Multi-Elemental Analysis of Aqueous Geological Samples by Inductively Coupled Plasma-Optical Emission Spectrometry

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U.S. Department of the Interior
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Conversion Factors

SI to Inch/Pound

Multiply	By	To obtain
	Length	
centimeter (cm)	0.3937	inch (in.)
millimeter (mm)	0.03937	inch (in.)
	Volume	
liter (L)	33.82	ounce, fluid (fl. oz)
	Flow rate	
liter per second (L/s)	15.85	gallon per minute (gal/min)
	Mass	
gram (g)	0.03527	ounce, avoirdupois (oz)
kilogram (kg)	2.205	pound avoirdupois (lb)

Concentrations of chemical constituents are given either in milligrams per liter (mg/L) or micrograms per liter (µg/L).

Multi-Elemental Analysis of Aqueous Geological Samples by Inductively Coupled Plasma-Optical Emission Spectrometry

By Todor I. Todorov[1], Ruth E. Wolf[2], and Monique Adams[2]

Principle

Typically, 27 major, minor, and trace elements are determined in natural waters, acid mine drainage, extraction fluids, and leachates of geological and environmental samples by inductively coupled plasma-optical emission spectrometry (ICP-OES). At the discretion of the analyst, additional elements may be determined after suitable method modifications and performance data are established. Samples are preserved in 1–2 percent nitric acid (HNO_3) at sample collection or as soon as possible after collection. The aqueous samples are aspirated into the ICP-OES discharge, where the elemental emission signals are measured simultaneously for 27 elements. Calibration is performed with a series of matrix-matched, multi-element solution standards.

Interferences

ICP-OES interferences may result from spectral interferences, background shifts, and matrix effects (Thompson and Walsh, 1983). Interelement correction factors (IECs) and background corrections are applied using PerkinElmer WinLab 32 version 4.0 proprietary data system software (Boss and Fredeen, 2004). Either one-point or two-point background correction is selected for all wavelengths determined by peak area or peak height measurements based on adjacent spectral features. Approximately 80 spectral interference corrections are performed on each sample. Further corrections are made when an element influences other elements beyond the normal range of correction. It is allowable, at the analyst's discretion, to not report an element affected by the extraordinary interference of another element or to flag its data as being estimated as a result of the interference. Interference corrections are generally made using the proprietary multicomponent spectral fitting (MSF) algorithm provided in the instrument operating software (Ivaldi and Barnard, 1993). Multicomponent spectral fitting requires that the two lines do not completely overlap. The MSF algorithm can correct the interference in a wide range of concentrations and analyte-to-interferent ratios. The MSF correction factors must be checked and regenerated on an annual basis or whenever significant instrument or method changes are made. Additionally, new MSF corrections should be generated every time a wavelength calibration is performed (not common unless the instrument is physically moved or a hardware problem is experienced). See appendix 1 for a detailed procedure explaining generation of new MSF correction factors.

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[1] U.S. Department of Health and Human Services, Food and Drug Administration

[2] U.S. Geological Survey

All analyses are performed using internal standardization (Boss and Fredeen, 2004). In this procedure, a known amount of the selected internal standardization element is added to all blanks, standards, and samples presented to the ICP-OES instrument. The ratios of the resulting analytical signals for each element of interest to the measured intensity of the internal standard element are then calculated. The purpose of the internal standard is twofold: (1) to compensate for instrument drift due to changes of the sample introduction system over time (for example, nebulizer or injector clogging and tubing degradation), and (2) to compensate for differences in sample matrix characteristics (for example, viscosity and ionization efficiency) that may affect sample uptake rates or ionization efficiencies. The internal standard element is selected by determining which element is a good proxy for the elements of interest, and it cannot be present to any significant extent in the samples or be one of the elements of interest. To use internal standardization, the internal standard element must be added to the calibration blanks, standards, and samples at the same concentration. Generally, internal standards are added online using a mixing tee in order to facilitate high-throughput analysis, reduce analyst time preparing individually spiked standards and samples, eliminate errors caused by spiking, and minimize the amount of internal standard used. However, the use of online internal standardization can cause undesirable dilution of the sample and the analyst may choose instead to individually and manually spike all solutions with the internal standard solution.

Scope

Analysis by ICP-OES for major and minor elements in aqueous samples is useful for a variety of geochemical investigations. The wavelengths used for this method, including those monitored for interference effects, are shown in table 1. Approximately 200 aqueous samples can be analyzed in 27 hours by the ICP-OES instrumentation.

Table 1. Elements and wavelengths for ICP-OES analysis.

[nm, nanometer; a, axial detection; r, radial detection; Lu, Lutetium was used as an internal standard]

Element	Wavelength, nm
Ag	328.066 (a), 338.288 (a)
Al	396.151 (r), 308.212 (r)
As	188.980 (r), 193.696 (r, a)
B	249.772 (r), 249.676 (r)
Ba	455.402 (r), 233.524 (r)
Be	313.104 (r), 313.042 (r)
Ca	317.932 (r), 422.673 (r)
Cd	226.502 (a), 214.434 (a)
Co	228.615 (a), 230.785 (r)
Cr	205.560 (a), 267.711 (r)
Cu	324.754 (r), 327.393 (a)
Fe	238.201 (r), 239.568 (a)
K	766.490 (r)
Li	670.784 (r)
Mg	285.212 (r), 279.076 (r)
Mn	257.607 (r), 293.305 (r)
Mo	202.032 (a), 203.846 (a)
Na	589.587 (r), 330.237 (r)
Ni	231.604 (r, a)
P	213.617 (r), 178.224 (r)
Pb	220.354 (r, a)
S[2]	180.671 (a), 181.975 (a)
Sb	206.834 (a), 217.582 (r)
Se[1]	196.029 (r, a)
Si	251.609 (r), 221.663 (r)
Sr	421.549 (r), 460.733 (r)
Th[1]	283.730 (r), 401.913 (r)
Ti[1]	334.943 (r), 368.519 (r)
Tl[1]	190.801 (r)
V	292.402 (a), 290.880 (r)
U[1]	409.014 (a, r)
W[1]	207.912 (r), 239.708 (r)
Zn	213.858 (a), 206.199 (a)
Lu	291.138 (r, a)

