



A Honeywell Company

Metals in Miscellaneous Samples by ICP-OES

UOP Method 714-07

Scope

This method is for determining metals in samples of unknown composition such as residues and deposits. Aluminum (Al), cadmium (Cd), calcium (Ca), chromium (Cr), cobalt (Co), copper (Cu), iron (Fe), lead (Pb), magnesium (Mg), manganese (Mn), molybdenum (Mo), nickel (Ni), potassium (K), sodium (Na), strontium (Sr), tin (Sn), titanium (Ti), vanadium (V), zinc (Zn), and zirconium (Zr) are quantitatively determined by Inductively Coupled Plasma - Optical Emission Spectrometry (ICP-OES). Concentrations determined generally cover the range of 0.02 to several mass-%, see Table 1. Sample dilution extends the working range for major (matrix) components. If requested, additional elements can be determined, if the sample preparation method is appropriate for the additional elements.

Table 1
Applicable Concentration Ranges, Mass-%

<u>Element</u>	<u>Range</u>	<u>Element</u>	<u>Range</u>
Aluminum	0.02 - 2.5	Manganese	0.01 - 2.5
Cadmium	0.02 - 1.25	Molybdenum	0.03 - 2.5
Calcium	0.01 - 10	Nickel	0.03 - 2.5
Chromium	0.03 - 2.5	Potassium	0.10 - 2.5
Cobalt	0.02 - 2.5	Sodium	0.01 - 10
Copper	0.01 - 1.25	Tin	0.05 - 2.5
Iron	0.02 - 50	Titanium	0.01 - 1.25
Lead	0.05 - 2.5	Vanadium	0.01 - 5
Magnesium	0.01 - 10	Zinc	0.02 - 2.5

Barium (Ba) and silicon (Si) cannot be determined by this method due to the acid treatment of the samples. Alpha-alumina will not dissolve in the acid media used in this method.

An alternative microwave-based dissolution technique is described in the *Appendix*. Silicon can be determined using the microwave-based dissolution technique; and instructions for the analysis of silicon are included.

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. MATERIAL SAFETY DATA SHEETS (MSDS) OR EXPERIMENTAL MATERIAL SAFETY DATA SHEETS (EMSDS) 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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References

ASTM Method D 1193, "Specification for Reagent Water," www.astm.org

UOP Method 954, "Loss on Ignition for Fresh, Regenerated, Used, and Spent Catalysts, Catalyst Supports, and Adsorbents," www.astm.org

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

Outline of Method

The samples are decomposed by digestion with sulfuric and hydrofluoric acids. The addition of hydrofluoric acid and heating the digest volatilizes silica. Additional acid treatments may be necessary, depending upon the sample matrix. Scandium is added to the solution as the internal standard and the sample solutions are analyzed by ICP-OES calibrated with appropriate, matrix(acid)-matched standards.

Apparatus

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

Balance, readability 0.1-mg

Beaker, Teflon™, PTFE, 250-mL, Fisher Scientific, Cat. No. 02-593-5B

Bottle, wash, polyethylene, 500-mL, VWR, Cat. No. 16651-187

Sample grinding equipment

Balls, grinding, 11.1-mm (7/16-inch), tungsten carbide, SPEX Industries, Inc., Cat. No. 8004A, or equivalent

Container, grinding, 57 x 64 mm (2-1/4 x 2-1/2 inches), tungsten carbide, SPEX Industries, Inc., Cat. No. 8004, or equivalent

Mixing mill, shaker type, SPEX Industries, Inc., Cat. No. 8000

Mortar and pestle, Agate, VWR, Cat. No. 50410-060

Cylinders, graduated, polypropylene, Class B, 10- and 100-mL, Fisher Scientific, Cat. Nos. 08-572A and -572D, respectively

Flasks, volumetric, Class B, Nalgene polypropylene, 100, 200, and 500-mL capacity, Fisher Scientific, Cat. Nos. 10-198-50B, -50C, and -50E respectively, several of each required

Hot plate, stirring, variable heat to at least 400°C

Pipet, microliter, 2500-µL capacity, Rainin Instrument Co., Cat. No. EP-2500

Pipet, 10-mL capacity, Rainin Instrument Co., Cat. No. EP-10ML

Pipets, volumetric, transfer, Class A, 1-, 2-, 5-, 10-, 20-, 25-, 40-, and 50-mL, Fisher Scientific, Cat. Nos. 13-650-2B, -2C, -2F, -2L, -2N, -2P, -2R, and -2S, respectively

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

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

Spectrometer, Inductively Coupled Plasma-Optical Emission Spectrometer, computer controlled, having sufficient resolving power and dispersion to separate the analytical lines in the 160-800 nm region. The computer must be capable of performing background corrections, blank corrections, and weight/volume and dilution corrections. A commercial grating spectrometer with a band pass of 0.018 nm or less in the first order is satisfactory. PerkinElmer Optima 5300 V

Watch glass, Teflon, PTFE, 75-mm diameter, Fisher Scientific, Cat. No. 02-610-5C

Weighing scoop, Fisher Scientific, Cat. No. 01-914-36

Reagents and Materials

References to catalog numbers and suppliers are included as a convenience to the method user. Other suppliers may be used. References to water mean ASTM D 1193 Type I reagent grade water.

Argon, 99.995% minimum purity, ICP feed gas

Hydrochloric acid, concentrated

Hydrofluoric acid, concentrated

Nitrogen, 99.99% minimum purity, ICP optics purge gas

Pipet tips, disposable, for EP-2500 microliter pipet, Rainin Instrument Co., Cat. No. RC-2500

Pipet tips, disposable, for EP-10ML pipet, Rainin Instrument Co., Cat. No. RC-2500

Pipets, disposable, polyethylene, general purpose, 1.7-mL, VWR, Cat. No. 16001-174

Scandium solution, aqueous, 1000 µg/mL, SPEX Industries, Inc., Cat. No. PLSC-2

Standard solutions of metals, Fe, Ni, V, Ca, Mg, Mn, Cr, Sn, Cu, Zn, Ti, Pb, Na, Mo, Co, Al, Cd, K, Sr, and Zr solutions of 1000 µg/mL concentrations, SPEX Industries, Inc., Cat. Nos. PLFE-2, PLNI-2, PLV-2, PLCA-2, PLMG-2, PLMN-2, PLCR-2, PLSN-2, PLCU-2, PLZN-2, PLTI-2, PLPB-2, PLNA-2, PLMO-2, PLCO-2, PLAL-2, PLCD-2, PLK-2, PLSR-2X, and PLZR3-2X respectively

Sulfuric acid, concentrated

Nitric acid, concentrated

Water, ASTM D 1193 Type I reagent grade

Procedure

Caution: All steps involving additions of acids and heating of samples must be performed in a properly operating fume hood, and appropriate personal protective equipment must be worn. See the MSDS and local regulations for each material used.

The analyst is expected to be familiar with general laboratory practices, the technique of ICP-OES, and the equipment being used.

Preparation of Standards

Due to the linear response of the ICP, single point standards are used.

Prepare four stock standard solutions at the 50-µg/mL level, labeled as Stock Standards 2 through 5.

- This labeling follows SPEX numbering convention, wherein standard number 1 is used for rare earth elements, which are not addressed in this method.