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An American National Standard

Standard Test Methods for Iron in Water[1]

This standard is issued under the fixed designation D1068; 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.

This standard has been approved for use by agencies of the Department of Defense.

ε[1] NOTE—Warning notes were moved into the text editorially in July 2005.

1. Scope

1.1 These test methods cover the determination of iron in water. Procedures are given for determining total iron, dissolved iron, and ferrous iron. Undissolved iron may be calculated from the total iron and dissolved iron determinations. The test methods are given as follows:

	Range	Sections
Test Method A—Atomic Absorption, Direct	0.1 to 5.0 mg/L	7 to 15
Test Method C—Atomic Absorption, Graphite Furnace	5 to 100 µg/L	16 to 24
Test Method D—Photometric Bathophenanthrolineµ g/L	40 to 1000 µg/L	25 to 36

1.2 It is the user's responsibility to ensure the validity of these test methods to waters of untested matrices.

1.3 *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.* Specific hazards statements are given in Note 3, section 11.7.1, and section X1.1.2.

1.4 Two former photometric test methods were discontinued. See Appendix X2 for historical information.

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D858 Test Methods for Manganese in Water
D1066 Practice for Sampling Steam
D1129 Terminology Relating to Water
D1192 Guide for Equipment for Sampling Water and Steam in Closed Conduits[3]
D1193 Specification for Reagent Water
D1687 Test Methods for Chromium in Water
D1688 Test Methods for Copper in Water
D1691 Test Methods for Zinc in Water
D1886 Test Methods for Nickel in 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
D3558 Test Methods for Cobalt in Water
D3559 Test Methods for Lead in Water
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 Molecular Absorption Spectrometry
E275 Practice for Describing and Measuring Performance of Ultraviolet and Visible Spectrophotometers

3. Terminology

3.1 *Definitions:* 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 iron*—an arbitrary analytical term relating to the recoverable forms of iron that are determinable by the digestion method which is included in these test methods.

4. Significance and Use

4.1 Iron is the second most abundant metallic element in the earth's crust and is essential in the metabolism of plants and

[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] 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.

[3] Withdrawn. The last approved version of this historical standard is referenced on www.astm.org.

animals. If presented in excessive amounts, however, it forms oxyhydroxide precipitates that stain laundry and porcelain. As a result, the recommended limit for iron in domestic water supplies is 0.3 mg/L. These test methods are useful for determining iron in many natural waters.

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.[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 these test methods. In addition, water used in preparing solutions for the determination of ferrous iron shall be freshly boiled and essentially oxygen free.

6. Sampling

6.1 Collect the sample in accordance with Practice D1066, Specification D1192, or Practices D3370, as applicable.

6.2 Samples should be preserved with HNO_3 or HCl (sp gr 1.42) to a pH of 2 or less immediately at the time of collection. If only dissolved iron is to be determined, the sample shall be filtered through a 0.45-µm membrane filter before acidification. The holding time for samples can be calculated in accordance with Practice D4841.

6.3 If ferrous iron is to be determined, the sample should be analyzed as soon as possible after collection and contact with atmospheric oxygen should be minimized.

6.4 Additional information on sampling requirements for Test Method D is provided in 34.1.

TEST METHOD A—ATOMIC ABSORPTION, DIRECT

7. Scope

7.1 This test method covers the determination of dissolved and total recoverable iron in most waters and wastewaters.

7.2 This test method is applicable in the range from 0.1 to 5.0 mg/L of iron. The range may be extended to concentrations greater than 5.0 mg/L by dilution of the sample.

7.3 This test method has been used successfully with reagent water; tap, ground, and surface waters; unspecified wastewaters; and a refinery primary treatment water. 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 Iron is determined by atomic absorption spectrophotometry. Dissolved iron is determined by atomizing the filtered sample directly with no pretreatment. Total recoverable iron is determined by atomizing the sample following hydrochloric-nitric acid digestion and filtration. The same digestion procedure may be used to determine total recoverable nickel (Test Methods D1886), chromium (Test Methods D1687), cobalt (Test Methods D3558), copper (Test Methods D1688), lead (Test Methods D3559), manganese (Test Methods D858), and zinc (Test Methods D1691).

9. Interferences

9.1 Sodium, potassium, barium, chloride and sulfate (5000 mg/L each), calcium, magnesium, chromium, manganese, cobalt, nickel, copper, zinc, palladium, silver, cadmium, tin, lead, lithium, mercury, selenium, aluminum, antimony, arsenic, vanadium, boron, and molybdenum (100 mg/L) do not interfere.

9.2 Background correction (or chelation-extraction) may be necessary to determine low levels of iron in some waters.

NOTE 1—Instrument manufacturers' instructions for use of the specific correction technique should be followed.

