



# Standard Test Method for Tritium in Drinking Water<sup>1</sup>

This standard is issued under the fixed designation D 4107; 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 ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

## 1. Scope

1.1 This test method covers the determination of tritium in drinking water (as T<sub>2</sub>O or HTO) by liquid scintillation counting of the tritium beta particle activity.

1.2 This test method is used successfully with drinking water. It is the user's responsibility to ensure the validity of this test method for waters of untested matrices.

1.3 The tritium concentrations, which can be measured by this test method utilizing currently available liquid scintillation instruments, range from less than 0.037 Bq/mL (1 pCi/mL) to 555 Bq/mL (15 000 pCi/mL) for a 10-mL sample aliquot. Higher tritium concentrations can be measured by diluting or using smaller sample aliquots, or both.

1.4 The maximum contaminant level for tritium in drinking water as given by the National Interim Primary Drinking Water Regulations (NIPDWR) is 0.740 Bq/mL (20 000 pCi/L). The NIPDWR lists a required detection limit for tritium in drinking water of 0.037 Bq/mL (1 pCi/mL), meaning that drinking water supplies, where required, should be monitored for tritium at a sensitivity of 0.037 Bq/mL (1 pCi/mL). In Appendix X1, Eq X1.3 is given for determining the necessary counting time to meet the required sensitivity for drinking water monitoring.

1.5 *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.*

## 2. Referenced Documents

### 2.1 ASTM Standards:

D 1129 Terminology Relating to Water<sup>2</sup>

D 1193 Specification for Reagent Water<sup>2</sup>

D 2777 Practice for Determination of Precision and Bias of Applicable Methods of Committee D-19 on Water<sup>2</sup>

D 3370 Practices for Sampling Water from Closed Conduits<sup>2</sup>

<sup>1</sup> 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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<sup>2</sup> *Annual Book of ASTM Standards*, Vol 11.01.

D 3648 Practices for the Measurement of Radioactivity<sup>3</sup>

## 3. Terminology

3.1 *Definitions*—For definitions of terms used in this test method, refer to Terminology D 1129. For terms not defined in this test method or in Terminology D 1129, reference may be made to other published glossaries.<sup>4</sup>

## 4. Summary of Test Method

4.1 In this test method, a 100-mL drinking water sample aliquot is treated with a small amount of sodium hydroxide and potassium permanganate, distilled, and a specified fraction of the distillate is collected for tritium analysis. The alkaline treatment is to prevent other radionuclides, such as radioiodine and radiocarbon from distilling over with the tritium. Some drinking water supplies will contain trace quantities of organic compounds, especially surface water sources that contain fish and other life. The permanganate treatment is to oxidize trace organics in the sample aliquots which could distill over and cause quenching interferences. A middle fraction of the distillate is collected for tritium analysis because the early and late fractions are more apt to contain interfering materials for the liquid scintillation counting process.

4.2 As the sample distills, there is a gradient in the tritium concentration in the accumulating distillate; therefore, it is important to collect the same fraction of the distillate for all samples and standards for tritium analysis.

4.3 The collected distillate fraction is thoroughly mixed and a portion (up to 10 mL) is mixed with liquid scintillator solution, and after dark adapting, is counted in the liquid scintillation counting system for tritium beta particle activity.

## 5. Significance and Use

5.1 This test method was developed for measuring tritium in water to determine if the concentration exceeds the regulatory statutes of drinking water. This test method also is applicable for the determination of tritium concentration in water as required by technical specifications governing the operations of nuclear power facilities. With suitable counting technique,

<sup>3</sup> *Annual Book of ASTM Standards*, Vol 11.02.

<sup>4</sup> *American National Glossary of Terms in Nuclear Science and Technology*, ANSI, 11 W. 42nd St., 13th Floor, New York, NY 10036.

sample size, and counting time a detection limit of less than 37 Bq/L (1000 pCi/L) is attainable by liquid scintillation.

## 6. Interferences

6.1 A reduced counting efficiency may result from quenching in the sample scintillator mixture. Quenching is caused by impurities in the sample, which can inhibit the transfer of energy, or by colored materials, which may absorb some of the emitted light. Corrections for quenching can be made by the use of internal standards<sup>4</sup> or by the ratio method.<sup>5</sup> The approach described in this test method, distillation after alkaline permanganate treatment, eliminates quenching substances, as well as radionuclides which might be present in a volatile chemical form such as radioiodine and radiocarbon. A boiling chip must be used with each distillation to avoid bumping, which can amount to a carry over excursion.

6.2 Scintillator stock solution or samples exposed to daylight must be dark-adapted. Also, toluene or xylene base scintillators exposed to fluorescent lighting should be dark-adapted for a minimum of 6 h and dioxane base scintillators exposed to fluorescent lighting for 24 h. All flours should be checked for excitation under lighting conditions being used, and if possible, they should be exposed only to red light.

## 7. Apparatus

7.1 *Liquid Scintillation Spectrometer*, coincidence-type.

7.2 *Liquid Scintillation Vials*, of low-potassium glass are recommended. Polyethylene vials may be used when dioxane scintillator solution is used.

7.3 *Distillation Apparatus*—For aqueous distillation, 250-mL and 1000-mL round bottom borosilicate flasks, connecting side arm adapter,<sup>6</sup> condenser, graduated cylinder, boiling chips, and heating mantle.

## 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, where such specifications are available.<sup>7</sup> Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

8.1.1 All chemicals should be of reagent-grade or equivalent whenever they are commercially available.

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

8.3 *Reagents of Distillation Treatment:*

8.3.1 *Sodium Hydroxide Pellets.*

<sup>5</sup> Bush, E.T., "General Applicability of the Channels Radio Method of Measuring Liquid Scintillation Counting Efficiencies," *Analytical Chemistry*, 35:1024, 1963.

<sup>6</sup> Corning part no. 9060 has been found satisfactory for this purpose.

<sup>7</sup> *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. Pharmaceutical Convention, Inc. (USPC), Rockville, MD.

8.3.2 *Potassium Permanganate.*

8.4 *Background Water*, with tritium activity below the minimum detectable activity (most deep well waters are low in tritium content).

8.5 *Scintillator Solutions:*

8.5.1 *Dioxane Liquid Scintillator Solution*—Dissolve 4 g of scintillation-grade PPO (2,5-diphenyloxazole), 0.05 g of scintillation-grade POPOP [1,4-bis(5-phenyloxazolyl-2-yl)-benzene], and 120 g of naphthalene in 1 L of spectroquality, 1,4-dioxane. Store the solution in a dark (amber) bottle. This solution can be used with glass or polyethylene vials.

8.5.2 *Solution G Scintillator Solution*—Dissolve 18 g of scintillation-grade PPO (2,5-diphenyloxazole) and 3.6 g of scintillation-grade BIS-MSB [p-bis(o-methylstyryl)benzene] in 2 L of spectroquality *p*-xylene. Add 1 L of Triton N-101<sup>8</sup> detergent to the *p*-xylene scintillator solution. Dissolve 50 g of SXS (sodium xylene sulfonate) in 100 mL of water and add this solution to the *p*-xylene scintillator-Triton solution. Mix thoroughly. Store the solution in a dark (amber) bottle. This solution should be used with glass vials since the *p*-xylene solvent evaporates slowly through the wall of the polyethylene vials.

8.5.3 Other commercially available scintillators can be used, such as the environmentally safe di-isopropyl naphthalene based scintillators. It is the responsibility of the user to verify the acceptability of a substitute scintillator.

## 9. Sampling

9.1 Collect the sample in accordance with Practices D 3370.

9.2 Since tritium in drinking water is likely to be in the form of T<sub>2</sub>O or HTO, there is no need for special handling or preservation.

## 10. Calibration

10.1 *Determination of Recovery and Counting Efficiency Factors:*

10.1.1 Prepare in a 1-L volumetric flask, a tritium standard solution containing approximately 16.67 disintegrations/s (dps)/mL using low level tritium background raw water (undistilled) and standard tritium activity. Label this solution as *raw water tritium standard solution*. Distill approximately 600 mL of water obtained from the same raw water source as above (without tritium activity added).

10.1.1.1 Use this distillate for background tritium determinations. Using the distillate and standard tritium activity, prepare a tritium standard solution in a 500-mL volumetric flask to contain the same specific activity as the raw water tritium standard solution. Label this solution as distilled water tritium standard solution.

10.1.2 *Aqueous Alkaline Permanganate Distillation*—Place a 100-mL aliquot of the raw water tritium standard solution in a 250-mL distillation flask. Add 0.5 g of sodium hydroxide, 0.1 g of potassium permanganate, and a boiling chip. Proceed with the distillate according to the procedure described in 10.1, discard 10 mL, and collect 50 mL of distillate for analysis. Mix

<sup>8</sup> Triton products are available from Rohm and Haas Company, Independence Mall West, Philadelphia, PA 19105.