

INTERNATIONAL STANDARD

ISO
17698

First edition
2003-10-15

Footwear — Test methods for uppers — Delamination resistance

*Chaussures — Méthodes d'essai des tiges — Résistance au
dé laminage*

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Reference number
ISO 17698:2003(E)

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Case postale 56 • CH-1211 Geneva 20
Tel. + 41 22 749 01 11
Fax + 41 22 749 09 47
E-mail copyright@iso.org
Web www.iso.org

Published in Switzerland

Foreword

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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 17698 was prepared by CEN (as EN 13514:2001) and was adopted, under a special “fast-track procedure”, by Technical Committee ISO/TC 216, *Footwear*, in parallel with its approval by the ISO member bodies.

For the purposes of international standardization, a list of corresponding International and European Standards for which equivalents are not given in EN 13514 has been added as Annex ZZ.

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Foreword

This European Standard has been prepared by Technical Committee CEN/TC 309 "Footwear", the secretariat of which is held by AENOR.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by May 2002, and conflicting national standards shall be withdrawn at the latest by May 2002.

This European Standard is based on the IULTCS/IUF 470 method (ISO 11644:1993 "Leather - Test for adhesion of finish").

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Czech Republic, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and the United Kingdom.

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EN 13514:2001 (E)

1 Scope

This European Standard specifies a test method for determining the delamination resistance of uppers irrespective of the material, in order to assess the suitability for the end use.

2 Normative references

This European Standard incorporates by dated or undated reference, provisions from other publications. These normative references are cited at the appropriate places in the text, and the publications are listed hereafter. For dated references, subsequent amendments to or revisions of any of these publications apply to this European Standard only when incorporated in it by amendment or revision. For undated references the latest edition of the publication referred to applies (including amendments).

EN 12222, *Footwear - Standard atmospheres for conditioning and testing of footwear and components for footwear*.

EN 12749, *Footwear - Ageing conditioning*.

EN 13400, *Footwear - Sampling location, preparation and duration of conditioning of samples and test pieces*.

EN ISO 3696, *Water for analytical laboratory use - Specification and test method*.

EN ISO 7500-1, *Metallic materials – Verification of static uniaxial testing machines. Part 1: Tension/compression testing machines*.

3 Term and definition

For the purposes of this European Standard, the following term and definition applies.

3.1

delamination resistance

strength of adhesion between a coating and its base material

4 Apparatus and material

The following apparatus and material shall be used:

4.1 Tensile testing machine with: a jaw separation rate of 100 mm/min $\pm$ 10 mm/min, a force range appropriate to the specimen under test (normally a range of 0 N to 200 N is suitable for test specimens of polyurethane coated fabric) and the capability of measuring the force to an accuracy greater than 2 % as specified by Class 2 in EN ISO 7500-1.

4.2 Autographic recorder or similar means of continuously recording the force.

4.3 Rapid acting platen press capable of applying a pressure of 550 kPa $\pm$ 50 kPa on an area of 50 mm x 70 mm.

4.4 Rubber pad of thickness at least 10 mm and hardness 40 IRHD $\pm$ 10 IRHD.

4.5 Radiant heater capable of heating a dry adhesive film on resin rubber to 80 °C to 90 °C within 15 s, normally mounting the adhesive film from 100 mm to 150 mm from a heater element of power approximately 3 kW and area of approximately 0,06 m² is satisfactory. Commercial equipment used for reactivating soles and uppers in footwear production is suitable.

4.6 Means of checking that the temperature of the adhesive film is within the range 80 °C to 90 °C. Heat sensitive crayons are suitable, preferably with a melting temperature of 83 °C. Also suitable are infra-red temperature measuring guns.

4.7 Resin rubber, thickness $3,5 \text{ mm} \pm 0,2 \text{ mm}$ and hardness 95 IRHD ± 2 IRHD with a surface peel tear strength greater than that of the test specimen.

4.8 A solvent-borne polyurethane adhesive which will bond well to resin rubber and the coated surface of the test specimen.

4.9 An adhesion primer, such as a halogenating solution used in footwear manufacture for rubber can be helpful in producing satisfactory bonds.

4.10 Cutting device such as a press knife or scissors capable of cutting rectangular test specimens of dimensions $(50 \text{ mm} \pm 1 \text{ mm}) \times (70 \text{ mm} \pm 1 \text{ mm})$. In addition, if carrying out the test on hydrolysed test specimens, a second cutting device is required to cut square test specimens $(70 \text{ mm} \pm 1 \text{ mm}) \times (70 \text{ mm} \pm 1 \text{ mm})$.

4.11 Cutting device such as a sharp knife or rotary disc cutter for cutting test specimens from bonded test assemblies. This device shall neither unduly compress nor force apart the layers of the test assembly at the edges during cutting and therefore a press knife is unsuitable.

4.12 If testing the wet adhesion strength, **distilled or deionised water** complying with grade 3 of EN ISO 3696.

4.13 Timer capable of recording times up to 30 s to the nearest 0,5 s.

5 Sampling

5.1 For the dry tests, mark six rectangular boxes $(70 \text{ mm} \pm 1 \text{ mm}) \times (50 \text{ mm} \pm 1 \text{ mm})$: two with their longer edges parallel to the along direction of sheet material (machine or backbone direction) or X-axis (as defined in EN 13400) of the upper, and four with their longer edges perpendicular to this, on the reverse of the sheet material or uppers.

5.2 For the wet tests mark either a further two rectangular boxes $(70 \text{ mm} \pm 1 \text{ mm}) \times (50 \text{ mm} \pm 1 \text{ mm})$, putting the 50 mm edge in the direction with the lowest dry peel strength (if already known), or a further six boxes as described in 5.1 on the reverse of the material or uppers.

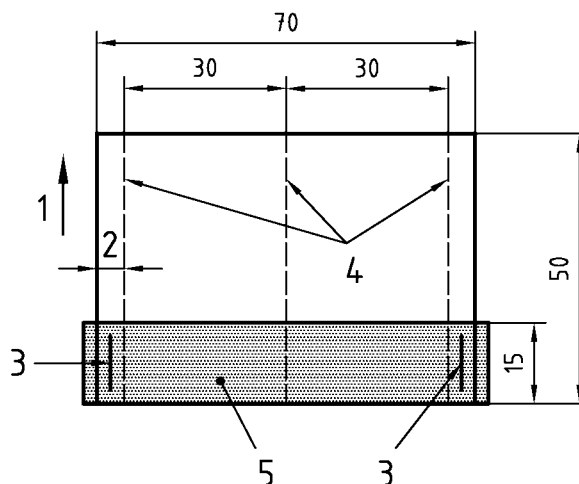
5.3 Make further marks on the material to divide each of the rectangles marked in 5.1 and 5.2 into two equal halves $(35 \text{ mm} \pm 0,5 \text{ mm}) \times (50 \text{ mm} \pm 1 \text{ mm})$. Mark the along direction or X-axis in each of the smaller rectangles. Use an arrow and ensure that the arrow heads point in the same direction. For uppers, the arrow head shall point towards the toe.

5.4 For hydrolysis tests, mark a further two $70 \text{ mm} \pm 1 \text{ mm}$ square boxes with their edges parallel to the along direction or X-axis. Mark the along direction or X-axis using an arrow as in 5.3.

NOTE Since ink marks can be removed by the wetting or hydrolysis treatments it is advisable to also use a code of cut off corners.

5.5 Cut from the sheet material or uppers, the rectangular boxes marked in 5.1 and 5.2 and the squares marked in 5.4. Each piece of material is later cut to give two test specimens $30 \text{ mm} \pm 0,5 \text{ mm} \times 50 \text{ mm} \pm 1 \text{ mm}$, see Figure 1. After bonding to resin rubber, each piece of material is later cut to give two peel test specimens. With some shoe styles it will not be possible to cut the correct size pieces of material. In such cases it will be necessary to reduce the size of the pieces to a minimum of $(40 \text{ mm} \pm 1 \text{ mm}) \times (50 \text{ mm} \pm 1 \text{ mm})$. In all such cases, the pieces will give one test specimen only and shall not be further sub-divided as in 5.3.

5.6 Cut a rectangular piece of resin rubber (see 4.7) measuring $(50 \text{ mm} \pm 1 \text{ mm}) \times (70 \text{ mm} \pm 1 \text{ mm})$ for each piece of material cut in 5.5. If the setting of the radiant heater unit (see 4.5) needs to be checked cut one or two additional pieces of rubber.

