Designation: D 4922 – 94ᵉ01

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

This standard is issued under the fixed designation D 4922; 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.

ε¹ NOTE—Editorial changes were made throughout in August 1995.

1. Scope

1.1 This test method covers the determination ~~of iron-55~~ of ^{55}Fe in the presence ~~of iron-59~~ of ^{59}Fe by liquid scintillation

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[1] This test method is under the jurisdiction of ASTM Committee D-19 on Water and is the direct responsibility of Subcommittee D19.04 on Methods of Radiochemical Analysis.

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counting. The *a-priori* minimum detectable concentration for this test method is 7.4 E-3 Bq/mL.[2]

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

1.3 This test method was used successfully with Type III reagent water conforming to Specification D 1193. 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:*
D 1068 Test Methods for Iron in Water[3]
D 1129 Terminology Relating to Water[3]
D 1193 Specifications for Reagent Water[3]
D 2777 Practice for Determination of Precision and Bias of Applicable Methods of Committee D-19 on Water[3]
D 3370 Practices for Sampling Water[3]

3. Terminology

3.1 *Definitions*—For definitions of terms used in this test method, refer to Terminology D 1129. For terms not defined in this test method or in Terminology D 1129, 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 ~~various molarity~~ 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 or any procedure described in Test Method D 1068.

5. Significance and Use

5.1 Radioactive iron is produced by neutron activation of ~~water,~~ stable iron. Its concentration in reactor ~~fuel cladding material, and reactor structural components. It is, therefore, of significance as an indicator of~~ coolant is used to monitor the corrosion of reactor parts such as reactor fuel-cladding material and reactor structural components.

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.

6. Interferences

6.1 Samples of reactor origin will also ~~contain iron-59~~ contain ^{59}Fe after other radioactive contaminants have been removed by anion exchange (see Fig. 1~~). Iron-59~~). ^{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 iron-55.~~ 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 iron-59~~ the ^{59}Fe spectral cross-talk in ~~the iron-55~~ the ^{55}Fe region.

6.2 Quenching, which may be caused by a number of factors, results in a reduction in the 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 iron-55~~ the ^{55}Fe efficiency and ~~the iron-59~~ the ^{59}Fe cross-talk in ~~the iron-55~~ 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.

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[2] Currie, L., "Limits for Qualitative Detection and Quantitative ~~Determination,"~~ Determination," *Analytical Chemistry*, 40, 1968, pp. 586–593.
[3] *Annual Book of ASTM Standards*, Vol 11.01.
[4] "American National Standard Glossary of Terms,'' *Nuclear Science and Technology (ANSI N1.1)*, American National Standards Institute, 1430 Broadway, New York, NY 10018.
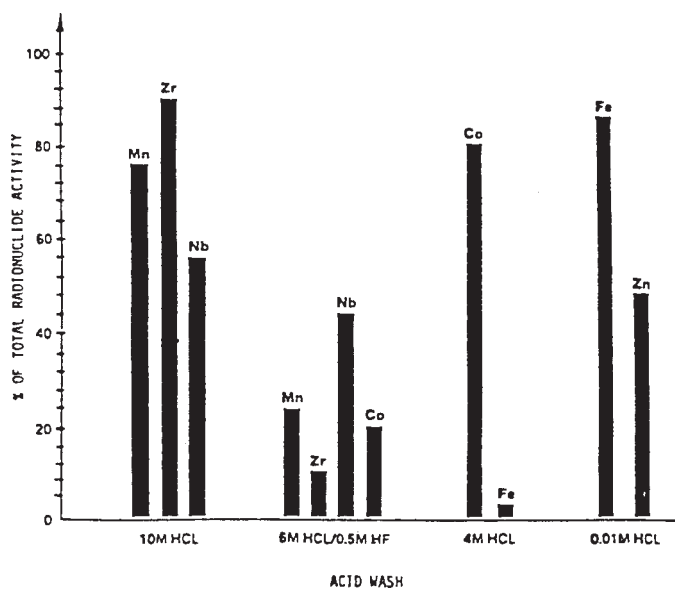
FIG. 1 Percent of Total Radionuclide Activity Removed Per Acid Wash

~~Note 1—At least 3.5 h of dark adaptation is suggested; however, since this period may vary based on the amount of liquid scintillation cocktails, it~~ 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 ~~or~~ 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 ~~Flask, 50 mL.~~ Flasks.*

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 D 1193, Type III.

