Designation: D4922 – 09 (Reapproved 2016)$^{\varepsilon 1}$

Standard Test Method for Determination of Radioactive Iron in Water[1]

This standard is issued under the fixed designation D4922; 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.

ε^1 NOTE—Editorial corrections were made to 5.1 in January 2016.

1. Scope

1.1 This test method covers the determination of ^{55}Fe in the presence of ^{59}Fe by liquid scintillation counting. The *a-priori* minimum detectable concentration for this test method is 7.4 Bq/L.[2]

1.2 This test method was developed principally for the quantitative determination of ^{55}Fe. However, after proper calibration of the liquid scintillation counter with reference standards of each nuclide, ^{59}Fe may also be quantified.

1.3 This test method was used successfully with Type III reagent water conforming to Specification D1193. It is the responsibility of the user to ensure the validity of this test method for waters of untested matrices.

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 a specific hazard statement, see Section 9.

2. Referenced Documents

2.1 *ASTM Standards:*[3]

D1068 Test Methods for Iron in Water
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
D5847 Practice for Writing Quality Control Specifications for Standard Test Methods for Water Analysis
D7282 Practice for Set-up, Calibration, and Quality Control of Instruments Used for Radioactivity Measurements

3. Terminology

3.1 *Definitions*—For definitions of terms used in this test method, refer to Terminology D1129. For terms not defined in this test method or in Terminology D1129, refer to other published glossaries.[4]

4. Summary of Test Method

4.1 This test method describes the effective separation of iron from the interfering cations of manganese, cobalt, zirconium, niobium, and cesium by anion exchange using acid washes of various molarities. Subsequent elution of the iron is followed by phosphate precipitation to remove any residual zinc. The iron phosphate precipitate is dissolved in phosphoric acid and water and mixed with liquid scintillation cocktail. The chemical yield is determined by the recovery of iron carrier using atomic absorption spectrophotometry. Alternatively, any procedure described in Test Methods D1068 may be used, but this will need to be validated by the user prior to reporting sample results.

5. Significance and Use

5.1 Fe-55 is formed in reactor coolant systems of nuclear reactors by activation of stable iron. The ^{55}Fe is not completely removed by waste processing systems and some is released to the environment by means of normal waste liquid discharges. Power plants are required to monitor these discharges for ^{55}Fe as well as other radionuclides.

5.2 This technique effectively removes other activation and fission products such as isotopes of iodine, zinc, manganese, cobalt, and cesium by the addition of hold-back carriers and an anion exchange technique. The fission products (zirconium-95 and niobium-95) are selectively eluted with hydrochloric-hydrofluoric acid washes. The iron is finally separated from Zn^{+2} by precipitation of $FePO_4$ at a pH of 3.0.

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[1] This test method is under the jurisdiction of ASTM Committee D19 on Water and is the direct responsibility of Subcommittee D19.04 on Methods of Radiochemical Analysis.

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[2] Currie, L., "Limits for Qualitative Detection and Quantitative Determination," *Analytical Chemistry*, Vol. 40, 1968, pp. 586–593.

[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.

[4] "American National Standard Glossary of Terms," *Nuclear Science and Technology (ANSI N1.1)*, American National Standards Institute, 1430 Broadway, New York, NY 10018.

