INTERNATIONAL

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Standard Test Methods for Selenium in Water[1]

This standard is issued under the fixed designation D3859; 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 cover the determination of dissolved and total recoverable selenium in most waters and wastewaters. Both test methods utilize atomic absorption procedures, as follows:

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Test Method A—Gaseous Hydride AAS[2, 3]	7 – 16
Test Method B—Graphite Furnace AAS	17 – 26

1.2 These test methods are applicable to both inorganic and organic forms of dissolved selenium. They are applicable also to particulate forms of the element, provided that they are solubilized in the appropriate acid digestion step. However, certain selenium-containing heavy metallic sediments may not undergo digestion.

1.3 These test methods are most applicable within the following ranges:

Test Method A—Gaseous Hydride AAS[2, 3]	1 µg ⁄L to 20 µg ⁄L
Test Method B—Graphite Furnace AAS	2 µg ⁄L to 100 µg/L

These ranges may be extended (with a corresponding loss in precision) by decreasing the sample size or diluting the original sample, but concentrations much greater than the upper limits are more conveniently determined by flame atomic absorption spectrometry.

1.4 The values stated in SI units are to be regarded as standard. The values given in parentheses are mathematical conversions to inch-pound units that are provided for information only and are not considered standard.

1.5 *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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.* For specific hazard statements, see 11.12 and 13.14.

1.6 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[4]

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 Flowing Process Streams

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

D5673 Test Method for Elements in Water by Inductively Coupled Plasma—Mass Spectrometry

D5810 Guide for Spiking into Aqueous Samples

D5847 Practice for Writing Quality Control Specifications for Standard Test Methods for Water Analysis

3. Terminology

3.1 *Definitions:*

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 selenium, n*—a descriptive term relating to the selenium forms recovered in the acid-digestion procedure specified in these test methods.

4. Significance and Use

4.1 In most natural waters selenium concentrations seldom exceed 10 µg/L. However, the runoff from certain types of seleniferous soils at various times of the year can produce concentrations as high as several hundred micrograms per litre.

[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] Lansford, M., McPherson, E. M., and Fishman, M. J., *Atomic Absorption Newsletter*, Vol 13, No. 4, 1974, pp. 103–105.

[3] Pollack, E. N., and West, S. J., *Atomic Absorption Newsletter*, Vol 12, No. 1, 1973, pp. 6–8.

[4] 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.

Additionally, industrial contamination can be a significant source of selenium in rivers and streams.

4.2 High concentrations of selenium in drinking water have been suspected of being toxic to animal life. Selenium is a priority pollutant and all public water agencies are required to monitor its concentration.

4.3 These test methods determine the dominant species of selenium reportedly found in most natural and wastewaters, including selenities, selenates, and organo-selenium compounds.

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 specifications are available.[5] Other grades may be used, provided it is 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, reference 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 samples in accordance with Practices D3370. Take the samples in acid-washed TFE-fluorocarbon or glass bottles. Other types of bottles may be used for sampling, but should be checked for selenium absorption. The holding time for the samples may be calculated in accordance with Practice D4841.

6.2 When determining only dissolved selenium, filter the sample through a 0.45 µm membrane filter as soon as possible after sampling. Add HNO_3 to the filtrate to bring the pH to <2.0.

6.3 When determining total recoverable selenium, add HNO_3 to the unfiltered sample to a pH of <2.0 within 15 min of collecting the sample.

NOTE 1—Alternatively, the pH may be adjusted in the laboratory if the sample is returned within 14 days. However, acid must be added at least 24 h before analysis to dissolve any metals that adsorb to the container walls. This could reduce hazards of working with acids in the field when appropriate.

[5] *ACS Reagent Chemicals, Specifications and Procedures for Reagents and Standard-Grade Reference Materials*, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar 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.

TEST METHOD A—GASEOUS HYDRIDE AAS

7. Scope

7.1 This test method covers the determination of dissolved and total recoverable selenium in the range from 1 µg/L to 20 µg/L. The range may be extended by decreasing the sample size or diluting the original sample.

