



A Honeywell Company

Trace Metals in Organics by ICP-MS

UOP Method 1005-14

Scope

This method is for determining the concentrations of aluminum (Al), arsenic (As), calcium (Ca), cadmium (Cd), chromium (Cr), cobalt (Co), copper (Cu), iron (Fe), gallium (Ga), lead (Pb), lithium (Li), magnesium (Mg), manganese (Mn), molybdenum (Mo), nickel (Ni), palladium (Pd), phosphorus (P), platinum (Pt), potassium (K), sodium (Na), strontium (Sr), tin (Sn), titanium (Ti), vanadium (V), zinc (Zn), and zirconium (Zr), in organic matrices such as crude petroleum, asphalts, vacuum tower bottoms, vacuum gas oils, atmospheric gas oils, diesel and jet fuels and their blending components, pyrolysis oils, and fatty acid derivatives by Inductively Coupled Plasma – Mass Spectrometry (ICP-MS). The lower limits of quantitation for the above elements, are listed in Table 1. Determination of additional elements is possible if they are compatible with other analytes during digestion.

Table 1
Lower Limits of Quantitation, mg/kg (mass-ppm)

Al	0.02	K	0.03	Pd	0.01
As	0.01	Li	0.01	Pt	0.01
Ca	0.03	Mg	0.02	Sn	0.01
Cd	0.01	Mn	0.01	Sr	0.01
Co	0.01	Mo	0.01	Ti	0.03
Cr	0.01	Na	0.06	V	0.01
Cu	0.01	Ni	0.01	Zn	0.01
Fe	0.02	P	0.50	Zr	0.01
Ga	0.01	Pb	0.01		

Alternatively, many of the elements listed above can be determined using UOP Method 389, “Trace Metals in Organics by ICP-OES.” Some of the elements listed above may be analyzed by Atomic Absorption Spectroscopy (AAS). See UOP Method 391, “Trace Metals in Petroleum Products or Organics by AAS,” for specific metals and their range of quantitation. Metals known to be non-volatile may be analyzed by UOP Method 407, “Trace Metals in Organics by Dry Ashing - ICP-OES.” Many of these elements can be determined in kerosene using ASTM Method D7111, “Determination of Trace Elements in Middle Distillate Fuels by Inductively Coupled Plasma Atomic Emission Spectrometry (ICP-AES).”

References

ASTM Specification D1193, “Reagent Water,” www.astm.org

IT IS THE USER'S RESPONSIBILITY TO ESTABLISH APPROPRIATE PRECAUTIONARY PRACTICES AND TO DETERMINE THE APPLICABILITY OF REGULATORY LIMITATIONS PRIOR TO USE. EFFECTIVE HEALTH AND SAFETY PRACTICES ARE TO BE FOLLOWED WHEN UTILIZING THIS PROCEDURE. FAILURE TO UTILIZE THIS PROCEDURE IN THE MANNER PRESCRIBED HEREIN CAN BE HAZARDOUS. SAFETY DATA SHEETS (SDS) OR EXPERIMENTAL SAFETY DATA SHEETS (ESDS) FOR ALL OF THE MATERIALS USED IN THIS PROCEDURE SHOULD BE REVIEWED FOR SELECTION OF THE APPROPRIATE PERSONAL PROTECTION EQUIPMENT (PPE).

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ASTM Method D7111, "Determination of Trace Elements in Middle Distillate Fuels by Inductively Coupled Plasma Atomic Emission Spectrometry (ICP-AES)," www.astm.org

ASTM Practice D7455, "Sample Preparation of Petroleum and Lubricant Products for Elemental Analysis," www.astm.org

ASTM Practice D7876, "Sample Decomposition Using Microwave Heating (With or Without Prior Ashing) for Atomic Spectroscopic Elemental Determination in Petroleum Products and Lubricants," www.astm.org

UOP Method 389, "Trace Metals in Organics by ICP-OES," www.astm.org

UOP Method 391, "Trace Metals in Petroleum Products or Organics by AAS," www.astm.org

UOP Method 407, "Trace Metals in Organics by Dry Ashing - ICP-OES," www.astm.org

UOP Method 999, "Precision Statements in UOP Methods," www.astm.org

Outline of Method

The sample is treated initially with water, then nitric and hydrofluoric acids, to reduce the volatility of some of the metals. Then the samples are digested. The digestate is quantitatively transferred to a polypropylene flask using purified water. Scandium and bismuth are added as internal standards and the mixture is diluted to the desired volume. The concentrations of elements in the resulting solutions are determined by ICP-MS.

Apparatus

References to catalog numbers and suppliers are included as a convenience to the method user. Other suppliers may be used.

Balance, readable to 0.0001 g

Beaker, PTFE, 600-mL, Fisher Scientific, Cat. No. 02-586-1K

Bottles, polypropylene, narrow mouth, screw cap, 125-mL, VWR, Cat. No. 16067-066

Bottle, wash, VWR, Cat. No. 2402-0500, for deionized water

Cover, PTFE, Milestone, Cat. No. MCL0435, with o-ring, Cat. No. OR0138, used to cover the stainless steel vessel in the microwave oven

Cylinders, graduated, Class B, 5-, 10-, 25-, and 100-mL, VWR, Cat. Nos. 14201-610, -462, -364, and -718, respectively

Flasks, volumetric, polypropylene, Class B, 25-, 50-, 100-, and 1000 mL, VWR, Cat. Nos. 83008-964, -974, -984, and 83009-008, respectively; several of each required

Fume hood

Hot plate, VWR, Cat. No. 11301-006

ICP-MS, PerkinElmer, DRC-e, equipped with an AS10 autosampler, or equivalent newer version

Microwave oven, 15-vessel capacity, with closed digestion vessels, quartz tubes and lids, CryoLab chiller, and computer, Milestone, UltraWAVE, or equivalent newer version

Pipet, automatic, 10-100- μ L, VWR, Cat. No. 89079-968; calibrated and/or certified according to manufacturer's recommendations

Pipet, automatic, 100-1000- μ L, VWR, Cat. No. 89079-974; calibrated and/or certified according to manufacturer's recommendations

Pipet, electronic, 1-10-mL, EDP 3-Plus, Rainin, Cat. No. SE3-10mL; calibrated and/or certified according to manufacturer's recommendations

Regulator, argon or nitrogen, two-stage, high purity, delivery pressure range 30-700 kPa (4-100 psi), Matheson Tri-Gas, Model 3122-580

Timer, capable of timing two hours, VWR, Cat. No. 62344-440

Watch glass, PTFE, 125-mm, Fisher Scientific, Cat. No. 02-617-1F

Reagents and Materials

References to catalog numbers and suppliers are included as a convenience to the method user. Other suppliers may be used. Unless otherwise specified, references to water mean deionized water.

Argon, 99.995% minimum purity

Bismuth, indium, and scandium stock solution, aqueous, 100- μ g/mL. To prepare 50 mL, pipet 0.5 mL of the 10,000- μ g/mL scandium, 5 mL of the 1000- μ g/mL indium, and 5 mL of the 1000- μ g/mL bismuth metal solutions into a 50-mL volumetric flask. Fill to the mark with deionized water. Cap and invert several times to mix. The solution should remain stable for two months.

Bismuth, indium, and scandium internal standard solution, 10- μ g/mL. To prepare 100 mL, pipet 10.0 mL of the 100- μ g/mL scandium, indium, and bismuth metal stock solution into a 100-mL volumetric flask. Fill to the volume mark with deionized water and mix. Store in a 125-mL polypropylene bottle. The solution should remain stable for two months.

Bismuth metal solution, aqueous, 1000- μ g/mL, SPEX CertiPrep, Inc., Cat. No. PLBI-2, for use as an internal standard

Cleaning compound, Alconox detergent, VWR, Cat. No. 21835-032

Gloves, neoprene/natural rubber, VWR, Cat. No. 32917-212 (size 10)

Gloves, Viton, VWR, Cat. No. 89236-768 (size 10)

Hydrofluoric acid, concentrated, trace metals grade, VWR, Cat. No. EM-HX0628-7

Hydrogen peroxide, 29-32%, VWR, Cat. No. VW3690-5

Indium metal solution, aqueous, 1000- μ g/mL, SPEX CertiPrep, Inc., Cat. No. PLIN2-2X, for use as a stabilizer

Lubricant, dry spray silicone or PTFE, W. W. Grainger, Cat. No. 4JB33, or local supply

Nitric acid, concentrated, trace metals grade, VWR, Cat. No. EM-NX0408-2

Nitric acid, 2%. Pipet 20 mL of concentrated nitric acid into a 1000-mL containing ~800-mL of deionized water; fill to mark with deionized water, and invert several times to mix.

Nitrogen, 99.995% minimum purity, optional, see *Microwave Digestion*, Step 6

O-rings, replacement, for microwave oven, Milestone, Cat. No. OR0159

O-ring, top of chamber, replacement, Milestone, Cat. No. OR0170

Pipet, dropping, VWR, Cat. No. 52950-206

Pipet tips, 10 mL, Rainin, Cat. No. RC-10 mL

Pipet tips, 1-200- μ L VWR, Cat. No. 53508-810

Pipet tips, 100-1250- μ L, VWR, Cat. No. 53508-922

Scandium metal solution, aqueous, 10,000- μ g/mL, SPEX CertiPrep, Inc., Cat. No. PLSC2-3X, for use as an internal standard

Standard metal solutions, Al, As, Ca, Cd, Co, Cr, Cu, Fe, Ga, K, Li, Mg, Mn, Mo, Na, Ni, P, Pb, Pd, Pt, Sn, Sr, Ti, V, Zn, and Zr solutions of 1000- μ g/mL concentrations, SPEX CertiPrep, Inc., Cat. Nos. PLAL1-2, PLAS2-2, PLCA1-2, PLCD2-2, PLCO2-2, PLCR1-2, PLCU2-2, PLFE2-2, PLGA2-2, PLK1-2, PLI2-2, PLMG2-2, PLMN2-2, PLMO2-2, PLNA2-2, PLNI2-2, PLP9-2, PLPB2-2, PLPD3-2, PLPT3-2, PLSN5-2, PLSR2-2, PLTI9-2, PLV2-2, PLZN2-2, and PLZR2-2, respectively

Sulfuric acid, concentrated, trace metals grade, VWR, Cat. No. EM-SX1248-6

Towels, paper, disposable, local supply

Vials, autosampler, 50 mL, with caps, PerkinElmer, Cat. No. BO193234

Vials, PTFE, 15-mL, Milestone, Cat. No. HB00086, with caps, Cat. No. DD00134

Water, deionized, ASTM D1193 Type III reagent grade water, unless otherwise specified

Water, hot, tap

Wipers, Kimwipes Ex-L, VWR, Cat. No. 21905-026

The following materials are not required to perform the analyses described herein and are not cited in the *Procedure* portion of the method. However, they may be useful for reference purposes, for verification of instrument performance, or as an aid in instrument maintenance. The multi-element standard, diluted, was used to develop part of the precision statement.

Kerosene solvent, metals free, VWR, Cat. No. AA36447-K7 or SPEX, Cat. No. KER-BLK-G

Reference material, SPEX CertiPrep 23 Multi-Element Standard, 100- μ g/g (mg/kg) each, SPEX CertiPrep, Inc., Cat. No. S23 1004. Dilute by weight in kerosene solvent to 10 μ g/g.

Procedure

The analyst is expected to be familiar with general laboratory practices, the technique of ICP-MS, and the equipment being used. Additional information regarding sample preparation may be found in ASTM Practice D7455, "Sample Preparation of Petroleum and Lubricant Products for Elemental Analysis." Additional information regarding the use of microwave digestion for sample decomposition may be found in ASTM Practice D7876, "Sample Decomposition Using Microwave Heating (With or Without Prior Ashing) for Atomic Spectroscopic Elemental Determination in Petroleum Products and Lubricants." Dispose of all supplies and samples in an environmentally safe manner according to applicable regulations.

CAUTION: All subsequent steps involving additions of reagents and heating of samples must be performed in a properly operating fume hood, and appropriate personal protective equipment must be worn. See the safety data sheet (SDS) and any local requirements for each material used.

Preparation of Standards

Standards are required for each element to be determined. They are prepared to contain a number of elements with compatible chemistries. Stock standard solutions are prepared first, from which lower level, matrix matched, calibration standard solutions are then prepared.

Preparation of Stock Standard Solutions

Prepare stock standard solutions of 10- $\mu\text{g/mL}$ concentration of each element (except Ca 20- $\mu\text{g/mL}$ and P 40- $\mu\text{g/mL}$) as follows:

1. Pipet 1.0 mL each of the Al, As, Ca (2.0 mL), Cd, Cr, Co, Cu, Fe, Ga, Mg, Mn, Ni, and Pb, 1000- $\mu\text{g/mL}$ commercial stock standards into a 100-mL polypropylene volumetric flask. Dilute to the mark with deionized water. Label as Stock Standard A. The solution should remain stable for two months.
2. Pipet 1.0 mL each of the Pd, Pt, Sn, V, and Zn 1000- $\mu\text{g/mL}$ commercial stock standards into a second 100-mL polypropylene volumetric flask. Dilute to the mark with deionized water. Label as Stock Standard B. The solution should remain stable for two months.
3. Pipet 1.0 mL each of the Mo, P (4.0 mL) Ti, and Zr, 1000- $\mu\text{g/mL}$ commercial stock standards into a third 100-mL polypropylene volumetric flask. Dilute to the mark with deionized water. Label as Stock Standard C. The solution should remain stable for two months.
4. Pipet 1.0 mL each of the K, Li, Na, and Sr 1000- $\mu\text{g/mL}$ commercial stock standards into a fourth 100-mL polypropylene volumetric flask. Dilute to the mark with deionized water. Label as Stock Standard D. The solution should remain stable for two months.
5. Pipet 1.0 mL of the K 1000- $\mu\text{g/mL}$ commercial stock standard into a fifth 100-mL polypropylene volumetric flask. Dilute to the mark with deionized water. Label as Stock Standard E. The solution should remain stable for two months.

Cleaning of PTFE Vials and Caps

Clean the PTFE vials and caps as follows:

1. Place PTFE vials and caps in a 600-mL beaker, cover with 2% nitric acid, and place a watch glass on top of the beaker.
2. Place the beaker on a hot plate. Set the hot plate to 200 °C and bring to a boil. Adjust the temperature of the hot plate to maintain a slow boil. Boil for two hours.
3. Remove the beaker from the hot plate and allow to cool.
4. Decant the acid and dispose of it in an environmentally safe manner according to applicable regulations.
5. Rinse the PTFE vials, caps, beaker, and watch glass with deionized water, and air dry.
6. Store the vials and caps in the beaker, covered with the watch glass until needed.

Microwave Digestion

The blank and the reference are digested in the same manner as the samples.

1. Weigh 0.20-0.25 g of each sample to the nearest 0.0001 g into the previously cleaned and air dried vials. Using a pipet or graduated cylinder, add 1 mL of water. Using a graduated cylinder, add 3 mL of concentrated nitric acid (HNO_3). Using a plastic dropping pipet, add three drops of hydrofluoric acid (HF) to the PTFE vials.

2. Prepare an additional three vials as digestion acid blanks by adding 1 mL water, 3 mL concentrated nitric acid and three drops of hydrofluoric acid to each of the pre-cleaned, air dried PTFE vials.
 - One vial is used for preparing the blank, two are used for standard preparation. This is to ensure matrix matching with the samples.
 - The addition of hydrofluoric acid is used to stabilize tin (Sn). If the results for tin (Sn) are inconsistent, increase the amount of hydrofluoric acid (HF) to five drops.
3. Close the digestion vessels and place them into the sample holder.
4. Set up the microwave as described below. Refer to the UltraWAVE Operator Manual for details. Add the basic load/absorption media as follows: Using graduated cylinders, add 130 mL of water, 5 mL of 30% hydrogen peroxide, and 2 mL of sulfuric acid into the PTFE vessel that goes into the stainless steel pressure vessel. This acts as absorption media.
 - Ensure that all o-rings are properly cleaned and greased.
5. Check the water level in the CryoLab chiller.
6. Turn on the gas (nitrogen or argon), CryoLab chiller, computer, and UltraWAVE.
7. Program the system using the settings shown in Table 2.
 - Other conditions may be used provided they produce equivalent results.

