

COBALT and compounds, as Co

7027

Co MW: 58.93 (Co) CAS: 7440-48-4 (Co) RTECS: GF8750000

METHOD: 7027, Issue 2

EVALUATION: FULL

Issue 1: 15 February 1984

Issue 2: 15 August 1994

OSHA : 0.1 mg/m³
NIOSH: 0.05 mg/m³
ACGIH: 0.05 mg/m³ (fume, dust)

PROPERTIES: magnetic, hard metal; valence +2, +3;
 MP 1495 °C; VP not significant

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:	cobalt
VOL-MIN:	30 L @ 0.1 mg/m ³	ASHING:	aqua regia, 3 mL, 140 °C; conc. HNO ₃ , 6 mL, 140 °C; room temperature, 30 min; 140 °C to near dryness
-MAX:	1500 L	FINAL SOLUTION:	5% HNO ₃ , 10 mL
SHIPMENT:	routine	FLAME:	air-acetylene, oxidizing
SAMPLE STABILITY:	stable	WAVELENGTH:	240.7 nm
FIELD BLANKS:	2 to 10 field blanks per set	BACKGROUND CORRECTION:	D2 or H2 lamp
ACCURACY		CALIBRATION:	Co ²⁺ in 5% HNO ₃
RANGE STUDIED:	0.03 to 0.26 mg/m ³ [1] (270-L samples)	RANGE:	3 to 90 µg per sample [2]
BIAS:	5.6%	ESTIMATED LOD:	0.6 µg per sample [3]
OVERALL PRECISION (\hat{S}_{rr}):	0.070 [1]	PRECISION (\hat{S}_{r}):	0.03 [1]; 0.02 [3]
ACCURACY:	± 18.0%		

APPLICABILITY: The working range is 0.01 to 0.3 mg/m³ for a 300-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: The use of D2 or H2 continuum background correction is required to control molecular or flame absorption. There are no known spectral line interferences.

OTHER METHODS: This method combines and replaces P&CAM 173 [4] and S203 [2] for cobalt. Method 7300 (Elements by ICP-AES) is an alternate analytical technique.

REAGENTS:

1. Nitric acid (HNO₃, conc.
2. Hydrochloric acid (HCl), conc.
3. Aqua regia: mix 3 volumes conc. HCl with 1 volume conc. HNO₃.
4. Nitric acid, 5% (w/v). Add 50 mL conc. HNO₃ to water; dilute to 1 L.
5. Calibration stock solution, 1000 µg/mL Co. Commercially available or dissolve 1.000 g Co metal in minimum volume of aqua regia. Dilute to 1 L with 5% HNO₃.
6. Distilled or deionized water.
7. Air, filtered.
8. Acetylene.

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 and cobalt hollow cathode lamp.
4. Regulators, two-stage, for air 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 60-µL.*
8. Hotplate, surface temperature 140°C.

* Clean with conc. HNO₃ 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 filter in line.
2. Sample at an accurately known flow rate between 1 and 3 L/min for a sample size of 30 to 1500 L. Do not exceed a filter loading of approximately 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 necessary for some samples, especially if several metals are to be determined on a single filter.

3. Open the cassette filter holders and transfer the samples and blanks to clean beakers.
4. Add 3 mL aqua regia, cover with a watchglass, let stand at room temperature 30 min. Start reagent blanks at this point.
5. Heat on hotplate (140°C) until most of the acid has evaporated (ca. 0.5 mL remains).
6. Repeat 2 more times using 3 mL conc. HNO₃ each time. Leave about 1 mL solution after the last digestion.
7. Cool each beaker. Remove watchglass and rinse into the beaker with 5% HNO₃.
8. Dissolve the residues in 2 to 3 mL 5% HNO₃.
9. Transfer the solution quantitatively to a 10-mL volumetric flask.
10. Dilute to volume with 5% HNO₃.

CALIBRATION AND QUALITY CONTROL:

11. Calibrate daily with at least six working standards. Add known amounts, covering the expected range of samples (0 to 60 µg Co per sample), of calibration stock solution to 10-mL volumetric flasks and dilute to volume with 5% HNO₃.
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 ($\mu\text{g/mL}$).
14. Aspirate a standard for every 10 samples to check instrument drift.
15. Check recoveries with at least one spiked media blank per 10 samples.
16. Use method of standard additions occasionally to check for interferences.

MEASUREMENT:

17. Set spectrophotometer according to the manufacturer's recommendations and to the conditions on page 7027-1.
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 5% HNO₃, reanalyze, and apply the appropriate dilution factor in the calculations.

CALCULATIONS:

19. Using the measured absorbances, calculate the corresponding concentrations ($\mu\text{g/mL}$) of cobalt 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/m^3), of cobalt in the volume of air sampled, V (L):

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

NOTE: $\mu\text{g/mL} \equiv \text{mg/m}^3$

EVALUATION OF METHOD:

Method S203 [2] was issued on February 18, 1977, and validated over the range 0.03 to 0.22 mg/m^3 Co fume and 0.04 to 0.26 mg/m^3 Co dust using spiked filters and generated atmospheres of cobalt dust and fume (Co₂O₃) [1]. Precision and accuracy data are given on page 7027-1. A separate check on this method showed 98% recovery from filters spiked with 12 or 96 μg soluble Co [3].

REFERENCES:

- [1] Documentation of the NIOSH Validation Tests, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 77-185 (1977).
- [2] NIOSH Manual of Analytical Methods, 2nd ed., V. 4, Method S203, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 78-175 (1978).
- [3] User check, UBTL, NIOSH Seq. #3990-N (unpublished, November 29, 1983).
- [4] 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).

METHOD REVISED BY:

Mark Millson, NIOSH/DPSE and R. DeLon Hull, Ph.D., NIOSH/DSHEFS; S203 originally validated under NIOSH Contract CDC-99-74-45.