METAL AND METALLOID PARTICULATES IN WORKPLACE ATMOSPHERES (ICP ANALYSIS)

Method Number: ID-125G

Matrix: Air, Wipe, or Bulk

OSHA Permissible Exposure Limits: Permissible Exposure Limits (PELs) are listed in Table 1 for elements commonly found in industrial environments. This method has the capability of sampling and analyzing more than these elements, the number being limited by instrumental capability, as well as digestion solubility and stability.

Collection Procedure: A calibrated personal sampling pump is used to draw a known volume of air through a mixed-cellulose ester membrane filter contained in a styrene cassette.

Minimum Recommended Air Volumes:
Time Weighted Average Samples - 480 L
Short-Term Exposure Limit Samples - 30 L*
Ceiling Samples - 30 L

Recommended Sampling Rate: 2 L/min

Analytical Procedure: Filters are digested with nitric acid, sulfuric acid and hydrogen peroxide. Dissolution of the elements is facilitated by addition of hydrochloric acid. Analysis is performed using Inductively Coupled Argon Plasma-Atomic Emission Spectroscopy (ICAP-AES).

Detection Limits: See Table 2

Validation Level: See Table 3

Precision and Accuracy: See Table 3

Method Classification: Validated analytical method

Chemists: Jerry Septon, Ray Abel, Michael Simmons

Date: November, 1988
Revised: September, 2002

* Take 60-L samples when evaluating STEL exposures to beryllium.

Division of Physical Measurements and Inorganic Analyses
OSHA Technical Center
Salt Lake City, Utah

1. Introduction

1.1 Scope

1.1.1 This method describes the collection and subsequent analysis of airborne metal and metalloid particulates by Inductively Coupled Argon Plasma-Atomic Emission Spectroscopy (ICAP-AES).

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 13 elements (Be, Cd, Co, Cr, Cu, Fe, Mn, Mo, Ni, Pb, Sb, V, and Zn). Other elements can be added to or subtracted from the method. The capability for expanding the analysis to other elements is mainly dependent on laboratory instrumentation and element solubility and stability in the acid matrix used for digestion.

1.2 History

1.2.1 Previous to the introduction of ICAP-AES, samples containing metallic particulates were digested in a variety of ways and analyzed by Atomic Absorption Spectroscopy (AAS) at the OSHA Analytical Laboratory.

1.2.2 A first generation plasma source and spectrometer (Jarrell-Ash Model 975 Atomcomp) was then used by the OSHA Analytical Laboratory. The analytical procedure for this instrument is described in OSHA Method No. ID-125 (8.1).

1.2.3 Procurement of new inductively coupled plasma (ICP) instruments, computers, and software allowed samples to be determined using later technology. This technology includes more sophisticated computer systems for data reduction and instrument control.

1.2.4 When this method was originally written, three different ICP instruments at the OSHA Salt Lake Technical Center (OSHA-SLTC) were used to apply this method:

Jobin-Yvon (JY) Model 32 (Instruments SA, Edison, NJ)
Jarrell-Ash Model 975 Atomcomp* (Thermo Jarrell-Ash Corp., Franklin, MA)
Applied Research Lab. (ARL) Model 3560 (ARL, Sunland, CA)

These instruments are further referred to as ICP1, ICP2, or ICP3, respectively.

The Jarrell-Ash system was upgraded with a new computer, generator, and software in 1989.

This method is applicable to any simultaneous spectrometer. This method was validated using ICP1 and the data is presented in a backup report (8.2). An additional evaluation was performed using ICP3 (8.3).

2. Detection Limits and Working Ranges (8.2)

2.1 OSHA Permissible Exposure Limits (PELs) (8.4) for the elements screened and validated are listed in Table 1. Detection limits and working ranges are in Table 2. All reported detection limits were calculated for 50-mL solution volumes.

2.2 The optimum working range for each element listed in Table 2 extends several orders of magnitude above each detection limit.

3. Method Performance (8.2)

3.1 The precision and accuracy data for the 13 validated elements using ICP1 are listed in Table 3. These values are based on six samples at each concentration level tested. Solutions of the 13 elements were spiked on mixed-cellulose ester filters. These samples were then digested and analyzed using procedures mentioned in this method and in reference 8.2.

3.2 Nine of the thirteen elements reported in Table 3 were spiked at 0.5, 1, and 2 times the PEL, assuming a 120-L air volume. Spikes for manganese were calculated assuming a 30-L air volume. Approximately 200-L air volumes were assumed for Pb, Ni, and Sb.

3.3 The analytical error (AE) at 95% confidence for each element listed in Table 3 was calculated as:

$$\pm AE\% = 100 \times [\,|\text{Mean Bias}| + 2(CV)]$$

Analytical errors for all elements tested were within ±25%; the greatest value was ±18.1% for V. This element was validated near its detection limit.

4. Interferences (8.6)

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.

4.4 The first effect (a) can be minimized by a careful selection of wavelengths for the reported elements. 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 and interference within the working range limits. A spectral interference correction equation typically used by ICP manufacturers is:

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

where: A_i is Correction factor
Cp_i is Concentration of the interfering element

Samples having analyte concentrations above the working range limits should be diluted into range; interelement corrections may not be accurate above the working range. Experimentally determined interelement corrections for the validated elements are listed in reference 8.3.

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

5. Sampling

5.1 Equipment

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). Cassettes, filters (MCE) and backup pads of 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 L/min.

5.1.4 Assorted flexible tubing.

5.1.5 Stopwatch and bubble tube or meter for pump calibration.

5.1.6 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.

5.1.7 Smear tabs, part no. 225-24 (SKC Inc., Eighty Four, PA), or Whatman no. 41 or no. 42 filters (Whatman LabSales Inc., Hillsboro, OR) for wipe sampling.

5.1.8 Gloves, disposable (for wipe sampling).

5.1.9 Ghost Wipes, 4” × 4” 1000/cs Wet with DI Water, part no. SC4250 (Environmental Express, Mt. Pleasant, SC) for wipe sampling.

5.2 Sampling Procedure - Air Samples

Welding fumes and samples requiring sample weights can be characterized using this method. Collect samples on pre-weighed 37-mm polyvinyl chloride (PVC) filters at 2 L/min flow rate. Conduct the welding fume sampling with the filter cassette located inside the welding helmet (8.7). If the free-space inside the hood precludes the use of 37-mm diameter cassettes and filters, 25-mm sampling assemblies with pre-weighed PVC filters can be used. Desiccate and post-weigh each sample and then calculate total welding fume exposure:

$$\text{mg/m}^3 = \frac{\text{net weight }(\mu\text{g}) - \text{net weight of blank }(\mu\text{g})}{\text{air volume (liters)}}$$

and determine compliance with the 5 mg/m^3 TLV for welding fumes. The samples are then analyzed at the laboratory for welding fume/ICP analysis to further characterize the samples.

5.2.1 Place a MCE filter and a cellulose backup pad in each two- or three-piece cassette. Seal each cassette with a gel band.

5.2.2 Calibrate each personal sampling pump with a prepared cassette in-line to approximately 2 L/min flow rate.

5.2.3 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. Collect the samples at about 2 L/min flow rates. Minimum sampling times recommended are:

Recommended Sampling Times

Sample Type	Time
Time Weighted Average Samples	240 min
Short-Term Exposure Limit Samples	15 min*
Ceiling Samples	15 min

* When determining compliance with the STEL for beryllium, take 30-min samples. The analytical sensitivity of a specific analyte may dictate using a larger sampling time.

Note: If soluble compounds (i.e., Cr^{2+}, Cr^{3+}, soluble salts of Al, Fe, Mo, Ni, $ZnCl_2$, etc.) are suspected to be present in the sampled air, take separate samples. Request analysis for the specific compound(s). These samples are analyzed using OSHA Method No. ID-121 and not by this method.

5.2.4 If the filter becomes overloaded while sampling, another filter cassette should be prepared. Consecutive samples using shorter sampling periods should be taken if overloading occurs.

5.2.5 Place plastic end caps on each cassette after sampling.

5.2.6 Attach an OSHA-21 seal around each cassette in such a way as to secure the end caps.

5.3 Sampling Procedure - Wipe Samples

5.3.1 Wear clean, impervious, disposable gloves when taking wipe samples to prevent sample contamination. Change gloves between samples to reduce the possibility of cross contamination.

5.3.2 Moisten Smear Tabs and Whatman filters with deionized water prior to use.

5.3.3 If using a Ghost Wipe remove it from its package and unfold it. Next fold the Ghost Wipe in half and wipe a 10-cm × 10-cm area by starting at the outside edge of the surface, applying firm pressure, wipe the surface and progress towards the center by making concentric squares of decreasing size. Fold wipe in half, with contaminant side in, and wipe the surface again by making concentric squares of decreasing size. Fold the wipe in half, contaminant side in, and wipe surface a third time.

If using a Smear Tab or Whatman filter, wipe a 10-cm × 10-cm area by starting at the outside edge of the surface, applying firm pressure, wipe the surface and progress towards the center by making concentric squares of decreasing size. If possible wipe the area at least 3 times.

5.3.4 Fold the wipe sample with exposed side in.

5.3.5 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.

5.4 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.

5.5 Shipment

When other compounds or elements are known or suspected to be present in the sampled air, such information should be transmitted with the sample(s) to the laboratory.

5.5.1 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 no air is drawn through the blank.

5.5.2 Send the samples to the laboratory with the OSHA 91A paperwork requesting ICP analysis.

5.5.3 Bulk samples should be shipped 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.

6. Analysis

6.1 Safety Precautions

6.1.1 Prepare 1:1 H_2SO_4 in DI H_2O cautiously.

1) Use a 1- or 2-L thick-walled, break- and heat-resistant bottle.
2) Wear thick rubber gloves, plastic apron, laboratory coat, and face shield.
3) Add 500 mL DI H_2O to the bottle.
4) Place the bottle over the drain in a sink which has a slot vent to provide ventilation. Begin running cold tap water over the side of the bottle, being careful not to get any tap water in the bottle. Let the level of water rise in the sink to provide cooling of the bottle.
5) Carefully and slowly begin adding 500 mL concentrated H_2SO_4 to the DI H_2O. Add a small quantity, swirl to mix, and allow contents to cool. Do **NOT** allow boiling of solution within the container.
6) After the acid has been added, loosely cap the bottle and allow it to remain in the sink with the water running for at least 15 min. Allow the solution to cool to room temperature.
7) A thick-walled beaker, Teflon-coated stirring bar, electronic stirrer and a ventilation hood can also be used to prepare 1:1 H_2SO_4 if precautions are taken to prevent solution overheating and splattering.

6.1.2 Digest all samples within a suitable exhaust hood.

6.1.3 To prevent splattering, add H_2O_2 (30%) to beakers in 2 to 3 drop groups.

6.1.4 Perchloric acid added to organic substances can produce fires and/or explosions. If $HClO_4$ solutions darken in color while heating, immediately remove beakers from the hotplate and carefully add a small amount of HNO_3. Only use $HClO_4$ in exhaust hoods designed and reserved for $HClO_4$ use.

6.1.5 Do not directly view the plasma.

6.1.6 Do not override the rf generator or torch box safety interlocks.

6.2 Equipment

6.2.1 Inductively coupled argon plasma/atomic emission direct-reading spectrometer, cooling unit for torch assembly, computer, and radio-frequency (rf) generator.

6.2.2 Nebulizer.

6.2.3 Automatic sampler.

6.2.4 Peristaltic pumps (optional). Use one pump for automatic sampler rinse. Use the other pump for sample introduction into the nebulizer.

6.2.5 Mass Flow Controller (optional). Use the controller to regulate nebulizer argon flow and sample uptake rate.

6.2.6 Borosilicate glass conical beakers, 125- and 250-mL.

6.2.7 Borosilicate glass volumetric flasks, 25-, 50-, 100-, 250-mL, and 1- or 2-L. Use the larger flasks for standard preparation.

6.2.8 Thick walled, 1- or 2-L heat- and break-resistant bottle.

6.2.9 Mixed cellulose ester filters (0.45-µm pore size) and a filtering apparatus. Use this system to remove any insoluble particulates from sample solutions.

6.2.10 Hot plate capable of reaching 300 °C.

6.2.11 Volumetric pipets, glass of various sizes.

6.2.12 Analytical balance (0.01 mg).

6.3 Reagents (reagent grade or better)

6.3.1 Deionized water (DI H_2O).

6.3.2 Concentrated sulfuric (H_2SO_4), hydrochloric (HCl), nitric (HNO_3), and perchloric ($HClO_4$) acids.

6.3.3 Prepare 1:1 H_2SO_4 (V/V) solutions as described in Section 6.1.1.

6.3.4 Sample dilution solution or reagent blank (8% HCl/4% H_2SO_4):

In an exhaust hood, slowly and carefully add 40 mL concentrated H_2SO_4 to approximately 500 mL of DI H_2O contained in a thick-walled, heat- and break-resistant bottle. Gently stir and allow the solution to cool to room temperature. Slowly and carefully add 80 mL concentrated HCl, allow to cool, and dilute to 1 L with DI H_2O.

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

6.3.6 Hydrogen peroxide, (H_2O_2), 30%.

Note: Some manufacturers use organotin compounds to stabilize H_2O_2. Since Sn is one of the elements screened, use H_2O_2 that does not contain this type of stabilizer.

6.3.7 Argon - quality as specified by the ICP manufacturer.

6.4 Standard Preparation

Prepare multielement calibration standards (STD) and continuing calibration verification (CCV) solutions (see Addendum A for examples of combinations) using 1,000 or 10,000 µg/mL stock solutions. An independent calibration verification (ICV) standard is a mixture of elements whose concentrations are within their respective analytical linear ranges and is used to verify the calibration standards. Whenever possible, prepare the ICV standard from different stock solutions than those used for calibration standards. The ICV standard should contain elements and concentrations reflecting what is expected in the majority of the samples, or problem elements. The final acid concentration of the STD, CCV and ICV standards is 8% HCl/4% H_2SO_4. These standards should be stable for at least 6 months.

6.5 Sample Preparation

The final acid concentration for the different sample matrices should be 8% HCl/4% H_2SO_4. All of the elements validated are soluble when using the following acid digestion procedures. Other elements not included in the validated element list (Table 3) should be evaluated for solubility and stability before using these procedures.

Note: Requests for analysis of compounds which have a PEL that specifically addresses the soluble fraction (i.e., Fe, Ni, Mo, etc.) are analyzed using OSHA Method No. ID-121 and not by this method.

Filters, backup pads, wipes, and bulks are prepared by the following procedures:

6.5.1 Mixed-cellulose ester (MCE) membrane filters

1) If the beakers used for the digestion have not been cleaned using a appropriate automated system, clean the insides of the 125-mL conical beakers by refluxing 1:1 HNO_3 using a hot plate in a ventilated hood. Carefully pour the used 1:1 HNO_3 into an appropriate labeled container. Allow beakers to cool, then rinse several times with DI H_2O and allow to dry.
   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_2O 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_2O 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.
2) For samples with air volumes >200 L: Add 4 mL of 1:1 H_2SO_4, followed by 2 mL of concentrated HNO_3 to each beaker containing the filter sample. For samples with air volumes ≤200 L: Add 2 mL of 1:1 H_2SO_4, followed by 2 mL of concentrated HNO_3.
3) To facilitate the digestion, allow the filters to sit at least an hour in the 1:1 H_2SO_4. Add several drops of H_2O_2 (30%) to each beaker before placing it on the hotplate.
4) Heat the beakers on a hot plate for approximately 10 min. The solutions should turn brown.
5) Cautiously add H_2O_2 in 2- to 3-drop groups until each solution becomes clear, colorless, or slightly yellow (the color is dependent on the concentration and type of analyte present).
6) Heat several more minutes until dense, white fumes of SO_3 just become evident. Remove the beakers from the hotplate and allow to cool.
7) Slowly and carefully add the following amount of concentrated HCl.

   (**CAUTION: SPLATTERING MAY OCCUR IF THE HCL IS ADDED TOO RAPIDLY OR THE H_2SO_4 SOLUTION IS STILL HOT**):

   Acid Addition Data

Acid Volume	Air Volume
2 mL	≤200 L
4 mL	>200 L

8) Rinse the sides of the beakers with DI H_2O and return the beakers to the hotplate. Heat the beakers until near boiling to promote solubilization of all elements present. Remove the beakers from the hotplate and allow to cool.
9) Quantitatively transfer the solutions into volumetric flasks using DI H_2O. For samples having air volumes >200 L, dilute to 50 mL; volumes ≤200 L, dilute to 25 mL, if the filter contains a lot of material, dilute to 50 mL.

6.5.2 Backup Pads

Contamination of the backup pad may be due to leakage of air around the filter during sampling.

6.5.3 Wipe or Polyvinyl Chloride (PVC) Filter Samples

Polyvinyl chloride filters can be used for sample collection and analysis by ICP. The industrial hygienist may sample for gravimetric determinations of total dust or total welding fumes using PVC filters and also submit these samples for ICP analysis if a sample weight is required.

---

1) If the beakers used for the digestion have not been cleaned using a appropriate automated system, reflux 1:1 HNO_3 in 250-mL (for wipes) or 125-mL (for PVC filters) conical beakers, empty and allow to cool. Rinse the beakers several times with DI H_2O and allow to dry.
2) Place each filter or wipe in a separate washed beaker.
3) Add the appropriate amount of 1:1 H_2SO_4 as listed below:

Acid Addition Data

Acid Volume*	Sample Type
8 mL	Wipe
4 mL	PVC filter (>200 L Air Volume)
2 mL	PVC filter ($\leq$200 L Air Volume)

* Concentrated HCl or 1:1 H_2SO_4

4) Add 10 mL concentrated HNO_3 to each beaker. Place the beakers on the hotplate.
5) Add H_2O_2 in 2- to 3-drop groups. PVC filters and wipes require more H_2O_2 for digestion than MCE filters.
6) If $HClO_4$ digestion precautions are followed (Section 6.1.4), 1 to 2 mL of $HClO_4$ can also be used to complete digestion. The PVC filter will not completely digest if $HClO_4$ is not added; therefore, thoroughly rinse the filter residue with DI H_2O during quantitative transfer of the sample solution.
7) Allow digested samples to cool and carefully add the same volume of concentrated HCl as the 1:1 H_2SO_4 used in Section 6.5.3, Step 3. above.
8) Rinse the sides of the beaker with DI H_2O and then heat to near boiling.
9) After beakers have cooled to room temperature, dilute digested wipe solutions to 100 mL. Dilute PVC filter solutions to final volumes as stated in Section 6.5.1, Step 9.

6.5.4 Ghost Wipes

1) If the beakers used for the digestion have not been cleaned using a appropriate automated system, reflux 1:1 HNO_3 in 125-mL conical beakers, empty and allow to cool. Rinse the beakers several times with deionized water and allow them to dry.
2) Place each Ghost Wipe in a separate washed beaker.
3) Add 4 mL of 1:1 H_2SO_4 to each beaker. Let sample sit for 5 minutes.
4) Add 2 mL of deionized water followed by 2 mL concentrated HNO_3 to each beaker. Let samples sit for 15 minutes. After 15 minutes, up to 8 mL more of concentrated HNO_3 can be added to facilitate digestion, especially if samples are heavily loaded. Place the beakers on the hotplate.
5) When solutions turns brown or black cautiously add H_2O_2 in 2- to 3-drop groups until each solution becomes clear, colorless, or slightly yellow (the color is dependent on the concentration and type of analyte present). Ghost Wipes may require up to 10 mL H_2O_2 before solution clears.
6) Heat solutions several more minutes until dense, white fumes of SO_3 just become evident. Remove beakers from hotplate and allow them to cool.
7) After samples have cooled carefully add 4 mL of concentrated HCl to each beaker.

8) Rinse the sides of the beaker with deionized water and then heat again on hot plate until samples are near boiling.
9) After beakers have cooled to room temperature, dilute digested Ghost Wipes solutions to 50 mL.

6.5.5 Bulks

1) 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.
2) Measure by volume or weight an appropriate aliquot of any liquid bulk sample. Weigh the appropriate amount of any solid bulk sample. Weigh an aliquot of any paint bulk by placing a small amount on a MCE filter, allow to air dry then take the dry weight.

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 needed, use a mortar and pestle to grind any nonhomogenous particulate bulk samples in an exhaust hood.

After measuring, transfer the aliquot to a previously cleaned or acid- washed 250-mL conical beaker.

3) Add 20 mL of 1:1 H_2SO_4 and digest on a hotplate. Hydrogen peroxide (dropwise) and a few mLs of HNO_3 can be carefully added to break up the matrix.

Caution: Do not add the HNO_3 to wet bulk materials containing organic solvents. Significant reactions could occur.

4) Remove the beaker from the hotplate and allow to cool. Carefully add 20 mL of concentrated HCl and then heat the solution to near boiling.
5) Allow to cool and quantitatively transfer to a 250-mL volumetric flask. Dilute to volume with DI H_2O.

6.5.6 Air, wipe, and bulk samples: If particulates are present in any of the sample solutions, filter this solution through a MCE filter (0.45-µm pore size) and then re-digest the particulate and filter. Save the filtrates for analysis.

6.6 Instrument Startup and Calibration

Follow the manufacturer's instructions for instrument start-up and calibration. An example of ICP operating parameters is shown below. These settings will vary from instrument to instrument:

Gas Used	Argon	
Gas Flow	Plasma	12 - 16 L/min
(Rotameter settings)	Nebulizer	*
	Auxiliary Plasma	0.14 - 0.18 L/min
RF Power	Incident	1.1 kW
	Reflected	< 5 W
Observation Height	Plasma	15 mm above work coil
Integration Time	Peak Signal	3 to 10 s
Wash Time	Automatic Sampler (AS)	60 s
	Without AS	10 s
Number Of Exposures	Standards & Samples	2 to 10
Nebulizer*	Solution Uptake Rate	0.8 - 1.6 mL/min
	Pressure	~30 psig
Mass Flow Controller	Flow Rate Range	varies*

* This flow will vary depending on the type of nebulizer in use.

6.6.1 Profile the instrument before beginning the calibration and analysis. Follow the Standard Operating Procedure (SOP) (8.7) or manufacturer's instructions for computer initialization and profiling.

6.6.2 Obtain a two-point calibration curve by nebulizing the working standards into the plasma and measuring atomic emission intensities. For most instruments, a first-order linear fit of the data is computer calculated and slope and intercept coefficients are obtained. Perform calibrations by following the instrument manufacturer's guidelines.

6.6.3 See Addendum A for the proper sequence of standards and samples during the analysis.

6.7 Analytical Procedure

For more details regarding analytical procedures, refer to the instrument manufacturer's software manual(s) or the SOP (8.7).

6.7.1 If necessary, determine detection limits using the manufacturer's software (if available). These limits normally do not significantly change during short time spans. A general rule is to recalculate detection limits 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) is shown:

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

where:
S is Solution volume in mL
K is Degree of confidence (sigma value)*
SDI is Standard deviation of reagent blank intensity (I_o)
C is Concentration of the calibration standard in µg/mL
I is Total intensity of standard containing concentration C
I_o is Background intensity (reagent blank)
* In most cases, K=2 or 3 for qualitative and K=10 for quantitative determinations.

Reporting limits may be equal to or larger than the calculated detection limits. Reporting limits should be verified annually for each matrix.

6.7.2 Analysis using an automatic sampler is described below:

1) Fill automatic sample vials to the minimum sample volume 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 (a general rule is to use a value less than ±10 to 15% of the known concentration), the analyst should recalibrate before proceeding with the analysis.
3) Aspirate each sample or standard for approximately 1 min 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 (Table 2). In particular, notice the reduced upper limit (8 µg/mL) for lead. 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 8% HCl/4% H_2SO_4.
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/m³ (air samples),

total micrograms (wipes), or percentage of total weight (bulks) using the equations shown below.

6.8 Calculations

6.8.1 Total amount of analyte in the sample:

$$\mu g\ A = (\mu g/mL\ A) \times (mL\ S) \times (DF) \qquad (1)$$

where:
µg A is Total µg of analyte in the sample
µg/mL A is Measured concentration of analyte in sample 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:

$$\mu g_c\ A = \mu g\ A - \mu g_b\ A \qquad (2)$$

where:
μg_c A is µg of analyte, blank corrected
μg_b A is µg of analyte in blank

6.8.3 For air samples, the concentration of analyte in the sample is expressed in mg analyte per cubic meter for each element or compound analyzed:

$$\frac{mg}{m^3} = \frac{(\mu g_c\ A) \times (GF)}{[\text{Air Volume (L)}]} \qquad (3)$$

where:
GF is Gravimetric Factor

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

1.4298 for Fe_2O_3 1.2447 for ZnO 1.7852 for V_2O_5

6.8.4 Convert bulk sample analytes to % composition using:

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

where:
μg_c A is analyte amount (µg)
Sample wt is aliquot (in mg) of bulk taken in Section 6.5.4

7. Reporting Results

7.1 Air sample results are reported as mg/m³. Results for analytes having a PEL as an oxide are reported as mg/m³ of the oxide.

7.2 Wipe sample concentrations are calculated and reported as total micrograms for each element.

7.3 Bulk sample results are calculated and reported as elemental percent by weight (or volume if liquid aliquots were used). Due to differences in sample matrices between bulks and standards, bulk results are approximate for each element determined. (Elemental values are to be reported for all bulk analyses, do not use a gravimetric factor.)

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 Analytical Laboratory: *OSHA Analytical Methods Manual* (USDOL/OSHA-SLCAL Method No. ID-125). Cincinnati, OH: American Conference of Governmental Industrial Hygienists (Pub. No. ISBN: 0-936712-66-X), 1985.

