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Standard Practice for Determination of Adsorptive Capacity of Activated Carbon by a Micro-Isotherm Technique for Adsorbates at ppb Concentrations[1]

This standard is issued under the fixed designation D5919; 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 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 values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.5 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:*[2]

D1129 Terminology Relating to Water
D1193 Specification for Reagent Water
D2652 Terminology Relating to Activated Carbon
D2867 Test Methods for Moisture in Activated Carbon
D3370 Practices for Sampling Water from Closed Conduits

3. Terminology

3.1 *Definitions:*

3.1.1 For definitions of terms used in this practice relating to activated carbon, refer to Terminology D2652.

3.1.2 For definitions of terms used in this practice relating to water, refer to Terminology D1129.

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 headspace 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

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[1] 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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[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.

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-µm) 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 D1193, Type II.

8.2 *Methanol,* high purity HPLC grade.

8.3 *Potassium Monobasic Phosphate* (KHPO4), 1 *M* solution.

8.4 *Sodium Hydroxide* (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 (µg/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-µm) 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 Methods D2867 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 into the isotherm bottle to ensure complete delivery, and causes the carbon particles to adhere to the container walls which will minimize carbon loss during bottle filling.

12.4 The isotherm bottles containing the carbon are kept tightly capped until a sample is to be introduced.

13. Calculation Procedures for Determining Carbon Dosages

13.1 Preliminary Freudlich constants, K and $1/n$, are either taken from published literature values or estimated using Polanyi adsorption potential theory.[3]

13.2 The carbon dosages are calculated to give a constituent removal of from 10 % at the lowest carbon weight to 90 % for the highest weight. For a target (final) constituent concentration, C_e, the carbon dosage is calculated based on the following mass balance within the isotherm bottle:

$$M = V[C_o - C_e]/[KC_e^{1/n}] \tag{1}$$

where:

K and $1/n$ = as determined in 13.1,
M = required carbon dosage, g,
V = volume of isotherm bottle, L,
C_o = initial constituent concentration, mg/L, and
C_e = target (final) constituent concentration, mg/L.

14. Solution Preparation and Handling

14.1 The source of the water used in this practice can originate from a contaminated water or wastewater source or can be prepared in the laboratory using pure constituents and reagent grade water (refer to Practices D3370 for water sampling). The source water can be used directly in this practice provided it is essentially free of particulate matter. The pH of the water should be checked and adjusted or corrected as appropriate.

14.2 The preparation of a laboratory solution requires the use of a 10-L, 316 stainless steel delivery container equipped with a floating cover and flow control ball valve.

14.3 If reagent grade water is to be used, it may be buffered to avoid pH effects on the adsorption of the organic constituent. The buffer is prepared by adding 1 mL of a 1 *M* potassium monobasic phosphate solution to 1 L of water and adjusting the pH to 6.0 using a 1 *M* sodium hydroxide solution. Use of a phosphate buffer will change the ionic strength of the solution and may promote biological activity. Other buffer relations may be used provided that they do not interfere with the adsorption process.

14.4 A stock solution containing the constituent(s) to be adsorbed is prepared by injecting the pure component(s) into reagent grade water contained in a 250-mL bottle. For poorly soluble compounds, heating the tightly closed container to a maximum of 40 °C or the use of a co-solvent such as HPLC grade methanol may be required. The tightly capped, essentially zero headspace bottle is tumbled using the equilibrator for a sufficient time (usually 1 to 3 days) to ensure the solute is completely dissolved. The contents of this stock solution bottle are analyzed to ensure desired concentrations of the solute(s) were achieved.

14.5 The buffered reagent water in the delivery container is spiked with the stock solution prepared in 14.4 and the floating cover is put in place on top of the solution to prevent volatilization.

14.6 The solution is made ready to fill the isotherm bottles by thoroughly mixing the contents of the delivery container by means of a PTFE stirring bar and magnetic stirrer. Mixing is continued until analysis of water taken through the bottle filling ball valve shows a constant constituent concentration.

