Designation: D4785 – 20

Standard Test Method for Low-Level Analysis of Iodine Radioisotopes in Water[1]

This standard is issued under the fixed designation D4785; 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 quantification of low levels of radioactive iodine in water by means of chemical separation and counting with a high-resolution gamma ray detector. Iodine is chemically separated from a 4 L water sample using ion exchange and solvent extraction and is then precipitated as cuprous iodide for counting.

1.2 The values stated in SI units are to be regarded as standard. The values given in parentheses after SI units are provided for information only and are not considered standard.

1.3 *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 specific hazard statements, see 8.16, 8.17, 8.18, Section 9, and 13.2.11.

1.4 *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:*[2]

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
D3648 Practices for the Measurement of Radioactivity
D3649 Practice for High-Resolution Gamma-Ray Spectrometry of Water
D4448 Guide for Sampling Ground-Water Monitoring Wells
D5847 Practice for Writing Quality Control Specifications for Standard Test Methods for Water Analysis
D6001 Guide for Direct-Push Groundwater Sampling for Environmental Site Characterization
D7282 Practice for Set-up, Calibration, and Quality Control of Instruments Used for Radioactivity Measurements
D7902 Terminology for Radiochemical Analyses

2.2 *Other Documents:*

ANSI N42.22 Traceability of Radioactive Sources to the National Institute of Standards and Technology (NIST) and Associated Instrument Quality Control[3]
BIPM-5 Decay Data Evaluation Project (DDEP)[4]
NUDAT2[5]

3. Terminology

3.1 *Definitions:*

3.1.1 For definitions of terms used in this standard, refer to Terminology D1129.

3.1.2 For definitions of terms used in this standard relating to radiochemical analysis, refer to Terminology D7902.

4. Summary of Test Method

4.1 Sodium iodide is added as a carrier prior to performing any chemical separations. The samples undergo an oxidation-reduction process to ensure exchange between the carrier and the radioactive iodide. Hydroxylamine hydrochloride and sodium bisulfite are added to convert all the iodine to iodide which is then removed by anion exchange. Subsequent elution of the iodide is followed by oxidation-reduction to elemental iodine. The elemental iodine is purified by solvent extraction, reduced to iodide, and precipitated as cuprous iodide. The chemical yield is determined from the net mass of recovered iodide carrier.

[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] 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.

[3] Available from Institute of Electrical and Electronics Engineers, Inc. (IEEE), 445 Hoes Ln., Piscataway, NJ 08854-4141, http://www.ieee.org.

[4] Available from BIPM, Sèvres Cedex, France, https://www.bipm.org.

[5] Available from National Nuclear Data Center at Brookhaven National Laboratory, W Princeton Ave, Yaphank, NY 11980, http://www.nndc.bnl.gov.

5. Significance and Use

5.1 This test method was developed for measuring low levels of radioactive iodine in water. The results of the test may be used to determine if the concentration of several radioisotopes of iodine in the sample exceeds the regulatory limits for drinking water. With suitable counting techniques, sample size, and counting time, a detection limit of less than 0.037 Bq/L (1 pCi/L) is attainable by gamma-ray spectrometry.

5.2 This test method is intended for the analysis of iodine radioisotopes with half-lives greater than 2 hours, which include ^{121}I, ^{123}I, ^{124}I, ^{125}I, ^{126}I, ^{129}I, ^{130}I, ^{131}I, ^{132}I, ^{133}I, and ^{135}I. The test method was tested according to Practice D2777 using only ^{131}I. The user of this test method is responsible for determining applicability, bias, and precision for the measurement of other iodine radioisotopes.

6. Interferences

6.1 Stable iodine in the sample will interfere with the chemical yield determination. One milligram of ambient iodine would produce a bias of about –4 %.

6.2 There are numerous characteristic iodine X-rays at and below 33.6 keV which are indicative of iodine, but not of a specific radioisotope of iodine. Only use discrete gamma energy lines at and above 35.5 keV for identification and quantification of iodine radioisotopes.

