

**ARSENIC and compounds, as As
(except AsH₃ and As₂O₃)**

7900

As MW: 74.92 CAS: 7440-38-2 RTECS: CG525000

METHOD: 7900, Issue 2

EVALUATION: PARTIAL

Issue 1: 15 February 1984

Issue 2: 15 August 1994

OSHA : 0.01 mg/m³

NIOSH: C 0.002 mg/m³/15 min; carcinogen

ACGIH: 0.01 mg/m³; carcinogen

PROPERTIES: soft, reactive metalloid; MP 848 °C;
valence ± 3, 5 in salts

SYNONYMS: vary depending upon the compound

SAMPLING		MEASUREMENT	
SAMPLER:	FILTER (0.8-µm cellulose ester membrane)	TECHNIQUE:	ATOMIC ABSORPTION, FLAME ARSINE GENERATION
FLOW RATE:	1 to 3 L/min	ANALYTE:	arsenic
VOL-MIN:	30 L @ 0.002 mg/m ³	ASHING:	conc. HNO ₃ , 3 mL; conc. H ₂ SO ₄ , 1 mL; conc. HClO ₄ , 1 mL; 140 °C
-MAX:	1000 L	FINAL SOLUTION:	4% H ₂ SO ₄ , 25 mL
SHIPMENT:	routine	FLAME:	hydrogen-argon
SAMPLE STABILITY:	stable if refrigerated	WAVELENGTH:	193.7 nm
BLANKS:	2 to 10 field blanks per set	BACKGROUND CORRECTION:	D ₂ or H ₂ continuum
ACCURACY		CALIBRATION:	As in 4% H ₂ SO ₄
RANGE STUDIED:	not studied	RANGE:	0.05 to 2.0 µg per sample [1]
BIAS:	see APPLICABILITY	ESTIMATED LOD:	0.02 µg per sample [1]
OVERALL PRECISION ($\hat{S}_{rt}$):	not evaluated	PRECISION ($\hat{S}_r$):	0.11 [1]
ACCURACY:	not determined		

APPLICABILITY: The working range is 0.00025 to 0.01 mg/m³ for a 200-L air sample and 0.002 to 0.07 mg/m³ for a 30-L air sample. **This method collects particulate arsenic only; if arsenic trioxide vapor is present, use the sampler in Method 7901.** This is an elemental analysis, not compound specific. Volatile organic arsenic compounds, As₂O₃ vapor, and arsine are not collected efficiently by this sampling method.

INTERFERENCES: Background absorption is overcome by the use of D₂ or H₂ continuum.

OTHER METHODS: This revises P&CAM 139 [1]; a similar method appears in the criteria document [2]. Method 7901 uses a sampler designed to collect As₂O₃ vapor and an alternate measurement technique (graphite furnace-AAS). Method 7300 (ICP-AES) also gives an alternate measurement technique.

REAGENTS:

1. Nitric acid, conc.
2. Hydrochloric acid, conc.
3. Sulfuric acid, conc.
4. Perchloric acid, conc.*
5. Calibration stock solution, 1000 mg/mL.*
Commercially available or dissolve 1.320 g primary standard As_2O_3 in 25 mL 20% (w/v) KOH. Neutralize with 20% (v/v) HNO_3 to a phenolphthalein endpoint. Add 10 mL conc. HNO_3 and dilute to 1 L with distilled or deionized water.
6. Ashing acid, 3 volumes HNO_3 , 1 volume H_2SO_4 , and 1 volume HClO_4 .
7. Hydrogen.
8. Argon.
9. Distilled or deionized water.
10. Sodium borohydride, pellets.
11. Air, compressed.

* See SPECIAL PRECAUTIONS.

EQUIPMENT:

1. Sampler: cellulose ester 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 appropriate hydrogen burner head or quartz tube furnace, and arsenic hollow cathode lamp or EDL and arsine generation system.
4. Regulators, two-stage, for air, hydrogen and argon.
5. Beakers, Phillips, 125-mL, or Griffin, 50-mL, with watchglass covers.*
6. Volumetric flasks, 25- and 100-mL.*
7. Pipets, volumetric, as needed.*
8. Hotplate, surface temperature 140 °C.

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

SPECIAL PRECAUTIONS: Arsenic is a recognized carcinogen. Handle appropriately [2]. Perform all perchloric acid digestions in a perchloric acid 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 30 to 1000 L. Do not exceed ca. 2 mg total dust loading on the filter.

SAMPLE PREPARATION:

3. Open the cassette filter holders and transfer the samples and blanks to clean beakers.
NOTE: Analyze the backup pad separately if qualitative indication of As_2O_3 vapor is desired.
Use Method 7901 if quantitative collection of As_2O_3 vapor is desired.
4. Add 5 mL ashing acid and cover with a watchglass.
5. Heat on hotplate (140 °C) until the solution is colorless.
6. Add 1 mL conc. HNO_3 and/or 70% HClO_4 drop by drop as needed to complete the ashing.
7. Remove the watchglass.
8. Heat on 140 °C hotplate until dense SO_3 fumes appear.
9. Allow the mixture to cool.
10. Transfer the solution quantitatively to a 25-mL volumetric flask.
11. Dilute to volume with distilled or deionized water.

CALIBRATION AND QUALITY CONTROL:

12. Prepare working standards. Add known amounts, covering the range 0.2 to 8 μg As/100 mL (0.05 to 2 μg As per sample), of 1000 $\mu\text{g}/\text{mL}$ As calibration stock solution to 100-mL volumetric flasks containing 4 mL conc. H_2SO_4 and dilute to volume with distilled or deionized water.

13. Analyze working standards together with the blanks and samples (steps 18 through 25).
14. Prepare calibration graph (absorbance vs. solution concentration, $\mu\text{g/mL}$).
15. Analyze a standard for every 10 samples.
16. Check analytical recoveries with at least one spiked media blank per 10 samples.
17. Use method of standard additions occasionally to check for interferences.

ANALYTICAL PROCEDURE:

18. Set spectrophotometer according to manufacturer's recommendations and to conditions on page 7900-1.
 19. Set up arsine generator per manufacturer's instructions.
 20. Pipet 5 mL aliquot of the 25-mL sample into the arsine generation flask.
 21. Add 25 mL distilled or deionized water, 3 mL conc. HCl, and mix well.
 22. Connect the flask to the generation system.
 23. Introduce a single sodium borohydride pellet or sodium borohydride solution to the sample solution.
 24. Allow the gases to flush into the flame of the atomic absorption instrument.
 25. Record the absorbance readings.
- NOTE: If the absorbance values of the samples are above the linear range of the standards, dilute the solutions, or use a smaller aliquot, reanalyze, and use the appropriate dilution factor in calculations.

CALCULATIONS:

26. Using the measured absorbances, calculate the corresponding solution concentrations ($\mu\text{g/mL}$) of As in the sample, C_s , and average media blank, C_b , from the calibration graph.
27. Using the solution volumes (mL) of the sample, V_s , and media blanks, V_b , calculate the concentration, C (mg/m^3), of As 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:

This method was evaluated in July, 1976, over the range 0.02 to 3 mg per sample by laboratory testing with spiked filters. Precision and accuracy data are given on page 7900-1 [1].

REFERENCES:

- [1] NIOSH Manual of Analytical Methods, 2nd. ed., V. 1, P&CAM 139, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 77-157-A (1977).
- [2] Criteria for a Recommended Standard...Occupational Exposure to Inorganic Arsenic, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 75-149 (1975).

METHOD WRITTEN BY:

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