Designation: D 4785 – 00a

Standard Test Method for
Low-Level Iodine-131 in Water [1]

This standard is issued under the fixed designation D 4785; 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 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:*

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[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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D 1129 Terminology Relating to Water[2]
D 1193 Specification for Reagent Water[2]
D 2777 Practice for Determination of Precision and Bias of Applicable Test Methods of Committee D-19 on Water[2]
D 3370 Practices for Sampling Water from Closed Conduits[2]
D 3648 Practices for Measurement of Radioactivity[3]
D 3649 Practice for High-Resolution Gamma-Ray Spectrometry of Water[3]

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 %.

6.2 Iodine isotopes other than iodine-131 will cause erroneously high results if the activity is determined using beta counting.

7. Apparatus

7.1 *Ion Exchange Column* *Analytical Balance*, glass tube, 35 ± 2-mm inside diameter, 150-mm length, fitted with No. 8 one-hole stoppers and perforated disk. readable to 0.1 mg.

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

7.3 *Flexible Polyvinyl Chloride (PVC) Tubing*, 6.35 mm (¼ 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 *Membrane Filters* *Glass Fiber Filter Paper*, 0.4 or 0.45-µm pore size, 25-mm diameter, with suitable filter holder and vacuum filter flask. 11.5-cm diameter.

7.5 *pH Meter.* *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 *Vortex Mixer.* *Membrane Filters*, 0.4 or 0.45-µm pore size, 25-mm diameter, with suitable filter holder and vacuum filter flask.

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

7.8 *Gamma-Ray Spectrometry System*—high resolution using a high-purity germanium or lithium-drifted germanium detector (see Practice D 3649). *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 *Glass Fiber Filter Paper*, 11.5-cm diameter. *Vacuum Desiccator.*

7.11 *Analytical Balance*, readable to 0.1 mg. *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.

[2] *Annual Book of ASTM Standards*, Vol 11.01.
[3] *Annual Book of ASTM Standards*, Vol 11.02.
[4] "Reagent Chemicals, American Chemical Society Specifications," Am. Chemical Soc., 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, and the" United States Pharmacopeia."

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 (NH $_4$OH).

8.5 *Ammonium Hydroxide* (1 + 9)(1.4 *M*)—Mix one volume of concentrated NH$_4$OH (sp gr 0.90) with nine volumes of water.

8.6 *Anion Exchange Resin*—Strongly basic, styrene, quarternary ammonium salt, 20–50 mesh, chloride form, Dowex 1-X8,[5] 1-X8, or equivalent.

8.7 *Cuprous Chloride Solution* (CuCl) (approximately(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* ~~(1 + 40)~~ (0.3 ~~M~~ *M*)—Dilute 25 mL of concentrated HCl to 1000 mL with water.

8.10 *Hydroxylamine Hydrochloride* (NH$_2$OH: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 HNO$_3$.

8.14 *Nitric Acid* ~~(1 + 10)~~ (1.4 *M*)—Mix 1 volume of 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* ~~(500 g/L)~~ (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 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 ~~2.45 using dilute HCl~~ between 2.40 to 2.50 with 0.3 *M* HCl or 1.4 *M* NH$_4$OH.

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. ~~Sampling~~

~~11.1 Collect a sample in accordance with Practice D 3370.~~

~~12.~~ Calibration of High-resolution Gamma-ray Spectroscopy System

1~~2~~1.1 Prepare a set of three calibration sources in accordance with the following:

1~~2~~1.1.1 Add 50 mL of reagent water to a 150-mL beaker.

1~~2~~1.1.2 Quantitatively add approximately 3.7 kBq of NIST-traceable iodine-131 solution and swirl gently to mix.

1~~2~~1.1.3 Follow 13.3.1-13.3.6.

1~~2~~1.1.4 Count the calibration sources individually and accumulate 20 ~~000~~ thousand to 50 ~~000~~ thousand counts in the 365.48-kV photopeak.

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[5] ~~Available from Dow Chemical USA, Midland, MI 48640.~~

[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.

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

$$E_{(n)} = R_{C(n)}/A_C \qquad (1)$$

$$E = C_r / (A_C * I) \qquad (1)$$

Ac

where:

$R_{C(n)}C_r$ = the net count rate for the 364.48-keV 364.5 keV gamma-ray line of one of the standard reference iodine-131 calibration sources in the same chemical form and geometerical arrangement as used for the sample and sample,

$A_{C}c$ = the activity in becquerels of that calibration source at the midpoint of the counting period, Bq

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 $NH_2OH:HCl$, stir, and add 5.0 mL of 2 *M* $NaHSO_3$. Adjust the pH to 6.5 using (500 g/L) 12.5 *M* NaOH or (1 + 10) NHO₃ 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 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.

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[6] CuCl solution is not stable. It can be oxidized to the Cu $^{+2}$ state by air after a period

[6] Firestone, R.B. and Shirley, V.S., *Table 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 Isotopes*, (Eighth Edition), John Wiley and then closing the container promptly. Sons, Inc., New York, 1996.
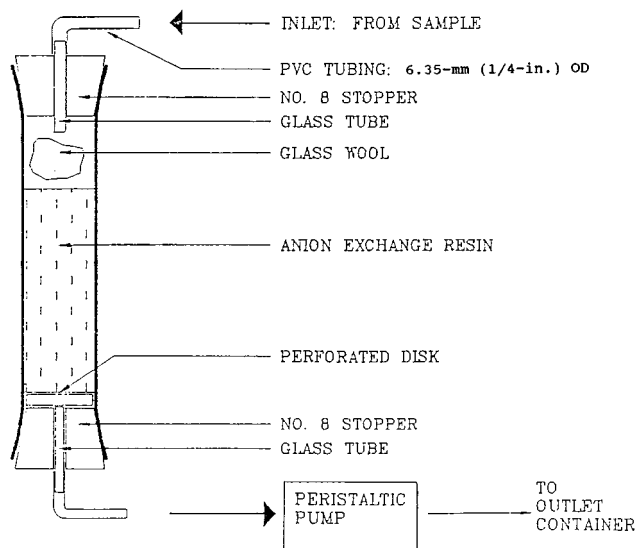

FIG. 1 Iodine Procedure: Ion Exchange

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.

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 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 9—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 10—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 occasionaly. 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 11—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.

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 ~~HCl (1 + 40)~~ 0.3 M HCl or 1.4 M NH₄OH ~~(1 + 9)~~ solution as required.

NOTE 12—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 yields.

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

NOTE 13—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 D 3648).

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 down 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 counting efficiency.

13.3.6 Using the high resolution gamma-ray spectrometry system, determine the net counting rate for the 364.48-keV gamma-ray line (Firestone and Shirley[6]).

14. Calculation

14.1 Calculate the radioactivity concentration, (AC) of iodine-131, corrected to sample collection time, as follows:

$$AC \text{ (Bq/L)} = \frac{(R_a - R_b)}{E \times V_a \times Y \times \epsilon^{-\lambda t}} \tag{2}$$

$$AC \text{ (Bq/L)} = \frac{(R_a - R_b)}{E \times V_a \times Y \times e^{-\lambda t}} \tag{2}$$

where:
E = absolute detection efficiency of the gamma-ray spectrometer, counts per disintegration (Eq 1),
λ = decay constant for iodine-131, (Reference 1) 6) 0.08640 d^{-1}
R_a = count rate of the sample aliquant, counts per second,
R_b = count rate of the instrument background, counts per second, at the 364.48-keV gamma-ray region of interest,
t = elapsed time from sample collection to midpoint of sample counting time, days,
V_a = volume of sample aliquant, L, and
Y = fraction of the total iodine carrier recovered from the sample, which is the weight of the sample determined in 13.3.3 13.3 divided by weight of standardized carrier in accordance with 10.8.

14.2 The sample aliquant has an uncertainty in its net count rate (counting uncertainty). The uncertainty in the iodine-131 ratio activity concentration in the sample from the counting uncertainty, U_R, is given at 68 % confidence level as follows:

$$U_R \text{ (Bq/L)} = \frac{(\sqrt{R_a + R_b}\,/\sqrt{t_a}}{E \times V_a \times \epsilon^{-\lambda t}} \tag{3}$$

$$U_R \text{ (Bq/L)} = \frac{(\sqrt{R_a + R_b}\,/\sqrt{t_a}}{E \times V_a \times e^{-\lambda t}} \tag{3}$$

where:
t_a = count duration of the sample aliquant, and
Other terms are as defined in 14.1

14.3 Calculate the total propagated combined standard uncertainty (TPU) (U_c) as follows:

$$TPU \text{ (Bq/L)} = AC \sqrt{(U_R/AC)^2 + U_E^2 + U_{V_a}^2 + U_Y^2 + U_O^2} \tag{4}$$

$$U_c \text{ (Bq/L)} = AC \sqrt{(U_R/AC)^2 + U_E^2 + U_{V_a}^2 + U_Y^2 + U_O^2} \tag{4}$$

where:
U_E = fractional uncertainty in the detection efficiency based on the standard deviation of the three calibration sources and the source uncertainty,
U_{V_a} = fractional uncertainty in the sample volume measurement,
U_Y = fractional uncertainty in the chemical yield (recovery) of the iodine carrier,
U_O = fractional systematic any additional fractional uncertainty in the method that is dependent on the sample matrix, has been determined or estimated, and
Other terms are as defined in 14.1 and 14.2.

14.4 Calculate minimum detectable radioactivity concentration (MDAC) as follows:

14.4 *Expanded Uncertainty (U)*:

$$U(Bq/L) = KU_C \tag{5}$$

where:
K = coverage factor for the desired confidence interval (nomally K=2 for 95 % confidence interval).

14.5 Calculate *a priori* minimum detectable radioactivity concentration (MDAC) as follows:

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$$MDC \text{ (Bq/L)} = \frac{(4.65 \sqrt{C_b} + 2.71)}{E \times t_a \times V_a \times Y \times \epsilon^{-\lambda t}} \tag{6}$$

$$MDC \text{ (Bq/L)} = \frac{(4.65 \sqrt{C_b} + 2.71)}{E \times t_a \times V_a \times Y \times e^{-\lambda t}} \tag{6}$$

where:

C_b = number of counts accumulated during the counting of the sample aliquant that are due to instrument background in the gamma-ray region of interest, and

Other terms are as defined previously.

NOTE 14—The counting times of the sample aliquant and of instrument background must be of equal length for Eq 3-5 3-6 to be valid. The values used for the terms in Eq 5 6 should be typical for the laboratory.

14.5 The absolute counting efficiency is determined as follows:

$$\epsilon = R_c/R_d \tag{6}$$

where:

R_c = net counting rate for the 364.48 KeV gamma ray line of a standard iodine-131 source in the same chemical form and geometrical arrangement as is used for the sample and

R_d = disintegration rate of the standard iodine-131 source at the time of counting.

15. Quality Control

15.1 Prior to using this test method, a quality control (QC) program for the instrumentation (high-resolution gamma-ray spectrometer) shall be established when the system is calibrated. Quality control or tolerance charts shall be established. Control limits shall be established with corrective actions when control limits are exceeded.

15.2 Before this test method is utilized for the analysis of samples, individuals performing this test method shall be qualified by analyses of samples spiked with iodine-131. The bias and precision of these measurements should be consistent with the data provided in Section 16.

15.3 A QC source shall be counted daily or prior to use. The results shall be tabulated or plotted on control charts and evaluated in accordance with Practices D 3648.

15.4 The counter background shall be determined periodically. The background data shall be maintained in a logbook or plotted on a trend chart, or both.

15.5 Precision and bias can be assessed in the following manner: the precision of an individual measurement can be approximated by the total propagated uncertainty and bias can be assessed by the analysis of NIST-traceable spiked samples with known quantities of iodine-131.

15.6 The project manager, as part of the external quality control program, shall submit blind quality control samples to the analysts along with routine samples. These external quality control samples, which usually include duplicate and blank samples, shall test sample collection and preparation as well as sample analysis whenever possible. In addition, analysts are expected to run internal quality control samples that will indicate whether the analytical method is in control. Both internal and external quality control samples need to be prepared in such a way to duplicate the chemical matrix of routine samples, insofar as this is practical. The quality control samples that are routinely used include blank samples, replicate samples, reference material, control samples, and spiked samples.

16. Precision and Bias [7]

16.1 The collaborative test conducted of this test method included 6 laboratories each with one operator. Three activity levels between 0.74 Bq/L (20 pCi/L) and 149 Bq/L (4020 pCi/L) were tested with three replicates per level. The determination of the precision and bias statements were made in accordance with Practice D 2777.

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 1.

17. Keywords

17.1 iodine-131; ion exchange; low-level activity; radioactivity; water

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[7] Firestone, R.B. and Shirley, V.S., *Table of the Isotopes*, 8th ed. 1995, p. 1151.
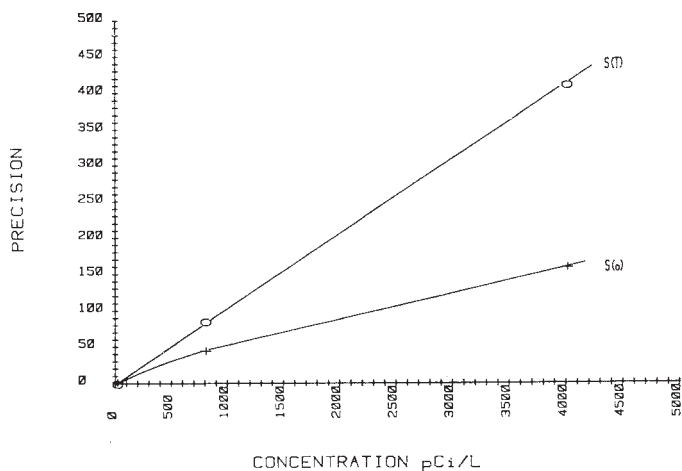[7] Supporting data are available from ASTM Headquarters. Request Research Report RR:D-19-1136.

FIG. 2 *S*(*T*) and *S*(*O*) versus Concentration

TABLE 1 Iodine-131 Precision and Bias Data [A]

Amount Added		Amount Found		Per-cent Bias	Rela-tive Preci-sion
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	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.