7. Apparatus

7.1 *Analytical Balance,* readable to 0.1 mg.

7.2 *Flexible Polyvinyl Chloride (PVC) Tubing,* 6.35 mm (¼ in.) outside diameter, 1 m length.

7.3 *Gamma-Ray Spectrometry System*—High resolution gamma spectrometer (high purity germanium or equivalent) with a useful energy range of approximately 30 keV to 1800 keV (see Practice D3649 and Practice D7282, Sections 8, and 9.2).

7.4 *Glass Fiber Filter Paper,* 11.5 cm diameter.

7.5 *Ion Exchange Column,* glass tube, 35 ± 2 mm inside diameter, 150 mm length, fitted with No. 8 one-hole rubber stoppers and perforated disk.

7.6 *Membrane Filters,* 0.45 µm (or 0.4 µm) pore size, 25 mm diameter, with suitable filter holder and vacuum filter flask.

7.7 *Peristaltic Tubing Pump,* variable speed, fitted with vinyl or silicone tubing.

7.8 *pH Meter.*

7.9 *Sintered Glass Filter,* Büchner funnel, 150 mL size, medium or coarse porosity with suitable one-hole stopper and vacuum filter flask.

7.10 *Vacuum Desiccator.*

7.11 *Vortex Mixer.*

8. Reagents and Materials

8.1 *Purity of Reagents*—Reagent grade chemicals shall be used in 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.[6] Other grades may be used provided they are of sufficiently high purity to permit their use without reducing the accuracy of the determination. Some reagents, even those of high purity, may contain naturally-occurring radioactivity, such as isotopes of uranium, radium, actinium, thorium, rare earths, potassium compounds, or artificially produced radionuclides, or combinations thereof. Consequently, when such reagents are used in the analysis of low-radioactivity samples, the activity of the reagents should be determined under analytical conditions that are as close as practicable to those used for the test sample. The activity contributed by the reagents should be accounted for and applied as a correction when calculating the test sample result.

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

8.3 *Ammonium Hydroxide* (sp gr 0.90)—Concentrated ammonium hydroxide (NH_4OH).

8.4 *Ammonium Hydroxide* (1.4 *M*)—Mix one volume of concentrated NH_4OH with nine volumes of water.

8.5 *Anion Exchange Resin*—Strongly basic, styrene, quaternary ammonium salt, 20–50 mesh, chloride form, Dowex[7] 1-X8, or equivalent.

8.6 *Cuprous Chloride Solution* (approximately 10 mg CuCl/mL)—Dissolve 10 g of CuCl (99.99 %) in 26 mL of concentrated HCl (sp gr 1.19). Add this solution to 1000 mL of NaCl solution (1 *M*) slowly with continuous stirring. Add a small quantity of metallic copper (for example, 5 to 10 copper metal shot) to the solution for stabilization.[8] Store the CuCl salt in a desiccator.

8.7 *Hydrochloric Acid* (sp gr 1.19)—Concentrated hydrochloric acid (HCl).

8.8 *Hydrochloric Acid Solution* (0.3 *M*)—Dilute 25 mL of concentrated HCl to 1000 mL with water.

8.9 *Hydroxylamine Hydrochloride* (NH_2OH:HCl)—Crystals.

8.10 *Iodide Carrier Solution* (25 mg I/mL)—Dissolve 14.76 g of NaI in approximately 80 mL of water in a 500-mL volumetric flask and dilute to volume. Standardize using the procedure in Section 10.

8.11 *Iodine-131 Standard Solution*—Solution traceable to the SI through a national metrology institute (NMI), such as

[6] *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.

[7] Dowex is a trademark of Dow Chemical Company, Midland, MI.

[8] CuCl solution is not stable. It can be oxidized to the Cu^{+2} state by air after a period of time, when the solution will turn dark green. If this happens, prepare a fresh solution. The shelf life of the solution can be extended by displacing the air over the remaining solution with nitrogen or argon gas after each use and then closing the container promptly.