8.2 Occupational Safety and Health Administration Technical Center: *ICP Backup Data Report (ID-125G)* by J.C. Septon. Salt Lake City, UT. Revised 1991.

8.3 Occupational Safety and Health Administration Technical Center: *ICP Backup Data Report (ARL 3560)* by J.C. Septon. Salt Lake City, UT. In progress.

8.4 "Toxic and Hazardous Substances," *Code of Federal Regulations* Title 29, Pt. 1910.1000, Subpart Z. 1987. pp 676-682.

8.5 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.

8.6 United States Department of Labor, OSHA: "Memorandum, Sampling for Welding Fumes" by Patricia Clark, Director Designate, Directorate of Compliance Programs. United States Department of Labor, OSHA, Washington, DC, February 14, 1989. [Memo].

8.7 Occupational Safety and Health Administration Analytical Laboratory: *ICP Standard Operating Procedure* by J.C. Septon. Salt Lake City, UT. 1988 (unpublished).

Table 1
Air Contaminants - OSHA Permissible Exposure Limits*

Element	Substance Exposed to	PEL (mg/m^3) TWA	PEL (mg/m^3) Ceiling
Ag**	Metal and soluble compounds (as Ag)	0.01	
As**	Inorganic compounds (as As) see 29 CFR 1910.1018		
Be#	Beryllium and compounds (as Be)	0.002	0.005##
Ca**	Calcium oxide	5	
Cd	Fume	0.1	0.3
	Dust	0.2	0.6
	(See 29 CFR 1910.1027)		
Co	Metal, dust and fume (as Co)	0.1	
Cr	Metal and insoluble salts (as Cr)	1	
Cu	Fumes (as Cu)	0.1	
	Dusts and mists (as Cu)	1	
Fe	Dicyclopentadienyl iron total dust	15	
	Iron oxide fume (as Fe_2O_3)	10	
Fe, V	Ferrovanadium	1	
Mg**	Magnesium oxide fume total particulate	15	
Mn	Mn compounds (as Mn)		5
	Mn fume (as Mn)		5
Mo	Insoluble compounds (as Mo) total dust	15	
Ni	Metal and insoluble compounds (as Ni)	1	
	Soluble compounds (as Ni)	1	

Element	Substance Exposed to	PEL (mg/m^3)	
		TWA	Ceiling
Pb	Inorganic (see 29 CFR 1910.1025)	0.05	
Sb	Sb and compounds (as Sb)	0.2	
Se**	Se and compounds (as Se)	2	
Sn**	Inorganic compounds except oxides (as Sn)	0.1	
Te**	Te and compounds (as Te)	5	
Zn	Zinc oxide fume	15	
	Zinc oxide total dust	15	
	Zinc stearate total dust		

* From reference 8.4.

** Elements screened - PELs are listed for information only, because the screened elements are not digested or analyzed using optimum conditions.

\# Beryllium also has a Peak PEL of 0.025 µg/m^3.

\## Both the Ceiling Limits and the STEL for beryllium are for a maximum 30-min duration.

Note: Compounds having total and respirable dust PELs of 15 and 5 mg/m^3, 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* and Upper Limits

Analyte	Qual Det Limit (µg)	Quan Det Limit (µg)	Upper Limit (µg/mL)
Ag**	1.1	3.8	**
Al**	6.8	23	200
As**	1.7	5.7	20
Be	0.013	0.043	5
Ca**	0.79	2.6	20
Cd	0.14	0.47	50
Co	1.2	4.0	100
Cr	0.40	1.3	50
Cu	0.64	2.1	50
Fe	8.9	30	200
Mg**	2.1	7.1	20
Mn	0.061	0.20	15
Mo	0.52	1.7	100
Ni	0.59	2.0	100
Pb	2.1	7.0	8
Sb	4.2	14	100
Se**	3.2	11	20
Si**	2.4	8.1	20
Sn**	3.5	12	**
V	0.57	1.9	100
Zn	0.14	0.47	50

* Calculations are based on a 50-mL solution volume and equations listed in Section 6.7.1. Each detection limit is dependent on the spectral wavelength and order used for analysis.

** Screened elements - Limits are approximate - the digestion and analysis are not optimized for these elements.

Both the detection limits and upper limits were determined using ICP1 (JY-32 ICP). Performance may vary from instrument to instrument. Upper limits are the upper linear range for each element. These were determined using a linear model (8.2).

Table 3
Precision and accuracy Data* for Spiked Samples

Element	CV	Bias	Range (µg)	Analytical error (±%)
Be	0.010	0.010	0.12-0.48	2.9
Cd[1]	0.014	-0.005	6-24	3.2
Co	0.020	-0.040	6-24	8.1
Cr[2]	0.015	-0.074	60-240	10.3
Cu[3]	0.017	0.054	60-240	8.7
Fe	0.018	-0.012	420-1680	4.8
Mn	0.032	0.077	75-300	14.1
Mo[4]	0.047	-0.029	225-900	12.3
Ni	0.025	0.017	105-420	6.6
Pb	0.040	0.04	5-20	12.1
Sb	0.014	0.012	60-240	3.9
V	0.045	0.090	3.5-14	18.1
Zn	0.007	0.014	240-960	2.9

* Reported values were obtained from ICP1 (ISA JY-32 ICP). Results may vary from instrument to instrument.
The following exceptions were used when calculating spike amounts:
[1] PEL for fume (0.1 mg/m^3) used
[2] PEL for insoluble forms (1 mg/m^3) used
[3] PEL for dust form (1 mg/m^3) was used
[4] 5 mg/m^3 target concentration was used. This was performed because a very large amount of spike would be necessary if the 15 mg/m^3 Transitional PEL for Mo was used
CV Coefficient of Variation

Addendum A

Instrument Calibration & Quality Control Sequence

QC Function	**Acronym**	**Frequency**	**Acceptance Criteria**	**Analytes & Nominal Concentrations (ppm)**
Initial Calibration[a]	**CB**	At the beginning of each analytical sequence, *and* as required by failing performance checks	n/a	
	STD - A		RSD < 2% (replicate readings)	*Target analytes reported to clients*: 1 - Be 5 - Cr, Cu, Pb, V 10 - Cd, Co, Mn, Mo, Ni, Sb, Zn 100 - Fe
	STD - B			*Anticipated interfering analytes*: 1 - Ag, Pt 10 - Al, As, Ce, Mg, Nb, Se, Sn, Ti
Initial Performance Checks	**ICV**	Immediately following the initial calibration	90 - 110 % Recovery	0.5 - Be 2.5 - Cr, Cu, Pb, V 5 - Cd, Co, Mn, Mo, Ni, Sb, Zn 50 - Fe
	CCV	Immediately following each calibration event in the analytical sequence		0.5 - Be 2.5 - Cr, Cu, Pb, V 5 - Cd, Co, Mn, Mo, Ni, Sb, Zn 50 - Fe

(IB must follow ICS)

QC Function	Acronym	Frequency	Acceptance Criteria	Analytes & Nominal Concentrations (ppm)
	RLV		75 - 125 % Recovery	0.002 - Be 0.01 - Cd 0.03 - V 0.05 - Co, Cu, Mn, 0.1 - Pb 0.2 - Cr 0.5 - Fe, Mo, Ni, Sb, Zn
	ICS		85 - 115 % Recovery (Target Analytes Only)	0.2 - Be, Cd 1 - Pb, V, Ag 5 - Co, Cr, Co, Mn, Mo, Ni, Sb, Zn, As, Se, Sn 10 - Ce, Nb, Pt 200 - Fe, Al, Mg, Ti
	IB		|X| < Reporting Limit (Target Analytes Only)	
Continuing Performance Checks	**CCV**	Every 10 field samples	90 - 110 % Recovery	0.5 - Be 2.5 - Cr, Cu, Pb, V 5 - Cd, Co, Mn, Mo, Ni, Sb, Zn 50 - Fe
	RLV		75 - 125 % Recovery	0.002 - Be 0.01 - Cd 0.03 - V 0.05 - Co, Cu, Mn, 0.1 - Pb 0.2 - Cr 0.5 - Fe, Mo, Ni, Sb, Zn
	IB		|X| < Reporting Limit (Target Analytes Only)	
Sequence Termination Performance Checks	**CCV**	At the end of the analytical sequence	90 - 110 % Recovery	0.5 - Be 2.5 - Cr, Cu, Pb, V 5 - Cd, Co, Mn, Mo, Ni, Sb, Zn 50 - Fe
	RLV		75 - 125 % Recovery	0.002 - Be 0.01 - Cd 0.03 - V 0.05 - Co, Cu, Mn, 0.1 - Pb 0.2 - Cr 0.5 - Fe, Mo, Ni, Sb, Zn
	IB		|X| < Reporting Limit (Target Analytes Only)	

Terms:

STD	Calibration Standard(s)
CB	Calibration Blank
ICV	Independent Calibration Verification (alternate source reference material)
CCV	Continuing Calibration Verification
IB	Instrument Blank (part of continuing calibration verification)
RLV	Reporting Limit Verification
ICS	Interference Check Sample

Target Analyte: any analyte that is reported to clients in the final report of results

[a] The linear calibration range for target analytes may be established/verified for each analytical sequence by analyzing a high level calibration verification standard at the end of the analytical sequence. In order to report sample results that are measured at levels between this high standard and the initial calibration standard, the high standard must exhibit a recovery between 90 and 110%.

Addendum B
Ghost Wipe Backup Data

1. Backup Data

General background information about the determination of detection limits and reproducibility of the overall procedure is found in the "Evaluation Guidelines for Surface Sampling Methods".[1] The Guidelines define analytical parameters, specific laboratory tests, statistical calculations and acceptance criteria.

Sample preparation of Ghost Wipes was performed as described in Section 6.5.4 of OSHA Method ID-125G. Instrument analytical conditions used are described in Section 1.9 of this document. Lot number of Ghost Wipes used was 9901, manufactured on May 20, 1999.

1.1 Detection Limit of the overall procedure (DLOP) and reliable quantitation limit (RQL).

The DLOP is measured as mass per sample. Seven Ghost wipes were spiked with descending increments of analyte. These spiked Ghost Wipes, and a sample blank, were analyzed and the data obtained used to calculate the required parameters (standards error of estimate and the slope) for the calculation of the DLOP.

The RQL is considered the lower limit for precise quantitative measurements. It is determined from the regression line parameters obtained for the calculation of the DLOP, providing 75% to 125% of the analyte is recovered.

The analyte target, calculated DLOPs, and RQLs for the 13 validated analytes are shown below:

Table 1.1.1
Target Concentrations, DLOPs, and RQLs

analyte and wavelength	target concn (µg)	DLOP (µg) in 50 mL	RQL (µg) in 50mL	recovery at RQL* (%)
Be 313.107	4	0.0052	0.017	112.8
Cd 214.440	10	0.053	0.18	94.6
Co 228.616	200	0.32	1.1	101.2
Cr 267.716	1000	1.0	3.4	101.2
Cu 324.752	200	0.45	1.5	106.0
Fe 238.204	2000	1.5	5.1	105.5
Mn 257.610	400	0.29	0.97	101.1
Mo 202.031	1000	0.8	2.7	103.8
Ni 232.003	1000	0.92	3.1	97.5
Pb 220.353	100	0.55	1.8	81.9
Sb 206.836	1000	1.5	5.1	105.8
V 292.402	56	0.08	0.29	103.1
Zn 213.857	800	2.2	7.3	95.4

* Percent recovery at or near the RQL.

---

[1] Lawrence, R. *Evaluation Guidelines for Surface Sampling Methods*; OSHA Salt Lake Technical Center, U.S. Department of Labor: Salt Lake City, UT, 2001.

Table 1.1.2
Detection Limit of the Overall Procedure
for Beryllium

mass per sample (µg)	intensity
0	-7.4
0.012	64.7
0.025	121.9
0.05	224.5
0.1	436.1
0.15	666.4
0.2	895.1
0.25	1110.4

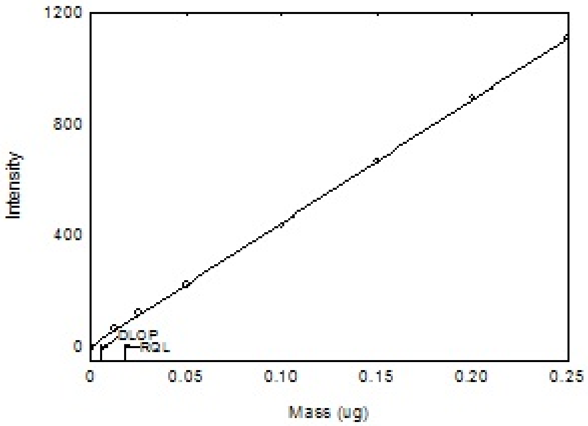

Figure 1.1.2. Plot of data to determine DLOP/RQL for Beryllium. (Y = 4436.7X + 2.2)

Table 1.1.3
Detection Limit of the Overall Procedure
for Cadmium

mass per sample (µg)	intensity
0	0.4
0.031	4.5
0.062	4.6
0.12	9
0.25	15.8
0.38	24.6
0.5	32.5
0.63	43

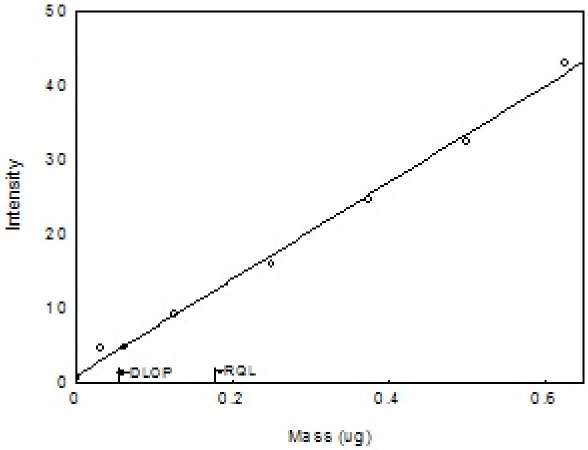

Figure 1.1.3. Plot of data to determine the DLOP/RQL for Cadmium. (Y = 65.2X + 0.7)

Table 1.1.4
Detection Limit of the Overall Procedure
for Cobalt

mass per sample (µg)	intensity
0	-2.1
0.62	26.7
1.25	52.9
2.5	104.8
5	206.7
7.5	318.5
10	428.2
12.5	519.8

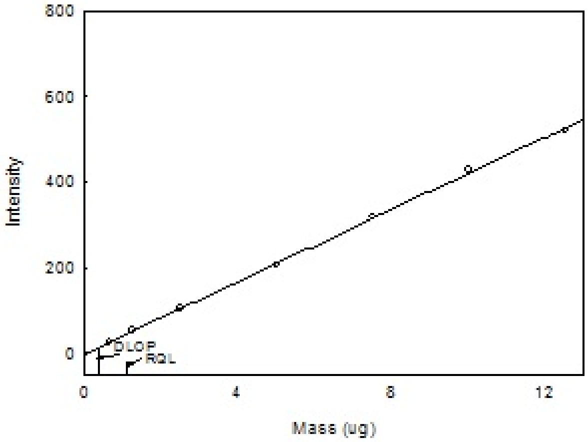

Figure 1.1.4. Plot of data to determine DLOP/RQL for Cobalt. (Y = 42.2X + 0.4)

Table 1.1.5
Detection Limit of the Overall Procedure for Chromium

mass per sample (µg)	intensity
0	46.3
3.1	673.8
6.2	1332.1
12.5	2749
25	5258
37.5	8015
50	10769.5
62.5	13546.3

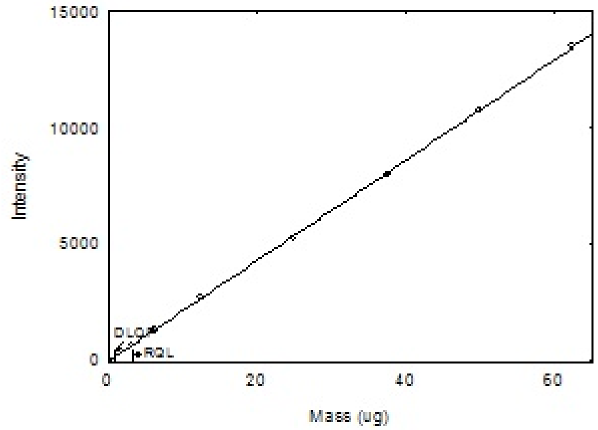

Figure 1.1.5. Plot of data to determine DLOP/RQL for Chromium. (Y = 215.6X - 6.5)

Table 1.1.6
Detection Limit of the Overall Procedure for Copper

mass per sample (µg)	intensity
0	1050.3
0.62	1404
1.3	1904.2
2.5	2687.7
5	4444.8
7.5	6128.2
10	7844.6
12.5	9893.2

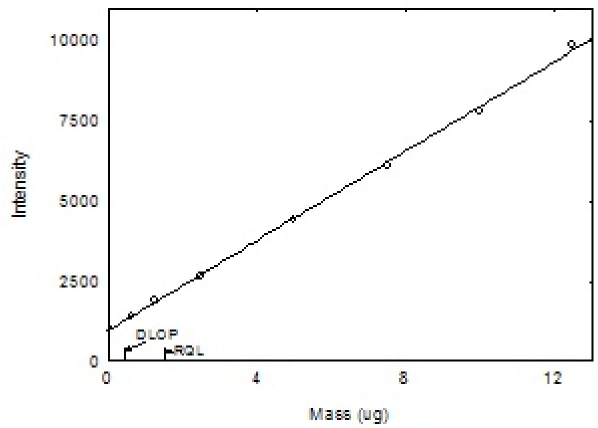

Figure 1.1.6. Plot of data to determine DLOP/RQL for Copper. (Y = 699.4X + 977.2)

Table 1.1.7
Detection Limit of the Overall Procedure for Iron

mass per sample (µg)	intensity
0	699.2
6.2	1546.4
12.5	2371.5
25	4035.6
50	7338.3
75	10830.9
100	14308.3
125	17759.7

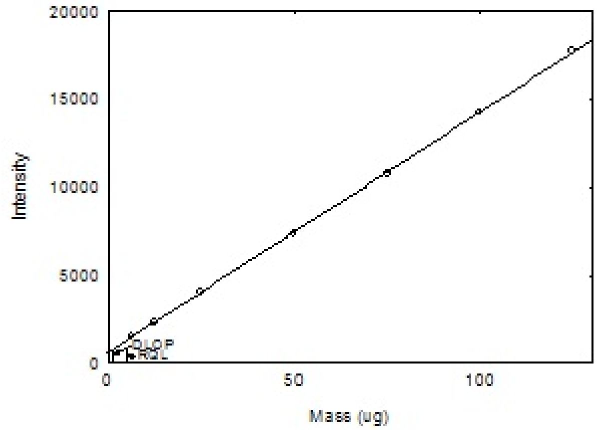

Figure 1.1.7. Plot of data to determine DLOP/RQL for Iron. (Y = 136.4X + 648.9)

Table 1.1.8
Detection Limit of the Overall Procedure for Manganese

mass per sample (µg)	intensity
0	166.6
1.2	2552.4
2.5	5015.1
5	9899.9
10	19028.4
15	28853.3
20	38573.7
25	48431.3

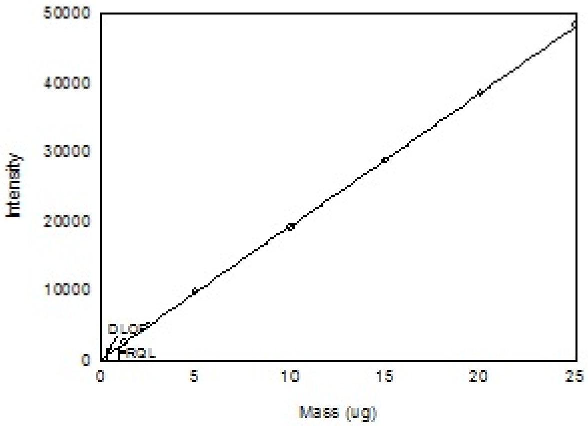

Figure 1.1.8. Plot of data to determine DLOP/RQL for Manganese. (Y = 1924.2X + 124.5

Table 1.1.9
Detection Limit of the Overall Procedure for Molybdenum

mass per sample (µg)	intensity
0	-2.7
3.1	32.3
6.2	63.2
12.5	130.3
25	251.6
37.5	388
50	515.2
62.5	641.4

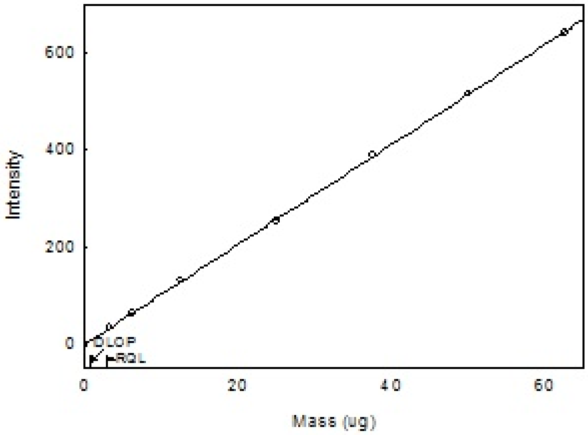

Figure 1.1.9. Plot of data to determine DLOP/RQL for Molybdenum. (Y = 10.3X - 1.1)

Table 1.1.10
Detection Limit of the Overall Procedure for Nickel

mass per sample (µg)	intensity
0	0.9
3.1	65.5
6.2	135.7
12.5	265.1
25	508.5
37.5	770.4
50	1040.4
62.5	1284

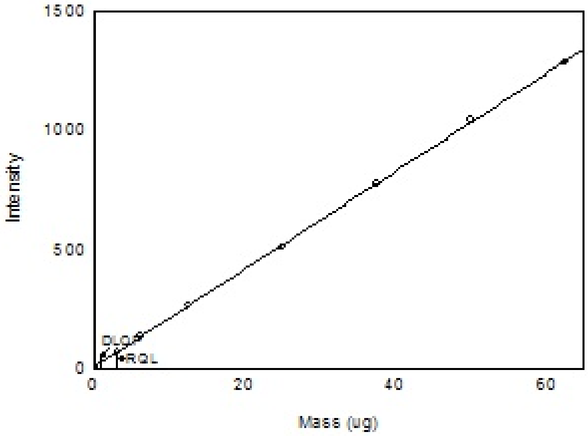

Figure 1.1.10. Plot of data to determine DLOP/RQL for Nickel. (Y = 20.6X + 3.0)

Table 1.1.11
Detection Limit of the Overall Procedure for Lead

mass per sample (µg)	intensity
0	0.3
0.31	-0.4
0.62	2.7
1.25	4.3
2.5	11.6
3.8	19.5
5	24.8
3.2	31.5

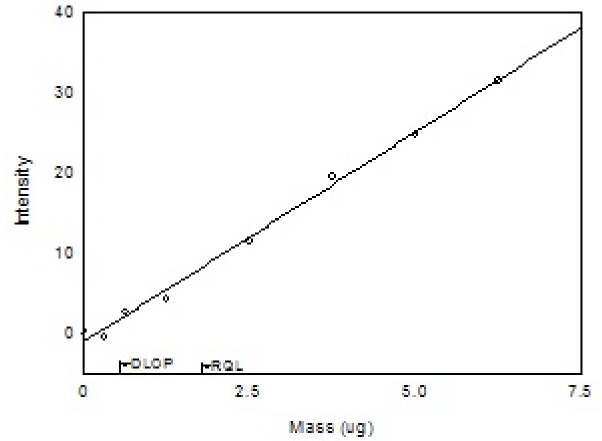

Figure 1.1.11. Plot of data to determine DLOP/RQL for Lead. (Y = 5.2X - 1.0)

Table 1.1.12
Detection Limit of the Overall Procedure for Antimony

mass per sample (µg)	intensity
0	-1.3
3.1	6
6.2	15.2
12.5	28.8
25	60.8
37.5	88.1
50	116.6
62.5	146.3

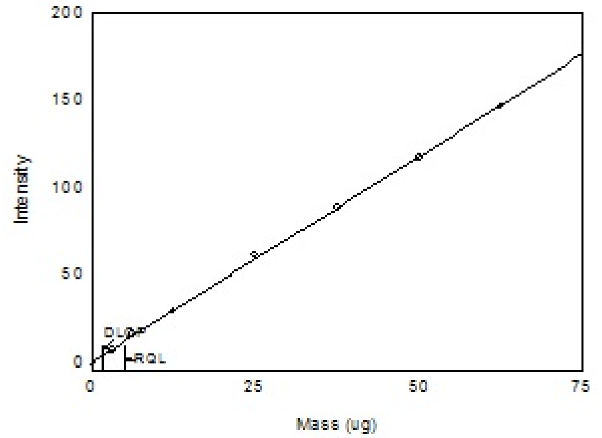

Figure 1.1.12. Plot of data to determine DLOP/RQL for Antimony. (Y = 2.4X - 0.4)

Table 1.1.13
Detection Limit of the Overall Procedure for Vanadium

mass per sample (µg)	intensity
0	5.7
0.17	39.8
0.35	64.5
0.7	114.6
1.4	230.2
2.1	352.4
2.8	472.4
3.5	586.7

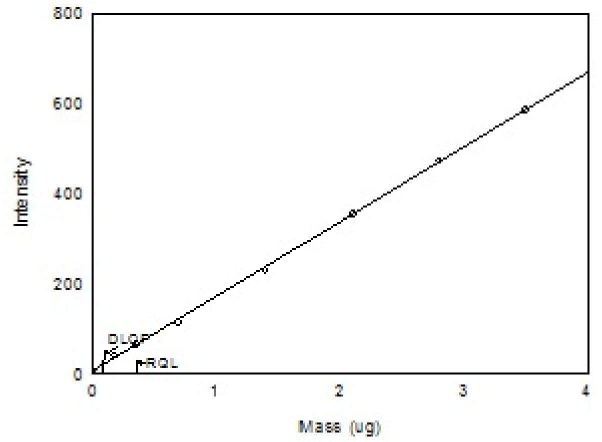

Figure 1.1.13. Plot of data to determine DLOP/RQL for Vanadium. (Y = 165.9X + 4.6)

Table 1.1.14
Detection Limit of the Overall Procedure for Zinc

mass per sample (μg)	intensity
0	805.8
2.5	879.8
5	1088.1
10	1445.5
20	2201.2
30	3046.4
40	3825.4
50	4482.3

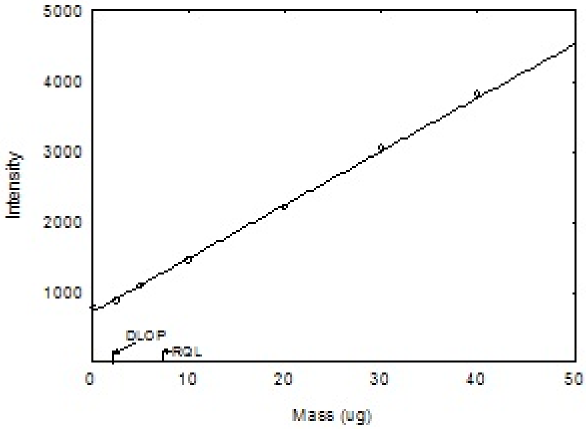

Figure 1.1.14. Plot of data to determine DLOP/RQL for Zinc (Y = 76.0X + 725.7)

1.2 Storage Test

Storage samples were prepared by liquid-spiking Ghost Wipes at the target concentration. Twelve storage samples were prepared. Three samples were analyzed on the day prepared. Nine of the samples were stored at ambient temperature (about 22°C). At 5-day intervals three samples were analyzed. Results were blank corrected.