7.2 This test method has been used successfully with reagent water, natural water, wastewater, and brines. The information on precision may not apply to waters of other matrices.

8. Summary of Test Method

8.1 The determination consists of the conversion of selenium in its various forms to gaseous selenium hydride (hydrogen selenide), with the subsequent analysis of the gas by flame AAS.

8.1.1 The conversion consists of (*1*) decomposition and oxidation to selenium (VI), (*2*) reduction to selenium (IV), and (*3*) final reduction to selenium hydride.

8.1.2 The absorbance is determined at 196.0 nm in a hydrogen-argon (air-entrained) flame.

8.2 Sample concentrations are obtained directly from a simple concentration versus absorbance calibration curve.

8.3 Total recoverable selenium is determined by treating the entire sample as the procedure indicates, and the dissolved selenium is determined by treating the filtrate after the sample is filtered through a 0.45 µm membrane filter.

9. Interferences

9.1 Mercury and arsenic at concentrations greater than 500 µg/L and greater than 100 µg/L, respectively, may inhibit the formation of selenium hydride.

10. Apparatus

10.1 An apparatus similar to that depicted in Fig. 1, with the components specified in 10.2 – 10.4.8, is recommended for this test method.[6]

10.2 *Atomic Absorption Spectrophotometer*—The instruments shall consist of an atomizer and burner, suitable pressure and flow regulation devices capable of maintaining constant diluent and fuel pressure for the duration of the test, a selenium lamp, an optical system capable of isolating the desired wavelength, an adjustable slit, a photomultiplier tube or other photosensitive devices such as a light measuring and amplifying device, and a readout mechanism for indicating the amount of absorbed radiation. A background corrector may be used, but is not absolutely essential.

10.2.1 *Selenium Electrodeless Discharge Lamp*—The sensitivity of selenium to atomic absorption spectroscopy is generally improved with this lamp, although some hollow-cathode lamps produce equivalent results. The intensity and stability of

