Designation: D2972 – 08

Standard Test Methods for Arsenic in Water[1]

This standard is issued under the fixed designation D2972; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 These test methods[2] cover the photometric and atomic absorption determination of arsenic in most waters and wastewaters. Three test methods are given as follows:

	Concentration Range	Sections
Test Method A—Silver Diethyldithiocarbamate Colorimetric	5 to 250 µg/L	7 to 15
Test Method B—Atomic Absorption, Hydride Generation	1 to 20 µg/L	16 to 24
Test Method C—Atomic Absorption, Graphite Furnace	5 to 100 µg/L	25 to 33

1.2 The analyst should direct attention to the precision and bias statements for each test method. It is the user's responsibility to ensure the validity of these test methods for waters of untested matrices.

1.3 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.4 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.* For specific hazard statements, see Note 1 *and* Note 6.

2. Referenced Documents

2.1 *ASTM Standards:*[3]

D1129 Terminology Relating to Water
D1193 Specification for Reagent Water
D2777 Practice for Determination of Precision and Bias of Applicable Test Methods of Committee D19 on Water
D3370 Practices for Sampling Water from Closed Conduits
D3919 Practice for Measuring Trace Elements in Water by Graphite Furnace Atomic Absorption Spectrophotometry
D4841 Practice for Estimation of Holding Time for Water Samples Containing Organic and Inorganic Constituents
D5810 Guide for Spiking into Aqueous Samples
D5847 Practice for Writing Quality Control Specifications for Standard Test Methods for Water Analysis
E60 Practice for Analysis of Metals, Ores, and Related Materials by Spectrophotometry
E275 Practice for Describing and Measuring Performance of Ultraviolet and Visible Spectrophotometers

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 For definitions of terms used in these test methods refer to Terminology D1129.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *total recoverable arsenic*—an arbitrary analytical term relating to the forms of arsenic that are determinable by the digestion method which is included in the procedure. Some organic-arsenic compounds, such as phenylarsonic acid, disodium methane arsonate, and dimethylarsonic acid, are not recovered completely during the digestion step.

4. Significance and Use

4.1 Herbicides, insecticides, and many industrial effluents contain arsenic and are potential sources of water pollution. Arsenic is significant because of its adverse physiological effects on humans.

5. Purity of Reagents

5.1 Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such

[1] These test methods are under the jurisdiction of ASTM Committee D19 on Water and are the direct responsibility of Subcommittee D19.05 on Inorganic Constituents in Water.

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[2] Similar to that appearing in *Standard Methods for the Examination of Water and Wastewater,* 12th edition, APHA, Inc., New York, NY, 1965, and identical with that in Brown, Eugene, Skougstad, M. W., and Fishman, M. J., "Methods for Collection and Analysis of Water Samples for Dissolved Minerals and Gases," *Techniques of Water-Resources Investigations of the U.S. Geological Survey,* Book 5, 1970 p. 46.

[3] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

*A Summary of Changes section appears at the end of this standard specifications are available.[4] Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

5.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water conforming to Specification D1193 Type I. Other reagent water types may be used provided it is first ascertained that the water is of sufficiently high purity to permit its use without adversely affecting the bias and precision of the test method. Type II water was specified at the time of round robin testing of this test method.

6. Sampling

6.1 Collect the sample in accordance with Practices D3370.

6.2 Preserve the samples with HNO_3 (sp gr 1.42) to a pH of 2 or less immediately at the time of collection; normally about 2 mL/L is required. If only dissolved arsenic is to be determined, filter the sample through a 0.45-µm membrane filter before acidification. The holding times for the samples may be calculated in accordance with Practice D4841.

TEST METHOD A—SILVER DIETHYLDITHIOCARBAMATE COLORIMETRIC

7. Scope

7.1 This test method covers the determination of dissolved and total recoverable arsenic in most waters and waste waters in the range from 5 to 250 µg/L of arsenic.

7.2 The precision and bias data were obtained on reagent water, river water, and process water. The information on precision and bias may not apply to other waters. It is the user's responsibility to ensure the validity of this test method for waters of untested matrices.

