Chapter G

The determination of forty elements in geological and botanical samples by inductively coupled plasma-atomic emission spectrometry

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Principle

Forty major, minor, and trace elements are determined in geological and botanical materials by inductively coupled plasma-atomic emission spectrometry (ICP-AES). The sample is decomposed using a mixture of hydrochloric, nitric, perchloric, and hydrofluoric acids at low temperature (Crock and others, 1983). The digested sample is aspirated into the ICP-AES discharge where the elemental emission signals are measured simultaneously for the forty elements. Calibration is performed with digested rock reference materials and a series of multi-element solution standards (Lichte and others, 1987).

Interferences

ICP-AES interferences may result from spectral interferences, background shifts, and matrix effects (Thompson and Walsh, 1983). Inter-element correction factors and background corrections are applied using the proprietary data system software (Perkin-Elmer, 1999). Approximately 100 spectral interference corrections are performed on each sample. Further corrections are made when an element influences other elements beyond the "normal correction." It is common to not report an effected element due to the extraordinary interference of the affecting element. Matrix effects can generally be negated by proper matching of standard and sample.

Sample decomposition using this multi-acid digestion technique is suited to dissolve most rock types, soils, sediments and ashed botanical samples. 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 rare earth elements in a monazite. Samples that contain elements in concentrations beyond the linear working range where normally the element is a trace constituent, will have to be diluted (i.e., Mg in a dolomite, Pb in a galena, Zn in a sphalerite, Cu in a chalcopyrite). This dilution increases the lower reporting limits.

Scope

Analysis by ICP-AES for major, minor, and trace elements is useful for a variety of geochemical investigations. The lower and upper reporting limits used for this method are shown in table 1. Approximately 150 samples can be analyzed daily by the ICP-AES instrumentation.

Table 1.—Reporting limits for 40 elements by ICP-AES

Element	*Concentration range*		*Element*	*Concentration range*	
Aluminum, Al	0.005	50 %	Gallium, Ga	4	50,000 ppm
Calcium, Ca	0.005	50 %	Holmium, Ho	4	5,000 ppm
Iron, Fe	0.005	25 %	Lanthanum, La	2	50,000 ppm
Potassium, K	0.005	50 %	Lithium, Li	2	50,000 ppm
Magnesium, Mg	0.005	5 %	Manganese, Mn	4	50,000 ppm
Sodium, Na	0.005	50 %	Molybdenum, Mo	2	50,000 ppm
Phosphorous, P	0.005	50 %	Niobium, Nb	4	50,000 ppm
Titanium, Ti	0.005	25 %	Neodymium, Nd	4	50,000 ppm
Silver, Ag	2	10,000 ppm	Nickel, Ni	3	50,000 ppm
Arsenic, As	10	50,000 ppm	Lead, Pb	4	50,000 ppm
Gold, Au	8	50,000 ppm	Scandium, Sc	2	50,000 ppm
Barium, Ba	1	35,000 ppm	Tin, Sn	5	50,000 ppm
Beryllium, Be	1	5,000 ppm	Strontium, Sr	2	15,000 ppm
Bismuth, Bi	10	50,000 ppm	Tantalum, Ta	40	50,000 ppm
Cadmium, Cd	2	25,000 ppm	Thorium, Th	4	50,000 ppm
Cerium, Ce	4	50,000 ppm	Uranium, U	100	100,000 ppm
Cobalt, Co	2	25,000 ppm	Vanadium, V	2	30,000 ppm
Chromium, Cr	2	50,000 ppm	Yttrium, Y	2	25,000 ppm
Copper, Cu	2	15,000 ppm	Ytterbium, Yb	1	5,000 ppm
Europium, Eu	2	5,000 ppm	Zinc, Zn	2	15,000 ppm

Apparatus

- Perkin Elmer Optima 3000 simultaneous ICP-AES
- Hot plate with 50-position aluminum heating block
- 30-mL Teflon vessels with caps (Savillex)
- Acid dispensers (Labindustries)
- Repeating pipet (Eppendorf)
- Drying oven set at 95°C
- 13x100 mm disposable polypropylene test tubes with caps

Reagents

Hydrochloric acid, HCl reagent grade, 37 percent
Nitric acid, HNO_3 reagent grade, 70 percent
Hydrofluoric acid, HF reagent grade, 48 percent
Perchloric acid, $HClO_4$ reagent grade, 70 percent
Deionized water (DI)

One percent nitric acid solution: 10 mL 70 percent conc HNO_3 diluted in 1000 mL DI water

Aqua regia: three parts conc HCl and one part conc HNO_3; solution is not stable and must be prepared immediately before use

Lutetium internal standard (Lu): 500 µg Lu/mL, as Lu_2O_3 in 5 percent (v/v) HCl

Safety precautions

All laboratory personnel are required to wear safety glasses, rubber gloves, and lab coats when working in the laboratory. All sample digestions must be performed in a perchloric acid hood; the latter is washed down after each day's use. Refer to the *CHP* and *MSDS* for specific precautions, effects of overexposure, disposal, and first-aid treatment, for reagents used in the digestion procedure and operating the ICP-AES instrumentation. Calcium gluconate gel should be available in labs where HF is in use.

