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Document Name: AATCC 118: Oil Repellency: Hydrocarbon Resistance Test

CFR Section(s): 10 CFR 430 Subpart B

Standards Body: American Association of Textile Chemists and Colorists



Official Incorporator:

THE EXECUTIVE DIRECTOR
OFFICE OF THE FEDERAL REGISTER
WASHINGTON, D.C.

Oil Repellency: Hydrocarbon Resistance Test

Developed in 1966 by AATCC Committee RA56; reaffirmed 1972, 1975, 1978, 1983, 1989; editorially revised 1985, 1986, 1990, 1995; revised 1992; editorially revised and reaffirmed 1997. Technically equivalent to ISO 14419.

1. Purpose and Scope

1.1 This test method detects the presence of a fluorochemical finish, or other compounds capable of imparting a low energy surface, on all types of fabrics, by evaluating the fabric's resistance to wetting by a selected series of liquid hydrocarbons of different surface tensions.

2. Principle

2.1 Drops of standard test liquids, consisting of a selected series of hydrocarbons with varying surface tensions, are placed on the fabric surface and observed for wetting, wicking, and contact angle. The oil repellency grade is the highest numbered test liquid which does not wet the fabric surface.

3. Terminology

3.1 **grade**, n.—*in textile testing*, the symbol for any step of a multistep standard reference scale for a quality characteristic.

NOTE: The grade is assigned to test specimens exhibiting a degree of the quality comparable to that step of the standard.

3.2 **oil repellency**, n.—*in textiles*, the characteristic of a fiber, yarn or fabric whereby it resists wetting by oily liquids.

4. Safety Precautions

NOTE: These safety precautions are for information purposes only. The precautions are ancillary to the testing procedures and are not intended to be all inclusive. It is the user's responsibility to use safe and proper techniques in handling materials in this test method. Manufacturers MUST be consulted for specific details such as material safety data sheets and other manufacturer's recommendations. All OSHA standards and rules must also be consulted and followed.

4.1 Good laboratory practices should be followed. Wear safety glasses and impervious gloves when handling test liquids in all laboratory areas.

4.2 The hydrocarbons specified in this method are flammable. Keep away from

heat, sparks and open flame. Use with adequate ventilation. Avoid prolonged breathing of vapor or contact with skin. Do not take internally.

4.3 Exposure to chemicals used in this procedure must be controlled at or below levels set by governmental authorities [e.g., Occupational Safety and Health Administration's (OSHA) permissible exposure limits (PEL) as found in 29 CFR 1910.1000 of January 1, 1989]. In addition, the American Conference of Governmental Industrial Hygienists (ACGIH) Threshold Limit Values (TLVs) comprised of time weighted averages (TLV-TWA), short term exposure limits (TLV-STEL) and ceiling limits (TLV-C) are recommended as a general guide for air contaminant exposure which should be met (see 12.1).

5. Uses and Limitations

5.1 This test method is not intended to give an absolute measure of the resistance of the fabric to staining by all oily materials. Other factors, such as composition and viscosity of the oily substances, fabric construction, fiber type, dyes, other finishing agents, etc., also influence stain resistance. This test can, however, provide a rough index of oil stain resistance, in that generally the higher the oil repellency grade, the better resistance to staining by oily materials, especially liquid oil substances. This is particularly true when comparing various finishes for a given fabric.

6. Apparatus and Materials

6.1 Test liquids prepared and numbered according to Table I (see 12.2).

6.2 Dropping bottles (see 12.3).

6.3 White AATCC Textile Blotting Paper (see 12.4).

6.4 Laboratory gloves (general purpose is sufficient).

7. Test Specimens

7.1 Test two specimens approximately 20 × 20 cm (8 × 8 in.) from each sample. Condition the test specimens for a minimum of 4 hr at 21 ± 1°C (70 ± 2°F) and 65 ± 2% RH prior to testing.

8. Procedure

8.1 Place the test specimen flat on the white textile blotting paper on a smooth, horizontal surface.

8.1.1 When evaluating open weave of "thin" fabrics, conduct the test on at least two layers of the fabric; otherwise, the test liquid may wet the underlying surface, not the actual test fabric, and thereby cause confusion in the reading of the results.

8.1.2 Equipment, benches and gloves must be free of silicone. Use of silicone containing products could adversely affect the oil repellency grade.

8.2 Wearing clean laboratory gloves, brush the pile of napped or pile fabrics with your hand in the direction giving the greatest lay of the surface prior to placing the drops of the test liquid.

8.3 Beginning with the lowest-numbered test liquid (AATCC Oil Test Grade Liquid No. 1), *carefully* place small drops [approximately 5 mm (0.187 in.) in diameter or 0.05 mL volume] on the test specimen in five locations along the filling direction. The drops should be approximately 4.0 cm (1.5 in.) apart. The dropper tip should be held at a height of approximately 0.6 cm (0.25 in.) from the fabric surface while placing drops. DO NOT TOUCH THE FABRIC WITH THE DROPPER TIP. Observe the drops for 30 ± 2 sec, from approximately a 45° angle.

