Antimony, atomic absorption spectrometric, hydride

Parameters and Codes:

Antimony, dissolved, I-1055-85 (µg/L as Sb): 01095
Antimony, total, I-3055-85 (µg/L as Sb): 01097
Antimony, suspended total, I-7055-85 (µg/L as Sb): 01096
Antimony, total-in-bottom material, dry wt, I-5055-85 (µg/g as Sb): 01098

1. Application

1.1 This method may be used to analyze water and water-suspended sediment containing at least 1 µg/L of antimony. Samples containing more than 15 µg/L need to be diluted.

1.2 Suspended total antimony is calculated by subtracting dissolved antimony from total antimony.

1.3 This method may be used to analyze bottom material containing at least 1 µg/g of antimony. Ordinarily, a 100-mg sample of prepared bottom material (method P-0520) is taken for analysis. However, if the sample contains more than 15 µg/g of antimony, a smaller sample needs to be used.

1.4 Total antimony in water-suspended sediment may be determined after each sample has been thoroughly mixed by vigorous shaking and a suitable sample portion has been rapidly withdrawn from the mixture.

2. Summary of method

Organic antimony-containing compounds are decomposed by adding sulfuric and nitric acids and by repeatedly evaporating the sample to fumes of sulfur trioxide. The antimony so liberated, together with inorganic antimony originally present, is subsequently reacted with potassium iodide and stannous chloride, and finally with sodium borohydride to form stibine. The stibine is removed from solution by aeration and swept by a flow of nitrogen into a hydrogen diffusion flame, where it is determined by atomic absorption at 217.6 nm.

3. Interferences

3.1 Since the stibine is freed from the original sample matrix, interferences in the flame are minimized.

3.2 Selenium and arsenic, which also form gaseous hydrides, do not interfere at concentrations of 100 µg/L. Greater concentrations were not tested.

4. Apparatus

4.1 *Atomic absorption spectrometer* and recorder.

4.2 Refer to the manufacturer's manual to optimize instrument for the following:

Wavelength -------------- 217.6 nn
Source (electrodeless
discharge lamp) -------- Antimony
Burner -------------------- Three-slot
Fuel ----------------------- Hydrogen
Diluent ------------------- Nitrogen
Carrier ------------------- Nitrogen

4.3 *Stibine vapor analyzer* (fig. 1) consisting of ________

4.3.1 *Beaker*, Berzelius, 200-mL capacity.

4.3.2 *Gas dispersion tube*, coarse frit (Scientific Glass Apparatus Co. No. JG-8500 has been found satisfactory).

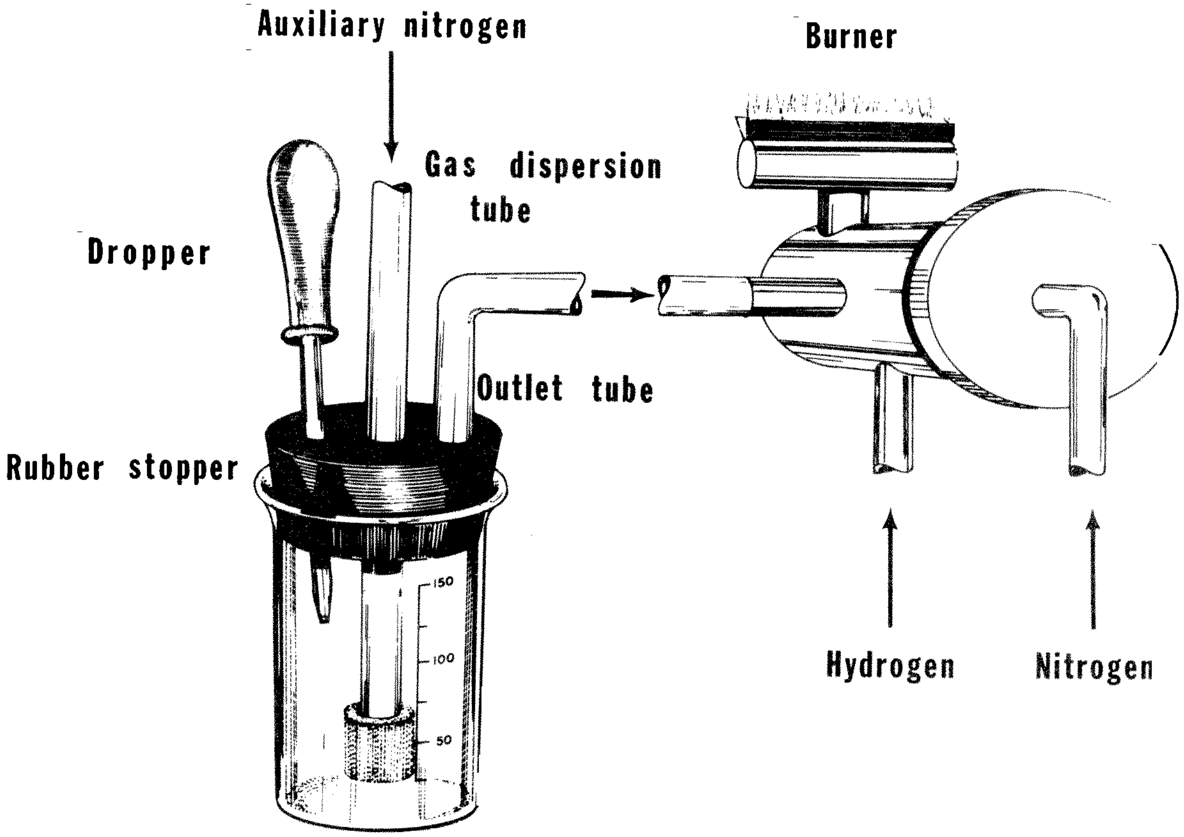
4.3.3 *Medicine dropper*, 2-mL capacity, minimum, or automatic pipettor, 5-mL capacity.