Procedure

Digestion of samples

1. Weigh 0.200 g sample into Teflon vessel.
2. Add 100 μL Lu internal standard to each vessel with repeating pipet.
3. Rinse side walls of Teflon vessel with a minimum amount of DI water.
4. In the fume hood, slowly add 3 mL HCl and allow any reaction to subside.
5. Add 2 mL HNO_3, 1 mL $HClO_4$, and 2 mL HF. Place sample solution vessel on hot plate with aluminum heat block at a controlled temperature of 110°C in a perchloric acid fume hood.
6. Evaporate sample solution to complete dryness on hot plate (usually overnight).
7. Remove from hot plate, cool to touch and add 1 mL $HClO_4$ and 2 to 3 mL DI water.
8. Return to hot plate and evaporate to hard dryness. The temperature of the hot plate is increased to 160°C. This step usually takes a few hours.
9. Remove dried sample from hot plate and cool.
10. Add 1.0 mL *aqua regia* with repeating pipet and let react for 15 min.
11. Add 9.0 mL 1 percent HNO_3 and thread screw cap tightly on vessel. Place vessel in drying oven for 1 hour at a controlled temperature of 95°C.
12. Remove sample solution and cool. Transfer solution into labeled disposable polypropylene test tube and cap with test tube cap.
13. Analyze sample solution by ICP-AES.

ICP-AES analysis

The ICP-AES instrument is calibrated at the start of each day using a well characterized in-house geological reference material (Pikes Peak granite) and nine single and or multi-element solution standards. The major and trace elements are determined by comparing the element intensities obtained from the standards to those obtained from the samples. There are three method preparation blanks digested with each sample set. A blank subtraction is performed to negate the effect of the reagents. Table 2 shows instrumental operating conditions and element wavelengths for this method.

Table 2.—Operating conditions for determination of 40 elements by ICP-AES

RF power to the torch 1300W
Plasma argon flow rate ..... 15 L/min
Auxiliary argon flow rate ..... 0.5 L/min
Nebulizer argon flow rate ..... 0.75 L/min
Sample pump rate ..... 1 mL/min
Observation height above
load coil ..... 15 mm
Equilibration time 15s
Auto integration time ..... 0.5 to 20 s
Nebulizer ..... Modified Babington

Element	*Wavelength, nm*	*Element*	*Wavelength, nm*
Ag	328.0	Mg	279.0
Al	308.2	Mn	257.6
As	188.9	Mo	202.0
Au	242.7	Na	589.5
Ba	413.1	Nb	292.7
Be	313.0	Nd	406.1
Bi	223.0	Ni	231.6
Ca	430.2	P	213.6
Cd	226.5	Pb	220.3
Ce	413.7	Sc	424.6
Co	228.6	Sn	189.9
Cr	267.7	Sr	460.7
Cu	324.7	Ta	240.0
Eu	381.9	Th	350.9
Fe	273.9	Ti	223.0
Ga	294.3	U	385.9
Ho	345.6	V	292.4
K	766.4	Y	371.0
La	408.6	Yb	328.9
Li	670.7	Zn	213.8

Most elements in each sample data set are normalized (i.e. standardized) using well defined in-house reference materials (RM) that have undergone the sample digestion process. The normalized sample set is quality control checked by an independent, established RM that has undergone the sample digestion process also. The RM QC check is accepted if the recovery is within the upper and lower control limits of three times the standard deviation of the certified value, and the concentrations of the elements are >10 times the lower reporting limit. If the standardization is not accepted the sample set and in-house standards are redigested and reanalyzed.

Calculation

Element concentration = IRU/IRS × CONSTD × WT SOLN/WT SAMPLE +IEC

where

IRU = intensity of element/intensity of Lu
IRS = intensity of calibration standard/intensity of Lu
CONSTD = conc of calibration standard
WT SOLN = weight of final solution
WT SAMPLE = weight of sample
IEC = interelement corrections

Assignment of Uncertainty

The analytical results for the selected reference materials, duplicate samples, and method blanks are summarized in table 3. Please note: Some *pv* data has been converted from the oxide using the conversion factors in appendix A, table A1. Proposed values taken from the following published reference materials compilations: GSD-3, GSD-5 AND AGV-1 (Govindaraju, 1994), SRM 2709, 2710 and 2711 (NIST Certificate of Analysis, 1992), TILL-4 (CCRMP, 1995), and SRM 1571 pv values converted to ash weight from ash% of 9.2 (NBS Certificate of Analysis, 1977) .

Table 3.—Analytical performance summary for 40 elements by ICP-AES See page ix of the introduction to this Methods Manual for an explanation of the abbreviations used in the analytical performance summary tables.

