

Standard Test Method for Sorbent Performance of Adsorbents¹

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This standard has been approved for use by agencies of the Department of Defense.

1. Scope

1.1 This test method covers laboratory tests that describe the performance of adsorbents in removing nonemulsified oils and other floating, immiscible liquids from the surface of water.

1.2 The values stated in SI units are to be regarded as the 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. Specific precautionary statements are given in 8.3.1.

2. Referenced Documents

2.1 ASTM Standards:²

D2859 Test Method for Ignition Characteristics of Finished Textile Floor Covering Materials

F716 Test Methods for Sorbent Performance of Absorbents 2.2 *Federal Standard:*

Fed. Std. No. 141a Paint, Varnish, Lacquer and Related Materials, Methods of Inspection, Sampling and Testing³

2.3 Military Specification:

MIL-I-631D Insulation, Electric, Synthetic Resin Composition, Nonrigid³

3. Terminology

3.1 General Terminology:

3.1.1 *gellant*—a material such as a colloidal network or other aggregate network that pervades and holds a liquid in a highly viscous fragile structure. Many gels may rapidly liquify

with added heat or ionic/polar addition. These materials are soluble/flowable in excess liquid.

3.1.2 *sorbent*—an insoluble material or mixture of materials used to recover liquids through the mechanisms of absorption or adsorption, or both.

3.1.3 *thickener*—a material (usually of higher molecular weight) that is soluble in excess liquid. These materials go from dry to gummy (viscoelastic) to flowable and then soluble. The final viscosity depends only on the liquid to solid ratio.

3.1.4 *universal sorbent*—an insoluble material or mixture of materials that will sorb both hydrophobic and hydrophilic liquid spills.

3.2 Definitions:

3.2.1 *absorbent*—a material that picks up and retains a liquid distributed throughout its molecular structure causing the solid to swell (50 % or more). The absorbent is at least 70 % insoluble in excess fluid.

3.2.2 *adsorbent*—an insoluble material that is coated by a liquid on its surface including pores and capillaries without the solid swelling more than 50 % in excess liquid.

3.2.3 *adsorbent cubage factor* "C"—this is the ratio of sorbent volume used to the liquid volume sorbed.

3.2.4 *cubage*—defines cubic content, volume, or displacement.

3.3 Definitions of Terms Specific to This Standard:

3.3.1 This test method does not apply to belt, rope, or weir type skimming devices.

3.3.2 *oil*—a substantially water immiscible organic liquid that will float on water (density less than 1 g/cm³), typically with surface tension less than 40×10^{-3} N/m.

3.3.3 *Type I adsorbent (roll, film, sheet, pad, blanket, web)*—a material with length and width much greater than thickness and which has both linear form and strength sufficient to be handled either saturated or unsaturated.

3.3.4 *Type II adsorbent (loose)*—an unconsolidated, particulate material without sufficient form and strength to be handled except with scoops and similar equipment.

3.3.5 Type III adsorbent (enclosed):

3.3.5.1 *IIIa, pillows*—adsorbent material contained by an outer fabric or netting that has permeability to oil, but with

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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.

³ Available from Standardization Documents Order Desk, Bldg. 4 Section D, 700 Robbins Ave., Philadelphia, PA 19111-5094, Attn: NPODS.

openings sufficiently small so as to substantially retain the sorbent material within the fabric or netting.

3.3.5.2 *IIIb, adsorbent booms*—adsorbent material contained by an outer fabric or netting that is permeable to oil but with openings sufficiently small so as to substantially retain the sorbent material within the fabric or netting. The lengthwise dimension substantially exceeds other dimensions and with strength members running parallel with length. Booms are also provided with connections for coupling adsorbent booms together.

3.3.6 *Type IV-agglomeration unit*—an assemblage of strands, open netting, or other physical forms giving an open structure that minimally impedes the intrusion into itself of high viscosity oils. Normally for use with viscous oils, typically above 10 000 cP viscosity. Said oils are then held in this structure permitting the composite oil/structure to be handled (pompoms).

3.3.7 *reuse*—the art of extracting adsorbed liquids from an adsorbent through rolls or other compression techniques permitting the adsorbent to be used once again; limitations on reuse may include the U.S. Clean Water Act or other legal restrictions.

4. Summary of Test Method

4.1 The adsorbent material is tested using established standard tests for factors relating to storage, while specially developed tests are used for covering other performance factors. Oil and water adsorption strength, buoyancy, and reusability tests are included among these latter tests.

5. Significance and Use

5.1 This test method is to be used as a basis for comparison of adsorbents in a consistent manner.

5.2 These tests are not appropriate for absorbent materials that are covered in Methods F716.

Note 1-Ensure that material compatibilities exist between the sorbent and the hazardous substance which may be sorbed.