8. Summary of Test Method

8.1 Organic arsenic-containing compounds are decomposed by adding sulfuric and nitric acids and repeatedly evaporating the sample to fumes of sulfur trioxide. The arsenic (V) so produced, together with inorganic arsenic originally present, is subsequently reduced to arsenic (III) by potassium iodide and stannous chloride, and finally to gaseous arsine by zinc in hydrochloric acid solution. The resulting mixture of gases is passed through a scrubber containing borosilicate wool impregnated with lead acetate solution and then into an absorption tube containing a solution of silver diethyldithiocarbamate in pyridine. Arsine reacts with this reagent to form a red-colored silver sol having maximum absorbance at about 540 nm. The absorbance of the solution is measured photometrically and the arsenic determined by reference to an analytical curve prepared from standards.

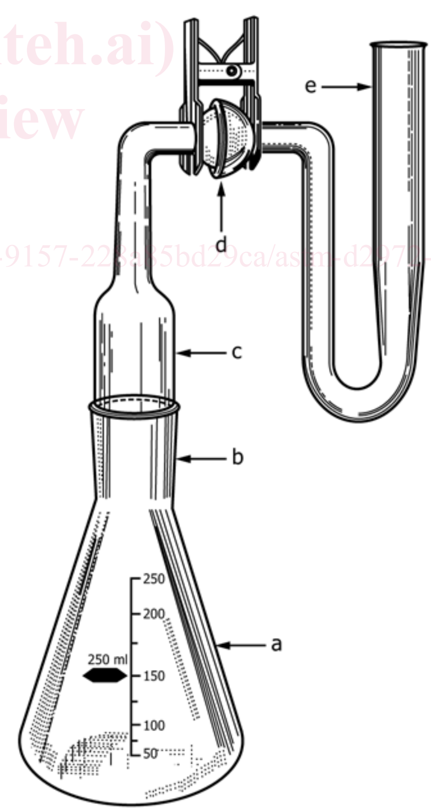
9. Interferences

9.1 Although many samples are relatively free of interferences, several metals, notably cobalt, nickel, mercury, silver, platinum, copper, chromium, and molybdenum, may interfere with the evolution of arsine and with the recovery of arsenic. The presence of any or all of these metals in a sample being analyzed must be considered as a potential source of interference, and the analyst must fully determine the extent of actual interference, if any. This could be accomplished by spiking.

9.2 Hydrogen sulfide and other sulfides interfere, but commonly encountered quantities are effectively removed by the lead acetate scrubber and the digestion.

9.3 Antimony interferes by forming stibine, which distills along with the arsine. Stibine reacts with the color-forming reagent to form a somewhat similar red sol having maximum absorbance near 510 nm. The sensitivity for antimony at 540 nm is only about 8 % that of arsenic (1 mg/L of antimony will show an apparent presence of 0.08 mg/L of arsenic).

9.4 Nitric acid interferes with the test and must be completely eliminated during the digestion.



(a) Generator flask, borosilicate glass, 250-mL capacity.
(b) Standard-taper neck 24/40.
(c) Scrubber, borosilicate glass wool impregnated with lead acetate.
(d) Ground-glass ball-and-socket joint.
(e) Absorber: add AgDDC solution and pack with glass beads.

FIG. 1 Arsine Generator, Scrubber, and Absorber [7]

[4] *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For Suggestions on the testing of reagents not listed by the American Chemical Society, see *Annual Standards for Laboratory Chemicals,* BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary,* U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

10. Apparatus

10.1 *Arsine Generator, Scrubber, and Absorber*,[5] assembled as shown in Fig. 1.

10.2 *Spectrophotometer or Filter Photometer,* suitable for use at 540 nm and providing a light path of at least 10 mm. The filter photometer and photometric practice prescribed in this method shall conform to Practice E60. The spectrophotometer shall conform to Practice E275.