Reference	Description	n	mean	s	pv	% RSD	% R
Aluminum, Al (%)							
SRM 1571	**orchard leaves**	7	0.44	0.01	---	2	---
GSD-3	**stream sediment**	35	6.11	0.1	6.4	2	95
SRM 2710	**soil**	5	6.33	0.08	6.44	1	98
SRM 2711	**soil**	46	6.53	0.02	6.53	0.3	100
TILL-4	**till**	17	7.32	0.2	7.62	2	96
SRM 2709	**soil**	46	7.42	0.2	7.5	2	99
GSD-5	**stream sediment**	40	8.08	0.2	8.19	2	99
AGV-1	**andesite**	6	9.13	0.2	9.07	2	101
Arsenic, As (ppm)							
AGV-1	**andesite**	6	<10	---	0.88	---	---
GSD-3	**stream sediment**	35	17.6	3	17.6	19	100
SRM 2709	**soil**	46	17.7	4	17.7	25	100
GSD-5	**stream sediment**	40	76.4	5	75	7	102
SRM 1571	**orchard leaves**	7	104	7	109	7	95
SRM 2711	**soil**	46	106	5	105	5	101
TILL-4	**till**	17	112	11	111	10	101
SRM 2710	**soil**	5	634	27	626	4	101

Table 3.—Analytical performance summary for 40 elements by ICP-AES--- Continued.

Reference	Description	n	mean	s	pv	% RSD	% R
Barium, Ba (ppm)							
TILL-4	**till**	17	407	15	395	4	103
SRM 1571	**orchard leaves**	7	456	4	478	1	95
GSD-5	**stream sediment**	40	464	24	440	5	106
GSD-3	**stream sediment**	35	626	19	615	3	102
SRM 2710	**soil**	5	707	7	707	1	100
SRM 2711	**soil**	46	723	7	726	1	100
SRM 2709	**soil**	46	939	20	968	2	97
AGV-1	**andesite**	6	1210	35	1226	3	99
Beryllium, Be (ppm)							
GSD-3	**stream sediment**	35	1.2	0.07	1.5	6	81
AGV-1	**andesite**	6	2.0	0.08	2.1	4	95
SRM 1571	**orchard leaves**	7	<2	---	---	---	---
SRM 2711	**soil**	46	2.2	0.1	---	5	---
GSD-5	**stream sediment**	40	2.2	0.1	2.3	5	98
SRM 2710	**soil**	5	2.5	0.07	---	3	---
TILL-4	**till**	17	3.0	0.1	3.7	4	82
SRM 2709	**soil**	46	3.7	0.6	---	16	---
Bismuth, Bi (ppm)							
SRM 2709	**soil**	46	<10	---	---	---	---
SRM 2710	**soil**	5	<10	---	---	---	---
SRM 2711	**soil**	46	<10	---	---	---	---
TILL-4	**till**	17	<10	---	40	---	---
GSD-3	**stream sediment**	35	<10	---	0.79	---	---
AGV-1	**andesite**	6	<10	---	0.057	---	---
GSD-5	**stream sediment**	40	<10	---	2.4	---	---
SRM 1571	**orchard leaves**	7	<20	---	---	---	---
Cadmium, Cd (ppm)							
TILL-4	**till**	17	<2	---	---	---	---
SRM 2709	**soil**	46	<2	---	0.38	---	---
GSD-5	**stream sediment**	40	<2	---	0.82	---	---
AGV-1	**andesite**	6	<2	---	0.069	---	---
GSD-3	**stream sediment**	35	<2	---	0.1	---	---
SRM 1571	**orchard leaves**	7	<4	---	1.2	---	---
SRM 2710	**soil**	5	20.3	2	21.8	9	93
SRM 2711	**soil**	46	40.1	3	41.7	7	96

Table 3.—Analytical performance summary for 40 elements by ICP-AES--- Continued.

Reference	Description	n	mean	s	pv	% RSD	% R
Calcium, Ca (%)							
GSD-3	**stream sediment**	35	0.127	0.0050	0.18	4	71
TILL-4	**till**	17	0.880	0.02	0.89	2	99
SRM 2710	**soil**	5	1.27	0.006	1.25	0.5	102
SRM 2709	**soil**	46	1.93	0.05	1.89	3	102
SRM 2711	**soil**	46	2.87	0.05	2.88	2	100
AGV-1	**andesite**	6	3.54	0.04	3.53	1	100
GSD-5	**stream sediment**	40	3.86	0.08	3.79	2	102
SRM 1571	**orchard leaves**	7	22.5	0.6	22.72	3	99
Cerium, Ce (ppm)							
SRM 1571	**orchard leaves**	7	11.2	0.8	---	7	---
SRM 2709	**soil**	46	45.9	2	42	5	109
SRM 2710	**soil**	5	59.4	3	57	5	104
GSD-3	**stream sediment**	35	67.4	4	64	6	105
AGV-1	**andesite**	6	69.3	3	67	4	103
SRM 2711	**soil**	46	74.6	4	69	6	108
TILL-4	**till**	17	81.1	5	78	7	104
GSD-5	**stream sediment**	40	93.2	6	89	6	105
Chromium, Cr (ppm)							
AGV-1	**andesite**	6	3.3	1	10.1	44	33
SRM 1571	**orchard leaves**	7	29.7	3	28.3	11	105
TILL-4	**till**	17	40.9	3	53	8	77
SRM 2711	**soil**	46	44.3	2	47	5	94
SRM 2710	**soil**	5	55.7	32	39	58	143
GSD-5	**stream sediment**	40	71.6	3	70	4	102
GSD-3	**stream sediment**	35	91.9	4	87	5	106
SRM 2709	**soil**	46	126	5	130	4	97
Cobalt, Co (ppm)							
SRM 1571	**orchard leaves**	7	<2	---	2.17	---	---
SRM 2710	**soil**	5	2.2	5	10	213	22
TILL-4	**till**	17	8.2	2	8	29	102
SRM 2711	**soil**	46	9.5	2	10	19	95
SRM 2709	**soil**	46	14.0	2	13.4	14	104
GSD-3	**stream sediment**	35	15.7	3	11.7	19	134
AGV-1	**andesite**	6	17.1	3	15.3	19	112
GSD-5	**stream sediment**	40	20.2	2	18.9	11	107

