Designation: D 5228 – 92 (Reapproved 2005)

A9

Standard Test Method for Determination of Butane Working Capacity of Activated Carbon[1]

This standard is issued under the fixed designation D 5228; 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.

1. Scope

1.1 This test method covers the determination of the butane working capacity (BWC) of new granular activated carbon. The BWC is defined as the difference between the butane adsorbed at saturation and the butane retained per unit volume of carbon after a specified purge. The test method also produces a butane activity value that is defined as the total amount of butane adsorbed on the carbon sample and is expressed as a mass of butane per unit weight or volume of carbon.

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 and health practices and determine the applicability of regulatory limitations prior to use.* For a specific hazard statement, see 7.1.

2. Referenced Documents

2.1 *ASTM Standards:* [2]

D 2652 Terminology Relating to Activated Carbon

D 2854 Test Method for Apparent Density of Activated Carbon

D 2867 Test Methods for Moisture in Activated Carbon

D 3195 Practice for Rotameter Calibration

E 177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods

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*—For definitions of terms used in this test method, refer to Terminology D 2652.

4. Summary of Test Method

4.1 An activated carbon bed of known volume and mass is saturated with butane vapor. The mass adsorbed at saturation is noted. The carbon bed is then purged under prescribed conditions with dry hydrocarbon free air. The loss of mass is the BWC and is expressed as mass of butane per unit volume of carbon.

5. Significance and Use

5.1 The BWC, as determined by this test method, is a measure of the ability of an activated carbon to adsorb and desorb butane from dry air under specified conditions. It is useful for quality control and evaluation of granular activated carbons that are used in applications where the adsorption of butane and desorption with dry air are of interest. The BWC can also provide a relative measure of the effectiveness of the tested activated carbons on other adsorbates.

5.2 The butane activity and retentivity can also be determined under the conditions of the test. The butane activity is an indication of the micropore volume of the activated carbon sample. The butane retentivity is an indication of the pore structure of the activated carbon sample.

6. Apparatus

6.1 *Water Bath*, capable of maintaining a temperature of 25° ± 0.2°C and of sufficient depth so the entire carbon bed in the sample tube is immersed in the water. A 6-mm OD copper tube with an immersed length of 1.9 m (Fig. 1) provides adequate heat transfer for gas temperature control.

6.2 *Sample Tube*, as shown in Fig. 2. The glass plate with holes is preferred to a fritted disk to support the carbon, since fritted disks can vary widely in pressure drop.

6.3 *Flow Meters*, one capable of delivering air at 0 to 500 mL/min, and one capable of delivering butane at 0 to 500 mL/min, both calibrated in accordance with Practice D 3195.

6.4 *Balance*, capable of weighing to within ± 0.01 g.

6.5 *Fill Device*—The vibration feed device used in Test Method D 2854, Figs. 1 through 4, is preferred.

[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] 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.

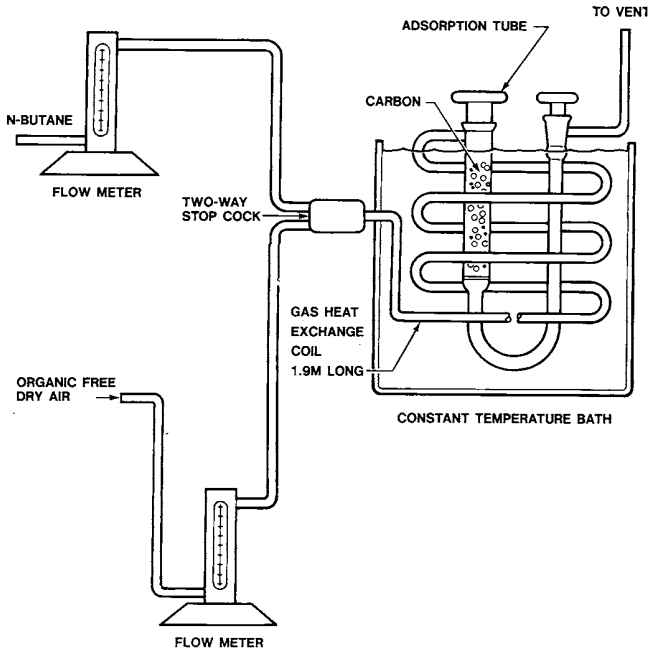
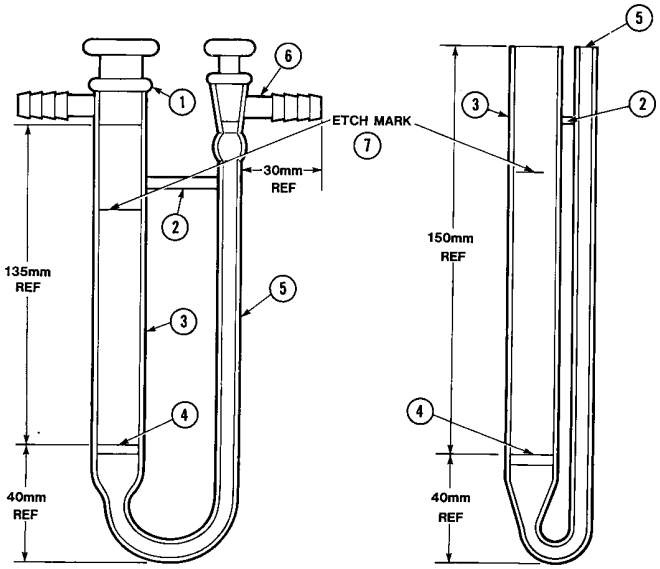