Table 2
Digestion Conditions

<u>Nr.</u>	<u>t(min)</u>	<u>E (w)</u>	<u>T1 °C</u>	<u>T2 °C</u>	<u>P</u>
1	00:30:00	1500	230	60	150
2	00:40:00	1500	230	60	150

Nr. 1 is for reaching to the prescribed condition,
and Nr. 2 is holding at that condition.

8. Load the samples into the UltraWAVE.
9. Press the down arrow on the touchscreen monitor to lower the samples to level 1, check that everything is OK, then press the down arrow again to lower it to level 2. At this point the sample holder is immersed into the absorption media.
10. Manually close the clamps.
11. Check the method and parameters, and then start the run.
 - At the end of the digestion process, after the materials have cooled to 30 °C or lower on the readout, open the UltraWAVE clamps, press the up arrow on the monitor to lift the sample holder, wait for the fumes to exhaust through the vent, and press the up arrow again to lift the sample holder fully. Cover the stainless steel vessel with the PTFE cover to protect it from possible dripping of the acid condensate .
12. Carefully remove the sample holder with the PTFE vials and place on disposable paper towels.
13. Lift each vial from the sample holder and wipe the bottom on a paper towel. Quantitatively transfer the contents of each vial into separate 25-mL polypropylene volumetric flasks using deionized water as the diluent.
14. Add 100 µL of the 10-µg/mL scandium, indium, and bismuth internal standard solution to each volumetric flask containing samples.
15. Fill each flask to the mark with water. Cap and invert several times to thoroughly mix the contents.

16. Immediately after use, to avoid corrosion of the UltraWAVE's stainless steel pressure vessel, dispose of the absorption media, and rinse with water. Carefully clean and wipe dry other exposed parts. Spray lubricant on a wiper and wipe as described in the UltraWAVE Operator Manual. Also clean the PTFE container and PTFE vials.

Preparation of Blank and Calibration Standard Solutions

Prepare calibration standards and a blank as follows. The blank and calibration standards for potassium (K) are prepared separately.

1. Into one of the three volumetric flasks containing the digestate of the acid blank, pipet 50 μL of each of the 10.0 $\mu\text{g/mL}$ stock standard solutions A through D. Label as calibration standard solution, 20 ng/mL (except Ca, 40-ng/mL, and P, 80-ng/mL).
2. Into a second flask containing the digestate of the acid blank, pipet 125 μL of each of the 10.0- $\mu\text{g/mL}$ standard stock solutions A through D. Label as calibration standard solution, 50-ng/mL (except Ca, 100-ng/mL, and P, 200-ng/mL).
 - The third flask containing the digestate of the acid blank is the blank. Label as blank solution.
3. Add 100 μL of the 10- $\mu\text{g/mL}$ scandium, indium, and bismuth internal standard solution to each volumetric flask.
4. Fill each flask to the mark with water. Cap and invert several times to thoroughly mix the contents.

Preparation of Blank and Calibration Standard Solutions for Potassium (K)

Prepare calibration standards and a blank as follows for potassium:

1. Add 10 mL of purified water, 2 mL of nitric acid (HNO_3) and 1 drop of hydrofluoric acid to each of the three 25-mL polypropylene volumetric flasks.
2. Pipet 50 μL of potassium (K) Stock Standard E into the flask labeled 20-ng/mL Calibration Standard 1 for potassium. Pipet 125 μL of potassium (K) Stock Standard E into the flask labeled 50-ng/mL Calibration Standard 2 for potassium.
3. The third flask will be used as the blank.
4. Pipet into each of the three flasks 100 μL of the 10.0- $\mu\text{g/mL}$ scandium, indium, and bismuth internal standard solution.
5. Fill each to the mark with deionized water. The solutions should remain stable for one month.

ICP-MS Determination of Analytes

The ICP-MS is calibrated with prepared standards using the autosampler. Three calibration points are obtained by analyzing a blank, 20- and 50-ng/mL mixed standards. Samples are then analyzed and the 20- and 50-ng/mL standards are reanalyzed as samples at the end of the run.