8.4 If no penetration or wetting of the fabric at the liquid-fabric interface and no wicking around the drops occur, place drops of the next higher-numbered test liquid at an adjacent site on the fabric and again observe for 30 ± 2 sec.

8.5 Continue this procedure until one of the test liquids shows obvious wetting or wicking of the fabric under or around the drop within 30 ± 2 sec.

9. Evaluation

9.1 The AATCC Oil Repellency Grade of a fabric is the numerical value of the highest-numbered test liquid which will

Table I—Standard Test Liquids

AATCC Oil Repellency Grade Number	Composition
0	None (Fails Kaydol)
1	Kaydol
2	65:35 Kaydol: n-hexadecane by volume
3	n-hexadecane
4	n-tetradecane
5	n-dodecane
6	n-decane
7	n-octane
8	n-heptane

not wet the fabric within a period of 30 ± 2 sec. A grade of zero (0) is assigned when the fabric fails the Kaydol test liquid. Wetting of the fabric is normally evidenced by a darkening of the fabric at the liquid-fabric interface or wicking and/or loss of contact angle of the drop. On black or dark fabrics, wetting can be detected by loss of "sparkle" within the drop.

9.2 Different types of wetting may be encountered depending on the finish, fiber, construction, etc.; and the determination of the end point can be difficult on certain fabrics. Many fabrics will show complete resistance to wetting by a given test liquid (as indicated by a clear drop with a high contact angle, see Fig. 1, Example A) followed by immediate penetration by the next higher-numbered test liquid. In these instances the end point, and oil repellency grade, is obvious. However, some fabrics will show progressive wetting under several test liquids as evidenced by a partial darkening of the fabric at the liquid-fabric interface (see Fig. 1, Examples B, C and D). For such fabrics, the point of failure is considered to be that test liquid which exhibits complete darkening of the interface or any wicking within 30 ± 2 sec.

9.3 A **failure** occurs when three (or more) of the five drops applied from a given test liquid show complete wetting (Fig. 1 [D]) or wicking with loss of contact angle (Fig. 1 [C]). A **pass** occurs if three (or more) of the five drops applied show clear well rounded appearance with high contact angle (Fig. 1 [A]). The grade is expressed as the integer value of the pass test liquid immediately prior to the

fail test liquid. A **borderline pass** occurs if three (or more) of the five drops applied show the rounded drop with partial darkening of the test specimen (Fig. 1 [B]). The grade is expressed to the nearest 0.5 value determined by subtracting one-half from the number of the borderline pass test liquid.

10. Report

10.1 The oil repellency grade should be measured on two separate specimens. If the two grades agree, report the value. When the two grades are not in agreement, a third determination should be made. Report the grade of the third determination if that value is the same as either of the first two determinations. When the third determination is different from either of the first two, report the median value. For example, if the first two grades are 3.0 and 4.0 and the third determination is a 4.5 value, report the median value of 4.0. Report the oil repellency grade to the nearest 0.5 value (see Fig. 1).

11. Precision and Bias

11.1 *Summary.* Interlaboratory tests were conducted in September 1990 and April 1991 to establish the precision of this test method. The September interlab involved two participants at each of nine laboratories rating two specimens of each of four fabrics each day for three days. The grades of this interlab were concentrated into the 1-2 and 4-5 regions of the scale. The April interlab was conducted with fabrics responding in the 2-3 and 5-7

portions of the scale. This interlab involved two participants at each of seven laboratories rating two specimens of each of two fabrics each day for two days. (Day interaction was shown not to be a significant factor in the analysis of the September interlab.) Results from both interlabs were combined for precision and bias statements. All materials necessary for the interlabs were provided to each laboratory by AATCC including the standard test liquids. A video cassette of the grading procedure prepared at the AATCC Technical Center by the subcommittee and visual examples of pass, borderline and fail conditions were included in the protocol. The fabrics were limited to polyester/cotton materials. The unit of measure was the median of the grades of the two (or three) specimens rated each day.