8.3 *Anion Exchange Columns*:

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

8.3.2 *Columns*—Commercially available plastic drying tubes and ends (40 mL volume, 1.5 cm diameter, 15 cm long).[6]

8.3.3 *Tubing*—Pump inlet tubing, approximately 18 in. in length. ~~P~~, and pump outlet tubing, approximately 30 in. in length.

8.3.4 *Polyethylene Porous Disc*—35 μm pore size and 3.2 mm thick.[7]

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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 *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.

[6] Bio-Rad drying tubes, available from Bio-Rad, 2200 Wright Ave., Richmond, CA 94804, have been found satisfactory for this purpose.

[7] Bel-Art porous polyethylene sheets, in various pore sizes and thicknesses, available from Bel-Art, Pequannock, NJ 07440, have been found satisfactory for this purpose.

8.4 *Scintillation Cocktail*—Commercially prepared Insta-Gel scintillator or equivalent non-ionic detergent scintillator of the *octyl*-phenyl polyglycol ether type.[8]

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.

8.5 *Ammonium Hydroxide* (NH₄OH)—Concentrated (sp gr 0.90).

8.6 *Ammonium Phosphate* (0.5 M)—Dissolve 66 g of ammonium monohydrogen phosphate [(NH₄)₂HPO₄] in 1000 mL of water.

8.7 *Cesium Carrier Solution* (1 mg = 1 mL)—Cesium as cesium chloride (CsCl) in dilute hydrochloric acid.[9]

8.8 *Cobalt Carrier Solution* (1 mg = 1 mL)—Cobalt as cobalt chloride (CoCl₂) in dilute hydrochloric acid.[9]

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

8.10 *Hydrochloric Acid* ~~(5 + 1)—Dilute 833 mL of concentrated hydrochloric acid to 1000 mL with water in a volumetric flask. This solution is 10~~10 M (5 + 1)—Dilute 833 mL of concentrated hydrochloric acid to 1000 mL with water in a volumetric flask.

8.11 *Hydrochloric Acid* ~~(1 + 1)—Dilute 500 mL of concentrated hydrochloric acid to 1000 mL with water in a volumetric flask. This solution is 6~~6 M (1 + 1)—Dilute 500 mL of concentrated hydrochloric acid to 1000 mL with water in a volumetric flask.

8.12 *Hydrochloric Acid* ~~(1 + 2)—Dilute 333 mL of concentrated hydrochloric acid to 1000 mL with water in a volumetric flask. This solution is 4~~4 M (1 + 2)—Dilute 333 mL of concentrated hydrochloric acid to 1000 mL with water in a volumetric flask.

8.13 *Hydrochloric Acid* ~~(1 + 23)—Dilute 42 mL of concentrated hydrochloric acid to 1000 mL with water in a volumetric flask. This solution is 0.5~~0.5 M (1 + 23)—Dilute 42 mL of concentrated hydrochloric acid to 1000 mL with water in a volumetric flask.

8.14 *Hydrochloric Acid* ~~.01 M~~ (1 + 1199)—Dilute 20 mL of ~~HCl (1 + 23) to 1000 mL with water in a volumetric flask. This solution is 0.01~~ 0.5 M HCl 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) to 1000 mL with water in a plastic or TFE-fluorocarbon volumetric flask.

8.16 *Iron Carrier Solution* (Fe⁺³ as ferric chloride)[9]—Dissolve 5.00 g of metallic iron in 300 mL of ~~HCl (1 + 1),~~ 6 M HCl, filter, dilute to 1 L, and calibrate using an atomic absorption spectrophotometer (Test Methods D 1068).

8.17 *Manganese Carrier Solution* (1 mg = 1 mL)—Mn ⁺² in dilute nitric acid (HNO₃).[9]

8.18 *Niobium Carrier Solution* (1 mg = 1 mL)—Nb⁺⁵ in 5 % hydrofluoric acid (1 + 9 M).[9]

8.19 *Nitric Acid* (sp gr 1.42)—Concentrated (HNO₃).

