

METHOD #: 206.5

Approved for NPDES and SDWA (Issued 1978)

TITLE:

Arsenic (Sample Digestion Prior to Total Arsenic Analysis by Silver Diethyldithiocarbamate or Hydride Procedures)

ANALYTE:

CAS # As Arsenic 7440-38-2

INSTRUMENTATION:

AA

1.0 Scope and Application:

- 1.1 Both the silver diethyldithiocarbamate spectrophotometric method and the AA hydride procedure measure inorganic arsenic. Therefore, if either of these procedures are being employed for the purpose of measuring total arsenic (inorganic plus organic), all organically bound arsenic must first be converted to an inorganic form prior to the analytical determination. This may be accomplished with $\text{H}_2\text{SO}_4 \cdot \text{HNO}_3$.

2.0 Procedure

- 2.1 To a suitable sample containing from 2 to 30 ug of arsenic, add 7 mL (1 + 1) H_2SO_4 and 5 mL conc HNO_3 . Evaporate the sample to SO_3 fumes. Caution: If the sample chars, stop the digestion immediately, cool and add additional conc HNO_3 . Continue digestion adding additional conc HNO_3 as necessary.
- 2.2 If the sample remains colorless, or straw-yellow during evolution of SO_3 fumes, the digestion is complete.
- 2.3 Cool the digested sample, add about 25 mL distilled water, and again evaporate to SO_3 fumes to expel oxides of nitrogen.
- 2.4 The sample is now ready for analysis using either the hydride or spectrophotometric procedure.

3.0 Interferences

- 3.1 All traces of nitric acid must be removed before either the spectrophotometric or the hydride procedures are applied. Oxides of nitrogen should be expelled by taking the sample to fumes of SO_3 .

4.0 Notes

- 4.1 The digestion step may be carried out in a flask on a hot-plate or in a Kjeldahl apparatus. This digestion step may also be used, in effect, to concentrate the sample, inasmuch as any size volume may be processed.

Bibliography

1. Standard Methods for the Examination of Water and Wastewater, p285, method 404B, step 4a. 14th Edition (1975).