INTERNATIONAL STANDARD

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Textiles — Aqueous liquid repellency — Water/alcohol solution resistance test

Textiles — Caractère hydrophobe — Essai avec une solution eau/alcool

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. 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.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 23232 was prepared by Technical Committee ISO/TC 38, *Textiles*, Subcommittee SC 2, *Cleansing, finishing and water resistance tests*.

Textiles — Aqueous liquid repellency — Water/alcohol solution resistance test

1 Scope

This International Standard provides a guide for aqueous stain resistance. It can provide a rough index of aqueous stain resistance as, generally, the higher the aqueous liquid repellency grade, the better the resistance to staining by water/alcohol-based materials, especially water/alcohol-based liquids. This is particularly true when comparing various finishes for a given substrate. This International Standard can also be utilized for determining if washing and/or dry-cleaning treatments have any adverse effects on the aqueous liquid 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 water/alcohol-based substances. Other factors, such as the composition and viscosity of the water/alcohol-based 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 water/alcohol-based chemicals; for this evaluation, see ISO 6530.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 139, *Textiles — Standard atmospheres for conditioning and testing*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

3.1
grade
⟨textile testing⟩ symbol for any step of a multi-step standard reference scale for a quality characteristic

NOTE A grade is assigned to test specimens exhibiting a degree of the quality comparable to that step of the standard reference scale.

3.2
aqueous repellency
⟨textiles⟩ characteristic of a fibre, yarn or fabric whereby it resists absorption by aqueous liquids

4 Principle

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

5 Uses and limitations

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

6 Safety precautions

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

The alcohol specified in this method is flammable. Keep it away from heat, sparks and open flame. Use it with adequate ventilation. Avoid prolonged inhalation of vapour or contact with skin. Do not take internally.

Exposure to chemicals used in this procedure must be controlled at or below levels set by governmental authorities.

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 details, such as material safety sheets and other manufacturer's recommendations.

7 Reagents

7.1 Test liquids, prepared and numbered according to Table 1.[1)]

The ratio of test liquids does affect the surface tension of the liquid. Use only analytical grades of test liquids. The surface tension of the liquids should be checked every month or the liquids in the dropper bottles should be replaced every month from a sealed stock solution bottle, since the isopropyl alcohol concentration decreases due to evaporation.

1) Test liquids are available from CBM Group of N.C. Inc., 1308 N. Ellis Ave., Dunn NC 28334 (Tel: +1 910-892-5701) or Textile Innovators Corp., Div. of SDL Atlas, 3934 Airway Dr., Rock Hill, SC 29732, USA; (Tel: +1 803-329-2110; Fax: +1 803-329-2133; info@sdlatlas.com). This information is given for the convenience of the users of this document 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.

Table 1 — Standard test liquids

Aqueous Solution Repellency Grade Number	Composition	Surface tension at 25 °C dyn/cm
0	None (fails 98 % water solution test liquid)	—
1	98:2/Water:isopropyl alcohol (by volume)	59,0
2	95:5/Water:isopropyl alcohol (by volume)	50,0
3	90:10/Water:isopropyl alcohol (by volume)	42,0
4	80:20/Water:isopropyl alcohol (by volume)	33,0
5	70:30/Water:isopropyl alcohol (by volume)	27,5
6	60:40/Water:isopropyl alcohol (by volume)	25,4
7	50:50/Water:isopropyl alcohol (by volume)	24,5
8	40:60/Water:isopropyl alcohol (by volume)	24,0

8 Apparatus and materials

8.1 Dropping bottles.

For convenience, it is desirable to transfer the test liquids from stock solutions to dropping bottles; each marked with the appropriate Aqueous Solution Repellency Grade Number. A typical system that is found to be useful consists of 60 ml dropping bottles with ground-in pipettes and neoprene bulbs. Standard dropping-bottle pipettes (medicine droppers) should deliver about 20 drops/ml. 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.

8.2 White textile blotting paper [2], approximately (0,71 ± 0,1) mm in thickness, (370 ± 5%) g/m^2, and with an absorbent capacity of (220 ± 30) %.

8.3 Laboratory gloves, general purpose.

9 Test specimens

Test two specimens from each sample, approximately no smaller than 20 cm × 20 cm and no larger than 20 cm × 40 cm. The test specimen size should be chosen to represent all physical and colour characteristics of the substrate and allow ample room for testing. Condition the test specimens for a minimum of 4 h prior to testing in accordance with ISO 139.

