

METHOD #: 206.4 Approved for NPDES and SDWA (Editorial Revision 1974)

TITLE: Arsenic (Spectrophotometric, SDDC)

ANALYTE: CAS # As Arsenic 7440-38-2

INSTRUMENTATION: Spectrophotometer

STORET No. 01002

Inorganic, Dissolved 00095

Inorganic, Total 00997

Inorganic, Suspended 00996

1.0 Scope and Application

- 1.1 The silver diethyldithiocarbamate method determines inorganic arsenic when present in concentrations at or above 10 $\mu\text{g/L}$. The method is applicable to drinking water and most fresh and saline waters in the absence of high concentrations of chromium, cobalt, copper, mercury, molybdenum, nickel, and silver. Domestic and industrial wastes may also be analyzed after digestion (see 3.3).
- 1.2 Difficulties may be encountered with certain industrial waste materials containing volatile substances. High sulfur content of wastes may exceed removal capacity of the lead acetate scrubber.

2.0 Summary of Method

- 2.1 Arsenic in the sample is reduced to arsine, AsH_3 , in acid solution in a hydrogen generator. The arsine is passed through a scrubber to remove sulfide and is absorbed in a solution of silver diethyldithiocarbamate dissolved in pyridine. The red complex thus formed is measured in a spectrophotometer at 535 nm.

3.0 Comments

- 3.1 In analyzing drinking water and most surface and ground waters, interferences are rarely encountered. Industrial waste samples should be spiked with a known amount of arsenic to establish adequate recovery.
- 3.2 It is essential that the system be airtight during evolution of the arsine, to avoid losses.
- 3.3 If concentration of the sample and/or oxidation of any organic matter is required, refer to Method 206.5. [Standard Methods, 14th Edition, Method 404B, p. 284, Procedure 4.a (1975)]. For sample handling and preservation, see part 4.1 of the Atomic Absorption Methods section of this manual.
 - 3.3.1 Since nitric acid gives a negative interference in this test, use sulfuric acid as a preservative if only inorganic arsenic is being measured.
- 3.4 1-Ephedrine in chloroform has been found to be a suitable solvent for silver

diethyldithiocarbamate if the analyst finds the odor of pyridine objectionable [Anal. Chem. 45, 1786 (1973)].

3.5 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.

4.0 Precision and Accuracy

4.1 In a round-robin study reported by Standard Methods a synthetic unknown sample containing $40 \mu\text{g/L}$, as As, with other metals was analyzed in 46 laboratories. Relative standard deviation was $\pm 13.8\%$ and relative error was 0%.

5.0 Reference

5.1 The procedure to be used for this determination is found in: Standard Methods for the Examination of Water and Wastewater, 14th Edition, p. 283, Method 404A (1975).