



## Standard Test Method for Gaseous Tritium Content of the Atmosphere<sup>1</sup>

This standard is issued under the fixed designation D 3442; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

ε<sup>1</sup> NOTE—Editorial corrections were made to Sections 1 and 9 and Section 16 was added in August 1995.

### 1. Scope

1.1 This test method covers the measurement of total tritium activity in the atmosphere in concentrations greater than 74 mBq/m<sup>3</sup> (2 pCi/m<sup>3</sup>). This test method covers sample collection, preparation, and radiometric assay of the sample.

1.2 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information 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.*

### 2. Referenced Documents

#### 2.1 ASTM Standards:

- D 1071 Test Methods for Volumetric Measurement of Gaseous Fuel Samples<sup>2</sup>
- D 1356 Terminology Relating to Sampling and Analysis of Atmospheres<sup>3</sup>
- D 1357 Practice for Planning the Sampling of the Ambient Atmosphere<sup>3</sup>
- D 2476 Test Method for Tritium in Water<sup>4</sup>
- D 3195 Practice for Rotameter Calibration<sup>3</sup>

### 3. Terminology

3.1 *Definitions*—For definitions of terms used in this test method, see Terminology D 1356.

### 4. Summary of Test Method

4.1 Air is pumped at a sampling rate of 100 to 500 mL/min through a tube furnace packed with palladium catalyst at a temperature of 500°C or a copper oxide catalyst at a temperature of 600° to 650°C for approximately 1 week. Gaseous tritium present in the airstream is oxidized to form tritiated

water vapor which is then adsorbed on silica gel with the ambient water vapor. The water is distilled from the silica gel and the tritium activity is measured by liquid scintillation counting. The volume of air sampled and the total volume of water vapor collected during the sampling period is determined and the tritium activity of the atmosphere is calculated.

4.2 Both the palladium and copper oxide catalysts have been found satisfactory for sampling where monitoring tritium in the form of water, hydrogen gas, and heavy hydrocarbon compounds; however, the copper oxide catalyst has low efficiency for oxidizing tritium in forms such as methane.

### 5. Significance and Use

5.1 This test method is useful for sampling the atmosphere for tritium activity, in the form of tritiated hydrogen gas (HT or T<sub>2</sub>), water, methane, and other gaseous hydrocarbons, and gases that can be oxidized to form water.

5.2 Tritiated particulate matter will not be collected and analyzed.

### 6. Interference

6.1 Volatile radioactive particulates and radioiodine can interfere and when they are expected they can be prefiltered by a charcoal cartridge or zeolite. Radioiodine and radioactive particulate matter can be filtered from the air prior to passing through the tube furnace.

### 7. Apparatus

7.1 *Liquid Scintillation Counter*. (See Test Method D 2476.)

7.2 *Tube Furnace* (100 mm heating length; range 750°C).

7.3 *Low-Volume Leakless Continuous Vacuum Pump*, capacity of 750 mL/min, with flow controller such as a limiting orifice.

7.4 Tubes, glass or metal, 30 to 50 mm in diameter and 250 to 350 mm long.

7.5 *Temperature-Resisting Tubing*, to hold catalyst inside tube furnace.

7.6 *Distillation Flask*, 1-L, with standard-taper connectors.

7.7 *Condenser*, with connectors to distillation flask, all “standard-taper” joints.

7.8 *Condensate Receiving Vessel*, with standard-taper joint.

7.9 *Rubber Stoppers*.

<sup>1</sup> This test method is under the jurisdiction of ASTM Committee D-22 on Sampling and Analysis of Atmospheres, and is the direct responsibility of Subcommittee D22.03 on Ambient Atmospheres and Source Emissions.

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<sup>2</sup> *Annual Book of ASTM Standards*, Vol 05.05.

<sup>3</sup> *Annual Book of ASTM Standards*, Vol 11.03.

<sup>4</sup> *Annual Book of ASTM Standards*, Vol 11.02.

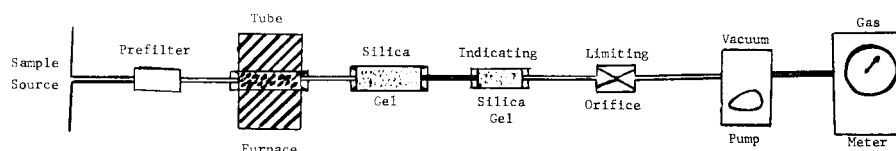


FIG. 1 Apparatus

7.10 *Vacuum Tubing*, to connect sampling system components.

7.11 *Vials*, polyethylene (20- to 25-mL, for liquid scintillation counting). (If sample is to be stored for any length of time glass vials should be used.) See Test Method D 2476 for precautions on type of glass.

7.12 *Heating Mantle and Temperature Controller*.

7.13 *Charcoal Cartridge*.

7.14 *Quartz Wool*.

7.15 *Volume Meter*, dry, diaphragm type.

7.16 Rotameter, ~750 mL/min.

## 8. Reagents

8.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. All reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.<sup>5</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.

8.2 *Catalyst Copper Wool*, or *Palladium Sponge* (40 + mesh).

8.3 *Indicating Silica Gel*, or other desiccant.

8.4 *Liquid Scintillation Solution*.<sup>6,7</sup> (See Test Method D 2476.)

8.5 *Nonindicating Silica Gel* (6 to 16 mesh), reagent grade.

## 9. Precautions

9.1 *Technical Precautions*—Ensure that the water retention capacity of the silica gel is not exceeded during the sampling period. The adsorbent capacity is a function of relative humidity and for very hot and dry regions can drop to about 5 mass % for an ambient relative humidity of 10 %. Examination of the local meteorological conditions will determine the need for shortened sampling periods.

## 10. Sampling

10.1 See Practice D 1357 for guidance on sampling procedures.

## 11. Preparation of Apparatus (See Fig. 1)

11.1 Dry about 0.5 kg of nonindicating silica gel for 24 h at 120° to 150°C prior to use for tritium sampling.

<sup>5</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.

<sup>6</sup> Moghissi, A., et al, *International Journal of Applied Radiation and Isotopes*, IJARA, Vol 20, 1969, pp. 145–156.

<sup>7</sup> Lieberman, R., and Moghissi, A., *ibid.* Vol 21, 1970, pp. 319–327.

11.2 Place a rubber stopper with a plug of glass wool in the end of the tritium sampling cylinder and fill the cylinder with dried silica gel from 11.1. Close the other end of the cylinder with a second plug of glass wool and a rubber stopper.

11.3 Place about 10 g of copper wool or 5 g of palladium sponge inside a length of high-silica glass tubing and retain in place with quartz wool. Place this length inside the tube furnace and fix in place.

11.4 Place a calibrated limiting orifice for 100 to 500 mL/min flow in its holder and attach with tubing to the vacuum pump.

11.5 Attach the combustion tube from 11.3 to the sampling point or prefilters and turn on furnace.

11.6 Remove the stoppers from the silica gel column at the sampling location and insert one-hole stoppers in the column ends. Attach the column to the combustion tube and connect a small clear glass tube of indicating anhydrous calcium sulfate desiccant or indicating silica gel to the outlet of the column.

11.7 Attach the sampling assembly from 11.6 to the limiting orifice and vacuum pump, and the volume meter (gas meter) to the outlet of the air pump.

11.8 Use a flow-measuring device such as a rotameter to check on the sampling flow rate.

11.9 Start the pump and check the differential pressure across the system components to assure that the orifice is controlling the flow rate.

## 12. Calibration and Standardization

12.1 Calibrate the gas meter in accordance with Test Methods D 1071.

12.2 Calibrate the rotameter in accordance with Practice D 3195.

## 13. Procedure

13.1 Record the date and time that the sampling starts and continue sampling for about 1 week, periodically checking the indicating silica gel to determine if and when the silica gel of the sampling column has become saturated with vapor. If a breakthrough is indicated, then record the date and time of the end of the sampling period. If a breakthrough occurs before the end of sampling, the data collected by the method will be suspect.

13.2 At the completion of the sampling period, record the time, date, and the sample location and stop the sampling pump. Record the volume of sample in m<sup>3</sup>.

13.3 Seal the sampling cylinder by replacing one-hole stoppers with solid stoppers and replace the column with a fresh one as in 11.6.

13.4 Transfer the silica gel from the used column to a 1-L distillation flask. Distill the adsorbed water over a period of about 24 h at about 300°C with a heating mantle and quantitatively recover all of the water. Record the volume of