



Designation: D 5919 – 96 (Reapproved 2001)

## Standard Practice for Determination of Adsorptive Capacity of Activated Carbon by a Micro-Isotherm Technique for Adsorbates at ppb Concentrations<sup>1</sup>

This standard is issued under the fixed designation D 5919; 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 ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

### 1. Scope

1.1 This practice covers the assessment of activated carbon for the removal of low concentrations of adsorbable constituents from water and wastewater using the bottle point isotherm technique. It can be used to characterize the adsorptive properties of virgin and reactivated activated carbons.

1.2 This practice can be used in systems with constituent concentrations in the low milligrams per litre or micrograms per litre concentration ranges.

1.3 This practice can be used to determine the adsorptive capacity of and Freundlich constants for volatile organic compounds provided the handling procedures described in this practice are followed carefully.

1.4 The following safety caveat applies to the procedure section of this practice: *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 1129 Terminology Relating to Water<sup>2</sup>

D 1193 Specification for Reagent Water<sup>2</sup>

D 3370 Practices for Sampling Water from Closed Conduits<sup>2</sup>

D 2652 Terminology Relating to Activated Carbon<sup>3</sup>

D 2867 Test Method for Moisture in Activated Carbon<sup>3</sup>

### 3. Terminology

#### 3.1 Definitions:

3.1.1 For definitions of terms used in this practice relating to activated carbon, refer to Terminology D 2652.

<sup>1</sup> This practice is under the jurisdiction of ASTM Committee D28 on Activated Carbon and is the direct responsibility of Subcommittee D28.02 on Liquid Phase Evaluation.

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<sup>2</sup> Annual Book of ASTM Standards, Vol 11.01.

<sup>3</sup> Annual Book of ASTM Standards, Vol 15.01.

3.1.2 For definitions of terms used in this practice relating to water, refer to Terminology D 1129.

### 4. Summary of Practice

4.1 This practice consists of the determination of the adsorptive capacity of activated carbon for adsorbable constituents by contacting the aqueous solution contained in an essentially zero head space container with activated carbon, determining the amount of the constituents removed, and calculating the adsorptive capacity and the Freundlich constants,  $K$  and  $1/n$ , from a Freundlich isotherm plot.

4.1.1 The weights of activated carbon used in this practice may have to be adjusted to achieve reasonable levels of removal of the constituent. The best data is obtained when carbon dosages are selected that result in no more than 90 % or no less than 10 % of the adsorbable constituents being removed from the water by the carbon.

4.1.2 If carbon dosages used are less than 1 mg, larger volumes of the aqueous solution may be used, such as 1000 mL.

### 5. Significance and Use

5.1 This practice allows the adsorption capacity at equilibrium of an activated carbon for adsorbable constituents present in water to be determined. The Freundlich  $K$  and  $1/n$  constants that can be calculated based upon information collected using this practice can be used to estimate carbon loading capacities and usages rates for the constituent present in a water stream at other concentrations.

### 6. Interferences

6.1 The water shall not contain any nondissolved components.

6.2 The presence of naturally occurring organic compounds such as humic acids in the water being studied may significantly affect the ability of the carbon to adsorb the constituent of interest. Results obtained when using water other than reagent grade water may be unique for the particular water used and it may not be possible to apply these results to other water systems.

6.3 The adsorption isotherm data collected using this practice can be affected by the ionic strength, pH and temperature of the water, and the presence and growth of microorganisms.

## 7. Apparatus

7.1 *Equilibrator* (or other rotating mixing device), a rotating device operating at 25 rpm which can rotate the isotherm bottles end over end ensuring good dispersion of the powdered activated carbon in the water being treated.

7.2 *Grinding Mill*, capable of grinding material so that 90 % passes through a U.S. No. 325-mesh (45  $\mu\text{m}$ ) sieve.

7.3 *Isotherm Bottles*, narrow neck amber bottles with polytetrafluoroethylene (PTFE) coated septum sealed caps of 250, 500, and 1000 mL capacity suitable for use in a centrifuge operating at 2000 rpm.

7.4 *Solution Delivery Tank*, a 10-L, 316 stainless steel container equipped with a PTFE coated floating lid and a 316 stainless ball valve to control flow during bottle filling.

7.5 *Analytical Balance*, capable of weighing to the nearest 0.1 mg.

7.6 *Oven*, forced-air circulation, capable of temperature regulation up to 250°C.

