



## Impurities in Catalysts by ICP-OES

### UOP Method 303-05

#### Scope

This method is for determining trace metals in fresh, regenerated and spent alumina-, silica- or aluminosilicate- (silica/alumina-) based catalysts by Inductively Coupled Plasma - Optical Emission Spectroscopy (ICP-OES). Ordinarily, the metals determined include: calcium (Ca), chromium (Cr), cobalt (Co), copper (Cu), iron (Fe), lead (Pb), magnesium (Mg), manganese (Mn), molybdenum (Mo), nickel (Ni), silicon (Si), sodium (Na), tin (Sn), titanium (Ti), vanadium (V), and zinc (Zn). Silicon is not determined when it is a component of the catalyst base. Arsenic (As) and phosphorous (P) can be determined but are not ordinarily present on fresh catalysts. They can, however, be present on regenerated or spent catalyst. Barium (Ba) and mercury (Hg) cannot be determined because barium precipitates and mercury volatilizes. Mercury can be determined by UOP Method 894, "Mercury in Catalysts by GF-AAS." Other elements also can be determined, if required. The specified elements can be determined within the concentration ranges listed in Table 1. Typical concentration ranges are 0.005-1.0 mass-%.

This method is not applicable to noble metals and modifiers on catalysts. Noble metals and modifiers can be determined by UOP Method 873, "Noble Metals and Modifiers in Catalysts by ICP-AES." This method cannot be used for determining metals in alpha-alumina based catalyst because alpha-alumina is not solubilized under the conditions of this procedure.

An alternative microwave-based dissolution technique is described in the *Appendix*.

#### References

- ASTM Method D 4307, "Preparation of Liquid Blends for Use as Analytical Standards," [www.astm.org](http://www.astm.org)
- UOP Method 873, "Noble Metals and Modifiers in Catalysts by ICP-AES," [www.astm.org](http://www.astm.org)
- UOP Method 879, "Aluminum in Catalysts by Atomic Absorption," [www.astm.org](http://www.astm.org)
- UOP Method 894, "Mercury in Catalysts by GF-AAS," [www.astm.org](http://www.astm.org)
- UOP Method 897, "Metal Impurities in Molecular Sieves by ICP-AES," [www.astm.org](http://www.astm.org)
- UOP Method 926, "Silicon and Aluminum in Fresh Catalysts by Lithium Metaborate Fusion/ICP-AES," [www.astm.org](http://www.astm.org)
- UOP Method 954, "Loss on Ignition (LOI) for Fresh, Regenerated, Used, and Spent Catalysts,"

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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Catalyst Supports, and Adsorbents,” [www.astm.org](http://www.astm.org)

UOP Method 999, “Precision Statements in UOP Methods,” [www.astm.org](http://www.astm.org)

## Outline of Method

The catalyst samples are digested in acids. Scandium is added to the digests as an internal standard. Hydrofluoric acid is used to remove silica from silica- or silica/alumina-based catalyst digests by volatilization. The digests are then aspirated into a polychromatic ICP-OES along with appropriate matrix-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, 250-mL, Fisher Scientific, Cat. No. 02-593-5B

*Bottle*, wash, for water, Fisher Scientific, Cat. No. 04-409-20E

*Catalyst grinding equipment* (optional, see *Sample Preparation*)

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

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

*Mixing mill*, shaker type, SPEX Industries, Inc., Cat. No. 2000

*Cylinders*, graduated, polypropylene, 10- and 100-mL, Fisher Scientific, Cat. Nos. 08-570-21A and -21D, respectively

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

*Hot plate*, variable heat to at least 150°C, Fisher Scientific, Cat. No. 11-600-49H

*Microliter pipet*, 2500- $\mu$ L capacity, Rainin Instrument Co., Cat. No. EP-2500

*Microliter pipet*, 10-mL capacity, Rainin Instrument Co., Cat. No. EP-10ML

*Pipettes*, volumetric, transfer, Class A, 5-, 10-, 15-, 20-, 25- and 50-mL, Fisher Scientific, Cat. Nos. 13-650-2F, -2L, 2M, 2N, 2P, and -2S, respectively

*Regulator*, argon, two-stage, high purity, delivery pressure range 30-700 kPa (4-100 psi), Matheson Tri-Gas, Model 3122-530

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

*Spectrometer*, polychromatic, Inductively Coupled Plasma-Optical Emission Spectrometer, having sufficient resolving power and dispersion to separate the analytical lines in the 160- to 800-nm region. The instrument should be capable of performing background corrections, blank corrections, and weight/volume and dilution calculations. A commercial grating spectrometer with a band pass of 0.018 nm or less in the first order is satisfactory. PerkinElmer, Optima 4300.

*Thermometer*, digital, capable of measuring to at least 150°C, to measure hot plate temperature, Fisher Scientific, Cat. No. 15-077-9E

*Watch glass*, Teflon, 75-mm diameter, Thomas Scientific, Cat. No. 9788C20

## 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 Type I reagent grade water.

*Argon*, 99.995% minimum purity, ICP feed gas

*Droppers*, disposable, polypropylene, Fisher Scientific, Cat. No. 13-711-10

*Gloves*, neoprene/natural rubber, Fisher Scientific, Cat. No. 11-394-114

*Hydrochloric acid*, concentrated, Certified ACS Plus, Fisher Scientific, Cat. No. A144-212

*Hydrofluoric acid*, concentrated, ACS Reagent, J. T. Baker, Mallinckrodt Baker, Cat. No. 9550

*Hydrogen peroxide*, 30%, Certified ACS, Fisher Scientific, Cat. No. H325-500

*Nitric acid*, concentrated, Certified ACS Plus, Fisher Scientific, Cat. No. A200-212

*Nitrogen or Argon*, 99.99% minimum purity, ICP optics purge gas

*Single element standard*, Aluminum (Al), 10,000 µg/mL, SPEX Industries, Inc., Cat. No. PLAL1-3X, used for matrix matching

*Single element standard*, Scandium (Sc), 1000 µg/mL, SPEX Industries, Inc., Cat. No. PLSC-2, internal standard

*Single element standards*, 1000 µg/mL each, As, Ca, Co, Cr, Cu, Fe, Mg, Mn, Mo, Na, Ni, P, Pb, Si, Sn, Ti, V, and Zn, SPEX Industries, Inc., Cat. Nos. PLAS2-2X, PLCA1-2X, PLCO2-2X, PLCR1-2X, PLCU2-2X, PLFE2-2X, PLMG2-2X, PLMN2-2X, PLMO9-2X, PLNA2-2X, PLNI2-2X, PLP9-2X, PLPB2-2X, PLSI9-2X, PLMN5-2X, PLTI9-2X, PLV2-2X, and PLZN1-2X, respectively

*Sulfuric acid*, concentrated, Certified ACS Plus, Fisher Scientific, Cat. No. A300-212

*Tips*, disposable, for EP-2500 microliter pipet, Rainin Instrument Co., Cat. No. RC-2500

*Tips*, disposable, for EP-10ML microliter pipet, Rainin Instrument Co., Cat. No. RC-10ML

*Water*, ASTM Type I reagent grade water

## Procedure

**Caution:** All subsequent steps involving additions of acids and heating of samples and digests must be performed in a properly operating fume hood, using appropriate precautions. Gloves, safety glasses and/or face shield, and other appropriate personal protective equipment must be worn. See the material safety data sheet (MSDS) for each material used.

### Preparation of Standards

To ensure accurate blending, use the techniques described in ASTM Method D 4307, "Preparation of Liquid Blends for Use as Analytical Standards." It has been found convenient to use volumetric pipets in the preparation of the Stock Solutions, and to use microliter pipets thereafter, although volumetric pipets may be used throughout except where hydrofluoric acid (HF) is present. A few Single Element Standards contain a trace of HF and will be so labeled. For these, use only microliter pipets with disposable tips.

Prepare four Stock Solutions from the Single Element Standards as follows: