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ANALYSIS OF METALS IN AIR WITH A MODIFIED NIOSH 7300 METHOD

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1.0 SCOPE AND APPLICATION

This method is a modification of NIOSH Method 7300 for metals in air (Appendix B). It is applicable to the analysis of indoor air samples collected on cellulose ester membrane filters. The filter sample is digested and metal concentrations are determined by Graphite Furnace Atomic Absorption (GFAA) or inductively coupled argon plasma (ICAP) spectroscopy techniques. The method is simple, rapid, and relatively free of matrix interferences.

Detection limits, sensitivity, and optimum ranges for metals analysis will vary with the filter material, volume of air sampled, and models of GFAA or ICAP instruments used.

These are standard (i.e., typically applicable) operating procedures, which may be varied or changed as required, depending upon site conditions, equipment limitations or limitations imposed by the procedure. In all instances, the ultimate procedures employed will be documented and associated with the final report.

Mention of trade names or commercial products does not constitute United States Environmental Protection Agency (U.S. EPA) endorsement or recommendation for use.

2.0 METHOD SUMMARY

Indoor air samples are collected on cellulose ester membrane filters contained in cassette filter holders according to NIOSH Method 7300. The filter from the cassette holder is quantitatively transferred to a clean 50-milliliter (mL) beaker and digested in nitric acid. After digestion is complete, the sample is transferred to a 25-mL volumetric flask and diluted to volume with dilution acid.

The sample is analyzed by ICAP and/or GFAA methods.

3.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING, AND STORAGE

All samples must have been collected according to NIOSH Method 7300. After collection, samples must be stored and packed securely for shipment.

4.0 INTERFERENCES AND POTENTIAL PROBLEMS

Spectral interferences are the primary interferences encountered in ICAP analysis. These are minimized by judicious wavelength selection, inter-element correction factors, and background correction. Cross-contamination and contamination of the sample can be major sources of error because of the sensitivities achieved with the GFAA spectroscopy technique. The sample preparation work area should be kept scrupulously clean. All glassware should be cleaned as directed in Section 5.4.

5.0 EQUIPMENT/APPARATUS

5.1 Graphite Furnace Atomic Absorption (GFAA) Spectrophotometer

Single- or dual-channel, single- or double-beam instrument having a grating monochromator, photomultiplier detector, adjustable slits, a wavelength range of 190 to 800 nm, and provisions for interfacing with a strip chart recorder or computer, printer, and autosampler.



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5.2 Strip-Chart Recorder, Integrator, or Printer

A recorder is useful to provide a permanent record and for easy recognition of any problems with the analysis.

5.3 Inductively Coupled Argon Plasma (ICAP) Spectrometer

A computer-controlled simultaneous ICAP emission spectrometer with inter-element and background correction capabilities and provisions for interfacing to a printer and an autosampler should be used.

5.4 Glassware and Containers

All glassware, polypropylene, or Teflon[®] containers, including sample bottles, should be washed in the following sequence: detergent, tap water, 1:1 nitric acid, tap water, 1:1 hydrochloric acid, tap water, and Type I water. If it can be documented through an active analytical quality control program, using spiked samples and reagent blanks, that certain steps in the cleaning procedure are not required for routine samples, those steps may be eliminated from the procedure.

5.5 Hotplate

Automatic temperature control, capable of reaching a surface temperature of $150^{\circ}\text{C} \pm 5^{\circ}\text{C}$.

6.0 REAGENTS

All standard solutions are prepared and documented in accordance with ERT/SERAS SOP #1012, Preparation of Standard Solutions.

6.1 Type I Water (ASTM D1193)

Use Type I water for the preparation of all reagents and calibration standards and as dilution water.

6.2 Concentrated Nitric Acid (HNO_3)

Use commercially available high-purity, spectro grade acid certified for ICAP/GFAA use.

6.3 Dilution Acid (4% HNO_3)

Add 40 mL concentrated HNO_3 to 500 mL Type I water; dilute to 1-L.

6.4 Calibration Stock Solutions

Use commercially available standard solutions, typically 1000 micrograms per milliliter ($\mu\text{g}/\text{mL}$).

6.5 Calibration Standards

Appropriate concentrations for standards must be selected depending on the typical analytical range of the



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instrumentation used. A calibration blank and one or more calibration standards should be prepared containing all elements of interest by combining appropriate volumes of the stock standard solutions in volumetric flasks. The calibration standards should be prepared using the same type or combination of acid(s) and in the same concentration range that will be present in the samples after they are processed.

For GFAA instruments that do not read out directly in concentration, a calibration curve is prepared to cover the appropriate concentration range. For best results, intermediate and working standards should be prepared fresh each time a batch of samples is analyzed. Standards may also be preserved at 4°C and used until their expiration date (maximum 6 months). A blank and a minimum of three working standards in the linear part of the calibration range should be used to calibrate the GFAA instrument.

7.0 PROCEDURES

7.1 Sample Preparation

1. Open the cassette holder and quantitatively transfer the filter sample to a clean 50-mL beaker.
2. Add 5 mL HNO₃, cover with a watchglass, and let stand 30-minutes at room temperature.
NOTE: All digestions must be performed under a working fume hood.
3. Heat on the hotplate (120°C) until approximately 0.5 mL of sample remains.
4. Add 2 mL HNO₃ and repeat step 3. Repeat this step until the solution is clear.
5. Remove watch glass and rinse into the beaker with Type I water.
6. Increase the temperature to 150°C and heat until approximately 0.5 mL of sample remains.
7. Transfer the solution quantitatively to a 25-mL volumetric flask and dilute to volume with dilution acid.

7.2 Media Blank

Prepare the media blank (lot blank) using blank filter media from the same lot as the unknown samples. Prepare the media blank using the procedure outlined in Section 7.1 at the same time the samples are prepared. A minimum of three media blanks per batch of samples (not to exceed 20 samples) must be prepared.

7.3 Media Blank Spike/Blank Spike Duplicate

Prepare the media blank spike (BS) and blank spike duplicate (BSD) samples by spiking blank filter media with known amounts of metals (see Table 1, Appendix A) using the procedure outlined in Section 7.1. BS/BSD samples are prepared in the laboratory to monitor the precision and accuracy of the method. Use the BS/BSD samples delivered to the laboratory with the unknown samples, or media blanks from the same lot, if none are provided with the samples.

7.4 Calibration and Measurement



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Differences between the various makes and models of satisfactory ICAP and GFAA instruments prevent the formulation of detailed instructions applicable to each system. The analyst should follow the manufacturer's operating instructions for a particular instrument.

Analyze the working standards together with the samples and blanks. Analyze the full set of working standards at the beginning of the run to establish the calibration curve. Analyze additional standards during sample analysis to confirm the initial calibration and continuing instrument response (see Section 9.0).

Samples with high concentrations of metals must be diluted into the linear calibration range (see Section 9.6).

8.0 CALCULATIONS

Read the metal concentration (B) in $\mu\text{g/L}$ from the calibration curve or directly from the read-out system of the instrument. Calculate the metal concentration in the sample (A) as follows:

$$A = \mu\text{g/L metal sample} = \frac{D + C}{D} B$$

where:

- B = Concentration of metal ($\mu\text{g/L}$)
- C = Amount of acid blank matrix used for dilution (mL)
- D = Sample aliquot used for dilution (mL)

Note: If a dilution was not required, $C = 0$, and $A = B$.

Calculate the amount of metal (W) for each filter:

$$W = \mu\text{g/filter metal} = A (0.025)$$

where:

- A = Concentration of metal in the sample ($\mu\text{g/L}$)
- 0.025 = Final solution volume (L)

Calculate the concentration ($\mu\text{g/m}^3$) of metal in the air volume sampled:

$$\mu\text{g/m}^3 \text{ metal} = 1000 \frac{W - BLK}{V}$$

where:

- W = Amount of metal in each filter ($\mu\text{g/filter}$)



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V = Volume of air sampled (L)
 BLK = Concentration in the media blank ($\mu\text{g}/\text{filter}$)

NOTE: BLK is the average of the media blank results (minimum 3). For blank values $<MDL$, substitute zero (0) for the raw data prior to calculating the average. The calculated average is then subtracted from each sample even if the average is $<MDL$.

NOTE: Report $\mu\text{g}/\text{filter}$ results for lot blank, trip blank, field blank(s), and BS/BSD samples because no air volume is collected for these samples.

9.0 QUALITY ASSURANCE/QUALITY CONTROL

All quality control (QC) data must be documented and available for reference or inspection.

9.1 Initial Calibration Verification (ICV)

The GFAA or ICAP instruments must be calibrated each day with a blank and the appropriate number of calibration standards (Section 7.4). The initial calibration must be verified by analysis of an ICV standard, the calibration blank, QC standards, and media blanks (Section 7.2). The ICV standard should be prepared (using an independent source) at or near the mid-range concentration for most elements, and must be within the calibration range for all elements. The ICV results must be within the Performance Acceptance Limits (PALs) supplied by the vendor. The QC standards should be prepared using an independent source and must have concentrations within the calibration range for all elements. QC standard results must be within the PALs supplied by the vendor. Calibration blank results must be less than the MDL.

9.2 Continuing Calibration Verification (CCV)

The working standard curve must be verified by analyzing the CCV standard and the calibration blank after every 10 samples and after the final sample. CCV results must be within ± 10 percent of the true value for ICAP analysis and ± 20 percent for GFAA analysis. Calibration blank results must be less than the MDL.

