Designation: D4785 – 08 (Reapproved 2013)ε1

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.

ε[1] NOTE—Warning notes were editorially updated throughout in June 2013.

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 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 8.17, 8.18, 8.19, Section 9, and 13.2.11.

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 Closed Conduits
D3648 Practices for the Measurement of Radioactivity
D3649 Practice for High-Resolution Gamma-Ray Spectrometry of Water
D5847 Practice for Writing Quality Control Specifications for Standard Test Methods for Water Analysis
D3856 Guide for Management Systems in Laboratories Engaged in Analysis of Water

3. Terminology

3.1 *Definitions*—For definitions of terms used in this test method, refer to Terminology D1129.

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 recovery 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 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 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. This method was tested for ^{131}I . Other iodine radioisotopes should behave in an identical manner in this procedure. However, other iodine radioisotopes have not been tested according to Practice D2777. The user of this method is responsible for determining applicability, bias, and precision for the measurement of other iodine radioisotopes using this method.

5.2 This procedure addresses 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.

6. Interferences

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

[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.

6.2 There are numerous characteristic iodine X-rays at and below 33.6 keV which are indicative of iodine, but not a specific radioisotope of iodine. It is recommended that only discreet gamma energy lines at and above 35.5 keV be used 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).

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,* 0.4 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,* 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.[3] 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 D1193, 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 (NH_4OH).

8.5 *Ammonium Hydroxide* (1.4 *M*)—Mix one volume of concentrated NH_4OH (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.[4] 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* (NH_2OH: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 standardizing body such as 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 HNO_3.

8.14 *Nitric Acid* (1.4 *M*)—Mix 1 volume of concentrated HNO_3 (sp gr 1.42) with 10 volumes of water.

8.15 *Sodium Bisulfite Solution,* (2 *M*)—Dissolve 104.06 g of $NaHSO_3$ 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. (**Warning—** The dissolution of sodium hydroxide may produce excessive heat.)

8.18 *Sodium Hypochlorite* (NaOCl)—Approximately 5 to 6 %. Commercially available bleach is acceptable. (**Warning—**Acidification of NaOCl produces toxic chlorine gas and must be handled in a fume hood.)

8.19 *Toluene.* (**Warning—**Toluene is a carcinogen and must be handled and disposed of in an approved manner.)

8.20 *Calibration Standard(s)*—Known amounts of ^{125}I, ^{129}I, and ^{131}I are used for calibration when determining these radionuclides. A mixed-gamma standard, for example, ^{241}Am, ^{109}Cd, ^{57}Co, ^{141}Ce, ^{113}Sn, ^{137}Cs, ^{88}Y, and ^{60}Co, is used for calibration over an extended energy range as required for the

[3] *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 *Annual 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.

[4] 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.

determination of additional radioisotopes of iodine. These standards should be mounted on the filter as described in 7.6. The known amounts of the radionuclides must be traceable to a national standardizing body such as NIST in the USA. The standard may be prepared by the laboratory performing this method or by a commercial supplier of such standards. Alternate radionuclides may be used for calibration provided that they have gamma ray energies covering the range of interest for the iodine radionuclides to be analyzed.

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 Cl_2 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* $NaHSO_3$ 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* NH_4OH.

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 Accumulate an energy spectrum using the calibration standard (8.20) traceable to a national standards body, in the geometrical position representing that of the samples to be analyzed. Accumulate sufficient net counts (total counts minus the Compton baseline) in each full-energy gamma-ray peak of interest to obtain a relative standard counting uncertainty of ≤1 %.

11.2 Using the gamma-ray emission data from the calibration standard and the peak location data from the calibration spectrum, establish the energy per channel relationship (energy calibration) as:

$$En = Offset + (Ch \times Slope) \quad (1)$$

where:

En = peak energy (keV),
Offset = energy offset for the energy calibration equation (keV),
Ch = peak location channel number, and
Slope = energy calibration equation slope (keV per channel).

NOTE 1—Most modern spectroscopy software packages perform this calculation, and may include higher-order polynomial terms to account for minor non-linearity in the energy calibration.

