Designation: D 3467 – 99 (Reapproved 2003)

Standard Test Method for Carbon Tetrachloride Activity of Activated Carbon[1]

This standard is issued under the fixed designation D 3467; 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.

INTRODUCTION

Carbon tetrachloride has been identified as a potential contributor to stratospheric ozone depletion. Amendments to the Montreal Protocol in 1990 mandate the global phase-out of carbon tetrachloride production by the year 2000. The 1990 Amendments to the U.S. Clean Air Act were even more aggressive, accelerating the U.S. phase-out deadline to the mid-1990's. A small amount of carbon tetrachloride will still be produced for critical industrial applications; however, in 1993 carbon tetrachloride will not be available for laboratory purposes. With these developments, use of this test method is not recommended.

Instead, the use of Test Method D 5742 is recommended. The correlation obtained between n-butane activity values and carbon tetrachloride activity values is contained in that test method.[2]

1. Scope

1.1 This test method covers the determination of the activation level of activated carbon. Carbon tetrachloride (CCl_4) activity is defined herein as the ratio (in percent) of the weight of CCl_4 adsorbed by an activated carbon sample to the weight of the sample, when the carbon is saturated with CCl_4 under conditions listed in this test method.

1.2 *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.* Specific hazards statements are given in Section 7.

2. Referenced Documents

2.1 *ASTM Standards:* [3]

D 2652 Terminology Relating to Activated Carbon
D 2854 Test Method for Apparent Density of Activated Carbon
D 2867 Test Method for Moisture in Activated Carbon
D 5742 Test Method for the Determination of Butane Activity of Activated Carbon
E 300 Practice for Sampling Industrial Chemicals
E 691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

3. Terminology

3.1 *Definitions*—Terms relating to this test method are defined in Terminology D 2652.

4. Summary of Test Method

4.1 Activity is determined by flowing CCl_4-laden air through a sample of carbon of known weight, under specified conditions, until there is no further increase in the weight of the sample, then determining the weight of the CCl_4 adsorbed. The apparatus required for the test consists essentially of means to control the supply air pressure, to remove oil and water in both liquid and vapor states from the supply air, to produce the specified concentration of CCl_4 in the air flowed through the carbon sample, and to control the flow rate of the gas (air + CCl_4) mixture through the sample.

5. Significance and Use

5.1 Activity as measured by this test method is basically a measure of the pore volume of the activated carbon sample. This test method is therefore a means of determining the degree of completion of the activation process, hence a useful means of quality control for gas-phase activated carbons. This activity number does not necessarily provide an absolute or relative measure of the effectiveness of the tested carbon on other adsorbates, or at other conditions of operation.

[1] This test method is under the jurisdiction of ASTM Committee D28 on Activated Carbon and is the direct responsibility of Subcommittee D28.04 on Gas Phase Evaluation Tests.

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[2] The data for this correlation is available from ASTM International Headquarters. Request RR: D 28–1000.

[3] 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 Document Summary page on the ASTM website.

6. Apparatus and Materials

6.1 *Carbon Tetrachloride*, reagent grade.

6.2 *Supply of Clean, Dry, Oil-Free Air*—The air must be passed through a HEPA filter and a bed of activated carbon containing at least 500 mL of carbon per 1670 mL/min of air flow. Relative humidity of the air must be less than 5 % at 25°C.

6.3 *Balance*, capable of weighing to within ±10 mg.

6.4 *Pressure Regulator*.

6.5 *CCl_4 Gas-Generating Device*, capable of maintaining a CCl_4 concentration of 250 ± 10 mg/L in the air stream at a temperature of 25 ± 1°C, equivalent to a relative saturation of 27.5 %. A typical generation device, shown in Fig. 1, consists of a gas-washing bottle and a refrigerated bath capable of maintaining a bath temperature of 0°C. See also Table 1.

6.6 *Stopcock*, three-way.

6.7 *Regulating Valve*, needle valve, flowmeter, and clock.

6.8 *Adsorption Tube* having the critical dimensions shown in Fig. 1.

6.9 *Thermostat*, capable of maintaining the CCl_4-laden air stream and sample tube at a temperature of 25 ± 1°C.

7. Hazards

7.1 Carbon tetrachloride vapor is toxic and should not be inhaled. It is advisable to handle carbon tetrachloride and the test equipment described in this test method in a well-designed chemical fume-hood. The most recent regulations issued by the Occupational Safety and Health Administration and published in the Federal Register should be followed with regards to allowable human exposure. The most recently obtained Material Safety Data Sheet (MSDS) obtained from the supplier or manufacturer of the carbon tetrachloride should be available as a guide, as well. Acceptable concentrations of carbon tetrachloride for stack release also should conform to the regulations of the United States Environmental Protection Agency, also available in the Federal Register.

8. Sampling

8.1 Guidance in sampling granular activated carbon is given in Practice E 300.

9. Calibration

9.1 Calibration of thermometers, flowmeters, and balances shall be maintained by standard laboratory methods. The concentration of CCl_4 in the gas stream is determined as described in 11.2.

10. Procedure

10.1 Dry the sample using the procedure described in Test Method D 2867.

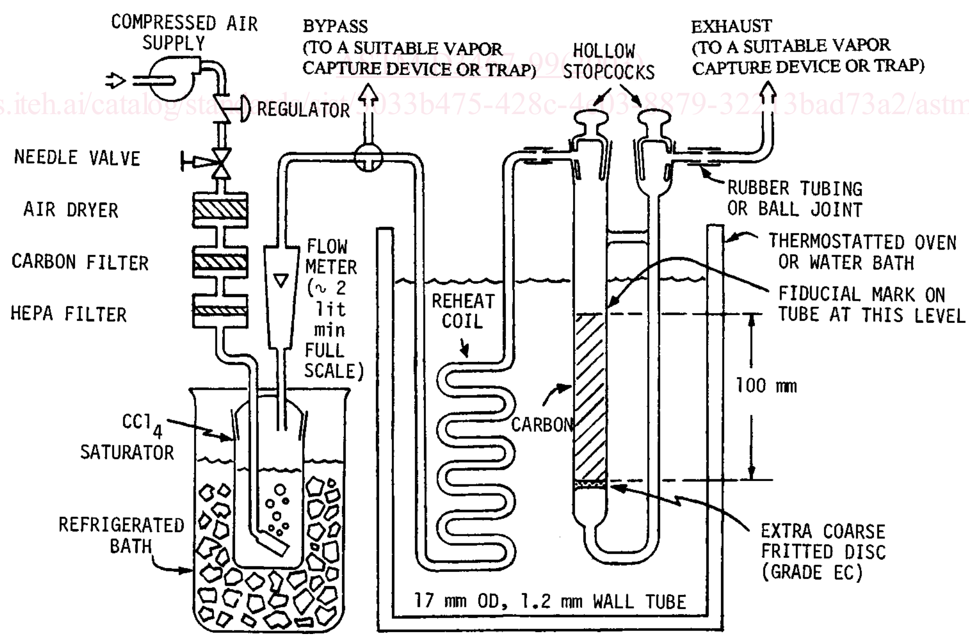
10.2 Weigh the dry sample tube to nearest the 10 mg and record the weight.

10.3 Fill the sample tube to the 100-mm mark ±1 mm, using the vibratory feeder described in Test Method D 2854. Isolate the sample by closing the sample tube stopcocks.

10.4 Weigh the filled sample tube and record the weight.

10.5 Place the filled sample tube vertically in the test assembly (Fig. 1).

10.6 Turn the three-way stopcock to vent the CCl_4-laden air stream, bypassing the sample.



NOTE 1—These items for the apparatus are critical: the dimensions of the sample tube; the location of the flowmeter downstream of the CCl_4 saturator; and the location of the stopcocks. Reheat coil must be adequate to bring the gas temperature to within 1°C of the water bath temperature (that is, bed temperature).

NOTE 2—Additional sampling tubes may be connected in parallel to allow several samples to be run simultaneously, provided flow through each sample is maintained within the limits set in 6.7.

NOTE 3—The pressure at the sample inlet must be maintained at 105 ± 3 kPa.

FIG. 1 Carbon Tetrachloride Activity Test Apparatus