[1] Monitored for interference, not reported

[2] S is reported as sulfate (SO_4)

Apparatus

- Simultaneous dual-view ICP-OES system equipped with an autosampler (PerkinElmer Optima 5300DV, PerkinElmer, Waltham, Mass., or equivalent)
- Burgener PEEK MiraMist Nebulizer PMM 4000 (Burgener Research, Inc., Mississauga, Ontario, Canada)

- 13 x 100 millimeter (mm) disposable, polypropylene test tubes with caps (VWR International, vials part number 60818-860 and caps part number 60828-738)
- Adjustable mechanical or electronic pipettes, typical volume ranges needed are 5–120 microliters (µL), 50–1,000 µL, and 100–5,000 µL
- 15 and 50 milliliter (mL) BD Falcon polypropylene tubes (BD Biosciences, San Jose, Calif.).
- Peristaltic pump tubing, polyvinyl chloride (PVC) type (Meinhard Inc., Golden, Colo.):
  - Internal standard: Green-orange (0.38 mm inner diameter [i.d.])
  - Sample: Black-black (0.76 mm i.d.)
  - Drain: Red-red (1.14 mm i.d.)
- Autosampler peristaltic pump tubing, Santoprene type: Red-red (1.14 mm i.d.), 3-stop (Meinhard Inc., Golden, Colo.)
- Plastic mixing tee (1/16” i.d.) (EW-30506-15), (Cole-Parmer, Vernon Hills, Ill.)
  - 20 milligrams per liter (mg/L) Lu internal standard solution (High Purity Standards, Charleston, S.C.)

Reagents

- Concentrated nitric acid, HNO_3, reagent grade, 70 percent weight/weight (wt/wt) for rinse solution preparation (VWR International, Radnor, Pa.)
- Concentrated ultra-pure double distilled HNO_3, 70 percent wt/wt, prepared via sub-boiling distillation (Reagent grade HNO_3, VWR International, Radnor, Pa., purified using Milestone DuoPur, Mileston, Shelton, Conn.)
- Deionized water (DI), 18 megohm (MΩ) (Milli-Q, EMD Millipore, Billerica, Mass.)
- 2 percent weight/volume (wt/vol) nitric acid solution: 56 mL double distilled 70 percent HNO_3 diluted to 2,000 mL using DI water
- Single element standards: 10,000 mg/L of Al, Ca, Fe, K, Mg, Na, and 1,000 mg/L of Ag, As, B, Ba, Be, Bi, Cd, Co, Cr, Cu, Li, Lu, Mn, Mo, Ni, P, Pb, S, Sb, Se, Si, Sr, Th, Ti, Tl, U, V, W, Zn, and Zr (High Purity Standards, Charleston, S.C.)
- Lutetium internal standard solution (20 mg/L Lu): 40.30 grams (g) 1,000 mg/L Lu diluted in 100 mL double distilled 70 percent HNO_3 and 1,860 mL DI water.

Safety Precautions

All laboratory personnel are required to wear safety glasses, gloves, closed toe shoes, and lab coats when working in the laboratory. Refer to the laboratory chemical hygiene plan and material safety data sheets for specific precautions, effects of overexposure, and first-aid treatment for reagents used in the preparation procedure and operation of the ICP-OES system.

Sample Preparation

Samples need to be preserved in 1–2 percent wt/vol HNO_3 at sample collection or as soon as possible after sample collection. Most sampling protocols require that the samples are filtered and acidified in the field (USGS, 2006; Wanty, 1999; Wilde, 2004). At the analyst’s discretion, unfiltered samples collected as raw acidified (RA) may be filtered or decanted prior to analysis if visible particulates or solids are present to prevent clogging of the sample introduction system (for example, sample probe or nebulizer). The analyst should notify the sample submitter if this or any other procedures were necessary to analyze the samples. If a sample result is over the range of the calibration curve, dilution and re-analysis of the sample(s) for the over range elements are required.

Calibration Standards

All calibration standards are prepared in 2 percent HNO_3 starting with 10,000 mg/L or 1,000 mg/L single-element solutions using six stock solutions (table 2). The final volume required is 50 mL for all calibration standard solutions. The preparation and concentrations of the calibration standards are shown in tables 3a and 3b, respectively. Alternatively, final calibration standards may be prepared using commercially available or custom-mixed multi-element standards.

Table 2. Stock solution preparation.

[mg/L, milligrams per liter; mL, milliliter; %, percent; HNO_3, nitric acid; all concentrations in mg/L; final standard volume is 50 mL]

Stock 1 preparation
2.5 mL each of 1,000 mg/L high purity standard solution of As, Cu, Mn, Mo, Pb, Si, Zn, P, S
0.25 mL each of 1,000 mg/L high purity standard solution of Ag, B, Ba, Be, Bi, Cd, Co, Cr, Li, Ni, Sb, Se, Sr, Th, Ti, Tl, U, V, W, Zr
Add 6 mL of concentrated distilled HNO_3
Dilute to 50 mL with 2% nitric acid
Stock 2 preparation
1 mL Stock 1
Dilute to 50 mL with 2% HNO_3
Stock 3 preparation
5 mL each of 10,000 mg/L high purity standard solution of Ca, K, Mg, Na
Dilute to 50 mL with 2% HNO_3
Stock 4 preparation
2.5 mL Stock 3
Dilute to 50 mL with 2% HNO_3
Stock 5 preparation
5 mL each of 10,000 mg/L high purity standard solution of Al, Fe
Dilute to 50 mL with 2% HNO_3
Stock 6 preparation
0.1 mL Stock 5
Dilute to 50 mL with 2% HNO_3

Table 3A. Calibration standard preparation.

[mL, milliliter; %, percent; HNO_3, nitric acid; mg/L, milligrams per liter]

	Volume of stock solutions (mL) to prepare 50 mL calibration standard						
	Stock 1	**Stock 2**	**Stock 3**	**Stock 4**	**Stock 5**	**Stock 6**	**2% HNO_3**
Standard 1		0.5		1		0.25	48.25
Standard 2		2		4		1	43
Standard 3		10	1			5	34
Standard 4	1		2.5		0.1		46.4
Standard 5	10		5		5		30

Table 3B. Calibration standard concentrations.

[mg/L, milligrams per liter]

Element	Standard 1	Standard 2	Standard 3	Standard 4	Standard 5
	Standard concentration, mg/L				
Ag	0.001	0.004	0.02	0.1	1
Al	0.01	0.04	0.2	2	100
As	0.01	0.04	0.2	1	10
B	0.001	0.004	0.02	0.1	1
Ba	0.001	0.004	0.02	0.1	1
Be	0.001	0.004	0.02	0.1	1
Bi	0.001	0.004	0.02	0.1	1
Ca	1	4	20	50	100
Cd	0.001	0.004	0.02	0.1	1
Co	0.001	0.004	0.02	0.1	1
Cr	0.001	0.004	0.02	0.1	1
Cu	0.01	0.04	0.2	1	10
Fe	0.01	0.04	0.2	2	100
K	1	4	20	50	100
Li	0.001	0.004	0.02	0.1	1
Mg	1	4	20	50	100
Mn	0.01	0.04	0.2	1	10
Mo	0.01	0.04	0.2	1	10
Na	1	4	20	50	100
Ni	0.001	0.004	0.02	0.1	1
P	0.01	0.04	0.2	1	10
Pb	0.01	0.04	0.2	1	10
S[1]	0.01	0.04	0.2	1	10
Sb	0.001	0.004	0.02	0.1	1
Se	0.001	0.004	0.02	0.1	1
Si	0.01	0.04	0.2	1	10
Sr	0.001	0.004	0.02	0.1	1
Th	0.001	0.004	0.02	0.1	1
Ti	0.001	0.004	0.02	0.1	1
Tl	0.001	0.004	0.02	0.1	1
U	0.001	0.004	0.02	0.1	1
V	0.001	0.004	0.02	0.1	1
W	0.001	0.004	0.02	0.1	1
Zn	0.01	0.04	0.2	1	10
Zr	0.001	0.004	0.02	0.1	1

[1] S is reported as sulfate (SO_4)

ICP-OES Analysis

Table 4 shows instrumental operating conditions for this method using a PerkinElmer Optima 5300DV. The elemental wavelengths and viewing position are given in table 1. All analyses are performed using online internal standardization. The internal standard solution delivered from the orange-green (0.38 mm i.d.) peristaltic pump tubing is mixed with the sample solutions coming from the black-black (0.76 mm i.d.) tubing using a polypropylene barbed tee-fitting (Cole Parmer, EW-30506-15) prior to introduction into the nebulizer. The internal standard is diluted by a 1:4 ratio, resulting in a Lu internal standard concentration of approximately 5 mg/L. The sample flow rate is set to 0.8 milliliter per minute (mL/min) in the instrument software.

Table 4. Instrument parameters and conditions for the determination of 27 elements in aqueous solutions by ICP-OES.

[W, watts; L/min, liters per minute; mL/min, milliliters per minute; mm, millimeters; i.d., inner diameter; s, seconds]

Instrument parameters	Conditions
RF power	1450 W
Cool gas (L/min)	13
Auxilary gas (L/min)	0.4
Nebulizer gas (L/min)	0.60-0.68
Injector	2 mm quartz
Spray chamber	Cyclonic, quartz
Sample flow (mL/min) (black/black tubing, 0.76 mm i.d.)	0.8
Integration time (s)	0.1 to 10 (Auto)
Nebulizer	Burgener PEEK Mira Mist
Rinse time (s)	120 at 2 mL/min uptake rate
Specific method name	water02TT 20120711

Once a 30-minute warm-up time has been completed after plasma ignition, the instrument sensitivity is checked while aspirating a 2 percent HNO_3 solution mixed with the 20 mg/L Lu internal standard solution using the online tee fitting. The method minimum specification for the Lu signal is 20,000 counts-per-second (cps) for radial and 150,000 cps for axial detection mode. The sensitivity requirements must be met for both modes since elements are analyzed in both radial and axial detection modes. The sensitivity readings should be recorded in the instrument logbook. If the minimum sensitivities are not met, corrective action must be taken before sample analyses are continued. The most common causes of low sensitivity include: worn or improperly tensioned pump tubing, clogging of the nebulizer, partial clogging of the torch injector, or improper positioning of radial viewing height or axial viewing position.

The ICP-OES instrument is calibrated at the start of each analytical run using a blank and five multi-element solution standards (see table 3) using a linear through zero calibration. The calibration blank should be prepared from the same source and at the same acid concentration used in the calibration standards. Blank subtraction is performed to negate the effect of the reagents in instances where the blank is higher than the detection limit. The performance of the method is checked using a minimum of three aqueous quality control samples or certified reference materials (CRMs) available from USGS Standard Reference Sample Project (*http://bqs.usgs.gov/srs/*), the National Institutes for Standards and Technology (NIST, *http://www.nist.gov*), and various other reference materials suppliers. Calibration is verified using the independent calibration verification (ICV) standard(s) prepared from a second, independent source of stock solutions. The ICV solution should be prepared at a level near the mid-point of the calibration curve for each element; for example, 25.5 mg/L Ca, Na; 5.5 mg/L K, Mg, Si; and 0.5 mg/L Ag, Al, As, B, Be, Ba, Cd, Co, Cu, Cr, Fe, Li, Mn, Mo, Ni, Pb, S, Sb, Sr, V, Zn, and P. The measured ICV concentrations should be within ± 10 percent of the true or prepared value. If the ICV results are not within acceptable limits, then the source of the error should be identified and corrected and the instrument recalibrated and the ICV solution(s) reanalyzed. Common errors include improperly prepared calibration standards or stock solutions. The results for all quality control sample or CRM sample analyses should be within ± 20 percent of the certified or proposed values, including stated errors. If the results are not within ± 20 percent of the stated values, the samples are prepared again and re-analyzed by ICP-OES. If the results are still not within acceptable values, the instrument conditions and method parameters are checked and the instrument is re-calibrated using freshly prepared calibration standards and the quality control samples are reanalyzed. For every 10 unknown samples, a blank, continuous calibration verification (CCV), and a CRM sample is analyzed. The CCV solution is prepared from the same stock standards used to prepare the calibration standards and has the same concentration as the ICV solution (see above). The measured CCV concentrations should be within ± 10 percent of the true or prepared value. If the CCV values are not within limits, the instrument conditions and method parameters are checked and the instrument is re-calibrated using freshly prepared calibration standards, and any quality control and unknown samples are re-analyzed. If the CRM sample is not within ± 20 percent of the accepted value, the instrument conditions and method parameters are checked and the instrument is re-calibrated using freshly prepared calibration standards, and any quality control and unknown samples are re-analyzed.