11.3 Using the gamma emission data from the calibration standard and the peak resolution data from the calibration spectrum, establish the resolution versus energy relationship (energy calibration) as:

$$FWHM = Offset + (Ch \times Slope) \quad (2)$$

where:

FWHM = full width of the peak at one-half the maximum counts in the centroid channel (keV),
Offset = width offset for the resolution calibration equation (keV),
En = peak energy (keV), and
Slope = resolution calibration equation slope (keV/keV).

NOTE 2—Most modern spectroscopy software packages perform this calculation, and may include higher-order polynomial terms to account for non-linearity in the resolution calibration.

11.4 For each gamma-ray photopeak, calculate the full-energy peak efficiency, ε_f, as follows:

$$\epsilon_f = \frac{R_n}{R_\gamma \times DF} \quad (3)$$

where:

ε_f = full-energy peak efficiency (counts per gamma ray emitted),
R_n = net gamma-ray count rate in the full-energy peak of interest, counts per second (s^{-1}),
R_γ = gamma-ray emission rate, in gamma-rays per second (s^{-1}), as of the reference date and time of the calibration standard,
DF = decay factor for the calibrating radionuclide, $e^{-\lambda(t_1-t_0)}$,
λ = (ln 2) / $t_{1/2}$,
$t_{1/2}$ = half-life of calibrating radionuclide (half-life unit must match that used for the time difference, $t_1 - t_0$),
t_0 = reference date and time of the calibration standard, and
t_1 = midpoint of sample count (date and time).

11.5 Many modern spectrometry systems are computerized and the determination of the gamma-ray detection efficiencies is performed automatically at the end of an appropriate counting interval. Refer to the manufacturer instructions for specific requirements and capabilities.

11.6 Plot the values for the full-energy peak efficiency (as determined in Section 11.5) versus gamma-ray energy. Compare the efficiency curve to the typical efficiency curve for the detector type. The curve should be smooth, continuous and have a shape similar to the detector being used. The plot will allow the determination of efficiencies at energies throughout the range of the calibration energies and will show that the algorithms used in computerized systems are providing valid efficiency calibrations. Select the fit that has the best 95 % confidence limit around the fitted curve, has all data points

within ±8 % of the value of the fitted curve, or both. This is accomplished by calculating the bias between the actual efficiency and the efficiency calculated with the fitted curve.

11.7 Save or store the values of energy versus efficiency for future reference, to be used in the calculation of activity for each iodine nuclide in Section 14.

12. Sampling

12.1 Collect a sample in accordance with Practice D3370 or other approved procedure.

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 NH_2OH:HCl, stir, and add 5.0 mL of 2 *M* $NaHSO_3$. Adjust the pH to 6.5 using 12.5 *M* NaOH or 1.4 *M* HNO_3. 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 3—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.

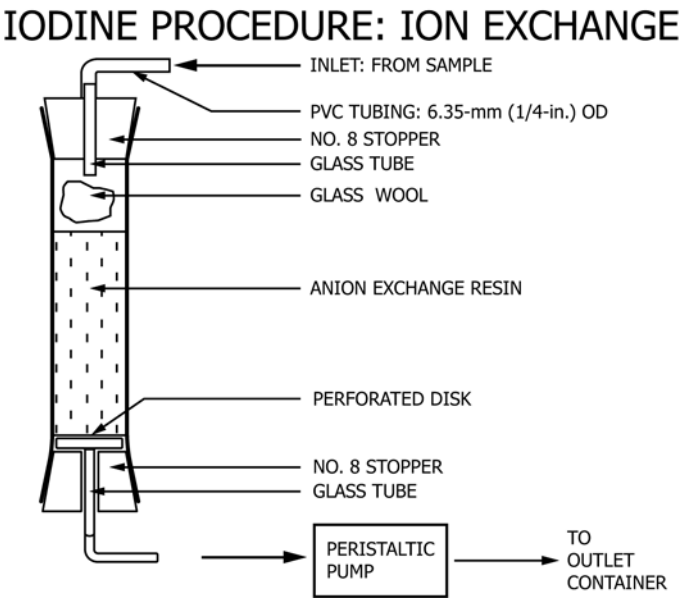

FIG. 1 Iodine Procedure: Ion Exchange

NOTE 4—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.

