

<b>METHOD #:</b> 206.2	Approved for NPDES and SDWA (Issued 1978)
<b>TITLE:</b>	Arsenic (AA, Furnace Technique)
<b>ANALYTE:</b>	CAS # As Arsenic 7440-38-2
<b>INSTRUMENTATION:</b>	AA
<b>STORET No.</b>	Total 01002 Dissolved 01000 Suspended 01001
<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: Dissolve 1.320 g of arsenic trioxide,  $\text{As}_2\text{O}_3$  (analytical reagent grade) in 100 mL of deionized distilled water containing 4 g NaOH. Acidify the solution with 20 mL conc.  $\text{HNO}_3$  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.  $\text{HNO}_3$ , 2mL of 30%  $\text{H}_2\text{O}_2$  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%  $\text{H}_2\text{O}_2$  and sufficient conc.  $\text{HNO}_3$  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  $\text{HNO}_3$  concentration of the sample to 1% (v/v) and add 2 mL of 30%  $\text{H}_2\text{O}_2$  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  $\pm 0.7$ ,  $\pm 1.1$  and  $\pm 1.6$  respectively. Recoveries at these levels were 105%, 106% and 101%, respectively.