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

This standard is issued under the fixed designation D3467; 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 is classified as a Class I ozone-depleting substance by the U.S. Environmental Protection Agency. Therefore, use of this test method is discouraged.

Instead, the use of Test Method D5742 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 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this 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.* Specific hazards statements are given in Section 7.

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:*[3]

D2652 Terminology Relating to Activated Carbon

D2854 Test Method for Apparent Density of Activated Carbon

D2867 Test Methods for Moisture in Activated Carbon

D5742 Test Method for Determination of Butane Activity of Activated Carbon

E300 Practice for Sampling Industrial Chemicals

E691 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 D2652.

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.

[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] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D28-1000. Contact ASTM Customer Service at service@astm.org.

[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.

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.

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

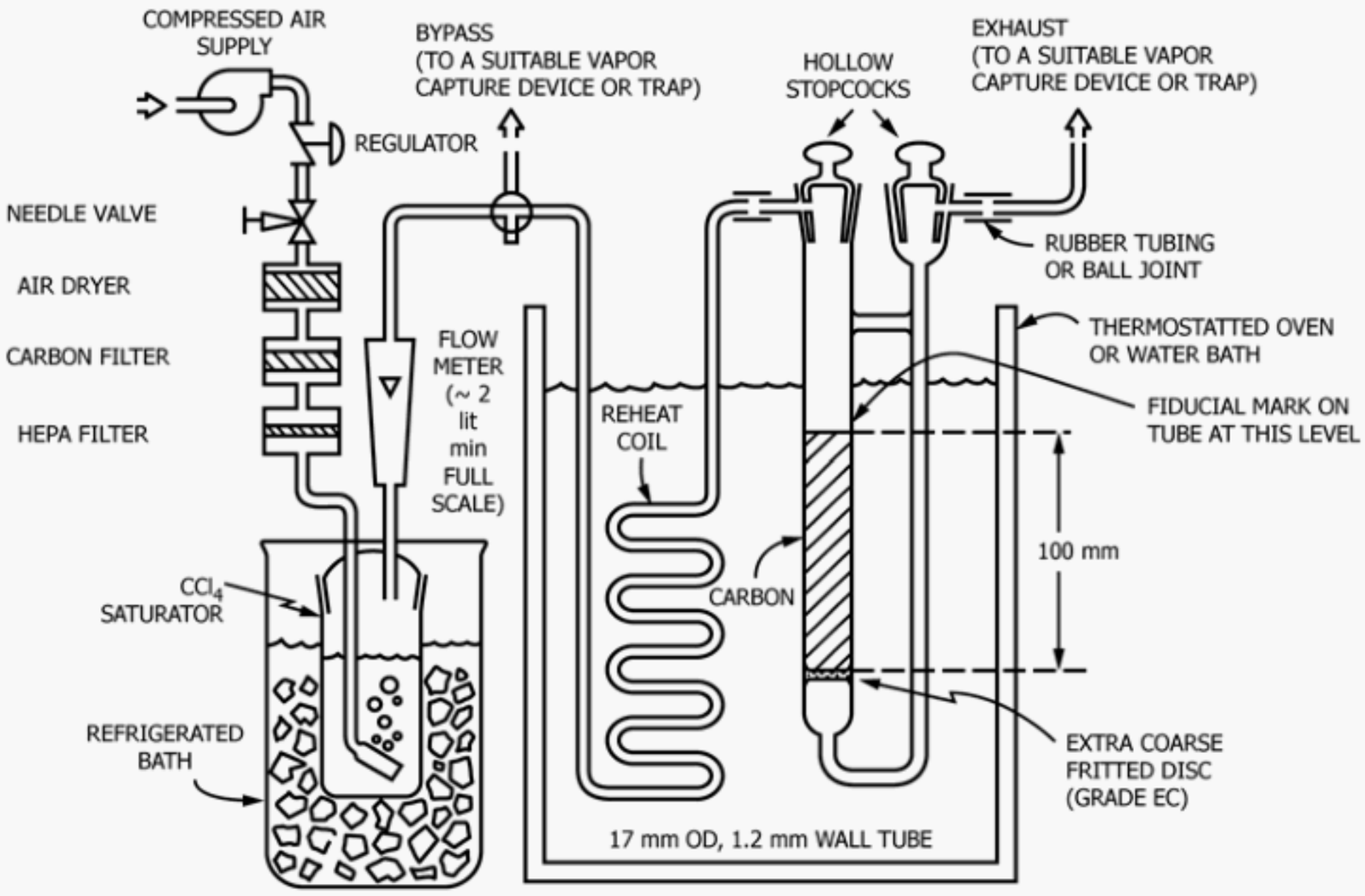
7.1 Carbon tetrachloride vapor is toxic and should not be inhaled. It is advisable to handle carbon tetrachloride and the test equipment described in this test method in a well-designed chemical fume hood. The most recent regulations issued by the Occupational Safety and Health Administration and published in the Federal Register should be followed with regard 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 E300.

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.



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

TABLE 1 CCl_4 Saturation Capacity (Activity) of Granular Activated Carbons

Granule Diameter (Avg) mm	Bed Diameter mm	Number of Runs	Average Activity %	Standard Deviation %	Relative Std. Dev. %
0.55	10.3	19	68.1	±1.8	2.6
0.72	10.3	20	66.2	±1.1	1.7
1.02	23.2	17	61.7	±0.7	1.2
1.30	23.2	20	59.0	±1.1	1.9

10. Procedure

10.1 Dry the sample using the procedure described in Test Methods D2867.

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 D2854. 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.

10.7 Open the sample tube stopcocks. Flow the clean, dry air through the CCl_4 generator until the CCl_4 concentration stabilizes at 250 ± 10 mg/L at 25 ± 1 °C at a total gas flow rate of 1670 ± 15 mL/min, corresponding to a superficial velocity of 10 m/min through the sample tube.

10.8 At the end of the equilibration period, open the sample-tube stopcocks, turn the three-way stopcock to pass the CCl_4-laden air through the sample; note the time. Air flow must be vertically downward through the carbon sample.

10.9 Maintain the gas flow at a rate of 1670 mL/min through the sample for 30 min. At the end of that time, turn the three-way stopcock to vent the gas flow (but maintain the gas flow through the stopcock). Close the sample tube stopcocks. Remove the sample tube from the test assembly, wipe the exterior dry, and weigh to the nearest 10 mg; record the weight.

10.10 Replace the sample tube in the assembly, turn the stopcocks to resume the gas flow through the sample for an additional 10 min.

10.11 At the end of 10 min, repeat the weighing procedure of 6.9.

10.12 Repeat 10.10 and 10.11 until the difference between two successive weighings is less than 10 mg.

11. Calculation

11.1 *Activity*—Determine the CCl_4 activity using Eq 1:

$$A = 100\,(D - C)/(C - B\,) \tag{1}$$

where:

A = carbon tetrachloride activity, as a percent of carbon weight,
B = initial weight of dry sample tube, without carbon, g,
C = initial weight of filled sample tube, g, and
D = final weight of filled sample tube, g.

11.2 *CCl_4 Concentration*—The concentration is normally determined by the first measurement of weight gain, using Eq 2:

$$S = 10^6\,(D_t - C\,)/Q\,t \tag{2}$$

where:

S = concentration of CCl_4, mg/L,
D_t = weight of filled sample tube after t min exposure, g,
C = initial weight of filled sample tube, g,
Q = measured gas flow rate, mL/min, and
t = measured exposure period, min (normally 10 min).

If the carbon sample shows no weight gain between 10 and 20 min exposure, the sample may have been saturated in less than 10 min, and the calculation given in Eq 2 will underestimate the actual concentration. A repeat test run at a time period too short to saturate the carbon will yield a more accurate measure of concentration.

12. Report

12.1 Report the name of the carbon supplier, the grade designation and nominal particle size range, the carbon tetrachloride activity, the name of the agency and technician making the test, and the identification number and date of the test.

13. Precision and Bias

13.1 In a recent round-robin experiment involving three laboratories and four different carbons, the analysis of the data using Practice E691 is shown in Table 2. From this data, the overall relative standard deviation of this test method is 1.2 to 2.6 %.

TABLE 2 Results of Round-Robin Test According to Practice E691

Sample No.	$\bar{x}_j$	$(Sr)_j$	$(S_L)_j$	$(Vr)_j$	$(V_L)_j$	$(S_R)_j$	$(S_R)_j$ %
1	104.76	1.32	1.41	1.26	1.34	1.93	1.84
2	80.89	1.78	1.07	2.20	1.32	2.07	2.56
3	68.17	0.83	0	1.22	0	0.83	1.22
4	67.03	0.79	0.80	1.17	1.19	1.12	1.67

14. Keywords

14.1 activated carbon; carbon tetrachloride