FIG. 1 Butane Working Capacity Apparatus Schematic



1– Ground glass stopper, hollow, medium length, 14/20, from Kontes Catalog No. K-89100 Schwartz adsorption tube, or equivalent.
2– 5-mm rod, brace.
3– 17-mm O.D. × 1.2 mm standard wall tubing.
4– Coor's perforated porcelain disk or extra coarse fritted disk, or equivalent.
5– 10-mm O.D. × 1.0 mm standard wall tubing.
6– Right angle stopcock, Kontes Catalog No. K-84700, size 4, 10 mm O.D. stem, with Kontes Catalog No. K-89340 size B serrated hose connector, or equivalent.
7– Dimension corresponding to a volume of 16.7 mL above the retainer plate.

FIG. 2 Butane Working Capacity Sample Tube

6.6 *Buret*, Class A, 25 mL capacity.

6.7 *Apparatus Assembly* shown in Fig. 1.

7. Reagents

7.1 *n-Butane*, C. P. Grade. (**Warning**—Butane is a flammable gas with a flash point of −138°C and a boiling point of 0.5°C. Its specific gravity is 2.046 relative to air. Butane may be narcotic in high concentrations and is considered a simple asphyxiant. If the entire apparatus is not set up in a fume hood, provision must be made to vent the gas coming from the discharge stem of the sample tube.)

7.2 *Dry Air*, free of organics, with a dew point no higher than −32°C.

8. Sampling

8.1 For guidance in sampling granular activated carbon, refer to Practice E 300.

9. Calibration of a Sample Tube

9.1 Clean and dry the sample tube to prevent any water droplets from adhering to the inner surface of the tube.

9.2 Using distilled water, carefully fill the sample tube through the narrow side stem to prevent the introduction of any air bubbles.

9.2.1 Hold the sample tube upright while slowly introducing the distilled water. Air bubbles have a tendency to form directly below the retainer plate of the tube.

9.3 Clamp the filled sample tube in an upright position to a ring stand and stopper the narrow side stem.

9.4 Using a pipet, carefully remove the water from the sample tube to the top of the retainer plate. Caution must be taken so no water is removed from below the retainer plate creating air bubbles that would result in a spurious calibration of the sample tube. If this occurs, the tube must be refilled by repeating 9.1 through 9.3.

9.5 Using the buret, fill the sample tube with 16.7 ± 0.05 mL of water, then etch the tube at the level of the meniscus.

10. Maintenance of Bath Water

10.1 In order to prevent mold formation, the bath water should be changed periodically.

11. Procedure

11.1 Dry an adequate sample as prescribed in Test Methods D 2867, Section 4.

11.2 Determine the apparent density in accordance with Test Method D 2854 and record.

11.3 Accurately weigh the empty, dry sample tube and stoppers to the nearest 0.01 g and record.

11.4 Fill the adsorption tube with carbon to the etch mark at a rate of 0.35 to 1.0 mL/s using the vibrating feeder apparatus described in Test Method D 2854 with a funnel modified to accommodate the adsorption tube. Larger particles will require the slower fill rate to achieve the required packing density.

11.5 Weigh the filled sample tube and stoppers to the nearest 0.01 g and record.

11.5.1 The sample packing density, carbon weight/16.7, must be equal to at least 94 % of its dry apparent density if the result of the determination is to be reproducible. Otherwise, the ratio of purge air volume to sample volume will vary, and the quantity of butane purged will also vary from one determination to the next. If the specified packing density is not achieved in the first attempt, the filling procedure must be repeated until

attempts the desired packing density cannot be achieved, the percentage should be noted and the procedure continued.

11.6 Set the water bath control to maintain a temperature of 25 ± 0.2°C.

11.7 Clamp the sample tube in a vertical position in the 25 ± 0.2°C water bath and attach the tube to the output of the flow meter. If the entire apparatus is not in a hood, attach a short length of tubing from the effluent side of the sample tube to an exhaust vent.

11.8 Regulate the flow to pass butane through the carbon bed at 250 ± 5 mL/min. Continue the flow of butane for at least 900 s.

11.9 Turn off the butane, disconnect the tubing, and immediately stopper the sample tube. Remove the sample tube from the water bath, dry the sample tube, and visually inspect the tube for any condensed water vapor. If any condensed water is observed, stop the testing and begin the test procedure again.