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

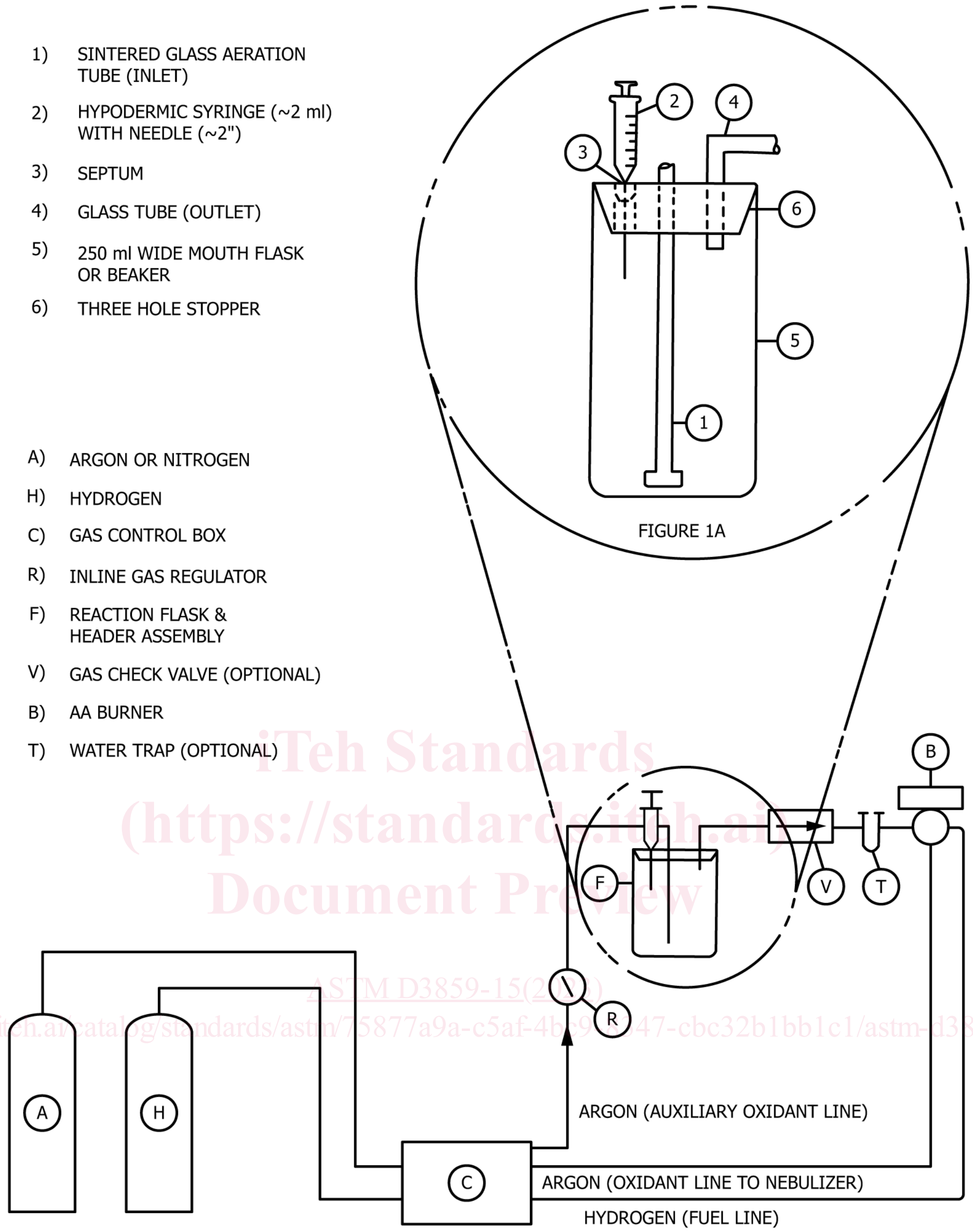

FIG. 1 Apparatus for Selenium Determination

the lamp shall be adequate to determine selenium in the range from 1 µg/L to 20 µg/L.

10.2.2 *Recorder or Digital Readout, or Both*—Any multirange, variable-speed recorder, or digital readout accessory that is compatible with the atomic absorption detection system, is suitable.

10.2.3 The manufacturer's instructions are to be followed for all instrument parameters.

10.3 *Gas System:*

10.3.1 See 11.14 for materials for the gas system.

10.3.2 *Pressure-Reducing Valves*—Pressure-reducing valves shall be capable of maintaining argon pressure at 275 kPa (40 psi) and hydrogen pressure at 138 kPa (20 psi).

10.4 *Additional Equipment:*

10.4.1 *Flask Header*—The flask header shall consist of a three-hole rubber stopper into which is inserted:

10.4.1.1 A sintered-glass aeration tube for the argon sweep gas,

10.4.1.2 A small gas chromatographic-type septum (5 mm to 10 mm in diameter), for injection of the borohydride solution, and

10.4.1.3 A glass outlet tube for the reaction gases to exit.

NOTE 2—Instead of the gas chromatographic-type septum, a more secure seal may be obtained by using a glass tube with a septum cap. These items are commercially available on an individual basis. A different header may be used if proven reliable.

10.4.2 *Fittings and Adapters*—Stainless steel fittings and adapters shall be used to install the reaction-flask header in series with the auxiliary oxidant line and the burner. Plastic or other metals may be substituted if proven acceptable.

10.4.3 *Tubing*—Any commercially available plastic tubing that is not susceptible to attack by hydrochloric acid, selenium hydride, or other gases from the reaction mixture is acceptable. Poly(vinyl chloride) tubing has been found acceptable.

10.4.4 *Gas-Flow Regulator*—A suitable in-line gas-flow valve shall be used to adjust the flow of argon to the reaction-flask header.

10.4.5 *Water Trap (optional)*—Any commercially available glass trap suitable to prevent carryover moisture from going to the burner is acceptable.

10.4.6 *One-Way Gas Check Valve (optional)*—A one-way check valve can be installed in series with the water trap and burner to prevent hydrogen from back flowing to the generating flask whenever samples are changed. However, precautionary measures could generally preclude the use of this device, since only when the flask header is removed for prolonged periods would there be significant hydrogen back flow.

