

**METHOD #: 204.2** Approved for NPDES (Issued 1978)  
**TITLE:** Antimony (AA, Furnace Technique)  
**ANALYTE:** CAS # Sb Antimony 7440-36-0  
**INSTRUMENTATION:** AA  
**STORET No.** Total 01097  
Dissolved 01095  
Suspended 01096  
**Optimum Concentration Range:** 20-300  $\mu\text{g/L}$   
**Detection Limit:** 3  $\mu\text{g/L}$

#### 1.0 Preparation of Standard Solution

- 1.1 Stock solution: Prepare as described under "direct aspiration method".
- 1.2 Prepare dilutions of the stock solution to be used as calibration standards at the time of analysis. These solutions are also to be used for "standard additions".
- 1.3 The calibration standard should be diluted to contain 0.2% (v/v)  $\text{HNO}_3$ .

#### 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 thru 4.1.3 of the Atomic Absorption Methods section of this manual should be followed including the addition of sufficient 1:1 HCl to dissolve the digested residue for the analysis of suspended or total antimony. The sample solutions used for analysis should contain 2% (v/v)  $\text{HNO}_3$ .

#### 4.0 Instrument Parameters (General)

- 4.1 Drying Time and Temp: 30 sec-125°C.
- 4.2 Ashing Time and Temp: 30 sec-800°C.
- 4.3 Atomizing Time and Temp: 10 sec-2700°C.
- 4.4 Purge Gas Atmosphere: Argon
- 4.5 Wavelength: 217.6 nm
- 4.6 Other operating parameters should be set as specified by the particular instrument manufacturer.

#### 5.0 Analysis Procedure

- 5.1 For the analysis procedure and the calculation, see "Furnace Procedure" part 9.3 of the Atomic Absorption Methods section of this manual.

## 6.0 Notes

- 6.1 The above concentration values and instrument conditions are for a Perkin-Elmer HGA-2100, based on the use of a 20  $\mu\text{L}$  injection, continuous flow purge gas and non-pyrolytic graphite. Smaller size furnace devices or those employing faster rates of atomization can be operated using lower atomization temperatures for shorter time periods than the above recommended settings.
- 6.2 The use of background correction is recommended.
- 6.3 Nitrogen may also be used as the purge gas.
- 6.4 If chloride concentration presents a matrix problem or causes a loss previous to atomization, add an excess of 5 mg of ammonium nitrate to the furnace and ash using a ramp accessory or with incremental steps until the recommended ashing temperature is reached.
- 6.5 For every sample matrix analyzed, verification is necessary to determine that method of standard addition is not required (see part 5.2.1 of the Atomic Absorption Methods section of this manual).
- 6.6 If method of standard addition is required, follow the procedure given earlier in part 8.5 of the Atomic Absorption Methods section of this manual.
- 6.7 Data to be entered into STORET must be reported as  $\mu\text{g/L}$ .

## 7.0 Precision and Accuracy

- 7.1 Precision and accuracy data are not available.