Method Performance—Blank Analysis and Reporting Limits

The instrument detection limits and reporting limits are shown in table 5. The preferred wavelengths are selected based on background equivalent concentration (BEC), achieved detection limits, analytical performance of CRMs, and minimal interferences present at the particular wavelength. For most elements, more than one wavelength is analyzed in order to rule out possible interferences in unusual samples. The results for Si and S are converted to SiO_2 and SO_4^{2-} using 2.14 and 2.995 conversion factors, respectively. The dual-view instrument used in this report offers the capability of detecting the elements in the plasma radially and axially. Axial detection provides a longer viewing distance of only the central channel where the analytes are in the plasma, and thus, it is more sensitive and provides improved sensitivity compared to radial or side-on view. The improvement of detection limits in clean samples (for example, drinking water) is on the order of 5–10 times. The main disadvantage of axial view is in cases with increased total dissolved solids (for example, natural waters from mine drainange, bore holes, and leachates). As the matrix is increased, the analytes of interest signal can be decreased or increased resulting in an inaccurate quantification (Alavosus, 1995). For example, Cu is analyzed at 324.754 (radial) and 327.393 (axial). The BEC for the two wavelengths are 0.18 µg/L and 0.32 µg/L, respectively (PerkinElmer, 2010, Winlab 32 Software, version 4.0). Although the 324.754 line provides better BEC, it has interferences from Mg and Th and requires the use of MSF corrections in order to achieve accurate quantification. Additionally, matrix effects from the major cations in waters are significant, and the use of radial detection is advantageous. Thus, the less sensitive Cu line at 327.393 provides better actual detection limits, and because of the lack of interferences, it is the preferred wavelength in this method. Similar reasoning is behind the selection of the preferred analytical wavelengths for all other analytes in this investigation. In table 5, the method detection limit (MDL) is calculated as a value three times the standard deviation (SD) of a large number (n=30) of blank analyses over time. The lower reporting limit in table 5 is established by multiplying the MDL by a reasonable value, generally between 2 and 10, and rounding to a suitable number of significant figures. The instrument MDLs and reporting limits should be re-evaluated on an annual basis or as instrument operating conditions are changed.

Table 5. Blanks analysis.

[SD, standard deviation; MDL, method detection limit; µg/L, micrograms per liter; mg/L, milligrams per liter; preferred lines are in bold]

Element and wavelength	Detection view	Calibration units	Number of samples	Mean	Standard deviation (SD)	MDL = 3 x SD	Lower reporting limit	Upper reporting limit
Ag 328.066	**axial**	**µg/L**	**30**	**0.01**	**0.71**	**2.14**	**5**	**10000**
Ag 338.288	axial	µg/L	30	1.0	0.9	2.8	5	10000
Al 396.151	radial	µg/L	30	–3	5	14	30	100000
Al 308.212	**radial**	**µg/L**	**30**	**–3**	**5**	**15**	**30**	**100000**
As 188.980	radial	µg/L	30	–2	6	17	50	10000
As 193.696	radial	µg/L	30	9	7	21	50	10000
As 193.696	**axial**	**µg/L**	**30**	**2**	**2**	**6**	**50**	**10000**
B 249.772	radial	µg/L	30	–0.7	0.2	0.7	5	10000
B 249.676	**radial**	**µg/L**	**30**	**–0.2**	**0.7**	**2.1**	**5**	**10000**
Ba 455.402	**radial**	**µg/L**	**30**	**0.8**	**0.2**	**0.5**	**1**	**10000**
Ba 233.524	radial	µg/L	30	–0.2	0.2	0.7	1	10000
Be 313.104	**radial**	**µg/L**	**30**	**–0.04**	**0.04**	**0.11**	**10**	**10000**
Be 313.042	radial	µg/L	30	0.02	0.03	0.10	10	10000
Ca 317.932	radial	mg/L	30	–0.291	0.002	0.006	0.1	1000
Ca 422.673	**radial**	**mg/L**	**30**	**–0.210**	**0.005**	**0.014**	**0.1**	**1000**
Cd 226.502	axial	µg/L	30	0.006	0.085	0.254	5	10000
Cd 214.434	**axial**	**µg/L**	**30**	**0.02**	**0.07**	**0.21**	**5**	**10000**
Co 228.615	axial	µg/L	30	0.2	0.1	0.3	10	10000
Co 230.785	**radial**	**µg/L**	**30**	**1.3**	**0.5**	**1.6**	**10**	**10000**
Cr 205.560	axial	µg/L	30	–0.3	0.2	0.6	10	10000
Cr 267.711	**radial**	**µg/L**	**30**	**–0.4**	**0.5**	**1.6**	**10**	**10000**
Cu 324.754	radial	µg/L	30	–1	2	7	10	10000
Cu 327.393	**axial**	**µg/L**	**30**	**4**	**1**	**3**	**10**	**10000**
Fe 238.201	radial	µg/L	30	0.2	0.3	0.9	20	100000
Fe 239.568	**axial**	**µg/L**	**30**	**3.5**	**0.1**	**0.4**	**20**	**100000**
K 766.490	**radial**	**mg/L**	**30**	**–0.02**	**0.05**	**0.14**	**0.5**	**1000**
Li 670.784	**radial**	**µg/L**	**30**	**–0.7**	**1.2**	**3.6**	**5**	**1000**
Mg 285.212	**radial**	**mg/L**	**30**	**–0.008**	**0.002**	**0.006**	**0.1**	**1000**
Mg 279.076	radial	mg/L	30	–0.050	0.004	0.011	0.1	1000
Mn 257.607	**radial**	**µg/L**	**30**	**0.13**	**0.08**	**0.25**	**10**	**10000**
Mn 293.305	radial	µg/L	30	0.6	0.6	1.7	10	10000
Mo 202.032	axial	µg/L	30	0.2	0.4	1.1	20	10000
Mo 203.846	**axial**	**µg/L**	**30**	**0.1**	**0.7**	**2.1**	**20**	**10000**
Na 589.587	**radial**	**mg/L**	**30**	**–0.31**	**0.02**	**0.06**	**0.1**	**1000**
Na 330.237	radial	mg/L	30	–0.4	0.1	0.4	1	1000
Ni 231.604	**radial**	**µg/L**	**30**	**0.5**	**1.2**	**3.7**	**10**	**10000**
Ni 231.604	axial	µg/L	30	–0.1	0.4	1.3	10	10000
P 213.617	**radial**	**mg/L**	**30**	**–0.006**	**0.006**	**0.017**	**0.5**	**10000**
P 178.224	radial	mg/L	30	0.15	0.09	0.26	0.5	10000
Pb 220.354	radial	µg/L	30	–2	5	14	50	10000
Pb 220.354	**axial**	**µg/L**	**30**	**0.4**	**0.7**	**2.1**	**50**	**10000**
S[1] 180.671	axial	mg/L	30	0.44	0.02	0.06	1	1000
S[1] 181.975	**axial**	**mg/L**	**30**	**–0.03**	**0.02**	**0.05**	**1**	**1000**

Table 5. Blanks analysis.—Continued

[SD, standard deviation; MDL, method detection limit; µg/L, micrograms per liter; mg/L, milligrams per liter; preferred lines are in bold]

Element and wavelength	Detection view	Calibration units	Number of samples	Mean	Standard deviation (SD)	MDL = 3 x SD	Lower reporting limit	Upper reporting limit
Sb 206.834	**axial**	**µg/L**	**30**	**14**	**1**	**4**	**50**	**10000**
Sb 217.582	radial	µg/L	30	18	14	41	50	10000
Si 251.609	**radial**	**mg/L**	**30**	**0.010**	**0.003**	**0.009**	**0.1**	**1000**
Si 221.663	radial	mg/L	30	0.023	0.004	0.013	0.1	1000
Sr 421.549	**radial**	**µg/L**	**30**	**–0.35**	**0.06**	**0.18**	**1**	**10000**
Sr 460.733	radial	µg/L	30	7	9	26	50	10000
V 292.402	**axial**	**µg/L**	**30**	**0.8**	**0.4**	**1.3**	**10**	**10000**
V 290.880	radial	µg/L	30	–3.3	0.2	0.7	10	10000
Zn 213.858	**axial**	**µg/L**	**30**	**4.7**	**0.1**	**0.4**	**20**	**10000**
Zn 206.199	axial	µg/L	30	1.75	0.08	0.24	20	10000

[1] S is reported as sulfate (SO_4)

Table 6 summarizes the interferences associated with the wavelength used in the method, the peak processing used for each wavelength, and the preferred wavelength for each element. The spectra obtained at each wavelength were processed either using peak height or MSF alogorithms. The MSF function is used in two different ways: (1) to change and remove the presence of interfences (examples include Be 313.104, Cd 226.502, S 180.6671), and (2) to minimize the background noise and improve detection limits.

Table 6. Interferences and peak processing algorithm.

[MSF, multicomponent spectral fitting]

Element and wavelength	Detection view	Interfering elements	Peak processing	Preferred wavelength
Ag 328.066	axial	Th, Zr, V	peak area	preferred
Ag 338.288	axial	Cr, Th	MSF	
Al 396.151	radial	Mo, Zr	peak area	
Al 308.212	radial	Ce, Th, V	MSF	preferred
As 188.980	radial	Cr, Mo	MSF	
As 193.696	radial	V	peak height	
As 193.696	axial	V	peak height	preferred
B 249.772	radial	Fe, Th	MSF	
B 249.676	radial	Fe, Zr	peak height	preferred
Ba 455.402	radial		peak height	preferred
Ba 233.524	radial	V	peak height	
Be 313.104	radial	Ce, Th	MSF	preferred
Be 313.042	radial	V	MSF	
Ca 317.932	radial	Th	peak height	
Ca 422.673	radial	Sr	peak height	preferred

Table 6. Interferences and peak processing algorithm.—Continued

[MSF, multicomponent spectral fitting]

Element and wavelength	Detection view	Interfering elements	Peak processing	Preferred wavelength
Cd 226.502	axial	Fe	MSF	
Cd 214.434	axial		MSF	preferred
Co 228.615	axial		MSF	
Co 230.785	radial	Mo	peak height	preferred
Cr 205.560	axial		peak area	
Cr 267.711	radial		MSF	preferred
Cu 324.754	radial	Th, Mg	MSF	
Cu 327.393	axial	Th	peak area	preferred
Fe 238.201	radial		peak height	
Fe 239.568	axial	Co	peak height	preferred
K 766.490	radial		peak area	preferred
Li 670.784	radial		peak area	preferred
Mg 285.212	radial		peak height	preferred
Mg 279.076	radial		peak height	
Mn 257.607	radial		peak height	preferred
Mn 293.305	radial	V, Th	MSF	
Mo 202.032	axial		peak height	
Mo 203.846	axial		peak height	preferred
Na 589.587	radial		peak height	preferred
Na 330.237	radial	Zn, Zr	peak height	
Ni 231.604	radial		peak area	preferred
Ni 231.604	axial		peak area	
P 213.617	radial	Cu	MSF	preferred
P 178.224	radial		peak height	
Pb 220.354	radial		peak area	preferred
Pb 220.354	axial		peak area	
S[1] 180.671	axial	Ca	MSF	
S[1] 181.975	axial		peak area	preferred
Sb 206.834	axial	Cr	peak area	preferred
Sb 217.582	radial		peak area	
Si 251.609	radial		peak height	preferred
Si 221.663	radial		peak height	
Sr 421.549	radial		peak height	preferred
Sr 460.733	radial		MSF	
V 292.402	axial		MSF	preferred
V 290.880	radial		MSF	
Zn 213.858	axial	Ca	peak height	
Zn 206.199	axial		peak height	preferred