Table 1.2.1
Storage Test for Beryllium

time (days)	recovery (%)		
0	90.2	91.3	92.8
5	90.7	93.7	91.2
10	90.1	88.9	92.0
15	91.1	91.3	91.9

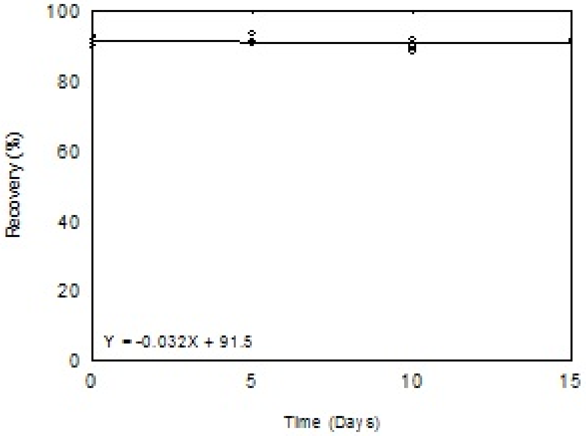

Figure 1.2.1. Storage test for Beryllium.

Table 1.2.2
Storage Test for Cadmium

time (days)	recovery (%)		
0	91.0	92.6	93.3
5	95.8	97.9	96.8
10	96.8	94.7	97.7
15	96.0	96.0	96.4

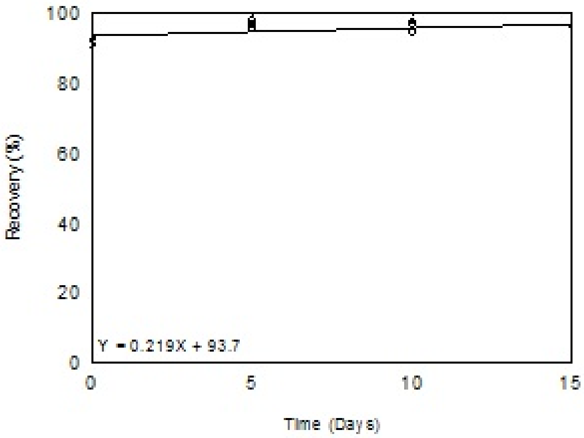

Figure 1.2.2. Storage test for Cadmium.

Table 1.2.3
Storage Test for Cobalt

time (days)	recovery (%)		
0	94.8	95.9	96.7
5	93.6	96.6	94.3
10	93.2	92.7	95.6
15	97.4	98.4	97.4

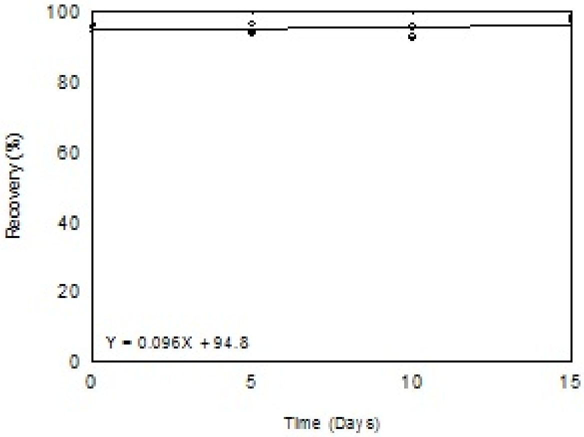

Figure 1.2.3. Storage test for Cobalt.

Table 1.2.4
Storage Test for Chromium

time (days)	recovery (%)		
0	96.7	100.1	98.1
5	95.6	98.7	97.6
10	96.4	94.1	97.0
15	96.7	98.5	97.0

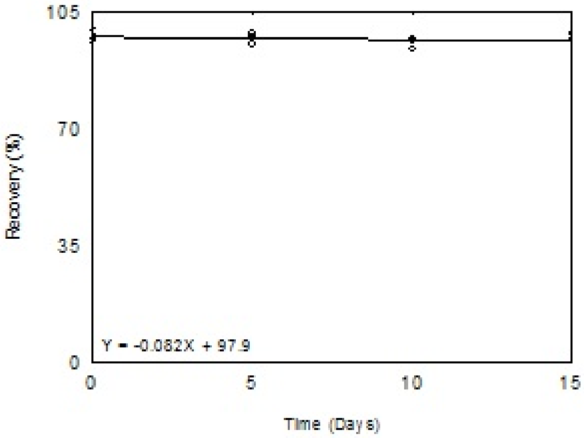

Figure 1.2.4. Storage test for Chromium.

Table 1.2.5
Storage Test for Copper

time (days)	recovery (%)		
0	95.7	96.6	98.2
5	95.9	97.8	96.3
10	96.3	93.8	96.6
15	96.8	97.8	96.8

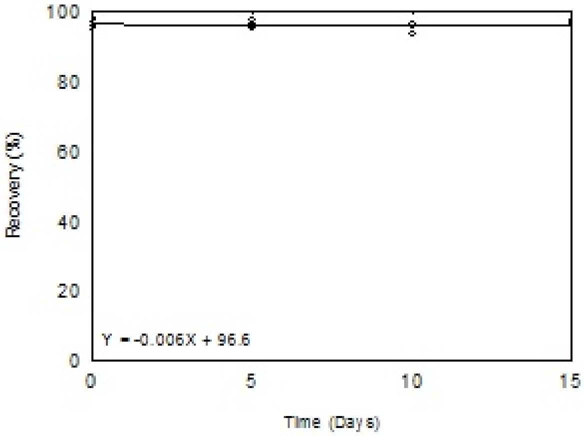

Figure 1.2.5. Storage test for Copper.

Table 1.2.6
Storage Test for Iron

time (days)	recovery (%)		
0	97.5	98.6	100.3
5	97.1	99.0	97.7
10	97.7	95.9	98.8
15	98.7	99.9	99.0

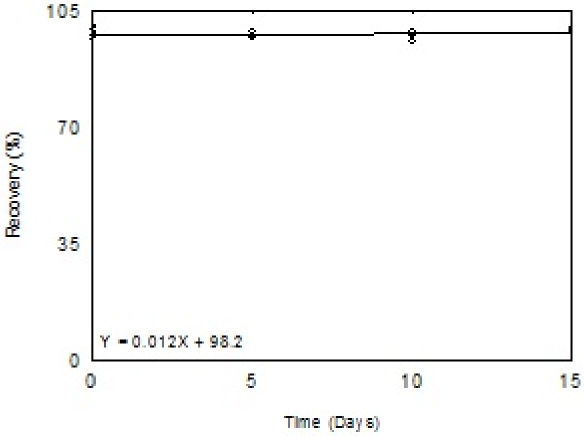

Figure 1.2.6. Storage test for Iron.

Table 1.2.7
Storage Test for Manganese

time (days)	recovery (%)		
0	95.2	98.5	96.8
5	94.6	97.5	96.5
10	96.0	93.8	96.6
15	96.9	98.7	97.2

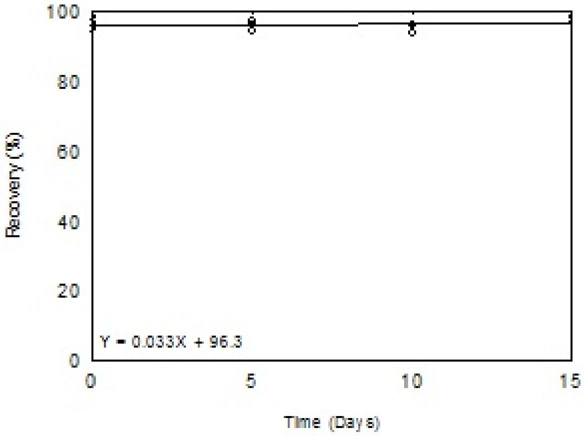

Figure 1.2.7. Storage test for Manganese.

Table 1.2.8
Storage Test for Molybdenum

time (days)	recovery (%)		
0	96.0	97.0	98.0
5	96.3	97.0	95.0
10	94.7	93.6	96.5
15	95.3	95.8	95.8

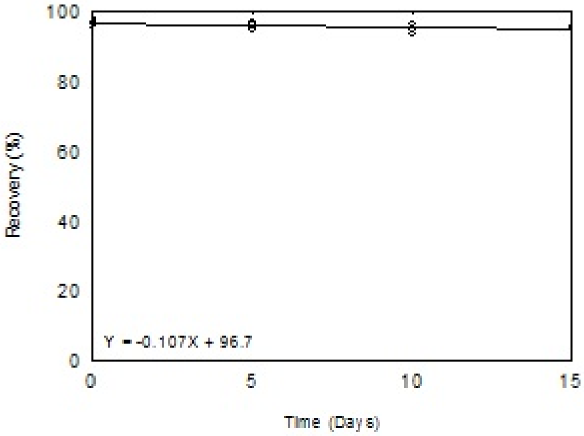

Figure 1.2.8. Storage test for Molybdenum.

Table 1.2.9
Storage Test for Nickel

time (days)	recovery (%)		
0	94.5	97.7	95.1
5	94.2	98.1	95.0
10	94.5	93.3	96.2
15	94.4	94.6	95.3

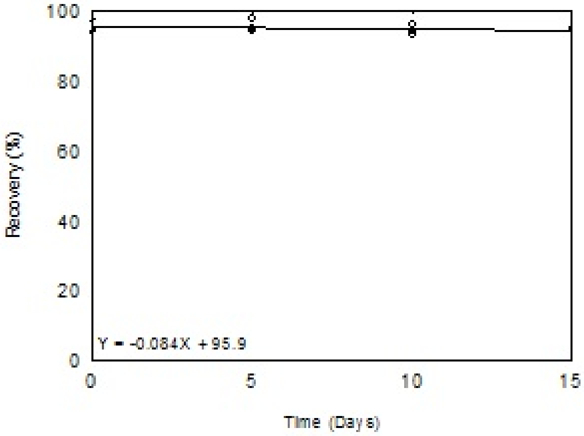

Figure 1.2.9. Storage test for Nickel.

Table 1.2.10
Storage Test for Lead

time (days)	recovery (%)		
0	93.6	94.8	96.3
5	93.0	96.6	95.7
10	95.2	93.4	96.7
15	94.3	95.3	95.0

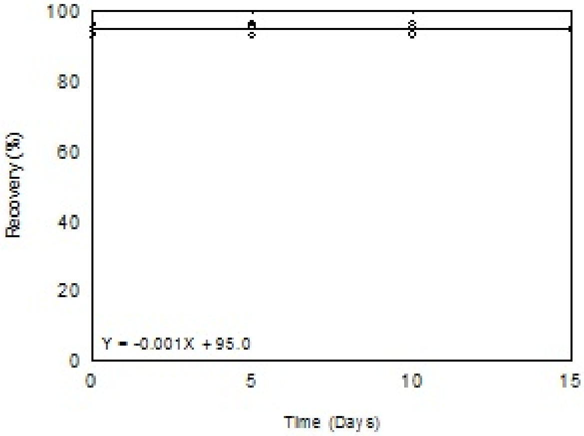

Figure 1.2.10. Storage test for Lead.