10. Apparatus

10.1 *Atomic Absorption Spectrophotometer*, for use at 248.3 nm.

NOTE 2—The manufacturer's instructions should be followed for all instrumental parameters. A wavelength other than 248.3 nm may be used if it has been determined to be equally suitable.

10.1.1 *Iron Hollow-Cathode Lamp*—Multielement hollow-cathode lamps are available and have also been found satisfactory.

10.2 *Pressure-Reducing Valves*—The supplies of fuel and oxidant shall be maintained at pressures somewhat higher than the controlled operating pressure of the instrument by suitable valves.

11. Reagents and Materials

11.1 *Hydrochloric Acid (sp gr 1.19)*—Concentrated hydrochloric acid (HCl).

NOTE 3—If the reagent blank concentration is greater than the method detection limit, distill the HCl or use a spectrograde acid. **Precaution**—When HCl is distilled an azeotropic mixture is obtained (approximately 6 *N* HCl). Therefore, when concentrated HCl is specified for the preparation of reagents or in the procedure, use double the volume specified if distilled acid is used.

11.2 *Nitric Acid (sp gr 1.42)*—Concentrated nitric acid (HNO_3).

NOTE 4—If the reagent blank concentration is greater than the method detection limit, distill the HNO_3 or use a spectrograde acid.

11.3 *Nitric Acid (1 + 499)*—Add 1 volume of HNO_3(sp gr 1.42) to 499 volumes of water.

[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 *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmaceutical Convention, Inc. (USPC), Rockville, MD.

11.4 *Iron Solution, Stock (1 mL = 1.0 mg Iron)*—Dissolve 1.000 g of pure iron in 100 mL of HCL (1 + 1) with the aid of heat. Cool and dilute to 1 L with water.

11.5 *Iron Solution, Standard (1 mL = 0.1 mg Iron)*—Dilute 100.0 mL of the iron stock solution to 1 L with water.

11.6 *Oxidant*:

11.6.1 *Air*, which has been passed through a suitable filter to remove oil, water, and other foreign substances is the usual oxidant.

11.7 *Fuel*:

11.7.1 *Acetylene*—Standard, commercially available acetylene is the usual fuel. Acetone, always present in acetylene cylinders can affect analytical results. The cylinder should be replaced at 50 psig (345 kPa). (**Warning—**"Purified" grade acetylene containing a special proprietary solvent rather than acetone should not be used with poly vinyl chloride tubing as weakening of the tubing walls can cause a potentially hazardous situation.)

12. Standardization

12.1 Prepare 100 mL each of a blank and at least four standard solutions to bracket the expected iron concentration range of the samples to be analyzed by diluting the standard iron solution with HNO_3 (1 + 499). Prepare the standards each time the test is to be performed.

12.2 When determining total recoverable iron add 0.5 mL of HNO_3 (sp gr 1.42) and proceed as directed in 13.1 through 13.5. When determining dissolved iron proceed as directed in Note 5, 13.1.

12.3 Aspirate the blank and standards and record the instrument readings. Aspirate HNO_3 (1 + 499) between each standard.

12.4 Prepare an analytical curve by plotting the absorbance versus concentration for each standard on linear graph paper. Alternatively read directly in concentration if this capability is provided with the instrument.

13. Procedure

13.1 Measure 100.0 mL of a well-mixed acidified sample into a 125-mL beaker or flask.

NOTE 5—If only dissolved iron is to be determined, start with 13.5.

13.2 Add 5 mL of HCl (sp gr 1.19) to each sample.

13.3 Heat the samples on a steam bath or hotplate in a well-ventilated hood until the volume has been reduced to 15 to 20 mL, making certain that the samples do not boil.

NOTE 6—When analyzing samples of brines or samples containing appreciable amounts of suspended matter or dissolved solids, the amount of reduction in volume is left to the discretion of the analyst.

13.4 Cool and filter the samples through a suitable filter (such as fine-textured, acid-washed, ashless paper), into 100-mL volumetric flasks. Wash the filter paper two or three times with water and adjust a volume.

13.5 Aspirate each filtered and acidified sample and determine its absorbance or concentration at 248.3 nm. Aspirate HNO_3 (1 + 499) between each sample.