11. Reagents

11.1 *Arsenic Solution, Stock* (1.00 mL = 1.00 mg As)—Commercially purchase or dissolve 1.320 g of arsenic trioxide (As_2O_3) (**Warning:** see Note 1), dried for at least 1 h at 110°C, in 10 mL of NaOH solution (420 g/L) and dilute to 1 L with water. This solution is stable.

NOTE 1—**Warning:** Arsenic trioxide is extremely toxic. Avoid ingestion or inhalation of dry powder during standard preparation. Wash hands thoroughly immediately after handling arsenic trioxide. Under no circumstances pipette any arsenic solutions by mouth.

11.2 *Arsenic Solution, Intermediate* (1.00 mL = 10.0 µg As)— Dilute 5.00 mL of arsenic stock solution to 500 mL with water.

11.3 *Arsenic Solution, Standard* (1.00 mL = 1.00 µg As)—Dilute 10.0 mL of arsenic intermediate solution to 100 mL with water. Prepare fresh before each use.

11.4 *Hydrochloric Acid* (sp gr 1.19)—Concentrated hydrochloric acid (HCl). Use analytical grade acid with an arsenic content not greater than 1×10^{-6} %.

11.5 *Lead Acetate Solution* (100 g/L)—Dissolve 10 g of lead acetate ($Pb(C_2H_3O_2)_2 \cdot 3H_2O$) in 100 mL of water. Store reagent in a tightly stoppered container.

11.6 *Nitric Acid* (sp gr 1.42)—Concentrated nitric acid (HNO_3). Use analytical grade acid with an arsenic content not greater than 1×10^{-6} %.

11.7 *Nitric Acid* (1 + 1)—Add 250 mL of concentrated nitric acid (sp gr 1.42) to 250 mL of water.

11.8 *Potassium Iodide Solution* (150 g/L)—Dissolve 15 g of potassium iodide (KI) in 100 mL of water. Store in an amber bottle.

11.9 *Silver Diethyldithiocarbamate Solution*—Dissolve 1 g of silver diethyldithiocarbamate (AgDDC) in 200 mL of pyridine. This solution is stable for at least several months when stored in an amber bottle.

11.10 *Sodium Hydroxide Solution* (420 g/L)—Dissolve 42 g of sodium hydroxide (NaOH) pellets in 100 mL of water. **Warning:** This is a very exothermic reaction.

11.11 *Stannous Chloride Solution* —Dissolve 40 g of arsenic-free stannous chloride ($SnCl_2 \cdot 2H_2O$) in 100 mL of HCl (sp gr 1.19). Add a few small pieces of mossy tin (which is the common name and is commercially available).

11.12 *Sulfuric Acid* (1 + 1)—Cautiously, and with constant stirring and cooling, add 250 mL of concentrated H_2SO_4 (sp gr 1.84) to 250 mL of water.

11.13 *Zinc, Granular,* 20-mesh. Arsenic content must not exceed 1×10^{-6} %.

12. Standardization

12.1 Clean all glassware before use by rinsing first with hot HNO_3 (1 + 1) and then with water. The absorbers must be additionally rinsed with acetone and then air-dried.

12.2 Prepare, in a 250-mL generator flask, a blank and sufficient standards containing from 0.0 to 25.0 µg of arsenic by diluting 0.0 to 25.0-mL portions of the arsenic standard solution to approximately 100 mL with water.

12.3 Proceed as directed in 13.3 – 13.9.

12.4 Construct an analytical curve by plotting the absorbances of standards versus micrograms of arsenic.

NOTE 2—The response is linear up to 15 µg of arsenic; however, because the curve is nonlinear above 15 µg, it is necessary to have sufficient standards above 15 µg to permit constructing an accurate curve.

13. Procedure

13.1 Clean all glassware before use by rinsing first with hot HNO_3 (1 + 1) and then with water. The absorbers must be additionally rinsed with acetone and then air-dried.

13.2 Pipette a volume of well-mixed acidified sample containing less than 25 µg of arsenic (100 mL maximum) into a generating flask and dilute to approximately 100 mL.

NOTE 3—If only dissolved arsenic is to be determined use a filtered and acidified sample (see 6.2).