Table 3.—Analytical performance summary for 40 elements by ICP-AES--- Continued.

Reference	Description	n	mean	s	pv	% RSD	% R
Copper Cu (ppm)							
SRM 2709	**soil**	46	32.7	1	34.6	3	94
AGV-1	**andesite**	6	57.5	1	60	2	96
SRM 2711	**soil**	46	115	2	114	2	101
GSD-5	**stream sediment**	40	141	4	137	3	103
SRM 1571	**orchard leaves**	7	145	9	130	6	112
GSD-3	**stream sediment**	35	178	6	177	3	100
TILL-4	**till**	17	246	10	237	4	104
SRM 2710	**soil**	5	2900	25	2950	1	98
Europium, Eu (ppm)							
SRM 2709	**soil**	46	<1	---	0.9	7	99
SRM 2711	**soil**	46	1.1	0.06	1.1	6	99
SRM 2710	**soil**	5	1.1	0.06	1	6	110
TILL-4	**till**	17	1.2	0.07	<1	6	---
GSD-3	**stream sediment**	35	1.2	0.08	1.3	7	89
GSD-5	**stream sediment**	40	1.2	0.08	1.4	7	89
AGV-1	**andesite**	6	1.7	0.1	1.64	6	107
SRM 1571	**orchard leaves**	7	<2	---	---	9	---
Galium, Ga (ppm)							
SRM 1571	**orchard leaves**	7	<8	---	0.87	---	---
GSD-3	**stream sediment**	35	14.8	1	15.9	8	93
SRM 2709	**soil**	46	15.2	1	14	8	108
SRM 2711	**soil**	46	15.4	1	15	6	103
TILL-4	**till**	17	16.8	1	---	6	---
GSD-5	**stream sediment**	40	20.5	2	20.3	8	101
AGV-1	**andesite**	6	20.6	0.7	20	3	103
SRM 2710	**soil**	5	38.3	1	34	4	113
Gold, Au (ppm)							
SRM 2709	**soil**	46	<8	---	---	---	---
SRM 2711	**soil**	46	<8	---	0.03	---	---
SRM 2710	**soil**	5	<8	---	0.6	---	---
GSD-3	**stream sediment**	35	<8	---	---	---	---
AGV-1	**andesite**	6	<8	---	---	---	---
TILL-4	**till**	17	<8	---	---	---	---
GSD-5	**stream sediment**	40	<8	---	---	---	---
SRM 1571	**orchard leaves**	7	<16	---	---	---	---

Table 3.—Analytical performance summary for 40 elements by ICP-AES--- Continued.

Reference	Description	n	mean	s	pv	% RSD	% R
Holmium, Ho (ppm)							
SRM 2709	**soil**	46	<4	---	0.54	---	---
SRM 2710	**soil**	5	<4	---	0.6	---	---
SRM 2711	**soil**	46	<4	---	---	---	---
TILL-4	**till**	17	<4	---	---	---	---
GSD-3	**stream sediment**	35	<4	---	0.9	---	---
AGV-1	**andesite**	6	<4	---	0.67	---	---
GSD-5	**stream sediment**	40	<4	---	1.1	---	---
SRM 1571	**orchard leaves**	7	<8	---	---	---	---
Iron, Fe (%)							
SRM 1571	**orchard leaves**	7	0.3	0.02	0.326	7	104
SRM 2711	**soil**	46	2.9	0.05	2.89	2	99
SRM 2710	**soil**	5	3.3	0.06	3.38	2	99
SRM 2709	**soil**	46	3.5	0.07	3.5	2	101
TILL-4	**till**	17	4.0	0.1	3.94	2	101
GSD-5	**stream sediment**	40	4.1	0.09	4.08	2	100
GSD-3	**stream sediment**	35	4.7	0.1	4.57	3	103
AGV-1	**andesite**	6	4.7	0.08	4.74	2	100
Lanthanum, La (ppm)							
SRM 1571	**orchard leaves**	7	14.4	0.9	---	6	---
SRM 2709	**soil**	46	24.5	1	23	5	106
SRM 2710	**soil**	5	32.1	2	34	5	94
GSD-3	**stream sediment**	35	37.9	3	39	7	97
SRM 2711	**soil**	46	40.6	3	40	6	101
AGV-1	**andesite**	6	42.3	2	38	4	111
TILL-4	**till**	17	43.6	3	41	8	106
GSD-5	**stream sediment**	40	44.0	3	46	7	96
Lead, Pb (ppm)							
SRM 2709	**soil**	46	16.9	2	18.9	12	89
AGV-1	**andesite**	6	36.1	1	36	4	100
GSD-3	**stream sediment**	35	40.5	3	40	7	101
TILL-4	**till**	17	48.1	3	50	7	96
GSD-5	**stream sediment**	40	112	7	112	6	100
SRM 1571	**orchard leaves**	7	516	56	489	11	105
SRM 2711	**soil**	46	1110	76	1162	7	95
SRM 2710	**soil**	5	5360	471	5532	9	97