10.4.7 *Reaction Flasks,* 250 mL spoutless beakers, or their equivalent, with graduations may be used. Conical and restricted neck flasks do not perform as reliably as spoutless beakers.

10.4.8 *Hypodermic Syringe,* 2 mL capacity with a 50 mm needle.

11. Reagents and Materials

11.1 *Calcium Chloride Solution* (30 g/L)—Commercially purchase or dissolve 30 g of calcium chloride ($CaCl_2 \cdot 2H_2O$) in water and dilute to 1 L.

11.2 *Hydrochloric Acid* (sp gr 1.19), concentrated hydrochloric acid (HCl).

11.3 *Hydrochloric Acid* (1 + 1)—Add 1 volume of HCl (sp gr 1.19) to 1 volume of water. Always add acid to water.

11.4 *Hydrochloric Acid* (1 + 99)—Add 1 volume of HCl (sp gr 1.19) to 99 volumes of water. Always add acid to water.

11.5 *Methyl Orange Indicator Solution* (25 mg/100 mL)—Dissolve 25 mg of methyl orange in 100 mL of water.

11.6 *Nitric Acid* (sp gr 1.42), concentrated nitric acid (HNO_3).

11.7 *Nitric Acid* (1 + 99)—Add 1 volume of HNO_3 (sp gr 1.42) to 99 volumes of water.

11.8 *Potassium Permanganate Solution* (0.3 g/L)—Dissolve 0.3 g of potassium permanganate ($KMnO_4$) in water and dilute to 1 L.

11.9 *Selenium Solution, Stock* (1.00 mL = 1.00 mg selenium)—Accurately weigh 1.000 g of gray elemental selenium and place in a small beaker. Add 5 mL of HNO_3 (sp gr 1.42). Warm until the reaction is complete, then cautiously evaporate to dryness. Redissolve with HCl (1 + 99) and dilute to 1 L with the same acid solution.

11.9.1 A purchased metal selenium stock solution of appropriate known purity is also acceptable.

11.10 *Selenium Solution, Intermediate* (1.00 mL = 10 µg selenium)—Dilute 5 mL of the selenium stock solution to 500 mL with HCl (1 + 99).

11.11 *Selenium Solution, Standard* (1.00 mL = 0.10 µg selenium)—Dilute 10 mL of the selenium intermediate solution to 1000 mL with HCl (1 + 99). Prepare fresh daily and store in a TFE-fluorocarbon or other acceptable container. To minimize waste only prepare 100 mL of the Selenium Standard Solution.

11.12 *Sodium Borohydride Solution* (4 g/100 mL)—Dissolve 4 g of sodium borohydride ($NaBH_4$) and 2 g of sodium hydroxide in water and dilute to 100 mL. Prepare fresh weekly. (**Warning—**Sodium borohydride reacts strongly with acids.)

11.13 *Sodium Hydroxide Solution* (4 g/L)—Dissolve 4 g of sodium hydroxide (NaOH) in water and dilute to 1 L.

11.14 *Gases:*

11.14.1 *Argon* (nitrogen may be used in place of argon)—Standard, commercially available argon is the usual diluent.

11.14.2 *Hydrogen*—Standard, commercially available hydrogen is the usual fuel.

12. Standardization

12.1 Transfer 0.0 mL, 0.5 mL, 1.0 mL, 2.0 mL, 5.0 mL, and 10.0 mL portions of the standard selenium solution (1.0 mL = 0.10 µg Se) (11.11) to freshly washed 250 mL reaction flasks. Adjust the volume to 50 mL with water. Analyze at least six working standards containing concentrations of selenium that bracket the expected sample concentration, prior to analysis of samples, to calibrate the instrument.

12.2 Proceed as directed in 13.3 – 13.15.

12.3 Calibrate the spectrophotometer to output micrograms of selenium directly, if provided with this capability or prepare a calibration curve by plotting absorbance (or recorder scale readings) versus micrograms of selenium on linear graph paper.

13. Procedure

13.1 It is emphasized that careful control of pH, oxidant concentration, temperature, and time are imperative if accurate and precise selenium determinations are to be obtained.