10 Test procedure

10.1 Place the first test specimen flat on the white textile blotting paper (8.2) on a smooth, horizontal surface with the face-side on top. Testing should be conducted in a 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.

2) White Textile Blotting Paper is the trade name of a suitable product supplied by AATCC, P.O. Box 12215, Research Triangle Park, NC 27709-2215, USA; (Tel: +1 919-549-8141; Fax: +1 919-549-8933; www.aatcc.org). This information is given for the convenience of the 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.

10.1.1 When evaluating open weave of “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.

10.1.2 Equipment, benches and gloves should be free of silicon.

10.2 Wearing clean laboratory gloves (8.3), brush the pile of napped or pile substrates lightly with your hand in the direction giving the greatest lay of the surface prior to depositing the drops of the test liquid. (This is the direction giving the lowest pile.)

10.3 Beginning with the lowest-numbered test liquid (Aqueous Solution Repellency Grade No. 1), *carefully* place small drops (approximately 5 mm in diameter or 0,05 ml volume) on the test specimen in a minimum of three 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 depositing drops. *Do not touch the substrate with the dropper tip*. Observe the drops for (10 ± 2) s, from approximately a 45° angle. Assess each drop in accordance with Figure 1.

10.4 If no penetration or wetting of the substrate at the liquid-substrate interface and no wicking around the drops occur, place drops of the next higher-numbered test liquid at an adjacent site on the substrate, so as not to interfere with the previous test, and again observe for (10 ± 2) s. Assess each drop in accordance with Figure 1.

10.5 Continue this procedure until one of the test liquids shows obvious wetting or wicking of the substrate under or around the drop within (10 ± 2) s.

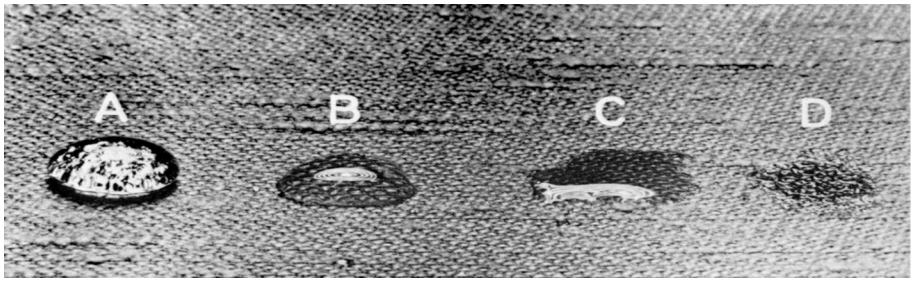
10.6 Repeat the procedure with the second specimen. A third specimen may be required (see Clause 12).

11 Evaluation

11.1 The Aqueous Solution 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 (10 ± 2) s. A grade of zero (0) is assigned when the substrate fails the 98 % water solution test liquid. 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 the contact angle of the drop. On black or dark substrates, wetting can be detected by loss of “sparkle” within the drop.

11.2 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 Aqueous Solution Repellency Grade, is obvious. However, some substrates will show progressive wetting under several test liquids as evidenced by a partial darkening of the fabric 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 (10 ± 2) s.

11.3 A **failure** occurs when two (or more) of the three drops applied from a given test liquid show complete wetting (Figure 1, Example D) or wicking with a loss of contact angle (Figure 1, Example C). A **pass** occurs if two (or more) of the three drops applied show a clear well-rounded appearance with a high contact angle (Figure 1, Example 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 two (or more) of the three drops applied show the rounded drop with partial darkening of the test specimen (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 — Grading examples [3)]

12 Evaluation of results

The Aqueous Solution Repellency Grade should be determined 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 values of 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. This grade variation may be an indication of a non-uniform fabric or contamination problems.

13 Test report

The test report shall include the following information:

a) a reference to this International Standard;

b) all information necessary for 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) Aqueous Solution Repellency Grade to the nearest 0,5 value (see Figure 1 and 11.3).

3) The Liquid Repellency Resistance Grading Scale is available from AATCC, P.O. Box 12215, Research Triangle Park, NC 27709-2215, USA; (Tel: +1 919-549-8141; Fax: +1 919-549-8933; www.aatcc.org). This information is given for the convenience of the users of this document 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.