13.3 To each flask, add 7 mL of H_2SO_4 (1 + 1) and 5 mL of concentrated HNO_3. Add a small boiling chip and carefully evaporate to dense fumes of SO_3, maintaining an excess of HNO_3 until all organic matter is destroyed. This prevents darkening of the solution and possible reduction and loss of arsenic. Cool, add 25 mL of water, and again evaporate to dense fumes of SO_3. Maintain heating for 15 min to expel oxides of nitrogen.

13.4 Cool, and adjust the volume in each flask to approximately 100 mL with water.

13.5 To each flask add successively, with thorough mixing after each addition, 8 mL of concentrated HCl, 4 mL of KI solution, and 1 mL of $SnCl_2$ solution. Allow about 15 min for complete reduction of the arsenic to the trivalent state.

13.6 Place in each scrubber a plug of borosilicate wool that has been impregnated with lead acetate solution. Assemble the generator, scrubber, and absorber, making certain that all parts fit and are correctly adjusted. Add 3.00 mL of silver diethyldithiocarbamate-pyridine solution to each absorber. Add glass beads to the absorbers until the liquid just covers them.

NOTE 4—Four millilitres of silver diethyldithiocarbamate-pyridine solution may be used with some loss of sensitivity.

13.7 Disconnect each generator, add 6 g of zinc, and reconnect immediately.

[5] Available commercially.

13.8 Allow 30 min for complete evolution of arsine. Warm the generator flasks for a few minutes to make sure that all arsine is released.

13.9 Pour the solutions from the absorbers directly into clean spectrophotometer cells and within 30 minutes measure the absorbance of each at 540 nm.

14. Calculation

14.1 Determine the weight of arsenic in each sample by referring to the analytical curve. Calculate the concentration of arsenic in the sample in micrograms per litre, using Eq 1:

$$\text{Arsenic, } \mu g/L = 1000\ W/V \tag{1}$$

where:

1000 = 1000 mL / Litre
V = volume of sample, mL, and
W = weight of arsenic in sample, µg.

15. Precision and Bias[6]

15.1 The single-operator and overall precision of this method for three laboratories, which included a total of six operators analyzing each sample on three different days, within its designated range varies with the quantity being tested in accordance with Table 1.

15.2 Recoveries of known amounts of arsenic (arsenic trioxide) in a series of prepared standards are given in Table 1.

15.3 The precision and bias data were obtained on reagent water, river water, and process water. The information on precision and bias may not apply to other waters. It is the user's responsibility to ensure the validity of this test method for waters of untested matrices.

15.4 Three independent laboratories participated in the round robin study. Precision and bias for this test method conform to Practice D2777 – 77, which was in place at the time of collaborative testing. Under the allowances made in 1.4 of Practice D2777 – 06, these precision and bias data do meet existing requirements for interlaboratory studies of Committee D19 test methods.

16. Quality Control

16.1 In order to be certain that analytical values obtained using these test methods are valid and accurate within the confidence limits of the test, the following QC procedures must be followed when analyzing arsenic.

16.2 *Calibration and Calibration Verification:*

16.2.1 Analyze at least three working standards containing concentrations of arsenic that bracket the expected sample concentration, prior to analysis of samples, to calibrate the instrument. The calibration correlation coefficient shall be equal to or greater than 0.990. In addition to the initial calibration blank, a calibration blank shall be analyzed at the end of the batch run to ensure contamination was not a problem during the batch analysis.

16.2.2 Verify instrument calibration after standardization by analyzing a standard at the concentration of one of the calibration standards. The concentration of a mid-range standard should fall within ±15 % of the known concentration.

16.2.3 If calibration cannot be verified, recalibrate the instrument.

16.3 *Initial Demonstration of Laboratory Capability:*

16.3.1 If a laboratory has not performed the test before, or if there has been a major change in the measurement system, for example, new analyst, new instrument, etc., a precision and bias study must be performed to demonstrate laboratory capability.

