METHOD #: 270.2	Approved for NPDES and SDWA (Issued 1978)
TITLE:	Selenium (AA, Furnace Technique)
ANALYTE:	CAS # Selenium 7782-49-2
INSTRUMENTATION:	AA
STORET No.	Total 01147 Dissolved 01145 Suspended 01146
Optimum Concentration Range: **Detection Limit:**	5-100 μg/L 2 μg/L

1.0 Preparation of Standard Solution

1.1 Stock Selenium Solution: Dissolve 0.3453 g of selenous acid (actual assay 94.6% H_2SeO_3) in deionized distilled water and make up to 200 mL. 1 mL = 1 mg Se (1000 mg/L).

1.2 Nickel Nitrate Solution, 5%: Dissolve 24.780 g of ACS reagent grade $Ni(NO_3)_2 \cdot 6H_2O$ in deionized distilled water and make up to 100 mL.

1.3 Nickel Nitrate Solution, 1%: Dilute 20 mL of the 5% nickel nitrate to 100 mL with deionized distilled water.

1.4 Working Selenium Solution: Prepare dilutions of the stock solution to be used as calibration standards at the time of analysis. Withdraw appropriate aliquots of the stock solution, add 1 mL of conc. HNO_3, 2 mL of 30% H_2O_2 and 2 mL of the 5% nickel nitrate solution. Dilute to 100 mL with deionized distilled water.

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 Transfer 100 mL of well-mixed sample to a 250 mL Griffin beaker, add 2 mL of 30% H_2O_2 and sufficient conc. HNO_3 to result in an acid concentration of 1 %(v/v). Heat for 1 hour at 95°C or until the volume is slightly less than 50 mL.

3.2 Cool and bring back to 50 mL with deionized distilled water.

3.3 Pipet 5 mL of this digested solution into a 10-mL volumetric flask, add 1 mL of the 1% nickel nitrate solution and dilute to 10 mL with deionized distilled water. The sample is now ready for injection into the furnace.

NOTE: If solubilization or digestion is not required adjust the HNO_3 concentration of the sample to 1% (v/v) and add 2 mL of 30% H_2O_2 and 2 mL of 5% nickel nitrate to each 100 mL of sample. The volume of the calibration standard should be adjusted with deionized distilled water to match the

volume change of the sample.

4.0 Instrument Parameters

4.1 Drying time and temperature: 30 sec @ 125°C
4.2 Charring time and temperature: 30 sec @ 1200°C
4.3 Atomizing time and temperature: 10 sec @ 2700°C
4.4 Purge Gas Atmosphere: Argon
4.5 Wavelength: 196.0 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 μL injection, purge gas interrupt 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 Selenium analysis suffers interference from chlorides (> 800 mg/L) and sulfate (> 200 mg/L). For the analysis of industrial effluents and samples with concentrations of sulfate from 200 to 2000 mg/L, both samples and standards should be prepared to contain 1% nickel.
6.4 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).
6.5 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.
6.6 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 entered into STORET must be reported as μg/L.

7.0 Precision and Accuracy

7.1 Using a sewage treatment plant effluent containing < 2 μg/L and spiked with a concentration of 20 μg/L, a recovery of 99% was obtained.
7.2 Using a series of industrial waste effluents spiked at a 50 μg/L level, recoveries ranged from 94 to 112%.
7.3 Using a 0.1% nickel nitrate solution as a synthetic matrix with selenium concentrations of 5, 10, 20, 40, 50, and 100 μg/L, relative standard deviations of 14.2, 11.6, 9.3, 7.2, 6.4 and 4.1%, respectively, were obtained at the 95% confidence level.
7.4 In a single laboratory (EMSL), using Cincinnati, Ohio tap water spiked at

concentrations of 5, 10, and 20 μg Se/L, the standard deviations were ±0.6, ±0.4, and ±0.5, respectively. Recoveries at these levels were 92%, 98%, and 100%, respectively.

8.0 Reference

8.1 "Determining Selenium in Water, Wastewater, Sediment and Sludge By Flameless Atomic Absorption Spectroscopy", Martin, T.D., Kopp, J. F. and Ediger, R.D. Atomic Absorption Newsletter 14, 109 (1975).