Table 3.—Analytical performance summary for 40 elements by ICP-AES--- Continued.

Reference	Description	n	mean	s	pv	% RSD	% R
Lithium, Li (ppm)							
SRM 1571	**orchard leaves**	7	6.4	0.2	6.52	2	99
AGV-1	**andesite**	6	10.6	0.9	12	8	88
SRM 2711	**soil**	46	27.3	0.8	---	3	---
TILL-4	**till**	17	30.0	1	30	4	100
GSD-3	**stream sediment**	35	32.5	0.9	33	3	99
SRM 2710	**soil**	5	40.2	0.7	---	2	---
GSD-5	**stream sediment**	40	47.4	1	45	3	105
SRM 2709	**soil**	46	55.9	1	---	2	---
Magnesium, Mg (%)							
GSD-3	**stream sediment**	35	0.39	0.007	0.41	2	94
GSD-5	**stream sediment**	40	0.57	0.009	0.58	2	98
TILL-4	**till**	17	0.73	0.01	0.76	2	96
SRM 2710	**soil**	5	0.85	0.006	0.853	1	100
AGV-1	**andesite**	6	0.91	0.006	0.92	1	99
SRM 2711	**soil**	46	1.05	0.01	1.05	1	100
SRM 2709	**soil**	46	1.52	0.02	1.51	2	101
SRM 1571	**orchard leaves**	7	6.69	0.1	6.74	2	99
Manganese, Mn (ppm)							
GSD-3	**stream sediment**	35	412	10	387	2	106
TILL-4	**till**	17	480	7	465	1	103
SRM 2709	**soil**	46	548	11	538	2	102
SRM 2711	**soil**	46	638	9	638	1	100
AGV-1	**andesite**	6	734	12	774	2	95
SRM 1571	**orchard leaves**	7	977	19	989	2	99
GSD-5	**stream sediment**	40	1150	25	1162	2	99
SRM 2710	**soil**	5	9500	163	10100	2	94
Molybdenum, Mo (ppm)							
GSD-5	**stream sediment**	40	<2	---	1.2	---	---
SRM 2709	**soil**	46	<2	---	2	---	---
AGV-1	**andesite**	6	<2	---	2.7	---	---
SRM 2711	**soil**	46	<2	---	1.6	---	---
SRM 1571	**orchard leaves**	7	<4	---	3.26	---	---
TILL-4	**till**	17	14.4	0.8	16	5	90
SRM 2710	**soil**	5	16.8	2	19	12	88
GSD-3	**stream sediment**	35	91.5	2	92	2	100

Table 3.—Analytical performance summary for 40 elements by ICP-AES--- Continued.

Reference	Description	n	mean	s	pv	% RSD	% R
Neodymium, Nd (ppm)							
SRM 1571	**orchard leaves**	7	<8	---	---	---	---
SRM 2710	**soil**	5	18.3	1	---	5	---
SRM 2709	**soil**	46	19.3	1	---	5	---
GSD-3	**stream sediment**	35	26.6	1	30	5	89
AGV-1	**andesite**	6	31.0	1	33	5	94
SRM 2711	**soil**	46	31.6	2	31	5	102
TILL-4	**till**	17	32.3	2	30	7	108
GSD-5	**stream sediment**	40	33.1	2	35	6	95
Nickel, Ni (ppm)							
SRM 1571	**orchard leaves**	7	16.0	0.3	14.1	2	113
SRM 2710	**soil**	5	16.4	0.3	14.3	2	114
AGV-1	**andesite**	6	16.8	0.2	16	1	105
TILL-4	**till**	17	19.4	0.7	17	3	114
SRM 2711	**soil**	46	21.5	0.7	20.6	3	105
GSD-3	**stream sediment**	35	28.5	1	25.6	4	111
GSD-5	**stream sediment**	40	37.2	1	34	4	109
SRM 2709	**soil**	46	80.6	3	88	4	92
Niobium, Nb (ppm)							
SRM 2710	**soil**	5	<4	---	---	---	---
SRM 1571	**orchard leaves**	7	<8	---	---	---	---
GSD-3	**stream sediment**	35	16.1	6	16	39	101
SRM 2709	**soil**	46	17.0	6	---	38	---
AGV-1	**andesite**	6	20.5	3	15	15	137
GSD-5	**stream sediment**	40	20.8	7	19	36	110
SRM 2711	**soil**	46	22.6	7	---	33	---
TILL-4	**till**	17	23.4	11	15	46	156
Phosphorus, P (%)							
GSD-5	**stream sediment**	40	0.062	0.003	0.06	5	104
GSD-3	**stream sediment**	35	0.063	0.003	0.06	5	104
SRM 2709	**soil**	46	0.065	0.003	0.062	4	105
SRM 2711	**soil**	46	0.085	0.004	0.086	4	99
TILL-4	**till**	17	0.095	0.002	0.09	2	106
SRM 2710	**soil**	5	0.118	0.004	0.106	4	111
AGV-1	**andesite**	6	0.224	0.009	0.22	4	102
SRM 1571	**orchard leaves**	7	2.50	0.1	2.28	5	110

