



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 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.