16.3.2 Analyze seven replicates of a standard solution prepared from an Independent Reference Material containing a mid-range concentration of arsenic. The matrix and chemistry of the solution should be equivalent to the solution used in the collaborative study. Each replicate must be taken through the complete analytical test method including any sample preservation and pretreatment steps.

16.3.3 Calculate the mean and standard deviation of the seven values and compare to the acceptable ranges of bias in Table 1. This study should be repeated until the recoveries are within the limits given in Table 1. If a concentration other than the recommended concentration is used, refer to Practice D5847 for information on applying the F test and t test in evaluating the acceptability of the mean and standard deviation.

16.4 *Laboratory Control Sample (LCS):*

16.4.1 To ensure that the test method is in control, analyze a LCS containing a known concentration of arsenic with each batch or 10 samples. If large numbers of samples are analyzed in the batch, analyze the LCS after every 10 samples. The laboratory control samples for a large batch should cover the analytical range when possible. The LCS must be taken through all of the steps of the analytical method including sample preservation and pretreatment. The result obtained for a mid-range LCS shall fall within ±15 % of the known concentration.

16.4.2 If the result is not within these limits, analysis of samples is halted until the problem is corrected, and either all the samples in the batch must be reanalyzed, or the results must be qualified with an indication that they do not fall within the performance criteria of the test method.

16.5 *Method Blank:*

16.5.1 Analyze a reagent water test blank with each batch. The known concentration of arsenic found in the blank should

[6] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D19-1049.

TABLE 1 Precision and Bias for Arsenic by Test Method A, Diethyldithiocarbamate Colorimetric

Water	Amount Added, µg/L	Amount Found, µg/L	S_t	S_o	Bias, %
Reagent Type II	25.0	23.66	1.76	1.78	−5.4
	100.0	95.28	5.21	5.24	−4.7
	200.0	194.99	8.43	8.79	−2.6
Water of Choice	25.0	24.76	2.07	1.84	−0.96
	100.0	97.00	4.15	3.78	−3.0
	200.0	189.01	9.96	9.70	−5.5

be less than 0.5 times the lowest calibration standard. If the known concentration of arsenic is found above this level, analysis of samples is halted until the contamination is eliminated, and a blank shows no contamination at or above this level, or the results must be qualified with an indication that they do not fall within the performance criteria of the test method.

16.6 *Matrix Spike (MS):*

16.6.1 To check for interferences in the specific matrix being tested, perform a MS on at least one sample from each batch by spiking an aliquot of the sample with a known concentration of arsenic and taking it through the analytical method.

16.6.2 The spike known concentration plus the background known concentration of arsenic must not exceed the high calibration standard. The spike must produce a known concentration in the spiked sample that is 2 to 5 times the analyte known concentration in the unspiked sample, or 10 to 50 times the detection limit of the test method, whichever is greater.

16.6.3 Calculate the percent recovery of the spike (P) using the following formula:

$$P = 100[A(V_s + V) - B\,V_s]/C\,V \quad (2)$$

where:

A = analyte known concentration (mg/L) in spiked sample,
B = analyte known concentration (mg/L) in unspiked sample,
C = known concentration (mg/L) of analyte in spiking solution,
V_s = volume (mL) of sample used, and
V = volume (mL) of spiking solution added.

16.6.4 The percent recovery of the spike shall fall within the limits, based on the analyte known concentration, listed in Guide D5810, Table 1. If the percent recovery is not within these limits, a matrix interference may be present in the sample selected for spiking. Under these circumstances, one of the following remedies must be employed: the matrix interference must be removed, all samples in the batch must be analyzed by a test method not affected by the matrix interference, or the results must be qualified with an indication that they do not fall within the performance criteria of the test method.

NOTE 5—Acceptable spike recoveries are dependent on the known concentration of the component of interest. See Guide D5810 for additional information.

16.7 *Duplicate:*

16.7.1 To check the precision of sample analyses, analyze a sample in duplicate with each batch. If the known concentration of the analyte is less than five times the detection limit for the analyte, a matrix spike duplicate (MSD) should be used.