Table 3.—Analytical performance summary for 40 elements by ICP-AES--- Continued.

Reference	Description	n	mean	s	pv	% RSD	% R
Potassium, K (%)							
GSD-5	**stream sediment**	40	1.8	0.05	1.69	3	105
GSD-3	**stream sediment**	35	1.9	0.05	2.02	3	96
SRM 2709	**soil**	46	2.0	0.1	2.03	6	97
SRM 2710	**soil**	5	2.1	0.03	2.11	2	98
SRM 2711	**soil**	46	2.4	0.1	2.45	5	98
AGV-1	**andesite**	6	2.5	0.09	2.42	3	102
TILL-4	**till**	17	2.5	0.07	2.7	3	94
SRM 1571	**orchard leaves**	7	13.8	1	15.98	9	86
Scandium, Sc (ppm)							
SRM 1571	**orchard leaves**	7	<4	---	---	---	---
SRM 2710	**soil**	5	9.6	0.3	8.7	3	111
SRM 2711	**soil**	46	9.7	0.3	9	3	108
TILL-4	**till**	17	11.2	0.2	10	2	112
SRM 2709	**soil**	46	12.3	0.3	12	3	102
AGV-1	**andesite**	6	12.8	0.2	12.2	2	105
GSD-3	**stream sediment**	35	13.8	0.4	14.3	3	97
GSD-5	**stream sediment**	40	14.4	0.3	14.5	2	99
Silver, Ag (ppm)							
TILL-4	**till**	17	<2	---	---	---	---
SRM 2709	**soil**	46	<2	---	0.41	---	---
GSD-5	**stream sediment**	40	<2	---	0.36	---	---
AGV-1	**andesite**	6	<2	---	0.078	---	---
GSD-3	**stream sediment**	35	<2	---	0.59	---	---
SRM 1571	**orchard leaves**	7	<4	---	---	---	---
SRM 2711	**soil**	46	4.6	0.2	4.63	4	99
SRM 2710	**soil**	5	35.1	1	35.3	4	100
Sodium, Na (%)							
SRM 1571	**orchard leaves**	7	0.092	0.005	0.089	5	103
GSD-3	**stream sediment**	35	0.20	0.006	0.24	3	85
GSD-5	**stream sediment**	40	0.27	0.008	0.3	3	91
SRM 2710	**soil**	5	1.12	0.03	1.14	3	98
SRM 2709	**soil**	46	1.16	0.03	1.16	3	100
SRM 2711	**soil**	46	1.17	0.03	1.14	2	103
TILL-4	**till**	17	1.81	0.06	1.82	3	99
AGV-1	**andesite**	6	3.13	0.04	3.2	1	98

Table 3.—Analytical performance summary for 40 elements by ICP-AES--- Continued.

Reference	Description	n	mean	s	pv	% RSD	% R
Strontium, Sr (ppm)							
GSD-3	**stream sediment**	35	87.1	3	90	3	97
TILL-4	**till**	17	116	2	109	2	106
GSD-5	**stream sediment**	40	210	4	204	2	103
SRM 2709	**soil**	46	231	5	231	2	100
SRM 2711	**soil**	46	247	4	245.3	2	101
SRM 2710	**soil**	5	325	3	240	1	135
SRM 1571	**orchard leaves**	7	438	10	402	2	109
AGV-1	**andesite**	6	669	4	662	1	101
Tantalum, Ta (ppm)							
SRM 2709	**soil**	46	<40	---	---	---	---
SRM 2710	**soil**	5	<40	---	---	---	---
SRM 2711	**soil**	46	<40	---	---	---	---
TILL-4	**till**	17	<40	---	1.6	---	---
GSD-3	**stream sediment**	35	<40	---	1.08	---	---
AGV-1	**andesite**	6	<40	---	0.9	---	---
GSD-5	**stream sediment**	40	<40	---	1.4	---	---
SRM 1571	**orchard leaves**	7	<80	---	---	---	---
Thorium, Th (ppm)							
AGV-1	**andesite**	6	5.4	0.5	6.5	10	82
SRM 1571	**orchard leaves**	7	<8	---	0.7	---	---
GSD-3	**stream sediment**	35	9.0	0.7	9.2	8	98
SRM 2709	**soil**	46	11.5	0.9	10.8	8	107
SRM 2711	**soil**	46	14.2	1	13.6	8	104
SRM 2710	**soil**	5	14.4	2	13.5	11	107
GSD-5	**stream sediment**	40	15.7	1	15.2	7	104
TILL-4	**till**	17	17.4	2	17.4	11	100
Tin Sn (ppm)							
SRM 2711	**soil**	46	<5	---	---	---	---
SRM 2709	**soil**	46	<5	---	---	---	---
AGV-1	**andesite**	6	<5	---	4.2	---	---
GSD-3	**stream sediment**	35	<5	---	3.4	---	---
GSD-5	**stream sediment**	40	<5	---	4.6	---	---
SRM 2710	**soil**	5	6.8	2	---	26	---
TILL-4	**till**	17	9.8	3	---	27	---
SRM 1571	**orchard leaves**	7	<10	---	---	---	---