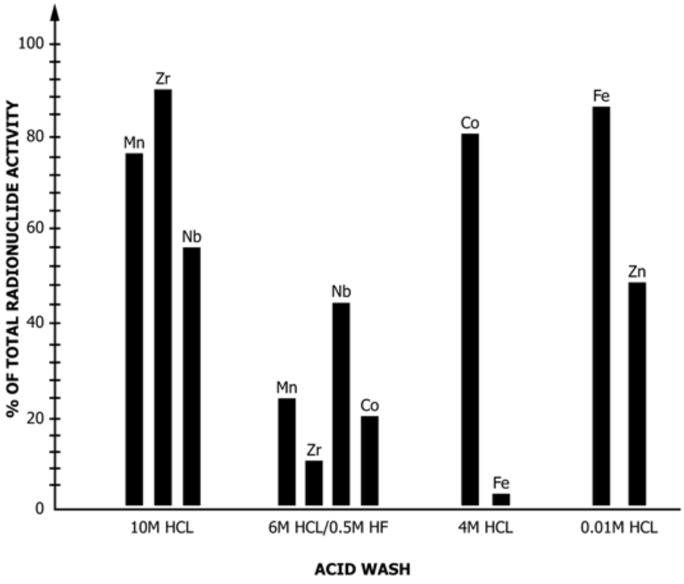

FIG. 1 Percent of Total Radionuclide Activity Removed Per Acid Wash

6. Interferences

6.1 Samples of reactor origin will also contain ^{59}Fe after other radioactive contaminants have been removed by anion exchange (see Fig. 1). ^{59}Fe is also an activation product which decays by β-γ emission and will be a source of interference in the quantitative determination of ^{55}Fe. The large difference in the energies of their characteristic decay emissions makes it possible to determine appropriate factors to correct for the ^{59}Fe spectral cross-talk in the ^{55}Fe region.

6.2 Quenching, which may be caused by a number of factors, results in a reduction in light output from the sample. The subsequent decrease in the spectral pulse height will cause variations in the counting efficiency with varying degrees of quench. For this reason, it is necessary to monitor both the changes in the ^{55}Fe efficiency and the ^{59}Fe cross-talk in the ^{55}Fe region as a function of quench. This technique recommends the use of the automatic external standard ratio supplied by most liquid scintillation counters to monitor the amount of quench in a sample.

6.3 The final heating of the sample solution will drive off all excess hydrochloric acid, ammonia, and water. These substances are, therefore, effectively removed as possible quenching agents.

6.4 Scintillation stock or sample solutions which have been exposed to light must be dark adapted to avoid erratic results due to light activation of the scintillator.

NOTE 1—It is the responsibility of the user to determine the required dark adaptation period for the specific cocktail used.

6.5 The stable iron content in a sample will interfere in the determination of the chemical recovery. Since the amount of stable iron in a sample will depend on its sources, a correction for the iron in the sample must be made.

7. Apparatus

7.1 *Liquid Scintillation Counter,* with an automatic external standard and multiple energy region of interest (ROI) capabilities.

7.2 *Glass Scintillation Vials,* 20-mL vials exhibiting suitable optical reproducibility so as not to cause erratic results among samples.

7.3 *Atomic Absorption Spectrophotometer.*

7.4 *Variable Speed Peristaltic Pump,* with controller. Pump speed should be between 5 and 8 mL/min.

7.5 *Centrifuge,* using 100 mL centrifuge tubes.

7.6 *Volumetric Flasks.*

7.7 *Anion Exchange Columns:*

7.7.1 *Columns*—Commercially available plastic drying tubes and ends (40 mL volume, 1.5 cm diameter, 15 cm long).

7.7.2 *Tubing*—Pump inlet tubing, approximately 45.7 cm (18 in.) in length, and pump outlet tubing, approximately 76.2 cm (30 in.) in length.

7.7.3 *Polyethylene Porous Disc*—35-µm pore size and 3.2 mm thick.

8. Reagents and Materials

8.1 *Purity of Reagents*—Reagent grade chemicals shall be used for 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 first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

8.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water conforming to Specification D1193, Type III.

8.3 *Resin*—AGl-X8, AG1-X10, 200–400 mesh; 25 mL previously equilibrated with 125 mL concentrated hydrochloric acid.

8.4 *Scintillation Cocktail*—Commercially prepared Insta-Gel scintillator or equivalent non-ionic detergent scintillator of the *octyl*-phenyl polyglycol ether type.[6]

NOTE 2—To obtain a clear aqueous final sample, the sample volume must be kept below 1.8 mL with the addition of 15 mL Insta-Gel. It is the responsibility of the user to determine the optimum sample volume to cocktail volume to obtain a clear homogeneous solution for any other liquid scintillation cocktail used.

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[5] *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.

[6] The sole source of supply of the apparatus known to the committee at this time is Insta-Gel scintillator, available from PerkinElmer Life and Analytical Sciences, 940 Winter Street, Waltham, MA 02451. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,[1] which you may attend.

8.5 *Ammonium Hydroxide* (NH_4OH)—Concentrated (approximately 15M) (sp gr 0.90).

8.6 *Ammonium Phosphate* (0.5 M)—Dissolve 66 g of ammonium monohydrogen phosphate [$(NH_4)_2HPO_4$] in water and dilute to 1000 mL with water in a volumetric flask.

8.7 *Cesium Carrier Solution* (1 mg/1 mL)—Cesium as cesium chloride (CsCl) in dilute hydrochloric acid.[7]

8.8 *Cobalt Carrier Solution* (1 mg/1 mL)—Cobalt as cobalt chloride ($CoCl_2$) in dilute hydrochloric acid.[7]

8.9 *Hydrochloric Acid* (sp gr 1.187)—Concentrated HCl.

8.10 *Hydrochloric Acid* 10 *M*—Dilute 833 mL of concentrated hydrochloric acid in 100 mL water and dilute to 1000 mL with water in a volumetric flask.

8.11 *Hydrochloric Acid* 6 *M*—Dilute 500 mL of concentrated hydrochloric acid in 400 mL of water, dilute to 1000 mL with water in a volumetric flask.

8.12 *Hydrochloric Acid* 4 *M*—Dilute 333 mL of concentrated hydrochloric acid in 600 mL of water, dilute to 1000 mL with water in a volumetric flask.

8.13 *Hydrochloric Acid* 0.5 *M*—Dilute 42 mL of concentrated hydrochloric acid in 900 mL of water, dilute to 1000 mL with water in a volumetric flask.

8.14 *Hydrochloric Acid* 0.01 *M*—Dilute 20 mL of 0.5 *M* HCl in 900 mL of water, dilute to 1000 mL with water in a volumetric flask.

8.15 *Hydrochloric* (6 *M*)*-Hydrofluoric Acid* (0.5 *M*)—Dilute 500 mL of concentrated hydrochloric acid (HCl) (sp gr 1.187) and 20 mL of 49 % concentrated hydrofluoric acid (HF) in 400 mL of water, dilute to 1000 mL with water in a plastic or TFE-fluorocarbon volumetric flask.

8.16 *Iron Carrier Solution* (Fe^{+3} as ferric chloride)[7]—Dissolve 5.00 g of metallic iron in 300 mL of 6 *M* HCl, filter, dilute to 1 L, and calibrate using an atomic absorption spectrophotometer (Test Methods D1068).

8.17 *Manganese Carrier Solution* (1 mg/1 mL)—Mn^{+2} in dilute nitric acid (HNO_3).[7]

8.18 *Niobium Carrier Solution* (1 mg/1 mL)—Nb^{+5} in 5 % hydrofluoric acid (1 + 9 *M*).[7]

8.19 *Nitric Acid* (sp gr 1.42)—Concentrated (HNO_3).

8.20 *Phosphoric Acid* (sp gr 1.834)—Concentrated (H_3PO_4).

8.21 *Sodium Hydroxide* (6 *M*)—Dissolve 24 g sodium hydroxide (NaOH) in 90 mL water, allow to cool, and dilute to 100 mL with water and store in plastic bottles; a commercially prepared solution may also be used.

8.22 *Zinc Carrier Solution* (1 mg/mL)—Zn^{+2} in dilute hydrochloric acid.[7]

8.23 *Zirconium Carrier Solution* (1 mg/mL)—Zr^{+4} in dilute hydrochloric acid.[7]

8.24 *^{55}Fe Standard Solution*—Traceable to a national standards laboratory such as National Institute of Standards and Technology (NIST) or UK National Physical Laboratory (NPL).

8.25 *^{59}Fe Standard Solution*—Traceable to a national standards laboratory such as NIST or NPL.

9. Hazards

9.1 HF is extremely hazardous and should be used in a well-ventilated hood. Wear rubber gloves, safety glasses or goggles, and a laboratory coat. Avoid breathing any HF fumes. Clean all spills and wash thoroughly after using HF. Also, do not add HF to any glassware for it is a significant hazard and can affect analytical accuracy.

10. Sampling

10.1 Collect samples in accordance with procedures presented in Practices D3370, as applicable.

10.2 If the sample is not acidified at the time of collection, 20 mL of concentrated HCl or HNO_3 should be added per 1000 mL of sample. After acidification of the sample, the sample should be allowed to sit overnight prior to analysis.

11. Calibration

11.1 The reference standard for both iron isotopes will contain 5 mg iron carrier. Add 6 drops of concentrated phosphoric acid to the carrier solution and heat on a hot plate until it clears. This will drive off any excess hydrochloric acid and water (to less than 0.5 mL but do not allow to bake dry). Add 1 mL of water and swirl in the glass vial. This final carrier solution should be colorless. Cool the vial to room temperature. Spike with the appropriate isotope and add 15 mL of scintillation cocktail. Cap and shake until the mixture is clear; this step ensures that the proper sample volume to scintillation cocktail volume ratio is obtained for a clear, homogeneous solution. The volume of the reference standard should be such that its addition to the sample does not cause additional quench.

11.2 Prepare a series of quenched ^{55}Fe standards and a series of quenched ^{59}Fe standards using various weights of iron carrier or concentration or volumes, or both, of acid. Use the least quenched standards in each set to optimize the liquid scintillation counter (LSC) discriminator settings and amplifier gain. Ensure that the ^{55}Fe spectrum does not spill over into the ^{59}Fe ROI. If your instrument does not allow for multiple nuclides to be optimized follow Practice D7282 for instrument optimization.

11.3 Count each set of standards with the LSC automatic external standard ratio selected on to obtain approximately 1 % counting statistics (approximately 10 000 counts) in the ROI.

11.4 Prepare a curve of the ^{55}Fe efficiency versus the external standard ratio.

11.5 Prepare a crosstalk (XT) curve of the ratio of ^{59}Fe counts in the ^{55}Fe ROI to the ^{59}Fe counts in the ^{59}Fe ROI versus the external standard ratio.

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[7] Commercially available atomic absorption reference standards in 1 mg = 1 mL concentrations may be used as carriers.

11.6 Additional guidance on the set-up, calibration and calibration verification of the instrument can be found in Practice D7282.

12. Procedure

12.1 Measure an acidified and filtered aliquant of the sample (approximately 0.05 L) into a 100 mL centrifuge tube.

12.1.1 A background subtraction sample shall be processed in accordance with 12.2 through 12.16 with each set of samples.

12.2 Add 5 mg iron carrier and mix well.

12.3 Add NaOH (6 *M*), mix, and heat to precipitate iron hydroxide.

12.4 Centrifuge and decant the supernatant solution to waste.

12.5 Wash the precipitate with 50 mL of hot water, centrifuge and decant the supernatant solution to waste.

12.6 Dissolve the precipitate in 5 mL of 10 *M* HCl and add 2 mL (2 mg) each of the appropriate hold-back carriers (Co^{+2}, Zn^{+2}, Zr^{+4}, Nb^{+5}, Mn^{+2}, and Cs^{+1} carriers). It is recommended that an aliquant of the sample be prepared for gamma isotopic analysis to verify the presence of potentially interfering nuclides. Transfer the solution with 10 *M* HCl washes to a beaker and dilute to approximately 50 mL with 10 *M* HCl. Pass the solution through an anion exchange column (AG1-X8 or AG1-X10, 200–400 mesh) previously equilibrated with 125 mL of concentrated HCl. The column volume is approximately 25 mL. The speed of the sample and washes through the column should be between 5 and 8 mL/min.

12.7 After the sample has been passed through the column, wash the column with 100 mL of 10 *M* HCl, then 150 mL of 6 *M* HCl followed by 300 mL of 4 *M* HCl. If $^{95}Zr/^{95}Nb$ activity is suspected, replace the 6 *M* HCl with the 6 *M* HCl-0.5 *M* HF. Discard the effluents.

NOTE 3—If 6 *M* HCl-0.5 *M* HF is used, use only plastic ware. Column plug should not be glass wool.

12.8 Elute the iron with approximately 150 mL of 0.01 *M* HCl (discard the first 20 mL) and collect the yellow band indicative of Fe^{+3} in a beaker. Do not allow the column to run dry.

12.9 Add 10 mL of 0.5 *M* $(NH_4)_2HPO_4$ to the beaker and add dropwise concentrated NH_4OH until the pH is 3.0. The use of pH paper may not be sensitive enough to accurately determine the pH. Gently heat the sample on a hot plate to completely precipitate the iron as $Fe(PO_4)$. Centrifuge, decant, and wash the precipitate with hot water.

12.10 Dissolve the precipitate with a minimal amount of 6 *M* HCl. Transfer the solution with approximately 10 mL water to a clean, preweighed glass scintillation vial (m_1).

12.11 Weigh the total solution plus the vial (m_2) and quantitatively transfer a 1-mL aliquant to a 50 mL volumetric flask and dilute to 50 mL with water. Weigh the remaining solution plus the vial (m_3).

12.12 Calculate the ratio (Eq 2), the amount remaining versus the amount transferred.

12.13 Determine the iron concentration by using an atomic absorption spectrophotometer (Test Methods D1068) on the filtered water sample (EC_o) and the diluted aliquant (EC_a) in 12.11.

12.14 Determine the chemical yield by Eq 1:

$$Y = \frac{EC_a \times V_D \times Ratio}{EC_o \times V_s + m_c} \tag{1}$$

where:

Y = chemical yield of iron,
EC_a = concentration of iron in the aliquant transferred and diluted in a proper size volumetric flask, (ppm (mg/L)),
EC_o = concentration of iron in the original sample (filtered), ppm (mg/L),
V_D = volume of the dilution in 12.11, L,
V_s = volume of the original sample aliquant in 12.1, L,
m_c = mass of iron carrier added, mg, and
$Ratio$ = mass of amount remaining/mass of amount transferred.

and

$$Ratio = \frac{m_3 - m_1}{m_2 - m_3} \tag{2}$$

where:

m_1 = mass of glass scintillation vial, clean and preweighed, mg,
m_2 = mass of total solution plus vial, mg, and
m_3 = mass of remaining solution plus vial, mg.

12.15 Add 6 drops of concentrated phosphoric acid to the remaining solution in the vial and heat the solution on a hot plate until it clears. This will drive off any excess hydrochloric acid and water (to less than 0.5 mL but do not allow to bake dry). Add 1 mL of water and swirl in the glass vial. This final solution should be colorless. Cool the vial to room temperature.

NOTE 4—If the final solution is still yellow, add a few more drops of concentrated phosphoric acid and repeat 12.15 until the solution clears.

12.16 Add 15 mL of liquid scintillation cocktail,[6] cap, and shake until the solution clears.

NOTE 5—Use of an ultrasonic bath may be necessary in 12.15 and 12.16 to dissolve the solids and to remove air bubbles caused by shaking.

12.17 Allow sample to dark-adapt before a count is initiated.

13. Calculation

13.1 Calculate the ^{55}Fe concentration ($AC_{Fe\text{-}55}$) in a sample on the reference day in becquerels per litre using Eq 3:

$$AC_{Fe-55} = \frac{R_{n,Fe-55} - (R_{n,Fe-59} \times XT)}{\varepsilon_{Fe-55} \times Y \times V_s \times DF} \tag{3}$$

where:

$AC_{Fe\text{-}55}$ = ^{55}Fe activity concentration, Bq/L,
$R_{n,Fe\text{-}55}$ = net count rate in the ^{55}Fe ROI, in counts per second, s^{-1},

$$u_c(AC_{Fe-55}) = \sqrt{\frac{u^2(R_{n,Fe-55}) + u^2(R_{n,Fe-59}) \times XT^2 + u^2(XT) \times R^2_{n,Fe-59}}{\varepsilon^2_{Fe-55} \times Y^2 \times V_s^2 \times DF^2} + AC^2_{Fe-55}\left(\frac{u^2(\varepsilon_{Fe-55})}{\varepsilon^2_{Fe-55}} + \frac{u^2(Y)}{Y^2} + \frac{u^2(V_s)}{V_s^2}\right)} \qquad (4)$$

where:

$u_c(AC_{Fe-55})$ = combined standard uncertainty of the ^{55}Fe activity concentration, Bq/L,
$u(R_{n,Fe-55})$ = standard uncertainty of the ^{55}Fe net count rate, s^{-1},
$u(R_{n,Fe-59})$ = standard uncertainty of the ^{59}Fe net count rate, s^{-1},
$u(\varepsilon_{Fe-55})$ = standard uncertainty of the ^{55}Fe detection efficiency,
$u(XT)$ = standard uncertainty of the crosstalk factor,
$u(Y)$ = standard uncertainty of the chemical yield, and
$u(V_s)$ = standard uncertainty of the sample volume, L.

$R_{n,Fe-59}$ = net count rate in the ^{59}Fe ROI, s^{-1}),
XT = cross talk factor obtained from ^{59}Fe quench curve, (see 11.5),
ε_{Fe-55} = detection efficiency for ^{55}Fe obtained from the $_{55}$Fe quench curve (see 11.4),
Y = chemical recovery as a fraction,
V_s = sample volume, L, (see 12.1),
DF = decay factor = $e^{-\lambda t}$,
λ = ^{55}Fe decay constant (d^{-1}) and
t = elapsed time between reference date and midpoint of counting period in days.

13.2 *Combined Standard Uncertainty:*

13.3 *Expanded Uncertainty:*

$$U(AC_{Fe-55}) = k \times u_c(AC_{Fe-55}) \qquad (5)$$

where:

$U(AC_{Fe-55})$ = expanded uncertainty of the ^{55}Fe activity concentration, (Bq/L), and
k = coverage factor for the desired level of confidence (normally $k = 2$ for ~95 % confidence).

13.4 *Critical Level/Decision Level (DLC) and Minimum Detectable Concentration (MDC):*

13.4.1 The Critical Level/Decision Level activity concentration can be estimated as follows:

$$DLC = \frac{1.645\sqrt{R_{b,Fe-55}\frac{t_a + t_b}{t_a t_b} + u^2(R_{n,Fe-59}) \times XT^2 + u^2(XT) \times R_{n,Fe-59}{}^2}}{\varepsilon_{Fe-55} \times Y \times V_s \times DF} \qquad (6)$$

NOTE 6—It may be concluded that ^{55}Fe is present in the sample when the measured activity concentration of ^{55}Fe (AC_{Fe-55}) exceeds the DLC.

