



Designation: ~~D4785 – 08 (Reapproved 2013)~~^{ε1} D4785 – 20

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

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.

^{ε1} 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~~ 4 L water sample using ion exchange and solvent extraction and is then precipitated as cuprous iodide for counting.

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

1.3 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety, health, and environmental practices and determine the applicability of regulatory limitations prior to use.* For specific hazard statements, see ~~8-178.16, 8-188.17, 8-198.18~~, Section 9, and 13.2.11.

1.4 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 ASTM Standards:²

[D1129 Terminology Relating to Water](#)

[D1193 Specification for Reagent Water](#)

[D2777 Practice for Determination of Precision and Bias of Applicable Test Methods of Committee D19 on Water](#)

[D3370 Practices for Sampling Water from Flowing Process Streams](#)

[D3648 Practices for the Measurement of Radioactivity](#)

[D3649 Practice for High-Resolution Gamma-Ray Spectrometry of Water](#)

[D4448 Guide for Sampling Ground-Water Monitoring Wells](#)

[D5847 Practice for Writing Quality Control Specifications for Standard Test Methods for Water Analysis](#)

~~D3856~~[D6001 Guide for Management Systems in Laboratories Engaged in Analysis of Water](#)[Direct-Push Groundwater Sampling for Environmental Site Characterization](#)

[D7282 Practice for Set-up, Calibration, and Quality Control of Instruments Used for Radioactivity Measurements](#)

[D7902 Terminology for Radiochemical Analyses](#)

2.2 Other Documents:

[ANSI N42.22 Traceability of Radioactive Sources to the National Institute of Standards and Technology \(NIST\) and Associated Instrument Quality Control](#)³

[BIPM-5 Decay Data Evaluation Project \(DDEP\)](#)⁴

[NUDAT2](#)⁵

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~~ standard's Document Summary page on the ASTM website.

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

⁴ Available from BIPM, Sèvres Cedex, France, <https://www.bipm.org>.

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

3. Terminology

3.1 ~~Definitions—Definitions:~~ For definitions of terms used in this test method, refer to Terminology [D1129](#).

3.1.1 For definitions of terms used in this standard, refer to Terminology [D1129](#).

3.1.2 For definitions of terms used in this standard relating to radiochemical analysis, refer to Terminology [D7902](#).

4. Summary of Test Method

4.1 Sodium iodide is added as a carrier prior to performing any chemical separations. The samples undergo an oxidation-reduction process to ensure exchange between the carrier and the radioactive iodide. Hydroxylamine hydrochloride and sodium bisulfite are added to convert all the iodine to iodide which is then removed by anion exchange. Subsequent elution of the iodide is followed by oxidation-reduction to elemental iodine. The elemental iodine is purified by solvent extraction, reduced to iodide, and precipitated as cuprous iodide. The chemical ~~recovery~~yield is determined from the ~~recovery~~net mass of the recovered 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~~limits for drinking water. With a ~~suitable counting technique, techniques,~~ 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 spectroscopy.~~ ¹³¹I. 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 test method~~ is intended for the analysis of iodine radioisotopes with half-lives greater than 2 hours, which include ¹²¹I, ¹²³I, ¹²⁴I, ¹²⁵I, ¹²⁶I, ¹²⁹I, ¹³⁰I, ¹³¹I, ¹³²I, ¹³³I, and ¹³⁵I. The test method was tested according to Practice [D2777](#) using only ¹³¹I. The user of this test method is responsible for determining applicability, bias, and precision for the measurement of other iodine radioisotopes.

6. Interferences

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

6.2 There are numerous characteristic iodine X-rays at and below 33.6 keV which are indicative of iodine, but not of a specific radioisotope of iodine. ~~It is recommended that only discreet~~ Only use discrete 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, ~~4-m-~~1 m length.

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

7.4 *Glass Fiber Filter Paper*, ~~4.5-cm-~~11.5 cm diameter.

7.5 *Ion Exchange Column*, glass tube, 35 ± ~~2-mm-~~2 mm inside diameter, ~~150-mm-~~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-0.45 µm (or 0.4 µm) pore size, 25 mm diameter, with suitable filter holder and vacuum filter flask.

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

7.8 *pH Meter*.

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

7.10 *Vacuum Desiccator*.

7.11 *Vortex Mixer*.

8. Reagents and Materials

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

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

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

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

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

8.5 *Anion Exchange Resin*—Strongly basic, styrene, ~~quarternary~~quaternary ammonium salt, 20–50 mesh, chloride form, Dowex⁷ 1-X8, or equivalent.

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

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

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

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

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

~~8.11 *Iodine-131 Standardizing Standard Solution*—National standardizing body Solution traceable to the SI through a national metrology institute (NMI), such as National Institute of Standards and Technology (NIST), traceable solution with a typical concentration range from 1 to 10 kBq/mL.~~

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

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

8.14 *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.

8.15 *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.

8.16 *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.)

8.17 *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.)

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

8.19 *Calibration Standard(s)*—~~Known Use known amounts of of~~ ¹²⁵I, ¹²⁹I, and ¹³¹I are used for calibration when determining these radionuclides. A mixed-gamma standard, for example, standard containing ²⁴¹Am, ¹⁰⁹Cd, ⁵⁷Co, ¹⁴¹Ce, ¹¹³Sn, ¹³⁷Cs, ⁸⁸Y, and ⁶⁰Co, is ~~Co~~ may be used for calibration over an extended energy range as required for the determination of additional radioisotopes of iodine. ~~These~~The standards should be mounted on the filter as ~~described~~(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~~) in a configuration

⁶ *Reagent Chemicals, American Chemical Society Specifications, ACS Reagent Chemicals, Specifications and Procedures for Reagents and Standard-Grade Reference Materials*, American Chemical Society, Washington, DC. For ~~Suggestions~~suggestions on the testing of reagents not listed by the American Chemical Society, see *Annual/Anal. Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

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

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

that closely matches that of sample test sources (STS) to be counted. Calibration standards may be prepared by the laboratory performing this test method or by a commercial supplier of such standards. The standards used must be traceable to the SI through an NMI. Standards obtained from ANSI N42.22 reference material providers will meet this requirement. Alternate radionuclides may be used for calibration provided that they have gamma-ray energies covering the calibration source covers gamma-ray energies spanning the range of interest for the iodine radionuclides to be analyzed.

9. Hazards

9.1 Due to the potential health and safety effects from handling these compounds, the steps utilizing NaOCl and toluene must be carried out in a fume hood. Toluene is ~~a carcinogen~~ highly flammable 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~~ 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.

NOTE 1—A sixth standard prepared in parallel to the five replicates may be used to determine how much HCl or NH₄OH needs to be added to adjust the pH to the range of 2.40–2.50.

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 ~~Accumulate an energy spectrum using a spectrum for calibration by counting the calibration standard (8.208.19) traceable to a national standards body, in the geometrical position representing in a geometry matching that of the samples~~ STS 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 centroid location data from the calibration spectrum, establish the energy per channel relationship (energy calibration) as:~~

~~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:~~

$$E_n = \text{Offset} + (Ch \times \text{Slope}) \quad (1)$$

where:

E_n = 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 2—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 (peak-resolution calibration) as:~~

~~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);

Offset = FWHM offset for the resolution calibration equation (keV),
E_n = peak centroid energy (keV), and
Slope = peak resolution calibration equation slope (keV/keV).

NOTE 3—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 Using the gamma-ray emission data from the calibration standard, calculate the full-energy peak efficiency, ϵ_f , as follows:
 For each gamma-ray photopeak, calculate the full-energy peak efficiency, ϵ_f , as follows:

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

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

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, $e^{-\lambda(t_1 - t_0)}$,
 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 Modern spectrometry systems are computerized and the determination of the calculate gamma-ray detection efficiencies is performed automatically at the end of an appropriate counting interval. automatically. 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 typical for the detector type. The curve should be smooth, continuous and have a shape similar to the that reflects the type of 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 difference between the actual efficiency and the efficiency calculated with using 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 Practices D3370, Guide D4448, Guide D6001, 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 $NH_2OH \cdot HCl$, 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 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~~ 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~~ Wash the resin 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, ~~stopper~~ fitted with a short length of ~~5-mm~~ 5 mm glass tubing, in the top of the column and connect it to a ~~1-m~~ 1 m length of flexible PVC tubing.

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

13.2.3 Pump approximately 100 mL of water through the resin-packed column and check the final effluent pH with pH paper. Repeat the wash if the test indicates residual ~~activity~~ acidity in excess of levels observed during the initial resin washes. 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~~ 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~~ adjust the speed control to give a flow rate of 40 mL/min.

NOTE 6—~~It is necessary to calibrate~~ 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~~ stopper and glass wool plug from the top of the column and pour the resin into a ~~600-mL~~ 600 mL beaker.

13.2.8 Wash the resin with three successive ~~100-mL~~ 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~~ 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~~ 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

IODINE PROCEDURE: ION EXCHANGE

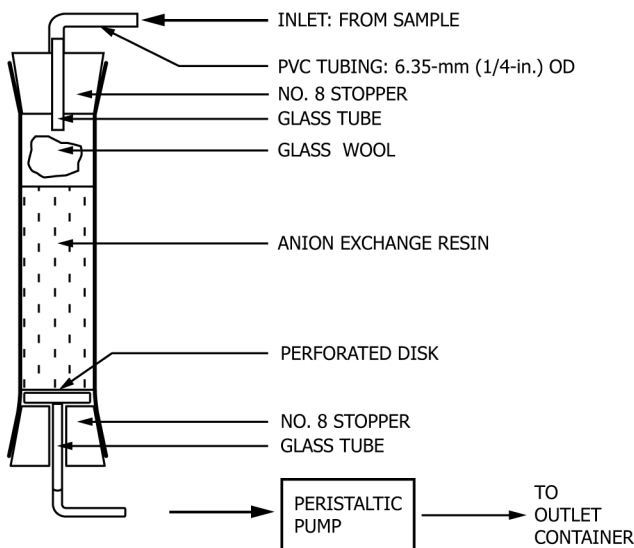


FIG. 1 Iodine Procedure: Ion Exchange

solution leads to decomposition of residual NaOCl that would interfere in the reduction of iodate to elemental iodine. ~~All Highly toxic chlorine gas is released (green color). Perform 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~~ 1000 mL separatory funnel containing 100 mL of toluene and 2 g of $\text{NH}_2\text{OH}\cdot\text{HCl}$.

NOTE 7—Hydroxylamine hydrochloride is a mild reducing agent capable of reducing iodate to iodine (I^0). Iodine is preferentially soluble in the toluene phase and can be separated by solvent extraction. When $\text{NH}_2\text{OH}\cdot\text{HCl}$ 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~~ 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 8—~~It is necessary to relieve~~ Relieve the pressure at the beginning of shaking and a few times during the ~~2-min~~ 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~~ 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~~ 100 mL centrifuge tube until the toluene phase enters the stopcock bore. Discard the toluene in an appropriate hazardous waste container.

NOTE 9—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~~ Using a pH meter, adjust the pH to between 2.40 to 2.50 with 0.3 M HCl or 1.4 M NH_4OH solution, ~~as required.~~ needed.

NOTE 10—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~~ yields.

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

NOTE 11—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 Practices D3648).

13.3.3 Mount a ~~preweighed membrane filter (0.4 or 0.45- μm pore size or equivalent)~~ tared membrane filter 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~~ desiccator and dry under vacuum for a minimum of 60 min or to constant weight. Remove the sample, weigh it, and record the ~~weight~~ gross mass of the filter and dry precipitate. Calculate the net mass of CuI precipitate as the difference of the gross mass and the tare mass of the filter.

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,~~ been calibrated for detection efficiency.

13.3.6 Using the high resolution gamma-ray spectrometry system, count the STS and 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.⁹

NOTE 12—Two online references for expertly evaluated nuclear data, including radionuclide half-lives and their uncertainties, are recommended: the BIPM-M Decay Data Evaluation Project (DDEP), and the NUDAT2 National Nuclear Data Center. Other sources of nuclear data may be used at the user's discretion. Regardless of which reference is used, the source should be clearly documented.

14. Calculation

NOTE 13—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.

NOTE 14—The following calculations for R_{net} , L_C , and MDC require 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, these calculations may be performed manually using a printout of channel contents.

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

⁹ National Nuclear Data Center, Information extracted from NUDAT Data Base, available at: <http://www.nndc.bnl.gov/nudat2> from <https://www.nndc.bnl.gov>.