TABLE 1 Determination of Bias, Atomic Absorption, Direct

Amount Added, mg/L	Amount Found, mg/L	Bias, mg/L	Bias, %	Statistically Significant (95 % Confidence Level)
Reagent Water Type II:				
0.2	0.2	±0.0	0.0	no
2.4	2.4	±0.0	0.0	no
4.4	4.3	−0.1	− 2.3	yes
Natural Water:				
0.2	0.2	±0.0	0	no
2.4	2.3	− 0.1	− 4.17	yes
4.4	4.2	− 0.2	− 4.55	yes

14. Calculation

14.1 Calculate the concentration of iron in the sample, in milligrams per litre, referring to 12.4.

15. Precision and Bias [5]

15.1 The precision of this test method for 10 laboratories, which include 16 operations within its designated range may be expressed as follows:

Reagent Water Type II:

$$S_T = 0.047\,X + 0.053$$

$$S_o = 0.030\,X + 0.037$$

Water of Choice:

$$S_T = 0.050\,X + 0.114$$

$$S_o = 0.024\,X + 0.078$$

where:
S_T = overall precision,
S_o = single-operator precision, and
X = determined concentration of iron, mg/L.

15.2 Recoveries of known amounts of iron in a series of prepared standards were as shown in Table 1.

15.3 The collaborative test data were obtained on reagent water; tap, lake, ground and surface water; unspecified wastewater; and a refinery primary treatment water. It is the user's responsibility to ensure the validity of this test method for waters of untested matrices.

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 – 98, 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

[5] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D19-1035.

confidence limits of the test, the following QC procedures must be followed when analyzing iron.

16.2 *Calibration and Calibration Verification*:

16.2.1 Analyze at least three working standards containing concentrations of iron 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 iron. 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. The replicates may be interspersed with samples.

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 iron 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 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 iron found in the blank should be less than 0.5 times the lowest calibration standard. If the known concentration of iron 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 known concentration of iron and taking it through the analytical method.

16.6.2 The spike known concentration plus the background known concentration of iron 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:

$$P = 100\,[A(V_s + V) - B\,V_s] / C\,V \tag{1}$$

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) added with spike.

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 7—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 C—ATOMIC ABSORPTION, GRAPHITE FURNACE

17. Scope

17.1 This test method covers the determination of dissolved and total recoverable iron in most waters and wastewaters.

17.2 This test method is applicable in the range from 5 to 100 µg/L of iron using a 20-µL injection. The range can be increased or decreased by varying the volume of sample injected or the instrumental settings. High concentrations may be diluted but preferably should be analyzed by direct aspiration atomic absorption spectrophotometry (Test Method A).

17.3 This test method has been used successfully with reagent grade water, filtered tap water, well water, demineralized water, boiler blowdown water, and condensate from a medium Btu-coal gasification process. It is the user's responsibility to ensure validity of this test method to waters of untested matrices.

17.4 The analyst is encouraged to consult Practice D3919 for a general discussion of interferences and sample analysis procedures for graphite furnace atomic absorption spectrophotometry.

18. Summary of Test Method

18.1 Iron is determined by an atomic absorption spectrophotometer used in conjunction with a graphite furnace. A sample is placed in a graphite tube, evaporated to dryness, charred (pyrolyzed or ashed), and atomized. The absorption signal generated during atomization is recorded and compared to standards. A general guide for the application of the graphite furnace is given in Practice D3919.

18.2 Dissolved iron is determined on a filtered sample with no pretreatment.

18.3 Total recoverable iron is determined following acid digestion and filtration. Because chlorides interfere with furnace procedures for some metals, the use of hydrochloric acid in any digestion or solubilization step is to be avoided. If suspended material is not present, this digestion and filtration may be omitted.

19. Interferences

19.1 For a complete discussion on general interferences with furnace procedures, the analyst is referred to Practice D3919.

20. Apparatus

20.1 *Atomic Absorption Spectrophotometer*, for use at 248.3 nm with background correction.

NOTE 8—A wavelength other than 248.3 nm may be used if it has been determined to be suitable. Greater linearity may be obtained at high concentrations by using a less sensitive wavelength.

NOTE 9—The manufacturer's instructions should be followed for all instrumental parameters.

20.2 *Iron Hollow-Cathode Lamp*—A single-element lamp is preferred, but multielement lamps may be used.

20.3 *Graphite Furnace*, capable of reaching temperatures sufficient to atomize the element of interest.

20.4 *Graphite Tubes*, compatible with furnace device. Pyrolytically coated graphite tubes are recommended to eliminate the possible formation of carbides.

20.5 *Pipets*, microlitre with disposable tips. Sizes may range from 1 to 100 µL, as required.

20.6 *Data Storage and Reduction Devices, Computer- and Microprocessor-Controlled Devices, or Strip Chart Recorders*, shall be utilized for collection, storage, reduction, and problem recognition (such as drift, incomplete atomization, changes in sensitivity, etc.). Strip chart recorders shall have a full scale deflection time of 0.2 s or less to ensure accuracy.

20.7 Automatic sampling should be used if available.

NOTE 10—Manual injection has been reported to cause widely scattered values even on purified waters due to contamination from pipetting technique.

21. Reagents and Materials

21.1 *Iron Solution, Stock (1.0 mL = 1000 µg Fe)*—See 11.4.