**Key**

- 1 Direction of peel
- 2 Approximately 5 mm
- 3 Staple
- 4 Cut lines
- 5 Paper strip

Figure 1 — Test specimen assembly

NOTE Specimens can be taken from materials likely to be used for uppers or from made-up uppers or finished footwear.

6 Test method**6.1 Principle**

The coated surface of a test specimen is bonded to a piece of resin rubber using a strong adhesive. The force required to peel the test specimen from the resin rubber leaving the coating attached to the rubber is measured using a tensile testing machine. The test can also be carried out on wet and hydrolysed test specimens.

6.2 Procedure

6.2.1 Place a strip of paper ($75 \text{ mm} \pm 5 \text{ mm}$) $\times$ ($15 \text{ mm} \pm 3 \text{ mm}$) to the coated side of each piece of material cut in 5.5 so that it is against one of the longer edges. Attach the paper strips with a staple (or similar) at each end so that the staples are positioned as shown in Figure 1. With the four test specimens cut for the dry test where the arrow points toward a longer edge, two shall have the paper strip attached along the edge to which the arrow is pointing and two shall have the paper strip along the opposite edge.

6.2.2 If an adhesion primer (see 4.9) is available, apply this in accordance with the suppliers instructions to the whole area of the reverse side of each piece of resin rubber cut in 5.6.

6.2.3 Allow the resin rubber pieces to dry full in accordance with the suppliers recommendations.

6.2.4 Apply the polyurethane adhesive (see 4.8) in accordance with the suppliers instructions to the whole area of the reverse of each piece of resin rubber.

6.2.5 Similarly apply adhesive to the coated surface of each piece of material under test so that it just overlaps the paper strip.

6.2.6 Allow the adhesive to dry for at least 1 h.

6.2.7 If the time required to heat the adhesive film to a temperature of between 80°C and 90°C using the radiant heater is not known, then measure this time using the extra pieces of rubber cut in 5.6 and the means of checking the temperature of the adhesive film (see 4.6). If this time is found to be longer than 15 s then either

increase the temperature of the heating element or reduce the distance between the element and the resin rubber until the time is below 15 s. Record the time taken to heat the adhesive film to the desired temperature as T_a to the nearest 1 s.

6.2.8 Preparation of bonded assemblies:

6.2.8.1 Place a piece of adhesive coated resin rubber in the radiant heater with the adhesive surface facing the heater element so that it is subjected to heat for T_a (in seconds).

6.2.8.2 Carefully and quickly place the adhesive coated surface of a piece of upper material (5.5) in contact with the adhesive coating on the piece of resin rubber so that the edges of both surfaces are aligned. This will subsequently be referred to as a test assembly.

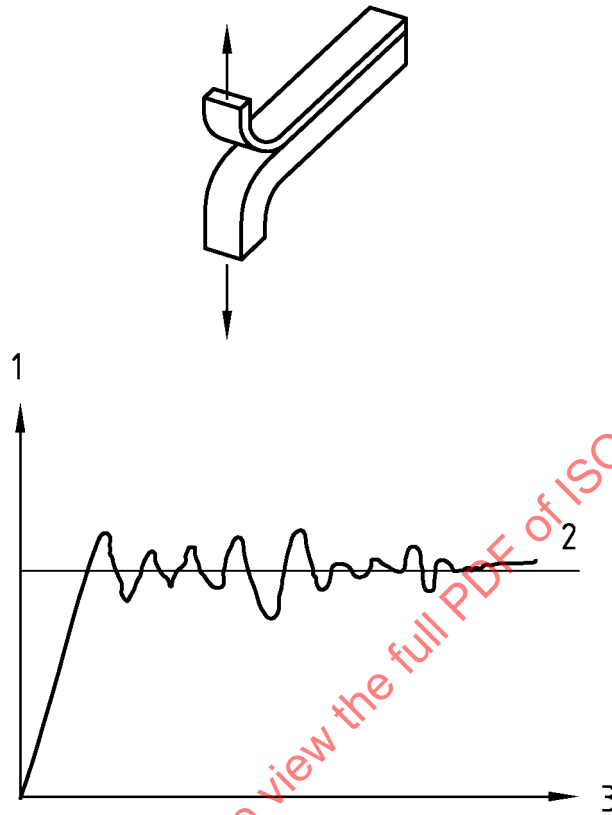
6.2.8.3 Immediately place the test assembly into the platen press (see 4.3) so that the resin rubber is lowermost and resting on the rubber pad (see 4.4). Apply a pressure of $550 \text{ kPa} \pm 50 \text{ kPa}$ to the assembly for $15 \text{ s} \pm 1 \text{ s}$.