1. Set up the instrument according to the manufacturer's recommended conditions, see Table 3. The recommended isotope to be used for the analysis of each analyte is shown in Table 4.
2. Transfer aliquots of the digested samples, blank, and calibration standards into autosampler vials.
3. Enter the locations of all the solutions to be analyzed into the instrument data system.

Table 3
ICP-MS Parameters

ICP/MS Instrument	Perkin Elmer DRC-e
Analytes	Al, As, Ca, Cd, Co, Cr, Cu, Fe, Ga, K, Li, Mg, Mn, Mo, Na, Ni, P, Pb, Pd, Pt, Sn, Sr, Ti, V, Zn, and Zr
Internal standard	⁴³ Sc, ²⁰⁹ Bi, ¹¹⁵ In (Sc and Bi are used as internal standards; In is used as a stabilizer)
RF power	1500 W
Plasma gas flow	15 L/min
Auxiliary gas flow rate	1.2 L/min
Nebulizer	Nebulizer Cross Flow II
Nebulizer gas flow rate	~0.85 L/min
Spray chamber	Ryton™ Scott-type
Ion sampling and skimmer cone	nickel cone
Sampling distance	10 mm from load coil
Sample wash	
Peristaltic pump speed	-20 rpm
Uptake	30 seconds
Wash	60 seconds
Resolution	standard
Detector mode	dual
Dwell Time	50 ms
Sweeps per reading	20
Readings per replicate	1
Replicates	3

Table 4
Recommended Isotope for Each Element

<u>Element</u>	<u>Concentration Range in Sample, mg/kg</u>	<u>Isotope</u>	<u>Element</u>	<u>Concentration Range in Sample, mg/kg</u>	<u>Isotope</u>
Al	0.01-10	27	Mo	0.01-10	98
As	0.01-10	75	Na	0.06-10	23
Bi	internal std	209	Ni	0.01-10	60
Ca	0.04-10	43	P	0.50-30	31
Cd	0.01-10	112	Pb	0.01-10	208
Co	0.01-10	59	Pd	0.01-10	106
Cr	0.01-10	52	Pt	0.01-10	195
Cu	0.01-10	63	Sc	Internal std	23
Fe	0.02-10	57	Sn	0.01-10	118
Ga	0.01-10	69	Sr	0.01-10	88
In	(stabilizer)	115	Ti	0.03-10	50
K	0.03-10	39	V	0.01-10	51
Li	0.01-10	7	Zn	0.01-10	66
Mg	0.02-10	24	Zr	0.01-10	90
Mn	0.01-10	55			

Scandium is used as an internal std for Li, Na, Mg, Al, P, K and Ca.
Bismuth is used as an internal std for the rest of the analytes.

4. Run the blank, calibration standards, and the samples. At the end of the series, analyze the blank and calibration standards as samples.
 - If the standards run at the end of the series do not repeat within the repeatability allowable difference shown under *Precision*, check the apparatus and technique, and after fixing any problems found, rerun the samples.

Analysis of Potassium

1. Transfer aliquots of the blank, samples, and potassium calibration standards into autosampler vials.
2. Enter the locations of all the solutions to be analyzed into the instrument data system.
3. Run the blank, calibration standards, and the samples. At the end of the series, analyze the blank and calibration standards as samples.
 - If the standards run at the end of the series do not repeat within the repeatability allowable difference shown under *Precision*, check the apparatus and technique, and after fixing any problems found, rerun the samples.
4. Dispose of the remaining solutions in an environmentally safe manner according to applicable regulations.

Calculations

All calculations are performed by the instrument data system after the sample mass and dilutions are entered, and sample results are reported directly in mg/kg. If all the data is not entered into the instrument data system, the results will be reported in ng/mL. Use Equation 1 to calculate the final sample concentration.

- If any analyte's concentration is more than 20% above that of the highest standard, dilute the sample further and reanalyze.

$$\text{Trace metal concentration, mg/kg} = \frac{25 C}{W} \quad (1)$$

where:

C = ng/mL analyte in the analyzed solution as reported by the instrument

W = sample weight, g

25 = diluted volume of the digest, mL

Report results in mg/kg to two significant figures.

- If the results for tin appear to be inconsistent, see Microwave Digestion, Step 2.

Precision

Precision statements were determined using UOP Method 999, "Precision Statements in UOP Methods," from precision data obtained using an autosampler.

Repeatability and Site Precision

A nested design was carried out for determining metals in organics by two analysts, with each analyst performing analyses on two separate days, performing two analyses each day for a total of eight analyses. Using a stepwise analysis of variance procedure, the within-day and within-lab estimated standard deviations (esd) were calculated at the concentration means listed in Table 5. Two analyses performed in one laboratory by the same analyst on the same day should not differ by more than the repeatability allowable differences shown in Table 5 with 95% confidence. Two analyses performed in one laboratory by different analysts on different days should not differ by more than the site precision allowable differences shown in Table 5 with 95% confidence.

The data in Table 5 represent short-term estimates of the repeatability of the method. When the test is run routinely, use of a control standard and a control chart is recommended to generate an estimate of long-term repeatability.

Table 5
Repeatability and Site Precision, mg/kg

<u>Component</u>	<u>Mean</u>	<u>Repeatability</u>		<u>Site Precision</u>	
		<u>Within- Day esd</u>	<u>Allowable Difference</u>	<u>Within- Lab esd</u>	<u>Allowable Difference</u>
Al	2.04	0.087	0.30	0.104	0.41
As	1.92	0.021	0.07	0.026	0.10
Ca	4.14	0.236	0.93	0.283	0.98
Cd	2.01	0.043	0.15	0.056	0.22
Co	2.06	0.040	0.16	0.046	0.16
Cr	2.02	0.052	0.20	0.135	0.47
Cu	2.02	0.051	0.20	0.066	0.23
Fe	1.80	0.046	0.18	0.132	0.46
Ga	2.05	0.047	0.16	0.058	0.23
K	2.12	0.075	0.30	0.147	0.51
Li	1.97	0.023	0.09	0.060	0.21
Mg	2.04	0.055	0.21	0.159	0.55
Mn	2.10	0.046	0.18	0.086	0.30
Mo	2.05	0.032	0.12	0.160	0.55
Na	2.00	0.048	0.19	0.154	0.53
Ni	2.05	0.037	0.14	0.082	0.28
P	6.57	0.408	1.55	0.449	1.60
Pb	2.04	0.039	0.15	0.050	0.17
Pd	2.01	0.036	0.14	0.082	0.28
Pt	1.96	0.076	0.30	0.089	0.31
Sn	2.09	0.043	0.17	0.055	0.19
Sr	2.06	0.036	0.14	0.066	0.23
Ti	2.09	0.099	0.39	0.121	0.42
V	2.11	0.054	0.21	0.100	0.35
Zn	1.95	0.045	0.18	0.098	0.34
Zr	2.08	0.033	0.13	0.165	0.57

Reproducibility

There is insufficient data to calculate the reproducibility of the test at this time.

Time for Analysis

The elapsed time for one analysis is approximately 4 hours, depending upon sample preparation. The labor requirement for one analysis is 1 hour. In groups of 5 or more samples, the labor requirement for each analysis is 0.5 hour.

Suggested Suppliers

Fisher Scientific, 300 Industry Dr., Pittsburgh, PA 15275, USA, 1-412-490-8300,
www.fishersci.com

Matheson Tri Gas, 150 Allen Rd # 302, Basking Ridge, NJ 07920, USA, 1-908-991-9200,
www.mathesontrigas.com

Milestone Inc., 25 Controls Dr., Shelton, CT 06484, USA, 1-203-925-4240, www.milestonesci.com

National Institute of Standards and Technology (NIST), Standard Reference Materials Group, 100
Bureau Dr., Gaithersburg, MD 20899, USA, 1-301-975-6776, www.nist.gov

PerkinElmer Analytical Instruments, 710 Bridgeport Ave., Shelton, CT 06484-4794, USA, 1-203-
925-4600, www.perkinelmer.com

Rainin Instrument Co., Mack Road, Woburn, MA 01801, USA, 1-800-472-4646, www.rainin.com

SPEX CertiPrep, Inc., 203 Norcross Ave., Metuchen, NJ 08840, USA, 1-732-549-7144,
www.spexcsp.com

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