15. Isotherm Bottle Filling and Equilibration

15.1 The isotherm bottles are filled by means of a PTFE tube attached to the ball valve on the delivery container. The bottles are filled as quickly as possible but in a way that causes the least amount of agitation of the solution. The PTFE tube is not allowed to come into contact with the solution in the isotherm bottles because carbon can cling to the PTFE tube and change the dosage. To prevent loss of carbon from the isotherm bottle when they are capped, approximately 1 mL of headspace is left in the bottle. This small amount of headspace aids mixing and does not result in a significant solute loss even for very volatile compounds.

15.2 During bottle filling, an empty initial concentration bottle containing no activated carbon is filled at the beginning, in the middle, and at the end of the bottle filling process.

15.3 Two blank bottles are filled with buffered reagent grade water containing no constituents at the beginning and at the end of the filling process.

15.4 The isotherm bottles are weighed before and after carbon addition and after filling with the solution being treated. Based on these weights, the exact weight of water added to each bottle is determined.

15.5 The isotherm bottles are placed in an equilibrator located in a constant temperature room (typically 20 ± 1 °C) and allowed to rotate at 25 rpm for five days. This length of time under most circumstances ensures that full equilibrium is achieved. If an equilibrator is not available, a magnetic stirring bar can be placed in each bottle prior to filling, and mixing can be achieved through the use of a magnetic stirrer.

15.6 After equilibration, the bottles are removed and centrifuged for 15 min at approximately 2000 rpm to settle the

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[3] Speth, T. F., "Predicting Equilibria from Single Solute and Multicomponent Aqueous Phase Adsorption onto Activated Carbon,'' Master's Thesis, Michigan Technical University, Houghton, MI, 1986.

TABLE 1 Format for Reporting Data

NOTE 1— Weight fraction moisture in equilibrated carbon = 0.040.

ID No.	Mass of Carbon,[A] M_w, g	Volume Solution Treated V, L	Residual Concentration C_f, mg/L	Constituent Adsorbed X, C_o–C_f[B]	X/M, mg/g
23-1	0.0102	0.247	4.0024	4.118	101.1
23-2	0.0219	0.247	1.9953	6.125	70.02
23-3	0.0562	0.247	0.5022	7.618	33.94
23-4	0.1005	0.249	0.1531	7.967	20.01
23-5	0.2003	0.247	0.0382	8.082	10.10
23-6	0.5007	0.249	0.0031	8.117	4.09
23-7	0.7508	0.247	0.0015	8.118	2.71
23-8	1.0002	0.247	0.0006	8.119	2.03

[A] The volume of liquid treated is calculated by dividing the mass of solution in kg by the density of water (at experiment temperature) in kg/L.

[B] C_o = 8.1196 g.

carbon. Using this procedure, no filtering of the carbon-treated solutions prior to analysis is required. A filter-equipped syringe may be used instead of a centrifuge to remove carbon particles after the bottles have been allowed to settle. The test solution should be poured into the syringe and filtration performed under pressure.

15.7 The caps are removed from the bottles and two 40-mL zero headspace samples are taken for analysis. It is important that care is exercised during sample taking to prevent loss of volatile organic compounds.

16. Calculations

16.1 For each isotherm bottle determine the amount of constituent adsorbed, X, as follows:

$$X = C_oV - C_fV \qquad (2)$$

where:

X = mass of constituent adsorbed, mg,
C_o = initial constituent concentration, mg/L,
C_f = constituent concentration after carbon treatment, mg/L, and
V = volume of test solution treated, L, and is calculated by dividing the weight of solution in kg by the density of water in kg/L at the temperature of the experiment.

16.2 Determine the mass of constituent adsorbed per unit weight of carbon, X/M, as follows:

$$X/M = [C_oV - C_fV]/M \qquad (3)$$

where:

X, C_o, C_f, and V are defined in 16.1,
M = mass dry carbon, g,

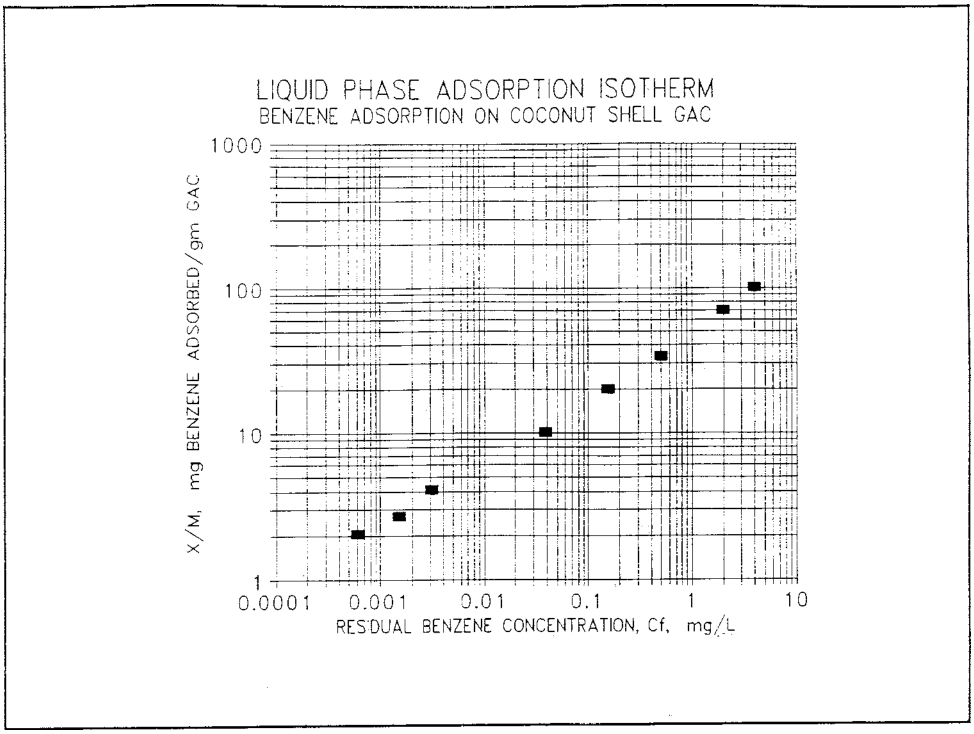

FIG. 1 Isotherm Plot

further where $M = (M_w\ (100- \%\ \text{Moisture})/100)$,
M_w = equilibrated carbon mass, g, and
X/M = constituent adsorbed per unit mass of carbon, mg/g.

17. Report

17.1 See Table 1 for recommended format for reporting data.

17.2 *Plotting of Data and Determination of Freundlich Parameters:*

17.2.1 Use three cycle log/log paper and plot concentration remaining, C_f, in mg/L on the abscissa and *X/M* on the ordinate, and draw the best fit straight line through the points (see Fig. 1).

17.2.2 Select the point on the abscissa axis where $C_f = 1$ and erect a vertical line that intersects the isotherm line and determine the *X/M* value on the ordinate scale that corresponds to this value. This value of *X/M* is defined as the *K* constant of the Freundlich isotherm equation. The slope of the line generated in 17.2.1 is defined as the 1/*n* constant of the Freundlich isotherm equation (see Table 2). Using a log/log plot, the slope is determined by dividing the difference of log values of two points on the abscissa axis by the difference in the log values of two points on the ordinate axis.

TABLE 2 Freundlich Constants Calculated From a Plot of the Data from Figure 1

$K = 49.2\ (\text{mg/g})/(\text{g/m}^3)^{1/n}$
$1/n = 0.44$
R squared = 0.9964

17.3 *Using Freundlich Isotherm Equation:*

17.3.1 Calculate the value for *X/M*, milligram constituent adsorbed per gram of carbon, for any desired constituent concentration, *C*, by using the following Freundlich equation:

$$X/M = KC^{1/n} \tag{4}$$

where:
K and 1/*n* are the Freundlich constants determined in 17.2.2.

18. Keywords

18.1 activated carbon; adsorption

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