ELEMENTS by ICP (Microwave Digestion) 7304

MW: Table 1	CAS: Table 2	RTECS: Table 2
METHOD: 7304, Issue 1	**EVALUATION: FULL**	**Issue 1: 25 June 2014**

OSHA: Table 2
NIOSH: Table 2
Other OELs: [1,2]*

PROPERTIES: Table 1

ELEMENTS:

aluminum	cadmium	iron	molybdenum	selenium	titanium
arsenic	calcium	lead	nickel	sodium	vanadium
barium	chromium	lithium	phosphorus	strontium	yttrium
beryllium	cobalt	magnesium	platinum	tellurium	zinc
boron	copper	manganese	potassium	thallium	zirconium

SAMPLING

SAMPLER: FILTER, (polyvinyl chloride (PVC), 37-mm diameter, 5.0 µm pore size)

FLOW RATE: 1 to 4 L/min

VOL-MIN:
-MAX: Table 1

SHIPMENT: Routine

SAMPLE STABILITY: Stable

BLANKS: 2 to 10 field blanks per set

ACCURACY

RANGE STUDIED: See Table 4

ACCURACY: See Table 4

BIAS: See Table 4

OVERALL PRECISION ($\hat{S}_{rT}$): See Table 4

MEASUREMENT

TECHNIQUE: INDUCTIVELY COUPLED ARGON PLASMA, ATOMIC EMISSION SPECTROSCOPY (ICP-AES)

ANALYTE: Elements listed above

REAGENTS: 12 mL of 5:1 concentrated nitric acid and ASTM Type ll water

FINAL SOLUTION: 20% HNO_3, 50 mL

WAVELENGTH: Depends upon element; Table 3

BACKGROUND CORRECTION: Spectral wavelength shift

CALIBRATION: Elements in 20% HNO_3

RANGE: See Table 4

ESTIMATED LOD: Table 3

PRECISION ($\bar{S}_r$): Table 3

APPLICABILITY: The working range of this method varies from element to element. This method is for the analysis of metal and nonmetal dust collected on PVC filters that are also used for gravimetric analysis. This is a simultaneous elemental analysis using a microwave digestion approach to simplify and expedite the analysis. Some elements such as antimony, silver, and tin do not form stable solutions in nitric acid when chloride from the PVC filters is present. In such cases a mixed cellulose ester (MCE) filter is necessary (See NMAM 7302). A different acid medium also helps but this technique is not described in this method.

INTERFERENCES: Spectral interferences are the primary interferences encountered in ICP-AES analysis. These are minimized by judicious wavelength selection, inter-element correction factors and background correction. [3,4,5,6]

OTHER METHODS: This method complements NIOSH hotplate digestion methods 7300 and 7301 for trace elements. Flame atomic absorption spectroscopy (e.g., Methods 7013 through 7082) is an alternative analytical technique for many of these elements. [7] Graphite furnace AAS (e.g., 7102 for Be, 7105 for Pb) is usually more sensitive. [7] NMAM 7301 and 7303 contain alternative extraction procedures.

REAGENTS

1. Nitric acid, conc., trace metal grade*
2. Calibration stock solutions, 1000 ug/mL and 10,000 ug/mL commercially available, or prepared per instrument manufacturer recommendation (see step 10)
3. Argon, liquid
4. De-ionized Water, ASTM Type II [8]
5. Dilution acid: 20% nitric acid in ASTM Type II water*

* See SPECIAL PRECAUTIONS

EQUIPMENT

1. Sampler: Polyvinyl chloride filter, 5.0-µm pore size, 37-mm diameter; in 2-piece cassette filter holder
2. Personal sampling pump, 1 to 4 L/min, with flexible connecting tubing
3. Inductively coupled plasma-atomic emission spectrometer, equipped as specified by the manufacturer for analysis of elements of interest
4. Regulator, two-stage for argon
5. Microwave, programmable power, active temperature control, minimum of 574 W, corrosion resistant ventilated oven and turntable
6. Microwave digestion vessels, high pressure, closed PTFE, 100-mL capacity
7. Volumetric flasks, 50 mL**
8. Assorted volumetric pipettes as needed**

** Acid wash all glassware and vessels before using.

SPECIAL PRECAUTIONS: Wear gloves, lab coat, and safety glasses while handling all chemicals. All work should be performed with adequate ventilation to personnel and equipment. Because this method involves the use of capped digestion containers, avoid the use of other acids such as perchloric acid in combination with nitric acid that could cause a violent reaction [1,9]. In the preparation of the digestion and dilution acid, it is imperative that acid be added to water in order to avoid a violent exothermic reaction.

