ALUMINUM and compounds, as Al 7013

Al	MW: 26.98 (Al); 101.96 ($Al_{2}0_{3}$)	CAS: 7429-90-5 (Al); 1344-28-1 ($Al_{2}0_{3}$)	RTECS: BD0330000 BD1200000

METHOD: 7013, Issue 2 **EVALUATION: PARTIAL**

Issue 1: 15 February 1984
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OSHA : no PEL
NIOSH: see table 1
ACGIH: see table 1

PROPERTIES: ductile metal; valence 3; MP 660 °C

SYNONYMS: vary depending upon the compound; alumina (Al_2O_3)

SAMPLING

SAMPLER:	FILTER (0.8-µm cellulose ester membrane)
FLOW RATE:	1 to 3 L/min
VOL-MIN:	10 L @ 5 mg/m^3
-MAX:	400 L
SHIPMENT:	routine
SAMPLE STABILITY:	stable
BLANKS:	2 to 10 field blanks per set

ACCURACY

RANGE STUDIED:	not studied
BIAS:	none identified
OVERALL PRECISION ($\hat{S}_{rT}$):	not evaluated
ACCURACY:	not evaluated

MEASUREMENT

TECHNIQUE:	ATOMIC ABSORPTION, FLAME
ANALYTE:	aluminum
ASHING:	conc. HNO_3, 6 mL; 140 °C
FINAL SOLUTION:	10% HNO_3, 10 mL 1000 µg/mL Cs
FLAME:	nitrous oxide-acetylene, reducing
WAVELENGTH:	309.3 nm
BACKGROUND CORRECTION:	none used
CALIBRATION:	Al^{3+} in 10% HNO_3
RANGE:	50 to 5000 µg per sample [1]
ESTIMATED LOD:	2 µg per sample [2]
PRECISION ($\bar{S}_r$):	0.03 [1,2]

APPLICABILITY: The working range is 0.5 to 10 mg/m^3 for a 100-L sample. This is an elemental analysis, not compound specific. Verify that the types of compounds in the samples are soluble with this ashing procedure. Aliquots of the samples can be analyzed separately for approximately four additional metals.

INTERFERENCES: Cesium at 1000 µg/mL controls ionization in the nitrous oxide-acetylene flame [3]. Iron and HCl at greater than 0.2% (w/w) decrease the sensitivity. Vanadium or H_2SO_4 require 1% (w/w) La as a releasing agent [4].

OTHER METHODS: This is Method P&CAM 173 for Al [1] in a revised format. Method 7300 (ICP-AES) is an alternate analytical method.

REAGENTS:

1. Nitric acid, conc.
2. Nitric acid, 10% (v/v). Add 100 mL conc. HNO_3 to 500 mL water; dilute to 1 L.
3. Calibration stock solution, 1000 µg Al/mL. Commercially available or dissolve 1.000 g Al wire in minimum volume of (1+1) HCl using small drop of Hg as catalyst. Dilute to 1 L with 1% (v/v) HCl.
4. Cs solution, 50 mg/mL. Dissolve 73.40 g $CsNO_3$ in 100 mL water; dilute to 1 L.
5. Nitrous oxide.
6. Acetylene.
7. Distilled or deionized water.

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 3 L/min, with flexible connecting tubing.
3. Atomic absorption spectrophotometer with a nitrous oxide-acetylene burner head and aluminum hollow cathode lamp.
4. Regulators, two-stage, for N_2O and acetylene.
5. Beakers, Phillips, 125-mL, or Griffin, 50-mL, with watchglass covers.*
6. Volumetric flasks, 10- and 100-mL.*
7. Micropipets, 5 to 500 µL.*
8. Hotplate, surface temperature 100 to 140 °C.

* Clean with conc. nitric acid and rinse thoroughly with distilled or deionized water before use.

SPECIAL PRECAUTIONS: Perform all acid digestions in a fume 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 3 L/min for a total sample size of 10 to 400 L. Do not exceed 2 mg total dust loading on the filter.