5. Reagents

5.1 *Antimony standard solution I*, 1.00 mL= 100 µg Sb: Dissolve 0.100 g Sb metal in a minimum amount of aqua regia. Add demineralized water to increase rate of dissolution and dilute to 1000 mL with demineralized water.

5.2 *Antimony standard solution II*, 1.00 mL = 10.0 µg Sb: Dilute 50.0 mL antimony standard solution I to 500.0 mL with demineralized water.

5.3 *Antimony standard solution III*, 1.0 mL = 0.10 µg Sb: Dilute 5.0 mL antimony standard



200-mL Berzelius beaker or 300-mL fleaker

solution II to 500.0 mL with demineralized water. Prepare fresh before each use.

5.4 *Hydrochloric acid,* concentrated (sp gr 1.19).

5.5 *Nitric acid,* concentrated (sp gr 1.41).

5.6 *Potassium iodide solution,* 15 g/100 mL: Dissolve 15 g KI in 100 mL demineralized water. This solution is stable when stored in an amber bottle.

5.7 *Sodium borohydride solution,* 4 g/100 mL: Dissolve 4 g $NaBH_4$ and 2 g NaOH in 100 mL demineralized water. Prepare fresh before each use.

5.8 *Stannous chloride solution,* 4.2 g/100 mL concentrated HCl: Dissolve 5 g $SnCl_2 \cdot 2H_2O$ in 100 mL concentrated HCl (sp gr 1.19). This solution is unstable. Prepare fresh daily.

5.9 *Sulfuric acid, 9M: Cautiously,* and with constant stirring and cooling, add 250 mL concentrated H_2SO_4 (sp gr 1.84) to 250 mL demineralized water.

6. Procedure

6.1 Follow instructions in paragraph 6.1.1 for water or water-suspended sediment and in paragraph 6.1.2 for bottom material.

6.1.1 Pipet a volume of well-mixed sample containing less than 1.5 µg Sb (100 mL max) into a 200-mL Berzelius beaker and dilute to 100 mL with demineralized water.

6.1.2 Weigh a portion of the prepared bottom-material sample containing less than 1.5 µg Sb (100 mg max); transfer to a 200-mL Berzelius beaker and add 100 mL demineralized water (NOTE 1).

NOTE 1. Do not use more than 100 mg of bottom material; otherwise, severe bumping and loss of antimony may occur during the subsequent digestion of the sample.

6.2 Prepare, in 200-mL Berzelius beakers, a blank and sufficient standards containing from 0.1 to 1.5 µg Sb by diluting 1.0 to 15.0 mL portions of antimony standard solution III to 100 mL with demineralized water.

6.3 To each beaker, add 7 mL 9*M* H_2SO_4 and 5 mL concentrated HNO_3. Add a small boiling chip and carefully evaporate to fumes of SO_3. Maintain an excess of HNO_3 until all organic matter is destroyed. This prevents darkening of the solution and possible reduction and loss of antimony. Cool, add 25 mL demineralized water, and again evaporate to fumes of SO_3 to expel oxides of nitrogen.

6.4 Cool, and adjust the volume of each beaker to approx. 100 mL with demineralized water.

6.5 To each beaker, add successively, with thorough mixing after each addition, 8 mL concentrated HCl, 1 mL KI solution, and 0.5 mL $SnCl_2$ solution. Allow about 15 min for reaction to proceed.

6.6 Attach one beaker at a time to the rubber stopper containing the gas dispersion tube.

6.7 Fill the medicine dropper with 2 mL $NaBH_4$ solution and insert into hole in rubber stopper.

6.8 Add the $NaBH_4$ solution to the sample solution. After the absorbance has reached a maximum and has returned to the baseline, remove the beaker. Rinse the gas dispersion tube in demineralized water before proceeding to the next sample. Treat each succeeding sample, blank, and standard in a like manner.

7. Calculations

7.1 Determine the micrograms of antimony in each sample from a plot of absorbances of standards. Exact reproducibility is not obtained, and an analytical curve must be prepared with each set of samples.

7.2 Determine the concentration of dissolved or total antimony in each sample as follows:

$$\text{Sb } (\mu g/L) = \frac{1{,}000}{\text{mL sample}} \times \mu g \text{ Sb in sample}$$

7.3 To determine the concentration of suspended total antimony, subtract dissolved-antimony concentration from total-antimony concentration.

7.4 Determine the concentration of antimony in air-dried bottom material as follows:

$$\text{Sb } (\mu g/g) = \frac{\mu g \text{ Sb in sample}}{\text{wt of sample (g)}}$$

8. Report

8.1 Report antimony, dissolved (01095), total (01097), and suspended-total (01096), concentrations as follows: less than 100 µg/L, nearest microgram per liter; 100 µg/L and above, two significant figures.

8.2 Report antimony, total - in - bottom-material (01098), concentrations as follows: less than 100 µg/g, nearest microgram per gram; 100 µg/g and above, two significant figures.

9. Precision

9.1 Precision for dissolved antimony for seven samples expressed in terms of percent relative standard deviation is as follows:

Number of laboratories	Mean (µg/L)	Relative standard deviation (percent)
3	2.0	0
3	2.0	50
3	2.3	26
3	4.3	28
3	4.3	35
3	4.7	13
3	8.0	0

9.2 It is estimated that the percent relative standard deviation for total and suspended total antimony and for total antimony in bottom material will be greater than that reported for dissolved antimony.