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 5—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.

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, step 13.2.9, and 13.2.10 into a 2000-mL beaker and discard the resin. (**Warning—**Chlorine gas released. Acidification of the residual NaOCl decomposes it, releasing chlorine gas (green color) which is highly toxic. This destroys residual NaOCl which would interfere in the reduction of iodate to elemental iodine. All subsequent steps through 13.2.16 are to be performed in a well-ventilated fume hood.)

13.2.12 In an adequate fume hood, slowly add concentrated HNO_3 (sp gr 1.42) to the NaOCl solution from 13.2.11 until the pH is brought to 1. (Approximately 45 mL of HNO_3 are required.) Stir magnetically until the bulk of the chlorine gas has evolved from the solution.

13.2.13 Pour the acidified solution into a 1000-mL separatory funnel containing 100 mL of toluene and 2 g of $NH_2OH{\cdot}HCl$.

NOTE 6—Hydroxylamine hydrochloride is a mild reducing agent capable of reducing iodate to iodine (I°). Iodine is preferentially soluble in the toluene phase and can be separated by solvent extraction. When

$NH_2OH{\cdot}HCl$ is added, some gas evolution will occur and the solution color will darken (straw to amber) due to the formation of the complex ions I_3^- (a combination of I_2 and I^-).

13.2.14 Shake the separatory funnel for a total of 2 min, relieving the pressure occasionally. Allow the phases to separate. Drain off the lower aqueous phase into a second clean 1000-mL separatory funnel containing 2 g of hydroxylamine hydrochloride, and 100 mL of toluene. Allow a few drops of the toluene to drain off with the aqueous phase. Save the toluene in the first separatory funnel.

NOTE 7—It is necessary to relieve the pressure at the beginning of shaking and a few times during the 2-min shaking. As the iodine transfers to the toluene phase, the dark color of the aqueous phase will be replaced by a violet color in the toluene due to dissolved elemental iodine.

13.2.15 Shake the second separatory funnel for 2 min, relieving the pressure occasionally. Allow the phases to separate, and discard the lower aqueous phase (a third extraction can be performed if desired). Combine this toluene with the first toluene fraction in the first separatory funnel.

13.2.16 To the combined toluene in the separatory funnel, add 50 mL of water containing 0.1 mL of 2 *M* $NaHSO_3$. Shake for 2 min. Allow the phases to separate and drain off the lower aqueous phase into a 100-mL centrifuge tube until the toluene phase enters the stopcock bore. Discard the toluene in an appropriate hazardous waste container.

NOTE 8—The $NaHSO_3$ reduces the iodine to iodide which is not soluble in toluene. The color in the toluene fades rapidly as the iodine is extracted into the aqueous phase. Remove any remaining toluene drops in the centrifuge tube with a disposable transfer pipet.

13.3 *CuI Precipitation and Mounting:*

13.3.1 Add 5 mL of the CuCl solution and stir thoroughly. Adjust the pH to between 2.40 to 2.50 with 0.3 *M* HCl or 1.4 *M* NH_4OH solution as required.

NOTE 9—The proper pH during the CuI precipitation is crucial. A pH of less than 2.4 causes incomplete iodide precipitation. A pH value of greater than 2.6 will cause a yellow to green color to appear in the precipitate and the coprecipitation of some form of the excess copper, resulting in artificially high chemical recoveries.

13.3.2 Allow the precipitate to stand with occasional mixing for 5 to 10 min.

NOTE 10—Paragraphs 13.3.3 through 13.3.6 presuppose that the radioactivity will be determined using gamma-ray spectrometry. Suitable adjustments may be made if beta-gamma coincidence counting is used (see Practice D3648).

13.3.3 Mount a preweighed membrane filter (0.4 or 0.45-µm pore size or equivalent) on a suction filtration apparatus and filter the CuI precipitate. Wash the walls of the filter holder and the precipitate with water.

13.3.4 Place the sample into the vacuum dessicator and dry under vacuum for a minimum of 60 min or to constant weight. Remove the sample, weigh it, and record the weight.

