5	486.60	8.41	465	1.7	105
NBS-2704	river sed	5	576.00	6.63	558	1.2	103
GSD-6	stream sed	5	911.80	19.02	1007	2.1	91
STSD-2	stream sed	5	1016.00	8.94	1084	0.9	94
LKSD-3	lake sed	5	1410.00	10.00	1394	0.7	101
Mo ppm							
LKSD-3	lake sed	5	1.13	0.06	<5	5.3	__
GSD-2	stream sed	5	2.32	0.05	2	2.2	116
NBS-2704	river sed	5	3.98	0.08	5.2	2.1	77
GSD-6	stream sed	5	8.30	0.12	7.7	1.4	108
STSD-2	stream sed	5	12.96	0.09	13	0.7	100
TILL-4	till	5	15.32	0.16	16	1.1	96
Nb ppm							
NBS-2704	river sed	5	10.88	0.47	__	4.3	__
LKSD-3	lake sed	5	11.06	0.31	5	2.8	221
GSD-6	stream sed	5	10.11	0.83	12	8.2	84
TILL-4	till	5	17.10	0.14	15	0.8	114
STSD-2	stream sed	5	26.52	0.76	20	2.9	133
GSD-2	stream sed	5	126.40	9.07	95	7.2	133

Table 3. Analytical performance summary for elements in geological materials by ICP-MS ---Continued

reference	*description*	*n*	*Mean*	*s*	*pv*	*% RSD*	*% R*
Nd ppm							
TILL-4	till	5	35.08	1.51	30	4.3	117
NBS-2704	river sed	5	29.66	1.35	32	4.6	93
GSD-6	stream sed	5	31.62	0.44	33	1.4	96
STSD-2	stream sed	5	47.78	1.29	43	2.7	111
LKSD-3	lake sed	5	48.38	0.70	44	1.4	110
GSD-2	stream sed	5	62.46	1.70	62	2.7	101
Ni ppm							
GSD-2	stream sed	5	5.01	0.21	5.5	4.1	91
TILL-4	till	5	16.30	0.53	17	3.2	96
NBS-2704	river sed	5	41.44	0.79	44.1	1.9	94
LKSD-3	lake sed	5	50.02	0.93	47	1.9	106
STSD-2	stream sed	5	55.30	0.62	53	1.1	104
GSD-6	stream sed	5	73.76	1.69	78	2.3	95
Pb ppm							
GSD-6	stream sed	5	25.58	0.39	27	1.5	95
LKSD-3	lake sed	5	32.34	1.88	29	5.8	112
GSD-2	stream sed	5	39.38	1.13	32	2.9	123
TILL-4	till	5	48.78	0.69	50	1.4	98
STSD-2	stream sed	5	70.24	1.02	66	1.5	106
NBS-2704	river sed	5	155.80	7.56	161	4.9	97
Sb ppm							
GSD-2	stream sed	5	0.49	0.08	0.46	16.7	108
TILL-4	till	5	1.01	0.11	1	10.4	101
GSD-6	stream sed	5	1.57	0.05	1.25	3.4	125
LKSD-3	lake sed	5	1.20	0.05	1.3	4.3	92
NBS-2704	river sed	5	3.71	0.38	3.79	10.2	98
STSD-2	stream sed	5	4.15	0.11	4.8	2.8	87
Sc ppm							
GSD-2	stream sed	5	4.04	0.12	4.4	2.8	92
TILL-4	till	5	10.78	0.45	10	4.2	108
NBS-2704	river sed	5	11.86	0.21	12	1.7	99
LKSD-3	lake sed	5	12.08	0.28	13	2.3	93
STSD-2	stream sed	5	14.16	0.32	16	2.3	89
GSD-6	stream sed	5	15.32	0.37	17	2.4	90
Sn ppm							
TILL-4	till	5	18.78	0.33	—	1.7	—
GSD-6	stream sed	5	3.53	0.33	2.8	9.5	126
LKSD-3	lake sed	5	2.87	0.56	3	19.5	96
STSD-2	stream sed	5	5.51	0.10	5	1.7	110
NBS-2704	river sed	5	14.78	1.13	9.5	7.7	156
GSD-2	stream sed	5	18.82	1.18	29	6.3	65

