SbH_3 MW: 124.77 CAS: 7803-52-3 RTECS: WJ0700000

METHOD: 6008, Issue 2 **EVALUATION: FULL** **Issue 1: 15 August 1987**
Issue 2: 15 August 1994

OSHA : 0.1 ppm
NIOSH: 0.1 ppm/10 h
ACGIH: 0.1 ppm
(1 ppm = 5.10 mg/m^3 @ NTP)

PROPERTIES: gas; BP -17 °C; MP -88 °C; VP >1 atm

SYNONYMS: antimony trihydride

SAMPLING

SAMPLER: SOLID SORBENT TUBE ($HgCl_2$-coated silica gel, 1 g/0.5 g)

FLOW RATE: 0.01 to 0.2 L/min

VOL-MIN: 4 L
-MAX: 50 L

SHIPMENT: routine

SAMPLE STABILITY: at least 7 days @ 25 °C [1]

BLANKS: 2 to 10 field blanks per set

ACCURACY

RANGE STUDIED: 0.12 to 1 mg/m^3 [1] (20-L samples)

BIAS: -1.4%

OVERALL PRECISION ($\hat{S}_{rT}$): 0.087 [1]

ACCURACY: ± 17.0%

MEASUREMENT

TECHNIQUE: VISIBLE SPECTROPHOTOMETRY

ANALYTE: antimony

EXTRACTION: 15 mL conc. HCl; 30 min

WAVELENGTH: 552 NM

CALIBRATION: standard solutions of Sb(III) in conc. HCl

RANGE: 2 to 20 µg Sb per sample [1]

ESTIMATED LOD: 0.4 µg SbH_3 per sample [1]

PRECISION ($\bar{S}_r$): not defined

APPLICABILITY: The working range is 0.1 to 3.5 mg/m^3 (0.02 to 0.7 ppm) for a 20-L air sample.

INTERFERENCES: Colorimetric determination of Sb is subject to interference by elements which form a Rhodamine B complex extractable into isopropyl ether. Am(III) (>250 µg); Au (>1000 µg); Fe(III) (>30,000 µg); Tl(I) (>1000 µg); and Sn(II) (>1000 µg) interfere [2]. If the possibility of an interference exists, an alternate measurement procedure should be used (e.g., IC P or AAS).

OTHER METHODS: This revises Method S243 [3].

REAGENTS:

1. Water, distilled or deionized.
2. Hydrochloric acid (HCl), conc.*
3. Antimony stock solution, 1000 µg/mL Sb(III).
4. Ceric sulfate, $Ce(SO_4)_2$, solid.
5. Isopropyl ether.
6. Rhodamine B stock solution, 0.2% in water. Dissolve 0.5 g Rhodamine B in 250 mL water. Prepare in duplicate.
7. Working Rhodamine B solution, 0.01% in 0.5 M HCl. Dilute 25 mL Rhodamine B stock solution and 24 mL conc. HCl to 500 mL. Prepare fresh daily.

* See SPECIAL PRECAUTIONS.

EQUIPMENT:

1. Sampler: glass tube, 7 cm long, 8-mm OD, 6-mm ID, with plastic caps, containing two sections of $HgCl_2$-coated silica gel (front = 1.0 g; back = 0.5 g) separated and contained by silylated glass wool. Pressure drop across the tube at 1 L/min airflow must be less than 3.4 kPa. Tubes are commercially available.
2. Personal sampling pump, 0.01 to 0.2 L/min, with flexible connecting tubing.
3. Visible spectrophotometer capable of measuring absorbance at 552 nm, with two matched 1-cm silica absorbance cells with tight-fitting PTFE caps.*
4. Separatory funnels, 125-mL.*
5. Beakers, Griffin, 50-mL.
6. Volumetric flasks, 25-, 50-, 250-, and 500-mL.
7. Pipets, 4-, 15-, and 20-mL.*
8. Graduated cylinders, 10- and 25-mL.*
9. Centrifuge, with centrifuge tubes, 15-mL, with plastic caps.*

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

SPECIAL PRECAUTIONS: Perform all concentrated acid handling in a fume hood.

SAMPLING:

1. Calibrate each personal sampling pump with a representative sampler in line.
2. Remove sampler caps immediately before sampling. Attach sampler to personal sampling pump with flexible tubing.
   NOTE: Use a cellulose ester membrane prefilter if particulate antimony compounds may be present.
3. Sample at an accurately known flow rate between 0.01 and 0.2 L/min for a total sample size of 4 to 50 L.
4. Cap the sampler and pack securely for shipment.

