

<b>METHOD #:</b> 239.2	Approved for NPDES and SDWA (Issued 1978)
<b>TITLE:</b>	Lead (AA, Furnace Technique)
<b>ANALYTE:</b>	CAS # Pb Lead 7439-92-1
<b>INSTRUMENTATION:</b>	AA
<b>STORET No.</b>	Total 01051 Dissolved 01049 Suspended 01050
<b>Optimum Concentration Range:</b>	5-100 $\mu\text{g/L}$
<b>Detection Limit:</b>	1 $\mu\text{g/L}$

#### 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  $\text{La}_2\text{O}_3$  in 100 mL conc.  $\text{HNO}_3$  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)  $\text{HNO}_3$ . To each 100 mL of diluted standard add 10 mL of the lanthanum nitrate solution.

#### 2.0 Sample Preservation

- 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 Prepare as described under "direct aspiration method". Sample solutions for analysis should contain 0.5% (v/v)  $\text{HNO}_3$ .
- 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  $\mu\text{L}$  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 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  $\mu\text{g/L}$ .

## 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  $\mu\text{g Pb/L}$ , 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.