SAMPLING:

1. Calibrate each personal sampling pump with a representative sampler connected to the pump (in line.)
2. Sample at an accurately known flow rate between 1 and 4 L/min. For estimated sampling volumes see Table 1. For TWA measurements see Table 2. Do not exceed a filter loading of approximately 2 mg total dust.
   NOTE: Filter overloading can be assessed by periodic visual checks. See NMAM Chapter O, "Factors Affecting Aerosol Sampling," for additional discussion on filter capacity. [http://www.cdc.gov/niosh/docs/2003-154/pdfs/chapter-o.pdf.]

SAMPLE PREPARATION:

NOTE: If total weights are desired, it should be done at this step. Follow NIOSH method 0500 for gravimetric analysis [12].

3. Open the cassette filter holders and transfer the samples, blanks, and Quality Control (QC) filters to clean PTFE digestion digestion vessels. Wipe the internal cassette surfaces with a 37 mm PVC filter wetted with deionized water and add to the digestion vessel to transfer non-filter aerosol deposits into the digestion vessels.
4. Add 2 mL of ASTM Type II water followed by adding (slowly) 10 mL concentrated nitric acid, then cap each vessel.

NOTE: In order to avoid a violent exothermic reaction, do not add water to concentrated nitric acid. Acid should be added after the water has been placed in the vessel.

5. Place digestion vessels in microwave and run preprogrammed PVC digestion procedure. Example microwave conditions for 12-vessel digestion: 1200 W power, ramp to 215 °C over 20 min, hold for 10 min at 215 °C followed by at least a 5 min cool down (power will be adjusted lower for fewer vessels).
6. Allow the samples to cool to room temperature.
7. Remove vessel lids and rinse contents into 50-mL volumetric flasks with ASTM Type II water.
8. Dilute to the mark with ASTM Type II water and mix.
9. Submit samples for analysis.

NOTE: A residual solid may be present after digestion. Filter/centrifuge the samples before analysis, as appropriate.

CALIBRATION AND QUALITY CONTROL:

10. Calibrate the spectrometer according to the manufacturers' recommendations.

NOTE: Typically an acid blank and a single or multi-element working standard are used. The following multi-element combinations are chemically compatible in 20% HNO3.

a. Al, As, Ba, Be, Ca, Co, Cr, Cu, Fe, Li, Mg, Mn, Mo, Na, Ni, Pb, Se, Sr, Ti, V, Y, Zn, Zr;
b. B, K, P, Te, Tl;
c. Cd;
d. Pt.

11. Analyze all applicable standards at least once every twenty (20) analyses (minimum frequency 5%).
12. Check recoveries with at least one media blank and two spiked media blanks per twenty samples. Use a spike level that is within the range of 10 to 20 times the Limit of Quantitation (LOQ.)

NOTE: Whenever possible, QA/QC samples should be prepared from certified reference materials in a matrix similar to the bulk material sampled. Liquid spiked filters are only surrogates for real world samples and QC data based upon certified samples would be ideal.

MEASUREMENT:

13. Set the ICP-AES spectrometer to conditions specified by manufacturer.
14. Analyze standards and samples at applicable wavelengths for each element (target analytes are in Table 3).

NOTE: If the values for the samples are above the linear range of the instrument, dilute the solutions with dilution acid, reanalyze, and apply the appropriate dilution factor in the calculations.

CALCULATIONS:

15. Obtain the solution concentrations for the sample, C_s (µg/mL), and the average media blank, C_b (µg/mL), from the instrument.
16. Using the solution volume of sample, V_s (mL), and media blank, V_b (mL), calculate the concentration for the sample, C (mg/m³), of each element in the air volume sampled, V (L), as follows:

$$C = \frac{(C_s V_s) - (C_b V_b)}{V}, \text{ mg/m}^3$$

NOTE: µg/Liter air is equivalent to mg/m^3.

