
**Textiles — Oil repellency — Hydrocarbon
resistance test**

Textiles — Oléofugation — Essai de résistance aux hydrocarbures



Foreword

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Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

International Standard ISO 14419 was prepared by ISO Technical Committee ISO/TC 38, *Textiles*, Subcommittee SC 2, *Cleansing, finishing and water resistance tests*.

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Textiles — Oil repellency — Hydrocarbon resistance test

1 Scope

This International Standard is used in the evaluation of a substrate's resistance to absorption of a selected series of liquid hydrocarbons of different surface tensions.

This International Standard is intended to provide a guide to oil stain resistance. It can provide a rough index of oil stain resistance as 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 substrate. This International Standard can also be utilized in determining if washing and/or dry cleaning treatments have any adverse effect on the oil repellency characteristics of a substrate. It is recommended that the washing and/or dry cleaning treatment procedures described in ISO 6330 or ISO 3175 be used for this purpose.

This International Standard is not intended to give an absolute measure of the resistance of the substrate to staining by all oily materials. Other factors, such as composition and viscosity of the oily substances, substrate construction, fibre type, dyes and other finishing agents, also influence stain resistance. This International Standard is not intended to estimate the resistance to penetration of the substrate by oil based chemicals; for this evaluation see ISO 6530, *Protective clothing – Protection against liquid chemicals – Determination of resistance of materials to penetration by liquids*.

2 Normative reference

The following normative document contains provisions which, through reference in this text, constitute provisions of this International Standard. For dated references, subsequent amendments to, or revisions of any of these publications do not apply. However, parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent edition of the normative document indicated below. For undated references, the latest edition of the normative document referred to applies. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 139:1973, *Textiles – Standard atmospheres for conditioning and testing*.

3 Definitions

For the purposes of this International Standard, the following definitions apply.

3.1 grade

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 reference scale.

3.2 oil repellency

characteristic of a fabric whereby it resists absorption of oily liquids

4 Principle

Drops of standard test liquids, consisting of a selected series of hydrocarbons with different surface tensions, are placed on the substrate surface and then observed for absorption, wicking and contact angle. The oil repellency grade is the highest numbered test liquid which is not absorbed by the substrate surface.

5 Safety precautions

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

5.2 Some of the hydrocarbons specified in this International Standard are flammable. Keep away from heat, sparks and open flame. Use with adequate ventilation. Avoid prolonged breathing of vapour or contact with skin. Do not take internally.

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 International Standard. Manufacturers should be consulted for specific detail such as material safety data sheets and other manufacturer's recommendations.

6 Reagents

All reagents must be of a suitable analytical grade. Ensure the standard test liquids are used and stored at a temperature of $(20 \pm 2) ^\circ\text{C}$.

6.1 Test liquids, prepared and numbered according to Table 1.

6.2 White mineral oil, USP white mineral oil 340-355 SSU @ $37,8 ^\circ\text{C}$ with Saybolt color + 30.

Table 1 — Standard test liquids

Composition	Oil test liquid number	Density kg/l	Surface tension N/m at $25 ^\circ\text{C}$
None (fails white mineral oil)	0	—	—
White mineral oil	1	0,84 – 0,87	0,0315
65:35 white mineral oil: <i>n</i> -hexadecane by volume	2	0,82	0,0296
<i>n</i> -hexadecane	3	0,77	0,0273
<i>n</i> -tetradecane	4	0,76	0,0264
<i>n</i> -dodecane	5	0,75	0,0247
<i>n</i> -decane	6	0,73	0,0235
<i>n</i> -octane	7	0,70	0,0214
<i>n</i> -heptane	8	0,69	0,0148

7 Apparatus

7.1 Dropping bottles, for convenience it is desirable to transfer the test liquids from stock solutions to dropping bottles, each marked with the appropriate oil test liquid 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.

7.2 White textile blotting paper, textile blotting paper is an absorbent textile blotting paper of 0,71 mm caliper and 141,32 g/m² blotter sheet weight. White Textile Blotting Paper is available from AATCC.¹⁾

7.3 Laboratory gloves, general purpose is sufficient.

8 Test specimens

Three test specimens approximately 20 cm × 20 cm are needed. Test specimen size should be chosen to represent all physical and colour characteristics of the fabric and allow ample room for testing. Condition the test specimens for a minimum of 4 h at (20 ± 2) °C and (65 ± 2) % RH prior to testing. See ISO 139.

