



Leather — Determination of water resistance of flexible leather —

Part 1: Repeated linear compression (penetrometer)

Cuir — Détermination de l'imperméabilité à l'eau des cuirs souples —

Partie 1: Compression linéaire répétée (pénétromètre)

(Revision of first edition ISO 5403:2002)

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ISO/CEN PARALLEL PROCESSING

This draft has been developed within the European Committee for Standardization (CEN), and processed under the **CEN-lead** mode of collaboration as defined in the Vienna Agreement.

This draft is hereby submitted to the ISO member bodies and to the CEN member bodies for a parallel five-month enquiry.

Should this draft be accepted, a final draft, established on the basis of comments received, will be submitted to a parallel two-month approval vote in ISO and formal vote in CEN.

This draft International Standard is submitted to all ISO member bodies for voting, as a standard prepared by an international standardizing body in accordance with Council Resolution 42/1999. The proposer, International Union of Leather Technologists and Chemists Societies (Commission IUP, IULTCS), has been recognized by the ISO Council as an international standardizing body for the purpose of Council Resolution 42/1999.

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Foreword

ISO (the International Organisation for Standardisation) 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 organisations, 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 standardisation.

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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 5403-1 was prepared by the Physical Tests Commission of the International Union of Leather Technologists and Chemists Societies (IUP Commission, IULTCS) and the Working Group 2 of CEN TC 289 Leather, the Secretariat of this Technical Committee is held by UNI. This method is a commonly used method in the leather industry to test for dynamic water resistance of shoe upper leathers.

The IULTCS, originally formed in 1897, is a world-wide organisation of professional leather societies to further the advancement of leather science and technology. IULTCS has 3 Commissions, which are responsible for establishing international methods for sampling and the testing of leather. ISO recognises IULTCS as an International Standardising Body for the preparation of test methods for leather.

This second edition cancels and replaces the first edition (EN ISO 5403:2002), which has been technically revised.

ISO 5403 consists of the following parts, under the general title *Leather — Determination of water resistance of flexible leather*.

- *Part 1: Repeated linear compression (penetrometer)*
- *Part 2: Repeated angular compression (Maeser)*

Leather — Determination of water resistance of flexible leather —

Part 1: Repeated linear compression (penetrometer)

1 Scope

This Standard specifies a method to determine the dynamic water resistance of leather by means of repeated linear compression. It is applicable to all flexible leathers but is particularly suitable for leathers intended for footwear applications.

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 2418 Leather - Chemical, physical and mechanical and fastness tests - Sampling location

ISO 2419 Leather - Physical and mechanical tests – Sample preparation and conditioning

ISO 3696:1987 Water for analytical laboratory use – Specification and test methods

3 Principle

A test piece is formed into the shape of a trough and flexed whilst partially immersed in water. The time taken for water to penetrate through the test piece is measured. The method also allows the percentage mass of water absorbed and the mass of water transmitted through the test piece to be determined.

4 Apparatus

4.1 Test machine, including the parts described in 4.1.1 to 4.1.3.

4.1.1 One or more pairs of cylinders, 30,0 mm \pm 0,5 mm diameter made of inert rigid material, mounted with their axes horizontal and co-axial. One cylinder shall be fixed and the other moveable along the direction of its axis such that the maximum separation of the cylinders is 40,0 mm \pm 0,5 mm.

4.1.2 Electric motor, which drives the moveable cylinder backwards and forwards along its axis with a crank motion of 50 cycles/min \pm 5 cycles/min and with amplitude of 1,0 mm \pm 0,1 mm, 1,50 mm \pm 0,15 mm, 2,0 mm \pm 0,2 mm or 3,0 mm \pm 0,3 mm about its mean position.

NOTE The four amplitudes of the crank motion are such that the test piece is compressed by 5%, 7,5%, 10% or 15% respectively when the cylinders approach one another.

4.1.3 Tank, made from non-corroding material, holding distilled or deionized water, in which the test piece can be partially immersed.

NOTE The test machine may also include an electrical circuit that indicates when water has penetrated through the test piece.

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.

4.2 Ring shaped clamps, with internal diameter adjustable between 30 mm and 40 mm.

4.3 Press knife, the inner wall of which is a rectangle $60 \text{ mm} \pm 1 \text{ mm} \times 75 \text{ mm} \pm 1 \text{ mm}$, conforming to the requirements of ISO 2419.

4.4 Distilled or deionized water, conforming to the requirements of grade 3 of ISO 3696: 1987.

4.5 Balance, weighing to 0,001 g.

4.6 Clock, reading to 1 s.

4.7 Abrasive paper, grade P180, as defined in the P-series grain size standard published by the Federation of European Producers of Abrasive Products, cut into rectangles $65 \text{ mm} \pm 5 \text{ mm} \times 45 \text{ mm} \pm 5 \text{ mm}$, fixed to a flat, rigid base of the same size and weighted to give a total mass of $1,0 \text{ kg} \pm 0,1 \text{ kg}$. A fresh piece of abrasive paper is to be used for each test.

4.8 Absorbent cloth, cut into rectangles $120 \text{ mm} \pm 5 \text{ mm} \times 40 \text{ mm} \pm 5 \text{ mm}$, machine washed prior to first use using the cycle recommended by the cloth manufacturer.

NOTE A suitable cloth is terry towelling (frotté) in 100% cotton and weighing about 300 g/m^2 . The absorbency of this material may not be optimal when new and therefore the cloths must be washed before the first use.

4.9 Auxiliary apparatus, to determine the stiffness of the test piece, consisting of one pair of cylinders, $30,0 \text{ mm} \pm 0,5 \text{ mm}$ diameter mounted with their axes horizontal and co-axial, a means of moving the cylinders together, a means of measuring the reduction in distance between the cylinders, to the nearest 0,1 mm, a means of measuring the force exerted along the axes of the cylinder to the nearest 5 N.

5 Sampling and sample preparation

5.1 Sample in accordance with ISO 2418. Cut four test pieces by applying the press knife (4.3) to the grain

surface. Cut two test pieces with the longer side parallel to the backbone and two test pieces with the longer side perpendicular to the backbone.

NOTE If there is a requirement for more than two hides or skins to be tested in one batch, then only one test piece in each direction need be taken from each hide or skin, provided that the overall total is not less than two test pieces in each direction.

5.2 Prepare the four test pieces by the following method:

Lightly buff the grain surface by placing the test piece grain upwards on a flat surface. Place the weighted abrasive paper (4.7) on the test piece and move the abrasive paper ten times backwards and forwards the full length of the test piece without applying any more downward force than is applied by the weighted abrasive.

NOTE 1 In some situations it may be more appropriate to flex a sample for 20 000 cycles using the method and apparatus specified in ISO 5402.

NOTE 2 Many leathers have on the grain a surface coat which greatly increases the water resistance of the leather. If microcracks develop rapidly in this coat as a result of flexing in wear or the coat is damaged by abrasion, then measurements made on the leather as received can be misleading. The abrasion and flexing treatments described above are intended to simulate the abrasion which the leather would receive in wear and the test piece should therefore be abraded or flexed before test. The purpose of this abrasion is not to remove the surface coat but merely to scratch it lightly

5.3 Store the test specimens in a standard controlled environment according to ISO 2419 for at least 48 hours. It is not necessary to carry out the test in this atmosphere.

5.4 If the water transmitted through the test piece is to be measured, condition a rectangle of absorbent cloth (4.8) in accordance with clause 5.3, weigh to the nearest 0,001 g and record the mass.

5.5 If the water absorption of the test piece is to be measured, weigh the test piece to the nearest 0,001 g and record the mass.

6 Procedure

6.1 Determination of stiffness and test amplitude

NOTE The stiffness and test amplitude are not determined if the test amplitude is otherwise specified.

6.1.1 Adjust the auxiliary apparatus (4.9) so that the cylinders are at the maximum separation.

6.1.2 Bend the test piece along the longer edges, with the grain or outer surface in wear facing outwards to form a trough and with the shorter edges parallel, and at the same level. Attach the longer edges to the cylinders by means of the ring clamps (4.2) with the same length of test piece (about 10 mm) overlapping each cylinder and with the test piece under sufficient tension to remove folds. The inner edges of the two ring clamps should lie as nearly as possible in the planes of the adjacent ends of the cylinders so that the length of the trough is the same as the distance between the cylinders. If the test piece and cylinders are to be transferred to the main test machine (4.1) ensure that the test piece forms a seal against the cylinder.

6.1.3 Move the cylinders $2,0 \text{ mm} \pm 0,1 \text{ mm}$ closer to each other evenly over a period of $5 \text{ s} \pm 2 \text{ s}$ and immediately return the cylinders to their original position over a period of $5 \text{ s} \pm 2 \text{ s}$.

6.1.4 Repeat the operations in 6.1.3 and record the force acting on the cylinders to the nearest 5 N.

6.1.5 Repeat the operation in 6.1.3 but this time moving the cylinders $4,0 \text{ mm} \pm 0,2 \text{ mm}$ closer to each other and record the force acting on the cylinders to the nearest 5 N.

6.1.6 Calculate the arithmetic mean of the forces recorded in 6.1.4 and 6.1.5. If the mean force is greater than or equal to 100 N then the amplitude of test is $1,0 \text{ mm} \pm 0,1 \text{ mm}$ (equivalent to 5% compression of the test piece).

If the mean force is greater than or equal to 50 N (but less than 100 N) then the amplitude of test is $1,50 \text{ mm} \pm 0,15 \text{ mm}$ (equivalent to a 7,5% compression of the test piece).

If the mean force is less than 50 N follow the procedure in 6.1.7 and 6.1.8.

6.1.7 Repeat the operation in 6.1.3 but this time moving the cylinders $6,0 \text{ mm} \pm 0,3 \text{ mm}$ closer to each other and record the force acting on the cylinder to the nearest 5 N.

6.1.8 Calculate the arithmetic mean of the forces recorded in 6.1.4, 6.1.5 and 6.1.7. If the mean force is greater than or equal to 20 N then the amplitude of the test is $2,0 \text{ mm} \pm 0,2 \text{ mm}$ (equivalent to a 10% compression of the

test piece). If the mean force is less than 20 N then the amplitude of test is $3,0 \text{ mm} \pm 0,3 \text{ mm}$ (equivalent to a 15% compression of the test piece).

6.2 Determination of penetration time

6.2.1 Set the test machine (4.1) so that the amplitude of test is as determined in (6.1) or as required by the specification.

6.2.2 Adjust the test machine (4.1) so that the cylinders (4.1.1) are at the maximum separation.

6.2.3 Bend the test piece along the longer edges, with the grain or outer surface in wear facing outwards, to form a trough with the shorter edges parallel and at the same level. Attach the longer edges to the cylinders by means of the ring clamps (4.2) with the same length of test piece (about 10 mm) overlapping each cylinder and with the test piece under sufficient tension to remove folds. The inner edges of the two ring clamps should lie as nearly as possible in the planes of the adjacent ends of the cylinders so that the length of the trough is the same as the distance between the cylinders. Ensure that the test piece forms a seal against the cylinders.

NOTE If the cylinders are removable then they and the attached test piece may be transferred from the auxiliary apparatus (4.9) to the test machine (4.1).

6.2.4 Raise the level of water in the freshly filled tank until the surface lies $10 \text{ mm} \pm 1 \text{ mm}$ below the top of the cylinders.

6.2.5 Start the motor and note the time.

6.2.6 Observe the test piece continuously for the first 15 min then at intervals of 15 min until water is seen to penetrate through the test piece. If the water penetrates between the test piece and the cylinder then reject the result and repeat the determination using a fresh test piece. Note the time when penetration occurs.

NOTE 1 An electrical device may be used to assist in the determination of water penetration but penetration should also be confirmed visually.

NOTE 2 Penetration may be seen as a damp patch or as a droplet (or droplets) of water formed on the surface.

6.3 Determination of water absorption

6.3.1 Carry out the steps given in 6.2.1 to 6.2.5.

6.3.2 After the required time has elapsed, stop the test machine, remove the test piece, blot gently to remove adhering water, weigh the test piece to the nearest 0,001 g and record the mass.

6.3.3 If other determinations are required, replace the test piece and continue the test.

6.4 Determination of water penetration

6.4.1 After initial water penetration has occurred, place a rolled up rectangle of absorbent material into the trough formed by the test piece.

6.4.2 Continue the test until the required time has elapsed. Remove the absorbent material and use it to mop up any excess water from the trough.

6.4.3 Weigh the absorbent material to the nearest 0,001 g and record the mass.

7 Expression of results

7.1 Penetration time

The penetration time shall be reported directly in minutes or hours and minutes, as convenient.

7.2 Water absorption

The percentage water absorption, W_a , shall be calculated using the formula:

$$W_a = \frac{(M_1 - M_2) \times 100}{M_0}$$

where:

M_1 is the mass of the test piece after any time period, in grams

M_0 is the initial conditioned mass of the test piece, in grams

7.3 Water transmission

The water transmission, W_t , in grams, shall be calculated using the formula:

$$W_t = W_1 - W_0$$

where:

W_1 is the mass of the absorbent material after test, in grams

W_0 is the initial conditioned mass of the absorbent material, in grams

8 Test report

The test report shall include the following

- a) reference to this International Standard, i.e. ISO 5403-1;
- b) the penetration time for each test piece tested;
- c) the water absorption, W_a , at each time interval, if measured;
- d) the water transmission, W_t , and the period over which it was determined, if measured;
- e) the conditions in ISO 2419 used to condition the test specimens, if different from reference standard conditions;
- f) any deviations from the method specified in this International Standard;
- g) full details for identification of the sample and any deviation from ISO 2418 with respect to sampling.

Annex A
(informative)

Sources of apparatus

Examples of suitable products available commercially are given below. This information is given for the convenience of users of this International Standard and does not constitute an endorsement by ISO of these products.

The recommended apparatus is the penetrometer, manufactured, for example, by:

Giuliani Apparecchi Scientifici, via Centrallo 68/18, I-10157 Torino, Italy;

SODEMAT, 29 rue Jean Moulin, ZA Coulmet, F – 10450 Breviandes, France;

SATRA Technology Centre, SATRA House, Rockingham Road, Kettering, Northamptonshire NN16 9JH, England;

Muver - Francisco Muñoz Irlles, Avda Hispanoamerica 42, E-03610 Petrer (Alicante), Spain

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Bibliography

ISO 5402 Leather - Physical and mechanical tests - Determination of flex resistance by flexometer method

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