EVALUATION OF METHOD:

This method is less time consuming and more convenient than using the acid hotplate approach. The elimination of perchloric acid in the sample digestion procedure helps to improve the safety of the method. [9] Use of the PVC filters allows for the acquisition of total mass per filter in addition to total metals concentration.

The evaluation of this method, 7304, for PVC filters was determined at six concentration levels based on the LOQ for each element listed on page 1 [13]. All of the precision data was evaluated for homogeneity for all concentration levels tested using the Bartlett's test and the results are listed in the method backup data report [12] and summarized in Tables 3 and 4. In many cases the highest concentration level (300 times the LOQ) was not poolable due in every case to the precision being so small relative to the other values, usually less than CV = 0.001 (<0.1%). Therefore, the overall precision ($\hat{S}_{rT}$) and accuracy as given in Table 4 is an upper limit predictor of precision; precision at concentration levels greater than 300 times the LOQ (see Table 3) will probably be much smaller.

For many of the metals, precision at the 3 times and/or 1 times the LOQ levels was reasonable (CV less than 10%) but were not poolable due to the precisions at the higher concentration levels being so much smaller. In one case (strontium) the lowest level was not poolable because its CV was an inlier (less than 1%), being much smaller than those at the higher concentration levels. In most cases the precision appeared to be a function of concentration. This is observable in Table 3 where the CVs for the 10 times the LOQ (lower level) and 300 times the LOQ (higher) levels are compared.

Three elements, antimony, silver, and tin, had poor recoveries. It is believed that the chloride ions produced in the digestion of the PVC filters is causing the formation of precipitates. These metals are preferably sampled on MCE filters. The values in Tables 3 and 4 were determined using several different ICP-AES instruments and also several different microwave ovens. All were operated according to the manufacturer's instructions.

REFERENCES:

[1] ACGIH [2013]. TLVs and BEIs based on the documentation of the Threshold Limit Values for chemical substances and physical agents and Biological Exposure Indices. Cincinnati, Ohio: American Conference of Governmental Industrial Hygienists. [www.acgih.org]. [Data accessed April 2014.]

[2] Institut fur Arbeitsschutz der Deutschen Gesetzlichen Unfallversicherung [2013]. GESTIS database on hazardous substances (German Social Accident Insurance). Sankt Augustin, FRG: [http://www.dguv.de/ifa/Gefahrstoffdatenbanken/GESTIS-Stoffdatenbank/index-2.jsp]. [Data accessed April 2014.]

[3] Hull, RD [1981]. Multi-element analysis of industrial hygiene samples, NIOSH internal report presented at the American Industrial Hygiene Conference, Portland, Oregon, May.

[4] NIOSH [1994]. Elements: Method 7300 (Supplement Issued 3/15/2003). In: Schlecht PC, O'Connor PF, eds. NIOSH Manual of analytical methods, 4th ed. Cincinnati, OH: U.S. Department of Health and Human Services, Centers for Disease Control and Prevention, National Institute for Occupational Safety and Health, DHHS Publication No. 94-113 [www.cdc.gov/niosh/nmam/].

[5] Harrison, GR [1969]. Massachusetts Institute of Technology wavelength tables, 2nd ed. West Hanover, MA: Halliday Lithograph, Volume 1.

[6] ASTM International [2010]. ASTM D7035, Standard test method for the determination of metals and metalloids in airborne particulate matter by inductively coupled plasma atomic emission spectrometry. West Conshohocken, PA: ASTM International [www.astm.org].

[7] NIOSH [1994]. NIOSH manual of analytical methods (NMAM). 4th ed. Schlecht PC, O'Connor PF, eds. Cincinnati, OH: U.S. Department of Health and Human Services, Centers for Disease Control and Prevention, National Institute for Occupational Safety and Health, DHHS (NIOSH) Publication No. 94-113 [www.cdc.gov/niosh/nmam/].