9 Procedure

9.1 Place the first test specimen flat on the white textile blotting paper (7.2) on a smooth, horizontal surface with the face-side on top. Testing should be conducted in standard atmosphere for testing in accordance with ISO 139. Testing should be completed within 30 min if test specimens are removed from a conditioning chamber.

9.1.1 When evaluating open weave or "thin" substrates, place the test specimen on at least two layers of the substrate; otherwise, the test liquid may wet the underlying surface, not the actual test substrate and thereby cause confusion in the reading of the results.

9.1.2 Equipment, benches and gloves should be free of silicone. Use of silicone-containing products could adversely affect oil repellency grades.

9.2 Wearing clean laboratory gloves (7.3), brush the pile of napped or pile substrates using the hand, in the direction giving the greatest lay of the surface prior to placing the drops of the test liquid. (The direction giving the lowest pile.)

9.3 Beginning with the lowest-numbered test liquid (oil test liquid No. 1), carefully place small drops (approximately 5 mm in diameter or 0,05 ml volume) on the test specimen in a minimum of five locations representing all physical and colour characteristics of the fabric. The drops should be approximately 4,0 cm apart. The dropper tip should be held at a height of approximately 0,6 cm from the substrate surface while placing drops. Do not touch the substrate with the dropper tip. Observe the drops for (30 ± 2) s from approximately a 45° angle. Assess each drop in accordance with Figure 1. Immediately examine the reverse of the fabric for any wetting.

9.4 If no penetration or wetting of the substrate at the liquid-substrate interface and also no wicking around the drops occurs, place drops of the next high-numbered test liquid at an adjacent site so as not to interfere with the previous test on the substrate and again observe for (30 ± 2) s. Assess each drop in accordance with Figure 1. Immediately examine the reverse of the fabric for any wetting.

1) White Textile Blotting Paper is the trade-name of a product supplied by AATCC P.O. Box 12215, Research Triangle Park, NC 27709-2215. This information is given for the convenience of users of this International Standard and does not constitute an endorsement by ISO of the product named. Equivalent products may be used if they can be shown to lead to the same results.

9.5 Continue this procedure until one of the test liquids shows obvious wetting or wicking of the substrate under or around the drop within (30 ± 2) s. A maximum of six tests (oil test liquids) may be performed on one test specimen.

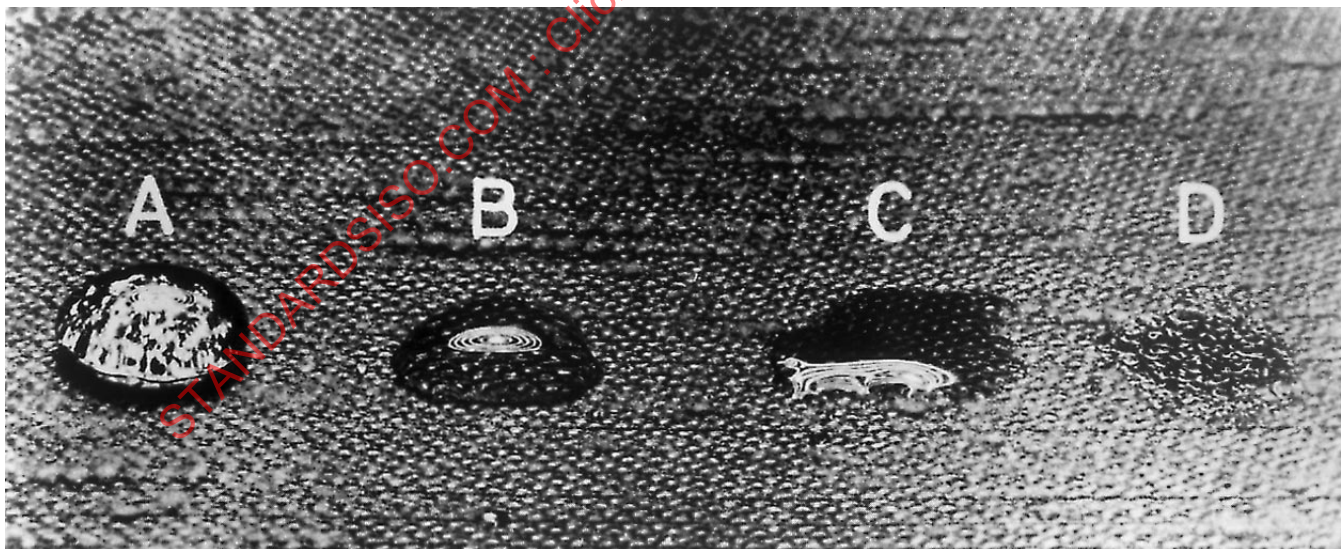
9.6 Repeat the procedure with the second sample. A third sample may be required (see clause 11).

