METHOD #: 252.2 Approved for NPDES (Issued 1978)

TITLE: Osmium (AA, Furnace Technique)

ANALYTE: CAS # Os Osmium 7440-04-2

INSTRUMENTATION: AA

**STORET No.** Total Not Assigned

**Optimum Concentration Range:**  $50-500 \mu g/L$  **Detection Limit:**  $20 \mu g/L$ 

- 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 1% (v/v) HNO<sub>3</sub>.
- 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 Prepare as described under "direct aspiration method", except omit the addition of  $H_2SO_4$  on the final volume adjustment. Sample solutions for analysis should contain 1% (v/v)  $HNO_3$ .
- 4.0 Instrument Parameters (General)
  - 4.1 Drying Time and Temp: 30 sec-105°C.
  - 4.2 Ashing Time and Temp: See NOTE 3 below.
  - 4.3 Atomizing Time and Temp: 10 sec-2700°C.
  - 4.4 Purge Gas Atmosphere: Argon
  - 4.5 Wavelength: 290.9 nm
  - 4.6 Other operating parameters should be set as specified by the particular instrument manufacturer.
- 5.0 Analysis Procedure
  - 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 Osmium tetroxide, the usual commercial form, is very volatile and highly toxic. Care should be exercised when working with this compound.
- 6.2 The above concentration values and instrument conditions are for a Perkin-Elmer HGA- 2100, based on the use of a 20 uL injection, continuous flow purge gas and non-pyrolytic graphite.
- 6.3 Since OsO<sub>4</sub> volatilizes near 150°C, the allowable ashing temperature must be verified in the sample matrix being analyzed.
- 6.4 The use of background correction is recommended.
- 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.

## 7.0 Precision and Accuracy

7.1 Precision and accuracy data are not available at this time.