[8] ASTM [1983]. ASTM D1193-77, Standard specification for reagent water. West Conshohocken, PA: ASTM International [www.astm.org].
[9] Furr KA [1995]. CRC Handbook of Laboratory Safety, 4th ed. Boca Raton, FL: CRC Press.
[10] NIOSH [1973]. The industrial environment - its evaluation & control. 3rd ed. U.S. Department of Health Education and Welfare, Center for Disease Control, National Institute for Occupational Safety and Health. DHEW (NIOSH) Publication No. 74-117.
[11] NIOSH [1994]. Particulates not otherwise regulated: Method 0500. In: Eller PM, Cassinelli ME, eds. NIOSH Manual of Analytical Methods, 4th ed. Cincinnati, OH: U.S. Department of Health and Human Services, Centers for Disease Control and Prevention, National Institute for Occupational Safety and Health. DHHS (NIOSH) Publication No. 94-113.
[12] Stone LC, Perkins JB, Rogers DM [2003]. Backup data report NMAM 7304: Elements by ICP on PVC filters using microwave digestion. Cincinnati OH: DataChem Laboratories, Inc. under NIOSH contract CDC-200-2001-08000. Unpublished.
[13] Lide DR [1993]. Handbook of chemistry and physics. 74th ed. Boca Raton FL: CRC Press.
[14] NIOSH [2005]. NIOSH pocket guide to chemical hazards. Barsan ME, ed. Cincinnati, OH: US Department of Health and Human Services, Centers for Disease Control and Prevention, National Institute for Occupational Safety and Health, DHHS (NIOSH) Publication No. 2005-149. [www.cdc.gov/niosh/npg].
[15] Code of Federal Regulations, 29 CFR Part 1910.1000, Table Z-1[2013]. Limits for air standards. National occupational safety and health standards. [https://www.osha.gov/pls/oshaweb/owadisp.show_document?p_table=STANDARDS&p_id=9992] Website accessed on December 13, 2013.

METHOD WRITTEN BY:

David M. Rogers, Lee C. Stone, John M. Reynolds, James B. Perkins, DataChem Laboratories, Salt Lake City, Utah; Yvonne Gagnon, Ronnee Andrews, Ph.D., DART/CEMB.

* Other OELs: Because exposure limits and guidelines may change over time, NIOSH recommends referring to the following sources for updated limits and guidelines on the use of this compound.

Table 1. PROPERTIES AND SAMPLING VOLUMES

Element (Symbol)	Properties [13]		Air Volume, L @ OSHA PEL [4]	
	Atomic Weight	MP, °C	MIN	MAX
Aluminum (Al)	26.98	660	5	100
Arsenic (As)	74.92	817	5	2000
Barium (Ba)	137.3	727	5(2)	200(2)
Boron (B)(1)	10.81	2300	5	2000
Beryllium(Be)	9.01	1278	1250	2000
Calcium (Ca)(1)	40.08	842.5	5	200
Cadmium (Cd)(3)	112.40	321	13	2000
Cobalt (Co)	58.93	1495	25	2000
Chromium (Cr)	52.00	1890	5	1000
Copper (Cu)	63.54	1083	5	1000
Iron (Fe)	55.85	1535	5	100
Potassium (K)(1)	39.10	63	5	2000
Lithium (Li)(1)	6.94	179	100	2000
Magnesium (Mg)	24.31	651	5	67
Manganese (Mn)	54.94	1244	5	200
Molybdenum (Mo)	95.94	651	5	67
Sodium (Na)(1)(3)	22.99	98	13	2000
Nickel (Ni)	58.71	1453	5	1000
Phosphorus (P)	30.97	44	25	2000
Lead (Pb)	207.19	328	50	2000
Platinum (Pt)(3)	195.09	1769	1250	2000
Selenium (Se)	78.96	217	13	2000
Strontium (Sr)(1)	87.62	769	5	2000
Tellurium (Te)	127.60	450	25	2000
Titanium (Ti)	47.90	1675	5	100
Thallium (Tl)	204.37	304	25	2000
Vanadium (V)	50.94	1890	5	2000
Yttrium (Y)	88.91	1495	5	1000
Zinc (Zn)	65.37	419	5	200
Zirconium (Zr)	91.22	1852	5	200

(1) No PEL, REL, or STEL data found [1,14].
(2) Air Volumes Estimated from TWA and LOQ's (see Tables 2, 3). [10]
(3) These metals, as well as tin and antimony, forms precipitates in nitric acid when chloride from the PVC filters is present.