It is important that the time between removing the resin rubber from the heater unit in 6.2.8.1 to placing the assembly in the press and applying pressure in 6.2.8.3 shall be no more than 7 s.

6.2.9 Store the bonded assemblies in a conditioned standard atmosphere as specified in EN 12222 for at least 24 h.

6.2.10 Use the cutting device (see 4.11) to make three cuts in each test assembly parallel to the 50 mm edges so that the centre portions form two test specimens of width $30,0 \text{ mm} \pm 0,5 \text{ mm}$ and length $50 \text{ mm} \pm 1 \text{ mm}$ and the two outer portions are waste strips of width approximately 5 mm, see Figure 1.

6.2.11 Open the unbonded portion of each test specimen, taking care not to weaken the bond line, and centrally clamp it between the jaws of the tensile tester (see 4.1) so that the unbonded tab of resin rubber is in one jaw and the unbonded tab of upper material is in the other jaw (see Figure 2).

**Key**

- 1 Delaminating force, in N
- 2 Average
- 3 Deformation

Figure 2 — Example of diagram force/deformation

6.2.12 Operate the tensile tester so that the jaws separate at a speed of $100 \text{ mm/min} \pm 10 \text{ mm/min}$ and note the type of separation that occurs, such as:

- a) failure of adhesion of the coating to the base fabric;
- b) surface failure of the base fabric;
- c) deep failure of the base fabric;
- d) failure of adhesion of a top coating to a cellular coagulated coating;
- e) failure within the cellular or coagulated coating;

f) separation between a cellular or coagulated layer and the base fabric.

If the separation is between the surface coating and the resin rubber, momentarily stop the test and without removing the test specimen from the tensile tester, using a sharp knife carefully cut through the surface coating close to the point where it meets the resin rubber to encourage separation of the coating from the supporting material.

6.2.13 From the trace of force versus jaw movement produced by the tensile testing machine, measure the:

6.2.13.1 Initial maximum force in newtons required to break through the coating.

6.2.13.2 Average force (see Figure 2) in newtons required to peel the coating from its base (average of all force readings after the initial maximum peak).

6.2.14 Repeat the procedure in 6.2.11 to 6.2.13 for the remaining test specimens that are to be tested dry.

6.2.15 For the test specimens that are to be tested wet:

6.2.15.1 Select the test specimens corresponding to the lowest dry peel strength calculated according to 7.1.1 and wet the test specimens by immersing in water (see 4.12) at $23\text{ °C} \pm 2\text{ °C}$ for $6,0\text{ h} \pm 0,5\text{ h}$.

6.2.15.2 Remove each test specimen from the water, blot the surfaces gently to remove surplus water and immediately carry out the test using the procedure in 6.2.11 to 6.2.13.

6.2.16 If testing test specimens after hydrolysis:

6.2.16.1 Follow the procedure in EN 12749 to produce two test specimens of hydrolysed material.

6.2.16.2 Cut a rectangular test specimen of material ($70\text{ mm} \pm 1\text{ mm}$) x ($50\text{ mm} \pm 1\text{ mm}$) from each piece of hydrolysed material so that the 50 mm edges are parallel to the direction of lowest dry peel strength calculated according to 7.1.2.

6.2.16.3 Repeat the procedure in 6.2.1 to 6.2.13 for the two test specimens of hydrolysed material.

7 Expression of results

7.1 For each test specimen, calculate:

7.1.1 The initial maximum peel strength, in N/mm to the nearest 0,1 N/mm, by dividing the initial maximum force measured in 6.2.13.1, by the test specimen width.

7.1.2 The average peel strength, in N/mm to the nearest 0,1 N/mm, by dividing the average force measured in 6.2.13.2, by the test specimen width.

7.2 For each peel direction, calculate:

7.2.1 The arithmetic mean of the initial maximum peel strengths calculated in 7.1.1.

7.2.2 The arithmetic mean of the average peel strengths calculated in 7.1.2.

8 Test report

The test report shall include the following information:

a) for each type of test specimen:

- any pre-treatment; either wetting or hydrolysis;