13.3.5 Mount the sample for counting in a reproducible geometrical arrangement for which the gamma-ray spectrometry system has been, or will be, calibrated for detection efficiency.

13.3.6 Using the high resolution gamma-ray spectrometry system, determine the net counting rate for the gamma-ray energy lines of each iodine nuclide to be assayed. Listed in Table 1 are recommended gamma energy lines and gamma emission fractions obtained from the National Nuclear Data Center.[5]

14. Calculation

NOTE 11—The following calculations assume that there are no interfering photopeaks in the background or sample spectrum, and none of the iodine photopeaks are found in multiplets.

14.1 For each photopeak the net count rate, R_{net}, and its standard uncertainty, u (R_{net}), are given by the following equations:

$$R_{net} = R_p - \left(R_b \times \frac{n_p}{n_b}\right) = \frac{C_p - (C_b \times n_p/n_b)}{t_s} \quad (4)$$

$$u(R_{net}) = \frac{\sqrt{C_p + (C_b \times n_p^2/n_b^2)}}{t_s} \quad (5)$$

where:

R_p = photopeak count rate (s^{-1}),
R_b = Compton baseline count rate (s^{-1}),
C_p = total photopeak counts,
C_b = total Compton baseline counts,
t_s = sample count time (s^{-1}),
n_p = number of channels in the photopeak, and
n_b = number of channels used in the baseline measurement.

[5] National Nuclear Data Center, Information extracted from NUDAT Data Base, available at: http://www.nndc.bnl.gov/nudat2.

TABLE 1 Nuclear Decay Data for Iodine Radioisotopes

Iodine Nuclide	Gamma Energy (keV)	Gamma Fraction	Half-Life
^{121}I	212.2	0.843	127.2 min
	532.08	0.0610	
^{123}I	158.97	0.833	13.27 h
^{124}I	511	0.4616	4.176 d
	602.72	0.605	
	722.78	0.0998	
	1691.02	0.1041	
^{125}I	35.49	0.06681	59.41 d
^{126}I	388.63	0.340	13.11 d
	666.33	0.331	
^{129}I	37.6	0.075	1.57×10^7 a
^{130}I	418.01	0.342	12.36 h
	536.09	0.99	
	668.54	0.996	
	739.48	0.820	
	1157.47	0.113	
^{131}I	284.3	0.0614	8.02 d
	364.49	0.817	
	636.99	0.0717	
^{132}I	522.65	0.16	137.7 min
	630.19	0.1330	
	667.72	0.987	
	772.6	0.756	
	812	0.0550	
	954.55	0.1760	
	1398.57	0.0701	
^{133}I	529.87	0.87	20.8 h
	875.33	0.0451	
^{135}I	546.56	0.072	6.57 h
	836.8	0.0673	
	1038.76	0.0801	
	1131.51	0.2274	
	1260.41	0.289	
	1457.56	0.0873	
	1678.03	0.0962	
	1791.2	0.0777	

14.2 When only one photopeak is used in the measurement (for example, the 364 keV photopeak of ^{131}I), the activity concentration, AC, is given by:

$$AC = \frac{R_{net}}{\varepsilon_f \times I_\gamma \times V \times Y \times e^{-\lambda t}} \quad (6)$$

where:

R_{net} = net count rate for the photopeak of interest (Eq 4),
ε_f = absolute detection efficiency of the gamma-ray spectrometer for the photopeak of interest (Eq 3),
λ = decay constant for the iodine isotope of interest, equal to (ln 2) / $t_{1/2}$, where the half-life, $t_{1/2}$, comes from Table 1,
t = elapsed time from sample collection to midpoint of the sample counting period, in days,
V = volume of sample aliquant (L),
Y = fraction of the total iodine carrier recovered from the sample, which is the weight of the sample determined in 13.3 divided by the weight of standardized carrier in accordance with 10.8,
I_γ = gamma-ray emission probability from Table 1.

14.3 When Eq 6 is used to calculate AC, the standard counting uncertainty of AC (uncertainty due to counting statistics only) is given by:

$$u_{cC}(AC) = \frac{u(R_{net})}{\varepsilon_f \times I_\gamma \times V \times Y \times e^{-\lambda t}} \quad (7)$$

where $u(R_{net})$ comes from Eq 5.