6. Apparatus

6.1 *Exterior Exposure Tester*, sufficient to be used under Federal Test Standard 141a, Method 6152, or actual exposure as detailed in 8.2.

6.2 *Test Cells*—The dimensions of the test cells shall be large enough to enable the adsorbent sample to float freely within the test cell. For Type I and Type II sorbents, the recommended test cell is a borosilicate 19 cm (diameter) by 10 cm (depth) crystallizing dish with a watch glass or glass plate cover. For larger samples, a 53 by 56 cm plastic sink (laundry tub or equivalent) to accommodate the sample is recommended.

6.3 *Mesh Baskets*—The basket shall be of a sufficient size and strength to accommodate the sample size and weight (150 cm^3 , 4 to 10 g minimum) when saturated. The basket must not be so tall as to interfere with a protective lid for the test cell.

Note 2—The mesh should retain the sorbent, yet allow free oil to drain away from the sorbent.

6.4 *Shaker Table*, capable of a frequency of 150 cycles/min and an amplitude of 2.5 cm.

6.5 *Top Loading Balance*—for Type I and II adsorbent, fitted with a hook or other handing mechanism, 400+ g maximum capacity with 0.1 g resolution, or equivalent.

6.6 *Continuous Reading Hanging Scale*, for Type III and IV adsorbent, fitted with a hook or other handing mechanism, 50+kg maximum capacity with 100 g resolution, or equivalent.

7. Conditioning

7.1 Condition all adsorbent test specimens at 23 ± 4 °C and 70 ± 20 % relative humidity for not less than 24 h prior to testing. Condition specimens in a fully exposed state with no coverings or wrapping that would hinder the ambient equilibration process.

7.2 If temperature conditions other than normal room temperature are expected to be important, then conditioning and testing should be carried out at temperatures of interest in addition to those specified in 7.1.

8. Tests for Storage Properties

8.1 *Storage Density*—The density of the sorbent sample is calculated by determining the weight of a known volume (standard sorbent package as delivered to the consumer) of the sorbent. If standard storage packages are not available, then the mass of at least 1 L of sorbent is determined to calculate the storage density.

8.2 *Mildew*—The susceptibility of an adsorbent to mildew under normal storage is defined under MIL-1-631D, Section 3.5.7. The objective of this test is to determine expected shelf life under conditions which could lead to mildew.

8.3 *Flammability*—The procedure for this test is described in Test Method D2859, the Methenamine Pill Test. This test relates to ignition from a spark, cigarette, or other point source such as might be encountered in normal shipping and storage. It should not be inferred that an adsorbent that passes this test will fail to burn if ignited in another manner such as full building involvement, bonfire, and so forth, and it should likewise be understood that the test is limited to and pertains to only unsaturated adsorbent samples as normally supplied by the manufacturer.

8.3.1 This test method should be used to measure and describe the properties of materials, products, or assemblies in response to heat and flame under controlled laboratory conditions and should not be used to describe or appraise the fire hazard or fire risk of materials, products, assemblies under actual fire conditions. However, results of this test may be used as elements of a fire risk assessment which takes into account all of the factors which are pertinent to an assessment of the fire hazard of a particular end use.

8.3.2 Test Type I adsorbents as a single sheet or pad as supplied by the vendor.

8.3.3 Uniformly arrange Type II adsorbents in a layer of sufficient size to fit the test apparatus. The thickness should be 1.5 mm uncompressed and arranged such that the bulk density is equal to that found in the packaged product as supplied by the manufacturer.

8.3.4 Any type adsorbent can be tested in the package in which it is supplied by placing the standard Methenamine test mask on a flat portion of the package surface, and placing the pill in the center of the mask on the package and igniting it. Results would be interpreted as in the normal test.

9. Tests for Performance Properties

9.1 These tests involve the use of oils with a range of viscosities and densities as indicated below.

Oil Type	Viscosity Range	Density Range	Example
Light	1 to 10 cP	0.820 to 0.870 g/cm ³	Diesel fuel, mineral oil
Medium	200 to 400 cP	0.860 to 0.970 g/cm ³	Crude oil, canola oil, mineral oil
Heavy	1500 to 2500 cP	0.930 to 1.000 g/cm ³	Bunker C or residual fuel, mineral oil
Weathered	8000 to 10 000 cP	0.930 to 1.000 g/cm ³	'

9.2 Dynamic Degradation Test—This procedure is designed to test for water take-up and to determine oleophilic properties of an adsorbent sample under dynamic conditions. This test is performed at $23 \pm 4^{\circ}$ C.