Table 3.—Analytical performance summary for 40 elements by ICP-AES--- Continued.

Reference	Description	n	mean	s	pv	% RSD	% R
Titanium, Ti (%)							
SRM 1571	**orchard leaves**	7	<.1	---	---	---	---
SRM 2710	**soil**	5	0.13	0.01	0.283	4	45
SRM 2711	**soil**	46	0.28	0.01	0.306	3	90
SRM 2709	**soil**	46	0.34	0.01	0.342	2	99
TILL-4	**till**	17	0.42	0.02	0.49	5	87
GSD-5	**stream sediment**	40	0.45	0.03	0.54	6	83
GSD-3	**stream sediment**	35	0.50	0.05	0.64	10	79
AGV-1	**andesite**	6	0.67	0.03	0.63	5	107
Uranium, U (ppm)							
SRM 2709	**soil**	46	<100	---	3	---	---
SRM 2710	**soil**	5	<100	---	25	---	---
SRM 2711	**soil**	46	<100	---	---	---	---
TILL-4	**till**	17	<100	---	5	---	---
GSD-3	**stream sediment**	35	<100	---	1.86	---	---
AGV-1	**andesite**	6	<100	---	1.92	---	---
GSD-5	**stream sediment**	40	<100	---	2.6	---	---
SRM 1571	**orchard leaves**	7	<200	---	0.32	---	---
Vanadium, V (ppm)							
SRM 1571	**orchard leaves**	7	5.6	0.2	---	3	---
TILL-4	**till**	17	69.1	1	67	1	103
SRM 2710	**soil**	5	73.8	1	76.6	2	96
SRM 2711	**soil**	46	81.2	1	81.6	2	100
GSD-5	**stream sediment**	40	105.6	2	109	2	97
SRM 2709	**soil**	46	112.8	1	112	1	101
GSD-3	**stream sediment**	35	119.3	2	120	2	99
AGV-1	**andesite**	6	122.1	0.8	121	1	101
Ytterbium, Yb (ppm)							
AGV-1	**andesite**	6	1.4	0.1	1.72	8	80
GSD-3	**stream sediment**	35	1.6	0.3	2.6	19	61
SRM 2709	**soil**	46	1.7	0.1	1.6	7	103
TILL-4	**till**	17	1.7	0.2	3.4	13	50
SRM 1571	**orchard leaves**	7	<2	---	---	---	---
GSD-5	**stream sediment**	40	2.1	0.2	2.9	10	72
SRM 2710	**soil**	5	2.2	0.08	1.3	4	172
SRM 2711	**soil**	46	2.7	0.2	2.7	6	100

Table 3.—Analytical performance summary for 40 elements by ICP-AES--- Continued.

Reference	Description	n	mean	s	pv	% RSD	% R
Yttrium, Y (ppm)							
SRM 1571	**orchard leaves**	7	5.3	0.5	---	9	---
GSD-3	**stream sediment**	35	16.1	2	22	12	73
SRM 2709	**soil**	46	16.4	0.7	18	4	91
TILL-4	**till**	17	17.9	0.8	33	4	54
AGV-1	**andesite**	6	19.3	0.8	20	4	97
GSD-5	**stream sediment**	40	20.4	1	26	6	79
SRM 2710	**soil**	5	20.9	0.7	23	3	91
SRM 2711	**soil**	46	26.4	1	25	5	106
Zinc, Zn (ppm)							
GSD-3	**stream sediment**	35	47.2	2	52	4	91
TILL-4	**till**	17	65.0	2	70	4	93
AGV-1	**andesite**	6	88.8	2	88	3	101
SRM 2709	**soil**	46	103	3	106	3	97
GSD-5	**stream sediment**	40	251	7	243	3	103
SRM 1571	**orchard leaves**	7	300	8	272	3	110
SRM 2711	**soil**	46	346	8	350.4	2	99
SRM 2710	**soil**	5	6540	405	6952	6	94

Table 3.—Continued--Duplicate samples results

Duplicate samples	k	n	mean	s	%RSD	min	max	<total	<pairs
Ag ppm	6	2	27.0	0.9	3	2	120	150	75
Al %	81	2	5.94	0.1	2	0.05	11	0	0
As ppm	29	2	127	4	3	10	2800	104	52
Au ppm	0	2	-	-	-	-	-	162	81
Ba ppm	81	2	625	14	2	24	1200	0	0
Be ppm	65	2	3.78	0.1	2	1	110	32	16
Bi ppm	0	2	-	-	-	-	-	162	81
Ca %	81	2	2.46	0.1	4	0.08	29	0	0
Cd ppm	26	2	28.7	1	4	2	190	110	55
Ce ppm	73	2	78.8	6	7	18	180	16	8
Co ppm	69	2	17.6	2	9	1	180	24	12
Cr ppm	80	2	181	7	4	1	8500	2	1
Cu ppm	81	2	70.5	4	6	4	1700	0	0
Eu ppm	2	2	2.10	0.02	0.9	2	2	158	79
Fe %	81	2	3.21	0.05	2	0.06	17	0	0
Ga ppm	70	2	16.0	0.5	3	5	26	22	11
Ho ppm	0	2	-	-	-	-	-	162	81
K %	81	2	3.45	0.2	5	0.1	25	0	0
La ppm	78	2	37.9	3	7	2	96	6	3
Li ppm	77	2	32.9	1	3	3	100	8	4
Mg %	81	2	1.24	0.02	2	0.09	5	0	0
Mn ppm	81	2	1069	51	5	49	7700	0	0
Mo ppm	25	2	25.6	2	6	2	210	112	56
Na %	79	2	1.01	0.03	3	0.02	3	4	2
Nb ppm	66	2	15.8	2	15	5	56	30	15
Nd ppm	74	2	34.0	2	7	8	83	14	7
Ni ppm	81	2	69.2	3	5	3	1700	0	0
P %	81	2	0.444	0.01	3	0.009	7	0	0
Pb ppm	73	2	497	38	8	6	9700	16	8
Sc ppm	69	2	11.5	0.2	2	2	25	24	12
Sn ppm	12	2	11.8	3	23	5	56	138	69
Sr ppm	81	2	217	3	2	26	910	0	0
Ta ppm	0	2	-	-	-	-	-	162	81
Th ppm	71	2	11.3	1	8	4	27	20	10
Ti %	74	2	0.304	0.01	4	0.05	0.69	14	7
U ppm	0	2	-	-	-	-	-	162	81
V ppm	79	2	74.5	1	1	3	160	4	2
Y ppm	78	2	18.1	0.6	3	2	95	6	3
Yb ppm	68	2	2.00	0.1	7	1	5	26	13
Zn ppm	81	2	583	24	4	8	6300	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)

Method blank	n	Mean	s	3s	5s
Ag ppm	30	-0.01	0.01	0.04	0.06
Al %	30	0.00006	0.00002	0.00005	0.0001
As ppm	30	-0.7	1	4	6
Au ppm	30	-0.1	0.06	0.2	0.3
Ba ppm	30	0.08	0.09	0.3	0.5
Be ppm	30	0.0005	0.001	0.002	0.003
Bi ppm	30	0.5	0.5	1	2
Ca %	30	-8E-06	0.0001	0.0003	0.0004
Cd ppm	30	0.005	0.02	0.05	0.08
Ce ppm	30	0.2	0.1	0.4	0.7
Co ppm	30	-0.2	0.1	0.3	0.5
Cr ppm	30	-0.1	0.2	0.6	0.9
Cu ppm	30	0.001	0.01	0.03	0.1
Eu ppm	30	0.01	0.003	0.01	0.01
Fe %	30	-0.00004	0.00001	0.00004	0.00006
Ga ppm	30	-0.06	0.08	0.2	0.4
Ho ppm	30	0.03	0.02	0.06	0.1
K %	30	0.00002	4E-05	0.0001	0.0002
La ppm	30	-0.02	0.03	0.08	0.1
Li ppm	30	0.0005	0.01	0.03	0.05
Mg %	30	-0.00002	1E-05	4E-05	7E-05
Mn ppm	30	0.0005	0.005	0.01	0.02
Mo ppm	30	-0.08	0.1	0.4	0.6
Na %	30	0.000002	6E-06	2E-05	3E-05
Nb ppm	30	-0.5	0.2	0.5	0.8
Nd ppm	30	0.05	0.07	0.2	0.4
Ni ppm	30	0.1	0.05	0.1	0.2
P %	30	0.00003	4E-05	0.0001	0.0002
Pb ppm	30	-0.3	0.3	1.0	2
Sc ppm	30	-0.005	0.004	0.01	0.02
Sn ppm	30	-0.2	0.5	1	2
Sr ppm	30	0.04	0.1	0.4	0.7
Ta ppm	30	-0.07	0.2	0.5	0.8
Th ppm	30	-0.09	0.05	0.1	0.2
Ti %	30	-0.00006	0.00009	0.0003	0.0005
U ppm	30	0.3	0.5	1	2
V ppm	30	-0.01	0.008	0.02	0.04
Y ppm	30	-0.005	0.001	0.003	0.005
Yb ppm	30	0.01	0.004	0.01	0.02
Zn ppm	30	-0.05	0.03	0.1	0.2

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