13.4.2 The *a priori* MDC for the ^{55}Fe region of interest only (assuming no interferences) can be estimated by the following equation:

14. Quality Control Samples

14.1 In order to provide reasonable assurance that the analytical results obtained using this test method are valid and accurate within the confidence limits of the method, Quality Control (QC) samples are analyzed with each batch of samples undergoing analysis. Each batch should include not more than 20 samples, excluding those used for QC purposes. Laboratory or project quality assurance plans may contain more restrictive process QC requirements. The following minimum QC procedures must be followed when running the test method:

14.2 *Initial Demonstration of Laboratory/Instrument Quality:*

14.2.1 If a laboratory or analyst has not performed this test before or there has been a major change in the measurement system, a precision and bias study must be performed to demonstrate laboratory/instrument capability. A significant change is defined as any change, repair, or alteration of any component in the system which maybe expected to affect the response of the measurement system.

14.2.2 Analyze seven replicates of a standard solution prepared from an independent reference material (ICRM) containing ^{55}Fe concentrations sufficient to reduce counting uncertainty to 1 % or less at one sigma. The matrix used for the demonstration should represent a water sample typical for which the procedure will be used, for example, surface water.

14.2.3 Calculate the mean and standard deviation of the seven values and compare to the acceptable ranges of precision and mean bias of 10 % and ±10 %, respectively, based on a review of the collaborative study data. Practice D5847 should be consulted on the manner by which precision and mean bias are determined from the initial demonstration study. The study should be repeated until the precision and bias are within the given limits.

14.2.4 Analyze three replicates of a blank solution matrix. The matrix used for the demonstration should represent a water sample typical for which the method will be used, for example, a surface water.

14.2.5 Calculate the ^{55}Fe activity for each of the three blank solutions. This study shall be repeated until the ^{55}Fe result of each of the three blank solutions is below the half of the associated MDC.

14.2.6 This method shall not be used for official samples until precision, bias, and blank requirements are met.

14.3 *Laboratory Control Sample (LCS):*

14.3.1 To ensure that the test method is in control, analyze an LCS with each batch of no more than 20 samples. The activity added to reagent water should be appropriate for the type of samples analyzed and should allow sufficient precision to ensure meaningful assessment of accuracy. The LCS must be taken through all the steps of the analytical method including sample preservation and pretreatment. The result for the LCS shall fall with the limit of ±25 % of the expected value.

$$MDC = \frac{\frac{2.71}{t_a} + 3.29\sqrt{R_{b,Fe-55}\frac{t_a + t_b}{t_a t_b} + u^2(R_{n,Fe-59}) \times XT^2 + u^2(XT) \times R_{n,Fe-59}{}^2}}{\varepsilon_{Fe-55} \times Y \times V_s \times DF} \quad (7)$$

where:

t_a = counting time of sample in seconds,
t_b = background counting time in seconds,
$R_{b,Fe-55}$ = ^{55}Fe ROI background count rate.

TABLE 1 ^{55}Fe Overall and Single Operator Precision Data

Amount Added, Bq	Overall Precision, S_t, Bq	Single Operator Precision, S_o, Bq
43.7	6.7	0.74
426	23	21.09
5030	290	81.40
35 800	2200	183.15

TABLE 2 ^{55}Fe Bias Data

Amount Added, Bq	Amount Found, Bq	Bias, %
43.7	47.7 ± 6.7	+ 9.3
426	437 ± 23	+ 5.2
5030	5160 ± 290	+ 2.6
35 800	37 300 ± 2200	+ 4.0

14.3.2 If the result is not within the limit, analyses should be stopped and the reason for failure identified and resolved. An indication of the occurrence should accompany the reported results.

14.4 *Method Blank:*

14.4.1 Analyze a reagent water test blank with each batch of no more than 20 samples. The concentration of ^{55} Fe found in the blank should be less than DLC.

14.4.2 If the concentration of ^{55}Fe is found above this level, provide an explanation in a case narrative.

14.5 *Matrix Spike (MS):*

14.5.1 Analyze at least one matrix spike sample with each batch of no more than 20 samples by spiking an aliquot of a sample within the batch with a known ^{55}Fe concentration.