16.7.2 Calculate the standard deviation of the duplicate values and compare to the precision in the collaborative study using an F test. Refer to 6.4.4 of Practice D5847 for information on applying the F test.

16.7.3 If the result exceeds the precision limit, the batch must be reanalyzed or the results must be qualified with an indication that they do not fall within the performance criteria of the test method.

16.8 *Independent Reference Material (IRM):*

16.8.1 In order to verify the quantitative value produced by the test method, analyze an Independent Reference Material (IRM) submitted as a regular sample (if practical) to the laboratory at least once per quarter. The known concentration of the IRM should be in the known concentration mid-range for the method chosen. The value obtained must fall within the control limits established by the laboratory.

TEST METHOD B—ATOMIC ABSORPTION, HYDRIDE GENERATION

17. Scope

17.1 This test method covers the determination of dissolved and total recoverable arsenic in most waters and wastewaters in the range from 1 to 20 µg/L of arsenic. The range may be extended by dilution of the sample.

17.2 The precision and bias data were obtained on reagent water, tap water, salt water, river water, and untreated wastewater. The information on precision and bias may not apply to other waters. It is the user's responsibility to ensure the validity of this test method for waters of untested matrices.

18. Summary of Test Method

18.1 Organic arsenic-containing compounds are decomposed by adding sulfuric and nitric acids and repeatedly evaporating the sample to fumes of sulfur trioxide. The arsenic (V) so produced, together with inorganic arsenic originally present, is subsequently reduced to arsenic (III) by potassium iodide and stannous chloride, and finally to gaseous arsine by zinc in hydrochloric acid solution. Alternatively, the arsenic is converted to arsine by sodium borohydride in hydrochloric acid solution. The arsine is removed from solution by aeration and swept by a flow of nitrogen into a hydrogen flame where it is determined by atomic absorption at 193.7 nm.

19. Interferences

19.1 See 9.1.

20. Apparatus

20.1 *Arsine Vapor Analyzer,* assembled as shown in Fig. 2.[7]

20.2 *Atomic Absorption Spectrophotometer* , (**Warning:** see Note 6) for use at 193.7 nm.

NOTE 6—**Warning:** Because of the toxicity of arsenic, a well-ventilated hood must be used with the atomic absorption spectrometer.

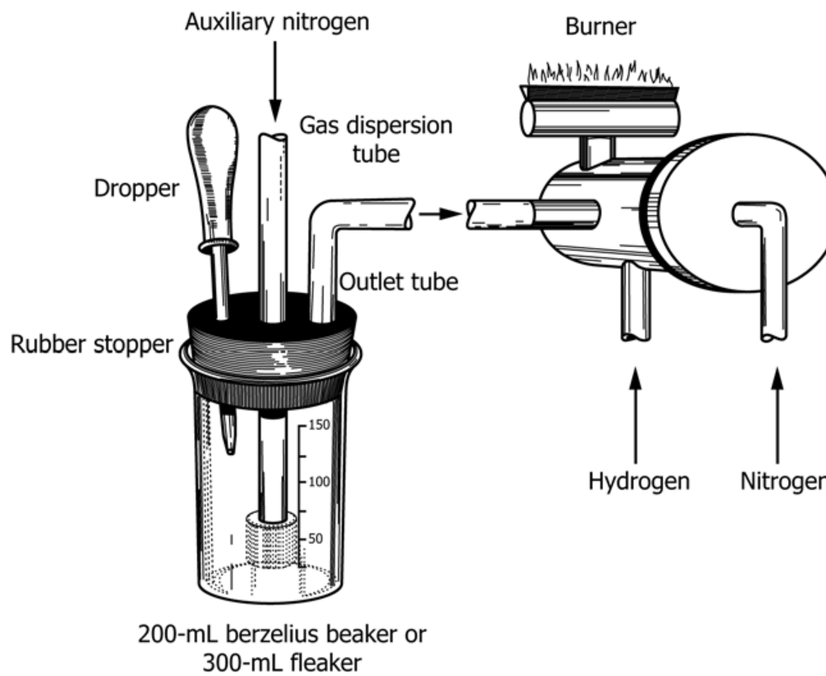
NOTE 7—Follow the manufacturer's instructions for all instrumental parameters.

