A Honeywell Company

Trace Metals in Methanol by ICP-OES

UOP Method 1003-13

Scope

This method is for determining trace concentrations of calcium (Ca), iron (Fe), magnesium (Mg), potassium (K), and sodium (Na) in methanol by Inductively Coupled Plasma – Optical Emission Spectrometry (ICP-OES). The lower limits of quantitation for these elements are listed in Table 1.

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

Ca	**0.04**	**K**	**0.08**	**Na**	**0.04**
Fe	**0.10**	**Mg**	**0.08**		

Determination of additional elements is possible if they are not volatilized during the evaporation step of the sample preparation.

References

ASTM Method D1193, "Specification for Reagent Water," 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 slowly evaporated with nitric acid on a hot plate to reduce the volatility of the metals. The residue is diluted with hydrochloric acid and an internal standard is added. The concentrations of elements in the resulting solution are determined by ICP-OES.

Apparatus

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

Balance, laboratory, readable to 0.01 g

Beaker, quartz, low form, 250-mL, Ace Glass, Inc., Cat. No. 5334-14

Bottle, wash, 500 mL, VWR, Cat. No. 2402-0500, for deionized water

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).

Cylinder, graduated, Class B, 10- and 100-mL, VWR, Cat. No. 89000-264 and -270, respectively

Flasks, volumetric, Class A, 10-, 50-, and 1000-mL, VWR, Cat. Nos. 89000-398, -402, and -412, respectively

Flasks, volumetric, polypropylene, 100- and 1000-mL, VWR, Cat. Nos. 29615-007, and -062, respectively

Hot plate, variable heat, with temperature display, VWR, Cat. No. 12365-474

Pipets, volumetric, Class A, 1-, 2-, 5-, 10-, and 100-mL, VWR, Cat. Nos. 89003-340, -342, -348, -350, and -368, respectively

Pipet filler, VWR, Cat. No. 53497-053

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

Spectrometer, ICP-OES, computer controlled, having sufficient resolving power and dispersion to separate the analytical lines in the 160 to 800 nm region. The data system shall be capable of performing background corrections, blank corrections, mass/volume corrections 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 8300 DV or equivalent. The use of an autosampler is recommended for efficiency

Tongs, beaker, VWR, Cat. No. 82027-374

Watch glasses, quartz, ribbed, 75-mm diameter, Wilmad-LabGlass, Cat. No. C-9990-75

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

Centrifuge tubes, 15 mL, polypropylene, Fisher Scientific, Cat. No. 05-538-51

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

Gloves, neoprene/natural rubber, VWR, Cat. No. 32917-206 (for size large)

Hydrochloric acid, concentrated, trace metals grade, VWR, Cat. No. EM-HX0608-2

Hydrochloric acid, 10%, To prepare 1000 mL, using a graduated cylinder, add 100 mL of the concentrated hydrochloric acid, into a 1000-mL volumetric flask. Fill to the mark with deionized water. Cap and invert several times to mix. The solution should remain stable for one month.

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

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

Scandium metal solution, aqueous, 1000-µg/mL, SPEX Industries, Inc., Cat. No. PLSC-2, for use as an internal standard

Scandium metal solution, aqueous, 10-µg/mL. To prepare 100 mL, pipet 1.0 mL of the 1000-µg/mL scandium metal solution into a 100-mL volumetric flask. Fill to the mark with 10% hydrochloric acid. Cap and invert several times to mix. The solution should remain stable for one month.