

METAL & METALLOID PARTICULATES IN WORKPLACE ATMOSPHERES  
(ATOMIC ABSORPTION)



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Method Number:	ID-121
Matrix:	Air, Wipes, or Bulks
OSHA Permissible Exposure Limits:	See Table 1
Collection Procedure:	Personal air samples are collected on mixed-cellulose ester filters using a calibrated sampling pump. Wipe or bulk samples are collected using grab sampling techniques.
Recommended Sampling Rate:	2 L/min
Recommended Air Volumes	
Time Weighted Average Samples:	480 to 960 L
Short-Term Exposure Limit Samples:	30 L
Ceiling Samples:	10 L*
Analytical Procedure:	Samples are desorbed or digested using water extractions or mineral acid digestions. Elemental analysis of the prepared sample solutions is performed by atomic absorption or emission spectroscopy.
Detection Limits:	See Table 2
Precision and Accuracy:	See Table 3
Method Classification:	Validated Analytical Method
Date:	1985
Date Revised:	February 2002

\* Alternate air volumes may be necessary to achieve good analytical sensitivity.

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Commercial manufacturers and products mentioned in this method are for descriptive use only and do not constitute endorsements by USDOL-OSHA. Similar products from other sources can be substituted.

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## 1. Introduction

This method can determine the amount of specific metal and metalloid particulates in the workplace atmosphere. The airborne particulates are collected on filters using calibrated sampling pumps. These samples are then analyzed using flame atomic absorption or emission spectrometry. This method can also determine specific metals and metalloids contained in wipe and bulk samples. The identification and quantification of the particulate is directly determined as the element. The elements are:

Aluminum (Al)	Silver (Ag)	Lithium (Li)	Cobalt (Co)
Gold (Au)	Bismuth (Bi)	Thallium (Tl)	Molybdenum (Mo)
Potassium (K)	Iron (Fe)	Cesium (Cs)	Yttrium (Y)
Antimony (Sb)	Sodium (Na)	Magnesium (Mg)	Copper (Cu)
Hafnium (Hf)	Cadmium (Cd)	Tin (Sn)	Nickel (Ni)
Selenium (Se)	Lead (Pb)	Chromium (Cr)	Zinc (Zn)
Barium (Ba)	Tellurium (Te)	Manganese (Mn)	Platinum (Pt)
Indium (In)	Calcium (Ca)	Titanium (Ti)	Zirconium (Zr)

For some analytes, there are alternate methods or procedures which may be more sensitive, accurate, or specific. When a separate OSHA method or procedure exists, that method shall take precedence over this method unless special circumstances render it inapplicable. Elements or compounds having alternate methods or stopgap procedures are:

<u>Element or Compound</u>	<u>OSHA Method No.</u>
Aluminum oxide	ID-198SG or ID-109SG
Barium sulfate	ID-204
Cadmium	ID-189
Chromic acid/chromates	ID-103
Ferrovandium	ID-125G
Vapors (i.e. Ni(CO) <sub>4</sub> , H <sub>2</sub> Se, TeF <sub>6</sub> , C <sub>5</sub> H <sub>4</sub> Mn(CO) <sub>3</sub> ]	In-House Methods
Organic tin compounds	ID-102SG
Platinum (soluble)	ID-130SG
Selenium	ID-133SG
Solders	ID-206
Stibine	NIOSH 6008, In House
Tetraethyl lead and tetramethyl lead	In-House Method
Titanium dioxide	ID-204
Welding fumes	ID-125G
Zinc oxide	ID-143

Depending on advances in technology or changes in exposure limits, substances may be added or deleted from the above lists.

### 1.1 History

Air and wipe samples containing metal and metalloid particulate have always been analyzed at the OSHA Salt Lake City Analytical Laboratory using atomic absorption or emission spectrometry (8.1). Constituents in bulk samples have been determined semi-quantitatively using this technique.

### 1.2 Principle

Air samples of the workplace are taken using calibrated sampling pumps with cassettes containing either mixed cellulose ester (MCE) or polyvinyl chloride (PVC) filters. These samples are prepared in the laboratory using concentrated (concd) acids or extracted with deionized water if a soluble fraction is required. The sample solution is diluted to a known volume after any necessary matrix modifiers are added. The sample is then aspirated into the flame of an atomic absorption or emission spectrophotometer (AAS or AES) and the molecules in the sample solution are subjected to the following processes:

- 1) nebulization
- 2) desolvation
- 3) liquefaction
- 4) vaporization
- 5) atomization
- 6) excitation (atoms converted from "ground" to excited state)
- 7) ionization

The absorption or emission of light occurring during processes 5 and 6 is then measured at the characteristic wavelength for the element of interest.

For absorption, a hollow cathode lamp or an electrodeless discharge lamp (EDL) is used as the light source. A double beam spectrophotometer is normally used where the lamp radiation alternately passes through and around a flame into which the sample is being aspirated. The sample is atomized and the metal or metalloid atoms absorb light from the source at their characteristic wavelengths. This absorption is proportional to the concentration of the element present in the sample solution. A monochromator isolates the characteristic radiation of the element being analyzed. A photosensitive device then measures the intensity of the transmitted radiation from the two light paths to determine the amount of absorbance occurring in the flame.

For emission, a light source is not used. The sample is introduced into the flame, atomized and excited, and then the light emission from excitation is isolated and measured. The intensity of the light emitted is proportional to the concentration of the element present.

The following flames are used in this method for absorption or emission:

- a) Air/Acetylene mixture (Air/C<sub>2</sub>H<sub>2</sub>)
- b) Nitrous oxide/Acetylene mixture (N<sub>2</sub>O/C<sub>2</sub>H<sub>2</sub>)
- c) Air/Hydrogen mixture (Air/H<sub>2</sub>)

The use of a specific flame is dependent on the respective element's analytical stability, sensitivity, and interferences.

### 1.3 Advantages and Disadvantages

- 1.3.1 This analytical method is specific for the element to be determined and does not distinguish different compounds. When an analysis for a compound is requested, an elemental analysis is performed on the sample. A gravimetric factor is then applied to calculate the compound value (Note: For some compounds, additional analytical procedures (i.e. ion chromatography or X-ray diffraction) can be used to confirm the presence of the particular compound).
- 1.3.2 The analysis will also not differentiate between different particle size ranges, such as dusts and fumes.
- 1.3.3 Metallic analytes having Permissible Exposure Limits (PELs) designated as the soluble form (i.e., iron soluble salts, nickel, etc.) can be analyzed using this method. Samples for soluble analytes are extracted with deionized water and an elemental analysis is performed on the extract.
- 1.3.4 Some compounds may not dissolve using the digestion procedures described herein. In these cases, an alternative digestion method should be used.
- 1.3.5 Several elements can be determined from the same filter sample using this method; however, digestion procedures may solubilize only certain metals. If a combination of metals is requested on the same filter, all of the metals must be soluble in the digestion procedure used.

1.3.6 The equipment used is inexpensive and does not require specialized training.

#### 1.4 Use of Metal and Metalloid Compounds in Industry

Metals, their alloys, and compounds are used in a wide variety of industries. In certain operations (e.g., welding, smelting, grinding, etc.), particulate matter containing metals and their compounds may be released into the workplace atmosphere. These substances pose a potential health hazard to workers exposed to them (8.2-8.4). Further documentation regarding industrial use, toxicity, and physical properties may be found in NIOSH criteria documents for the particular substance.

### 2. Analytical Range and Sensitivity

This method uses detection limit, linearity, and sensitivity terms which are characteristic of atomic absorption. These terms are further defined in Appendix A. Any detection limits, linear ranges, and sensitivities mentioned in this method are for analyses using the primary analytical wavelength, a flow spoiler, an Air/C<sub>2</sub>H<sub>2</sub> flame, and a hollow cathode lamp unless otherwise noted.

2.1 The qualitative detection limits listed in Table 2 were taken from reference 8.5. The analytical detection limits (8.1) listed were determined from routine laboratory analyses using the definition listed in Appendix A. These limits are approximate since they are dependent on instrument performance and optimization, sample characteristics, and the range of standards analyzed.

2.2 The upper linear range for each element is also given in Table 2. These ranges were taken from reference 8.6. Instrument response is linear to greater concentrations if an alternate wavelength is used; however, the detection limit may also increase. Samples can be diluted to bring the concentration of the element(s) within the linear range. The upper linear range for most elements is usually found near 0.25 to 0.30 absorbance units (ABS).

2.3 The sensitivity for each element is also listed in Table 2. These values are for a nebulizer which has been optimized to give an ABS of 0.25 for an aqueous solution containing 5 µg/mL Cu (8.6). The actual sensitivity obtained will depend on the particular instrument and flame used, the sample matrix, and instrument operating parameters.

### 3. Method Performance - Precision and Recoveries

Listed in Table 3 are data compiled from quality control (QC) samples which were spiked with aqueous solutions of various analytes and then analyzed in single blind tests. Each analyte was spiked onto an individual MCE filter, allowed to dry, and then prepared and analyzed along with survey samples previously taken by industrial hygienists. These samples were analyzed from 1986 to 1989. Due to the limited number of survey samples received for a few substances, QC samples were not prepared and analyzed for all analytes included in this method.

### 4. Interferences

Interferences occur at the analytical level and can be characterized as chemical, matrix, ionization, spectral, or as background absorption.

