BRITISH STANDARD

BS EN
1392:2006

Adhesives for leather and footwear materials — Solvent-based and dispersion adhesives — Testing of bond strength under specified conditions

The European Standard EN 1392:2006 has the status of a British Standard

ICS 61.060; 83.180

BSI
British Standards

National foreword

This British Standard is the official English language version of EN 1392:2006. This document, together with BS EN 15062:2006 supersedes BS EN 1392:1998 which is withdrawn.

The UK participation in its preparation was entrusted to Technical Committee PRI/52, Adhesives, which has the responsibility to:

— aid enquirers to understand the text;

— present to the responsible international/European committee any enquiries on the interpretation, or proposals for change, and keep UK interests informed;

— monitor related international and European developments and promulgate them in the UK.

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Summary of pages

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May 2006

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English Version

Adhesives for leather and footwear materials - Solvent-based and dispersion adhesives - Testing of bond strength under specified conditions

Adhésifs pour cuir et matériaux de la chaussure - Adhésifs à base de solvant ou à dispersion- Méthodes d'essai pour mesurer la résistance de collage dans certaines conditions spécifiées

Klebstoffe für Leder und Schuhwerkstoffe - Lösemittel- und Dispersionsklebstoffe - Prüfung der Festigkeit von Klebungen unter festgelegten Bedingungen

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Management Centre: rue de Stassart, 36 B-1050 Brussels

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Foreword

This document (EN 1392:2006) has been prepared by Technical Committee CEN/TC 193 “Adhesives”, 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of an identical text or by endorsement, at the latest by November 2006, and conflicting national standards shall be withdrawn at the latest by November 2006.

This document, together with EN 15062, supersedes EN 1392:1998.

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1 Scope

This European Standard specifies the testing of some strength properties of bonds of leather and footwear materials, in stuck-on assemblies using solvent-based and dispersion adhesives, under different conditions. These can be chosen taking into account the different stresses that such bonds are subjected to, depending on the type of footwear.

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.

EN 923:2005, *Adhesives –Terms and definitions*

EN 1067, *Adhesives – Examination and preparation of samples for testing*

EN 12961, *Adhesives for leather and footwear materials – Determination of optimum activation temperatures and maximum activation life of solvent-based and dispersion adhesives*

EN 15307:2005, *Adhesives for leather and footwear materials – Sole-upper bonds - Minimum strength requirements*

EN ISO 868, *Plastics and ebonite – Determination of indentation hardness by means of a durometer (Shore hardness) (ISO 868:2003)*

EN ISO 7500-1, *Metallic materials - Verification of static uniaxial testing machines - Part 1: Tension/compression testing machines - Verification and calibration of the force-measuring system (ISO 7500-1:2004)*

EN ISO 10365, *Adhesives – Designation of main failure patterns (ISO 10365:1992)*

EN ISO 15605, *Adhesives – Sampling (ISO 15605:2000)*

ISO 554, *Standard atmospheres for conditioning and/or testing – Specifications*

ISO 2602, *Statistical interpretation of test results – Estimation of the mean – Confidence interval*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in EN 923:2005 and the following apply.

3.1 leather
tanned animal skin, usually free of hair

3.2 footwear materials
natural and synthetic materials which are suitable for footwear manufacture or repair and have adequate wear properties as upper or soling material

3.3 adhesives for leather and footwear materials
adhesives which are intended to produce firm and durable bonds of leather and footwear materials

4 Principle

The surface of the leather or the footwear material used is treated specifically to the nature of the material. Then strips of specified length and width are cut from the treated material.

Two of these strips or one strip of the above mentioned material and one strip of a suitable different material are bonded by an adhesive to test pieces of specified form.

The test pieces are stored under specified conditions and their bond strength is determined under specified conditions.

5 Test methods

5.1 Types of tests

5.1.1 Peel tests at (23 ± 2) °C

See 5.4.2

5.1.2 Peel test under constant load and at a constant elevated temperature ("creep test")

See 5.4.3

5.2 Adhesives and materials

5.2.1 Footwear adhesive

Identify the adhesive used completely in the test report, in particular note name and/or designation, manufacturer, date of manufacture/supply and/or lot number, main polymer, type (solvent-based or dispersion) and colour. For two-part adhesives identify the nature of the crosslinking agent and the mixing ratio of the components. Record this adhesive identification in the test report.

Some one or two part reference footwear test adhesives with specific properties have been developed for research, development and quality certification purposes (see EN 15307:2005, Annex A). Record the designation, source and date of supply of the reference test adhesive, if used, in the test report.

5.2.2 Leather and Footwear materials

Completely identify the footwear material(s) used in the test report.

Record name and/or designation, manufacturer, date of manufacture/supply, type of leather or of footwear material, e.g. soling or upper material. For leathers list colour, thickness and type of tannage (if known), for rubber and plastic materials colour, polymer base and Shore-hardness according to EN ISO 868. Include this identification of the material in the test report. Some reference footwear test materials with specified properties have been developed for research, development and quality certification purposes (see EN 15307:2005, Annex A). Record the designation, source and date of supply of the reference footwear test materials, if used, in the test report.