9.3 BS/BSD Samples

A minimum of one media BS and BSD sample must be analyzed with each batch of samples to verify precision and accuracy of the method.

BS/BSD percent recovery (%R) should be within 75-125 percent (advisory only) and calculated as:

$$\%R = \frac{SSR - BLK}{SA} \times 100$$

where:

SSR = spiked (BS or BSD) sample result ($\mu\text{g}/\text{filter}$)
SA = Spike added ($\mu\text{g}/\text{filter}$)



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BLK = Concentration in the media blank ($\mu\text{g}/\text{filter}$)

NOTE: BLK is the average of the media blank results. For blank values $<MDL$, substitute zero (0) for the raw data prior to calculating the average. The calculated average is then subtracted from each sample even if the average is $<MDL$.

The Relative Percent Difference (RPD) for duplicate samples (BS/BSD) should be within 80-120 percent (advisory only) and calculated as:

$$RPD = \frac{(BSR - BSDR)}{(BSR + BSDR)/2} \times 100$$

where:

BSR = %R for BS sample
 $BSDR$ = %R for BSD sample

9.4 Linear Analytical Range

For ICAP analyses, one or more linear analytical range (LAR) standards must be analyzed to determine the maximum linear range of the calibration for each element. Recovery must be within 90-110%. If the recovery is outside these limits, the maximum calibration standard concentration defines the linear range. LAR standards must be analyzed and reported on a quarterly basis.

For GFAA analyses, the maximum calibration standard concentration defines the linear range.

9.5 Serial dilution

A five-fold dilution of a typical sample is prepared and analyzed. The concentration in the undiluted sample must be greater than or equal to (\geq) 50x the Instrument Detection Limit (IDL) and the diluted sample must be $\geq 10x$ the IDL in order to obtain a meaningful comparison. Results are multiplied by the dilution factor and compared to the original determination (undiluted sample). Agreement within $\pm 10\%$ between the concentrations for the undiluted sample and the diluted sample indicates the absence of matrix interferences. If recovery is outside these limits, a chemical or physical effect should be suspected.

If the concentrations of all analytes in all samples are less than 50x the IDL, serial dilution will not be performed.

9.6 Dilution Analysis

If the concentration of any analyte in any sample exceeds the linear range, the sample must be diluted and reanalyzed. An appropriate dilution or series of dilutions (for example, 5x, 10x, 20x) may be required depending on the concentration in the undiluted sample. Results are reported from the lowest dilution that falls within the linear range.



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For analytes that saturate the detector, a series of dilutions is required. For a series of dilutions, comparisons are first made with respect to the undiluted sample and then, within the series. Based on the analyst's professional judgement, results are reported from the diluted sample that has the smallest dilution factor and indicates the absence of interferences.

10.0 DATA VALIDATION

Data will be assessed by the Data Validation and Report Writing Group using the most current revision of the ERT/SERAS SOP #1017, *Data Validation Procedure for Routine Inorganic Analysis*. However, data is considered satisfactory for submission purposes when all of the requirements listed in the method are met.

11.0 HEALTH AND SAFETY

The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined. However, each chemical compound should be treated as a potential health hazard. The laboratory is responsible for following the chemical hygiene plan and laboratory safety program regarding the safe handling of the chemicals specified in this method.

When working with potentially hazardous materials, refer to U.S. EPA, Occupational Safety and Health Administration (OSHA) and corporate health and safety practices. More specifically, refer to ERT/SERAS SOP #3013, *SERAS Laboratory Safety Program*.

The analyst should consult all appropriate MSDS information prior to running an analysis for the first time.

12.0 REFERENCES

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2. National Institute for Occupational Safety and Health. 1996. *NIOSH Manual of Analytical Methods. Method 7300*. 4th ed. Suppl.
3. ERT/SERAS SOP #1818, *Determination of Metals by Atomic Absorption (AA) Methods*.
4. ERT/SERAS SOP #1811, *Determination of Metals by Inductively Coupled Plasma (ICP) Methods*.