20.2.1 *Arsenic Light Source*—Arsenic electrodeless discharge lamp or hollow-cathode lamp.

21. Reagents and Materials

21.1 *Arsenic Solution, Stock* (1.00 mL = 1.00 mg As)—See 11.1.

[7] A static system, such as one using a balloon, has been found satisfactory for this purpose. See McFarren, E. F., "New Simplified Methods for Metal Analysis," *Journal of American Water Works Association*, Vol 64, 1972, p. 28.



NOTE 1—Fleaker, trademarked product of Corning Glass Works, and Berzelius beaker are available from most laboratory apparatus dealers.

FIG. 2 Arsine Vapor Analyzer

21.2 *Arsenic Solution, Intermediate* (1.00 mL = 10.0 µg As)—See 11.2.

21.3 *Arsenic Solution, Standard* (1.00 mL = 0.10 µg As)—Dilute 10.0 mL of arsenic intermediate solution to 1000 mL with water. Prepare fresh before each use.

21.4 *Hydrochloric Acid* (sp gr 1.19)—See 11.4.

21.5 *Nitric Acid* (sp gr 1.42)—See 11.6.

21.6 *Nitric Acid* (1 + 1)—See 11.7.

21.7 *Nitric Acid* (1 + 4)—Add 20 mL of nitric acid (sp gr 1.42) to 80 mL of water.

21.8 *Potassium Iodide Solution* (150 g/L)—See 11.8.

21.9 *Sodium Borohydride Solution* (4 g/100 mL)—Dissolve 4 g of sodium borohydride ($NaBH_4$) in 100 mL of water. Prepare fresh before each use.

21.10 *Stannous Chloride Solution* (400 g/L)—See 11.11.

21.11 *Sulfuric Acid* (1 + 1)—See 11.12.

21.12 *Zinc Metal* (*Dust*)*Suspension*—Add 10 g of zinc dust to 20 mL of water.

21.13 *Hydrogen*—Set burner control box to a gauge pressure of 8 psi (55 kPa) and adjust the flowmeter to approximately 6 L/min.

21.14 *Nitrogen or Argon*—Set burner control box to a gauge pressure of 30 psi (207 kPa) and adjust the flowmeter for maximum sensitivity by volatilizing standards. A flow of approximately 8 L/min has been found satisfactory for this purpose. This will depend on the burner used.

22. Standardization

22.1 Clean all glassware before use by rinsing first with hot HNO_3 (1 + 1) and then with water.

22.2 Prepare, in 200-mL Berzelius beakers or similar apparatus, a blank and sufficient standards containing from 0.0 to 1.0 µg of arsenic by diluting 0.0 to 10.0-mL portions of the arsenic standard solution to approximately 50 mL.

22.3 Proceed as directed in 23.1.3 – 23.1.8 or 23.2.3 – 23.2.7.

22.4 Prepare an analytical curve by plotting recorder scale readings versus micrograms of arsenic on linear graph paper. Alternatively, read directly in concentration if a concentration readout is provided with the instrument.

23. Procedure

23.1 *Determination of Arsenic with Zinc:*

23.1.1 Clean all glassware before use by rinsing first with hot HNO_3 (1 + 1) and then with water.

23.1.2 Pipette a volume of well-mixed acidified sample containing less than 1.0 µg of arsenic (50-mL maximum) into a 200-mL Berzelius beaker (or similar apparatus) and dilute to approximately 50 mL.

NOTE 8—If only dissolved arsenic is to be determined use a filtered and acidified sample (see 6.2).

23.1.3 To each beaker, add 7 mL of H_2SO_4 (1 + 1) and 5 mL of concentrated HNO_3 (sp gr 1.42). Add a small boiling chip and carefully evaporate to fumes of SO_3, maintaining an excess of HNO_3 until all organic matter is destroyed. This prevents darkening of the solution and possible reduction and loss of