METHOD #: 272.1 Approved for NPDES and SDWA (Technical Rev.

1978)

TITLE: Silver (AA, Direct Aspiration)

ANALYTE: CAS # Ag Silver 7440-22-4

INSTRUMENTATION: AA

STORET No. Total 01077

Dissolved 01075 Suspended 01076

Optimum Concentration Range: 0.1-4 mg/L using a wavelength of 328.1 nm

Sensitivity: 0.06 mg/L **Detection Limit:** 0.01 mg/L

1.0 Preparation of Standard Solution

1.1 Stock Solution: Dissolve 1.575 g of $AgNO_3$ (analytical reagent grade) in deionized distilled water, add 10 mL conc. HNO_3 and make up to 1 liter. 1 mL = 1 mg Ag (1000 mg/L).

- 1.2 Prepare dilutions of the stock solution to be used as calibration standards at the time of analysis. The calibration standards should be prepared using nitric acid and at the same concentration as will result in the sample to be analyzed either directly or after processing.
- 1.3 Iodine Solution, 1 N: Dissolve 20 grams of potassium iodide, KI (analytical reagent grade) in 50 mL of deionized distilled water, add 12.7 grams of iodine, I₂ (analytical reagent grade) and dilute to 100 mL. Store in a brown bottle.
- 1.4 Cyanogen Iodide (CNI) Solution: To 50 mL of deionized distilled water add 4.0 mL conc. NH $_4$ OH, 6.5 grams KCN, and 5.0 mL of 1.0 N $_2$ I solution. Mix and dilute to 100 mL with deionized distilled water. Fresh solution should be prepared every two weeks. (1)

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 4.1.3 of the Atomic Absorption Methods section of this manual have been found to be satisfactory; however, the residue must be taken up in dilute nitric acid rather than hydrochloric to prevent precipitation of AgCl.

4.0 Instrumental Parameters (General)

- 4.1 Silver hollow cathode lamp
- 4.2 Wavelength: 328.1 nm
- 4.3 Fuel: Acetylene
- 4.4 Oxidant: Air
- 4.5 Type of flame: Oxidizing

5.0 Analysis Procedure

5.1 For the analysis procedure and the calculation, see "Direct Aspiration", part 9.1 of the Atomic Absorption Methods section of this manual.

6.0 Notes

- 6.1 For levels of silver below 30 μ g/L, either the Special Extraction Procedure, given in part 9.2 of the Atomic Absorption Methods section or the furnace procedure, Method 272.2, is recommended.
- 6.2 Silver nitrate standards are light sensitive. Dilutions of the stock should be discarded after use as concentrations below 10 mg/L are not stable over long periods of time.
- 6.3 If absorption to container walls or the formation of AgCl is suspected, make the sample basic using conc. NH_4OH and add 1 mL of (CNI) solution per 100 mL of sample. Mix the sample and allow to stand for 1 hour before proceeding with the analysis.(1)
- 6.4 The 338.2 nm wavelength may also be used. This has a relative sensitivity of 2.
- 6.5 Data to be entered into STORET must be reported as μ g/L.

7.0 Precision and Accuracy

7.1 In a round-robin study reported by Standard Methods, a synthetic sample containing 50 μ g Ag/L was analyzed by 50 laboratories with a reported standard deviation of \pm 8.8 and a relative error 10.6%.

8.0 References

- 8.1 "The Use of Cyanogen Iodide (CNI) as a Stabilizing Agent for Silver in Photographic Processing Effluent Sample", Owerbach, Daniel, Photographic Technology Division, Eastman Kodak Company, Rochester, N.Y. 14650.
- 8.2 Standard Methods for Examination of Water and Wastewater, 14th Edition, p. 148, Method 301A.