Table 3. Analytical performance summary for elements in geological materials by ICP-MS ---Continued

reference	*description*	*n*	*Mean*	*s*	*pv*	*% RSD*	*% R*
Sr ppm							
GSD-2	stream sed	5	25.54	0.52	28	2.0	91
TILL-4	till	5	119.00	1.87	109	1.6	109
NBS-2704	river sed	5	129.60	1.67	130	1.3	100
LKSD-3	lake sed	5	251.20	4.15	240	1.7	105
GSD-6	stream sed	5	267.00	4.95	266	1.9	100
STSD-2	stream sed	5	415.20	6.42	400	1.5	104
Ta ppm							
LKSD-3	lake sed	5	<1	—	0.7	—	—
GSD-6	stream sed	5	<1	—	0.72	—	—
NBS-2704	river sed	5	<1	—	0.97	—	—
TILL-4	till	5	1.10	0.03	1.6	2.4	69
STSD-2	stream sed	5	1.39	0.06	1.6	4.4	87
GSD-2	stream sed	5	12.32	0.85	15.3	6.9	81
Th ppm							
GSD-6	stream sed	5	8.19	0.36	9	4.4	91
NBS-2704	river sed	5	8.45	1.05	9.2	12.5	92
LKSD-3	lake sed	5	10.88	0.24	11.4	2.2	95
STSD-2	stream sed	5	15.10	0.32	17.2	2.1	88
TILL-4	till	5	16.56	0.36	17.4	2.2	95
GSD-2	stream sed	5	71.02	1.49	70	2.1	101
Tl ppm							
STSD-2	stream sed	5	<1	—	—	—	—
TILL-4	till	5	1.33	0.06	—	4.5	—
LKSD-3	lake sed	5	<1	—	—	—	—
NBS-2704	river sed	5	1.13	0.04	1.06	3.2	107
GSD-6	stream sed	5	1.11	0.04	1.08	3.4	102
GSD-2	stream sed	5	2.29	0.06	1.9	2.6	120
U ppm							
GSD-6	stream sed	5	2.34	0.05	2.4	2.3	98
NBS-2704	river sed	5	2.77	0.06	3.13	2.1	88
LKSD-3	lake sed	5	4.48	0.08	4.6	1.8	97
TILL-4	till	5	3.97	0.19	5	4.8	79
GSD-2	stream sed	5	18.54	0.96	17	5.2	109
STSD-2	stream sed	5	17.16	0.27	18.6	1.6	92
V ppm							
GSD-2	stream sed	5	14.64	0.75	16.5	5.1	89
TILL-4	till	5	69.62	2.21	67	3.2	104
LKSD-3	lake sed	5	80.72	1.16	82	1.4	98
NBS-2704	river sed	5	94.48	1.40	95	1.5	99
STSD-2	stream sed	5	97.70	1.17	101	1.2	97
GSD-6	stream sed	5	134.00	2.83	142	2.1	94

Table 3. Analytical performance summary for elements in geological materials by ICP-MS ---Continued

reference	*description*	*n*	*Mean*	*s*	*pv*	*% RSD*	*% R*
Y ppm							
NBS-2704	river sed	5	23.54	0.24	__	1.0	__
GSD-6	stream sed	5	19.78	0.17	23.8	0.9	83
LKSD-3	lake sed	5	31.08	0.44	30	1.4	104
TILL-4	till	5	19.54	0.33	33	1.7	59
STSD-2	stream sed	5	38.28	0.45	37	1.2	103
GSD-2	stream sed	5	48.00	0.80	67	1.7	72
Yb ppm							
GSD-6	stream sed	5	2.29	0.07	2.1	2.8	109
LKSD-3	lake sed	5	3.48	0.11	2.7	3.2	129
NBS-2704	river sed	5	2.97	0.11	2.8	3.6	106
TILL-4	till	5	2.42	0.06	3.4	2.5	71
STSD-2	stream sed	5	4.01	0.02	3.7	0.4	108
GSD-2	stream sed	5	9.65	0.58	11	6.0	88
Zn ppm							
GSD-2	stream sed	5	40.50	1.51	44	3.7	92
TILL-4	till	5	65.68	2.45	70	3.7	94
GSD-6	stream sed	5	138.00	2.83	144	2.0	96
LKSD-3	lake sed	5	142.80	2.39	152	1.7	94
STSD-2	stream sed	5	241.40	3.21	246	1.3	98
NBS-2704	river sed	5	439.80	3.96	438	0.9	100

n= number of samples
s= standard deviation
pv= proposed value taken from published reference material compilations
GSD-2 and GSD-6 (Govindaraju, 1994)
LKSD-3 and STSD-2 (Lynch, 1990)and (Govindaraju, 1994)
TILL-4 (CCRMP, 1995)
NBS-2704 (Govindaraju, 1994)
%RSD= percent relative standard deviation
%R= percent recovery
__= no data

Table 3.—Continued—Duplicate samples results

Duplicate samples	**k**	**n**	**Mean**	**s**	**%RSD**	**MIN**	**MAX**	**<TOTAL**	**<PAIRS**
Al %	12	2	5.90	0.29	5.0	1.8	8.1	0	0
Ca %	12	2	4.09	0.26	6.3	0.27	15	0	0
Fe %	12	2	3.01	0.08	2.6	0.84	5.1	0	0
K %	12	2	1.74	0.10	5.9	0.77	2.9	0	0
Mg %	12	2	1.29	0.05	4.0	0.41	2.3	0	0
Na %	12	2	1.61	0.23	14	0.4	10	0	0
P %	12	2	0.09	0.01	6.5	0.029	0.14	0	0
Ti %	12	2	0.31	0.02	5.4	0.088	0.46	0	0
Ag ppm	11	2	0.32	0.06	18	0.17	0.58	3	1
As ppm	12	2	12.3	0.41	3.3	2.7	26	0	0

Table 3.—Continued—Duplicate samples results

Duplicate samples	k	n	Mean	s	%RSD	MIN	MAX	<TOTAL	<PAIRS
Au ppm	-	-	-	-	-	-	-	24	12
Ba ppm	12	2	664	16	2.4	280	1400	0	0
Be ppm	12	2	1.71	0.20	12	0.31	2.9	0	0
Bi ppm	-	-	-	-	-	-	-	24	12
Cd ppm	11	2	0.45	0.03	5.9	0.1	1	3	1
Ce ppm	12	2	57.7	1.6	2.7	18	120	0	0
Co ppm	12	2	11.4	0.22	2.0	5.6	18	0	0
Cr ppm	12	2	65.5	2.7	4.1	24	140	0	0
Cu ppm	12	2	31.0	0.79	2.6	10	58	0	0
Eu ppm	9	2	1.26	0.03	2.3	1	1.6	6	3
Ga ppm	12	2	13.5	0.24	1.8	3.6	20	0	0
Ho ppm	3	2	1.03	0.03	2.8	1	1.1	18	9
La ppm	12	2	31.3	0.92	2.9	8.6	58	0	0
Li ppm	12	2	47.0	0.89	1.9	9.1	85	0	0
Mn ppm	12	2	731	42	5.7	230	1800	0	0
Mo ppm	11	2	10.5	0.21	2.0	0.58	100	2	1
Nb ppm	11	2	11.9	0.56	4.7	7.7	19	2	1
Nd ppm	12	2	26.8	0.90	3.4	8.1	50	0	0
Ni ppm	12	2	31.5	0.58	1.8	8.4	70	0	0
Pb ppm	12	2	33.2	0.68	2.0	7	190	0	0
Sb ppm	12	2	1.49	0.05	3.1	0.15	3.9	0	0
Sc ppm	12	2	10.5	0.64	6.1	3.5	16	0	0
Sn ppm	12	2	2.34	0.13	5.7	1	4.1	2	0
Sr ppm	12	2	624	4.6	0.7	75	4800	0	0
Ta ppm	7	2	1.29	0.14	11.2	1	1.8	11	5
Th ppm	12	2	9.03	0.25	2.8	3.2	14	0	0
Tl ppm	-	-	-	-	-	-	-	24	12
U ppm	12	2	5.49	0.21	3.9	1.3	29	0	0
Y ppm	12	2	96.5	1.10	1.1	31	170	0	0
Y ppm	12	2	19.15	0.47	2.4	6.1	26	0	0
Yb ppm	11	2	2.01	0.10	4.8	1.3	2.8	2	1
Zn ppm	12	2	139	9.0	6.4	34	640	0	0

Table 3.–Continued--Method blank results 3s values are considered the lower limit of detection (LOD), and 5s values are considered the lower limits of determination (LLD)