SAMPLE PREPARATION:

5. Place front and back sorbent sections of the sampler tube in separate 50-mL beakers. Discard the glass wool.
6. Add 15.0 mL conc. HCl to each beaker.
7. Mildly agitate (swirl) the beaker contents every 10 min for a total desorption time of 30 min. Pour the solution into a 25-mL volumetric flask.
8. Add 4.0 mL conc. HCl to the sorbent in the beaker. Swirl and decant into the volumetric flask from step 7.
9. Repeat step 8. Dilute to volume with conc. HCl and swirl to mix.
   NOTE: Perform steps 10 through 15 within a 15-min period.
10. Pipet 15.0 mL of the sample extract into a separatory funnel.

11. Add 15 ± 5 mg ceric sulfate, swirl to dissolve and let stand unstoppered 60 s.
12. Add 15.0 mL isopropyl ether to the funnel, stopper, and shake 30 s, releasing pressure only at the beginning and end of that period.
13. Add 7.0 mL water, Shake 60 s. Wait 60 s to allow for separation. Remove and discard the aqueous layer. Be careful to remove all of the aqueous layer.
14. Add 20.0 mL working Rhodamine B solution, shake 60 s, releasing pressure only at the beginning and end of that period. Allow 60 s for the phases to separate. Remove and discard aqueous (lower) layer. Drain ca. 10 mL of the remaining solution into a 15-mL centrifuge tube and cap tightly.
15. Centrifuge 120 s at 2000 rpm. Transfer ca. 3 mL of the supernatant liquid to an absorption cell and cap. Proceed immediately to step 18.

CALIBRATION AND QUALITY CONTROL:

16. Calibrate daily with at least six working standards over the range 2 to 20 µg antimony per sample.
    a. Add known amounts of antimony stock solution and conc. HCl to cover the antimony concentration of 2 to 20 µg Sb per 15 mL and a reagent blank.
    b. Proceed as in steps 10 through 15 and 18 and 19.
    c. Prepare a calibration graph (absorbance vs. µg antimony/15 mL).
17. Analyze three quality control spikes to ensure that the calibration graph is in control.

MEASUREMENT:

18. Set the spectrophotometer to manufacturer's recommendations and to conditions given on page 6008-1.
19. Measure the absorbance using isopropyl ether as a blank.

CALCULATIONS:

20. Determine the mass, µg, of stibine found in the sample front (W_f) and back (W_b) sorbent sections, and in the average blank front (B_f) and back (B_b) sorbent sections by multiplying the mass of antimony/15 mL found for each of these sections by 1.708, a combination conversion and dilution factor (MW SbH_3/MW Sb) · (25 mL/15 mL).
    NOTE: If $W_b > W_f/10$, report breakthrough and possible sample loss.
21. Calculate concentration, C, of stibine in the air volume sampled, V (L):

$$C = \frac{(W_f + W_b - B_f - B_b)\ 1.708}{V}, \text{ mg/m}^3.$$

EVALUATION OF METHOD:

Method S243 was evaluated over the range 0.119 to 1.01 mg/m^3 using 20-L air samples [1]. Recovery averaged 98.6% in the range 0.12 to 1.0 µg SbH_3 per sample. The capacity of the sampling tubes (at a flow rate of 0.22 L/min, a SbH_3 concentration of 1.326 mg/m^3, and a relative humidity of 85%) was shown to be greater than 70 µg (as SbH_3). Previously, activated charcoal tubes were tried as a collection medium for stibine but were rejected because of poor collection efficiency [4].

REFERENCES:

[1] S243, Backup Data Report for Stibine, prepared under NIOSH Contract No. 210-76-123 (March 18, 1977), available as "Ten NIOSH Analytical Methods, Set 2," Order #PB271-464 from NTIS, Springfield, VA 22161.
[2] Ward, F.N. and H.W. Lakin. Anal. Chem., 26, 1168 (1954).
[3] NIOSH Manual of Analytical Methods, 2nd ed., Vol. 4, S243, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 78-175 (1978).
[4] Failure Report S243, Stibine, prepared under NIOSH Contract CDC-99-74-45 (unpublished, 1977).

METHOD REVISED BY:

R. DeLon Hull, Ph.D., NIOSH/DBBS.