BARIUM, soluble compounds 7056

Ba MW: 137.34 CAS: 7440-39-3 RTECS: CQ8370000

METHOD: 7056, Issue 2	**EVALUATION: FULL**	**Issue 1: 15 August 1987** **Issue 2: 15 August 1994**

OSHA : 0.5 mg/m^3
NIOSH: 0.5 mg/m^3
ACGIH: 0.5 mg/m^3

PROPERTIES: solubility @ 100 °C:

$BaCO_3$:	0.006 g/100 g H_2O;
$BaCl_2$:	59 g/100 g H_2O;
$Ba(NO_3)_2$:	34 g/100 g H_2O;
BaO:	91 g/100 g H_2O

SYNONYMS: vary depending upon compound

SAMPLING

SAMPLER:	FILTER (0.8-µm cellulose ester membrane)
FLOW RATE:	1 to 4 L/min
VOL-MIN:	50 L @ 0.5 mg/m^3
-MAX:	2000 L
SHIPMENT:	routine
SAMPLE STABILITY:	stable
FIELD BLANKS:	2 to 10 field blanks per set

ACCURACY

RANGE STUDIED:	0.28 to 1.08 mg/m^3 [1] (168-L samples)
BIAS:	- 0.55%
OVERALL PRECISION ($\hat{S}_{rT}$):	0.054 [1]
ACCURACY:	± 10.81%

MEASUREMENT

TECHNIQUE:	ATOMIC ABSORPTION, FLAME
ANALYTE:	barium ion (Ba^{2+})
EXTRACTION:	hot water leach, 10 mL, 10 min, twice; conc. HCl, 3 drops; evaporate to dryness
FINAL SOLUTION:	5% HCl/1.1 mg/mL Na^+, 5 mL
FLAME:	nitrous oxide-acetylene reducing
WAVELENGTH:	553.6 nm
CALIBRATION:	standard solutions of Ba^{2+} in 5% HCl/1.1 mg/mL Na^+
RANGE:	25 to 200 µg per sample [1]
ESTIMATED LOD:	2 µg per sample [1]
PRECISION ($\bar{S}_r$):	0.025 @ 43 to 180 µg per sample [1]

APPLICABILITY: The working range is 0.13 to 10 mg/m^3 for a 200-L air sample. This method determines Ba^{2+} in water-soluble barium compounds. Insoluble barium compounds (e.g., $BaSO_4$) require an ashing procedure.

INTERFERENCES: Ionization of barium in the flame is controlled by addition of sodium chloride to samples and standards. Calcium, at >0.1%, gives a positive interference unless background correction is used.

OTHER METHODS: This revises Method S198 [2].

REAGENTS:

1. Water, distilled or deionized.
2. Nitric acid (HNO_3), conc.*
3. Hydrochloric acid (HCl), conc.*
4. Sodium chloride (NaCl).
5. 5% HCl (v/v)/1.1 mg/mL Na^+: Dilute 5 mL conc. HCl and 0.28 g NaCl to 100 mL with deionized water.
6. Calibration stock solution, 1000 µg Ba/mL. Commercially available or dissolve 1.437 g $BaCO_3$ in minimum volume of (1+1) HCl and dilute to 1 L with 1% (v/v) HCl.
7. Nitrous oxide, 98%.
8. Acetylene, 99.6%.

* 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. Atomic absorption spectrophotometer with nitrous oxide-acetylene burner head and barium hollow cathode lamp.
   NOTE: Background correction (e.g., D_2 or H_2 lamp) needed for samples with >0.1% (w/v) Ca^{2+}.
4. Regulators, two-stage, for nitrous oxide and acetylene.
5. Beakers, Phillips, 125-mL, or Griffin, 50-mL, with watchglass covers.*
6. Volumetric flasks, 10- and 100-mL.*
7. Pipets, 4- to 400-µL, and 5-mL.
8. Hotplate, surface temperature 140 °C.
9. Forceps or tweezers, plastic-tipped.
10. Centrifuge, and centrifuge tubes, 50-mL.

* Clean with conc. HNO_3 and rinse thoroughly with distilled or deionized water before use.

SPECIAL PRECAUTIONS: Nitric and hydrochloric acids are corrosive liquids. Wear protective clothing and work in a fume head.

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 sample size of 50 to 2000 L. Do not exceed a filter loading of ca. 2 mg total dust.

SAMPLE PREPARATION:

3. Open cassette filter holders and transfer samples and blanks to clean beakers.
4. Add 10 mL boiling distilled water. Let stand 10 min with occasional swirling. Decant extract to a centrifuge tube.
5. Wash filter and beaker twice with ca. 2 mL hot distilled water and add to centrifuge tube.
6. Repeat extraction and washing (steps 4 and 5), adding the solutions to the centrifuge tube.
7. Remove filter with forceps and rinse with stream of hot distilled water into centrifuge tube.
8. Rinse original beaker three times with ca. 2 mL hot distilled water and add to centrifuge tube. Centrifuge and decant the solution to a second beaker.
9. Add three drops conc. HCl to sample and evaporate until ca. 0.5 mL liquid remains.
10. Cool each beaker. Pipet 5.0 mL 5% HCl/1.1 mg/mL Na^+ solution into each beaker. Swirl to dissolve residue.

CALIBRATION AND QUALITY CONTROL:

11. Add known amounts of calibration stock solution to 10-mL volumetric flasks and dilute to volume with 5% HCl/1.1 mg/mL Na^+ solution to produce Ba^{2+} concentrations in the range 0.4 to 40 µg/mL (0.002 to 0.2 mg per sample). Prepare fresh daily.
12. Analyze working standards with the blanks and samples (steps 17 and 18).
13. Prepare calibration graph (absorbance vs. solution concentration, µg/mL).
14. Aspirate a standard for every ten samples to check instrument drift.
15. Check recoveries with at least one spiked media blank per ten samples.
16. Use method of standard additions occasionally to check for interferences.

MEASUREMENT:

17. Set spectrophotometer according to manufacturer's recommendations and to conditions on page 7056-1.
18. Aspirate standards and samples. Record absorbance readings.

    NOTE: If absorbance values for samples are above the linear range of the standards, dilute with 5% HCl/1.1 mg/mL Na^+ solution, reanalyze, and apply the appropriate dilution factor in calculations.

CALCULATIONS:

19. Using the measured absorbances, calculate the corresponding concentrations (µg/mL) of barium 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 of barium, C (mg/m^3), in the volume of air sampled, V (L):

$$C = \frac{(C_sV_s - C_bV_b)}{V}, \text{ mg/m}^3.$$

EVALUATION OF METHOD:

Method S198 was validated using atomized aqueous solutions of barium chloride to generate atmospheres at approximately 0.3 to 1.1 mg/m^3 [1]. Samples taken at 1.4 L/min showed 100% collection efficiency. Recovery of barium chloride standards spiked on filters was 102% with $\bar{S}_r = 1.4\%$ in the range 0.043 to 0.18 mg barium per sample.

REFERENCES:

[1] Documentation of the NIOSH Validation Tests, S198, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 77-185 (1977), available as Stock No. PB 274-248 from NTIS, Springfield, VA 22161.

[2] NIOSH Manual of Analytical Methods, 2nd ed., Vol. 3, S198, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 77-157-C (1977).

METHOD REVISED BY:

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