Designation: D6385 – 22

Standard Test Method for Determining Acid-Extractable Content in Activated Carbon by Ashing[1]

This standard is issued under the fixed designation D6385; 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 is to be used for determination of the acid-extractable content of a sample of activated carbon. This test method presupposes the existence of substances other than carbon to be present with activated carbon but does not purport to address or identify those substances which may be present. This test method should be applicable to any form in which activated carbon may exist and requires the performance of Test Method D2866.

1.2 This test method requires the use of concentrated hydrochloric acid, which should be used in an appropriate and safe manner, with eye protection, skin protection, and handling carried out in a properly operating fume hood. The proper use of a muffle furnace is addressed in Test Method D2866.

1.3 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.4 *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.*

1.5 *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:*[2]

D1193 Specification for Reagent Water

D2866 Test Method for Total Ash Content of Activated Carbon

D2867 Test Methods for Moisture in Activated Carbon

E300 Practice for Sampling Industrial Chemicals

3. Summary of Test Method

3.1 The percent acid-extractable content of an activated carbon sample is determined by the difference between the percent mass of total ash of a sample and the percent mass of total ash of a sample which has been acid extracted.

4. Significance and Use

4.1 The quantitative determination of acid-extractable content is useful in evaluating activated carbon samples that contain acid-soluble impurities. These acid-soluble impurities can affect applications of activated carbon.

4.2 *Limitations of Method*—Hydrochloric acid is used in this test method as the extracting acid. All elements or compounds present in the activated carbon sample, which can be acid extracted, are assumed to form water-soluble chloride salts. Hydrochloric acid may not solubilize all impurities of activated carbon.

5. Apparatus

5.1 *250 mL Glass Beakers or 125 mL Capacity Block Heater Digestion Vessel,* for boiling and drying the sample.

5.2 *Watch Glass,* suitable for 250 mL beaker or 125 mL Capacity Block Heater Digestion Vessel.

5.3 *Graduated Cylinders,* of 100 mL (TD) and 25 mL (TD) size.

5.4 *Buchner Vacuum Funnel,* 7cm ID or equivalent filtering device.

5.5 *Vacuum Filter Flask,* with side arm, 500 mL.

5.6 *Vacuum Filter Collar,* for the above filter flask.

5.7 *Filter Paper,* which is hardened, ashless paper, ≤ 8 micron pore size and 7 cm in diameter is recommended, or a diameter which matches the vacuum funnel. The paper will be ashed with the carbon, so ashless paper is necessary.

5.8 *Drying Oven,* maintained at 150 °C ± 5 °C.

[1] This test method is under the jurisdiction of ASTM Committee D28 on Liquid Phase Evaluation.

Current edition approved Sept. 1, 2022. Published October 2022. Originally approved in 1999. Last previous edition approved in 2017 as D6385 – 99 (2017). DOI: 10.1520/D6385-22.

[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.

5.9 *Muffle Furnace,* as specified in Test Method D2866.

5.10 *Porcelain Crucibles,* as specified in Test Method D2866.

5.11 *Desicator,* containing indicating silica gel or any other desicant, which is effective in maintaining dryness of cooling hot crucibles or oven dried carbon samples.

5.12 *Balance,* capable of measurement of 0.1 mg.

5.13 *Carbon Particle Size Reduction,* a mortar and pestle, or any other means of systematically reducing activated carbon particle size to pass through a No. 325 U.S. mesh screen. The size reduction device must be constructed of material that will not contaminate the carbon sample with acid-soluble materials.

5.14 *No. 325 U.S. Mesh Screen,* for sieving the sample after size reduction.

6. Reagents

6.1 *Concentrated Hydrochloric Acid,* ACS reagent grade is recommended, 37 % HC1 w/w ± 2 %.

6.2 *ASTM Type II Water,* or better is recommended for low residue content. Whenever water is used in this test method, Type II or better is implied.

7. Test Sample

7.1 A test sample or samples should be representative of the material being tested in accordance with Practice E300. Samples should be uniformly dried to a constant weight before proceeding with this test method. If drying is not possible, then percent moisture should be determined by Test Methods D2867 and the appropriate correction to the sample weight should be made.

8. Procedure

8.1 A representative sample or samples should be ground or milled so that 95 % or more of the sampled mass will be passed through a No. 325 U.S. mesh screen. Retain all the sample for the test.

8.2 Determine the total ash content of this sample by Test Method D2866.

8.3 Weigh out to the nearest 0.1 mg sufficient dried activated carbon so that the estimated amount of ash will be at least 0.1 g.

8.4 Quantitatively transfer to a 250 mL beaker or 125 mL capacity digestion vessel.

8.5 Add reagents to the carbon in the beaker (or digestion vessel) in the following order: slowly add 100 mL (±1 mL) of water, then slowly add 25 mL (±1mL) of concentrated hydrochloric acid. Swirl beaker (or digestion vessel) to wet sample thoroughly.

8.6 Place the beaker (or digestion vessel) and contents on a hot plate and bring to a boil. Allow the carbon acid-water mixture to boil for 5 min ± 10 s. A watch glass should be used to cover the beaker (or digestion vessel) while boiling the carbon acid-water mixture.

8.7 After boiling, remove the beaker (or digestion vessel) from heat, and then vacuum filter the hot carbon acid-water mixture immediately through the 7 cm filter funnel with hardened filter paper in place. Wash the carbon retained on the filter paper with five (5), 5 mL portions of water to thoroughly remove all acid residue. Discard filtrate unless analysis of acid-soluble components in this extract is desired.

8.8 Vacuum filter the mixture through the 7 cm filter funnel with hardened filter paper in place. Wash the carbon retained on the filter paper with several portions of water to thoroughly remove all acid residue. Discard filtrate unless analysis of acid-soluble components in this extract is desired.

8.9 Transfer the filter, filter paper and carbon together into an oven and dry for at least 30 min at 150 °C. This process permits easy separation of the paper and carbon from the Buchner filter.

8.10 Quantitatively transfer the paper and carbon to a tared crucible.

8.11 Determine the ash content of the dried, acid-extracted carbon, using the mass of the carbon sample from 8.3. Using a suitable muffle furnace at 650 °C, thoroughly ash the carbon and paper as described in Test Method D2866.

9. Calculation of Result

9.1 The acid-extractable content as previously defined is calculated as follows (correction for water content is necessary if the sample was not dried before ashing):

$$\text{Acid Extractable Content}\,(\%) = (A - B) \tag{1}$$

where:

A = percent total ash content of activated carbon sample as determined by Test Method D2866, and

B = percent total ash content of acid-extracted sample, also as determined by Test Method D2866.

10. Precision and Bias

10.1 Based on limited information from one laboratory, the repeatability standard deviation and 95 % repeatability limits are as follows:

$$\textit{Repeatability Standard Deviation}: 9.3\,\% \tag{2}$$

$$95\,\%\ \textit{Repeatability Limits}: 0.90 \tag{3}$$

10.2 The percent acid-soluble ash is dependent on the percent total ash content of the original sample. The precision and repeatability of Test Method D2866 also is a factor in the repeatability of this test method.

11. Keywords

11.1 acid; acid extractable; activated carbon; ash