



Designation: D 4785 – 00a

Standard Test Method for Low-Level Iodine-131 in Water¹

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1. Scope

1.1 This test method covers the quantification of low levels of iodine-131 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 are provided for information purposes only.

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 and health practices and determine the applicability of regulatory limitations prior to use.* For specific hazard statements, see [Note 2](#), [Note 3](#), [Note 8](#), and [Section 9](#).

2. Referenced Documents

2.1 ASTM Standards:

[D 1129](#) Terminology Relating to Water²

[D 1193](#) Specification for Reagent Water²

[D 2777](#) Practice for Determination of Precision and Bias of Applicable Test Methods of Committee D-19 on Water²

[D 3370](#) Practices for Sampling Water from Closed Conduits²

[D 3648](#) Practices for Measurement of Radioactivity³

[D 3649](#) Practice for High-Resolution Gamma-Ray Spectrometry of Water³

3. Terminology

3.1 *Definitions*—For definitions of terms used in this test method, refer to Terminology [D 1129](#).

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 followed by oxidation-reduction yields 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 recovery of the iodide carrier.

5. Significance and Use

5.1 This test method was developed for measuring low levels of iodine-131 in water. The results of the test may be used to determine if the concentration of iodine-131 in the sample exceeds the regulatory statutes for drinking water. With a suitable counting technique, sample size, and counting time, a detection limit of less than 0.037 Bq/L (1 pCi/L) is attainable by gamma-ray spectroscopy.

6. Interferences

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

7. Apparatus

7.1 *Analytical Balance*, readable to 0.1 mg.

7.2 *Flexible Polyvinyl Chloride (PVC) Tubing*, 6.35 mm ($1/4$ in.) outside diameter, 1-m length.

7.3 *Gamma-Ray Spectrometry System*—high resolution using a high-purity germanium or lithium-drifted germanium detector (see Practice [D 3649](#)).

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 stoppers and perforated disk.

7.6 *Membrane Filters*, 0.4 or 0.45- 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*, Buchner funnel, 150-mL size, medium or coarse porosity with suitable one-hole stopper and vacuum filter flask.

7.10 *Vacuum Desiccator*.

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 *Annual Book of ASTM Standards*, Vol 11.01.

³ *Annual Book of ASTM Standards*, Vol 11.02.

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.⁴ Other grades may be used provided they are of sufficiently high purity to permit their use without reducing the accuracy of the determination.

8.2 *Purity of Water*— Unless otherwise indicated, reference to water shall be understood to mean reagent water conforming to Specification **D 1193**, Type III.

8.3 *Radioactive Purity*—Radioactive purity shall be such that the measured radioactivity of blank samples does not exceed the calculated probable error of the measurement.

8.4 *Ammonium Hydroxide (sp gr 0.90)*—Concentrated ammonium hydroxide (NH₄OH).

8.5 *Ammonium Hydroxide (1.4 M)*—Mix one volume of concentrated NH₄OH (sp gr 0.90) with nine volumes of water.

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

8.7 *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.⁵ Store the CuCl in a desiccator.

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

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

8.10 *Hydroxylamine Hydrochloride (NH₂OH:HCl)*—Crystals.

8.11 *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.

8.12 *Iodine-131 Standardizing Solution*—National Institute of Standards and Technology (NIST), traceable solution with a typical concentration range from 1 to 10 kBq/mL.

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

8.14 *Nitric Acid (1.4 M)*—Mix 1 volume of HNO₃ (sp gr 1.42) with 10 volumes of water.

8.15 *Sodium Bisulfite Solution (2 M)*—Dissolve 104.06 g of NaHSO₃ in approximately 300 mL of water in a 500-mL volumetric flask and dilute to volume.

8.16 *Sodium Chloride Solution (1 M)*—Dissolve 58.45 g of NaCl in approximately 500 mL of water in a 1000 mL volumetric flask and dilute to volume.

8.17 *Sodium Hydroxide Solution (12.5 M)*—Dissolve 500 g of NaOH in 800 mL of water and dilute to 1 L.

NOTE 1—**Caution:** The dissolution of sodium hydroxide may produce excessive heat.

8.18 *Sodium Hypochlorite (NaOCl)*—Approximately 5 to 6 %. Commercially available bleach is acceptable.

NOTE 2—**Warning:** Acidification of NaOCl produces toxic chlorine gas and must be handled in a fume hood.

8.19 *Toluene.*

NOTE 3—**Warning:** Toluene is a carcinogen and must be handled and disposed of in an approved manner.

9. Hazards

9.1 Due to the potential health effects from handling these compounds, the steps utilizing NaOCl and toluene must be carried out in a fume hood. Toluene is a carcinogen and acidification of NaOCl liberates toxic Cl₂ gas.

10. Standardization of Iodide Carrier

10.1 Pipet 1.0 mL of iodide carrier reagent into each of five 100-mL centrifuge tubes containing 50 mL of deionized water.

10.2 Add 0.1 mL of 2 M NaHSO₃ to each solution and stir vigorously using a vortex mixer. Add 5.0 mL of freshly prepared CuCl solution.

10.3 Using a pH meter, check the pH of each solution and adjust the pH to between 2.40 to 2.50 with 0.3 M HCl or 1.4 M NH₄OH.

10.4 Place each solution in a warm (approximately 50 to 60°C) water bath for 5 to 10 min stirring occasionally.

10.5 Rinse each CuI precipitate onto a separate preweighed 0.45-μm membrane filter mounted in a vacuum filtration assembly. Rinse the walls of the filter holder with approximately 50 mL of water.

10.6 Dry all samples in a vacuum desiccator for a minimum of 60 min or to constant weight. Remove and weigh the filter and precipitate. Record all data.

10.7 Determine the net weight of each CuI precipitate.

10.8 Use the mean of the five weights for the standard weight. The relative standard deviation of the mean should not exceed 0.025.

11. Calibration of High-resolution Gamma-ray Spectroscopy System

11.1 Prepare a set of three calibration sources in accordance with the following:

11.1.1 Add 50 mL of reagent water to a 150-mL beaker.

11.1.2 Quantitatively add approximately 3.7 kBq of NIST-traceable iodine-131 solution and swirl gently to mix.

11.1.3 Follow 13.3.1-13.3.6.

11.1.4 Count the calibration sources individually and accumulate 20 thousand to 50 thousand counts in the 365.48-kV photopeak.

⁴ *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. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

⁵ 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.

11.1.5 The absolute detection efficiency, E for the 364.5 keV gamma-ray, based on one calibration source is calculated as follows:

$$E = C_r / (A_c * I) \quad (1)$$

where:

- C_r = the net count rate for the 364.5 keV gamma-ray line of one of the reference iodine-131 sources in the same chemical form and geometrical arrangement as the sample,
- A_c = the activity in becquerels of that calibration source at the midpoint of the counting period,
- I = the intensity for the 364.8 keV gamma-ray line, 0.812.⁶

Use the mean of three results. The relative standard deviation of the mean should not exceed 5.0 %.

12. Sampling

12.1 Collect a sample in accordance with Practice D 3370.

13. Procedure

13.1 Sample Preparation:

13.1.1 Measure or weigh 4 L of the sample into a suitable plastic container. While stirring, add 1.0 mL of iodide carrier and 5.0 mL of 5 to 6 % NaOCl. Stir approximately 3 to 5 min.

13.1.2 Add 2.0 g of NH₂OH:HCl, stir, and add 5.0 mL of 2 M NaHSO₃. Adjust the pH to 6.5 using 12.5 M NaOH or 1.4 M HNO₃. Stir for 30 min.

13.1.3 Filter the sample through a glass fiber filter and discard the residue.

13.2 Anion Exchange Separation:

13.2.1 Slurry 100 mL (wet volume) of washed anion exchange resin into a 35-mm inside diameter glass column fitted at the lower end with a one-hole rubber stopper, perforated disk, and a short length of 5-mm glass tubing connecting to the inlet side of the peristaltic pump (see Fig. 1).

NOTE 4—The resin should be washed with water until the wash water shows no change in pH. This is most conveniently done by batch sequential washing of a relatively large quantity of resin and storing the washed resin as a slurry.