13.0 APPENDICES

- A. Tables
- B. NIOSH Method 7300 for Metals in Air



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APPENDIX A
Tables
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Table 1. Typical Spike Ranges for Blank Spike (BS) / Blank Spike Duplicate (BSD)	
Metal	Spike Amount ($\mu\text{g}/\text{filter}$)
Aluminum	50 – 200
Antimony	0.50 - 2.00
Arsenic	0.50 - 2.00
Barium	2.50 - 10.0
Beryllium	2.50 - 10.0
Cadmium	2.50 - 10.0
Calcium	50 – 200
Chromium	2.50 - 10.0
Cobalt	2.50 - 10.0
Copper	2.50 - 10.0
Iron	50 – 200
Lead	0.50 - 2.00
Magnesium	50 - 200
Manganese	2.50 - 10.0
Nickel	2.50 - 10.0
Potassium	100 - 400
Selenium	0.50 - 2.00
Silver	2.50 - 10.0
Sodium	50 - 200
Thallium	0.50 - 2.00
Vanadium	2.50 - 10.0
Zinc	2.50 - 10.0



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APPENDIX B
NIOSH Method 7300 for Metals in Air
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ELEMENTS by ICP 7300

MW: Table 1 CAS: Table 2 RTECS: Table 2

METHOD: 7300, Issue 2 EVALUATION: PARTIAL Issue 1: 15 May 1989
 Issue 2: 15 August 1994

OSHA : Table 2 PROPERTIES: Table 1
 NIOSH: Table 2
 ACGIH: Table 2

ELEMENTS:	aluminum*	chromium*	lithium*	phosphorus	tellurium	zinc
	arsenic	cobalt*	magnesium	platinum*	thallium	zirconium*
	beryllium*	copper	manganese*	selenium	titanium	
	cadmium	iron	molybdenum*	silver	vanadium	
	calcium	lead*	nickel	sodium	yttrium	

* Some compounds of those elements require special sample treatment.

SAMPLING		MEASUREMENT	
SAMPLER:	FILTER (0.8-µm, cellulose ester membrane)	TECHNIQUE:	INDUCTIVELY COUPLED ARGON PLASMA, ATOMIC EMISSION SPECTROSCOPY
FLOW RATE:	1 to 4 L/min	ANALYTE:	elements above
VOL-MIN:	Table 1	ASHING	
-MAX:	Table 1	REAGENTS:	conc. HNO ₃ , 4 mL; and conc. HClO ₄ , 1 mL
SHIPMENT:	routine	CONDITIONS:	room temperature, 30 min; 150 °C to near dryness
SAMPLE STABILITY:	stable	FINAL SOLUTION:	4% HNO ₃ , 1% HClO ₄ , 10 mL
BLANKS:	2 to 10 field blanks per set	WAVELENGTH:	depends upon element; Table 3
ACCURACY		BACKGROUND CORRECTION:	spectral wavelength shift
RANGE STUDIED:	not studied	CALIBRATION:	elements in 4% HNO ₃ , 1% HClO ₄
BIAS:	none identified	RANGE:	2.5 to 1000 µg per sample [1]
OVERALL PRECISION (S_{r,r}):	not evaluated	ESTIMATED LOD:	1 µg per sample [1]
ACCURACY:	not determined	PRECISION (S_r):	Table 3

APPLICABILITY: The working range of this method is 0.005 to 2.0 mg/m³ for each element in a 500-L air sample. This is simultaneous elemental analysis, not compound specific. An alternative microwave digestion procedure is included. Verify that the types of compounds in the samples are soluble with the ashing procedure selected.

INTERFERENCES: Spectral interferences are the primary interferences encountered in ICP-AES analysis. These are minimized by judicious wavelength selection, interelement correction factors and background correction [1,2].



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OTHER METHODS: This method replaces P&CAM 351 [2] for trace elements. Flame atomic absorption spectroscopy (e.g., Methods 70XX) is an alternate analytical technique for many of these elements. Graphite furnace AAS (e.g., 7102 for Be, 7105 for Pb) is more sensitive.

REAGENTS:

1. Nitric acid, conc., ultra pure.
2. Perchloric acid, conc., ultra pure.*
3. Ashing acid: 4:1 (v/v) HNO_3 : HClO_4 . Mix 4 volumes conc. HNO_3 with 1 volume conc. HClO_4 .
4. Calibration stock solutions, 1000 $\mu\text{g/mL}$. Commercially available, or prepared per instrument manufacturer's recommendation (see step 12).
5. Dilution acid, 4% HNO_3 , 1% HClO_4 . Add 50 mL ashing acid to 600 mL water; dilute to 1 L.
6. Argon.
7. Distilled, deionized water.

* See SPECIAL PRECAUTIONS.

EQUIPMENT:

1. Sampler: cellulose ester membrane filter, 0.8- μm pore size, 37-mm diameter; in cassette filter holder.
2. Personal sampling pump, 1 to 4 L/min, with flexible connecting tubing.
3. Inductively coupled plasma-atomic emission spectrometer, equipped as specified by the manufacturer for analysis of elements of interest.
4. Regulator, two-stage, for argon.
5. Beakers, Phillips, 125-mL, or Griffin, 50-mL, with watchglass covers.**
6. Volumetric flasks, 10- and 100- mL.**
7. Assorted volumetric pipets as needed.**
8. Hotplate, surface temperature 150 °C.

