

METHOD #: 204.1 Approved for NPDES (Editorial Revision 1978)

TITLE: Antimony (AA, Direct Aspiration)

ANALYTE: CAS # Sb Antimony 7440-36-0

INSTRUMENTATION: AA

STORET No. Total 01097
Dissolved 01095
Suspended 01096

Optimum Concentration Range: 1-40 mg/L using a wavelength of 217.6 nm

Sensitivity: 0.5 mg/L

Detection Limit: 0.2 mg/L

1.0 Preparation of Standard Solution

- 1.1 Stock Solution: Carefully weigh 2.7426 g of antimony potassium tartrate $K(SbO)C_4H_4O_6 \cdot \frac{1}{2}H_2O$ (analytical reagent grade) and dissolve in deionized distilled water. Dilute to 1 liter with deionized distilled water. 1 mL = 1 mg Sb(1000 mg/L).
- 1.2 Prepare dilutions of the stock solution to be used as calibration standards at the time of analysis. The calibration standards should be prepared using the same type of acid and at the same concentration as will result in the sample to be analyzed either directly or after processing.

2.0 Sample Preservation

- 2.1 For sample handling and preservation, see part 4.1 of the Atomic Absorption Methods section of this manual.

3.0 Sample Preparation

- 3.1 The procedures for preparation of the sample as given in parts 4.1.1 through 4.1.4 of the Atomic Absorption Methods section of this manual have been found to be satisfactory.

4.0 Instrumental Parameters (General)

- 4.1 Antimony hollow cathode lamp
- 4.2 Wavelength: 217.6 nm
- 4.3 Fuel: Acetylene
- 4.4 Oxidant: Air
- 4.5 Type of flame: Fuel lean

5.0 Analysis Procedure

- 5.1 For analysis procedure and calculation, see "Direct Aspiration", part 9.1 of the

Atomic Absorption Methods section of this manual.

6.0 Interferences

- 6.1 In the presence of lead (1000 mg/L), a spectral interference may occur at the 217.6 nm resonance line. In this case the 231.1 nm antimony line should be used.
- 6.2 Increasing acid concentrations decrease antimony absorption. To avoid this effect, the acid concentration in the samples and in the standards should be matched.

7.0 Notes

- 7.1 Data to be entered into STORET must be reported as $\mu\text{g/L}$.
- 7.2 For concentrations of antimony below 0.35 mg/L, the furnace procedure (Method 204.2) is recommended.

8.0 Precision and Accuracy

- 8.1 In a single laboratory (EMSL), using a mixed industrial-domestic waste effluent at concentrations of 5.0 and 15 mg Sb/L, the standard deviations were ± 0.08 and 0.1, respectively. Recoveries at these levels were 96% and 97%, respectively.