

METHOD #: 246.1 Approved for NPDES (Editorial Revision 1978)

TITLE: Molybdenum (AA, Direct Aspiration)

ANALYTE: CAS # Mo Molybdenum 7439-98-7

INSTRUMENTATION: AA

STORET No. Total 01062
Dissolved 01060
Suspended 01061

Optimum Concentration Range: 1-40 mg/L using a wavelength of 313.3 nm

Sensitivity: 0.4 mg/L

Detection Limit: 0.1 mg/L

1.0 Preparation of Standard Solution

- 1.1 Stock Solution: Dissolve 1.840 g of ammonium molybdate $(\text{NH}_4)_6\text{Mo}_7\text{O}_{24}\cdot 4\text{H}_2\text{O}$ (analytical reagent grade) in deionized distilled water and dilute to 1 liter.
1 mL = 1 mg Mo (1000 mg/L).
- 1.2 Aluminum nitrate solution: Dissolve 139 g aluminum nitrate, $\text{Al}(\text{NO}_3)_3\cdot 9\text{H}_2\text{O}$, in 150 mL of deionized distilled water; heat to effect solution. Allow to cool and make up to 200 mL.
- 1.3 Prepare dilutions of the stock molybdenum 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. To each 100 mL of standard and sample alike, add 2 mL of the aluminum nitrate solution.

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 4.1.4 of the Atomic Absorption Methods section of this manual have been found to be satisfactory.

4.0 Instrumental Parameters (General)

- 4.1 Molybdenum hollow cathode lamp
- 4.2 Wavelength: 313.3 nm
- 4.3 Fuel: Acetylene
- 4.4 Oxidant: Nitrous Oxide
- 4.5 Type of flame: Fuel rich

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 With the recommended nitrous oxide-acetylene flame, interferences of calcium and other ions may be controlled by adding 1000 mg/L of a refractory metal such as aluminum [Anal. Chem. Acta 44, 437 (1969)]. This should be done to both samples and standards alike.

7.0 Notes

7.1 Data to be entered into STORET must be reported as $\mu\text{g/L}$.

7.2 For concentrations of molybdenum below 0.2 mg/L, the furnace procedure, Method 246.2, is recommended.

8.0 Precision and Accuracy

8.1 In a single laboratory (EMSL), using a mixed industrial-domestic waste effluent at concentrations of 0.30, 1.5 and 7.5 mg Mo/L, the standard deviations were ± 0.007 , ± 0.02 and ± 0.07 , respectively. Recoveries at these levels were 100%, 96% and 95%, respectively.