Table 1.2.11
Storage Test for Antimony

time (days)	recovery (%)		
0	85.8	85.5	90.1
5	86.9	88.2	85.6
10	78.2	81.7	86.6
15	85.5	89.6	89.0

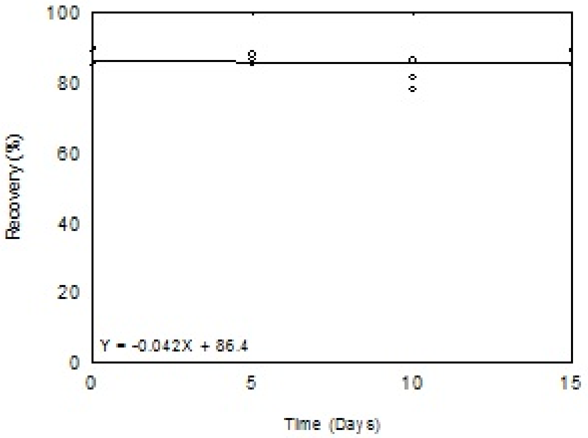

Figure 1.2.11. Storage test for Antimony.

Table 1.2.12
Storage Test for Vanadium

time (days)	recovery (%)		
0	91.3	93.6	92.4
5	91.2	94.4	91.7
10	91.0	89.5	91.6
15	91.1	90.7	91.2

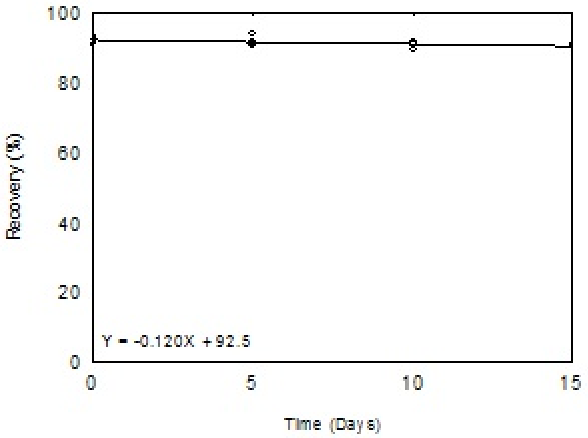

Figure 1.2.12. Storage test for Vanadium.

Table 1.2.13
Storage Test for Zinc

time (days)	recovery (%)		
0	90.0	90.9	91.4
5	89.9	92.8	90.7
10	89.6	88.7	91.4
15	90.3	91.4	90.9

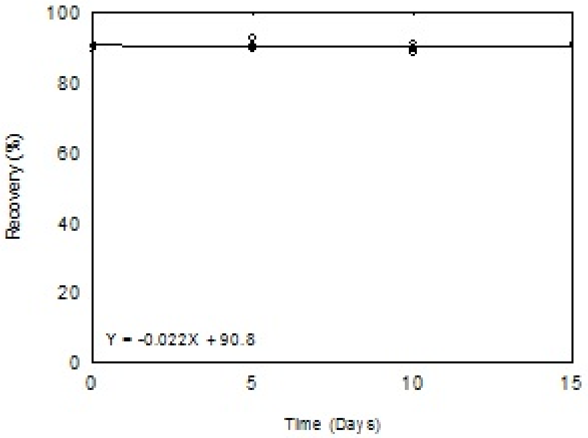

Figure 1.2.13. Storage test for Zinc.

1.3 Sampler Removal Efficiency

Six 100-cm^2 glass plates were liquid-spiked at the target concentrations and allowed to dry. Samples were collected from each surface by placing a Ghost Wipe folded in half at an outside edge of the glass plate and progressing towards the center making concentric squares of decreasing size while applying firm pressure. The Ghost Wipe was folded in half with the contaminant side in and the glass plate was wiped a second time. The Ghost Wipe was again folded in half and the surface wiped a third time. Results were blank corrected.

Table 1.3.1
Sampler Removal Efficiency
Data for Beryllium on Ghost Wipes

theoretical (µg/surface)	recovered (µg/sample)	recovery (%)
4	3.770	94.2
4	3.755	93.9
4	3.938	98.4
4	3.880	97.0
4	3.848	96.0
4	3.785	84.4

Table 1.3.2
Sampler Removal Efficiency
Data for Cadmium on Ghost Wipes

theoretical (µg/surface)	recovered (µg/sample)	recovery (%)
10	9.300	93.0
10	9.168	91.6
10	9.625	96.2
10	9.535	95.4
10	9.595	96.0
10	8.330	83.3

Table 1.3.3
Sampler Removal Efficiency
Data for Cobalt on Ghost Wipes

theoretical (µg/surface)	recovered (µg/sample)	recovery (%)
200	182.1	91.0
200	182.0	91.0
200	190.6	95.3
200	187.5	93.8
200	187.8	93.9
200	162.8	81.4

Table 1.3.4
Sampler Removal Efficiency
Data for Chromium on Ghost Wipes

theoretical (µg/surface)	recovered (µg/sample)	recovery (%)
1000	964.5	96.4
1000	889.5	88.9
1000	1001.5	100.1
1000	917.5	91.7
1000	992.0	99.2
1000	927.0	92.7

Table 1.3.5
Sampler Removal Efficiency
Data for Copper on Ghost Wipes

theoretical (µg/surface)	recovered (µg/sample)	recovery (%)
200	194.1	97.0
200	193.6	96.8
200	200.7	100.4
200	195.6	97.9
200	197.3	98.6
200	172.5	86.2

Table 1.3.6
Sampler Removal Efficiency
Data for Iron on Ghost Wipes

theoretical (µg/surface)	recovered (µg/sample)	recovery (%)
2000	1914	95.7
2000	1913	95.6
2000	1987	99.4
2000	1940	97.0
2000	1957	97.8
2000	1704	85.2

Table 1.3.7
Sampler Removal Efficiency
Data for Manganese on Ghost Wipes

theoretical (µg/surface)	recovered (µg/sample)	recovery (%)
400	385.1	96.3
400	356.8	89.2
400	400.1	100.0
400	367.1	91.8
400	395.7	98.9
400	371.1	92.8

Table 1.3.8
Sampler Removal Efficiency
Data for Molybdenum on Ghost Wipes

theoretical (µg/surface)	recovered (µg/sample)	recovery (%)
1000	892.0	89.2
1000	698.5	69.8
1000	791.0	79.1
1000	780.5	78.0
1000	724.5	72.4
1000	749.0	74.9

Table 1.3.9
Sampler Removal Efficiency
Data for Nickel on Ghost Wipes

theoretical (µg/surface)	recovered (µg/sample)	recovery (%)
1000	913.5	91.4
1000	843.0	84.3
1000	955.0	95.5
1000	881.0	88.1
1000	941.5	94.2
1000	875.0	87.5

Table 1.3.10
Sampler Removal Efficiency
Data for Lead on Ghost Wipes

theoretical (µg/surface)	recovered (µg/sample)	recovery (%)
100	90.95	91.0
100	89.00	89.0
100	93.60	93.6
100	91.55	91.6
100	92.75	92.8
100	80.40	80.4

Table 1.3.11
Sampler Removal Efficiency
Data for Antimony on Ghost Wipes

theoretical (µg/surface)	recovered (µg/sample)	recovery (%)
1000	809.5	81.0
1000	654.5	65.4
1000	733.0	73.3
1000	717.5	71.8
1000	698.5	69.8
1000	690.5	69.0

Table 1.3.12
Sampler Removal Efficiency
Data for Vanadium on Ghost Wipes

theoretical (µg/surface)	recovered (µg/sample)	recovery (%)
56	52.08	93.0
56	49.41	88.2
56	54.23	96.8
56	50.23	89.7
56	52.43	93.6
56	50.98	91.0

Table 1.3.13
Sampler Removal Efficiency
Data for Zinc on Ghost Wipes

theoretical (µg/surface)	recovered (µg/sample)	recovery (%)
800	710.7	88.8
800	712.2	89.0
800	754.2	94.3
800	739.2	92.4
800	735.2	91.9
800	641.7	80.2

1.4 Analytical Method Recovery and Stability of Digested Samples

1.4.1 Analytical Method Recovery

Analytical method recovery (AMR) was determined by liquid-spiking Ghost Wipes with the analytes at the RQL, 0.1, 1.0, and 10 times the target concentrations. These samples were stored overnight at ambient temperature and then analyzed. Results were blank corrected.

Table 1.4.1.1
Analytical Method Recovery of Beryllium from Ghost Wipes

level		sample number				
× target concn	µg per sample	1	2	3	4	mean
RQL	0.017	114.7	82.4	94.1	82.4	93.4
0.1	0.4	95.6	99.5	96.1	97.4	97.2
1.0	4	94.5	95.1	90.6	94.4	93.6
10	40	92.2	92.9	93.6	94.4	93.3
AMR						94.4

Table 1.4.1.2
Analytical Method Recovery of Cadmium from Ghost Wipes

level		sample number				
× target concn	µg per sample	1	2	3	4	mean
RQL	0.18	83.3	86.1	86.1	80.6	84.0
0.1	1	99.5	102.5	98.0	99.0	99.8
1.0	10	99.4	99.80	96.5	99.4	98.8
10	100	98.3	100.4	102.4	102.5	100.9
AMR						95.9

Table 1.4.1.3
Analytical Method Recovery of Cobalt from Ghost Wipes

level		sample number				
× target concn	µg per sample	1	2	3	4	mean
RQL	1.1	105.9	92.7	94.1	97.7	97.6
0.1	20	99.2	101.9	100.2	101.1	100.6
1.0	200	98.2	98.1	93.7	97.7	96.9
10	2000	96.6	97.6	98.2	99.3	97.9
AMR						98.2

Table 1.4.1.4
Analytical Method Recovery of Chromium from Ghost Wipes

level		sample number				
× target concn	µg per sample	1	2	3	4	mean
RQL	3.4	116.9	102.5	107.5	107.9	108.7
0.1	100	98.8	102.9	99.8	101.6	100.8
1.0	1000	99.3	98.9	95.3	99.1	98.2
10	10000	96.8	99.3	101.0	101.6	99.7
AMR						101.8

Table 1.4.1.5
Analytical Method Recovery of Copper from Ghost Wipes

level		sample number				
× target concn	µg per sample	1	2	3	4	mean
RQL	1.5	110.0	111.7	100.0	89.7	102.8
0.1	20	99.8	102.7	101.4	101.0	101.2
1.0	200	98.0	97.4	93.4	97.4	96.6
10	2000	94.8	97.5	99.0	99.4	97.7
AMR						99.6

Table 1.4.1.6
Analytical Method Recovery of Iron from Ghost Wipes

level		sample number				
× target concn	µg per sample	1	2	3	4	mean
RQL	5.1	131.9	112.9	107.4	93.0	111.3
0.1	200	102.3	106.2	102.9	104.2	103.9
1.0	2000	101.2	100.8	97.0	101.1	100.0
10	20000	98.5	101.7	103.8	103.8	102.0
AMR						104.3

Table 1.4.1.7
Analytical Method Recovery of Manganese from Ghost Wipes

level		sample number				
× target concn	µg per sample	1	2	3	4	mean
RQL	0.97	121.0	107.4	137.7	165.6	132.9
0.1	40	99.2	103.0	100.3	101.9	101.1
1.0	400	99.1	98.6	95.1	98.7	97.9
10	4000	96.4	99.0	100.7	101.2	99.3
AMR						107.8

Table 1.4.1.8
Analytical Method Recovery of Molybdenum from Ghost Wipes

level		sample number				
× target concn	µg per sample	1	2	3	4	mean
RQL	2.7	122.6	100.0	108.2	96.3	106.8
0.1	100	101.8	103.5	102.2	103.0	102.6
1.0	1000	98.9	98.7	94.6	98.5	97.7
10	1000	97.4	98.4	98.8	99.8	98.6
AMR						101.4

Table 1.4.1.9
Analytical Method Recovery of Nickel from Ghost Wipes

level		sample number				
× target concn	µg per sample	1	2	3	4	mean
RQL	3.1	122.1	105.5	121.1	106.1	113.7
0.1	100	98.0	101.0	99.4	99.6	99.5
1.0	1000	98.7	98.2	94.7	97.4	97.2
10	10000	96.9	97.7	97.8	99.1	97.9
AMR						102.1

Table 1.4.1.10
Analytical Method Recovery of Lead from Ghost Wipes

level		sample number				
× target concn	µg per sample	1	2	3	4	mean
RQL	1.8	53.9	65.0	80.6	67.2	66.7
0.1	10	90.7	93.6	101.0	104.0	97.3
1.0	100	98.8	98.8	94.5	98.5	97.6
10	1000	96.6	99.8	101.3	102.4	100.0
AMR						90.4

Table 1.4.1.11
Analytical Method Recovery of Antimony from Ghost Wipes

level		sample number				
× target concn	µg per sample	1	2	3	4	mean
RQL	5.1	76.9	59.8	37.1	47.8	55.4
0.1	100	90.9	90.6	90.6	92.5	91.2
1.0	1000	92.3	92.1	89.2	91.7	91.3
10	10000	88.7	85.9	93.2	94.5	90.6
AMR						82.1

Table 1.4.1.12
Analytical Method Recovery of Vanadium from Ghost Wipes

level		sample number				
× target concn	µg per sample	1	2	3	4	mean
RQL	0.29	94.8	86.2	96.6	101.7	94.8
0.1	5.6	93.0	96.8	94.2	97.2	95.3
1.0	56	94.6	93.9	91.1	93.3	93.2
10	560	92.6	93.6	93.9	94.4	93.6
AMR						94.2

Table 1.4.1.13
Analytical Method Recovery of Zinc from Ghost Wipes

level		sample number				
× target concn	µg per sample	1	2	3	4	mean
RQL	7.3	125.8	132.7	104.6	91.1	113.6
0.1	80	92.3	96.8	91.7	95.4	94.0
1.0	800	93.6	93.2	89.6	92.9	92.3
10	8000	93.5	92.6	93.2	94.1	93.4
AMR						98.3

1.4.2 Stability of Digested Samples

Stability of the digested samples were investigated by reanalyzing the four 1.0 times the target concentration samples seven days after initial analysis. After the original analysis was performed the samples were stored at ambient temperature in the 50 mL volumetric flasks in which they were brought to volume in. For the second analysis new aliquots were taken from each volumetric. Results were blank corrected.