11.10 Weigh the filled sample tube and its stoppers to the nearest 0.01 g and record.

11.11 Reconnect the sample tube to the apparatus and flow butane for an additional 600 s and weigh and record. Repeat to constant weight.

11.12 Reconnect the saturated carbon and sample tube and purge with dry organic free air for 2400 ± 20 s at 300 ± 5 mL/min.

11.13 Turn off the purge flow, disconnect tubing, install stoppers, remove the sample tube from the water bath, and dry.

11.14 Weigh to the nearest 0.01 g and record.

12. Calculation

12.1 The calculations described in this section are based upon the following determinations made during the course of the procedure:

A = Apparent density from 11.2,
B = Weight of sample tube and stoppers,
C = Weight of carbon sample, sample tube, and stoppers,
D = Weight of saturated carbon, sample tube, and stoppers, and
E = Weight of purged carbon, sample tube, and stoppers.

NOTE 1—A sample data and calculations sheet for BWC determinations is given in Annex A1.

12.2 Calculate the BWC on weight and volume bases as follows:

$$\text{BWC, W/W \%} = \frac{(D - E)}{(C - B)} \times 100 \quad (1)$$

$$\text{BWC, W/V g/100 mL} = \frac{(D - E)}{(C - B)} \times A \times 100 \quad (2)$$

12.3 Calculate the butane activity on weight and volume bases as follows:

$$\text{Butane activity, W/W \%} = \frac{(D - C)}{(C - B)} \times 100 \quad (3)$$

$$\text{Butane activity, W/V g/100 mL} = \frac{(D - C)}{(C - B)} \times A \times 100 \quad (4)$$

12.4 Calculate the butane retentivity on weight and volume bases as follows:

$$\text{Butane retentivity, W/W \%} = \frac{(E - C)}{(C - B)} \times 100 \quad (5)$$

$$\text{Butane retentivity, W/V g/100 mL} = \frac{(E - C)}{(C - B)} \times A \times 100 \quad (6)$$

13. Report

13.1 The analysis report shall include the following information:

13.1.1 Name of activated carbon supplier,
13.1.2 Grade designation of the sample,
13.1.3 Nominal partial size range,
13.1.4 Butane working capacity,
13.1.5 Butane activity,
13.1.6 Butane retentivity,
13.1.7 Name of the agency and technician running the test,
13.1.8 Identification number and date of the test, and
13.1.9 Lot number from which the sample was taken.

14. Precision and Bias

14.1 An interlaboratory study of this test method was conducted in 1990.[3] Each of eight laboratories tested three randomly drawn test specimens from each of three different activated carbons. Practice E 691 and its computer software were followed for the design of the study and the data analysis.

14.2 *95 % Limit on Repeatability (within Laboratory) in Percent*:

	Activated Carbon		
	A	B	C
Activity %, weight/weight	1.95	2.34	.97
Retentivity %, weight/weight	2.52	1.80	1.77
Working capacity %, weight/weight	2.05	3.14	2.19
Activity g/100 mL, weight/vol	0.68	0.70	0.40
Retentivity g/100 mL, weight/vol	0.72	0.52	0.95
Working capacity g/100 mL, weight/vol	0.51	0.92	1.04

14.3 *95 % Limit on Reproducibility (between Laboratories) in Percent*:

	Activated Carbon		
	A	B	C
Activity %, weight/weight	3.57	3.15	1.05
Retentivity %, weight/weight	3.75	3.79	3.79
Working capacity %, weight/weight	5.06	4.70	3.83
Activity g/100 mL, weight/vol	0.91	1.08	0.57
Retentivity g/100 mL, weight/vol	1.05	1.22	1.84
Working charge g/100 mL, weight/vol	1.41	1.51	2.06

14.4 *Apparent Density's 95 % Repeatability and Reproducibility Limits:*

	Activated Carbon		
	A	B	C
Repeatability g/100 mL	0.012	0.004	0.008
Reproducibility g/100 mL	0.019	0.021	0.025

NOTE 2—The terms repeatability and reproducibility limit are used as specified in Practice E 177.

[3] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR: D-28-1003.

ANNEX

(Mandatory Information)

A1. DATA AND CALCULATIONS SHEET FOR BWC DETERMINATIONS

A1.1 See Fig. A1.1:

Laboratory:_____
Test Performed By:_____ Date:_____
Sample Description:_____

A Sample AD	_____
B Weight of sample tube and stoppers	_____
C Weight of carbon sample, tube, and stoppers	_____
D Weight of saturated carbon, tube, and stoppers	_____
E Weight of purged carbon, tube, and stoppers	_____
Butane working capacity W/W %	_____
Butane working capacity W/V g/100 mL	_____
Butane activity W/W %	_____
Butane activity W/V g/100 mL	_____
Butane retentivity W/W %	_____
Butane retentivity W/V g/100 mL	_____

FIG. A1.1 Data and Calculations Sheet for BWC Determinations