13.2 For each sample, transfer 50 mL or less (to contain not more than 1.0 µg selenium) to a freshly washed 250 mL reaction flask. Make up to 50 mL with water if necessary.

13.3 To each sample, standard, and blank, add a few drops of methyl orange solution (11.5), 0.5 mL of $CaCl_2$ solution (11.1) and three or four boiling stones.

13.4 Adjust the pH to the red end point of methyl orange (pH = 3.1) with HCl (1 + 99) (11.4) or NaOH solution (4 g/L) (11.13). Add 0.5 mL of HCl (1 + 99) in excess. A pH meter may be used in place of the indicator if the sample is sufficiently discolored to affect the methyl orange end point.

13.5 Add potassium permanganate solution (11.8) dropwise (about 3 drops) to maintain the purple tint indicating excess $KMnO_4$. Boil the solution on a hotplate, carefully maintaining the purple tint until the volume is reduced to about 25 mL. Add 2 mL of NaOH solution (4 g/L) (11.13) and concentrate the solutions to dryness, being careful not to overheat the residue.

13.6 Cool and add 15 mL of concentrated HCl (sp gr 1.19) (11.2). Heat on a hot water or steam bath for 20 min. Do not boil. This step reduces the selenium (VI) to selenium (IV).

NOTE 3—Many laboratories have found block digestion systems a useful way to digest samples for trace metals analysis. Systems typically consist of either a metal or graphite block with wells to hold digestion tubes. The block temperature controller must be able to maintain uniformity of temperature across all positions of the block. For trace metals analysis, the digestion tubes should be constructed of polypropylene and have a volume accuracy of at least 0.5 %. All lots of tubes should come with a certificate of analysis to demonstrate suitability for their intended purpose.

13.7 Cool and add HCl (1 + 1) (11.3) to adjust the volume to 50 mL. Hold these solutions until all samples and standards are brought to this stage.

13.8 Set the atomic absorption instrument parameters in accordance with the manufacturer's instructions. Typical settings are as follows:

Grating	ultraviolet
Wavelength	196.0 nm
Burner	triple-slot or equivalent
Radiation Source	selenium electrodeless discharge lamp or equivalent
Slit	2.0 nm
Flame	hydrogen-argon (nitrogen may be used in place of argon)

13.9 If the gas control box is not equipped with separate controls for argon and hydrogen, simply connect the oxidant inlet line for the control box to the argon tank regulator and connect the fuel inlet line for the control box to the hydrogen tank regulator. The oxidant controls will then control the argon diluent gas and the fuel controls will control the hydrogen gas. To preclude the possibility of accidentally mixing the hydrogen fuel with the air oxidant normally used with atomic absorption spectroscopy, shut off all sources of air oxidant to the system. Set the tank pressures, the burner control box pressures, and the flow rates in accordance with the manufacturer's instructions for argon and hydrogen.

13.10 Center the burner about 5 mm below the optical light path. Ignite the flame. Since the flame does not give off visible light, optical flame sensors must be bypassed, but the presence of the low-temperature flame may be verified by aspirating tap water, which contains soluble salts that impart color to the flame. Optimize the burner position to give maximum absorbance while aspirating the intermediate selenium standard (1.0 mL = 10 µg selenium).

13.11 Interrupt the auxiliary oxidant line at the burner connection and attach the gas lines, the flask header, and the associated equipment. Connect in series, in this order, the auxiliary oxidant line, the in-line gas flow regulator, and the header aeration tube. Then connect the header outlet tube, the water trap (optional), the one-way check valve (optional), and the auxiliary oxidant inlet. Use minimum lengths of tubing to minimize dilution of the selenium hydride. Attach a reaction flask containing 50 mL of water to the flask header. With argon flowing through the system, adjust the in-line flow regulator to permit a maximum flow of the argon sweep gas to the reaction flask, with negligible solution carryover into the outlet line. The set-up is then complete.

13.12 If a recorder is used, adjust the span so that an absorbance of 0.500 from the spectrophotometer reads full scale on the recorder.

13.13 Rinse the reaction flask and header with water and introduce the blank, sample, or standard into the reaction flask. Replace the header and secure to form a tight seal. Allow the system to stabilize and prepare to record the peak absorbance or the total absorbance.