[1] S is reported as sulfate (SO_4)

Method Performance—Analyses of CRMs and Duplicate Samples

Table 7 summarizes the results obtained for six standard reference materials analyzed by this method: T131, T135, T143, T175, M158, and PPREE. These samples, in addition to ICV samples discussed in the ICP-OES analyses section, are used to check the methods performance and for QC/QA purposes. Unknown samples range from pristinely clean natural waters to mine waste waters and various leachates, and they vary largely in the concentrations of their trace and major elements. The six CRMs shown in this report represent trace and major elements in natural waters collected at various locations in Colorado in which the trace and major constituents are adjusted (USGS Standard Reference Sample Project, *http://bqs.usgs.gov/srs/*). These samples represent trace and major elements in natural waters, and for many elements, the levels are near the detection limits and are useful for evaluating accuracy at low concentrations. The accepted or most probable value (MPV) for the USGS reference waters is determined via multi-laboratory round-robin analysis. The PPREE sample is a rare-earth element CRM developed using mine water collected in western San Juan Mountains, Colo. (Verplank, 2001). This sample is elevated in Al, Ca, Fe, Mg, Mn, Sr, and rare-earth elements. Many of the interferences shown in table 6 are caused by these elements, and the obtained concentrations are within ± 20 percent of the accepted value for this CRM. Table 8 shows the results for 71 duplicate pairs, selected randomly from a variety of geological water samples, to demonstrate the reproducibility of the method.

Table 7. Standard reference material analysis.

[n, number; SD, standard deviation; %, percent; µg/L, micrograms per liter; <, less than; N/A, not applicable; mg/L, milligrams per liter]

Reference material		Description	n	Average determined value	SD	Accepted value	% Recovery
Silver, Ag	µg/L						
T131		trace constituents	20	< 5		1.26	
T135		trace constituents	15	12	1	9.81	124
M158		major constituents	15	< 5		N/A	
T175		trace constituents	15	< 5		1.85	
T143		trace constituents	15	23	1	19.8	118
PPREE		trace constituents	15	< 5		N/A	
Aluminum, Al	µg/L						
T131		trace constituents	20	127	5	132	96
T135		trace constituents	15	< 30		10.5	
M158		major constituents	15	< 30		N/A	
T175		trace constituents	5	45	10	52.0	87
T143		trace constituents	15	< 30	3	22.1	116
PPREE		trace constituents	15	11400	120	11500	99
Arsenic, As	µg/L						
T131		trace constituents	20	58	3	56.6	103
T135		trace constituents	15	< 50		10.0	
M158		major constituents	15	< 50		N/A	
T175		trace constituents	15	< 50		7.38	
T143		trace constituents	15	< 50		15.2	
PPREE		trace constituents	15	< 50		1.8	

Table 7. Standard reference material analysis.—Continued

[n, number; SD, standard deviation; %, percent; µg/L, micrograms per liter; <, less than; N/A, not applicable; mg/L, milligrams per liter]

Reference material		Description	n	Average determined value	SD	Accepted value	% Recovery
Boron, B	µg/L						
T131		trace constituents	20	143	11	141	102
T135		trace constituents	15	10	1	13.1	77
M158		major constituents	15	23	0	23.4	97
T175		trace constituents	15	48	1	48.3	99
T143		trace constituents	15	35	1	35.0	100
PPREE		trace constituents	15	< 5		N/A	
Barium, Ba	µg/L						
T131		trace constituents	20	526	19.8	507	104
T135		trace constituents	15	71	2.0	67.8	105
M158		major constituents	15	25	0.4	N/A	
T175		trace constituents	15	21	0.6	18.0	115
T143		trace constituents	15	91	2.2	81.9	111
PPREE		trace constituents	15	9	0.3	7.1	126
Berilium, Be	µg/L						
T131		trace constituents	20	12	0.4	12.2	101
T135		trace constituents	15	59	0.6	59.0	100
M158		major constituents	15	< 10		N/A	
T175		trace constituents	15	< 10		2.92	
T143		trace constituents	15	< 10		8.50	
PPREE		trace constituents	15	< 10		2.5	
Calcium, Ca	mg/L						
T131		trace constituents	20	30	1	30.6	99
T135		trace constituents	15	11	0	10.4	103
M158		major constituents	15	40	1	38.1	106
T175		trace constituents	15	9	0	8.76	106
T143		trace constituents	15	57	2	53.7	105
PPREE		trace constituents	15	411	8	348	118
Cadmium, Cd	µg/L						
T131		trace constituents	20	6	0.3	6.12	101
T135		trace constituents	15	52	1.0	50.5	103
M158		major constituents	15	< 5		N/A	
T175		trace constituents	15	9	0.3	8.20	111
T143		trace constituents	15	20	0.4	19.1	107
PPREE		trace constituents	15	< 5		0.4	

Table 7. Standard reference material analysis.—Continued

[n, number; SD, standard deviation; %, percent; µg/L, micrograms per liter; <, less than; N/A, not applicable; mg/L, milligrams per liter]

