

METHOD #: 206.2	Approved for NPDES and SDWA (Issued 1978)
TITLE:	Arsenic (AA, Furnace Technique)
ANALYTE:	CAS # As Arsenic 7440-38-2
INSTRUMENTATION:	AA
STORET No.	Total 01002 Dissolved 01000 Suspended 01001
Optimum Concentration Range:	5-100 $\mu\text{g/L}$
Detection Limit:	1 $\mu\text{g/L}$

1.0 Preparation of Standard Solution

- 1.1 Stock solution: Dissolve 1.320 g of arsenic trioxide, As_2O_3 (analytical reagent grade) in 100 mL of deionized distilled water containing 4 g NaOH. Acidify the solution with 20 mL conc. HNO_3 and dilute to 1 liter. 1 mL = 1 mg As (1000 mg/L).
- 1.2 Nickel Nitrate Solution, 5%: Dissolve 24.780 g of ACS reagent grade $\text{Ni}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ in deionized distilled water and make up to 100mL.
- 1.3 Nickel Nitrate Solution, 1%: Dilute 20 mL of the 5% nickel nitrate to 100 mL with deionized distilled water.
- 1.4 Working Arsenic 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 , 2mL of 30% H_2O_2 and 2mL 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 (General)

- 4.1 Drying Time and Temp: 30 sec-125°C.
- 4.2 Ashing Time and Temp: 30 sec-1100°C.
- 4.3 Atomizing Time and Temp: 10 sec-2700°C.
- 4.4 Purge Gas Atmosphere: Argon
- 4.5 Wavelength: 193.7nm
- 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 uL 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 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).
- 6.4 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.
- 6.5 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 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 a mixed industrial-domestic waste effluent containing 15 $\mu\text{g/L}$ and spiked with concentrations of 2, 10 and 25 $\mu\text{g/L}$, recoveries of 85%, 90% and 88% were obtained respectively. The relative standard deviation at these concentrations levels were $\pm 8.8\%$, $\pm 8.2\%$, $\pm 5.4\%$ and $\pm 8.7\%$, respectively.
- 7.2 In a single laboratory (EMSL), using Cincinnati, Ohio tap water spiked at concentrations of 20, 50 and 100 $\mu\text{g As/L}$, the standard deviations were ± 0.7 , ± 1.1 and ± 1.6 respectively. Recoveries at these levels were 105%, 106% and 101%, respectively.