Designation: D4922 – 21

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.

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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.* For a specific hazard statement, see Section 9.

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

2. Referenced Documents

2.1 *ASTM Standards:*[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 Flowing Process Streams
D5847 Practice for Writing Quality Control Specifications for Standard Test Methods for Water Analysis
D7282 Practice for Setup, Calibration, and Quality Control of Instruments Used for Radioactivity Measurements
D7902 Terminology for Radiochemical Analyses

3. Terminology

3.1 *Definitions*—For definitions of terms used in this test method, refer to Terminologies D1129 and D7902. For terms not defined in this test method or in Terminologies D1129 or D7902, 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,

[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.

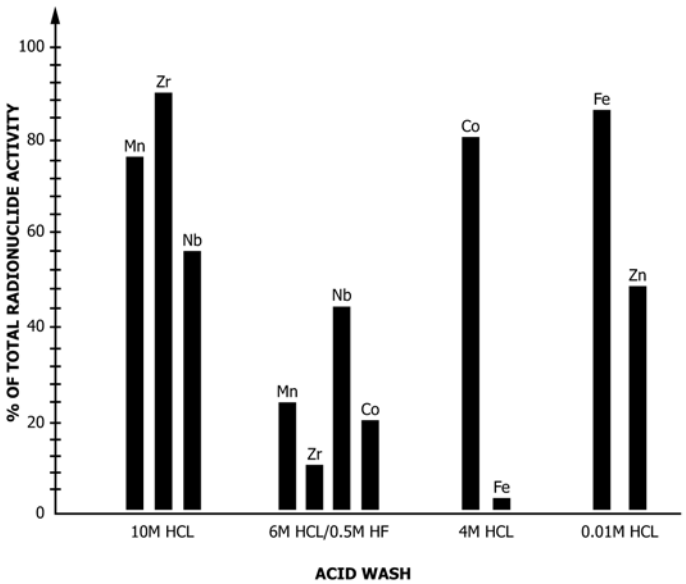

FIG. 1 Percent of Total Radionuclide Activity Removed Per Acid Wash

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 ^{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 yield. 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]

[5] *ACS Reagent Chemicals, Specifications and Procedures for Reagents and Standard-Grade Reference Materials*, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

[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.