Reference material	Description	n	Average determined value	SD	Accepted value	% Recovery
Cobalt, Co µg/L						
T131	trace constituents	20	25	0.7	24.6	100
T135	trace constituents	15	42	1.3	40.0	104
M158	major constituents	15	< 10		N/A	
T175	trace constituents	15	< 10		7.4	
T143	trace constituents	15	16	0.5	17.0	96
PPREE	trace constituents	15	111	1.6	98	114
Chromium, Cr µg/L						
T131	trace constituents	20	19	0.3	18.6	102
T135	trace constituents	15	81	1.6	79.0	103
M158	major constituents	15	< 10		N/A	
T175	trace constituents	15	< 10		1.9	
T143	trace constituents	15	39	0.5	37.0	105
PPREE	trace constituents	15			N/A	
Copper, Cu µg/L						
T131	trace constituents	20	20	1.2	20.2	98
T135	trace constituents	15	63	1.0	62.0	102
M158	major constituents	15	< 10		N/A	
T175	trace constituents	15	< 10		1.85	
T143	trace constituents	15	21	0.7	22.3	95
PPREE	trace constituents	15	< 10		0.40	
Iron, Fe µg/L						
T131	trace constituents	20	85	3	91	94
T135	trace constituents	15	212	4	228	93
M158	major constituents	15	< 20		N/A	
T175	trace constituents	15	34	1	40	87
T143	trace constituents	15	210	3	222	95
PPREE	trace constituents	15	60600	666	68300	89
Potassium, K mg/L						
T131	trace constituents	20	2.21	0.09	2.39	92
T135	trace constituents	15	0.85	0.08	0.96	88
M158	major constituents	15	1.78	0.04	1.71	104
T175	trace constituents	15	3.62	0.08	3.83	95
T143	trace constituents	15	2.50	0.07	2.50	100
PPREE	trace constituents	15	0.96	0.09	0.94	102

Table 7. Standard reference material analysis.—Continued

[n, number; SD, standard deviation; %, percent; µg/L, micrograms per liter; <, less than; N/A, not applicable; mg/L, milligrams per liter]

Reference material		Description	n	Average determined value	SD	Accepted value	% Recovery
Lithium, Li	µg/L						
T131		trace constituents	20	18	1.5	17.00	103
T135		trace constituents	15	75	1.9	73.7	102
M158		major constituents	15	< 5		N/A	
T175		trace constituents	15	6	0.5	3.20	181
T143		trace constituents	15	19	1.1	18.00	103
PPREE		trace constituents	15	8	1.7	6.0	130
Magnesium, Mg	mg/L						
T131		trace constituents	20	7.27	0.16	8.00	91
T135		trace constituents	15	1.83	0.05	2.00	92
M158		major constituents	15	10.9	0.09	11.8	93
T175		trace constituents	15	1.91	0.06	2.03	94
T143		trace constituents	15	9.73	0.11	10.4	94
PPREE		trace constituents	15	34.0	0.40	34.6	98
Manganese, Mn	µg/L						
T131		trace constituents	20	39	1.4	37.8	103
T135		trace constituents	15	452	5.8	423	107
M158		major constituents	15	< 10		N/A	
T175		trace constituents	15	55	1.4	49.4	112
T143		trace constituents	15	20	0.5	18.2	109
PPREE		trace constituents	15	5870	36.2	5300	111
Molybdenum,	µg/L						
T131		trace constituents	20	110	3	112	98
T135		trace constituents	15	56	2	63	89
M158		major constituents	15	< 20		N/A	
T175		trace constituents	15	< 20		2	
T143		trace constituents	15	31	0.9	36.1	85
PPREE		trace constituents	15	< 20		N/A	
Sodium, Na	mg/L						
T131		trace constituents	20	21	0.9	21.4	98
T135		trace constituents	15	31	1.3	30.8	100
M158		major constituents	15	73	2.0	71.7	102
T175		trace constituents	15	9	0.2	8.6	100
T143		trace constituents	15	34	1.2	34.0	101
PPREE		trace constituents	15	9	0.1	8.1	107

Table 7. Standard reference material analysis.—Continued

[n, number; SD, standard deviation; %, percent; µg/L, micrograms per liter; <, less than; N/A, not applicable; mg/L, milligrams per liter]

Reference material		Description	n	Average determined value	SD	Accepted value	% Recovery
Nickel, Ni	µg/L						
T131		trace constituents	20	64	2.6	56.3	113
T135		trace constituents	15	73	2.1	65.6	112
M158		major constituents	15	< 10		N/A	
T175		trace constituents	15	< 10		3.2	
T143		trace constituents	15	81	2.0	71.0	114
Phosphorus, P	mg/L						
T131		trace constituents	20	< 0.5		N/A	
T135		trace constituents	15	< 0.5		N/A	
M158		major constituents	15	< 0.5		0.06	
T175		trace constituents	15	< 0.5		N/A	
T143		trace constituents	15	< 0.5		N/A	
PPREE		trace constituents	15	< 0.5		0.18	
Lead, Pb	µg/L						
T131		trace constituents	20	< 50		18.1	
T135		trace constituents	15	105	2.3	103	102
M158		major constituents	15	< 50		N/A	
T175		trace constituents	15	< 50		3.0	
T143		trace constituents	15	92	1.5	83.4	110
PPREE		trace constituents	15	< 50		0.1	
Antimony, Sb	µg/L						
T131		trace constituents	20	52	6	56	93
T135		trace constituents	15	58	2	76	76
M158		major constituents	15	< 50		N/A	
T175		trace constituents	15	< 50		2	
T143		trace constituents	15	< 50		16.6	
PPREE		trace constituents	15	< 50		N/A	
Silica, SiO_2	mg/L						
T131		trace constituents	20	5.8	0.2	5.81	100
T135		trace constituents	15	4.1	0.1	4.28	96
M158		major constituents	15	14.6	0.1	15.0	98
T175		trace constituents	15	5.3	0.1	5.60	95
T143		trace constituents	15	22.5	0.4	23.4	96
PPREE		trace constituents	15	29.4	0.3	27.8	106
Sulfate, SO_4	mg/L						
T131		trace constituents	20	< 1		N/A	
T135		trace constituents	15	6	0.1	N/A	
M158		major constituents	15	115	1.3	105	110
T175		trace constituents	15	14	0.3	N/A	
T143		trace constituents	15	23	0.4	N/A	
PPREE		trace constituents	15	1410	89	1210	116

Table 7. Standard reference material analysis.—Continued

[n, number; SD, standard deviation; %, percent; µg/L, micrograms per liter; <, less than; N/A, not applicable; mg/L, milligrams per liter]