Table 1.4.2.1
Stability of Digested Samples for Beryllium

initial (%)	after 7 days (%)	difference (%)
94.5	91.9	2.6
95.1	92.3	2.8
90.6	87.3	3.3
94.4	91.1	3.3

Table 1.4.2.2
Stability of Digested Samples for Cadmium

initial (%)	after 7 days (%)	difference (%)
99.4	97.2	2.2
99.8	98.4	1.4
96.5	94.0	2.5
99.4	97.9	1.5

Table 1.4.2.3
Stability of Digested Samples for Cobalt

initial (%)	after 7 days (%)	difference (%)
98.2	98.3	0.1
98.1	98.7	0.6
93.7	94.0	0.3
97.7	97.7	0.0

Table 1.4.2.4
Stability of Digested Samples for Chromium

initial (%)	after 7 days (%)	difference (%)
99.3	97.8	1.5
98.9	97.2	1.7
95.3	93.4	1.9
99.1	97.2	1.9

Table 1.4.2.5
Stability of Digested Samples for Copper

initial (%)	after 7 days (%)	difference (%)
98.0	96.8	1.2
97.4	95.9	1.5
93.4	91.9	1.5
97.4	95.8	1.6

Table 1.4.2.6
Stability of Digested Samples for Iron

initial (%)	after 7 days (%)	difference (%)
101.2	99.2	1.0
100.8	98.6	2.2
97.0	94.1	2.9
101.1	98.7	2.4

Table 1.4.2.7
Stability of Digested Samples for Manganese

initial (%)	after 7 days (%)	difference (%)
99.0	97.4	1.6
98.6	96.6	2.0
95.1	93.0	2.1
98.7	96.6	2.1

Table 1.4.2.8
Stability of Digested Samples for Molybdenum

initial (%)	after 7 days (%)	difference (%)
98.9	99.2	0.3
98.7	99.8	1.1
94.6	99.8	5.2
98.5	98.6	0.1

Table 1.4.2.9
Stability of Digested Samples for Nickel

initial (%)	after 7 days (%)	difference (%)
98.7	96.6	2.1
98.2	96.8	1.9
94.7	92.4	2.3
97.4	95.4	2.0

Table 1.4.2.10
Stability of Digested Samples for Lead

initial (%)	after 7 days (%)	difference (%)
98.8	97.0	1.8
98.8	97.6	1.2
94.5	94.2	0.3
98.5	98.2	0.3

Table 1.4.2.11
Stability of Digested Samples for Antimony

initial (%)	after 7 days (%)	difference (%)
92.3	93.2	0.9
92.1	93.7	1.6
89.2	89.9	0.7
91.7	93.6	1.9

Table 1.4.2.12
Stability of Digested Samples for Vanadium

initial (%)	after 7 days (%)	difference (%)
94.6	92.1	2.5
93.9	92.0	1.9
91.0	87.3	3.7
93.3	90.3	3.0

Table 1.4.2.13
Stability of Digested Samples for Zinc

initial (%)	after 7 days (%)	difference (%)
93.6	93.5	0.1
93.2	93.9	0.7
89.6	89.6	0.0
92.9	92.7	0.2

1.5 Reproducibility

1.5.1 Sampling Reproducibility

Six 100-cm^2 glass plates were liquid-spiked at the target concentrations and allowed to dry. Two chemist, other than the one developing the method, conducted surface sampling. Results were blank corrected.

Table 1.5.1
Sampling Reproducibility Data for Beryllium on Ghost Wipes

	Chemist 1		Chemist 2	
theoretical (μg/surface)	recovered (μg/sample)	recovery (%)	recovered (μg/sample)	recovery (%)
4	3.821	95.5	3.621	90.5
4	3.788	94.7	3.646	91.2
4	3.687	92.2	3.678	91.9
4	3.973	99.3	3.640	91.0
4	4.023	100.6	3.711	92.8
4	3.899	97.5	3.599	90.0

Table 1.5.2
Sampling Reproducibility Data for Cadmium on Ghost Wipes

	Chemist 1		Chemist 2	
theoretical (μg/surface)	recovered (μg/sample)	recovery (%)	recovered (μg/sample)	recovery (%)
10	9.615	96.2	9.480	94.8
10	9.545	95.4	9.520	95.2
10	9.310	93.1	9.510	95.1
10	9.865	98.6	9.550	95.5
10	10.06	100.6	9.665	96.6
10	9.805	98.0	9.390	93.9

Table 1.5.3
Sampling Reproducibility Data for Cobalt on Ghost Wipes

	Chemist 1		Chemist 2	
theoretical (μg/surface)	recovered (μg/sample)	recovery (%)	recovered (μg/sample)	recovery (%)
200	182.1	91.1	183.5	91.8
200	179.5	89.7	185.5	92.9
200	173.3	86.6	185.4	92.7
200	187.6	93.8	185.0	92.5
200	190.5	95.2	188.2	94.1
200	184.0	92.0	182.5	91.2

Table 1.5.4
Sampling Reproducibility Data for Chromium on Ghost Wipes

	Chemist 1		Chemist 2	
theoretical (µg/surface)	recovered (µg/sample)	recovery (%)	recovered (µg/sample)	recovery (%)
1000	976.0	97.6	918.0	91.8
1000	981.0	98.1	956.0	95.6
1000	905.5	90.6	944.5	94.4
1000	991	99.1	935.0	93.5
1000	1025	102.5	980.5	98.0
1000	1012	101.2	954.5	95.4

Table 1.5.5
Sampling Reproducibility Data for Copper on Ghost Wipes

	Chemist 1		Chemist 2	
theoretical (µg/surface)	recovered (µg/sample)	recovery (%)	recovered (µg/sample)	recovery (%)
200	196.0	98.0	189.5	94.8
200	193.9	96.9	190.4	95.2
200	185.1	92.5	189.6	94.8
200	198.9	99.5	188.2	94.0
200	202.3	101.2	190.7	95.3
200	195.4	97.7	185.6	92.7

Table 1.5.6
Sampling Reproducibility Data for Iron on Ghost Wipes

	Chemist 1		Chemist 2	
theoretical (µg/surface)	recovered (µg/sample)	recovery (%)	recovered (µg/sample)	recovery (%)
2000	1961	98.1	1933	96.6
2000	1939	97.0	1949	97.5
2000	1838	91.9	1948	97.4
2000	2002	100.0	1936	96.8
2000	2039	101.9	1962	98.1
2000	1975	98.7	1909	95.5

Table 1.5.7
Sampling Reproducibility Data for Manganese on Ghost Wipes

	Chemist 1		Chemist 2	
theoretical (µg/surface)	recovered (µg/sample)	recovery (%)	recovered (µg/sample)	recovery (%)
400	390.5	97.6	369.0	92.3
400	392.4	98.1	383.7	95.9
400	363.5	90.9	379.0	94.8
400	396.2	99.1	374.8	93.7
400	409.1	102.2	391.8	98.0
400	403.3	100.8	382.6	95.6

Table 1.5.8
Sampling Reproducibility Data for Molybdenum on Ghost Wipes

	Chemist 1		Chemist 2	
theoretical (µg/surface)	recovered (µg/sample)	recovery (%)	recovered (µg/sample)	recovery (%)
1000	737.5	73.8	906.0	90.6
1000	862.0	86.2	932.0	93.2
1000	803.5	80.4	952.0	95.2
1000	934.0	93.4	908.0	90.8
1000	839.0	83.9	970.5	97.0
1000	929.5	93.0	938.5	93.8

Table 1.5.9
Sampling Reproducibility Data for Nickel on Ghost Wipes

	Chemist 1		Chemist 2	
theoretical (µg/surface)	recovered (µg/sample)	recovery (%)	recovered (µg/sample)	recovery (%)
1000	925.0	92.5	895.5	89.6
1000	928.0	92.8	932.5	93.2
1000	875.5	87.6	920.5	92.0
1000	945.0	94.5	915.0	91.5
1000	967.0	96.7	959.0	95.9
1000	959.0	95.9	936.0	93.6

Table 1.5.10
Sampling Reproducibility Data for Lead on Ghost Wipes

	Chemist 1		Chemist 2	
theoretical (µg/surface)	recovered (µg/sample)	recovery (%)	recovered (µg/sample)	recovery (%)
100	94.55	94.6	92.65	92.6
100	92.30	92.3	93.30	93.3
100	89.45	89.5	92.90	92.9
100	96.15	96.2	92.40	92.4
100	97.10	97.1	94.65	94.6
100	94.95	95.0	91.60	91.6

Table 1.5.11
Sampling Reproducibility Data for Antimony on Ghost Wipes

	Chemist 1		Chemist 2	
theoretical (µg/surface)	recovered (µg/sample)	recovery (%)	recovered (µg/sample)	recovery (%)
1000	757.0	75.7	751.5	75.2
1000	836.0	83.6	780.5	78.0
1000	813.0	81.3	797.5	79.8
1000	911.5	91.2	814.5	81.4
1000	823.0	82.3	882.5	88.2
1000	925.0	92.5	827.5	82.8

Table 1.5.12
Sampling Reproducibility Data for Vanadium on Ghost Wipes

	Chemist 1		Chemist 2	
theoretical (µg/surface)	recovered (µg/sample)	recovery (%)	recovered (µg/sample)	recovery (%)
56	52.58	93.9	48.54	86.7
56	51.68	92.3	50.88	90.9
56	49.37	88.2	50.78	90.7
56	53.28	95.2	50.48	90.2
56	54.38	97.1	52.48	93.7
56	53.83	96.1	51.23	91.5

Table 1.5.13
Sampling Reproducibility Data for Zinc on Ghost Wipes

	Chemist 1		Chemist 2	
theoretical (µg/surface)	recovered (µg/sample)	recovery (%)	recovered (µg/sample)	recovery (%)
800	725.5	90.7	729.2	91.2
800	720.7	90.1	727.7	91.0
800	697.7	87.2	742.2	92.8
800	750.7	93.8	731.2	91.4
800	754.7	94.3	751.7	94.0
800	732.7	91.6	728.2	91.0

1.5.2 Analytical Reproducibility

Six samples were prepared by spiking media in the same manner that was used in the preparation of samples for the storage study. The samples were submitted to the OSHA SLTC for analysis and the samples were analyzed by a chemist other than the one developing the method. The samples were analyzed after being stored for 15 days at ambient temperature. Results were blank corrected.