SAMPLE PREPARATION:

NOTE: Alumina (Al_2O_3) will not be dissolved by this procedure. Lithium borate fusion is necessary to dissolve alumina. The following sample preparation gave quantitative recovery for soluble aluminum compounds (see EVALUATION OF METHOD). Steps 4 through 9 of Method 7300 or other quantitative ashing techniques may be substituted, especially if several metals are to be determined on a single filter.

3. Open the cassettes and transfer the samples and blanks to separate clean beakers.
4. Add 6 mL conc. HNO_3 and cover with a watchglass. Start reagent blanks at this point.
5. Heat on hotplate (140 °C) until sample dissolves and a slightly yellow solution is produced. Add acid as needed to completely destroy organic material.
6. When the sample solution is clear, remove watchglass and rinse into the beaker with 10% HNO_3.
7. Place the beakers on a hotplate and allow to go to a small liquid volume (ca. 0.5 mL).
8. When sample is dry, rinse walls of beaker with 3 to 5 mL 10% HNO_3. Reheat for 5 min to dissolve the residue, then allow to air cool.
9. Transfer the solution quantitatively to a 10-mL volumetric flask containing 0.2 mL 50 mg/mL Cs solution. Dilute to volume with 10% HNO_3.
   NOTE: If vanadium or sulfuric acid are present, add 1% (w/w) La as a releasing agent [1,3].

CALIBRATION AND QUALITY CONTROL:

10. Add known amounts, covering the range 0 to 500 mg Al per sample, of calibration stock solution to 100-mL volumetric flasks containing 2.0 mL 50 mg/mL Cs solution and dilute to volume with

10% HNO_3.

11. Analyze the working standards together with the samples and blanks (steps 16 and 17).
12. Prepare a calibration graph of absorbance vs. solution concentration (µg/mL).
13. Aspirate a standard for every 10 samples to check instrument drift.
14. Check recoveries with at least one spiked media blank per 10 samples.
15. Use method of additions occasionally to check for interferences.

MEASUREMENT:

16. Set spectrophotometer according to manufacturer's instructions and to conditions on page 7013-1.
17. Aspirate standards and samples. Record absorbance readings.
    NOTE: If the absorbance values for the samples are above the linear range of the standards, dilute the solutions with 10% HNO_3 and an appropriate amount of the 50 mg/mL Cs solution, reanalyze, and apply the appropriate dilution factor in the calculations.

CALCULATIONS:

18. Using the measured absorbances, calculate the corresponding concentrations (µg/mL) of aluminum in the sample, C_s, and average media blank, C_b, from the calibration graph.
19. Using the solution volumes (mL) of the sample, V_s, and media blanks, V_b, calculate the concentration, C (mg/m^3), of aluminum in the volume of air sampled, V (L):

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

EVALUATION OF METHOD:

Estimated LOD was 2 µg Al per sample [2]. Only the analytical procedure was evaluated for this method.

REFERENCES:

[1] NIOSH Manual of Analytical Methods, 2nd ed., V. 5, P&CAM 173, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 79-141 (1979).

[2] User check, UBTL, NIOSH Seq. #3990-0 (unpublished, November 29, 1983).

[3] Winefordner, J. D., Ed., Spectrochemical Methods of Analysis, John Wiley & Sons (1971).

[4] Analytical Methods for Atomic Absorption Spectrophotometry, Perkin-Elmer (1976).

METHOD REVISED BY:

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TABLE 1 - ALUMINUM EXPOSURE LIMITS

	OSHA mg/m^3	NIOSH mg/m^3	ACGIH mg/m^3
Total Dust	no PEL	10	10
Respirable Fraction	no PEL	5	--
Pyro powders	no PEL	5	5
Welding fumes	no PEL	5	5
Soluble salts	no PEL	2	2

Alkyls	no PEL	2	2