Reference material		Description	n	Average determined value	SD	Accepted value	% Recovery
Strontium, Sr	µg/L						
T131		trace constituents	20	315	15	295	107
T135		trace constituents	15	52	1	46.0	112
M158		major constituents	15	69	2	63.6	109
T175		trace constituents	15	69	2	63.6	109
T143		trace constituents	15	344	12	306	113
PPREE		trace constituents	15	3100	38	2680	116
Vanadium, V	µg/L						
T131		trace constituents	20	33	2	34	97
T135		trace constituents	15	52	1	53	98
M158		major constituents	15	11.1	0.5	11.3	99
T175		trace constituents	15	< 10		N/A	
T143		trace constituents	15	29.1	0.9	30.0	97
PPREE		trace constituents	15	< 10		3	
Zinc, Zn	µg/L						
T131		trace constituents	20	68	2.5	72	94
T135		trace constituents	15	45	0.9	48	93
M158		major constituents	15	< 20		N/A	
T175		trace constituents	15	68	1.4	72	94
T143		trace constituents	15	< 20		20	
PPREE		trace constituents	15	562	33	490	115

Table 8. Duplicate analysis, based on the analyses of 71 duplicate pairs.

[%, percent; RSD, relative standard deviation; µg/L, micrograms per liter; mg/L, milligrams per liter; MDL, method detection limit]

Element	Concentration units	Mean	Standard deviation	% RSD	Minimum	Maximum	Maximum % difference between duplicates	Number of analysis higher than MDL
Ag	µg/L	9	2	24	5	18	18%	22
Al	µg/L	12900	1900	15	20	174000	19%	118
As	µg/L	1210	15	1	50	17300	14%	58
B	µg/L	315	13	4	5	5290	17%	78
Ba	µg/L	137	75	55	4	776	18%	132
Be	µg/L	< 10					0%	0
Ca	mg/L	38.8	5	12	0.5	469	13%	134
Cd	µg/L	10	1	14	5	18	13%	38
Co	µg/L	55	2	3	11	224	13%	58
Cr	µg/L	45	1	2	10	111	6%	34
Cu	µg/L	488	10	2	11	8420	17%	64
Fe	µg/L	43700	700	2	21	626000	12%	114
K	mg/L	6.8	2.5	37	0.1	39.2	13%	130
Li	µg/L	36	7	21	5	213	16%	104
Mg	mg/L	14.7	0.4	2	0.1	130	8%	130
Mn	µg/L	1270	180	14	11	7630	14%	106
Mo	µg/L	129	4	3	22	878	13%	26
Na	mg/L	115	8.0	7	0.2	1950	16%	136
Ni	µg/L	184	4	2	11	3250	11%	70
P	mg/L	1.6	0.0	2	0.6	6.2	10%	48
Pb	µg/L	96	7	7	81	110	14%	10
Sb	µg/L	120	1	1	76	220	3%	8
SiO_2	mg/L	17.0	1.8	10	0.3	83.0	12%	100
SO_4	mg/L	177	3	2	2	2240	13%	114
Sr	µg/L	257	27	11	1	2720	14%	138
V	µg/L	106	9	8	13	311	9%	50
Zn	µg/L	226	5	2	25	1220	13%	72

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Appendix 1. Generation of Multicomponent Spectral Fitting (MSF) and Interelement Correction Factors (IEC) Correction Equations

Required solutions:

- Calibration blank (2 percent HNO_3 blank)
- Low-level standard (lowest standard in method calibration, contains all elements of interest)
- 500 mg/L pure Ca Standard (Inductively coupled plasma [ICP] grade standard)
- 500 mg/L pure Mg Standard (ICP grade standard)
- 500 mg/L pure Al Standard (ICP grade standard)
- 200 mg/L pure Fe Standard (ICP grade standard)
- Mixed Interferent Check Solution (ICS); mix Ca, Mg, Al, Fe at above levels
- 1 mg/L single element solutions of Ag, Al, As, B, Ba, Be, Bi, Ca, Cd, Co, Cr, Cu, Fe, K, Li, Mg, Mn, Mo, Na, Ni, P, Pb, S, Sb, Se, Si, Sr, Th, Ti, Tl, U, V, W, Zn, Zr

Open the auto-analysis window; save the data to a file titled: your method name_MSF. This is important if you want to edit your MSF model in the future. To recall MSF models to edit them, all the samples used to create the model must be in the open results file.

Load auto-sampler and Create Sample information file:

Autosampler position	Sample name
21	Calibration blank
22	Standard 1 (Name specified in method)
23	Ca 500 mg/L
24	Mg 500 mg/L
25	Al 500 mg/L
26	Fe 200 mg/L
27	ICS (mixed interferent solution)
28	Ag 1 mg/L
29	Al 1 mg/L
30	B 1 mg/L
31–end	Rest of single element 1 mg/L solutions each in separate vials

Optimize instrument and perform a Hg realign before proceeding with analysis. Performing a Hg realignment now will ensure that any subsequent drift can be corrected for, by simply doing a daily Hg realignment, and the MSF models will continue to be valid. Shifting peaks will invalidate MSF models.

Analyze each of the solutions using the analytical method you are determining MSF models for (for example, water02TT 20120711).

Once the analysis has completed, shut off the instrument. Use the “Examine Spectra” tool in the WinLab version 4.0 software and reprocess all of the data to validate corrections. The following are the general steps used to finalize the method and add interferent corrections.

Step 1:

Use examine spectra window to align wavelengths, and set background points. While doing this, make note of the elements and wavelengths that show problems to be corrected by MSF. Note: the window will show spectra for all solutions overlayed so you can see where potential interferences are.

Step 2:

Switch to the MSF model builder in the examine spectra tool and begin building MFS models for elements that showed spectral interferences. MSF does not correct for direct spectral overlap, so if direct overlaps are observed, alternative wavelengths should be used.

Once the MSF models have been created, use the reprocessing mode of the software to reprocess the data collected and review changes to the data. Use the ICS or mixed matrix sample run to evaluate the effect of the corrections. Results for analyte elements should be very close to zero for all elements, except for the matrix elements of Ca, Mg, Al, and Fe, which are contained in this solution.

See PerkinElmer WinLab version 4.0 Software Manual, Section 9, p. 388–402, for more detailed information.

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