Table 2. EXPOSURE LIMITS, CAS #, RTECS [1,14,15]

Element (Symbol)	CAS #	RTECS #	Exposure Limits, mg/m^3 (C = ceiling limit) OSHA	NIOSH
Aluminum (Al)	7429-90-5	BD0330000	15 (total dust) 5 (respirable)	10 (total dust) 5 (respirable, fume)
Arsenic (As)	7440-38-2	CG0525000	0.010 (inorganic)	C 0.002(1)
Barium (Ba)	7440-39-3	CQ8370000	0.5 (soluble)	0.5 (soluble)
Beryllium (Be)	7440-41-7	DS1750000	0.002, C 0.005	C 0.0005(1)
Cadmium (Cd)	7440-43-9	EU9800000	0.005	lowest feasible conc.(1)
Calcium (Ca)	7440-70-2		No OEL	No OEL
Cobalt (Co)	7440-48-4	GF8750000	0.1	0.05 (dust, fume)
Chromium (II) (Cr)	22541-79-3	GB6260000	0.5	0.5
Chromium (III) (Cr)	16065-83-1	GB6261000	0.5	0.5
Chromium (VI) (Cr)	18540-29-9	GB6262000	0.005	0.0002
Cobalt (Co)	7440-48-4	GF8750000	0.1	0.05 (dust, fume)
Copper (Cu)	7440-50-8	GL5325000	1 (dust, mists) 0.1 (fume)	1 (dust, mists) 0.1 (fume)
Iron (Fe)	1309-37-1	NO7400000	10 (fume) as oxide	5 (dust, fume) as oxide
Magnesium (Mg)	1309-48-4	OM3850000	15 (dust) as oxide	--
Manganese (Mn)	7439-96-5	OO9275000	C 5	1; STEL 3
Molybdenum (Mo)	7439-98-7	QA4680000	5 (soluble) 15 (total insoluble)	--
Nickel (Ni)	7440-02-0	QR5950000	1	0.015(1)
Phosphorus (P)	7723-14-0	TH3500000	0.1	0.1
Lead (Pb)	7439-92-1	OF7525000	0.05	0.05
Platinum (Pt)	7440-06-4	TP2160000	0.002 (soluble)	1 (metal)
Selenium (Se)	7782-49-2	VS7700000	0.2	0.2
Silver (Ag)	7440-22-4	VW3500000	0.01 (soluble, metal)	0.01 (soluble, metal)
Tellurium (Te)	13494-80-9	WY2625000	0.1	0.1
Titanium (Ti)	7440-32-6	XR1700000	15 (as TiO_2)	lowest feasible(1)
Thallium (Tl)	7440-28-0	XG3425000	0.1 (soluble)	0.1(soluble)
Vanadium (V)	7440-62-2	YW240000	C 0.5 (respirable) as V_2O_5 C 0.1 (fume) as V_2O_5	C 0.05
Yttrium (Y)	7440-65-5	ZG2980000	1	1
Zinc (Zn)	1314-13-2	ZH4810000	5 (ZnO fume) 15 (ZnO dust) 5 (ZnO respirable)	5; STEL 10 (ZnO fume) 5; C 15 (ZnO dust)
Zirconium (Zr)	7440-67-7	ZH7070000	5	5, STEL 10