8.20 *Phosphoric Acid* (sp gr 1.834)—Concentrated (H₃PO₄).

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. This solution is 6 M.~~ water and store in plastic bottles.

8.22 *Zinc Carrier Solution* (1 mg = 1 mL)—Zn⁺² in dilute hydrochloric acid.[9]

8.23 *Zirconium Carrier Solution* (1 mg = 1 mL)—Zr⁺⁴ in dilute hydrochloric acid.[9]

9. Hazards

9.1 HF is very 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 D 3370, as applicable.

10.2 If the sample is not acidified at the time of collection, 20 mL of concentrated HCl or HNO₃ should be added per 1000 mL of sample.

11. Calibration

11.1 The reference standard for both iron isotopes will contain 5 mg iron carrier ~~h~~. 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 ~~6 drops of concentrated phosphoric acid~~ swirl in the glass vial. This final carrier solution should be colorless. Cool the vial to ~~obtain a colorless solution.~~ 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 iron-55~~ quenched ⁵⁵Fe standards and a series of ~~quenched iron-59~~ quenched ⁵⁹Fe standards using various weights of iron carrier or concentration or volumes, or both, of acid. Use the least quenched standards in each set

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[8] Insta-Gel scintillator, available from United Technologies Packard, Packard Instrument Company, 2200 Warrensville Rd., Downers Grove, IL 60515 has been found to be suitable for this purpose.

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

to optimize the liquid scintillation counter (LSC) discriminator settings and amplifier gain. Ensure that ~~the iron-55~~ the ^{55}Fe spectrum does not spill over into ~~the iron-59~~ the ^{59}Fe ROI.

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 iron-55~~ the ^{55}Fe efficiency versus the external standard ratio.

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

12. Procedure

12.1 Weigh an acidified and filtered aliquant of the sample (approximately 50 g) into a 100 mL centrifuge tube.

12.2 Add 1 mL of the iron carrier solution and mix well.

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

12.4 Centrifuge and decant the supernate to waste.

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

12.6 Dissolve the precipitate in ~~50~~ 5 mL of 10 M HCl ~~(5 + 1)~~ 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 ~~(5 + 1)~~ washes to a beaker and dilute to approximately 50 mL with ~~HCl (5 + 1).~~ 10 M HCl. Pass the solution through an anion exchange column (AGl-X10 or 1-X8, 200-400 mesh) previously equilibrated with 125 mL of concentrated HCl (sp gr 1.19). 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 ~~HCl (5 + 1),~~ 10 M HCl, then 150 mL of ~~HCl (1 + 1)~~ 6 M HCl followed by 300 mL of ~~HCl (1 + 2).~~ 4 M HCl. If zirconium-95/niobium-95 activity is suspected, ~~substitute~~ replace the ~~HCl (1 + 1)~~ 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 ~~HCl~~ 0.01 ~~(1 + 1199)~~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 ~~HCl (1 + 1).~~ 6 M HCl. Transfer the solution with approximately 10 mL water to a clean, preweighed glass scintillation vial (~~W~~M_1).

12.11 Weigh the total solution plus the vial (~~W~~M_2) and quantitatively transfer a 1-mL aliquant to a 50 mL volumetric flask and dilute to 50 mL. Weigh the remaining solution plus the vial (~~W~~M_3).

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

12.13 Determine the iron concentration by using an atomic absorption spectrophotometer (Test Method D 1068) on the filtered water sample (~~A~~EC_o) and the diluted aliquant (~~B~~EC_a) in 12.11.

12.14 Determine the chemical yield by Eq 1:

$$\text{Yield, } Y = \frac{B \times V_1 \times R}{A \times V_2 + C_{\,D}} \tag{1}$$

$$\text{Yield, } Y = \frac{EC_a \times V_D \times Ratio}{EC_o \times V_s + M_c} \tag{~~1~~}$$

~~V~~ ~~D~~ ~~V~~ ~~s~~ ~~M~~ ~~c~~ ~~Ratio~~

where:
~~B~~ EC_a = concentration of iron in the aliquant transferred and diluted in a proper size volumetric flask, ppm (mg/mL),

~~A~~ EC_o = concentration of iron in the original sample (filtered), ppm (mg/mL),

~~V~~$_1$ V_D = volume of the dilution in 12.11, L,

~~V~~$_2$ V_s = volume of the sample processed, L,

~~C~~ M_c = iron carrier, mg, and

~~Ratio~~ = weight of amount remaining/weight of amount transferred.

