METHOD #: 239.2 Approved for NPDES and SDWA (Issued 1978)

TITLE: Lead (AA, Furnace Technique)

ANALYTE: CAS # Pb Lead 7439-92-1

INSTRUMENTATION: AA

STORET No. Total 01051

Dissolved 01049 Suspended 01050

Optimum Concentration Range: 5-100 μ g/L

Detection Limit: $1 \mu g/L$

1.0 Preparation of Standard Solution

- 1.1 Stock solution: Prepare as described under "direct aspiration method".
- 1.2 Lanthanum Nitrate Solution: Dissolve 58.64 g of ACS reagent grade La_2O_3 in 100 mL conc. HNO $_3$ and dilute to 1000 mL with deionized distilled water. 1 mL = 50 mg La.
- 1.3 Working Lead Solution: Prepare dilutions of the stock lead solution to be used as calibration standards at the time of analysis. Each calibration standard should contain 0.5% (v/v) HNO₃. To each 100 mL of diluted standard add 10 mL of the lanthanum nitrate solution.

2.0 Sample Preservation

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

3.0 Sample Preparation

- 3.1 Prepare as described under "direct aspiration method". Sample solutions for analysis should contain 0.5% (v/v) HNO₃.
- 3.2 To each 100 mL of prepared sample solution add 10 mL of the lanthanum nitrate solution.

4.0 Instrument Parameters (General)

- 4.1 Drying Time and Temp: 30 sec-125°C.
- 4.2 Ashing Time and Temp: 30 sec-500°C.
- 4.3 Atomizing Time and Temp: 10 sec-2700°C.
- 4.4 Purge Gas Atmosphere: Argon
- 4.5 Wavelength: 283.3 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 in 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 μ 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 Greater sensitivity can be achieved using the 217.0 nm line, but the optimum concentration range is reduced. The use of a lead electrodeless discharge lamp at this lower wavelength has been found to be advantageous. Also a lower atomization temperature (2400°C) may be preferred.
- 6.4 To suppress sulfate interference (up to 1500 ppm) lanthanum is added as the nitrate to both samples and calibration standards. (Atomic Absorption Newsletter Vol. 15, No. 3, p 71, May-June 1976.)
- 6.5 Since glassware contamination is a severe problem in lead analysis, all glassware should be cleaned immediately prior to use, and once cleaned, should not be open to the atmosphere except when necessary.
- 6.6 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).
- 6.7 For quality control requirements and optional recommendations for use in drinking water analyses, see part 10 of the Atomic Absorption Methods section of this manual.
- 6.8 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.
- 6.9 Data to be entered into STORET must be reported as μ g/L.

7.0 Precision and Accuracy

7.1 In a single laboratory (EMSL), using Cincinnati, Ohio tap water spiked at concentrations of 25, 50, and 100 ug Pb/L, the standard deviations were \pm 1.3, \pm 1.6, and \pm 3.7, respectively. Recoveries at these levels were 885%, 92%, and 95% respectively.