



Designation: ~~D4785-00a~~ Designation: D 4785 - 08

Standard Test Method for ~~Low-Level Iodine-131 in Water~~ Low-Level Analysis of Iodine Radioisotopes in Water¹

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~~ 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 Note 2, Note 3, ~~Note 8~~ Note 9, and Section 9.

2. Referenced Documents

2.1 ASTM Standards:²

D 1129 Terminology Relating to Water

D 1193 Specification for Reagent Water

D 2777 Practice for Determination of Precision and Bias of Applicable Test Methods of Committee ~~D-19~~ D19 on Water

D 3370 Practices for Sampling Water from Closed Conduits

D 3648 Practices for the Measurement of Radioactivity

D 3649 ~~Practice for High-Resolution Gamma-Ray Spectrometry of Water~~³ Practice for High-Resolution Gamma-Ray Spectrometry of Water

D 5847 Practice for Writing Quality Control Specifications for Standard Test Methods for Water Analysis

D 3856 Guide for Good Laboratory Practices in Laboratories Engaged in Sampling and Analysis of Water

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 is followed by oxidation-reduction ~~yieldsto~~ elemental iodine. The elemental iodine is purified by solvent extraction, reduced to iodide, and precipitated as cuprous iodide. The chemical ~~yieldrecovery~~ 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.~~

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.

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*, Vol 11.01, volume information, refer to the standard's Document Summary page on the ASTM website.

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 D 2777. 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 yield determination. One milligram of iodine would produce a bias of about -4%.

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 %.

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 ($\frac{1}{4}$ 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 D3649—high resolution gamma spectrometer (high purity germanium or equivalent) with a useful energy range of approximately 30 keV to 1800 keV (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 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*, Buchner/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 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_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, quarternary 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).

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

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.

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 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 HNO₃.

8.14 *Nitric Acid (1.4 M)*—Mix 1 volume of concentrated 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.

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

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

Calibration of High-resolution Gamma-ray Spectroscopy System

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.

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 $\leq 1\%$.

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:

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

$$E_n = \text{Offset} + (Ch \times \text{Slope})$$

E_n

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, peak energy (keV),

A_c = the activity in becquerels of that calibration source at the midpoint of the counting period, energy offset for the energy calibration equation (keV),

I = the intensity for the 364.8 keV gamma-ray line, 0.812⁶, peak location channel number, and

$Slope$ = energy calibration equation slope (keV per channel).

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

NOTE 4—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:

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

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),

E_n = peak energy (keV), and

$Slope$ = resolution calibration equation slope (keV/keV).

NOTE 5—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:

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

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_s = 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 $\pm 8\%$ 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 D 3370 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 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~~ 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~~ 5 mm glass tubing connecting to the inlet side of the peristaltic pump (see Fig. 1).

~~NOTE 4—The 6—~~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 7—~~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 8—~~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.

IODINE PROCEDURE: ION EXCHANGE

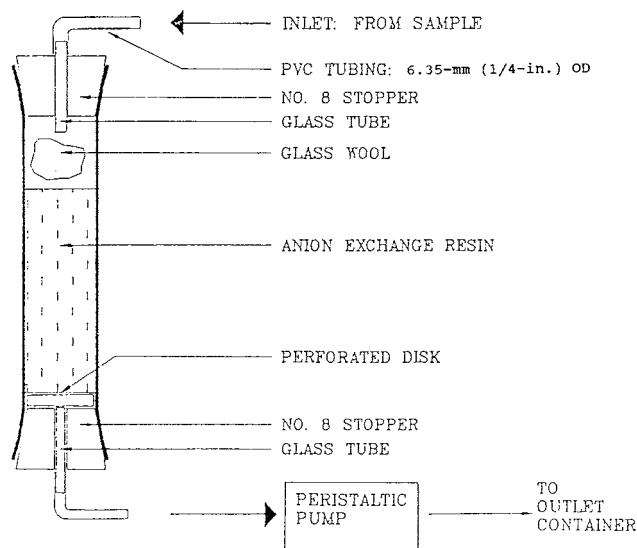


FIG. 1 Iodine Procedure: Ion Exchange

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 ~~and 13.2.11~~ into a 2000-mL beaker and discard the resin.

~~NOTE 8—~~ **9—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 HNO₃ (sp gr 1.42) to the NaOCl solution from 13.2.11 until the pH is brought to 1. (Approximately 45 mL of HNO₃ 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 NH₂OH·HCl.

~~NOTE 9~~ **10—**Hydroxylamine hydrochloride is a mild reducing agent capable of reducing iodate to iodine (I⁰). Iodine is preferentially soluble in the toluene phase and can be separated by solvent extraction. When NH₂OH·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 I₃⁻ (a combination of I₂ 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 11—**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 NaHSO₃. 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 NaHSO₃ 12—**The NaHSO₃ 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~~ **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 NH₄OH solution as required.

~~NOTE 12—~~ **The 13—**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—recoveries.

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

~~NOTE 13—~~ **Paragraphs 14—**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- μ m 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 counting detection 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 Shirley⁶).~~ 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

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

⁶ 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.

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