~~R = weight of amount remaining weight of amount transferred = (W3 – W1)(W2 – W3)~~

$$\frac{(M_3 - M_1)}{(M_2 - M_3)} \tag{2}$$

~~M2~~M3

where:

~~W~~M'	=	weight of glass scintillation vial, clean and preweighed, mg,
~~W~~M_2	=	weight of total solution plus vial, mg, and
~~W~~M_3	=	weight of remaining solution plus vial, mg.

12.15 Add 6 drops of <u>concentrated</u> phosphoric acid ~~(sp gr 1.834)~~ to the remaining solution <u>in the vial</u> and heat the solution on a hot plate until it clears. This will drive off any excess hydrochloric acid and water (<u>to less than 0.5 mL but</u> do not allow ~~the solution to~~ ~~be baked dry or to exceed 0.5 mL).~~ <u>bake dry).</u> 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[8], 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 A reagent blank containing 5 mg of iron carrier solution shall be processed in accordance with 12.1 through 12.16 with each set of samples.

12.18 Allow the sample to dark-adapt before a count is initiated. ~~Note 6—At least 3.5 h of dark adaptation is suggested.~~

13. Calculation

13.1 Calculate ~~the iron-55 concentration (C~~ ~~the~~ <u>the</u> ^{55}Fe concentration (AC_{Fe-55}) in a sample on the reference day in ~~microcuries~~ <u>Becquerels</u> per gram using Eq ~~2:~~ <u>3:</u>

$$\text{Iron}-55\ \text{activity, Bq/gm} = \frac{a - (b \times c)}{E \times Y \times W \times D}$$

$$A_{CFe-55} = \frac{R_{n^{55}Fe} - (R_{n^{59}Fe} \times XT)}{E_{^{55}Fe} \times Y \times M \times DF} \qquad (3)$$

Rn59FeXTE55FeYMDF

where:

~~a~~$AC^{55}Fe$	=	^{55}Fe activity concentration, Bq/g ~~net count rate (S $^{-1}$) in the ^{55}Fe ROI,~~
$R_n^{55}Fe$	=	net count rate (S^{-1}) in ~~the iron-55~~ <u>the</u> ^{55}Fe ROI,
~~b~~$R_n^{59}Fe$	=	net count rate (S^{-1}) in ~~the iron-59~~ <u>the</u> ^{59}Fe ROI,
~~c~~XT	=	cross talk factor obtained ~~from iron-59~~ <u>from</u> ^{59}Fe quench curve, <u>(see 11.5)</u>,
$E^{55}Fe$	=	~~fractional iron-55~~<u>fractional</u> ^{55}Fe efficiency obtained from ~~the iron-55~~ <u>the</u> ^{55}Fe quench curve (c~~p~~s/d~~p~~s), <u>(see 11.4)</u>,
Y	=	chemical recovery as a fraction,
~~W~~M	=	sample weight, g, <u>(see 12.1)</u>,
D~~F~~	=	decay factor = e$^{-\lambda t}$,
λ	=	~~iron-55~~ ^{55}Fe decay c
t	=	time between reference date and count date, days.

13.2 ~~Total Propagated~~<u>Combined Standard</u> Uncertainty:

$$TPU_\sigma = C_{55} * \left[\frac{\sigma^2_a + c^2\sigma^2_b + b^2\sigma^2_c}{[a - (b \times c)]^2} \right.$$

$$\left. + \left(\frac{\sigma_E}{E}\right)^2 + \left(\frac{\sigma_y}{Y}\right)^2 + \left(\frac{\sigma_w}{W}\right)^2 + \left(\frac{\sigma_D}{D}\right)^2 \right]^{\frac{1}{2}} \qquad (3)$$

$$AC_{55Fe} \left[\frac{S^2_{55Fe} + XT^2_{59Fe} + R_{n^{59}Fe}U_{XT}}{[R_{n^{55}Fe} - (R_{n^{59}Fe} \times XT)]^2} + \left(\frac{U_{E^{55}Fe}}{E_{55Fe}}\right)^2 + \left(\frac{U_y}{Y}\right)^2 + \left(\frac{U_m}{M}\right)^2 + \left(\frac{U_{DF}}{DF}\right)^2 \right]^{\frac{1}{2}} \qquad (4)$$