14.4 Calculate the combined standard uncertainty of AC as follows:

$$u_c(AC) = \quad (8)$$

$$\sqrt{u_{cC}^2(AC) + AC^2 \times (u_r^2(\varepsilon_f) + u_r^2(I_\gamma) + u_r^2(V) + u_r^2(Y) + u_r^2(\ldots))}$$

where:

$u_{cC}^2(AC)$ = standard counting uncertainty of AC, from Eq 7,
$u_r(\cdot)$ = relative standard uncertainty of the measured quantity in parentheses,
$u_r(\ldots)$ = relative standard uncertainty due to other causes identified by the user.

14.5 *Expanded Uncertainty (U)*—The combined standard uncertainty, $u_c(AC)$, may be multiplied by a coverage factor, k, to obtain an expanded uncertainty, U.

$$U = k \times u_c(AC) \quad (9)$$

The coverage factor k = 2 is often used. Assuming the measurement process produces normally distributed results, this coverage factor gives a coverage interval, $AC \pm U$, with a coverage probability of approximately 95 %.

14.6 Calculate the critical activity concentration (L_C) for each sample measurement as follows:

$$L_C = \frac{1.645\sqrt{\frac{R_b}{t_s} \times (F + F^2)}}{\varepsilon_f \times I_\gamma \times V \times Y \times e^{-\lambda t}} \quad (10)$$

where $F = n_p/n_b$, and the other symbols are as defined above. To decide whether the iodine isotope of interest is clearly present in the sample, compare the measured activity concentration, AC, to L_C.

NOTE 12—The following calculation for MDC requires the determination of the background area. Most automated gamma-ray software performs this calculation using a similar equation. If the software does not provide this calculation, the MDC calculation may be performed manually using a printout of channel contents.

14.7 When the detection criterion of 14.6 is used, the *a priori* minimum detectable activity concentration (MDC) may be calculated as follows:

$$MDC = \frac{2.71 + 3.29\sqrt{R_b \times t_s \times (F + F^2)}}{t_s \times \varepsilon_f \times I_\gamma \times V \times Y \times e^{-\lambda t}} \quad (11)$$

15. Quality Control

15.1 In order to provide reasonable assurance that the analytical results obtained using this test method are valid and accurate within the confidence limits of the method, Quality Control (QC) samples are analyzed with each batch of samples undergoing analysis. Each batch should include not more than 20 samples excluding those used for QC purposes. Laboratory or project quality assurance plans may contain more restrictive process QC requirements. The following minimum QC procedures must be followed when running the test method:

15.2 *Internal Standard:*

15.2.1 As indicated in 13.1.1, an accurately added amount of stable iodide is used as a carrier in the determination of radioiodine in the sample.

15.2.2 The recovery of the stable iodide carrier will be calculated for each sample and associated QC samples. This recovery should be reported along with the reported analytical data. The relative standard uncertainty of the carrier recovery measurement should be less than 5 %. As noted in Section 10.8, the relative standard deviation of the mean of the five calibration samples shall not exceed 0.025.

15.3 *Calibration and Calibration Verification:*

15.3.1 Standards used in the method shall be traceable to a national standards laboratory (such as NIST or NPL).

15.3.2 The detector counting efficiency should be determined using at least three standards as noted in Section 11.

15.3.3 The detector efficiency shall be verified on a daily basis or prior to use.

15.4 *Initial Demonstration of Laboratory/Instrument/Analyst Capability:*

15.4.1 If a laboratory or analyst has not performed this test before or there has been a major change in the measurement system, for example, significant instrument change, new instrument, etc., a precision and bias study must be performed to demonstrate laboratory, analyst, or instrument capability. A significant change is defined as any change, repair, or alteration of any component in the system, which may be expected to affect the response of the measurement system.

15.4.2 Analyze seven replicates of a standard solution prepared from a independent reference material (IRM) containing iodine-131 activity sufficient to reduce counting uncertainty to 1 % or less at one sigma. The matrix used for the demonstration should represent a water sample typical for which the method will be used, for example, a surface water.