14.5.2 The spike should produce a concentration of ^{55}Fe that is 2 to 5 times the anticipated sample concentration or as specified by the laboratory, whichever is greater.

14.5.3 The Matrix Spike must be taken through all the steps of the method.

14.5.4 Calculate the per cent recovery of the matrix spike using the following formula:

$$R = \frac{AC_{as} - AC_a}{AC_s} \times 100\,\% \quad (8)$$

where:

AC_{as} = concentration of ^{55}Fe in Bq/L measured in the spiked aliquot,
AC_a = concentration of ^{55}Fe in Bq/L in the original sample,
AC_s = spiked concentration of ^{55}Fe in Bq/L.

14.5.5 The percent recovery, *R*, should fall within ±25 % of the expected value. If the percent recovery is not within these limits, provide an explanation in a case narrative.

14.6 *Duplicate:*

14.6.1 To check the precision of sample analyses, analyze a sample in duplicate with each batch of no more than 20 samples. Calculate the statistical agreement [duplicate error ratio (DER)] between the two results. This calculation is performed using the combined standard uncertainty of each result as shown below:

$$DER = \frac{|\,AC_{original} - AC_{dup}\,|}{\sqrt{u_c^2(AC_{original}) + u_c^2(AC_{dup})}} \quad (9)$$

where:

$AC_{original}$ = original sample activity concentration,
AC_{dup} = duplicate sample activity concentration
$u_c(AC_{original})$ = combined standard uncertainty of the original sample, and
$u_c(AC_{dup})$ = combined standard uncertainty of the duplicate sample.

14.6.2 In those cases where there is insufficient sample volume to allow performance of a duplicate sample analysis, a duplicate LCS (LCS-D) should be performed and analyzed using the same DER criteria.

14.6.3 The value of DER should be less than or equal to 3.0. If the sample duplicate or LCS duplicate result is greater than 3.0 all samples in the batch must be reanalyzed, or an explanation must be provided in a case narrative.

14.7 *Independent Reference Material (IRM):*

14.7.1 In order to verify the quantitative value produced by the test method, analyze an IRM sample, which was submitted on at least a single-blind basis (if practical) to the laboratory at least once per quarter. The concentration of analyte in the traceable reference material should be appropriate to the typical purpose for which the method is used. The value obtained shall demonstrate acceptable performance as defined by the program or the outside source.

14.7.2 In the absence of other acceptance criteria for the IRM sample, compare the IRM sample result to the IRM known value as follows:

$$R = \frac{|\,IRM_{found} - IRM_{known}\,|}{\sqrt{u_c^2(IRM_{found}) + u_c^2(IRM_{known})}} \quad (10)$$

where:

R = relative difference,
IRM_{found} = found concentration of the IRM,
IRM_{known} = known concentration of the IRM,

$u_c(IRM_{found})$ = combined standard uncertainty of the IRM found concentration, and

$u_c(IRM_{known})$ = combined standard uncertainty of the IRM known concentration.

14.7.3 The value of R should be less than or equal to 3.0. If the value of R is greater than 3.0, the method should be investigated to determine the cause.

15. Precision and Bias[8]

15.1 The collaborative test conducted on this test method included six laboratories each with one operator. Four activity levels between 44 Bq/L (6 × MDC) and 36 kBq/L were tested with three replicates per level. The determination of the precision and bias statements were made in accordance with Practice D2777 – 85.

15.2 These collaborative test data were obtained using reagent grade water containing ^{55}Fe. For other matrices, these data may not apply.

15.3 The overall and single operator precision have been found to vary with ^{55}Fe level according to Table 1.

15.4 The bias of this test method, based upon the collaborative test data, was found to vary with ^{55}Fe level according to Table 2.

16. Keywords

16.1 anion exchange; ^{55}Fe; liquid scintillation counting; radioactive iron; radioactivity

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[8] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D19-1141. Contact ASTM Customer Service at service@astm.org.