Annex A
(informative)

Precision and bias

A.1 Summary

A.1.1 Intralaboratory tests were conducted in November 2002 and January 2003 to establish the precision of this test method. Both intralaboratory tests involved two participants in one laboratory rating two specimens of each of seven fabrics each day for three days. The grades of these intralaboratory tests use the scale from 1 to 8. All materials necessary for the intralaboratory tests were provided by customer fabric trials and were processed at two finish-load levels. The fabrics used included nylon, polyester, cotton and 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 Aqueous Solution Repellency Grade were calculated as given in Table A.1.

Table A.1 — Components of variance for alcohol/water test

Single operator	0,26
Between operator within laboratories	0,43

A.1.3 For the components of variance in A.1.2, two observations should be considered significantly different at the 95 % probability level if the difference equals or exceeds the critical differences given in Table A.2.

Table A.2 — Critical differences

No. of observations [a]	Single operator	Within laboratory
1	0,50	0,79
2	0,18	0,59
3	0,15	0,48
NOTE The critical differences were calculated using $t = 1{,}950$ from the Student's 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.		

Table A.3 gives mean values obtained from testing of specimens.

Table A.3 — Mean values from testing of specimens

Fabric	Finish load level	
	low	high
Cotton	3,5	5,5
Polyester	—	7,5
Cotton/Polyester	1,5	2,5
Nylon	6,0	8,0

A.2 Bias

The true value of the Aqueous Solution Repellency Grade can only be defined in terms of this test method. Within this limitation, this test method has no known bias.

Annex B
(informative)

Interlaboratory results

B.1 Summary

An interlaboratory trial was completed in March 2007. Five international laboratories (see Table B.1) participated. The fabric used was a khaki 100 % cotton twill with three varying finishes labeled as: 39P-2, 39P-3, 39P-11. Three replicates of each finish were sent to each laboratory (Lab.). The grades for these interlaboratory tests used the scale according to 7.1, Table 1.

B.2 Results

Table B.1 — Results

Finish	Specimen	Lab. 1 UK	Lab. 2 France	Lab. 3 Spain	Lab. 4 Portugal	Lab. 5 USA	Overall median
39P-2	Specimen 1	1,0 [a]	0,0	0,0	0,0	2,0 [b]	0,0
	Specimen 2	0,0 [a]	0,0	0,0	0,0	2,0 [b]	
	Specimen 3	2,0 [a]	0,0	0,0	0,0	2,0 [b]	
39P-3	Specimen 1	3,0	3,0	0,0 [b]	0,5 [b]	2,5	3,0
	Specimen 2	3,0	3,0	0,0 [b]	0,5 [b]	3,0	
	Specimen 3	3,0	3,0	0,0 [b]	0,5 [b]	3,0	
39P-11	Specimen 1	8,0	8,0	6,5 [b]	7,5	8,0	8,0
	Specimen 2	8,0	8,0	6,5 [b]	7,5	8,0	
	Specimen 3	8,0	8,0	6,5 [b]	7,5	8,0	

[a] Excluded from the final calculation of the overall median because of the dispersion of the results from the laboratory.

[b] Excluded from the final calculation of the overall median because of the statistical extreme median value of the laboratory.

B.3 Comments

B.3.1 Laboratory 1

A large variation was noticed depending on the height/angle of drop being exactly correct.

Samples were not conditioned; air system was under repair.

First time this test method was run.

B.3.2 Laboratory 2

39P-2: specimens were totally wet out with the liquid No.0 (water).

39P-11: specimens always passed, up to liquid No.8, then testing ended without wetting the specimens.

B.3.3 Laboratory 5

Very different results were obtained for 39P-2.

Specimens were actually tested two more times and obtained lots of variation:

— first test: when tested for the first time conditioned, the results were 1,5; 1,0; and 1,0;

— second test: when tested for the second time unconditioned, the results were 1,5; 2,0; and 2,0;

— third test: when tested for the third time conditioned, the results were 3,0; 3,0; and 3,0.

Bibliography

[1] ISO 3175 (all parts), *Textiles — Professional care, drycleaning and wetcleaning of fabrics and garments*

[2] ISO 6330, *Textiles — Domestic washing and drying procedures for textile testing*

[3] ISO 6530, *Protective clothing — Protection against liquid chemicals — Test method for resistance of materials to penetration by liquids*

[4] AATCC Test Method 193, *Aqueous Liquid Repellency: Water/Alcohol Solution Resistance Test*

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