BLANK	N	MEAN	s	3s	5s
Ag ppm	39	-0.00034	0.0007	0.0021	0.0036
Al ppm	39	-0.00000	11.0000	32.0000	53.0000
As ppm	39	-0.00007	0.0360	0.1100	0.1800
Au ppm	39	-0.00037	0.0007	0.0020	0.0033
Ba ppm	39	0.00000	0.0510	0.1500	0.2600
Be ppm	39	-0.00000	0.0058	0.0170	0.0290
Bi ppm	39	-0.01600	0.0110	0.0340	0.0560
Ca ppm	39	-0.00000	25.0000	76.0000	130.0000
Cd ppm	39	0.00000	0.0014	0.0042	0.0069
Ce ppm	39	0.00000	0.0180	0.0540	0.0900
Co ppm	39	-0.00005	0.0049	0.0150	0.0250
Cr ppm	39	0.00000	0.0960	0.2900	0.4800
Cu ppm	39	-0.00130	0.2900	0.8600	1.4000
Eu ppm	39	-0.00007	0.0003	0.0009	0.0015
Fe ppm	39	-0.00000	9.0000	27.0000	45.0000
Ga ppm	39	-0.00058	0.0026	0.0077	0.0130
Ho ppm	39	-0.00000	0.0002	0.0006	0.0010
K ppm	39	-0.00000	2.9000	8.8000	15.0000
La ppm	39	0.00000	0.0095	0.0290	0.0480
Li ppm	39	-0.00000	0.0630	0.1900	0.3100
Mg ppm	39	-0.00000	1.2000	3.5000	5.8000
Mn ppm	39	-0.00000	0.1400	0.4200	0.7100
Mo ppm	39	0.00000	0.0110	0.0320	0.0530
Na ppm	39	-0.00000	5.5000	16.0000	27.0000
Nb ppm	39	0.00024	0.0220	0.0650	0.1100
Nd ppm	39	0.00000	0.0050	0.0150	0.0250
Ni ppm	39	-0.00061	0.0520	0.1600	0.2600
P ppm	39	-0.00000	1.0300	3.0900	5.1500
Pb ppm	39	-0.00000	0.0740	0.2200	0.3700
Sb ppm	39	0.00000	0.0086	0.0260	0.0430
Sc ppm	39	0.00009	0.0069	0.0210	0.0340
Sn ppm	39	-0.00000	0.0250	0.0750	0.1200
Sr ppm	39	-0.00000	0.1700	0.5000	0.8300
Ta ppm	39	-0.00000	0.0024	0.0073	0.0120
Th ppm	39	-0.00000	0.0190	0.0570	0.0950
Ti ppm	39	-0.00000	0.8200	2.4600	4.1000
Tl ppm	39	0.00000	0.0160	0.0470	0.0790
U ppm	39	0.00000	0.0045	0.0140	0.0230
V ppm	39	0.00000	0.0290	0.0860	0.1400
Y ppm	39	0.00001	0.0094	0.0280	0.0470
Yb ppm	39	-0.00002	0.0012	0.0037	0.0061
Zn ppm	39	-0.00220	0.4500	1.4000	2.3000

Bibliography

Briggs, P.H., 1996, Forty elements by inductively coupled plasma-atomic emission spectrometry, in Arbogast, B.F., ed., Analytical methods manual for the Mineral Resource Program, U.S. Geological Survey Open-File Report 96-525, p.77-94.

Canadian Certified Reference Materials Project, 1995, Certificate of Analysis, TILL-1, TILL-2, TILL-3, and TILL-4.

Crock, J.G., Lichte, F.L., and Briggs, P.H., 1983, Determination of elements in National Bureau of Standards geological materials SRM 278 obsidian and SRM 688 basalt by inductively coupled plasma-atomic emission spectroscopy: Geostandards Newsletter, v.7, no.2, p. 335-340.

Govindaraju, K., ed.,1994, 1994 Compilation of Working Values and Sample Description for 383 Geostandards: Geostandards Newsletter, v. 18, Special Issue, 158 p.

Lichte, F.L., Meier, A.L., and Crock, J.G., 1987, Determination of the rare earth elements in geological materials by inductively coupled plasma mass spectrometry: Analytical Chemistry, v. 59, no.8, p. 1150-1157.

Lynch, J. 1990, Provisional elemental vales for eight new geochemical lake sediment and stream sediment reference materials LKSD-1, LKSD-2, LKSD-3, LKSD-4, STSD-1, STSD-2, STSD-3, AND STSD-4: Geostandards Newsletter, v.14, no.1, p.153-167.

Perkin Elmer Sciex Elan 6000 Software Guide, 1997.