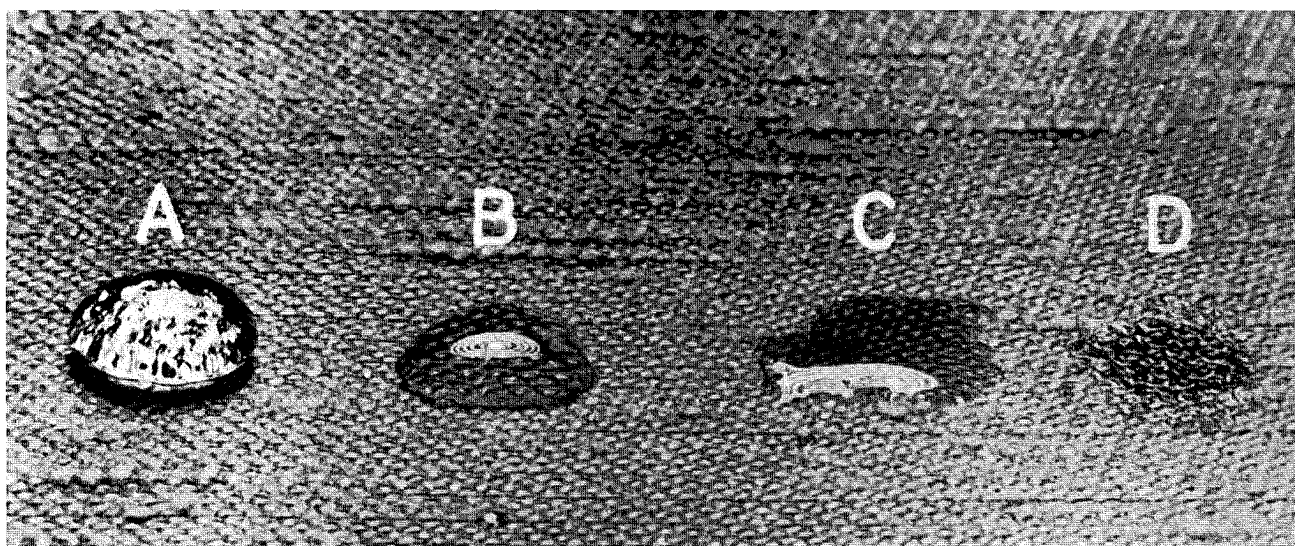
11.2 The components of variance as standard deviations of the oil repellency grade were calculated to be as follows:

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Single operator	0.27
Between operators/ within laboratories	0.30
Between laboratories	0.39

11.3 *Critical differences.* For the components of variance in 11.2, two observations should be considered significantly different at the 95% probability level if the difference equals or exceeds the critical differences shown in Table II.

11.4 *Bias.* The true value of the oil repellency grade can only be defined in terms of this test method. Within this limitation, this test method has no known bias.



- A = Passes; clear well-rounded drop
- B = Borderline pass; rounding drop with partial darkening
- C = Fails; wicking apparent and/or complete wetting
- D = Fails; complete wetting

Fig. 1—Grading example.

Table II—Critical Differences^a

No. of Observations ^b	Single Operator	Within Laboratory	Between Laboratory
1	0.75	1.12	1.55
2	0.53	0.99	1.45
3	0.43	0.94	1.42

^a The critical differences were calculated using *t* - 1.950, which is based on infinite degrees of freedom.

^b An observation is a unit of measure obtained from the median of the grades for 2 (or 3) specimens.

12. Notes

12.1 Available from Publications Office, ACGIH, Kemper Woods Center, 1330 Kemper Meadow Dr., Cincinnati OH 45240; tel: 513/742-2020.

12.2 Kaydol™ is available from CBM Group of N.C. Inc., 1308 N. Ellis Ave., Dunn

NC 28334; tel: 910/892-8985; fax: 910/892-5701; or Textile Innovators Corp., P.O. Box 8, Windsor NC 27983; tel: 252/794-9703; fax: 252/794-9704. All other hydrocarbon liquids should be laboratory quality available through most chemical supply houses. One source is Fisher Scientific Inc. That concern's catalog designations are as follows:

Test Liquid	Catalog Number	Specified Melting Point or Boiling Point Range	N*
n-hexadecane	O3035	17 to 18C	27.3
n-tetradecane	O4595	4 to 6C	26.4
n-dodecane	O2666	-10.5 to -9.0C	24.7
n-decane	O2128	173 to 175C	23.5
n-octane	O3980	124 to 126C	21.4
n-heptane	O3008	98 to 99C	14.8
Kaydol		348C	31.5

*N = dynes/cm at 25C

12.3 For convenience, it is desirable to transfer the test liquids from stock solutions to dropping bottles, each marked with the appropriate AATCC Oil Repellency Grade number. A typical system found useful consists of 60 mL dropping bottles with ground-in pipettes and Neoprene bulbs. Prior to use the bulbs should be soaked in heptane for several hours and then rinsed in fresh heptane to remove soluble substances. It has been found helpful to place the test liquids in sequential order in a wooden platform on the grading table. NOTE: Purity of test liquids does affect surface tension of the liquid. Use only analytical grades of test liquids.

12.4 AATCC White Textile Blotting Paper is available from AATCC, P.O. Box 12215, Research Triangle Park NC 27709; tel: 919/549-8141; fax: 919/549-8933; e-mail: orders@aatcc.org.