(1) Carcinogen

Table 3. MEASUREMENT WAVELENGTHS AND RECOVERY DATA

			Lower Level[4,5]				Higher Level[5]			
Element[1]	Wavelength (nm)	LOD (µg/ sample)	µg/ sample	N =	Percent Recovery	Precision (S_r)	µg/ sample	N =	Percent Recovery	Precision (S_r)
Ag	328.07	0.1	3.00	5	63.01	0.0739	300	6	3.92	0.0865
Al	308.22	2	50.25[4]	6	89.78	0.0565	1500	6	100.71	0.0055
Al[2]	308.214	0.5	15.0	5	115.05	0.0199	1500	6	105.17	0.0056
As	193.76	2	15.0	5	93.29	0.0570	1500	6	115.84	0.0174
Ba	493.41	0.2	1.50	5	107.16	0.0295	150	6	102.22	0.0104
B	249.68	0.4	7.50	5	86.38	0.0277	750	6	101.19	0.0082
Be	313.04	0.008	0.152	6	102.38	0.0861	15.2	6	107.71	0.0091
Ca	315.89		151[4]	6	94.64	0.0512	4500	6	116.25	0.0153
Ca[2]	315.88	2	45.0	5	104.82	0.0090	4500	6	98.13	0.0066
Cd	228.80	0.2	3.00	5	109.65	0.0316	300	6	111.68	0.0152
Co	228.62	0.7	7.50	5	89.87	0.0338	750	6	114.15	0.0141
Cr	267.72	0.7	7.50	5	112.65	0.0233	750	6	118.65	0.0136
Cr[2]	267.71	0.3	7.50	5	102.60	0.0048	750	6	92.98	0.0066
Cu	324.75	0.08	1.50	5	106.84	0.0364	150	6	100.42	0.0058
Cu[2]	324.75	0.08	1.50	5	117.16	0.0361	150	6	103.13	0.0150
Fe	259.94	15	30	5	120.58	0.0405	3000	6	112.41	0.0083
Fe[2]	259.94	5	30	5	112.55	0.0489	3000	6	97.20	0.0085
K	766.49	3	100[4]	6	85.57	0.0254	3000	6	86.46	0.0260
K[2]	766.49		100[4]	6	99.40	0.0300	3000	6	90.02	0.0205
Li	670.78	0.06	1.50	5	97.51	0.0253	150	6	81.96	0.0378
Mg	279.08	0.9	15.0	5	105.25	0.0088	1500	6	97.47	0.0077
Mg[2]	279.07	0.4	15.0	5	107.33	0.0043	1500	6	101.75	0.0058
Mn	257.61	0.09	1.50	5	110.24	0.0150	150	6	115.56	0.0090
Mo	202.03	0.4	4.50	5	87.79	0.0433	450	6	120.57	0.0093
Mo[2]	202.029	0.3	4.50	5	89.75	0.0215	450	6	100.44	0.0154
Na	589.00	5	75.0	6	124.56	0.0859	7500	6	83.07	0.0248
Ni	231.60	0.3	4.50	5	102.93	0.0475	450	6	110.59	0.0080
Ni[2]	231.60	0.2	4.50	5	109.91	0.0047	450	6	101.77	0.0139
P	214.92	2	30.0	5	81.82	0.0511	3000	6	107.20	0.0103
P[2]	214.91	2	30.0	5	86.36	0.0077	3000	6	103.33	0.0174
Pb	220.35	1	15.0	5	95.85	0.0308	1500	6	100.54	0.0154
Pt	203.65	9	150	5	104.67	0.0182	15000	6	105.19	0.0088
Sb[3]	206.84	0.7	15.0	6	25.29	0.5861	1500	6	111.95	0.0086

(1) Values reported were obtained with a Fisons ARL Accuris ICP-AES unless otherwise noted; performance may vary with instrument and should be independently verified.
(2) Values reported were obtained with a Perkin Elmer Optima 3000 DV ICP-AES.
(3) Elements that were evaluated and found not suitable for analysis by this method.
(4) Values given (lower level) are for the 10xLOQ level due to low recoveries at the 3xLOQ level.
(5) LOQ = Estimated limit of quantitation

Table 3. MEASUREMENT WAVELENGTHS AND RECOVERY DATA

			Lower Level[4,5]				Higher Level[5]			
Element[1]	Wavelength (nm)	LOD (µg/ sample)	µg/ sample	N =	Percent Recovery	Precision (S_r)	µg/ sample	N =	Percent Recovery	Precision (S_r)
Se	196.09	5	75.0	5	102.05	0.0531	7500	6	111.35	0.0063
Se[2]	196.02	2	75.0	5	99.93	0.0051	7500	6	99.72	0.0082
Sn	189.9		75.0	5	30.82	0.0502	7500	6	79.56	0.0124
Sn[2,3]	189.9	0.4	75.0	5	37.87	0.0816	7500	6	92.34	0.0129
Sr	421.55	0.04	7.50	5	100.00	0.0049	750	6	99.54	0.0055
Te	214.27	4	30.0	5	95.80	0.0624	3000	6	110.81	0.0094
Te[2]	214.28	2	30.0	5	97.18	0.0100	3000	6	99.64	0.0074
Ti	337.28	0.2	3.00	5	81.66	0.0392	300	6	103.42	0.0101
Ti[3]	334.94	0.1	3.00	5	82.68	0.0374	300	6	96.13	0.0121
Tl	190.86	2	15.0	5	96.38	0.0605	1500	6	97.25	0.0148
Tl[3]	190.79	1	15.0	5	97.75	0.0032	1500	6	92.04	0.0119
V	292.40	0.1	1.50	5	104.54	0.0528	150	6	111.15	0.0160
V[2]	292.40	0.09	1.50	5	100.99	0.0146	150	6	99.38	0.0232
Y	371.03	0.07	0.752	5	105.98	0.0245	75.2	6	105.03	0.0073
Zn	213.85	0.2	3.00	5	110.76	0.0327	300	6	116.84	0.0153
Zn[2]	213.86	0.4	3.00	5	93.45	0.0351	300	6	94.01	0.0055
Zr	339.20	0.2	1.50	5	102.61	0.0242	150	6	101.56	0.0144

