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100 Barr Harbor Dr., West Conshohocken, PA 19428
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Standard Test Method for Water Solubles in Activated Carbon[1]

This standard is issued under the fixed designation D 5029; 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 water soluble content of (unused) granular and powdered activated carbons. Water solubles are materials that can be extracted by distilled water under reflux conditions and are expressed as a percentage of dry carbon weight.

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.*

2. Referenced Documents

2.1 *ASTM Standards:*

D 1193 Specification for Reagent Water[2]
D 2652 Terminology Relating to Activated Carbon[3]
D 2867 Test Method for Moisture in Activated Carbon[3]
D 3838 Test Method for pH of Activated Carbon[3]
E 177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods[4]
E 300 Practice for Sampling Industrial Chemicals[5]

3. Terminology

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

4. Summary of Test Method

4.1 A known weight of activated carbon is placed into a reflux apparatus with Type II reagent water (see Specification D 1193). The mixture is refluxed for 15 min under specified conditions. This extraction is performed using the method and apparatus described in Test Method D 3838. After extraction, the carbon is separated by filtration and an aliquot of the filtrate is evaporated to dryness. Water solubles are determined by weighing the dry residue and expressing the result as a percentage of the dry carbon weight.

5. Significance and Use

5.1 In certain applications, the ash, color, conductivity, or pH of the finished activated carbon product may be influenced by the quantity of water solubles it contains. This water solubles test provides a relative indication of the quantity of soluble materials that may be extracted from various activated carbons.

6. Apparatus and Materials

NOTE 1—All volumetric measuring equipment should meet or exceed the requirements of National Institute of Standards and Technology Circular 602, *Testing of Glass Volumetric Apparatus*, available from the National Institute of Standards and Technology, Gaithersburg, MD 20899. Volumetric glassware meeting these specifications is generally designated as Class A.

6.1 *Flask*, 250 mL with 24/40 ST (standard taper) neck.
6.2 *Condenser*, with 24/40 inner ST (standard taper) joint.
6.3 *Buchner Funnel*, 9 or 12.5 cm.
6.4 *Filter Paper, Ashless*, (~5 to 10 µm particle retention).
6.5 *Glass or Porcelain Evaporating Dishes*, 100 mL capacity.
6.6 *Analytical Balance*, precision 0.1 mg.
6.7 *Drying Oven*.
6.8 *Desiccator*.
6.9 *Hot Plate*.
6.10 *Pipet*, 50 mL.
6.11 *Indicating Desiccant*.
6.12 *Water*, ASTM Type II or better, in accordance with Specification D 1193, Type II.
6.13 *Thermometer*, approximately 20 to 55°C.
6.14 *Steam Bath*, optional.
6.15 *Beakers*, 250 mL.
6.16 *Graduated Cylinder*, 100 mL.
6.17 *Laboratory Timer*.
6.18 *Filter Flasks*, vacuum, 500 mL.

7. Sampling

7.1 Conducted sampling according to Practice E 300.

8. Procedure

8.1 Determine the moisture content of the carbon in accordance with Test Method D 2867.

8.2 Weigh a sample of carbon equivalent to 10.00 ± 0.01 g on a dry basis. Remove boiler flask from apparatus (see

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[2] *Annual Book of ASTM Standards*, Vol 11.01.
[3] *Annual Book of ASTM Standards*, Vol 15.01.
[4] *Annual Book of ASTM Standards*, Vol 14.02.
[5] *Annual Book of ASTM Standards*, Vol 15.05.

Boiler-Reflux Condenser Figure in Test Method D 3838) and add carbon sample.

8.3 Bring approximately 110 mL of reagent water to a boil. Measure 100.0 ± 0.5 mL into a graduated cylinder while the water is hot. Add a small portion of the 100.0 ± 0.5 mL of water to wet the carbon. Wash down the sides of the flask with the remaining portion. Connect the flask to the condenser and place on a hot plate.

8.4 Bring the water to a gentle boil to ensure that no carbon splashes onto the side of the flask.

8.5 Boil gently for 900 ± 10 s.

8.6 Remove the flask from the hot plate and filter its contents immediately through the filter paper premoistened with the Type II water used for the test. Catch the filtrate in a 500-mL vacuum filter flask, being careful to prevent carbon fines from entering the filtrate.

8.7 Cool the filtrate to ambient temperature. (The pH may be measured on a portion of the filtrate.)

8.8 Dry the glass or porcelain evaporating dishes at 150 ± 5°C to a constant weight (±0.1 mg). Evaporating dishes must be cooled to ambient temperature and stored in a desiccator between weighings. Weigh the dry evaporating dish to the nearest 0.1 mg and record.

8.9 Using a pipet, transfer a 50-mL aliquot of the filtrate to a tared glass or porcelain evaporating dish.

8.10 Evaporate the filtrate to dryness in an oven or on a steam bath until the liquid disappears. Avoid boiling to prevent loss of residue.

8.11 Dry the residue at 150 ± 5°C for a minimum of 1 h and ensure dryness to constant weight (±0.1 mg). The evaporating dish containing the residue must be cooled to ambient temperature and stored in a desiccator between weighings. Weigh the evaporating dish and residue to the nearest 0.1 mg and record.

8.12 If the residue is less than 10 mg, repeat the procedure. Add the new aliquot during 8.9 to the evaporating dish containing residue from the previous aliquot.

8.13 Make two determinations on each carbon sample tested.

9. Calculation

9.1 The following equation is used for a general calculation of water solubles:

$$\text{Water Solubles, \%} = \frac{(B-A)\,(D)\,(100)}{(C)\,(E)} \quad (1)$$

where:

A = mass of evaporating dish, g,
B = mass of evaporating dish plus residue, g,
C = mass of carbon, g,
D = volume of water used in extraction, mL, and
E = volume of aliquot used, mL.

9.1.1 As an example, for extraction of one carbon sample, and evaporation of a 50-mL aliquot, the water solubles calculation is:

$$\text{Water Solubles, \%} = \frac{RW}{CW} \times 200$$

10. Report

10.1 Report the following:

10.1.1 Source of sample.

10.1.2 Type or designation of activated carbon.

10.1.3 Supplier name.

10.1.4 Supplier grade designation.

10.1.5 Supplier lot and batch number.

10.1.6 Moisture content in accordance with Test Method D 2867.

10.1.7 Water solubles content.

10.1.8 Date of test.

10.1.9 Name and signature of technician performing test.

10.1.10 Name and signature of supervisor approving test.

11. Precision and Bias

11.1 *Precision*:

11.1.1 *Repeatability*—Repeatability of this test method is ±20 % of the average value from three or more determinations. This range corresponds to 2*S* % as defined in Practice E 177.

11.1.2 *Reproducibility*—Reproducibility for this test method is ±35 % (2*S* %) of the calculated value.

11.1.3 These statements are based on a round robin trial of this test method on activated carbons from five different raw material bases tested by four different laboratories.

12. Keywords

12.1 activated carbon