4.1 Chemical or condensed phase interferences occur when the element of interest combines with another species in the flame, thus altering the number of atoms available for emission or absorption. This can result in either a positive or negative bias (usually negative) in the results obtained. Chemical interferences can be controlled by using a hotter flame, or by the addition of a releasing agent which inhibits the reaction between the metal and the interfering species.

4.2 Matrix interferences occur when the physical characteristics (viscosity, surface tension, etc.) of the sample and standard solutions differ considerably. This may occur when samples contain large amounts of dissolved salts or acid, when different solvents are used for samples and standards, or when the temperatures of samples and standards are appreciably different. To control this, samples

and standards must be matrix matched, or the sample must be diluted until any matrix effect becomes insignificant.

- 4.3 Ionization interferences occur when the flame temperature is sufficiently high to ionize the atoms of interest. This changes the absorption spectrum of the analyte and effectively removes atoms from the flame, causing a loss of sensitivity. Ionization interferences are controlled by adding large amounts (usually >0.1%) of an easily ionized metal such as Na, K, Cs, or rubidium (Rb). The excess electrons released in the flame greatly reduces the degree of ionization of the metal being determined.
- 4.4 Spectral interferences occur when an element other than the one analyzed absorbs at the same wavelength. This causes a positive bias in the results obtained when the interfering element is present in the samples. In this case, an alternate line should be used. Spectral interferences also occur when a multi-element hollow cathode lamp is used which contains elements with absorbing wavelengths close to one another and the analytical slit width used is wide enough to allow the wavelengths of more than one element to pass. If the sample contains two or more of these elements, a positive bias will occur. To resolve this, a single element lamp, an alternate wavelength, or in certain cases, a narrower slit width can be used.
- 4.5 Background absorption interferences include flame absorption, molecular absorption, and light scattering:
  - a) Flame absorption is most severe below 250 nm. This absorption can be controlled by careful optimization of fuel and oxidant flow rates. Other mechanisms of control are: Use of flames which are more transparent at these wavelengths (i.e. Air/H<sub>2</sub> or argon/hydrogen flames), or deuterium arc background correction (DABC).
  - b) Molecular absorption is controlled by using hotter flames to break down molecular species or by DABC.
  - c) Light scattering occurs at shorter wavelengths when samples have a large salt content; this is controlled using DABC.
- 4.6 Large amounts of silicates or other particulates may interfere and may also cause aspiration problems (8.7). If present, they should be removed by filtration. The particulate should then be re-digested and analyzed to ensure the analyte(s) of interest have been completely extracted.
- 4.7 This analytical method is normally not compound-specific. Compounds are only determined as the element, and a significant positive bias can occur when any sample has additional analytes containing the same element. Other analytical procedures may be necessary to identify a specific compound. An assessment of the industrial operation sampled may also provide information regarding the potential existence of other analytes that could cause a positive bias.
- 4.8 Potential interferences for several of the elements determined by this method are listed in Appendix B.

## 5. Sampling

### 5.1 Equipment - Air Filter Samples

- 5.1.1 Mixed cellulose ester (MCE) filters (0.8 µm pore size), cellulose backup pads, and cassettes, 37-mm diameter (part no. MAWP 037 A0, Millipore Corp., Bedford, MA). Filters and cassettes having a 25-mm diameter can also be used.
- 5.1.2 Gel bands (Omega Specialty Instrument Co., Chelmsford, MA) for sealing cassettes.
- 5.1.3 Sampling pumps capable of sampling at 2 liters per minute (L/min).

- 5.1.4 Assorted flexible tubing.
- 5.1.5 Stopwatch and bubble tube or meter for pump calibration.

5.2 Equipment - Wipe Samples

- 5.2.1 Smear tabs (part no. 225-24, SKC Inc., Eighty Four, PA), or wipe filters (Whatman no. 41 or no. 42 filters, Whatman Labsales Inc., Hillsboro, OR).
- 5.2.2 Deionized water.
- 5.2.3 Scintillation vials, 20-mL (part no. 74515 or 58515, Kimble, Div. of Owens-Illinois Inc., Toledo, OH) with polypropylene or Teflon cap liners. Metal cap liners should not be used.

5.3 Equipment - Bulk Samples

- 5.3.1 High-volume sampling pump with appropriate sized MCE collection filters.
- 5.3.2 Scintillation vials, 20-mL (same as Section 5.2.3).

5.4 Sampling Procedure - Air Filter Samples

- 5.4.1 Place a MCE filter and a cellulose backup pad in each two- or three-piece cassette. Seal each cassette with a gel band.
- 5.4.2 Calibrate each personal sampling pump with a prepared cassette in-line to approximately 2 L/min.
- 5.4.3 Attach prepared cassettes to calibrated sampling pumps (the backup pad should face the pump) and place in appropriate positions on the employee or workplace area.
- 5.4.4 Collect the samples at approximately 2 L/min for the recommended sampling times (unless otherwise noted):

TWA	240 to 480 min
STEL Samples	15 min
Ceiling Samples	5 min*

The analytical sensitivity of a specific analyte may dictate the use of a different sampling time.

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\* When determining compliance with the Ceiling PEL for sodium hydroxide, take 15-min samples.

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- 5.4.5 Place plastic end caps on each cassette after sampling. Attach an OSHA-21 seal around each cassette in such a way as to secure the end caps.

5.5 Sampling Procedure - Wipe Samples

Certain analytes may have a skin designation (See Table 1).

- 5.5.1 Wear clean, impervious, disposable gloves when taking each wipe sample.
- 5.5.2 Moisten the wipe filters with deionized water prior to use.
- 5.5.3 If possible, wipe a surface area covering 100 cm<sup>2</sup>.
- 5.5.4 Fold the wipe sample with the exposed side in.

- 5.5.5 Transfer the wipe sample into a 20-mL scintillation vial and seal with vinyl or electrical tape. Securely wrap an OSHA-21 seal length-wise from vial top to bottom.

## 5.6 Sampling Procedure - Bulk Samples

- 5.6.1 In order of laboratory preference, bulk samples may be one of the following:

- 1) a high-volume (>1,000 L) filter sample of the workplace area,
- 2) a representative settled dust (rafter) sample,
- 3) a sample of the bulk material in the workplace.

- 5.6.2 If possible, transfer the bulk material or filter into a 20-mL scintillation vial and seal with vinyl or electrical tape. Securely wrap an OSHA-21 seal length-wise from vial top to bottom.

## 5.7 Shipment

- 5.7.1 Submit at least one blank sample with each set of air or wipe samples. Blank filter samples should be handled in the same manner as other samples, except that an air or wipe sample is not taken.
- 5.7.2 The type of bulk sample should be stated on the OSHA 91A and cross-referenced to the appropriate air sample(s). Bulk samples should be shipped with Material Safety Data Sheets (if available) and should be sent separately from air samples. Check current mailing restrictions and ship bulks to the laboratory by an appropriate method.
- 5.7.3 Send all samples to the laboratory with the OSHA 91A paperwork requesting the specific analyte(s) of interest. If analysis of a mixture of different elements or compounds is necessary, contact the lab to ascertain which analytes can be analyzed together.

## 6. Analysis

### 6.1 Safety Precautions

- 6.1.1 Care should be exercised when handling any acidic solutions. Acid solution contact with work surfaces should be avoided. If any acid contacts the eyes, skin, or clothes, flush the area immediately with copious amounts of water. Medical treatment may be necessary.
- 6.1.2 All work with concentrated acids is potentially hazardous. Always wear safety glasses and protective clothing. Prepare all mixtures, samples, or dilutions in an exhaust hood. To avoid exposure to acid vapors, do not remove any beakers from the hoods until they have returned to room temperature.
- 6.1.3 **Extra care** should be used when handling perchloric acid ( $\text{HClO}_4$ ). Perchloric acid should only be used in a hood that has been approved for  $\text{HClO}_4$  use. In this hood:
- a) Organic reagents should not be used or stored near  $\text{HClO}_4$ .
  - b) A water wash down system for the ducts and work surface must be installed and periodically used.
  - c) Precautions should be taken to ensure that explosions or spontaneous ignition of sample material from  $\text{HClO}_4$  is prevented.

Working with  $\text{HClO}_4$  is very hazardous. Be sure to wear safety glasses, a laboratory coat, and gloves. Always add nitric acid ( $\text{HNO}_3$ ) with  $\text{HClO}_4$ . When digesting backup pads or other samples with  $\text{HClO}_4$ , watch them carefully since there is a chance they

could ignite. Always keep  $\text{HNO}_3$  nearby when using  $\text{HClO}_4$ . In the event of sample media ignition, quickly douse the sample with a small portion of  $\text{HNO}_3$ .

- 6.1.4 Care should be exercised when using laboratory glassware. Chipped pipettes, volumetric flasks, beakers, or any glassware with sharp edges exposed should not be used.
- 6.1.5 Pipetting is always performed using an automatic pipet or pipette bulb, never by mouth.
- 6.1.6 Before using any instrument, the operator should consult the Standard Operating Procedure (SOP) (8.8) and any instrument manuals.
- 6.1.7 Since metallic elements and other toxic substances are vaporized during flame operation, it is imperative that an exhaust hood is installed and used directly above the burner chamber of the spectrometer. Always ensure the exhaust system is operating before proceeding with the analysis.

## 6.2 Equipment

### 6.2.1 Atomic absorption spectrophotometer consisting of a(an):

Nebulizer and burner head.

Pressure-regulating devices capable of maintaining constant oxidant and fuel pressures.

Optical system capable of isolating the desired wavelength of radiation.

Adjustable slit.

Light measuring and amplifying device.

Display, strip chart, or computer interface for indicating the amount of absorbed or emitted radiation.

Deuterium Arc Background Corrector. This is usually required for determinations at short (<250 nm) wavelengths.

Light source for absorption:

- a) Hollow cathode lamp for the specific element or multielement (Note: Please see specific limitations of multielement lamps in Appendix B)
- b) Electrodeless Discharge Lamp (EDL) for the specific element. This type of lamp may provide better sensitivity and detection limits for some elements, especially Se, Sn, and Sb. If used, a separate EDL power supply is usually necessary.

6.2.2 Oxidant: Compressed, filtered air free from water, oils and other contaminants.

6.2.3 Nitrous oxide ( $\text{N}_2\text{O}$ ).

6.2.4 Fuel (Use flash arrestors when using flammable gases. Consult with the manufacturer for appropriate use.):

- a) Acetylene, commercially available acetylene dissolved in acetone. CAUTION: Do not use grades of acetylene that contain solvents other than acetone. These solvents may damage PVC tubing in some instruments. Do not use acetylene when the tank pressure drops below 520 kPa (75 psi).
- b) Hydrogen is used as the fuel in the determination of certain elements.

6.2.5 Pressure regulators, Two-stage.

6.2.6 Flash arrestors (model 6103, Matheson Gas Products, East Rutherford, NJ).

6.2.7 Glassware



- a) Conical beakers, 125- and 250-mL
  - b) Volumetric flasks, Class A: 10-, 25-, 50- and 100-mL
  - c) Pipettes, Class A: Assorted sizes
- 6.2.8 Forceps.
- 6.2.9 Exhaust hood and hotplate, or microwave digestion system (model no. MDS-81, CEM Corp., Matthews, NC).
- 6.2.10 Filtering apparatus consisting of MCE filters, 0.45- $\mu$ m pore size, 47-mm diameter (cat. no. HAWP 047 00, Millipore Corp., Bedford, MA) and filtering apparatus (cat. no. XX15 047 00, Millipore).
- 6.2.11 Analytical balance (0.01 mg).
- 6.3 Reagents (All chemicals should be reagent grade or better. Many of the chemicals listed below are only used in specific instances. Specific reagents are listed within the additional procedures in Table 4 and also in Table 5.)
- 6.3.1 Deionized water (DI H<sub>2</sub>O) with a specific conductance of less than 10  $\mu$ S.
  - 6.3.2 Ammonium fluoride (NH<sub>4</sub>F) solutions (used for specific insoluble compounds, see AP 6, Table 4).
    - a) Ammonium fluoride, 1 M: Dissolve 37.04 g NH<sub>4</sub>F and dilute to 1 L in DI H<sub>2</sub>O. Store in a polyethylene bottle.
    - b) Ammonium fluoride, 0.1 M in 4% HNO<sub>3</sub>: Carefully add 40 mL concd HNO<sub>3</sub> and 100 mL of the 1 M NH<sub>4</sub>F solution to 500 mL DI H<sub>2</sub>O and dilute to 1 L in a polyethylene volumetric flask. Store in a polyethylene bottle since acidic solutions of NH<sub>4</sub>F may form small amounts of HF and etch glass containers.
  - 6.3.3 Hydrogen peroxide (H<sub>2</sub>O<sub>2</sub>), 30% (used for digestions of Cr, see AP 5, Table 4).
  - 6.3.4 Mineral acids (used for digestions) CAUTION: Refer to Section 6.1.2 before using acids.
    - a) Hydrochloric acid (HCl), concd (36.5 to 38%).
    - b) Hydrofluoric acid (HF), concd (49%).
    - c) Nitric acid (HNO<sub>3</sub>), concd (69 to 71%).
    - d) Perchloric acid (HClO<sub>4</sub>), concd (69 to 72%). Please see Section 6.1.3 before using HClO<sub>4</sub>.
    - e) Sulfuric acid (H<sub>2</sub>SO<sub>4</sub>), concd (95 to 98%).
    - f) Acid mixture for platinum digestions: Prepare a mixture of HCl/HNO<sub>3</sub> by slowly and carefully adding 82 mL concd HCl to 18 mL concd HNO<sub>3</sub> (CAUTION: Do not store this solution; dispose of properly after use).
  - 6.3.5 Mineral acids (used for dilutions or cleaning glassware) CAUTION: Refer to Section 6.1.2 before using acids.

- a) Nitric acid, 1:1 HNO<sub>3</sub>/DI H<sub>2</sub>O mixture: Carefully add a measured volume of concd HNO<sub>3</sub> to an equal volume of DI H<sub>2</sub>O.
- b) Nitric acid, 4% v/v: Carefully add 40 mL concd HNO<sub>3</sub> to 500 mL DI H<sub>2</sub>O and dilute to 1 L.
- c) Nitric acid 10% v/v: Carefully add 100 mL of concd HNO<sub>3</sub> to 500 mL of DI H<sub>2</sub>O and then dilute to 1 L.
- d) Nitric and hydrochloric acid v/v mixture (4% HNO<sub>3</sub>/X% HCl, where X% is listed below): Carefully add the appropriate amount of concd HCl to 500 mL of DI H<sub>2</sub>O:

4% HCl 40 mL  
16% HCl 160 mL  
32% HCl 320 mL

Then carefully add 40 mL concd HNO<sub>3</sub> and dilute to 1 L with DI H<sub>2</sub>O.

#### 6.3.6 Chemical or ionization interference suppressants

- a) Aluminum ion, 5,000 µg/mL: Dissolve 69.52 g aluminum nitrate (Al(NO<sub>3</sub>)<sub>3</sub>•9H<sub>2</sub>O) and dilute to 1 L in DI H<sub>2</sub>O.
- b) Potassium ion, 5,000 µg/mL: Dissolve 9.54 g potassium chloride (KCl) in DI H<sub>2</sub>O and dilute to 1 L.
- c) Sodium ion, 5,000 µg/mL: Dissolve 12.71 g sodium chloride (NaCl) in DI H<sub>2</sub>O and dilute to 1 L.

#### 6.3.7 Stock standard solutions

Commercially available aqueous standards are used. Expiration dates for standards should be followed. If there is no expiration date, dispose of after 1 year. As an alternative, standards can be prepared using the procedures described in the SOP (8.8) or instrument manufacturer manuals (i.e., 8.6, 8.9, 8.10).

### 6.4 Glassware Preparation

- 6.4.1 Place the conical beakers in an exhaust hood and add approximately 10 mL of a 1:1 HNO<sub>3</sub>/DI H<sub>2</sub>O mixture in each 125- or 250-mL conical beaker. Apply moderate heat until refluxing occurs. Decant the acid mixture into a waste container and allow the beakers to cool before removing from the hood. Rinse the beakers thoroughly with DI H<sub>2</sub>O.
- 6.4.2 Rinse all volumetric flasks with 10% v/v HNO<sub>3</sub> and then rinse thoroughly with DI H<sub>2</sub>O.

### 6.5 Working Standards

- 6.5.1 Dilute stock standard solutions to the appropriate ranges using a diluent that will match the sample matrix. Use information in Tables 1 and 2 as guides for the ranges; use Table 5 for matrices. The standard concentrations should bracket the expected sample concentrations and the standard/sample matrices should match.
- 6.5.2 Store standards in appropriate containers. Protect Ag standards from light by storing them in actinic or brown plastic bottles. Store standards containing NH<sub>4</sub>F in polyethylene containers.

## 6.6 Sample Preparation

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Note: Always prepare blank samples with every sample set. Prepare an additional blank media sample any time an extra procedure is used (i.e., wiping out the particulate contained inside a cassette with an MCE filter or preparing a contaminated backup pad). This blank media should be from the same manufactured lot as the prepared filter or backup pad.

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### 6.6.1 Preparation of air and wipe samples

Use 125-mL conical beakers for air samples and smear tabs; use 250-mL beakers for large wipe samples. Carefully transfer any loose dust from the cassette into a labeled beaker. Using forceps transfer the sample filter into the same digestion beaker. If the backup pad appears contaminated, include it with the sample filter. If there is loose dust present, rinse the cassette top (and ring, if present) with a small amount of DI H<sub>2</sub>O and pour the water into the beaker with the sample filter. Wipe out the cassette top (and ring, if present) interior surface with a clean Smear Tab (or 1×2 inch section of Ghost Wipe) that has been moistened with DI H<sub>2</sub>O and place it in the same digestion beaker with the rinse and sample filter. Similarly wipe out the cassette bottom interior surface if the cassette contains loose dust or if the backup pad is contaminated. Ensure that blank samples are prepared and analyzed using the same materials and procedures as used for air samples.

If the backup pad appears to be discolored, it may be due to leakage of air around the filter during sampling.

### 6.6.2 Preparation of bulk samples

Review any available material safety data sheets to determine safe bulk handling. The safety data may also offer a clue as to the aliquot amount needed for adequate detection of the element(s) of interest.

Measure by volume or weight an appropriate aliquot of any liquid bulk sample.

Weigh the appropriate amount of any solid bulk sample.

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Note: Aliquot amounts of bulks are dependent on the analytical sensitivity, detection limit, and solubility of the material used. If uncertain, a 20- to 50-mg aliquot of a solid material can be taken as a starting point. Make sure the aliquot taken is representative of the entire bulk sample. If necessary, use a mortar and pestle to grind any nonhomogenous particulate bulk samples in an exhaust hood.

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After measuring, transfer the aliquot to a 250-mL conical beaker.

### 6.6.3 Extraction or digestion - all samples

Consult Tables 4 and 5 to determine the reagents used during extraction or digestion for each element to be analyzed. Some elements (Ba, Sn, etc.) or compounds are not digested with concd HNO<sub>3</sub>, but are prepared using alternate procedures (APs) listed in Table 4. These elements or compounds and their AP numbers are:

Ag	AP 1	LiH	AP 7	Te	AP 1
Al (soluble)	AP 2	MgO	AP 3	TiO <sub>2</sub>	AP 8
Al (pyro powders)	AP 3	Na cmpds	AP 7	Tl (soluble)	AP 2
Au	AP 4	Ni (soluble)	AP 2	Y	AP 3
Ba (soluble)	AP 2	Mo (soluble)	AP 2	Zr	AP 6
Ca cmpds	AP 3	Mo (insoluble)	AP 3	Cr (II or III)	AP 2
Pb	AP 1	Cr (metal)	AP 5	Pt (metal)	AP 4
CsOH	AP 7	Sb	AP 1	Fe (soluble)	AP 2
Se	AP 1	Hf	AP 6	Sn (inorganic)	AP 4
KOH	AP 7	SnO	AP 4		

For the element or compounds listed above, follow the APs recommended and then proceed with Section 6.6.4. For other elements or compounds, follow the procedures a, b, or c listed below:

a) All MCE air filters and smear tabs requiring HNO<sub>3</sub> digestion

Place the beakers in an exhaust hood and add 3 to 5 mL concd HNO<sub>3</sub> to cover the filter. Place the beakers on a hot plate and heat the samples until about 1 mL remains. Add a second portion of approximately 1 to 2 mL of concd HNO<sub>3</sub>. Apply heat until the appropriate amount of HNO<sub>3</sub> remains in the beaker (1 mL of HNO<sub>3</sub> will give a 4% HNO<sub>3</sub> matrix when diluted to 25 mL final volume).

b) Large wipe, PVC filters, or backup pads

Place the beakers in an exhaust hood and add the following amount of concd HNO<sub>3</sub> to the beakers:

Large wipes and backup pads - 10 to 15 mL  
PVC filters - 3 to 5 mL

Place the beakers on a hot plate and heat the samples until about 1 mL remains. Add 2 mL of concd HClO<sub>4</sub> along with a second portion of 2 mL HNO<sub>3</sub>, heat the sample, and then remove when about 1 mL remains. (Note: Please see Section 6.1.3 before using HClO<sub>4</sub>.)

As an alternative, an extraction of the backup pad or wipe sample using only HNO<sub>3</sub> may be used. Add HNO<sub>3</sub> to the media, digest on a hotplate, and continue to add HNO<sub>3</sub> until the solution becomes clear. Remove the beaker from the hotplate when the appropriate amount of HNO<sub>3</sub> remains.

c) Bulk samples

Add 10 to 30 mL HNO<sub>3</sub>, place the beaker on a hot plate, and digest the bulk sample until the material dissolves and the appropriate amount of solution remains (about 1 mL if diluting samples to 25 mL, 2 mL if 50 mL final volume, etc. After dilution this will give a final volume of 4% HNO<sub>3</sub>). If necessary, use other acids, or use a microwave digestion system to facilitate digestion [For further information regarding microwave digestion, see the Standard Operating Procedure (8.11)].

#### 6.6.4 Filtration - all samples

1) Samples Previously Extracted:

Samples extracted with DI H<sub>2</sub>O should normally be filtered. If particulate is present, filter the extract through a 0.45-µm MCE filter. Save the extract as the soluble portion. If necessary, digest the particulate on both filters using procedure (a) above or the

applicable AP to prepare the remaining insoluble material for additional analyses. To control for potential contamination, prepare blank samples in the same fashion as the filtered samples.

2) Samples Previously Digested:

If particulate matter is present after digesting, cool the sample, add approximately 10 mL DI H<sub>2</sub>O, then filter the solution through a 0.45-µm MCE filter. Save the filtrate. Repeat digestion procedure (a) above for the filter containing the particulate.

6.6.5 Dilution - all samples

Allow all digested samples to cool to room temperature in an exhaust hood before proceeding. Additional sample or filtrate treatment may be required for certain elements. Perform any special sample treatments recommended in Table 5, and then quantitatively transfer each sample and each filtrate solution to individual volumetric flasks. Add any reagents necessary to achieve the final solution concentrations listed in Table 5 for specific analytes. Dilute to volume with DI H<sub>2</sub>O and then mix well. Solution volumes are dependent on the following factors:

- a) The amount of sample the industrial hygienist has collected (air volume and/or filter loading).
- b) The detection limit of the analytical method.
- c) The PEL of the analyte.
- d) The number of analytes requested.

Air samples are normally diluted to 25 mL unless one or more of the above factors suggests an alternate volume should be used. For routine analysis, at least 1/10 of the OSHA PEL should be detectable. Final solution volumes can be estimated using the following equation:

$$\text{FV Factor} = \frac{0.1 \times \text{PEL} \times \text{Air Vol}}{\text{QnDL} \times \text{GF}}$$

where:

PEL is Permissible Exposure Limit (mg/m<sup>3</sup>)

Air Vol is Air Volume taken (L)

QnDL = Quantitative Detection Limit (µg/mL)

GF = Gravimetric Factor (if required - some factors are listed in Table 6)

Quantitative detection limits are listed in Table 2. The FV factor assists in determining the final volume. Sample solution volumes normally used are: 5-, 10-, 25-, 50-, or 100-mL. Final volumes of 50- and 100-mL are normally reserved for wipe or bulk samples. If possible, FV should always be larger than the final solution volume. For example, if a sample has a 200-L air volume, a PEL of 0.05 mg/m<sup>3</sup>, a GF of 1, and a QnDL of 0.09 µg/mL, then:

$$\text{FV Factor} = 11.1$$

and a final volume should be 10-mL. Due to the limited amount of solution available for analysis and the potential for sample loss during transfer, 5-mL solution volumes are only used when absolutely necessary.

## 6.7 Instrument Setup and Analysis

- 6.7.1 Set up the AAS or AES according to the SOP (8.8) or the manufacturer's instructions. Use the flame and wavelength recommended in Table 7. If alternate conditions are necessary, consult the instrument manufacturer's manual for other settings and operating procedures. Install an EDL or hollow cathode lamp for the element of interest and allow it to warm up for 10-20 min or until the energy output stabilizes. Optimize conditions such as lamp position, burner head alignment, fuel and oxidant flow rates, etc. See the SOP (8.8) or specific instrument manuals for details.
- 6.7.2 Aspirate and measure the ABS of a standard solution for the element of interest. The standard concentration should be within the linear range for the element. Compare the ABS to an expected sensitivity value (Note: Some values are listed in Table 7; these were adapted from reference 8.6 or obtained at the OSHA laboratory). Then aspirate the smallest standard to be used and assure the ABS reading is above the background level of the instrument.
- 6.7.3 Make any adjustments necessary for the particular analysis, such as: scale expansion, burner head rotation, background correction, or alternate wavelength.
- 6.7.4 Aspirate and measure the ABS of a prepared standard solution, then determine the baseline by aspirating DI H<sub>2</sub>O and measuring the ABS.
- 6.7.5 Analyze standards, samples, and blanks. Repeat the baseline determination after each solution is analyzed. The baseline readings will assist in correcting any instrument drift. If more than one solution has been prepared for a sample (i.e. filtrate and sample, or soluble and insoluble portions), analyze each for all requested elements. Standards must bracket the sample concentrations. Analyze a standard after every four or five samples. Standard readings should be within 10 to 15% of the readings obtained at the beginning of the analysis.
- 6.7.6 If any samples exceed the linear range, they should be diluted. When diluting a sample, be sure that the diluted sample has the same matrix as the original sample and standards. If a number of samples must be diluted, it may be more advantageous to use a less sensitive wavelength.

## 6.8 Analytical Recommendations

- 6.8.1 When a fresh standard is prepared, analyze the old and new standards and compare results to verify the new standard is correct. If two or more stock solutions are available for working standard preparations, rotate the preparation from one stock solution to the next to verify the quality.
- 6.8.2 Keep a permanent record of all standard preparation and comparison data. Assign and follow expiration dates for all standards.
- 6.8.3 Always analyze blank samples along with the other samples. Treat blanks in the same fashion as samples, including any filtration steps.
- 6.8.4 When analyzing for Ag, carry-over from a large concentration sample or standard to the next sample can occur, causing erroneous readings. To remedy this, aspirate 4% HNO<sub>3</sub> instead of water between samples.
- 6.8.5 In this method, many different matrices are used to digest and keep analytes in solution. Occasionally, during multiple element analysis of the same sample, matrix effects can occur if standards are not matrix-matched with samples. Also, it is sometimes necessary to prepare samples in a matrix substantially different from recommendations. If these conditions occur, one or two standards should be prepared in the same matrix to determine

any matrix effects. A reagent blank should also be prepared and analyzed to determine any effect on the background signal. If a significant difference is noted in the analytical signals for the two different matrices, a full set of standards should always be prepared in the sample matrix and analyzed with the samples.

## 7. Calculations

7.1 Subtract each baseline ABS from the corresponding standard ABS, and plot the net ABS versus the standard concentrations. Using a least squares method, determine the equation for the best curve fit.

7.2 Subtract each baseline ABS from the corresponding sample or blank ABS, and use the standard curve to calculate the concentration of each analyte in  $\mu\text{g/mL}$ .

7.3 Calculate the concentration for each air sample as:

$$C = \frac{[(A \times SA \times D \times GF) - (B \times SB \times GF)]}{\text{Air Vol}}$$

where:

C is analyte ( $\text{mg/m}^3$ )

A is concn of analyte in the sample solution ( $\mu\text{g/mL}$ )

B = concn of analyte in the blank solution ( $\mu\text{g/mL}$ )

SA = sample solution volume (mL)

SB = blank solution volume (mL)

D = dilution factor (if any)

GF = gravimetric factor (if any; see Table 6)

Air Vol = air volume sampled (L)

7.4 For wipe or bulk samples, calculate the total amount (in  $\mu\text{g}$ ) of analyte in each sample using the equation above. An air volume is not used. Convert bulk sample analytes to % composition using:

$$\text{Analyte \% (w/w)} = \frac{(C)(100\%)}{(\text{Sample wt})(1000 \mu\text{g/mg})} \quad (\text{Bulk Samples})$$

where:

C is analyte amount ( $\mu\text{g}$ )

Sample wt aliquot (in mg) of bulk taken in Section 6.6

## 7.5 Reporting Results to the Industrial Hygienist

For those samples only extracted with DI  $\text{H}_2\text{O}$ , report the sample results as the soluble fraction of the sample. If more than one solution exists for a sample, and it is not necessary to report results separately, then combine these results. An example is a sample that was filtered due to insoluble particulate. The results from the filtrate plus results from the second particulate digestion are added together.

7.5.1 Report air sample results as  $\text{mg/m}^3$  analyte.

7.5.2 Report wipe sample concentrations as total micrograms or milligrams analyte.

7.5.3 Report bulk sample results as approximate percent by weight analyte (note: Sample results for bulk liquids may be reported as approximate percent by volume if volumetric aliquots were taken during sample preparation.) Due to differences in sample matrices between bulks and standards, bulk results are approximate.

## 8. References

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Table 1  
Air Contaminants - OSHA Permissible Exposure Limits\*

Element	Substance Exposed to	Transitional PEL (mg/m <sup>3</sup> )		Final Rule PEL (mg/m <sup>3</sup> )		
		TWA	Ceiling	TWA	STEL	Ceiling
Ag	Metal and soluble cmpds (as Ag)	0.01		0.01		
Al	Soluble salts (as Al) Pyro powders			2	5	
Ba	Soluble compounds (as Ba)	0.5		0.5		
Bi	Bismuth telluride (SE doped)**			5		
Ca	Calcium oxide	5		5		



Element	Substance Exposed to	Transitional PEL (mg/m <sup>3</sup> )		Final Rule PEL (mg/m <sup>3</sup> )		
		TWA	Ceiling	TWA	STEL	Ceiling
Cd	Calcium cyanamide	0.1	0.3	0.5		0.3
Co	Fume	0.2	0.6	0.1		0.6
	Dust	0.1		0.2		
	Metal dust and fume (as Co)			0.05		
Cr	Cobalt Carbonyl or hydrocarbonyl (as Co)	0.5		0.1		
Cs	Cr (II or III) compounds (as Cr)	1		0.5		
	Cr metal (as Cr)			1		
Cu	Cesium hydroxide	0.1		2		
	Fumes (as Cu)	1		0.1		
Fe	Dusts and mists (as Cu)	15		1		
	Dicyclopentadienyl iron total dust	10		10		
	Iron oxide fume (as Fe <sub>2</sub> O <sub>3</sub> )			10		
Hf	Iron salts (soluble) (as Fe)	0.5		1		
In	Hafnium			0.5		
K	Indium and compounds (as In)			0.1		2
Li	Potassium hydroxide	0.025				
Mg	Lithium hydride	15		0.025		
Mn	Magnesium oxide fume total particulate		5	10		5
	Mn compounds (as Mn)		5		3	
	Mn fume (as Mn)			1		
Mo	Manganese tetraoxide (as Mn)	5		1		
	Soluble compounds (as Mo)	15		5		
Na	Insoluble compounds (as Mo) total dust			10		
	Sodium bisulfite			5	0.15	
	Sodium fluoroacetate	2		0.5		2
	Sodium hydroxide					
	Sodium metabisulfate			5		
Ni	Tetrasodium pyrophosphate***	1		5		
	Metal and insoluble compounds (as Ni)	1		1		
Pb	Soluble compounds (as Ni)			0.1		
Pt	Inorganic (see 29 CFR 1910.1025)					
Sb	Pt metal	0.5		1		
Se	Sb and compounds (as Sb)	0.2		0.5		
Sn	Se and compounds (as Se)	2		0.2		
	Inorganic compounds except oxides (as Sn)			2		
Te	Tin oxide (as Sn)	0.1		2		
Ti	Te and compounds (as Te)	15		0.1		
Tl	Titanium dioxide total dust	0.1		10		
Y	+Soluble compounds (as Tl)	1		0.1		
Zn	Yttrium	1		1	2	
	Zinc chloride fume	5		1	10	
	Zinc oxide fume	15		5		
	Zinc oxide total dust	15		10		
Zr	Zinc stearate	5		10	10	
	Zr compounds (as Zr)			5		

\* From reference 8.12 - Final rule PELs were voided by court ruling and are not applicable.

\*\* Sample is also analyzed for Te and Se content

\*\*\* Also can be analyzed for total phosphate content by ion chromatography

+Skin Designation

Note: Compounds having total and respirable dust PELs of 15 and 5 mg/m<sup>3</sup>, respectively, are normally analyzed gravimetrically. Elements contained in these dust samples can be identified by this or other methods, if necessary.

Table 2  
Detection Limits, Sensitivities, and Ranges

Element	Qualitative DL* (µg/mL)	Analytical DL* (µg/mL)	Sensitivity* (µg/mL)	Upper Linear Range* (µg/mL)
Ag	0.002	0.005	0.06	4
Al+	0.02	0.3	1	50
Au	0.01	0.05	0.25	20
Ba+	0.008	0.5	0.4	25

Element	Qualitative DL* (µg/mL)	Analytical DL* (µg/mL)	Sensitivity* (µg/mL)	Upper Linear Range* (µg/mL)
Bi	0.025	0.2	0.5	30
Ca	<0.0005	0.03+	0.08 (0.029)+	7
Cd	0.0002	0.004	0.025	2
Co	0.01	0.04	0.15	5
Cr	0.003	0.04 (0.04)+	0.1 (0.31)+	5 (10)+
Cs	0.005++		0.2	15
Cu	0.001	0.005	0.09	5
Fe	0.005	0.03	0.12	5
Hf+	2.0		15	500
In	0.02	0.1	0.7	50
K	<0.002	0.02	0.04	2
Li	0.0003	0.004	0.035	2
Mg	<0.0001	0.01	0.007	0.5
Mn	0.002	0.01	0.055	3
Mo+	0.02	0.04	0.5	60
Na	<0.0002	0.009	0.015	1
Ni	0.002	0.1	0.15	5
Pb	0.01	0.05	0.5	20
Pt		2.0	13	
Sb**	(0.08)	0.1	1.0	50
Se***	(0.05)	0.3	0.25	25
Sn***	(0.01)	0.1	0.6	40
Te	0.05	0.2	1.0	25
Ti	0.04		1.8	
Tl	0.03	0.05	0.5	20
Y	0.05	0.7	1.8	200
Zn	<0.01	0.01	0.018	1
Zr+	1.0	8	10.0	800

\* DL = Detection Limit. See Appendix A for more information regarding definitions or calculations. Analytical DLs are approximate.

\*\* Alternate line of 231.2 nm was used with one exception: The qualitative detection limit value is for the primary line (217.6 nm).

\*\*\* Air/H<sub>2</sub> flame used with the exception of the qualitative detection limit determination. This value is for Air/C<sub>2</sub>H<sub>2</sub> flame.

+ N<sub>2</sub>O/C<sub>2</sub>H<sub>2</sub> flame used.

++ Flame emission used to determine qualitative detection limit.

Table 3  
Precision and Accuracy\*

Element	CV	% Ave Recovery	Range**	N
Ag	0.083	97.8	1-4	270
Al	0.076	94.5	100-500	27
Au				
Ba	0.10	104.7	50-75	45
Bi				
Ca	0.162	98.3	100-500	51
Cd	0.087	99.5	10-15	93
Co	0.052	99.3	10-15	39
Cr (Soluble)				
Cr (Insoluble)	0.052	95.7	45-75	72
Cs				
Cu	0.043	96.8	100-150	45
Fe	0.084	98.2	300-400	69
Hf				
In				
K	0.063	93.3	125-200	30
Li				
Mg	0.073	112.1	100-300	24
Mn	0.044	100.2	100-150	60
Mo (Soluble)				
Mo (Insoluble)	0.075	91.2	100-250	27
Na	0.058	97.5	100-250	68
Ni	0.065	99.1	100-150	18
Pb	0.047	99.3	20-40	300
Pt	0.055	98.1	80-1800	24+

Element	CV	% Ave Recovery	Range**	N
Sb	0.081	98.4	50-75	36
Se	0.122	104.9	20-100	30
Sn	0.079	97.4	100-150	63
Te				
Ti				
Tl				
Y				
Zn	0.039	101.2	100-150	69
Zr				

CV Coefficient of Variation

\* Table updated January, 1990 (8.13)

\*\* Range (in µg) of analyte spiked onto MCE filters. Samples were spiked with aqueous solutions of dissolved metals or their salts. All samples were prepared and analyzed using conditions stated in the method.

+ These samples were prepared by weighing the metal on filters. A single blind study was not performed.

Table 4  
Alternate Procedures

AP 1: Ag, Pb, Sb, Se, Te

- 1) Digest samples with HNO<sub>3</sub>. Heat until the liquid is nearly gone. Allow the samples to cool to room temperature.
- 2) For 25 mL final sample solution volumes, add the following amount of concd HCl (Adjust accordingly for alternate solution volumes):

<u>Analyte Suspected to be Present</u>	<u>Amount of HCl</u>
Sb	8 mL
Pb or Ag	4 mL
Se or Te	1 mL

- 3) Warm gently and swirl to dissolve the analyte. Allow samples to cool and dilute to a 25-mL volume with DI H<sub>2</sub>O.

AP 2: Soluble Compounds of Al, Ba, Cr (II or III), Fe, Ni, Mo, Tl, Zn

- 1) Place the sample in a beaker and add an aliquot of room-temperature DI H<sub>2</sub>O into the beaker (15 mL is typically used for a full-shift sample).
- 2) Place the beaker in an ultrasonic bath for approximately 10 min.
- 3) Filter the sample through a 0.45 µm MCE filter and transfer the filtrate to a 25-mL volumetric flask. If an insoluble fraction is also requested, digest both sample filters according to the appropriate procedure.
- 4) Add reagents to achieve the final solution concentrations listed:

<u>Analyte Suspected to be Present</u>	<u>Final Concentration</u>
Cr(II or III), Fe, Ni, Tl, Zn (as ZnCl <sub>2</sub> )	4% HNO <sub>3</sub>
Al, Ba	4% HNO <sub>3</sub> /1000 µg/mL Potassium ion
Mo	4% HNO <sub>3</sub> /1000 µg/mL Aluminum ion

AP 3: Al (pyro powders), Ca, Mg, Mo (insoluble), Y

- 1) Digest the sample using the procedure described in Section 6.6.3.a.
- 2) Transfer the sample to a volumetric flask.
- 3) Dilute the samples and add ionization suppressants to achieve the final solution concentrations listed:

<u>Analyte Suspected to be Present</u>	<u>Final Concentration</u>
Al (pyro powders), Ca, Mg, Y	4% HNO <sub>3</sub> /1000 µg/mL Potassium ion
Mo (insoluble)	4% HNO <sub>3</sub> /1000 µg/mL Aluminum ion

AP 4: Au, Pt (metal), Sn, or Tin Oxide (SnO)

- 1) For Au, Sn, or SnO, add 9 mL HCl to each beaker, swirl, and then add 2 mL HNO<sub>3</sub>. CAUTION: Make sure the entire filter or sample is wetted with HCl and allow the filter/HCl solution to sit for a period of at least 2 to 3 min before adding the HNO<sub>3</sub>.
- 2) Digest the sample on a hot plate until nearly dry.
- 3) Allow the samples to cool and then quantitatively transfer the sample, using a small amount of DI H<sub>2</sub>O to rinse the beaker, to a clean volumetric flask. Dilute to volume, making the final solution 10% HCl. For example, add 2.5 mL concd HCl to a sample if the total solution volume is 25 mL.
- 4) Results for either Sn or SnO are reported as total Sn.

Table 4  
Alternate Procedures

**AP 5: Cr [Samples which potentially contain Cr(VI)]**

For samples requiring analysis of total Cr, the following procedure should be used. This procedure avoids the loss of any Cr(VI) as chromyl chloride( $\text{CrO}_2\text{Cl}_2$ ). For chromate or chromic acid analysis, see OSHA Method ID-215.

- 1) Digest the samples collected on MCE filters with  $\text{HNO}_3$  and then allow to cool to room temperature. If PVC filters were used, digest with  $\text{HNO}_3$  plus 2 mL of  $\text{HClO}_4$  and then allow to cool.
- 2) Add 1 or 2 mL of 30%  $\text{H}_2\text{O}_2$  to the cooled solution to reduce any Cr(VI) that may be present. Let the sample sit for several minutes.
- 3) Heat approximately 5 min to boil off the  $\text{H}_2\text{O}_2$  and then allow to cool. At this stage HCl may be added if needed to dissolve other metals.
- 4) Dilute to volume with DI  $\text{H}_2\text{O}$  and analyze.

NOTE: Do not add  $\text{HClO}_4$  to the sample solution if a large amount of HCl is already present [any Cr(VI) in the sample would be lost as  $\text{CrO}_2\text{Cl}_2$ ]. Add concd  $\text{HNO}_3$ , boil off the HCl, and then add the  $\text{HClO}_4$ .

**AP 6: Elements or Compounds\* which are Insoluble in Nitric Acid Digestions**

- 1) For compounds such as zirconium dioxide or hafnium dioxide, place the sample filter in a platinum crucible, char at 300 °C, then heat the residue at 800 °C in a muffle furnace. [As an alternative, the digestion can be performed using a microwave digestion system (8.11).]
- 2) Add 1 to 2 mL of HF, swirl the solution, and then heat on a hot plate to dissolve the residue.
- 3) Evaporate the solution to approximately 0.4 mL and then transfer to a 10-mL polyethylene volumetric flask. Dilute to volume with a solution of 0.1 M ammonium fluoride in 4%  $\text{HNO}_3$ .

Another procedure can be used for elements which do not need to be converted to their fluoride salts:

- 1) Heat the HF solution on the hot plate until the liquid is nearly gone.
- 2) Add 2 to 3 mL HCl, and warm the solution until about 1 mL remains.
- 3) Quantitatively transfer the solution to a 10 mL volumetric flask and dilute to volume with the appropriate diluents mentioned in Table 5.
- 4) It is recommended to prepare quality control samples of the substance of concern. Digest the samples and analyze by the same procedure to check the recovery efficiency.

For platinum:

- 1) Place the sample filter in a Teflon microwave digestion vessel and add 5 mL of the "acid mixture (HCl/ $\text{HNO}_3$ ) for platinum digestions" prepared in Section 6.3.4, part f).
- 2) Digest the sample according to Microwave Digestion Standard Operating Procedure (8.11) or manufacturer guidelines.
- 3) Allow the sample to cool and then transfer to a 25-mL volumetric flask. Dilute to volume with DI  $\text{H}_2\text{O}$ .

\*Some Zr compounds, such as the oxide and sulfate, may be insoluble when using the  $\text{HNO}_3$  digestion (8.6, 8.7). Hafnium dioxide may also be insoluble.

**AP 7: CsOH, KOH, LiH, and Na Compounds**

- 1) Place the sample filter in a beaker and desorb with 15 mL of DI  $\text{H}_2\text{O}$  for approximately 5 min.
- 2) Decant the sample solution into a 25-mL volumetric flask and add any reagents to achieve the final solution concentrations:

Analyte Suspected to be Present	Final Concentration
CsOH	DI $\text{H}_2\text{O}$ /1000 $\mu\text{g}/\text{mL}$ potassium ion
LiH, Na cmpds	DI $\text{H}_2\text{O}$
KOH	DI $\text{H}_2\text{O}$ /1000 $\mu\text{g}/\text{mL}$ sodium ion

For example, add 5 mL of 5,000  $\mu\text{g}/\text{mL}$  potassium ion for Cs analysis and dilute to volume with DI  $\text{H}_2\text{O}$ . Add 5 mL of 5,000  $\mu\text{g}/\text{mL}$  sodium ion for KOH analysis.

- 3) Analyze by flame emission or atomic absorption.

**AP 8: Titanium Dioxide**

- 1) Digest the filter with 1 mL  $\text{HNO}_3$  and 2 mL  $\text{H}_2\text{SO}_4$  in a conical beaker and heat until about 1 mL remains.
- 2) Quantitatively transfer the solution to a 25-mL volumetric flask, add 5 mL of 5,000  $\mu\text{g}/\text{mL}$  potassium ion, then dilute to volume with DI  $\text{H}_2\text{O}$ .

Table 5  
Digestion or Extraction Reagents

Substance	Reagents Used	Final Volume Concentration+
Ag	HNO <sub>3</sub> /HCl	4% HNO <sub>3</sub> /16% HCl
Al (soluble cmpds)	DI H <sub>2</sub> O	4% HNO <sub>3</sub> /1000 µg/mL K <sup>+</sup>
Al (pyro powders)	HNO <sub>3</sub>	4% HNO <sub>3</sub> /1000 µg/mL K <sup>+</sup>
Au	HCl/HNO <sub>3</sub>	10% HCl
Ba (soluble cmpds)	DI H <sub>2</sub> O	4% HNO <sub>3</sub> /1000 µg/mL K <sup>+</sup>
Bi <sub>2</sub> Te <sub>3</sub>	HNO <sub>3</sub>	4% HNO <sub>3</sub>
Ca & cmpds	HNO <sub>3</sub> /HCl*	4% HNO <sub>3</sub> /1000 µg/mL K <sup>+</sup>
Cd	HNO <sub>3</sub>	4% HNO <sub>3</sub>
Co & cmpds	HNO <sub>3</sub> /HCl*	4% HNO <sub>3</sub>
Cr(III or III) soluble cmpds	DI H <sub>2</sub> O	4% HNO <sub>3</sub>
Cr metal	HNO <sub>3</sub> /H <sub>2</sub> O <sub>2</sub> /HCl	4% HNO <sub>3</sub>
CsOH	DI H <sub>2</sub> O	DI H <sub>2</sub> O/1000 µg/mL K <sup>+</sup>
Cu	HNO <sub>3</sub>	4% HNO <sub>3</sub>
Fe & cmpds	HNO <sub>3</sub> /HCl*	4% HNO <sub>3</sub>
Fe (soluble salts)	DI H <sub>2</sub> O	4% HNO <sub>3</sub>
Hf	HF	4% HF/4% HNO <sub>3</sub> /0.1 M NH <sub>4</sub> F
In & cmpds	HNO <sub>3</sub>	4% HNO <sub>3</sub>
KOH	DI H <sub>2</sub> O	4% HNO <sub>3</sub> /1000 µg/mL Na <sup>+</sup>
LiH	DI H <sub>2</sub> O	DI H <sub>2</sub> O
MgO	HNO <sub>3</sub> /HCl*	4% HNO <sub>3</sub> /1000 µg/mL K <sup>+</sup>
Mn & cmpds	HNO <sub>3</sub> /HCl*	4% HNO <sub>3</sub>
Mo (soluble cmpds)	DI H <sub>2</sub> O	4% HNO <sub>3</sub> /1000 µg/mL Al
Mo (insoluble cmpds)	HNO <sub>3</sub>	4% HNO <sub>3</sub> /1000 µg/mL Al
Na & cmpds	DI H <sub>2</sub> O	DI H <sub>2</sub> O
Ni metal & insoluble cmpds	HNO <sub>3</sub> /HCl*	4% HNO <sub>3</sub>
Ni (soluble cmpds)	DI H <sub>2</sub> O	4% HNO <sub>3</sub>
Pb	HNO <sub>3</sub> /HCl	4% HNO <sub>3</sub> /16% HCl
Pt metal	HCl/HNO <sub>3</sub>	4% HNO <sub>3</sub> /16% HCl
Sb & cmpds	HNO <sub>3</sub> /HCl	4% HNO <sub>3</sub> /32% HCl
Se & cmpds	HNO <sub>3</sub> /HCl	4% HNO <sub>3</sub> /4% HCl
Sn (and SnO)	HCl/HNO <sub>3</sub>	10% HCl
Te & cmpds	HNO <sub>3</sub> /HCl	4% HNO <sub>3</sub> /4% HCl
TiO <sub>2</sub>	HNO <sub>3</sub> /H <sub>2</sub> SO <sub>4</sub>	4% H <sub>2</sub> SO <sub>4</sub> /1000 µg/mL K <sup>+</sup>
Tl (soluble cmpds)	DI H <sub>2</sub> O	4% HNO <sub>3</sub>
Y	HNO <sub>3</sub>	4% HNO <sub>3</sub> /1000 µg/mL K <sup>+</sup>
ZnCl <sub>2</sub>	DI H <sub>2</sub> O	4% HNO <sub>3</sub>
Zn & cmpds	HNO <sub>3</sub>	4% HNO <sub>3</sub>
Zr & cmpds	HF	4% HF/4% HNO <sub>3</sub> /0.1 M NH <sub>4</sub> F

+ Standards should be prepared in this matrix.

\* After completing the digestion with HNO<sub>3</sub>, add 1 or 2 drops of concd HCl to facilitate particulate dissolution.

Table 6  
Gravimetric Factors

Element	Compound	Gravimetric Factor
Bi	Bismuth telluride (Bi <sub>2</sub> Te <sub>3</sub> )	1.916
Ca	Calcium cyanamide (CaCN <sub>2</sub> )	1.998
Ca	Calcium hydroxide [Ca(OH) <sub>2</sub> ]	1.849
Ca	Calcium oxide (CaO)	1.399
Cr	Chromic acid (CrO <sub>3</sub> )	1.923
Cs	Cesium hydroxide (CsOH)	1.128
Fe	Dicyclopentadienyl iron [(C <sub>5</sub> H <sub>5</sub> ) <sub>2</sub> Fe]	3.331
Fe	Iron oxide (Fe <sub>2</sub> O <sub>3</sub> )	1.430
Li	Lithium hydride (LiH)	1.145
Mg	Magnesium oxide (MgO)	1.658
Na	Sodium bisulfite (NaHSO <sub>3</sub> )	4.525
Na	Sodium fluoroacetate (FCH <sub>2</sub> COONa)	4.351
Na	Sodium hydroxide (NaOH)	1.740
Na	Sodium metabisulfite (Na <sub>2</sub> S <sub>2</sub> O <sub>5</sub> )	4.134
Na	Tetrasodium pyrophosphate (Na <sub>4</sub> P <sub>2</sub> O <sub>7</sub> )	2.891

Element	Compound	Gravimetric Factor
Ti	Titanium oxide (TiO <sub>2</sub> )	1.668
Zn	Zinc chloride (ZnCl <sub>2</sub> )	2.085
Zn	Zinc oxide (ZnO)	1.245
Zn	Zinc stearate [Zn(C <sub>18</sub> H <sub>35</sub> O <sub>2</sub> ) <sub>2</sub> ]	9.671

Table 7  
Analytical Parameters

Element	λ (nm)	Slit (nm)	Optimization*	Flame Used	Comments
Ag	328.1	0.7	4 µg/mL=0.3 ABS	1	For multielement lamps containing Cu, use 0.2 nm slit
Al	309.3	0.7	50 µg/mL=0.22 ABS	3	
Au	242.8	0.7	15 µg/mL=0.26 ABS	1	
Ba	553.6	0.4	15 µg/mL=0.16 ABS	3	
Bi	223.1	0.2	20 µg/mL=0.18 ABS	1	
Ca**	442.7	0.7	4 µg/mL=0.22 ABS	4	
Cd**	228.8	0.7	2 µg/mL=0.35 ABS	1	
Co	240.7	0.2	5 µg/mL=0.015 ABS	2	
Cr**	357.9	0.7	2 µg/mL=0.05 ABS	3	
Cs	852.1	1.4	10 µg/mL=0.22 ABS	1	
Cu**	324.7	0.7	5 µg/mL=0.25 ABS	1	For multielement lamps containing Ni or Fe, use 0.2 nm slit
Fe**	248.3	0.2	5 µg/mL=0.18 ABS	2	In the presence of Co, do not use a multielement lamp containing Co at 248.3 nm. Use 248.8 or 72.0 nm
	248.8	0.2	5 µg/mL=0.11 ABS	2	
Hf	286.6	0.2	300 µg/mL=0.2 ABS	3	
In	303.9	0.7	25 µg/mL=0.15 ABS	1	
K	766.5	1.4	2 µg/mL=0.3 ABS	1	
Li	670.8	1.4	1 µg/mL=0.13 ABS	1	
Mg**	285.2	0.7	0.3 µg/mL=0.19 ABS	3	
Mn**	279.5	0.2	2 µg/mL=0.16 ABS	1	
Mo**	313.5	0.7	2 µg/mL=0.20 ABS	3	
Na	589.6	0.4	0.8 µg/mL=0.2 ABS	1	
Ni**	232.0	0.2	5 µg/mL=0.15 ABS	1	For multielement lamps containing Fe, use the secondary Ni line, 352.4 nm
Pb	283.3	0.7	20 µg/mL=0.18 ABS	1	
Pt	265.9	0.7	100 µg/mL=0.033 ABS	3	
Sb	217.6	0.2	20 µg/mL=0.18 ABS	1	For determination in the presence of Pb, use the 231.2 nm line
	231.2	0.7	20 µg/mL=0.07 ABS	1	
Se	196.0	2.0	20 µg/mL=0.18 ABS	5	Use an EDL
Sn	224.6	0.7	50 µg/mL=0.28 ABS	5	
Te	214.3	0.2	25 µg/mL=0.11 ABS	1	
Ti	365.3	0.2	120 µg/mL=0.3 ABS	3	
Tl	276.8	0.7	20 µg/mL=0.18 ABS	1	
Y	410.2	0.2	100 µg/mL=0.24 ABS	3	
Zn**	213.9	0.7	0.5 µg/mL=0.12 ABS	1	In the presence of Cu, do not use a multielement lamp containing Cu
Zr	360.1	0.2	400 µg/mL=0.17 ABS	3	

\* Adapted from reference 8.6 or from laboratory determinations

\*\* Due to the limited upper linear range, samples may have to be diluted, the burner head rotated, or an alternate wavelength used. The burner head is routinely rotated for Fe and Mg before analysis.

Flame Types:

- 1 Air/Acetylene mixture, lean, blue flame
- 2 Air/Acetylene mixture, very lean, blue flame
- 3 Nitrous oxide/Acetylene mixture, rich, red flame
- 4 Nitrous oxide/Acetylene mixture, slightly rich, red flame
- 5 Air/Hydrogen mixture

## Appendix A Terminology

For the purposes of this method, the following definitions are used:

### Qualitative detection limit

The concentration ( $\mu\text{g/mL}$ ) of an element which would yield an absorbance (ABS) equal to twice the standard deviation of a series of measurements of an aqueous solution containing the element. The signal obtained from the aqueous solution must be distinctly greater than the baseline (8.10). These detection limits were taken from reference 8.5.

### Analytical detection limit

The lowest concentration ( $\mu\text{g/mL}$ ) of an element that can be reliably quantitated. This limit is the largest value obtained from any of the three calculations:

- Three times the smallest possible non-zero instrument reading,
- Two times the average baseline variation, or
- The lowest standard used to construct a concentration-response curve. One-tenth the concentration of this standard is considered to be the detection limit if:

The average reading for this standard is within 20% of its linear response. The linearity is determined by the other standards used to construct the least-squares curve fit.

If the lowest standard ABS reading is more than 20% in error, then an algorithm is used and the concentration value is increased in 10% increments until a concentration is achieved that would display less than 20% error or until the lowest standard concentration is reached.

### Sensitivity

The concentration ( $\mu\text{g/mL}$ ) of an element in aqueous solution which will produce an ABS of 0.0044 (8.6).

### Linear Range

The working range of a specific analyte. The range is considered linear if doubling the concentration of a standard results in at least a 75% increase in ABS.

## Appendix B Potential Interferences

- Ag If a multielement lamp containing Cu is used, a spectral interference may occur when determining Ag in a sample containing Cu. A narrow slit should be used in this instance (8.6).
- Thorium (Th) is a reported chemical interference (8.14); however, this element is extremely rare in workplace environments. Analyze the sample for Th first if both are suspected to be present.
- Al Acetic acid, fluoroborate, Fe, and Ti enhance the Al signal. Ionization should be controlled by adding an alkali salt (potassium or lanthanum) to samples and standards.
- Au Spectral interferences from Fe have been observed. Palladium, platinum, and cyanide complexes are reported interferences (8.6).
- Ba This element is partially ionized in the  $\text{N}_2\text{O}/\text{C}_2\text{H}_2$  flame. To control this interference, the samples and standards should contain 1,000  $\mu\text{g/mL}$  potassium ion (8.6).

When analyzing using the primary Ba line (553.6 nm), background correction should be used if a large amount of Ca is present. The Ca can cause molecular absorption at this line.

Ca Sulfate, aluminate, phosphate, and silicate decrease sensitivity (8.14). Silicon (Si), Ti, Al, and Zr have also been reported as interferences (8.6). Using a  $N_2O/C_2H_2$  flame will control these interferences; however, samples and standards should contain 1,000  $\mu\text{g/mL}$  potassium ion to control any ionization.

Acetone from acetylene tanks has been reported to decrease sensitivity. Tanks should be changed when the pressure drops below 75 to 85 psig to prevent acetone from entering the flame (8.9).

Cd A possible interference is Si; however, Si is not significantly soluble using the mentioned digestion procedures.

Co A reported interference is Ni in concentrations greater than 1,500  $\mu\text{g/mL}$  (8.10). Such levels of Ni are unusual in industrial environments. If a large amount is expected, samples should be analyzed for Ni first and then analyzed using an alternate Co line if Ni concentrations exceed 1,500  $\mu\text{g/mL}$ .

Cr Co, Fe, Ni, Cu, Ba, Al, Mg, Ca, Na, and other metals have been reported as chemical interferences (8.6, 8.9, 8.10). Determining Cr in a lean flame will control these interferences, but with a decrease in sensitivity (8.9, 8.10). The instrument should be optimized using a mixed standard containing Fe and Ni in addition to the Cr when using the Air/ $C_2H_2$  flame. The above interferences are not noticed when a  $N_2O/C_2H_2$  flame is used.

Cs Solutions should contain 1,000  $\mu\text{g/mL}$  potassium ion to control ionization.

Strong acids may suppress the signal; therefore, samples and standards should be matrix-matched.

Cu Spectral interferences may occur when Ni or Fe is contained in the multielement lamp and in the sample solution. Use a single element Cu lamp or a narrow slit to circumvent this problem.

A large amount of Zn in the sample may interfere but can be controlled by using a lean flame (8.10).

Fe A spectral interference may be observed if the multielement lamp and the sample solution contain Co. An alternate line for Fe should be used (8.6).

Citric acid, Ni, and  $HNO_3$  may interfere but can be controlled by using a lean flame and by carefully optimizing burner height (8.6, 8.10). Silica may also interfere (8.14), but is not appreciably soluble in the acid digestion procedures mentioned.

Hf The presence of fluoride greatly enhances the sensitivity in the determination of Hf. Samples and standards should contain 0.1 M  $NH_4F$  to control this effect and to obtain the best sensitivity (8.6).

In A 100-fold or greater excess of Al, Mg, Cu, Zn, or phosphate will suppress the signal.

Mg Al,  $H_2SO_4$ ,  $HNO_3$ , Si, Ti, and HF are reported to interfere. Addition of a suppressant (lanthanum or potassium) will control these interferences (8.6, 8.14). Interferences can also be controlled using a  $N_2O/C_2H_2$  flame.

Mn Phosphate, perchlorate, Fe, Ni, and Co may interfere but can be controlled by using a lean flame (8.10). Tungsten (W), Mo, and Si have been reported to interfere when the pressure in the acetylene tanks is low (8.14).

Mo Many interferences have been reported for Mo including Fe, Mn, Ni, Cr, Si, and strontium (Sr). Addition of Al controls these interferences (8.9, 8.10, 8.14).

Na Ionization in the flame can occur; an ionization suppressant should be added to the standards and samples (8.6).



- Ni A spectral interference from Fe will result when determining Ni in a sample containing Fe with a multielement lamp containing Fe. An alternate line should be used.
- Cr, Co, and Fe (8.9), or HCl and HClO<sub>4</sub> in the presence of these metals (8.10) have been reported as interferences. They are controlled by using a lean Air/C<sub>2</sub>H<sub>2</sub> flame (8.10, 8.14).
- Pb Al, Be, Th, and Zr in a 1,000-fold molar excess over the Pb concentration decrease sensitivity (8.14). The digestion procedure used for Pb does not solubilize a significant amount of Al, Be, or Zr for them to be a problem in the analysis. Workplace environments rarely contain significant amounts of Th along with Pb; however, if suspected to be present, the sample should also be analyzed for Th since it is very toxic.
- Phosphate, carbonate, iodide, fluoride, and acetate at a 10-fold excess may also interfere (8.10). Sulfate and Ca in excess have also been reported as interferences (8.7).
- Pt A number of elements interfere with the determination when using an Air/C<sub>2</sub>H<sub>2</sub> flame (8.6). These interferences are minimized when using a N<sub>2</sub>O/C<sub>2</sub>H<sub>2</sub> flame.
- Sb A spectral interference occurs when Sb is determined at 217.6 nm in the presence of large amounts of Pb, which has an adjacent line at 217.0 nm. It has been reported that large concentrations of Cu also absorb at 217.6 nm. In either situation, the alternate 231.2 nm line for Sb should be used (8.6, 8.7)
- Cu and Ni have been reported to suppress Sb sensitivity, but can be controlled by using a lean flame (8.9, 8.10).
- Se Background absorption is severe at the wavelengths used to determine Se. Background correction should be used (8.6).
- Large amounts of Ni, Co, Fe, Cu, Mn, Pb, and other metals, if present in the sample may form selenides in the flame, decreasing sensitivity (8.9).
- Increased sensitivity is noted when using an Air/H<sub>2</sub> flame as compared to an Air/C<sub>2</sub>H<sub>2</sub> flame. For greatly enhanced sensitivity, analyze Se by graphite furnace atomic absorption using a modified matrix containing Ni (8.15).
- Sn Alkali metals and alkaline earths, Cu, Co, Zn, Al, Ti, phosphoric acid (H<sub>3</sub>PO<sub>4</sub>), and H<sub>2</sub>SO<sub>4</sub> have been reported as interferences when Air/H<sub>2</sub> flames are used. Interferences are reduced or eliminated in hotter flames, but sensitivity is greatly reduced (8.6, 8.10).
- Te A spectral interference may occur when Cu is contained in the multielement lamp and in the sample (8.14).
- Enhanced sensitivity can be obtained for this element using graphite furnace atomic absorption analysis of sample solutions modified to contain a Ni matrix (8.15).
- Ti Samples and standards should contain 1,000 µg/mL potassium ion to control ionization.
- The Ti signal is enhanced by many other metals (8.6).
- Y Samples and standards should contain 1,000 µg/mL potassium ion to control ionization.
- Strong acids may suppress the signal; therefore, samples and standards should be matrix-matched.
- Zn A spectral interference may occur if the multielement lamp and the sample contain Cu (8.14).
- Zr Fluoride, chloride, and ammonium enhance Zr sensitivity. Sulfate, nitrate, and nickel bromide decrease sensitivity. Addition of NH<sub>4</sub>F will control these interferences (8.6).