(1) Values reported were obtained with a Fisons ARL Accuris ICP-AES unless otherwise noted; performance may vary with instrument and should be independently verified.
(2) Values reported were obtained with a Perkin Elmer Optima 3000 DV ICP-AES.
(3) Elements that were evaluated and found not suitable for analysis by this method.
(4) Values given (lower level) are for the 10xLOQ level due to low recoveries at the 3xLOQ level.
(5) LOQ = Estimated limit of quantitation

Table 4. OVERALL PRECISION AND ACCURACY DATA [13]

Element	Instrument[(1)]	Range Studied (µg/sample)		Bias	Range of Bias		Precision S_{rT}	Accuracy (%)	Lowest Level[(2)]
		From	To		From	To			
Aluminum	Fisons	5.025	1500	-0.0318	-0.1022	0.0240	0.0419	9.9	50.25
Aluminum	P-E Optima	5.025	1500	0.0833	0.0567	0.1505	0.0379	15.1	15
Antimony	Fisons	5.025	1500	Poor and variable recoveries across study range.					
Arsenic	Fisons	5.025	1500	0.0630	-0.0671	0.1584	0.0461	14.3	15
Barium	Fisons	0.5038	150.4	0.0433	0.0222	0.0716	0.0182	7.6	0.5
Beryllium	Fisons	0.0509	15.2	0.0652	0.0366	0.0980	0.0163	9.5	0.0509
Boron	Fisons	2.514	750.4	-0.0387	-0.1362	0.0118	0.0164	6.4	7.504
Cadmium	Fisons	1.005	300.0	0.0923	0.0718	0.1167	0.0307	14.8	1.005
Calcium	Fisons	15.08	4500	0.0779	-0.0536	0.1624	0.0313	13.4	150.75
Calcium	P-E Optima	15.08	4500	0.0453	0.0098	0.0963	0.0245	8.8	15.08
Chromium	Fisons	2.514	750.4	0.1395	0.0974	0.1865	0.0214	18	2.514
Chromium	P-E Optima	2.514	750.4	-0.0018	-0.0701	0.1245	0.0131	<5	2.514
Cobalt	Fisons	2.514	750.4	0.0592	-0.1013	0.1508	0.0264	10.4	7.504
Copper	Fisons	0.5038	150.4	0.0475	0.0272	0.0684	0.0240	8.9	0.5038
Copper	P-E Optima	0.5038	150.4	0.0829	0.0313	0.1716	0.0217	12.1	1.504
Iron	Fisons	10.05	3000	0.1101	0.0630	0.2057	0.0397	18.6	30
Iron	Fisons	10.05	3000	0.0836	0.0630	0.0974	0.0396	15.4	100.5
Iron	P-E Optima	10.05	3000	0.0445	-0.0205	0.1255	0.0404	11.4	30
Lead	Fisons	5.025	1500	-0.0241	-0.0668	0.0124	0.0279	6.9	5.025
Lithium	Fisons	0.5038	150.4	-0.0690	-0.1804	0.0132	0.0276	11.1	0.5038
Magnesium	Fisons	5.025	1500	0.0156	-0.0253	0.0524	0.0171	<5	5.025
Magnesium	P-E Optima	5.025	1500	0.0715	0.0421	0.1372	0.0249	11.5	5.025
Manganese	Fisons	0.5038	150.4	0.1357	0.1005	0.1755	0.0201	17.3	0.5038
	Fisons	1.509	450.4	-0.0388	-0.1597	0.1353	0.0795	16.7	1.509
	P-E Optima	1.509	450.4	-0.0489	-0.2033	0.0969	0.0179	7.7	1.509
Nickel	Fisons	1.509	450.4	0.0787	0.0293	0.1274	0.0338	13.8	4.504
Nickel	P-E Optima	1.509	450.4	0.0645	0.0177	0.1406	0.0159	9.2	1.509
Phosphorus	Fisons	10.05	3000	-0.0546	-0.1818	0.0011	0.0417	12	30
Phosphorus	P-E Optima	10.05	3000	-0.0163	-0.1364	0.0333	0.0124	<5	10.05
Platinum	Fisons	50	15000	0.0423	0.0097	0.0671	0.0226	8.2	150
Potassium	Fisons	10.05	3000	-0.0909	-0.1443	-0.0316	0.0265	13.1	100.5
Potassium	P-E Optima	10.05	3000	-0.0499	-0.0998	-0.0060	0.0249	8.8	100.5
Selenium	Fisons	25.12	7500	0.0941	0.0675	0.1150	0.0150	12.1	25.12
Selenium	P-E Optima	25.12	7500	0.0026	-0.0027	0.0115	0.0127	<5	25.12
Silver	Fisons	1.005	300	Poor and variable recoveries across study range.					
Sodium	Fisons	25.12	7500	-0.0492	-0.1694	0.0718	0.0246	8.8	251.2
Strontium	Fisons	2.514	750.4	0.0172	-0.00002	0.0373	0.0153	<5	2.514
Tellurium	Fisons	10.05	3000	0.0295	-0.0420	0.1037	0.0404	9.8	30