21.2 *Iron Solution, Intermediate (1.0 mL = 10 µg Fe)*—Dilute 10.0 mL of iron solution, stock (20.1) and 1 mL of HNO_3(sp gr 1.42) to 1 L with water.

21.3 *Iron Solution, Standard (1.0 mL = 0.2 µg Fe)*—Dilute 20.0 mL of iron solution, intermediate (20.2) and 1 mL of HNO_3(sp gr 1.42) to 1 L water. This standard is used to prepare working standards at the time of the analysis.

21.4 *Nitric Acid (sp gr 1.42)*—Concentrated nitric acid (HNO_3) (see Note 4).

21.5 *Argon, Standard*, welders grade, commercially available. Nitrogen may also be used if recommended by the instrument manufacturer.

22. Standardization

22.1 Initially, set the instrument according to the manufacturer's specifications. Follow the general instructions as provided in Practice D3919.

23. Procedure

23.1 Clean all glassware to be used for preparation of standard solutions or in the solubilization step, or both, by rinsing first with HNO_3(1 + 1) and then with water.

23.2 Measure 100.0 mL of each standard and well-mixed sample into 125-mL beakers or flasks. For total recoverable iron add HNO_3(sp gr 1.42) to each standard and sample at a rate of 5 mL/L and proceed as directed in 23.4 through 23.6.

23.3 If only dissolved iron is to be determined, filter the sample through a 0.45-µm membrane filter prior to acidification and proceed to 23.6.

23.4 Heat the samples at 95°C on a steam bath or hotplate in a well-ventilated fume hood until the volume has been reduced to 15 to 20 mL, making certain that the samples do not boil (see Note 6).

23.5 Cool and filter the sample through a suitable filter (such as fine-textured, acid-washed, ashless paper) into a

TABLE 2 Determination of Bias, Atomic Absorption, Graphite Furnace

Reagent Water: Amount Added, µg/L	Amount Found, µg/L	S_T	± Bias, µg/L	± % Bias	Statistically Significant
8.0	11.3	6.18	+ 3.3	+ 41.3	no
20	21.1	12.35	+ 1.1	+ 5.5	no
68	67.1	30.62	– 0.9	– 1.3	no
Natural Water: Amount Added, µg/L	**Amount Found, µg/L**	**S_T**	**± Bias, µg/L**	**± % Bias**	**Statistically Significant**
8.0	6.9	3.17	–1.1	–13.8	no
20	19.0	8.33	–1.0	–5.0	no
68	70.1	21.63	+ 2.1	+ 3.1	no

100-mL volumetric flask. Wash the filter paper 2 or 3 times with water and bring to volume (Note 11). The acid concentration at this point should be 0.5 % HNO_3.

NOTE 11—If suspended material is not present, this filtration may be omitted. The sample must be diluted to 100 mL.

23.6 Inject a measured aliquot of sample into the furnace device following the directions as provided by the particular instrument manufacturer. Refer to Practice D3919.

24. Calculation

24.1 Determine the concentration of iron in each sample by referring to Practice D3919.

25. Precision and Bias [6]

25.1 The precision for this test method was developed by 13 laboratories using reagent water and 7 laboratories using tap water, filtered tap water, well water, demineralized water, boiler blowdown water, and condensate from a medium Btu coal gasification process. Although multiple injections may have been made, the report sheets provided allowed only for reporting single values. Thus, no single-operator precision data can be calculated. See Table 2 for bias data and overall precision data.

25.2 These data may not apply to waters of other matrices, therefore, it is the responsibility of the analyst to ensure the validity of this test method in a particular matrix.

25.3 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 – 98, these precision and bias data do meet existing requirements of interlaboratory studies of Committee D19 test methods.

26. Quality Control

26.1 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 iron.

26.2 *Calibration and Calibration Verification*:

26.2.1 Analyze at least three working standards containing known concentrations of iron that bracket the expected sample known 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.

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

26.2.3 If calibration cannot be verified, recalibrate the instrument.

26.3 *Initial Demonstration of Laboratory Capability*:

26.3.1 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.

26.3.2 Analyze seven replicates of a standard solution prepared from an Independent Reference Material containing a mid-range known concentration of iron. 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. The replicates may be interspersed with samples.

26.3.3 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 2. If a known concentration other than the recommended known 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.

26.4 *Laboratory Control Sample (LCS)*:

26.4.1 To ensure that the test method is in control, analyze a LCS containing a known concentration of iron 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 known concentration.

26.4.2 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.

26.5 *Method Blank*:

26.5.1 Analyze a reagent water test blank with each batch. The known concentration of iron found in the blank should be less than 0.5 times the lowest calibration standard. If the known concentration of iron is found above this level, analysis of

[6] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D19-1102.