9.2.1 Type I Adsorbent-Sample pieces of the adsorbent (four pieces cut with a sharp edge (to minimize compaction) into squares of approximately 6 by 6 cm) are first weighed then placed in a 4 L jar that is half-filled with water and sealed. The container is then placed on its side and mounted on a shaker table, or similar device, set at a frequency of 150 cycles per minute and an amplitude of 2.5 cm for a duration of 15 min. The contents of the jar are allowed to settle for a period of 2 min. Observations pertaining to the condition of the adsorbent and the condition of the water are recorded. Any adsorbent pieces that do not remain floating at the surface of the water are considered to have failed this test. The contents of the jar are strained through a mesh basket to catch the adsorbent samples, which are then weighed after a 30 s drain period. The water pick-up ratio is calculated from the weight measurements (see 9.5).

9.2.1.1 The jar is half-filled with fresh water and 3 mL of oil (medium crude, 300 cP oil, or equivalent) is added. The adsorbent sample is returned to the jar, which is then sealed. The jar is placed on its side and mounted on a shaker table, or similar device, set at a frequency of 150 cycles per minute and an amplitude of 2.5 cm for a duration of 15 min. The contents of the jar are allowed to settle for a period of 2 min, at which time observations are noted. Observations include but are not limited to: quantity of adsorbent submerged, physical appearance of adsorbent and water, and the persistence and color of residual test liquid sheen.

9.2.2 Type II Adsorbent—An adsorbent sample (approximately 4 to 10 g or a maximum of 150 cm^3) is first weighed then placed in a 4L jar that is half-filled with water and sealed. The container is then placed on its side and mounted on a shaker table, or similar device, set at a frequency of 150 cycles per minute and an amplitude of 2.5 cm for a duration of 15 min. The contents of the jar are allowed to settle for a period of 2 min. Observations pertaining to the condition of the adsorbent and the condition of the water are recorded. If 10 % or more of the adsorbent material has sunk, then the adsorbent is considered to have failed this test. The contents of the jar are strained

through a mesh basket to catch the adsorbent samples, which are then weighed after a 30 s drain period. The water pick-up ratio is calculated from the weight measurements (see 9.5).

9.2.2.1 The jar is half-filled with fresh water and 3 mL of oil (medium crude, 300 cP oil, or equivalent) is added. The adsorbent sample is returned to the jar, which is then sealed. The jar is placed on its side and mounted on a shaker table, or similar device, set at a frequency of 150 cycles per minute and an amplitude of 2.5 cm for a duration of 15 min. The contents of the jar are allowed to settle for a period of 2 min, at which time observations are noted. Observations include but are not limited to: quantity of adsorbent submerged, physical appearance of adsorbent and water, and the persistence and color of residual test liquid sheen.

9.2.3 *Types IIIa, IIIb, IV Adsorbents*—Both the outer fabric or netting and the filler material are tested independently for Type III adsorbents. Samples are prepared according to the protocol listed in 9.2.1 for the outer fabric or netting of Type III adsorbents and Type IV adsorbents, and the protocol listed in 9.2.2 is used for any particulate filler material used in Type III adsorbents. If the adsorbent material fails to remain floating as described in 9.2.1 or 9.2.2, then the adsorbent is deemed to have failed the dynamic degradation test.

9.3 Oil Adsorption-Short Test—This test gives idealized laboratory data that can be used to compare one adsorbent's oil capacity with another and likewise give relative cost effectiveness. It should be recognized that under normal use conditions, an adsorbent will not be exposed to sufficient oil layer thickness to become completely or rapidly saturated. This test will, therefore, give maximum possible capacity data and idealized time to saturation. The objective of this test is to determine optimum adsorbent without the competing presence of water. As such, this data relates only to oil layer thicknesses that approximate or exceed that of the adsorbent. All adsorption test procedures to be run with adsorbent samples conditioned as in Section 7 and using specified oils at $23 \pm 4^{\circ}$ C.

9.3.1 *Type I Adsorbent*—The test liquid layer should be of a minimum thickness of 2.5 cm if the thickness of the adsorbent is under 2.5 cm. If the adsorbent is thicker than 2.5 cm, then a liquid layer at least as thick as the adsorbent sample should be used.

9.3.1.1 The adsorbent sample to be tested shall be a minimum weight of 4 g. Cut the sample with a sharp edge (to minimize compaction) to minimum dimensions of 13 by 13 cm^2 . The adsorbent is then weighed and the value is recorded. The test cell is filled with an initial layer of test liquid. The adsorbent is lowered into the cell. The adsorbent shall be allowed to float freely within the test cell. After 15 min \pm 20 s, remove the adsorbent in a vertical orientation along an edge with a clip and let drain for 30 ± 3 s (use a 2 min ± 3 s drain time for heavy and weathered oil). Place a tared weighing pan under the adsorbent to catch any additional drips and immediately transfer the adsorbent to the pan. Determine and record the sample weight. All tests are triplicated with the mean of the three runs being used for calculations. The oil pick-up ratio on a weight basis is calculated, as is the volume of oil per adsorbent sample (see 9.5). If the value of any run (g/g)