

CADMIUM and compounds, as Cd

7048

Cd **MW: 112.40 (Cd);** **CAS: 7440-43-9 (Cd);** **RTECS: EU9800000 (Cd)**
 128.40 (CdO) **1306-19-0 (CdO)** **EV1930000(CdO)**

METHOD: 7048, Issue 2

EVALUATION: FULL

Issue 1: 15 February 1984

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OSHA : 0.1 mg/m³, C 0.3 (fume);
 0.2, C 0.6 (dust)

NIOSH: lowest feasible; carcinogen

ACGIH: 0.01 mg/m³ (total dust); 0.002 mg/m³ (respirable);
 carcinogen

PROPERTIES: soft metal; valence +2; BP 765 °C;
 MP 320.9 °C

SYNONYMS: Vary depending upon the compound.

SAMPLING		MEASUREMENT	
SAMPLER:	FILTER (0.8-µm cellulose ester membrane)	TECHNIQUE:	ATOMIC ABSORPTION, FLAME
FLOW RATE:	1 to 3 L/min	ANALYTE:	cadmium
VOL-MIN:	25 L @ 0.1 mg/m ³	ASHING:	conc. HNO ₃ , 6 mL; 140 °C conc. HCl, 6 mL; 400 °C
-MAX:	1500 L	FINAL SOLUTION:	0.5 N HCl; 25 mL
SHIPMENT:	routine	FLAME:	air-acetylene, oxidizing
SAMPLE STABILITY:	stable	WAVELENGTH:	228.8 nm
BLANKS:	2 to 10 field blanks per set	BACKGROUND CORRECTION:	D ₂ or H ₂ continuum
ACCURACY		CALIBRATION:	Standard solutions of Cd in 0.5 N HCl
RANGE STUDIED:	0.12 to 0.98 mg/m ³ [1] (25-L samples)	RANGE:	2.5 to 30 µg per sample
BIAS:	- 1.57%	ESTIMATED LOD:	0.05 µg per sample [2]
OVERALL PRECISION (S_{rT}):	0.06 [1]	PRECISION (S_p):	0.05 @ 3 to 23 µg per sample [1,3,4]
ACCURACY:	± 13.23%		

APPLICABILITY: The working range is 0.1 to 2 mg/m³ for a 25-L air sample, and 0.01 to 0.2 mg/m³ for a 250-L air sample. This is an elemental analysis and it will not distinguish Cd fume from Cd dust. Aliquots of the ashed samples can be analyzed separately for many additional metals.

INTERFERENCES: Background correction is required to control molecular or flame absorption. Iron does not interfere at 20 parts Fe to 1 part Cd [1].

OTHER METHODS: This method combines and replaces P&CAM 173 [5], S312 [4], and S313 [6]. A similar method appears in the criteria document [7]. Method 7300 (ICP-AES) is an alternate, multielement measurement method with approximately the same sensitivity. Aliquots of the ashed samples can be analyzed by graphite furnace atomic absorption with greatly increased sensitivity.

REAGENTS:

1. Nitric acid, conc., ultrapure.
2. Hydrochloric acid, conc., ultrapure.
3. Hydrochloric acid, 0.5 N. Add 41.5 mL conc. HCl to water; dilute to 1 L.
4. Calibration stock solution, 100 µg Cd/mL.* Commercially available or dissolve 0.100 g Cd metal in minimum volume of (1:1) HCl. Dilute to 1 L with 0.5 N HCl.
5. Distilled-deionized water.
6. Air, filtered.
7. Acetylene.

* 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 3 L/min, with flexible connecting tubing.
3. Atomic absorption spectrophotometer with an air-acetylene burner head, cadmium hollow cathode lamp, and background correction.
4. Regulators, 2-stage, for air and acetylene.
5. Beakers, Phillips, 125-mL, or Griffin, 50-mL, with watchglass covers.*
6. Volumetric flasks, 25- and 100-mL.*
7. Micropipets, 5 to 300 µL.*
8. Hotplate, surface temperature 400 °C.

