



Standard Test Method for Determination of Radioactive Iron in Water¹

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 in the presence of iron-59 by liquid scintillation counting. The *a-priori* minimum detectable concentration for this test method is 7.4 E-3 Bq/mL.

1.2 This test method was developed principally for the quantitative determination of iron-55. However, after proper calibration of the liquid scintillation counter with reference standards of each nuclide, iron-59 may also be quantitated.

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

2. Referenced Documents

2.1 ASTM Standards:

- D 1068 Test Methods for Iron in Water³
- D 1129 Terminology Relating to Water³
- D 1193 Specifications for Reagent Water³
- D 2777 Practice for Determination of Precision and Bias of Applicable Methods of Committee D-19 on Water³
- D 3370 Practices for Sampling Water³

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. 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, reactor fuel cladding material, and reactor structural components. It is, therefore, of significance as an indicator of the corrosion of reactor parts.

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 FePO₄ at a pH of 3.0.

6. Interferences

6.1 Samples of reactor origin will also contain iron-59 after other radioactive contaminants have been removed by anion exchange (see Fig. 1). Iron-59 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. The large difference in the energies of their characteristic decay emissions makes it possible to determine appropriate factors to correct for the iron-59 spectral cross-talk in the iron-55 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

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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² Currie, L., "Limits for Qualitative Detection and Quantitative 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.

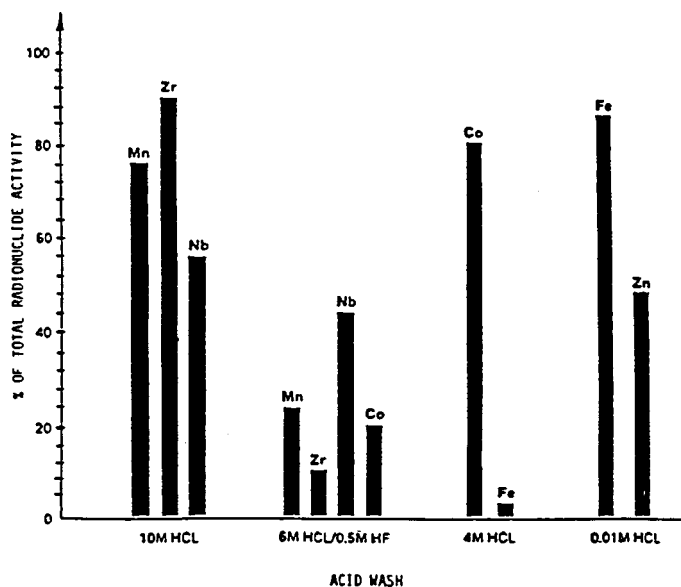


FIG. 1 Percent of Total Radionuclide Activity Removed Per Acid Wash

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 efficiency and the iron-59 cross-talk in the iron-55 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—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 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 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.

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*—AGI-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. Pump outlet tubing, approximately 30 in. in length.

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

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 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 M.

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 M.

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 M.

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 Pharmacopoeia 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 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.