** Clean all glassware with conc. nitric acid and rinse thoroughly in distilled water before use.

SPECIAL PRECAUTIONS: Perform all perchloric acid digestions in a perchloric acid hood.

SAMPLING:

1. Calibrate each personal sampling pump with a representative sampler in line.
2. Sample at an accurately known flow rate between 1 and 4 L/min for a total sample size of 200 to 2000 L (see Table 1) for TWA measurements. Do not exceed a filter loading of approximately 2 mg total dust.

SAMPLE PREPARATION:

3. Open the cassette filter holders and transfer the samples and blanks to clean beakers.
4. Add 5 mL ashing acid. Cover with a watchglass. Let stand 30 min at room temperature.
NOTE: Start a reagent blank at this step.
5. Heat on hotplate (120 °C) until ca. 0.5 mL remains.
NOTE 1: Recovery of lead from some paint matrices may require other digestion techniques. See Method 7082 (Lead by Flame AAS) for an alternative hotplate digestion procedure or the Appendix for a microwave digestion procedure [8].
NOTE 2: Some species of Al, Be, Co, Cr, Li, Mn, Mo, V, and Zr will not be completely solubilized by this procedure. Alternative solubilization techniques for most of these elements can be found elsewhere [2-7]. For example, aqua regia may be needed for Mn [4,9].
6. Add 2 mL ashing acid and repeat step 5. Repeat this step until the solution is clear.
7. Remove watchglass and rinse into the beaker with distilled water.
8. Increase the temperature to 150 °C and take the sample to near dryness (ca. 0.5 mL).
9. Dissolve the residue in 2 to 3 mL dilution acid.
10. Transfer the solutions quantitatively to 10-mL volumetric flasks.
11. Dilute to volume with dilution acid.



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ELEMENTS (ICP): METHOD 7300, Issue 2, dated 15 August 1994 - Page 3 of 10



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ELEMENTS (ICP): METHOD 7300, Issue 2, dated 15 August 1994 - Page 4 of 10

CALIBRATION AND QUALITY CONTROL:

12. Calibrate the spectrometer according to the manufacturers recommendations.
NOTE: Typically, an acid blank and 10 µg/mL multielement working standards are used. The following multielement combinations are chemically compatible in 4% HNO₃/1% HClO₄:
 - a. Ag, Ca, Co, Mn, Pb, V, Zn;
 - b. Al, Be, Cd, La, Li, Ni, Ti;
 - c. As, B, Ba, Mg, Mo, P;
 - d. Cu, Fe, Na, Pt, Sr, Te, Y;
 - e. Cr, K, Se, Ti, Zr; and
 - f. Si, W (distilled water only)
13. Analyze a standard for every ten samples.
14. Check recoveries with at least two spiked media blanks per ten samples.

MEASUREMENT:

15. Set spectrometer to conditions specified by manufacturer.
16. Analyze standards and samples.
NOTE: If the values for the samples are above the range of the standards, dilute the solutions with dilution acid, reanalyze and apply the appropriate dilution factor in the calculations.

CALCULATIONS:

17. Obtain the solution concentrations for the sample, C_s (µg/mL), and the average media blank, C_b (µg/mL), from the instrument.
18. Using the solution volumes of sample, V_s (mL), and media blank, V_b (mL), calculate the concentration, C (mg/m³), of each element in the air volume sampled, V (L):

$$C = \frac{C_s V_s - C_b V_b}{V}, \text{ mg/m}^3.$$

EVALUATION OF METHOD:

Method P&CAM 351 was evaluated in 1981 [1,2]. The precision and recovery data were determined at 2.5 and 1000 µg of each element per sample on spiked filters. The precision and recovery data, instrumental detection limits, sensitivity, and analytical wavelengths are listed in Table 3. The values in Table 3 were determined with a Jarrell-Ash Model 1160 ICP operated according to manufacturer's instructions.

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- [2] NIOSH Manual of Analytical Methods, 2nd ed., V. 7, P&CAM 351, U.S. Department of Health and Human Services, Publ. (NIOSH) 82-100 (1981).
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- [5] Ibid, V. 4, P&CAM 271 (Tungsten), U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 78-175 (1978).
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METHOD WRITTEN BY:

Mark Millson, NIOSH/DPSE, and R. DeLon Hull, Ph.D., NIOSH/DBBS.

James B. Perkins, David L. Wheeler, and Keith Nicholson, DataChem Laboratories, Salt Lake City, UT, prepared the microwave digestion procedure in the Appendix.



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TABLE 1. PROPERTIES AND SAMPLING VOLUMES

Element (Symbol)	Properties		Air Volume, L @ OSHA PEL	
	Atomic Weight	MP, °C	MIN	MAX
Silver (Ag)	107.87	961	250	2000
Aluminum (Al)	26.98	660	5	100
Arsenic (As)	74.92	817	5	2000
Beryllium (Be)	9.01	1278	1250	2000
Calcium (Ca)	40.08	842	5	200
Cadmium (Cd)	112.40	321	13	2000
Cobalt (Co)	58.93	1495	25	2000
Chromium (Cr)	52.00	1890	5	1000
Copper (Cu)	63.54	1083	5	1000
Iron (Fe)	55.85	1535	5	100
Lithium (Li)	6.94	179	100	2000
Magnesium (Mg)	24.31	651	5	67
Manganese (Mn)	54.94	1244	5	200
Molybdenum (Mo)	95.94	651	5	67
Sodium (Na)	22.99	98	13	2000
Nickel (Ni)	58.71	1453	5	1000
Phosphorus (P)	30.97	44	25	2000
Lead (Pb)	207.19	328	50	2000
Platinum (Pt)	195.09	1769	1250	2000
Selenium (Se)	78.96	217	13	2000
Tellurium (Te)	127.60	450	25	2000
Titanium (Ti)	47.90	1675	5	100
Thallium (Tl)	204.37	304	25	2000
Vanadium (V)	50.94	1890	5	2000
Yttrium (Y)	88.91	1495	5	1000
Zinc (Zn)	65.37	419	5	200
Zirconium (Zr)	91.22	1852	5	200



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TABLE 2. EXPOSURE LIMITS, CAS #, RTECS

Element (Symbol)	CAS #	RTECS	OSHA	Exposure Limits, mg/m ³ (Ca = carcinogen)	
				NIOSH	ACGIH
Silver (Ag)	7440-22-4	VW350000 0	0.01 (dust, fume, metal)	0.01 (metal, soluble)	0.1 (metal) 0.01 (soluble)
Aluminum (Al)	7429-90-5	BD0330000	15 (total) 5 (respirable)	5	10 (dust) 5 (fume)
Arsenic (As)	7440-38-2	CG0525000	varies	C 0.002, Ca	0.01, Ca
Beryllium (Be)	7440-41-7	DS1750000	0.002, C 0.005	0.0005, Ca	0.002, Ca
Calcium (Ca)	--	--	varies	varies	varies
Cadmium (Cd)	7440-43-9	EU9800000	0.2, C 0.6 (dust) 0.1, C 0.3 (fume)	lowest feasible, Ca	0.01 (total), Ca 0.002 (respir.), Ca
Cobalt (Co)	7440-48-4	GF8750000	0.1	0.05	0.05 (dust, fume)
Chromium (II) (Cr)	22541-79-3	GB6260000	0.5	0.5	0.5
Chromium (III) (Cr)	16065-83-1	GB6261000	0.5	0.5	0.5
Chromium (VI) (Cr)	18540-29-9	GB6262000	C 0.1	0.001 (dust)	0.05 (soluble) 0.05 (insoluble), Ca
Copper (Cu)	7440-50-8	GL5325000	1 (dust, mists) 0.1 (fume)	1 (dust) 0.1 (fume)	1 (dust, mists) 0.2 (fume)
Iron (Fe)	1309-37-1	NO7400000	10 (dust, fume)	5 (dust, fume)	5 (fume)
Lithium (Li)	--	--	--	--	--
Magnesium (Mg)	1309-48-4	OM3850000	15 (dust) as oxide 5 (respirable)	10 (fume) as oxide	10 (fume) as oxide
Manganese (Mn)	7439-96-5	OO9275000	C 5	1; STEL 3	5 (dust) 1; STEL 3 (fume)
Molybdenum (Mo)	7439-98-7	QA4680000	5 (soluble) 15 (total insoluble) 5 (respirable insol.)	5 (soluble) 10 (insoluble)	5 (soluble) 10 (insoluble)
Nickel (Ni)	7440-02-0	QR5950000	1	0.015, Ca	0.05, Ca
Lead (Pb)	7439-92-1	OF7525000	0.05	<0.1	0.15
Platinum (Pt)	7440-06-4	TP2160000	0.002	1 (metal)	1 (metal)
Selenium (Se)	7782-49-2	VS7700000	0.2	0.2	0.2
Tellurium (Te)	13494-80-9	WY2625000	0.1	0.1	0.1
Titanium (Ti) TiO ₂	7440-32-6 13463-67-7	XR1700000 XR2275000	as TiO ₂ , 15 as TiO ₂ , 5 (respirable)	lowest feasible, Ca	10
Thallium (Tl)	7440-28-0	XG3425000	0.1 (skin) (soluble)	0.1 (skin) (soluble)	0.1 (skin)
Vanadium (V) V ₂ O ₅	7440-62-2 1314-62-1	YW2400000 YW1355000	C 0.5 (respirable) as V ₂ O ₅ C 0.1 (fume) as V ₂ O ₅	C 0.05	0.05 (respir.) as V ₂ O ₅
Yttrium (Y)	7440-65-5	ZG2980000	1	1	1
Zinc (Zn)	1314-13-2	ZH4810000	5 (ZnO fume) 15 (ZnO dust) 5 (ZnO respirable)	5; STEL 10 (ZnO fume) 5; C 15 (ZnO dust)	5; STEL 10 (ZnO fume) 10 (ZnO dust)



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Element (Symbol)	CAS #	RTECS	OSHA	Exposure Limits, mg/m ³ (Ca = carcinogen)	
				NIOSH	ACGIH
Silver (Ag)	7440-22-4	VW350000 0	0.01 (dust, fume, metal)	0.01 (metal, soluble)	0.1 (metal) 0.01 (soluble)
Zirconium (Zr)	7440-67-7	ZH7070000	5	5, STEL 10	5, STEL 10

TABLE 3. MEASUREMENT PROCEDURES AND DATA^(a).

Element	Wavelength (nm)	Instrumental LOD (ng/mL)	Sensitivity (Intensity/ µg/mL)	Recovery		Precision (\bar{S}_r) (N = 3)	
				@ 2.5 µg/ filter ^(b)	@ 1000 µg/ filter	@ 2.5 µg/ filter	@ 1000 µg/ filter
Ag	328.3	26	0.65	111	91	0.02	0.075
Al	308.2	14	0.23	93	100	0.092	0.023
As	193.7	13	0.57	103	99	0.062	0.026
Be	313.0	1.5	1.29	107	90	0.040	0.034
Ca	315.9	10	0.49	99	95	0.036	0.014
Cd	226.5	1.6	0.83	107	99	0.032	0.020
Co	231.2	7.4	0.38	101	95	0.040	0.005
Cr	205.6	1.3	0.50	98	106	0.053	0.016
Cu	324.8	2.1	0.72	98	99	0.036	0.022
Fe	259.9	3.9	0.13	94	97	0.068	0.016
Li	670.8	2.8	0.48	89	95	0.171	0.043
Mg	279.6	24	0.22	105	106	0.084	0.027
Mn	257.6	0.4	0.74	84	93	0.062	0.035
Mo	281.6	7.0	0.18	94	88	0.023	0.049
Na	589.0	10	0.76	(c)	101	(c)	0.045
Ni	231.6	3.4	0.41	105	97	0.027	0.020
P	214.9	22	0.17	(c)	91	(c)	0.056
Pb	220.4	17	0.42	105	95	0.060	0.011
Pt	203.7	15	0.69	106	91	0.041	0.075
Se	190.6	21	0.28	105	97	0.068	0.049
Sn ^(d)	190.0	64	0.49	74	67	0.33	0.16
Te	214.3	29	0.41	102	94	0.050	0.063
Ti	334.9	1.2	0.55	96	108	0.051	0.029
Tl	190.9	17	0.22	103	99	0.043	0.017
V	310.2	3.2	0.88	99	94	0.043	0.014
W ^(d)	207.9	13	2.58	35	23	0.053	0.60
Y	371.0	0.8	2.35	99	100	0.015	0.013
Zn	213.9	0.6	0.60	101	94	0.013	0.013
Zr	339.2	1.9	0.88	75	98	0.049	0.008

- (a) Values reported were obtained with a Jarrell-Ash Model 1160 ICP; performance may vary with instrument and should be independently verified.
 (b) 2.5 µg/filter corresponds to 5 µg/m³ for a 500-L air sample.
 (c) Blank levels too high to make accurate determinations.
 (d) Qualitative only because of low recovery.



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APPENDIX - MICROWAVE DIGESTION FOR LEAD IN PAINT CHIPS (AND OTHER MATRICES)

This procedure is an alternative to the procedure presented in the Sample Preparation section of this method. It provides a rapid, complete acid digestion prior to analysis by flame atomic absorption (FAA), heated graphite furnace atomic absorption (HGFAA), and inductively coupled plasma spectroscopy (ICP) [10].

Apparatus and Material [11-16]

1. Microwave apparatus requirements
 - a. The microwave unit provides programmable power with a minimum of 574 W and can be programmed to within ± 10 W of the required power.
 - b. The microwave unit cavity is corrosion resistant as well as ventilated. All electronics are protected against corrosion for safe operation.
 - c. The system requires Teflon PFA digestion vessels (120-mL capacity) capable of withstanding pressures up to 7.5 ± 0.7 atm (110 ± 10 psi) and capable of controlled pressure relief at pressures exceeding 7.5 ± 0.7 atm (110 ± 10 psi).
 - d. A rotating turntable is employed to ensure homogeneous distribution of microwave radiation within the unit. The speed of the turntable should be a minimum of 3 rpm.
 - e. A safety concern relates to the use of sealed containers without pressure relief valves in the unit. Temperature is the important variable controlling the reaction. Pressure is needed to attain elevated temperatures but must be safely contained [12].
 - f. Polymeric volumetric ware in plastic (Teflon or polyethylene), 50- or 100-mL capacity.
 - g. Disposable polypropylene filter funnel.
 - h. Analytical balance, 300-g capacity, and minimum ± 0.001 g.

Reagents

1. Nitric acid, concentrated, spectroscopy grade.
2. Reagent Water. Reagent water shall be interference free. All references to water in the method refer to reagent water that meets the ASTM Type 2 standard.

Procedure

1. Calibration of Microwave Equipment
Calibrate microwave equipment in accordance with manufacturer's instructions. If calibration instructions are not available, see EPA Method 3051 [11].
2. All digestion vessels and volumetric ware must be carefully acid washed and rinsed with reagent water. All digestion vessels should be cleaned by leaching with hot (1:1) nitric acid for a minimum of fifteen minutes, rinsed with reagent water, and dried in a clean environment.
3. Sample Digestion
 - a. Tare the Teflon PFA digestion vessel.
 - b. Weigh out 0.1 g paint chip sample to the nearest 0.001 g into the tared Teflon PFA sample vessel. With large paint chip samples, measure out a 2 cm² piece, weigh to the nearest 0.001 g, and quantitatively transfer it to the vessel.



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- c. Add 5.0 ± 0.1 mL concentrated nitric acid to the sample vessel in a fume hood. If a vigorous reaction occurs, allow the reaction to stop before capping the vessel. Cap the vessel and torque the cap to 12 ft-lb (16 N-m) according to the manufacturer's directions. The sample vessel may be connected to an overflow vessel using Teflon PFA connecting tubes. Place the vessels in the microwave carousel. Connect the overflow vessels to the center well of the unit.
- d. Place the vessels evenly distributed in the turntable of the microwave unit using groups of two, six, or 12 sample vessels. Any vessels containing 5 mL of nitric acid for reagent blank purposes are counted as sample vessels. When fewer than the recommended number of samples are to be digested, i.e., three samples plus one blank, the remaining vessels should be filled with 5 mL of nitric acid to achieve the full complement of vessels. This provides an energy balance since the microwave power absorbed is proportional to the total mass in the cavity [14]. Irradiate each group of samples to achieve a temperature of 180°C in five minutes at a pressure of 50 psi. Continue to irradiate to achieve a temperature of 180°C at 100 psi after 25 minutes. Continue digestion for five minutes. A sample digestion program for 12 samples is presented in Table 1.

Table 1

Program Variables for Paint Chips Sample Digestion with Nitric Acid

Stage	(1)	(2)	(3)
Power	90%	90%	0%
Pressure, psi	50	100	0
Run Time, min	10:00	20:00	05:00
Time @ P, min	05:00	15:00	00:00
Temperature	180°C	180°C	0°C
Fan Speed	100%	100%	100%
Number of Vessels:	12		
Liquid Volume per Vessel:	5 mL		
Sample Weight:	0.1 g		

If the analyst wishes to digest other than two, six, or 12 samples at a time, use different values of power as long as they result in the same time and temperature conditions.

- e. At the end of the microwave program, allow the vessels to cool for a minimum of five minutes before removing them from the microwave unit. If a loss of sample is detected (e.g., material in overflow collection vessel, liquid outside liner), determine the reason for the loss (e.g., loss of vessel seal integrity, use of a digestion time longer than 30 minutes, too large a sample, or improper heating conditions). Once the source of the loss has been corrected, prepare a new sample beginning at Section 2. If insufficient material is available for reanalysis, dilute remaining digestate and note that some sample loss may have occurred.
- f. Uncap and vent each vessel in a fume hood. Add 20 mL reagent water, then reseal vessels and shake to mix thoroughly. Transfer the sample to an acid-cleaned polyethylene bottle. If the digested sample contains particulates which may clog nebulizers or interfere with injection of the sample into the instrument, allow the sample to settle or filter it:



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Settling: Allow the sample to stand until the supernatant is clear (usually, overnight is sufficient). If it does not clear, filter the sample.

Filtering: The filtering apparatus must be thoroughly precleaned and rinsed with dilute nitric acid. Filter the sample through quantitative filter paper into a second acid-cleaned container.

The digestate is now ready for analysis for elements of interest using the appropriate method.

4. Calculations: Report the concentrations based on the actual weight of the original sample.