10 Evaluation

10.1 The oil repellency grade of a substrate is the numerical value of the highest-numbered test liquid which will not wet the substrate within a period of (30 ± 2) s. A grade of zero (0) is assigned when the substrate fails the white mineral oil liquid test. Wetting of the substrate is normally evidenced by a darkening (greying/shadowing) of the substrate at the liquid-substrate interface or wicking and/or loss of contact angle of the drop. On black or dark substrates, wetting can be detected by loss of "sparkle" within the drop.

10.2 Different types of wetting may be encountered depending on the finish, fibre, construction, etc.; and the determination of the end point can be difficult on certain substrates. Many substrates will show complete resistance to wetting by a given test liquid [as indicated by a clear drop with a high contact angle (see Figure 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 substrates will show progressive wetting under several test liquids as evidenced by a partial darkening of the substrate at the liquid-substrate interface (see Figure 1, examples B, C and D). For such substrates, the point of failure is considered to be that test liquid which exhibits complete darkening of the interface or any wicking within (30 ± 2) s.

10.3 A **failure** occurs when three (or more) of the five drops applied from a given test liquid show complete wetting (see Figure 1, example D) or wicking with loss of contact angle (see Figure 1, example C). A **pass** occurs if three (or more) of the five drops applied show a clear, well-rounded appearance with high contact angle (see Figure 1, example A). The grade is expressed as the value of the pass oil test liquid immediately prior to the fail oil 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 (see Figure 1, example 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.



Key

- 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

Figure 1 — Example of assigned grades

11 Evaluation of results

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. This grade variation may be an indication of ununiform fabric or contamination problems.

12 Test report

The test report shall include the following information:

- a) reference to this International Standard, i.e. ISO 14419;
- b) all information necessary to complete identification of the sample tested;
- c) the number of specimens tested;
- d) the conditioning and testing atmosphere used;
- e) any deviation from the procedure specified;
- f) test results;
- g) oil repellency grade.

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Annex A (informative)

Precision and bias statement

A.1 Precision

A.1.1 Interlaboratory studies were conducted in September 1990 and April 1991 to establish the precision of this test method. The September interlaboratory study involved two participants at each of nine laboratories rating two specimens of each of four fabrics each day for three days. The grades from this study were concentrated into the 1-2 and 4-5 regions of the scale. The April interlaboratory study was conducted with fabrics responding in the 2-3 and 5-7 regions of the scale. This study 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 study.) Results from both interlaboratory studies were combined for precision and bias statements. All materials necessary for the studies 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.

A.1.2 The components of variance as standard deviations of the oil repellency grade were calculated to be as follows:

Oil repellency test	
Single operator	0,27
Between operators/within laboratories	0,30
Between laboratories	0,39

A.1.3 Critical differences: For the components of variance in A.1.2, two observations should be considered significantly different at 95 % probability level if the difference equals or exceeds the critical differences shown in Table A.1.

Table A.1 — Critical Differences

No. of observations ^a	Single operator	Within laboratory	Between laboratories
1	0,75	1,12	1,55
2	0,53	0,99	1,45
3	0,43	0,94	1,42
NOTE The critical differences were calculated using $t = 1,950$ from the Students' t distribution which is based on infinite degrees of freedom.			
^a An observation is a unit of measure obtained from the median of the grades for 2 (or 3) specimens.			

A.2 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.