13.2.2 Leave approximately 25 mL of water on top of the resin bed and insert a glass wool plug, being careful not to touch the resin. Place a one-hole rubber stopper, fitted with a short length of 5-mm glass tubing, in the top of the column and connect it to a 1-m length of flexible PVC tubing.

NOTE 5—If a peristaltic pump is not available, the sample can be passed through the column by gravity flow using an appropriate reservoir.

13.2.3 Pump approximately 100 mL of water through the resin-packed column and check the final effluent pH with pH paper. Repeat the wash if the test indicates residual activity. Be sure to leave approximately 25 mL of water standing on top of the resin bed in the glass column or be certain that the feed tube remains full of water in order to prevent air from entering the resin bed before the sample reaches the column.

IODINE PROCEDURE: ION EXCHANGE

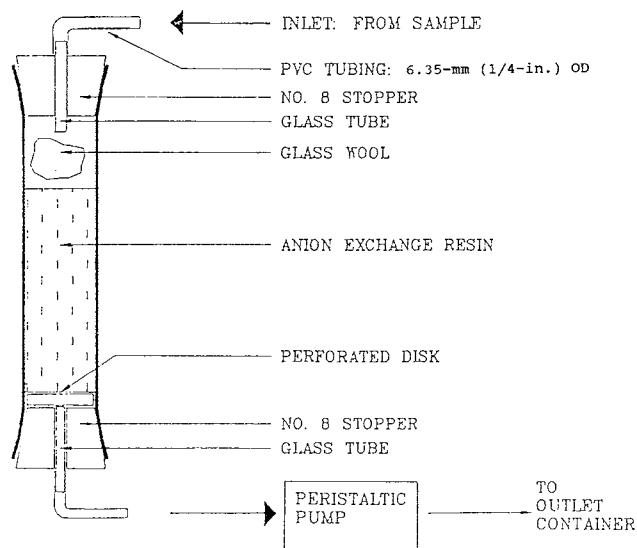


FIG. 1 Iodine Procedure: Ion Exchange

13.2.4 Place the flexible PVC inlet tube into the sample container. It may be desirable to attach a 250 to 300-mm length of glass tubing to the sample container end of the PVC to facilitate removal of the sample from the container.

13.2.5 Place the pump discharge tube into a beaker or bottle to collect the column effluent.

13.2.6 Start the pump and vary the speed control to give a flow rate of 40 mL/min.

NOTE 6—It is necessary to calibrate the variable speed control of the peristaltic pump by timing the flow of known liquid quantities at each setting of the control.

13.2.7 When the sample container is empty, remove the upper stopper, and glass wool plug from the top of the column and pour the resin into a 600-mL beaker.

13.2.8 Wash the resin with three successive 100-mL portions of water. Stir briefly and allow the resin to settle to the bottom of the beaker. Decant and discard the wash water.

13.2.9 Place a magnetic stirring bar in the beaker with the washed resin and add 250 mL of 5 to 6 % NaOCl. Place the beaker on a magnetic stirrer and stir for 10 min. Allow the resin to settle. Filter the NaOCl solution by suction through a glass fiber filter supported in a sintered glass Büchner-type funnel. Save the filtrate.

NOTE 7—The NaOCl solution oxidizes the iodide absorbed on the resin to periodate which is released to the solution.

13.2.10 Add 250 mL of fresh 5 to 6 % NaOCl solution to the resin remaining in the beaker and stir for another 10 min. Allow the resin to settle and filter the NaOCl solution into the Büchner funnel. Save the filtrate.

13.2.11 Add 50 mL of water solution to the resin remaining in the beaker and stir for 5 min. Filter the solution and resin into the Büchner funnel and rinse the resin thoroughly with water. Save the filtrate. Transfer the NaOCl solution from this step and 13.2.10 and 13.2.11 into a 2000-mL beaker and discard the resin.

NOTE 8—**Precaution:** Chlorine Gas Released. Acidification of the

⁶ Firestone, R.B. and Shirley, V.S., *Table of the Isotopes*, (Eighth Edition), John Wiley and Sons, Inc., New York, 1996.