13.14 The precision of this test method is highly dependent on the use of a consistently reproduced technique in this final step. Inject the 2 mL hypodermic needle through the septum and quickly add 2.0 mL $NaBH_4$ solution (4 g/100 mL) (11.12) to the sample. The H_2Se evolution will peak within a few seconds, but will trail off for up to 30 s afterward. After the H_2Se is swept from the system, remove the header and rinse well with water. (**Warning—**Selenium hydride is toxic to certain organs of the body. Avoid inhalation.)

13.15 Treat each succeeding sample, blank, and standard in a like manner.

14. Calculation

14.1 Determine the weight of selenium in each sample by referring to 12.3. Calculate the concentration of selenium in the sample in micrograms per litre, using Eq 1:

$$\text{Selenium } \mu g/L = (1000/V) \times W \quad (1)$$

where:

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

15. Precision and Bias[7]

15.1 The overall and single-operator precision of this test method within its designated range for reagent water and nonreagent water varies with the quantity being measured in accordance with Table 1. These values were established for four laboratories, using six operators over three consecutive days. The nonreagent waters included natural, waste, and brine waters.

[7] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D19-1056. Contact ASTM Customer Service at service@astm.org.

TABLE 1 Overall (S_T) and Single-Operator (S_O) Interlaboratory Precision for Selenium by Gaseous Hydride AAS, Test Method A

Concentration (*X*), µg/L	S_T	S_O
	Reagent Water	
2.79	0.95	0.72
8.50	1.62	1.44
17.89	2.98	1.71
	Natural Water	
2.69	0.69	0.78
8.56	1.70	1.63
18.35	2.13	1.67

15.1.1 The overall precision for reagent water varies linearly with the quantity being measured, and it may be expressed mathematically using Eq 2:

$$S_t = 0.146X + 0.49 \tag{2}$$

where:

S_t = overall precision, µg/L, and
X = concentration of selenium, µg/L.

15.2 The bias of this test method determined from recoveries of known amounts of selenium from selenium dioxide and selenium triphenylchloride in a series of prepared standards are given in Table 2.

15.3 The information on precision and bias may not apply to other wastewaters.

15.4 This section on precision and bias conforms to Practice D2777 – 77 which was in place at the time of collaborative testing. Under the allowances made in 1.4 of Practice D2777 – 13, these precision and bias data do meet existing requirements of 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 selenium.

16.2 *Calibration and Calibration Verification:*

16.2.1 Analyze at least six working standards containing concentrations of selenium that bracket the expected sample concentration, prior to analysis of samples, to calibrate the instrument (see 12.1). The calibration correlation coefficient shall be equal to or greater than 0.990.

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. Analyze a calibration blank to verify system cleanliness.

16.2.3 If calibration cannot be verified, recalibrate the instrument.

TABLE 2 Recovery and Bias Data, Test Method A (Gaseous Hydride AAS)

Amount Added, µg/L	Amount Found, µg/L	Recovery, %	Bias, %	Statistically Significant at 95 % Confidence Level
Reagent Water (Type II)				
3	2.8	93	−7	no
8	8.5	106	+ 6	no
17	17.9	105	+ 5	no
Nonreagent Water (Natural, Waste, and Brine)				
3	2.7	90	−10	no
8	8.6	107	+ 7	no
17	18.4	108	+ 8	yes

16.2.4 It is recommended to analyze a continuing calibration blank (CCB) and continuing calibration verification (CCV) at a 10 % frequency. The results should fall within the expected precision of the method or ±15 % of the known concentration.

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, and so forth, 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 midrange concentration of selenium. 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 2. 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, prepare and analyze a LCS containing a known concentration of selenium with each batch (laboratory-defined or twenty samples). The laboratory control samples for a large batch should cover the analytical range when possible. It is recommended, but not required to use a second source, if possible and practical for the LCS. 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 laboratory-defined batch. The concentration of selenium found in the blank should be less than 0.5 times the lowest calibration standard. If the concentration of selenium 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 laboratory-defined batch by spiking an aliquot of the sample