5.3 Apparatus and reagents

5.3.1 General

The items required will depend on the types of materials used:

5.3.2 Cutting knife

sharp knife, for cutting test pieces according to 5.6.2. The angle between the inner and outer cutting surface shall be approximately 20°;

5.3.3 Splitting machine

for splitting outsole leather;

5.3.4 Roughing machine

consists of a rotary wire brush with a wire diameter between 0,1 mm and 0,4 mm. The linear speed of revolution shall be between 10 m/s and 25 m/s;

5.3.5 Scouring machine

with a drum covered with emery paper or emery cloth of 60 grit size and a linear speed of revolution of between 10 m/s and 20 m/s;

5.3.6 Hard felt disc

made from wool for removing thin PVC coats from PVC upper materials;

5.3.7 Brush for dust removing

hand or mechanical device for removing the dust from strips of material after roughing or scouring;

5.3.8 Material for solvent wiping

suitable lintfree fabric or cotton wool. The material for solvent wiping must remain unaffected by the solvent used. Size about 150 mm x 150 mm;

5.3.9 Solvents

ethyl acetate (acetic acid ethyl ester) acetone or light petroleum, boiling range 80 °C to 110 °C;

5.3.10 Halogenation agent, solvent borne, 1- or 2-part

for treating rubber surfaces;

5.3.11 Brush for halogenation

hard and soft with a non-metallic bristle holder for halogenation; bristle length about (20 ± 5) mm;

5.3.12 Adhesive applicator

e.g. brush, roller, coating machine etc. for uniform coating of the adhesive under test;

5.3.13 Heat activator

for heating adhesive coats to the required temperature;

5.3.14 Equipment for measurement of temperature

thermocouple, infrared thermometer, thermoindicator papers or melting powders for measuring the temperature of activated adhesive coats;

5.3.15 Pressing device

providing a precise and even pressure of up to 0,6 MPa over the whole of the surface to be bonded;

5.3.16 Tensile testing machine

according to EN ISO 7500-1 for measuring maximum separation forces up to 10 kN and suitable force measurement ranges.

The tensile testing machine shall record automatically the separation forces determined, and to be adjustable to a constant rate of traverse of (100 ± 10) mm/min during the test.

5.3.17 Oven

with forced ventilation capable of storing test pieces at (50 ± 2) °C.

5.3.18 Warm air cabinet

with forced ventilation capable of maintaining the contents at temperatures between 40 °C and 100 °C with tolerances of ± 2 °C and suitable for testing test pieces in accordance with 5.4.The test cabinet shall be fitted with a window and devices for clamping five test pieces. These devices shall consist of upper clamps fixed on a metal bar in the cabinet and lower clamps which shall have means for attaching weights which pass through cylindrical holes in the base of the cabinet. All lower clamps shall be of the same mass and capable of being loaded with weights so that total loads applied by the individual clamps are 0,5 kg, 1,0 kg, 1,5 kg, 2,0 kg and 2,5 kg.

5.3.19 Weights

with a tolerance of ± 1 % for applying loads of 0,5 kg, 1,0 kg, 1,5 kg, 2,0 kg and 2,5 kg, including the weight of a clamp to the test pieces.

5.4 Form of test pieces

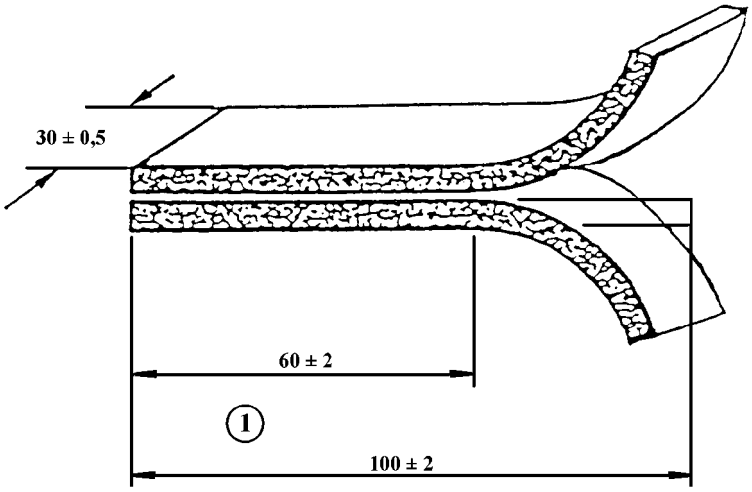
5.4.1 General

Prepare from the leather and footwear materials strips as specified in 5.6.2. If a material, does not allow the cutting of strips of the required dimensions, smaller and/or shorter strips may be used for preparing test pieces of the specified form, providing that their dimensions and overlap are recorded in the test report.

5.4.2 Peel test at (23 ± 2) °C

Two strips of material up to (100 ± 2) mm long and (30 ± 0,5) mm wide are bonded to cover each other over a length of at least (60 ± 2) mm (see Figure 1).

Dimensions in mm



Key
1 length of the bond in millimetres

Figure 1 — Form and dimensions of test pieces for the peel tests

5.4.3 Peel test under constant load and at a constant elevated temperature ("creep test")

Use the same test pieces as specified in 5.4.2

5.5 Number of test pieces

Prepare for every test and storage condition chosen at least three peel test pieces, at least 15 peel test pieces for the preliminary creep tests necessary for assessing the test temperature (five for each preliminary test) and at least 15 for the final creep tests (five for the creep test at each chosen test weight).

5.6 Preparation of test pieces

5.6.1 General

Condition all leathers and footwear materials to an equilibrium state in the standard atmosphere 23/50 according to ISO 554.

5.6.2 Cutting of material strips

When cutting the materials for test pieces, the surface that is to be bonded shall be uppermost. The inner surface of the knife shall be at right angles to the plane of the material to be cut. The depth of the cut shall be greater than the thickness of the material being cut.

NOTE: It is recommended that a thick sheet of paper be laid between the material and the cutting block.

5.6.3 Preparation of bonding surfaces

5.6.3.1 General

Prepare the bonding surfaces of the footwear materials for the test pieces in accordance with the instructions for the use of the adhesive. If there is no information available apply the procedures given in 5.6.3.2 to 5.6.3.4 appropriate to the type of material.

5.6.3.2 Roughing

Completely remove all loose fibres from the inner (flesh) surface of outsole leathers and reduce to thickness of use by splitting with a splitting machine (5.3.3). Then rough the inner surface by a roughing machine (5.3.4) to produce an even, velvet like surface free of loose fibres.

Rough upper leathers so that the finish and grain layer is completely removed and an even surface is produced (see Figure 2).

Rough synthetic upper materials comprising a coating supported by a woven or nonwoven material into the surface of the support or into the cellular layer if present; poromeric upper materials with a fabric interlayer into this layer; poromerics upper materials without a fabric interlayer into the cellular structure of the material (see Figure 2).

5.6.3.3 Scouring

Scour soling materials, e.g. vulcanised natural or synthetic rubber, crepe rubber and materials based on polyurethane, polyamide or ethylene vinyl acetate copolymers, in the testing direction with a scouring machine (5.3.5) in such a manner that their bonding surfaces are completely clean and even.

Before bonding with poly- chloroprene adhesives remove PVC coats from PVC upper materials carrying thin PVC coats with a hard wool felt disc (5.3.6).

Ensure that the emery cloth or paper is always in a good condition. After scouring remove the dust from the material strips by brushing in the testing direction by a suitable brush (5.3.7).

5.6.3.4 Cleaning

Clean material surfaces which will be bonded without a preceding mechanical treatment with a cloth or cotton wool (5.3.8) and a suitable solvent (5.3.9) so that they are free from release agents, impurities and/or ingredients which may have migrated from the material to its surface.

Clean PVC sole or upper materials with ethyl acetate or acetone, thermoplastic rubber sole materials with light petroleum. Renew cloth (or cotton wool) and solvent after cleaning 10 bonding surfaces.

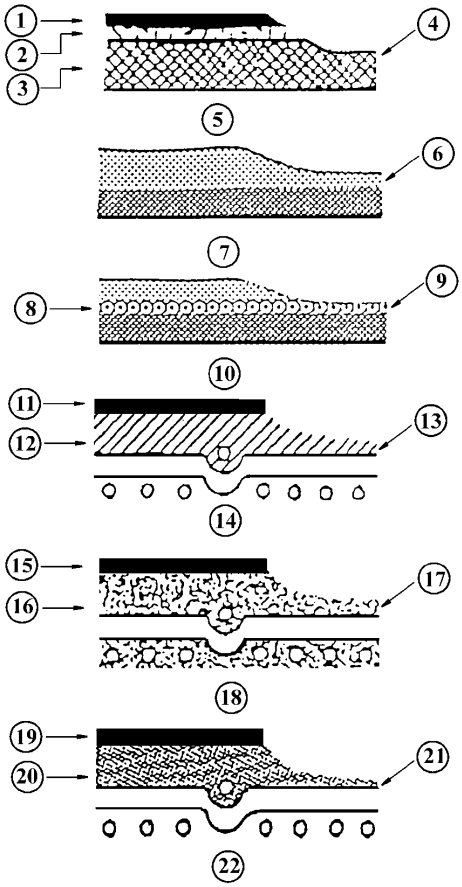
5.6.3.5 Halogenation

Usually halogenation is used before application of polyurethane adhesives. The halogenation agent (5.3.10) shall be applied in accordance with the instruction of the supplier.

Scrub the halogenation agent vigorously with a hard brush (5.3.11) onto surfaces of vulcanised rubber taking care that even and complete wetting is achieved.

However, apply the halogenation agent on surfaces of thermoplastic rubber materials with a soft brush (5.3.11) taking care to achieve even and complete wetting, with gentle brushing to avoid damaging the material.

Use a fresh portion of halogenation agent after coating each 10 surfaces. Before applying the adhesive, allow the solvents to evaporate and to complete the reaction. