

ICP ANALYSIS OF METAL/METALLOID PARTICULATES  
FROM SOLDER OPERATIONS



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Method Number: ID-206

Matrix: Air, Wipe (Smear Tab), or Bulk

OSHA Time Weighted  
Average (TWA) Permissible  
Exposure Limits: 0.01 mg/m<sup>3</sup> Silver (Ag)  
0.002 mg/m<sup>3</sup> Beryllium (Be)\*  
0.1 mg/m<sup>3</sup> Cadmium (Cd) fume  
0.1 mg/m<sup>3</sup> Copper (Cu) fume  
0.05 mg/m<sup>3</sup> Lead (Pb)  
0.5 mg/m<sup>3</sup> Antimony (Sb)  
2.0 mg/m<sup>3</sup> Tin (Sn)  
5.0 mg/m<sup>3</sup> Zinc Oxide (ZnO) fume

Collection Procedure: A personal sampling pump is used to draw a known volume of air through a mixed cellulose ester membrane filter contained in a polystyrene cassette. Wipe (smear tab) and bulk material are collected by grab sampling techniques.

Recommended Sampling Rate: 2 liters per minute

Recommended Air Volumes  
Be, Cd, Cu, Pb, Sb, Sn, ZnO: 480 L  
If Ag is determined: 960 L

Analytical Procedure: Filters are digested with hydrochloric and nitric acids. Analysis is performed using Inductively Coupled Plasma-Atomic Emission Spectroscopy.

Detection Limits: See Section 2

Validation Range: See Section 3

Method Classification: Validated analytical method

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Date: May, 1991

\* This method may be used to determine STEL (0.005 mg/m<sup>3</sup>) or Ceiling (0.025 mg/m<sup>3</sup>) exposures to Be. At least a 30-min sample (2 L/min) should be taken for STEL determinations; at least 5-min for Ceiling monitoring.

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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

### 1.1 Scope

- 1.1.1 This method describes the collection and analysis of airborne metal and metalloid particulates from solder operations in industry. Time Weighted Average (TWA) air samples are collected using personal sampling pumps and mixed-cellulose ester (MCE) filters. Analysis is by Inductively Coupled Plasma-Atomic Emission Spectroscopy (ICP-AES). Bulk and wipe samples can also be collected and analyzed by this method.
- 1.1.2 This method provides rapid simultaneous analysis and data reduction for a wide range of elements, eliminating the necessity of separate analyses by conventional atomic absorption techniques.
- 1.1.3 This method was validated for 8 elements (Ag, Be, Cd, Cu, Pb, Sb, Sn, and Zn). A total of 21 elements are analyzed, but 13 of these are determined for screening purposes only (Al, As, Ca, Co, Cr, Fe, Ni, Mg, Mn, Mo, Se, Si, and V). Other elements can be added to or subtracted from the method. The capability for expanding the analysis to other elements is dependent on laboratory instrumentation, and element solubility and stability in the acid matrix used for digestion.
- 1.1.4 The elements validated are those commonly found in solders or soldering operations. The acid matrices used for sample digestion (4:1 ratio of HCl:HNO<sub>3</sub>) and dilution (32% HCl/4% HNO<sub>3</sub>) were selected to ensure solubility of the eight elements.

### 1.2 History

- 1.2.1 Previous to the introduction of ICP-AES, samples containing metallic particulates were digested in a variety of ways and analyzed by Atomic Absorption Spectroscopy (AAS) at the OSHA Salt Lake City Analytical Laboratory (SLCAL).
- 1.2.2 An ICP simultaneous spectrometer [Instruments SA (ISA), Model JY-32, Edison, NJ] was used to validate this method.

This method is applicable to any simultaneous spectrometer. The validation of the JY-32 is described in the backup report (8.1).

## 2. Detection Limits and Working Ranges (8.1)

### 2.1 Detection limits are listed below:

Element	Qualitative Detection Limit		Quantitative Detection Limit	
	(µg/mL)	(µg*)	(µg/mL)	(µg*)
Ag	0.018	0.45	0.061	1.5
Be	0.00029	0.0072	0.00086	0.022
Cd	0.0062	0.15	0.0205	0.51
Cu	0.0078	0.19	0.026	0.65
Pb	0.071	1.8	0.237	5.9
Sb	0.14	3.5	0.47	12
Sn	0.074	1.8	0.246	6.1
Zn	0.0075	0.19	0.025	0.63

\* 25-mL solution volume

The quantitative limit is considered as the lower working range limit. These values are based on instrument performance and will normally change over time.

- 2.2 The upper working range limit is 100 µg/mL for all validated elements except Be. Due to the high sensitivity resulting from instrument settings and choice of wavelength, the upper limit for Be is 10 µg/mL.

### 3. Precision and Accuracy (8.1)

3.1 The precision and accuracy data for the 8 validated elements are listed below:

Element	Range (µg)		Bias	CV <sub>1</sub> (pooled)	Overall Error (±%)(Total)
Ag	4.6	19	0.006	0.054	11.5
Be	0.47	2.0	-0.075	0.037	14.9
Cd	23	100	0.048	0.041	13.1
Cu	23	94	0.065	0.038	14.2
Pb	11	48	-0.038	0.060	15.7
Sb	126	510	0.014	0.043	10.1
Sn	476	2000	-0.008	0.043	9.4
Zn	973	2900	0.031	0.039	10.9

These values are based on six samples at each of three concentration levels tested. Solutions of the eight elements were spiked on MCE filters. These samples were then digested and analyzed as in Section 6. of this method.

3.2 The eight elements reported above were spiked at 0.5, 1, and 2 times the PEL, assuming a 480-L air volume for all elements except Ag where a 960-L air volume was used. Overall analytical errors for the elements tested were within ±25%; the greatest value was 15.7% for Pb. The large overall error may be attributed to the fact that Pb was validated near its detection limit.

### 4. Interferences (8.2)

High temperatures present in the plasma (5,000 to 8,000 °C) minimize most chemical and matrix interferences. Interferences do exist, however, and can be categorized as follows:

- 4.1 Physical interferences such as nebulization and transport effects are influences that determine the rate and particle size in which analytes are delivered to the plasma. These effects are minimized by matching the acid concentrations of samples and standards.
- 4.2 Chemical interferences are characterized by molecular compound formation, ionization effects, and solute volatilization effects. These effects are not severe in ICP analysis and are minimized by matrix matching and careful selection of operating conditions such as: Incident plasma source power, sample uptake rate, and plasma observation height.
- 4.3 Spectral interferences include:
  - a) Unresolved overlap of molecular band spectra.
  - b) Overlap of a spectral line from another element.
  - c) Background from continuous or recombination phenomena.
  - d) Background from stray light.

The first effect (a) can be minimized by a careful selection of wavelengths for the reported elements. The wavelengths selected for analysis using the JY-32 are shown in Table 1. The other types of spectral interferences (spectral overlap and elevated background) are minimized by software which performs interelement corrections. This software assumes a linear relationship between the analyte concentration and interference within the working range limits. The spectral interference correction equation typically used by ICP manufacturers is:

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$$\text{Corrected Conc} = \text{Calculated Conc} - A_i \times CP_i$$

where:

$A_i$  is Correction factor

$CP_i$  is Concentration of the interfering element

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Samples having analyte concentrations above the working range limits listed in Section 2.2 should be diluted into range; interelement corrections may not be accurate above the working range. Further information and experimentally determined interelement corrections for the validated elements are listed in the backup report (8.1).

- 4.4 If necessary, supplemental background correction can be performed with additional software supplied by the instrument manufacturer.

## 5. Sampling

### 5.1 Safety Precautions

- 5.1.1 Attach the sampling equipment to the worker such that it will not interfere with work performance or safety.
- 5.1.2 Follow all safety practices that apply to the work area being sampled.
- 5.1.3 Wear impermeable gloves when taking wipe or bulk samples.

### 5.2 Equipment

- 5.2.1 Mixed cellulose ester (MCE) filters (0.8- $\mu$ m pore size), cellulose backup pads, and two- or three-piece cassettes, 37-mm diameter, (part no. MAWP 037 A0, Millipore Corp., Bedford, MA).
- 5.2.2 Gel bands (Omega Specialty Instrument Co., Chelmsford, MA) for sealing cassettes.
- 5.2.3 Sampling pumps capable of sampling at 2 liters per minute (L/min).
- 5.2.4 Assorted flexible tubing.
- 5.2.5 Stopwatch and bubble tube or meter for pump calibration.
- 5.2.6 Scintillation vials, 20 mL, (part no. 74515 or 58515, Kimble, Div. of Owens-Illinois Inc., Toledo, OH) with polypropylene or Teflon cap liners. If possible, submit bulk or wipe samples in these vials for ICP analysis.
- 5.2.7 Smear tabs, (part no. 225-24, SKC Inc., Eighty Four, PA) for wipe sampling.
- 5.2.8 Gloves, disposable (for wipe sampling).

### 5.3 Sampling Procedure - Air Samples

- 5.3.1 Place a MCE filter and a cellulose backup pad in each two- or three-piece cassette. Seal each cassette with a gel band.
- 5.3.2 Calibrate each personal sampling pump with a prepared cassette in-line to within  $\pm 10\%$  of the recommended flow rate of 2 L/min.
- 5.3.3 Attach prepared cassettes to calibrated sampling pumps (the backup pad should face the pump) and place in appropriate positions on the employees or the workplace areas. Whenever possible, collect samples using the air volumes listed below:

<u>Suspected Analyte</u>	<u>Recommended Air Volume</u>
All analytes except Ag	480 L
If Ag is also suspected	960 L
STEL or Ceiling for Be	60 L (STEL), 10 L (Ceiling)

- 5.3.4 If the filter becomes overloaded with particulate while sampling, another filter cassette should be prepared. Consecutive samples using shorter sampling periods should be taken if overloading occurs.
- 5.3.5 Place plastic end caps on each cassette after sampling.
- 5.3.6 Attach an OSHA-21 seal around each cassette in such a way as to secure the end caps.

#### 5.4 Sampling Procedure - Wipe Samples

- 5.4.1 Wear clean, impervious, disposable gloves when taking each wipe sample to prevent sample contamination.
- 5.4.2 Moisten the wipe filters with deionized water prior to use.
- 5.4.3 If possible, wipe a surface area covering 100 cm<sup>2</sup>.
- 5.4.4 Fold the wipe sample with the exposed side in.
- 5.4.5 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.

#### 5.5 Sampling Procedure - Bulk Samples

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

- 1) a high-volume filter sample,
- 2) a representative settled dust (i.e. rafter) sample,
- 3) a sample of the bulk material in the workplace.

Transfer the bulk material 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 Shipment

- 5.6.1 When other compounds or elements are known or suspected to be present in the sampled air, such information should be transmitted with the sample.
- 5.6.2 Request ICP/Solder analysis. If silver is suspected in any of the samples, request ICP/Solder-Silver analysis.
- 5.6.3 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 an actual sample is not taken.
- 5.6.4 Send the samples to the laboratory with the OSHA-91A paperwork.
- 5.6.5 Ship bulk samples separately from air samples. They should be accompanied by Material Safety Data Sheets, if available. Check current shipping restrictions and ship to the laboratory by the appropriate method. The type of bulk sample should be stated on the OSHA-91A and cross-referenced to the appropriate air sample(s).

### 6. Analysis

#### 6.1 Safety Precautions

- 6.1.1 Handle reagents and samples carefully. Use protective equipment such as: gloves, laboratory coats, safety glasses, and an exhaust hood. Wear a fit-tested respirator if necessary. Clean up spills immediately.
- 6.1.2 Carefully prepare any solutions or samples (especially those containing acid) within a suitable ventilated exhaust hood. The acids used to digest the samples are very corrosive and volatile.
- 6.1.3 Do not view the plasma directly.
- 6.1.4 Do not override the radio-frequency generator or torch box safety interlocks.

## 6.2 Equipment

- 6.2.1 Inductively coupled plasma/atomic emission direct-reading spectrometer, nebulizer, cooling unit for torch assembly, computer, and radio-frequency (rf) generator.
- 6.2.2 Automatic sampler.
- 6.2.3 Peristaltic pumps (optional). Use one pump for automatic sampler rinse. Use the other pump for sample introduction into the nebulizer.
- 6.2.4 Mass flow controller (optional). Use the controller to regulate nebulizer argon flow and sample uptake rate.
- 6.2.5 Borosilicate glass Phillips beakers, 125 and 250-mL.
- 6.2.6 Borosilicate glass volumetric flasks, 10-, 25-, 100-mL, and 1-L or 2-L. Use the larger flasks to prepare standard solutions.
- 6.2.7 Hot plate capable of reaching 300 °C.
- 6.2.8 Mixed cellulose ester filters (0.45- $\mu$ m pore size) and a filtering apparatus.
- 6.2.9 Amber colored bottle (1-L) with screw-top cap and Teflon cap liner.
- 6.2.10 Volumetric pipets, glass. Various sizes for sample dilutions and standard preparation.
- 6.2.11 Analytical balance (0.01 mg).

## 6.3 Reagents (Reagent grade or better)

Please see precautions in Sections 6.1.1-6.1.2 before preparing any reagents having an acid matrix.

- 6.3.1 Deionized water (DI H<sub>2</sub>O).
- 6.3.2 Hydrochloric acid (HCl), concentrated (36.5 to 38% w/w).
- 6.3.3 Nitric acid (HNO<sub>3</sub>), concentrated (69 to 71% w/w).
- 6.3.4 Reagent blank - sample diluting solution (32% HCl/4% HNO<sub>3</sub> by volume). Prepare as described below:
  - 1) Slowly and carefully add 320 mL concentrated HCl to 500 mL DI H<sub>2</sub>O in a 1-L volumetric flask. Gently swirl the solution and let cool.

- 2) Slowly and very carefully add 40 mL concentrated HNO<sub>3</sub> to the flask, gently swirl, and let cool.
- 3) Dilute to volume with DI H<sub>2</sub>O. Mix thoroughly.

6.3.5 Stock solutions of 1,000 µg/mL for the various elements.

6.3.6 Argon - grade or quality specified by the ICP instrument manufacturer.

#### 6.4 Standard Preparation

Prepare multielement working and control standard solutions using 1,000 or 10,000 µg/mL stock solutions. Multielement standard concentrations recommended for the JY-32 are shown in Table 1. A suggested control standard is shown in Table 2. Whenever possible, prepare the control standard from different stock solutions than those used for calibration standards. The control standard should contain elements and concentrations reflecting what is expected in the majority of the samples, or problem elements. Any standards containing silver should be prepared in amber colored containers to protect the silver from exposure to light.

The final acid concentration of the working and control standards is 32% HCl/4% HNO<sub>3</sub>. These standards should be stable for at least a year. Also prepare a reagent blank (32% HCl/4% HNO<sub>3</sub>).

#### 6.5 Sample Preparation

The final acid concentration for the different sample matrices should be 32% HCl/4% HNO<sub>3</sub>. All of the elements validated are soluble when using the following acid digestion procedures. Filters, backup pads, wipes, and bulks are prepared by the following procedures:

6.5.1 Mixed cellulose ester membrane filters, wipe samples (smear tabs), and contaminated back-up pads.

- 1) Acid-wash the insides of the 125-mL Phillips beakers by refluxing 1:1 HNO<sub>3</sub> using a hot plate in a ventilated hood. Remove the beakers from the hotplate and carefully pour the used 1:1 HNO<sub>3</sub> into an appropriate labeled container. Allow beakers to cool, then rinse several times with DI H<sub>2</sub>O and allow to dry.
- 2) Carefully transfer any loose dust from the cassette into a separately labeled and acid-washed 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 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. Do not use Ghost Wipes to wipe out cassettes for this method. Ensure that blank samples are prepared and analyzed using the same materials and procedures as used for air samples.

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Note: Do not combine sample filters (i.e. if an industrial hygienist has taken two consecutive samples from an employee). Always prepare each filter separately. If necessary, combine the results of consecutive samples.

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- 3) If the backup pad appears to be discolored, it may be due to leakage of air around the filter during sampling.
- 4) Slowly add 8 mL HCl to each beaker and allow to sit for 5 minutes while swirling gently. Add 2 mL HNO<sub>3</sub>.

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Note: Always add the HCl first to prevent conversion of Sn to SnO<sub>2</sub> or Sb to Sb<sub>2</sub>O<sub>3</sub>. Always slowly add the HCl and allow the filter to become saturated before adding the HNO<sub>3</sub>. Loss of Sb or Sn is possible if the procedure is not strictly followed. See reference 8.1 for further details.

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- 5) Digest the filters in the beakers on a hot plate until the solution volume is approximately:

<u>Solution volume</u>	<u>Air volume</u>
1 mL	>400 L
0.5 mL	≤400 L

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**Note: Do not allow the solutions to evaporate to dryness. Loss of analyte may occur.**

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Remove the beakers from the hotplate and allow to cool in the exhaust hood.

- 6) For samples with air volumes >400 L: Carefully add 8 mL HCl to the beaker, and gently swirl. Quantitatively transfer the sample to a clean 25-mL volumetric flask with DI H<sub>2</sub>O rinses. Be sure to rinse the sides of the beaker. Dilute to volume with DI H<sub>2</sub>O.
- 7) For samples with air volumes ≤400 L or if Ag is a requested analyte: Add 3 mL of HCl to the beaker and gently swirl. Quantitatively transfer the sample with DI H<sub>2</sub>O rinses to a 10-mL volumetric flask and then dilute to volume with DI H<sub>2</sub>O.
- 8) If particulate matter is present after dilution, filter the solution through a 0.45-μm MCE filter contained in a filtering apparatus. Save the filtrate. Repeat the digestion procedure above for the filter containing the particulate.

#### 6.5.2 Polyvinyl Chloride (PVC) Filter Samples

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Note: Polyvinyl chloride filters are not routinely used for solder sample collection and analysis by ICP. In some cases an industrial hygienist will sample for total or respirable dust using PVC filters and also submit these samples for ICP analysis. The PVC filter will not be completely digested using this procedure.

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For PVC filters, proceed as in Section 6.5.1, Step 1. Since the PVC filter will not completely digest, thoroughly rinse the filter residue with DI H<sub>2</sub>O during quantitative transfer of the sample solution.

#### 6.5.3 Bulk samples

- 1) Weigh a representative portion of the bulk sample and transfer to an acid-washed 250-mL Phillips beaker.



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.

- 2) Slowly add 16 mL HCl and allow to sit for 5 minutes while swirling gently. Then add 4 mL HNO<sub>3</sub>.
- 3) Digest the bulk until approximately 4 mL of sample solution remains. Remove the sample from the hot plate and allow to cool.
- 4) Slowly and carefully add 32 mL of HCl.
- 5) Quantitatively transfer the sample to a 100-mL volumetric flask with DI H<sub>2</sub>O rinses, and gently swirl. Dilute to volume with DI H<sub>2</sub>O.
- 6) If particulate matter is present after dilution, filter the solution through a 0.45- $\mu$ m MCE filter contained in a filtering apparatus. Save the filtrate. Repeat the digestion procedure above for the filter containing the particulate.

## 6.6 Instrument Startup and Calibration

Follow the instrument manufacturer's instructions for instrument start-up and calibration. An example of ICP operating parameters for the JY-32 with a Meinhard-type nebulizer is shown below. These settings may vary from instrument to instrument:

Gas used	Argon	
Gas flow	Plasma	12 to 16 L/min
	Nebulizer	*
	Auxiliary plasma	0.14 to 0.18 L/min
Power (rf)	Incident	1.1 kW
	Reflected	<5 W
Observation height	Plasma	15 mm above work coil
Wash time	With automatic sampler	60 s
	Without automatic sampler	10 s
Number of exposures	Standards & samples	5
Integration time	Peak signal	10 s
Nebulizer	Solution uptake rate	0.8 to 1.6 mL/min
	Pressure	~30 psig
Mass flow controller	Flow rate range	varies*

\* Nebulizer is controlled by a mass flow controller. The nebulizer flow will vary depending on the type in use.

- 6.6.1 Profile the instrument before beginning the calibration and analysis. Follow the standard operating procedure (SOP) (8.3) or manufacturer's instructions for computer initialization and profiling.
- 6.6.2 A two-point calibration curve is normally obtained by nebulizing the working standards into the plasma and measuring atomic emission intensities. For calibrations using the JY-32, a first-order linear fit of the data is computer calculated and slope and intercept coefficients are obtained. Calibrations should be performed by following the instrument manufacturer's guidelines.

## 6.7 Analytical Procedure

For more details regarding analytical procedures, refer to the SOP (8.3) or instrument manufacturer's software manual(s).

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Note: The samples and standards are made up in 32% HCl/4% HNO<sub>3</sub>. These solutions are very caustic. Care should be exercised during analysis to prevent physical contact with these solutions.

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- 6.7.1 If necessary, calculate detection limits. The manufacturer's software (if available) should be used for determining detection limits. These limits normally do not significantly change during short time spans. A general rule would be to calculate detection limits every 2 to 4 months or when an integral component (nebulizer, torch, mass flow controller, etc.) of the ICP has been replaced or adjusted. A typical calculation of detection limit (DL) in µg is shown:

$$DL = \frac{K \times SDI \times C}{I - I_0} \times S$$

where:

K is Degree of confidence (sigma value)\*

SDI is Standard deviation of reagent blank intensity (I<sub>0</sub>)

C is Concentration of the calibration standard in µg/mL

S is Solution volume in mL

I is Total intensity of standard containing concentration C

I<sub>0</sub> is Background intensity (reagent blank)

\* In most cases K=2 or 3 for qualitative and K=10 for quantitative determinations.

- 6.7.2 Analysis using an automatic sampler is described:

- 1) Fill automatic sample vials with the minimum sample volume needed for one analysis and a potential rerun.
- 2) Load the automatic sampler with labeled standard and sample vials. A multielement working standard should be analyzed after every 5 to 6 samples. A control standard should be occasionally analyzed to ensure proper instrument operation. If an element or elements contained in the control standard are not within specification, the analyst should recalibrate before proceeding with the analysis. A general rule is to use a value less than ±10 or 15% of the known concentration. See Table 2 for additional details.
- 3) Aspirate each sample or standard for approximately 1 minute prior to initiating the exposure cycle. This ensures equilibration in the plasma and minimizes carry-over effects.
- 4) Dilute and reanalyze any samples containing elements (both screened and validated) exceeding the working range. Interelement corrections may not be accurate above the working range. Prepare the dilutions by pipetting an appropriate aliquot from the original solution and dilute with 32% HCl/4% HNO<sub>3</sub>.
- 5) Based on the calibration curve initially obtained, convert the sample intensities to concentrations. Then, using the air volume, solution volume, dilution factor, and sample weight, calculate the concentration for each element analyzed as mg/m<sup>3</sup> (air samples), total micrograms (wipes), or percentage of total weight (bulks) using the equations shown below. Such calculations are usually performed by software programs within the instrument's dedicated computer or by an external data reduction method.

## 6.8 Calculations

6.8.1 The amount of analyte in each sample or blank is calculated as:

$$\mu\text{g A} = (\mu\text{g/mL A}) \times (\text{mL S}) \times (\text{DF}) \quad (1)$$

where:

$\mu\text{g A}$  is Total  $\mu\text{g}$  of analyte in the sample or blank

$\mu\text{g/mL A}$  is Measured concentration of analyte in solution (derived from calibration curve)

mL S is total volume of the solution analyzed

DF is amount of dilution applied to an aliquot of the original solution (ratio of final volume divided by the aliquot volume)

6.8.2 The blank value, if any, is subtracted from each sample:

$$\mu\text{g}_c \text{ A} = \mu\text{g A} - \mu\text{g}_b \text{ A} \quad (2)$$

where:

$\mu\text{g}_c \text{ A}$  is  $\mu\text{g}$  of analyte, blank corrected

$\mu\text{g A}$  is  $\mu\text{g}$  of analyte from equation (1) above

$\mu\text{g}_b \text{ A}$  is  $\mu\text{g}$  of analyte in blank

6.8.3 For air samples, the concentration of analyte in the sample is expressed in  $\text{mg}/\text{m}^3$  analyte for each element or compound analyzed.

$$\frac{\text{mg A}}{\text{m}^3} = \frac{(\mu\text{g}_c \text{ A}) \times (\text{GF})}{\text{Air Volume (L)}} \quad (3)$$

where:

GF is Gravimetric Factor

For those elements having a PEL listed as an oxide, the gravimetric factors are:

1.2447 for ZnO, 1.4298 for  $\text{Fe}_2\text{O}_3$ , 1.7852 for  $\text{V}_2\text{O}_5$

6.8.4 Convert bulk sample analytes to % composition using:

$$\text{Analyte \% (w/w)} = \frac{(\mu\text{g}_c \text{ A})(100\%)}{(\text{Sample wt})(1000 \mu\text{g}/\text{mg})} \quad (4)$$

where:

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

Sample wt is aliquot (in mg) of bulk taken in Section 6.5.3

## 7. Reporting Results

7.1 Wipe sample concentrations are calculated and reported as total micrograms ( $\mu\text{g}_c \text{ A}$ ) or milligrams for each element.

7.2 Bulk sample results are calculated and reported as elemental percent by weight. Although the acid matrix is constant for samples and standards, the differences in the overall sample matrix between a bulk and standard can be large. Therefore, bulk results are reported as approximate values for each element determined.

7.3 Air sample results are reported in units of  $\text{mg}/\text{m}^3$ . Results for analytes having an oxide PEL are reported as  $\text{mg}/\text{m}^3$  of the oxide.

7.4 Determinations of the screened elements or compounds are not routinely reported. Spectral

interference corrections for these analytes are not included, and validations have not been performed. If a sample has a screened analyte over the PEL, the analyst should contact her/his supervisor. Additional sampling or, if possible, additional analysis of the original sample should be performed to quantitate the potential overexposure.

## 8. References

- 8.1 Occupational Safety and Health Administration Technical Center: ICP Backup Data Report (ID-206 Solders) by D.C. Cook. Salt Lake City, UT. 1991.
- 8.2 National Institute for Occupational Safety and Health: NIOSH Manual of Analytical Methods, 2nd ed., Vol. 7 (DHEW/NIOSH Pub. No. 82-100). Cincinnati, OH, 1981. Method No. 351.
- 8.3 Occupational Safety and Health Administration Technical Center: ICP Standard Operating Procedure by J.C. Septon. Salt Lake City, UT. 1988 (unpublished).

Table 1  
Calibration Standards and Wavelengths

No.	Ele	Std Soln	Concn	Wavelength (nm)
1	Ag	1	1.00	328.0
2	Be	1	1.00	313.0
3	Cd	1	10.00	226.5
4	Pb	1	10.00	220.3
5	Cu	2	1.00	324.7
6	Sb	2	10.00	217.5
7	Sn	2	10.00	189.9
8	Zn	2	10.00	213.8
9	Al	3	10.00	396.1
10	As	3	10.00	189.0
11	Ca	3	10.00	393.3
12	Co	3	10.00	237.9
13	Cr	3	10.00	267.7
14	Fe	3	100.00	271.4
15	Mg	3	10.00	279.5
16	Mn	3	10.00	257.6
17	Mo	3	10.00	202.0
18	Ni	3	10.00	231.6
19	Se	3	10.00	196.0
20	Si	3	10.00	212.4
21	V	3	10.00	310.2

where:

Ele Element

Std Soln Number of Calibration standard

Concn Concentration ( $\mu\text{g/mL}$ ) of calibration standard

Notes: STD SOLN 1 - Prepare in an amber-colored glass bottle to protect the Ag from photo-decomposition. STD SOLN 3 - This standard is used only for calibration of the screened elements. The digestion and analysis are not optimized for these elements.

For the ISA JY-32 ICP, calibration is accomplished using a two-point calibration curve with the concentration for each element listed above. A reagent blank was used as the low standard. Each element calibrated is contained in one of three separate calibration standards (STD SOLN). For example, STD SOLN 1 contains Ag, Be, Cd, and Pb.

The three mixed calibration standards were selected because of chemical compatibility and potential interferences. Other combinations of elements or concentrations can be used; however, compatibility and possible interferences have to be considered when combining elements other than the mixtures listed above.

Table 2  
Control Standard

No.	Ele	Upper Conc	Lower Conc	Std Conc
1	Pb	5.75	4.25	5.0
2	Cd	5.75	4.25	5.0
3	Ag	0.575	0.425	0.5
4	Be	0.575	0.425	0.5

where:

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Upper Conc The upper concentration limit in  $\mu\text{g/mL}$

Lower Conc The lower concentration limit in  $\mu\text{g/mL}$

Std Conc The calibration concentration in  $\mu\text{g/mL}$

A control standard should be prepared and analyzed as an analytical monitor of ICP performance. Some instrument manufacturers have instituted analytical software routines which will evaluate control standard results during the analysis. The control standard example listed above is used for the JY-32 ICP. An alternate control mixture can be prepared.

To illustrate the control standard concept, 5  $\mu\text{g/mL}$  Pb is used. For this control standard, Pb has a limit of  $\pm 15\%$  (upper and lower concentration limits of 5.75  $\mu\text{g/mL}$  and 4.25  $\mu\text{g/mL}$ , respectively). If a calculated value greater than  $\pm 15\%$  is obtained while analyzing this standard, the analysis will automatically halt. The ICP operator should then re-calibrate the instrument.