Table 1.5.2.1
Analytical Reproducibility Data for
Beryllium on Ghost Wipes

theoretical (µg/surface)	recovered (µg/sample)	recovery (%)
4	3.767	94.2
4	3.903	97.6
4	3.958	99.0
4	4.008	100.2
4	4.024	100.6
4	3.892	97.3

Table 1.5.2.2
Analytical Reproducibility Data for
Cadmium on Ghost Wipes

theoretical (µg/surface)	recovered (µg/sample)	recovery (%)
10	10.10	101.0
10	10.30	103.0
10	10.46	104.6
10	10.74	107.4
10	10.50	105.0
10	10.34	103.4

Table 1.5.2.3
Analytical Reproducibility Data for
Cobalt on Ghost Wipes

theoretical (µg/surface)	recovered (µg/sample)	recovery (%)
200	193.8	96.9
200	199.4	99.7
200	201.3	100.6
200	205.2	102.6
200	203.9	102.0
200	199.2	99.6

Table 1.5.2.4
Analytical Reproducibility Data for
Chromium on Ghost Wipes

theoretical (µg/surface)	recovered (µg/sample)	recovery (%)
1000	973.4	97.3
1000	1004	100.4
1000	1025	102.5
1000	1033	103.3
1000	1016	101.6
1000	1006	100.6

Table 1.5.2.5
Analytical Reproducibility Data for
Copper on Ghost Wipes

theoretical (µg/surface)	recovered (µg/sample)	recovery (%)
200	193.0	96.5
200	198.0	99.0
200	202.1	101.0
200	203.0	101.5
200	202.7	101.4
200	196.4	98.2

Table 1.5.2.6
Analytical Reproducibility Data for
Iron on Ghost Wipes

theoretical (µg/surface)	recovered (µg/sample)	recovery (%)
2000	2016	100.8
2000	2070	103.5
2000	2119	106.0
2000	2134	106.7
2000	2104	105.2
2000	2069	103.4

Table 1.5.2.7
Analytical Reproducibility Data for
Manganese on Ghost Wipes

theoretical (µg/surface)	recovered (µg/sample)	recovery (%)
400	392.4	98.1
400	404.4	101.1
400	412.6	103.2
400	415.6	103.9
400	409.9	102.5
400	404.8	101.2

Table 1.5.2.8
Analytical Reproducibility Data for
Molybdenum on Ghost Wipes

theoretical (µg/surface)	recovered (µg/sample)	recovery (%)
1000	982.4	98.2
1000	879.9	88.0
1000	1157	115.7
1000	1040	104.0
1000	1043	104.3
1000	1016	101.6

Table 1.5.2.9
Analytical Reproducibility Data for
Nickel on Ghost Wipes

theoretical (µg/surface)	recovered (µg/sample)	recovery (%)
1000	955.1	95.5
1000	983.6	98.4
1000	990.1	99.01
1000	1005	100.5
1000	997.6	99.8
1000	981.6	98.16

Table 1.5.2.10
Analytical Reproducibility Data for
Lead on Ghost Wipes

theoretical (µg/surface)	recovered (µg/sample)	recovery (%)
100	96.10	96.1
100	97.75	97.8
100	98.00	98.0
100	101.2	101.2
100	99.60	99.6
100	97.85	97.8

Table 1.5.2.11
Analytical Reproducibility Data for
Antimony on Ghost Wipes

theoretical (µg/surface)	recovered (µg/sample)	recovery (%)
1000	907.4	90.7
1000	729.9	73.0
1000	1107	111
1000	942.4	94.2
1000	943.4	94.3
1000	917.9	91.8

Table 1.5.2.12
Analytical Reproducibility Data for
Vanadium on Ghost Wipes

theoretical (µg/surface)	recovered (µg/sample)	recovery (%)
56	51.53	92.0
56	53.55	95.6
56	53.85	96.2
56	54.60	97.5
56	54.80	97.9
56	53.50	95.5

Table 1.5.2.13
Analytical Reproducibility Data for
Zinc on Ghost Wipes

theoretical (µg/surface)	recovered (µg/sample)	recovery (%)
800	733.4	91.7
800	751.9	94.0
800	761.4	95.2
800	771.9	96.5
800	770.9	96.4
800	750.9	93.9

1.6 Interferences

1.6.1 Media, Reagents, and Surface.

Six blank Ghost Wipes were analyzed to determine interferences due to contamination from the media and reagents.

Table 1.6.1.1
Contamination to Analysis from the Media and Reagents (µg found)

analyte	wipe 1	wipe 2	wipe 3	wipe 4	wipe 5	wipe 6	average
Be	0.009	0.004	0.000	0.008	0.008	0.008	0.006
Cd	0.000	0.015	0.070	0.000	0.000	0.000	0.014
Co	0.090	0.060	0.100	0.000	0.020	0.035	0.051
Cr	0.030	0.015	0.000	0.09	0.020	0.100	0.042
Cu	1.690	1.190	1.040	1.010	1.280	1.355	1.261
Fe	4.900	3.265	3.140	3.475	3.840	4.795	3.902
Mn	0.103	0.022	0.021	0.020	0.037	0.026	0.038
Mo	0.000	0.000	0.000	0.000	0.060	0.075	0.022
Ni	0.000	0.000	0.000	0.070	0.000	0.000	0.012
Pb	0.430	0.000	0.000	0.515	0.000	0.000	0.158
Sb	0.000	0.435	0.000	0.000	0.000	0.000	0.072
V	0.040	0.000	0.015	0.020	0.030	0.000	0.018
Zn	13.88	8.755	8.300	6.065	9.080	9.560	9.273

Six blank Ghost Wipes were analyzed, after each being used to wipe a separate cleaned glass plate, to determine interferences due to contamination from the glass surface, media, and reagents.

Table 1.6.1.2
Contamination to the Analysis from the Glass Surface, Media, and Reagents
(µg found)

analyte	wipe 1	wipe 2	wipe 3	wipe 4	wipe 5	wipe 6	average
Be	0.000	0.000	0.000	0.000	0.002	0.000	0.000
Cd	0.030	0.020	0.020	0.005	0.005	0.020	0.017
Co	0.090	0.055	0.090	0.075	0.000	0.145	0.076
Cr	0.155	0.355	0.220	0.070	0.370	0.370	0.257
Cu	1.325	1.170	1.015	1.040	1.340	1.405	1.216
Fe	4.865	5.130	4.425	4.170	6.200	6.130	5.153
Mn	0.132	0.198	0.138	0.088	0.120	0.205	0.147
Mo	0.125	0.355	0.410	0.000	0.000	0.165	0.176
Ni	0.440	0.450	0.470	0.355	0.305	0.675	0.449
Pb	0.000	0.000	0.335	0.245	0.640	0.120	0.223
Sb	1.390	0.950	0.000	0.360	0.525	1.240	0.744
V	0.105	0.055	0.040	0.000	0.020	0.015	0.039
Zn	21.59	12.30	11.07	14.79	12.07	9.970	13.63

1.6.2 Spectral Interferences

The following Inter-Element Corrections (IEC) were used to correct for spectral interferences:

Table 1.6.2.1
Radial ICP IEC Factors[a,b,c]

analyte & wavelength	interfering analytes								
	Co	Cr	Cu	Fe	Mn	Mo	Ni	Ti	V
Cd 313.107	-	-	-	0.0576	-	-	-	-	-
Co 214.44	-	-	-	-	-	-	0.1681	1.6467	-
Cr 228.616	-	-	-	-	0.2	-	-	-	-
Cu 267.716	-	-	-	-	-	0.4168	-	-	-
Fe 238.204	-	0.0478	-	-	-	-	-	-	-
Ni 232.003	-	47.35	-	-	-	6.187	-	-	-
Pb 220.353	0.1	-	-	-	-	-	-	-	-
Sb 206.836	0.7907	11.628	-	-	-	-	-	-	-
V 292.402	-	-	-	-	-	-	-	0.8331	-
Zn 213.857	-	-	1.329	0.0518	-	-	4.691	0.2	-

a The IEC factor[2] is the apparent analyte concentration in ppb divided by the interferent concentration in ppm and is calculated as follows:

$$\text{IEC Factor} = \frac{\text{Apparent Analyte Concn (ppb)}}{\text{Interferent Concn (ppm)}}$$

Where the apparent analyte concentration is the concentration of the interferent measured at the analyte wavelength in the absence of the analyte.

The corrected analyte concentration is calculated as follows:

---

[2] The Perkin-Elmer Corporation. *WinLab32 Instrument Control Software Guide*; Norwalk, Ct, 1999.

Corrected Analyte Concn (ppb) =
Measured Analyte Concn (ppb) - (Interferent Concn (ppm) * IEC Factor)

b A dash indicates that no interference was observed on the analyte from the interfering analyte. Interfering analytes were analyzed at the following concentration:

200 ppm:	Fe
100 ppm:	Cr, Cu, Co, Mo, Ni
50 ppm:	Mn, V, Ti

c IEC factors are instrument dependent. Interferences listed in this table do not represent all possible interferences.

1.7 Solubility and Stability of Lead Sulfate

The solubility of lead sulfate was investigated by weighing out various amounts and placing it in a conical beaker, along with a Ghost Wipe, and digested. The samples were reanalyzed four days latter to determine if any lead had precipitated out of solution. Results were not blank corrected.

Table 1.7.1
Lead Sulfate Solubility and Stability

		day 1		day 4	
$PbSO_4$ used (mg)	theoretical for lead* (µg)	lead found (µg)	recovery (%)	lead found (µg)	recovery (%)
0.025	16.73	15.92	95.2	16.82	100.5
0.078	52.23	49.82	95.4	50.30	96.3
0.162	108.5	105.9	97.6	107.8	99.3
0.230	154.0	153.2	99.5	155.2	100.7
0.252	168.7	162.7	96.4	167.0	98.9
0.477	319.4	313.7	98.2	319.8	100.1
0.595	398.4	395.9	99.4	402.7	101.1
0.698	467.4	472.6	101.1	472.6	101.1
0.821	549.7	544.1	99.0	555.7	101.1
1.125	753.3	739.1	98.1	751.2	99.7
1.254	839.6	826.1	98.4	838.7	99.9
1.715	1148	1121	97.6	1144	99.6
2.025	1356	1326	97.8	1346	99.3
2.738	1833	1821	99.3	1845	100.6
4.584	3069	2993	97.5	3048	99.3

* Purity of lead sulfate used was 98%

1.8 Analysis of three NIST Standard Reference Materials

Approximately 100 mg of material was placed in a conical beaker along with a Ghost Wipe. Samples were digested and then filtered through an MCE filter to remove all particulate. The MCE filter for each sample, along with the remaining particulate, were digested a second time. Results were not blank corrected.

Table 1.8.1
Standard Reference Material 2580 Lead Paint

element	sample 1 weight = 101.6 mg				sample 2 weight = 100.9 mg				sample 3 weight = 100.6 mg			
	first digest (µg)	second digest (µg)	theor (µg)	recovery (%)	first digest (µg)	second digest (µg)	theor (µg)	recovery (%)	first digest (µg)	second digest (µg)	theor (µg)	recovery (%)
Pb	2466	1653	4409	93.4	2345	992.0	4379	76.2	1730	1674	4366	78.0
Fe*	533.5	27.08	508.0	110	520.0	17.50	504.0	107	513.0	24.42	503.0	107
Zn*	2842	127.1	3048	97.4	2790	83.95	3027	94.9	2750	114.2	3018	94.9

* Results not certified by NIST

Table 1.8.2
Standard Reference Material 2583 Trace Elements in Indoor Dust

element	sample 1 weight = 103.8 mg				sample 2 weight = 100.3 mg				sample 3 weight = 100.4 mg			
	first digest (µg)	second digest (µg)	theor (µg)	recovery (%)	first digest (µg)	second digest (µg)	theor (µg)	recovery (%)	first digest (µg)	second digest (µg)	theor (µg)	recovery (%)
Cd	0.565	0.000	0.757	74.6	0.695	0.000	0.732	94.9	0.465	0.000	0.733	63.4
Cr	5.215	0.900	8.304	73.6	5.585	0.905	8.024	80.8	5.395	0.755	8.032	76.6
Pb	6.820	0.365	8.916	80.6	6.425	0.050	8.616	75.2	6.710	0.160	8.624	79.7

Table 1.8.3
Standard Reference Material 1648 Urban Particulate Matter

element	sample 1 weight = 101.4 mg				sample 2 weight = 101.7 mg				sample 3 weight = 101.1 mg			
	first digest (µg)	second digest (µg)	theor (µg)	recovery (%)	first digest (µg)	second digest (µg)	theor (µg)	recovery (%)	first digest (µg)	second digest (µg)	theor (µg)	recovery (%)
Pb	608.0	30.88	664.2	96.2	618.0	33.12	666.1	97.8	595.0	30.35	662.2	94.4
Cd	7.185	0.255	7.605	97.8	7.215	0.280	7.628	98.3	7.185	0.270	7.583	98.3
Cr	10.38	3.990	40.86	35.2	9.595	2.920	40.99	30.5	10.76	3.185	40.74	34.2
Cu	53.95	2.925	61.75	92.1	54.70	3.020	61.94	93.2	53.50	2.645	61.57	91.2
Mn	71.80	4.101	79.70	95.2	73.63	4.495	79.93	97.7	71.44	3.766	79.46	94.6
Ni	6.600	0.490	8.315	85.3	6.660	0.500	8.339	85.9	6.760	0.530	8.290	87.9
V	9.805	0.990	12.88	83.8	9.725	1.070	12.92	83.6	9.680	0.900	12.84	82.4

1.9 Instrument Analytical Conditions

Instrument:	Perkin-Elmer Optima 4300 DV ICP
Replicates:	2
Read Time:	Auto, 5-20 sec
Sample Flow Rate:	2.20 mL/min
Gas:	Argon
Plasma Gas Flow:	15 L/min
Auxiliary Gas Flow:	0.20 L/min
Nebulizer Gas Flow:	0.60 L/min
RF Power:	1300 watts
Plasma View:	Radial
View Distant:	15.0 mm
Peak Algorithm:	Peak Area
Overlap Correction:	IEC
Background Correction:	2-Point
Calibration:	2-Point