13.3 <u>Expanded Uncertainty, U(Bq/g)</u>:

$$U(Bq/g) = kU_c \qquad (5)$$

S55FeS59FeUXTUE55FeUyUM

where:

~~σ~~k	=	coverage factor for the desired confidence interval (normally K=2 for 95% confidence interval),
U_c	=	combined standard uncertainty (Eq 4),
S^{55}_{Fe}	=	standard deviation of the net count rate for the ^{55}Fe ROI,
~~σ~~$_bS^{59}_{Fe}$	=	standard deviation of the net count rate for the ^{59}Fe ROI,

$\sigma_{\overline{c}U_{XT}}$ = uncertainty of the cross talk factor ~~c~~XT,

$\sigma_{\overline{U}E^{55}F\overline{e}}$ = uncertainty of the ^{55}Fe efficiency factor $E^{55}Fe$,

$\sigma_{\overline{Y}U_x}$ = uncertainty of the chemical recovery factor Y,

$\sigma_{\overline{W}U_M}$ = uncertainty of the sample weight, ~~W~~M, and

$\sigma_{\overline{D}U_{DF}}$ = uncertainty of the decay factor $D\underline{F}$.

13.~~3~~4 *Minimum Detectable Concentration (MDC)*:

13.~~3~~4.1 The *a priori* minimum detectable concentration for the ^{55}Fe region of interest only (assumi~~n~~ng no interferences) is:

$$MDC\ (\text{Bq/g}) = \frac{\dfrac{2.71}{T} + 4.65 \times \sigma_B}{E_{\times Y \times W \times D}} \tag{6}$$

$$MDC\ (\text{Bq/g}) = \frac{\dfrac{2.71}{T} + 4.65 \times S_B}{E_{Fe55} \times Y \times M \times DF} \tag{(6)}$$

$\underline{S_B}$

where:

T = counting time of sample in seconds,

$\sigma\underline{S_B}$ = standard deviation of the background count rate (s^{-1}) when counted for T time(s), and $E^{55}Fe$, Y, ~~W~~M, $D\underline{F}$, typical values of the parameters described above.

14. Quality Control Samples

14.1 Whenever possible, the project leader, as part of the external quality control program, should submit quality control samples to the analyst along with routine samples in such a way that the analyst does not know which of the samples are the quality control samples. These external qual~~ti~~ty control samples, which usually include duplicate and blank samples, should test sample collection and preparation as well as sample analysis whenever this is possible. In addition, analysts are expected to run internal quality control samples that will indicate to them whether the analytical procedures are in control. Both the external and internal quality control samples should be prepared in such a way as to duplicate the chemical matrix of the routine samples, insofar as this is practical. The quality control samples that are routinely used consist of five basic types: blank samples, replicate samples, reference materials, control samples, and "spiked" samples.

15. Precision and Bias [10]

15.1 The collaborative test conducted on this test method included six laboratories each with one operator. Four activity levels between 37 Bq/L (6 × MDC) and 37 kBq/L were tested with three replicates per level. The determination of the precision and bias statements were made in accordance with Practice D 2777 – 85.

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

15.3 The overall and single operator precision have been found to vary ~~with Fe-55~~ 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 Fe-55 level according to Table 2.~~

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; iron-55;~~ exchange; ^{55}Fe; liquid scintillation counting; radioactive iron; radioactivity

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

Amount Added, Bq	Overall Precision, S_t	Single Operator Precision, S_o
43.7	6.7	0.74
426	23	21.09
5030	2.81	81.40
35 800	2200	183.15

TABLE 2 Iron-^{55}Fe Bias Data

Amount Added, Bq	Amount Found, nCi/L	Bias, %
43.7	47.7 $\pm$ 6.7	+ 9.3
426	437 $\pm$ 23	+ 5.2
5030	5160 $\pm$ 290	+ 2.6
35 800	37 300 $\pm$ 2200	+ 4.0