The total dissolved solids (TDS) of the matrix should approximate that which may be encountered in normal use. In addition interference radioisotopes should be included in the matrix because they can interfere in the determination of the radioisotope of interest. The interference radioisotopes should be included at a level of at least ten times the *a priori* MDC of the analysis.

15.4.3 Calculate the mean and standard deviation of the seven values and compare to the acceptable ranges of precision and mean bias of 10 % and ±15 %, respectively, based on a review of the collaborative study data. Test Method D5847 should be consulted on the manner by which precision and mean bias are determined from the initial demonstration study. The study should be repeated until the precision and bias are within the given limits.

15.4.4 Analyze three replicates of a blank (in ^{131}I) solution matrix. The matrix used for the demonstration should represent a water sample typical for which the method will be used, for example, surface water. The total dissolved solids (TDS) of the matrix should approximate that which may be encountered in normal use. In addition interference isotope or isotopes should be included in the matrix because they can interfere in the determination of radioisotope of interest. The interference radioisotopes should be included at a level of at least five times the *a priori* MDC of the analysis.

15.4.5 Calculate the iodine-131 activity for each of these three blank solutions. This study should be repeated until the iodine-131 result of each of the three blank solutions is below half the associated MDC for each of the analytes.

15.4.6 This method shall not be used for official samples until precision, bias, and blank requirements are met.

15.5 *Laboratory Control Sample (LCS):*

15.5.1 To ensure that the test method is within control limits, analyze an LCS with each batch of no more than 20 samples. The activity added to reagent water should be appropriate for the type of samples analyzed and allow sufficient precision to ensure a meaningful assessment of accuracy. The LCS must be taken through all the steps of the analytical method including sample preservation and pretreatment. The result obtained for the LCS should fall within the limit of ±25 % of the expected value.

15.6 *Method Blank:*

15.6.1 Analyze a reagent water test blank with each batch of no more than 20 samples. The concentration of analytes found in the blank should be less than half the MDC of the analytes of interest. If the concentration of the analytes is found above this level, provide an explanation in a case narrative. If the result is not within the limit, analyses should be stopped and the reason for the failure should be identified and resolved.

15.7 *Matrix Spike:*

15.7.1 Analyze at least one matrix spike sample with each batch of no more than 20 samples by spiking an aliquot of a sample within the batch with a known concentration of radioiodine.

15.7.2 The spike should produce a concentration that is 2 to 5 times the anticipated sample concentration or as specified by the laboratory, whichever is greater.

15.7.3 The Matrix Spike must be taken through all the steps of the method.

15.7.4 Calculate the percent recovery of the matrix spike (R) using the following formula:

$$R = \frac{AC_{as} - AC_a}{AC_s} \times 100\,\% \tag{12}$$

where:

AC_{as} = the concentration of analyte in becquerels per litre (Bq/L) measured in the spiked aliquot,
AC_a = the concentration of analyte in becquerels per litre (Bq/L) in the sample, and
AC_s = the spiked concentration of analyte in becquerels per litre (Bq/L).

15.7.5 The percent recovery, R, should fall within ±25 % of the expected value. If the concentration is not within these limits, provide an explanation in a case narrative.

15.8 *Duplicate:*

15.8.1 To check the precision of sample analyses, analyze a sample in duplicate with each batch of no more than 20 samples. Calculate the statistical agreement (duplicate error ratio (DER)) between the two results. This calculation is performed using the combined standard uncertainty of each result as shown below.

$$DER = \frac{|\,AC_{original} - AC_{dup}\,|}{\sqrt{u_c^2(AC_{original}) + u_c^2(AC_{dup})}} \tag{13}$$

where:

$AC_{original}$ = original sample activity concentration,
AC_{dup} = duplicate sample activity concentration,
$u_c(AC_{original})$ = combined standard uncertainty of the original sample, and
$u_c(AC_{dup})$ = combined standard uncertainty of the duplicate sample.

15.8.2 In those cases where there is insufficient sample volume to allow performance of a duplicate sample analysis, a duplicate LCS (LCS-D) should be performed and analyzed using the same DER criteria.