* Clean with conc. nitric acid and rinse thoroughly before use.

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

Cadmium compounds are very toxic and should be considered carcinogens [3,7]. Handle with extra care.

SAMPLING:

1. Calibrate each personal sampling pump with a representative sampler in line.
2. Sample at an accurately known flow rate between 2 and 3 L/min for 15 min (30 to 45 L) for ceiling measurements, or at 1 to 3 L/min for a total sample size of 25 to 1500 L for TWA measurements. Do not exceed a filter loading of ca. 2 mg total dust.

SAMPLE PREPARATION:

NOTE: The following sample preparation gave quantitative recovery (see EVALUATION OF METHOD). Steps 4 through 9 of Method 7300 or other quantitative ashing techniques may be substituted.

3. Open the cassette filter holders and transfer the samples and blanks to separate clean beakers.
4. Add 2 mL conc. HNO₃, cover with a watchglass, and heat on hotplate (140 °C) until the volume is reduced to ca. 0.5 mL. Start reagent blanks at this point.
5. Repeat 2 more times using 2 mL conc. HNO₃ each time.
6. Add 2 mL conc. HCl, cover with a watchglass, and heat on hotplate (400 °C) until the volume is reduced to ca. 0.5 mL.
7. Repeat 2 more times using 2 mL conc. HCl. Do not allow the solution to go to dryness at any point.
8. Cool solution and add 10 mL distilled water.
9. Transfer the solution quantitatively to a 25-mL volumetric flask.
10. Dilute to volume with distilled water.

CALIBRATION AND QUALITY CONTROL:

11. Calibrate daily with at least six working standards. Add known amounts, covering the range 0 to 30 µg Cd per sample, of calibration stock solution to 100-mL volumetric flasks and dilute to volume with 0.5 N HCl.
12. Analyze the working standards together with the blanks and samples (steps 17 and 18).
13. Prepare a calibration graph of absorbance vs. solution concentration (µg/mL).
14. Aspirate a standard after every 10 samples to check for instrument drift.
15. Check recoveries with at least 2 spiked media blanks per 10 samples.
16. Use method of additions occasionally to check for interferences.

MEASUREMENT:

17. Set spectrophotometer according to manufacturer's recommendations and to conditions on page 7048-1.
18. 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 0.5 N HCl, reanalyze, and use the appropriate dilution factor in calculations.

CALCULATIONS:

19. Using the measured absorbances, calculate the corresponding concentrations (µg/mL) of cadmium in the sample, C_s , and average media blank, C_b , from the calibration graph.
20. Using the solution volumes (mL) of the sample, V_s , and media blanks, V_b , calculate the concentration, C (mg/m³), of cadmium 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:

Method S312 was issued on June 12, 1976 [4], and validated over the range 0.12 to 0.98 mg/m³ for a 25-L sample of CdO dust [1]. Method S313 was issued on November 26, 1976 [6], and validated over the range 0.12 to 0.57 mg/m³ for a 25-L sample of Cd fume, and over the range 0.04 to 0.18 mg/m³ for a 140-L sample of Cd fume [1].

REFERENCES:

- [1] Documentation of the NIOSH Validation Tests, S312 and S313, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 77-185 (1977).
- [2] User check, UBTL, NIOSH Seq. #3990-M (unpublished, November 29, 1983).
- [3] Current Intelligence Bulletin 42: Cadmium. U.S. Department of Health and Human Services, Publ. (NIOSH) 84-116 (Sept. 27, 1984).
- [4] NIOSH Manual of Analytical Methods, V. 3, Method S312, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 77-157-C (1977).
- [5] Ibid., 2nd ed., V. 5, P&CAM 173, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 79-141 (1979).

[6] Ibid, Method S313.

[7] Criteria for a Recommended Standard...Occupational Exposure to Cadmium, Appendix II, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 76-192 (1976).

METHOD REVISED BY:

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