7.7 *Centrifuge*, capable of handling isotherm bottles up to 1 L in size at 2000 rpm.

7.8 *Magnetic Stirring Bars and Stirrers*.

## 8. Reagents

8.1 *Reagent Water*, in accordance with Specification D 1193, Type II.

8.2 *Methanol*, high purity HPLC grade.

8.3 *Potassium Monobasic Phosphate* ( $\text{KHPO}_4$ ), 1 M solution.

8.4 *Sodium Hydroxide* ( $\text{NaOH}$ ), 1 M solution.

## 9. Cleaning Procedures

9.1 This practice is capable of generating activated carbon adsorption capacity data on aqueous solutions containing ppbw ( $\mu\text{g/L}$ ) levels of adsorbable constituents. It is therefore very important that all equipment and glassware that come in contact with the activated carbon or the water being treated be cleaned thoroughly to remove trace organic compounds.

9.2 All equipment and glassware should first be rigorously cleaned using procedures recommended by the EPA for priority pollutant analysis, hot water and detergent wash, reagent grade water, and solvent (high purity methanol) rinse followed by a bake-out.

9.3 The glassware is baked out in an oven at 250°C for a minimum of one hour. All PTFE and stainless steel apparatus are dried at 110°C for one hour.

## 10. Preparation of the Activated Carbon

10.1 This practice requires the use of well washed activated carbon that has been reduced in particle size so that 90 % or greater passes through a U.S. No. 325-mesh (45  $\mu\text{m}$ ) sieve by wet screening or equivalent.

10.2 Approximately 25 g of the powdered activated carbon sample is placed into each of four clean 250-mL bottles. The remainder of the bottle is filled with reagent grade water.

10.3 The bottle is tightly capped and inverted three to five times to mix the contents.

10.4 The bottles are then centrifuged at 2000 rpm for 15 min to settle the activated carbon. The supernate is poured off and the procedure is repeated until the supernatant is clear. Allowing the mixture to sit for a period of time to allow the carbon to settle prior to decanting is also acceptable.

10.5 The wet carbon is next dried in an oven at 110°C to a constant weight and placed in a desiccator to cool.

10.6 As an alternate technique to drying the carbon sample, carbon may be placed in a Soxhlet extraction device and extracted for a period of up to 1 week with reagent grade (Type II) water.

10.7 The dry activated carbon is transferred to clean 1-L brown borosilicate bottles with PTFE liners in the caps and stored in an inert atmosphere such as nitrogen for future use.

## 11. Activated Carbon Sample Weighing Procedure

11.1 This procedure allows the carbon to be handled at ambient conditions by calculating a correction for water adsorbed from the air.

11.2 The powdered activated carbon sample is allowed to come to equilibrium in a desiccator containing a saturated salt solution that will produce a relative humidity comparable to ambient laboratory conditions. During the 24 h conditioning period care shall be taken not to expose the carbon to organic vapors.

11.3 The moisture picked up by the conditioned activated carbon is determined by weighing approximately 500 mg into a tared (constant weight) bottle, drying for 2 h at 110°C, cooling in a desiccator, and re-weighing to determine weight change (refer to Test Method D 2867 for standard procedures). The ratio of change in weight between the activated carbon at equilibrium with air and after drying is calculated and used as a correction factor for the weighed carbon dosages.

11.4 The carbon dosages are weighed by first taking a weighing boat, adding the desired mass of equilibrated activated carbon, and re-weighing the boat after transferring the carbon to the bottle. The carbon dosage is the difference between the carbon plus the boat weight and the final boat weight. The weighed activated carbon dosage is then corrected for ambient conditions and the actual dried carbon dosage recorded.

## 12. Alternative Procedure for Addition of Known Quantities of Activated Carbon to Isotherm Bottles

12.1 This alternate procedure makes use of a clean dry activated carbon sample prepared according to procedures described in Section 10. Desired concentrations of carbon are added to each isotherm bottle volumetrically using a carbon slurry of known concentration.

12.2 The concentrations of the slurries are chosen so that 5, 10, and 20 mL volumes of each slurry would contain appropriate amounts of carbon for 250 mL isotherm bottles.

12.3 The slurries are pipetted into a pre-weighed baked-out isotherm bottle, baked dry in a 105°C oven, cooled, and re-weighed to determine the exact quantity of carbon added to the bottle. This drying technique eliminates any dilution of the water sample to be tested, allows the slurry pipet to be rinsed