(1) Values reported were obtained with a Fisons ARL Accuris ICP-AES or a Perkin Elmer Optima 3000 DV ICP-AES.
(2) Lowest level in range studied at which recoveries were between 81 and 121% recovery and relative standard deviation (S_r) less than 0.1100 on 5 or 6 replicates. Performance may vary with instrument and should be independently verified.

Table 4. OVERALL PRECISION AND ACCURACY DATA [13]

Element	Instrument(1)	Range Studied (µg/sample)		Bias	Range of Bias		Precision S_{rT}	Accuracy (%)	Lowest Level(2)
		From	To		From	To			
Tellurium	P-E Optima	10.05	3000	-0.0043	-0.0282	0.0163	0.0155	<5	10.05
Thallium	Fisons	5.025	1500	-0.0081	-0.0362	0.0334	0.0407	8.2	15
Thallium	P-E Optima	5.025	1500	-0.0505	-0.0688	-0.0048	0.0250	9	5.025
Tin	Fisons	25.12	7500	Poor and variable recoveries across study range.					
Tin	P-E Optima	25.12	7500	Poor and variable recoveries across study range.					
Titanium	Fisons	1.005	300	-0.0827	-0.1834	0.0342	0.0269	12.3	3
Titanium	P-E Optima	1.005	300	-0.1072	-0.1732	-0.0387	0.0321	15.3	1.005
Vanadium	Fisons	0.5038	150.4	0.0704	0.0438	0.1114	0.0195	10.5	0.5038
Vanadium	P-E Optima	0.5038	150.4	-0.0063	-0.0217	0.0099	0.0198	<5	0.5038
Yttrium	Fisons	0.2519	75.2	0.0598	0.0466	0.0795	0.0164	8.9	0.2519
Zinc	Fisons	1.005	300	0.1452	0.0630	0.2976	0.0340	22	1.005
Zinc	Fisons	1.005	300	0.1190	0.0630	0.1683	0.0356	18.7	3
Zinc	P-E Optima	1.005	300	-0.0502	-0.0655	-0.0388	0.0295	9.6	3
Zirconium	Fisons	0.5025	150	0.0164	-0.0096	0.0350	0.0175	<5	0.5025

(1) Values reported were obtained with a Fisons ARL Accuris ICP-AES or a Perkin Elmer Optima 3000 DV ICP-AES.
(2) Lowest level in range studied at which recoveries were between 81 and 121% recovery and relative standard deviation (S_r) less than 0.1100 on 5 or 6 replicates. Performance may vary with instrument and should be independently verified.