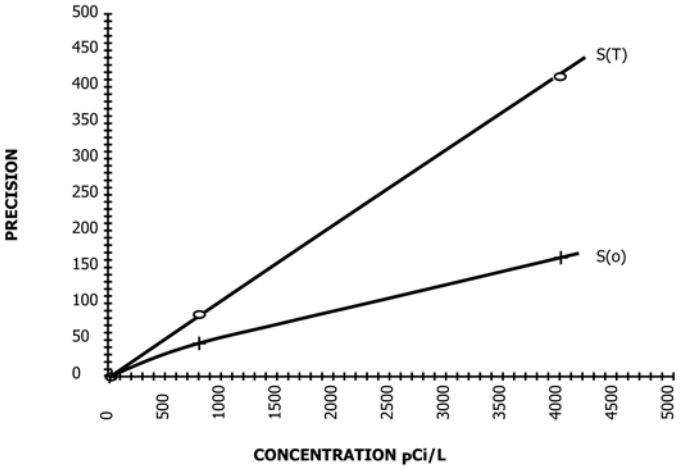

FIG. 2 $S_{(T)}$ and $S_{(o)}$ versus Concentration

TABLE 2 ^{131}I Precision and Bias Data [A]

Amount Added		Amount Found		Relative Bias (%)	Relative Precision (%)
Bq/L	pCi/L	Bq/L	pCi/L		
0.74 ± 0.01	20.2 ± 0.4	0.78 ± 0.12	21.1 ± 3.1	4.4	14.7
30 ± 1	809 ± 17	29.6 ± 4.0	802 ± 94	– 0.8	11.7
149 ± 9	4020 ± 82	152 ± 16	4100 ± 426	1.9	10.4

[A] For the 0.74-Bq/L (20-pCi/L) sample, the random uncertainty associated with counting statistics ranged up to 10 % at the 2-sigma level.

15.8.3 The value of *DER* should be less than or equal to 3.0. If the sample duplicate or LCS duplicate result is not within these limits all samples in the batch must be reanalyzed, or an explanation must be provided in a case narrative.

15.9 *Independent Reference Material (IRM):*

15.9.1 In order to verify the quantitative value produced by the test method, analyze an IRM sample, which was submitted on at least a single-blind basis (if practical) to the laboratory at least once per quarter. The concentration of analyte in the traceable reference material should be appropriate to the typical purpose for which the method is used. The value obtained shall demonstrate acceptable performance as defined by the program or the outside source.

15.9.2 In the absence of other acceptance criteria for the IRM sample, compare the IRM sample result to the IRM known value as follows:

$$R = \frac{|\, IRM_{\text{found}} - IRM_{\text{known}} \,|}{\sqrt{u_c^2(IRM_{\text{found}}) + u_c^2(IRM_{\text{known}})}} \quad (14)$$

where:

R = relative difference,
IRM_{found} = found concentration of the IRM,
IRM_{known} = known concentration of the IRM,
$u_c(IRM_{\text{found}})$ = combined standard uncertainty of the IRM found concentration, and
$u_c(IRM_{\text{known}})$ = combined standard uncertainty of the IRM known concentration.

15.9.3 The value of R should be less than or equal to 3.0. If the value of R is greater than 3.0, the method should be investigated to determine the cause.

16. Precision and Bias[6]

16.1 The collaborative test conducted of this test method included 6 laboratories each with one operator. Three activity levels of ^{131}I between 0.74 Bq/L (20 pCi/L) and 149 Bq/L (4020 pCi/L) were tested with three replicates per level. The determinations of the precision and bias statements were made in accordance with Practice D2777.

16.2 These collaborative test data were obtained using reagent grade water.

16.3 The overall precision $S_{(T)}$ and single-operator precision $S_{(O)}$ have been found to vary with level in a manner according to Fig. 2.

16.4 The bias of this test method, based upon the collaborative test data, was found to vary with level according to Table 2.

17. Keywords

17.1 iodine; ion exchange; low-level activity; radioiodine; radioactivity; solvent extraction water

[6] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D19-1136. Contact ASTM Customer Service at service@astm.org.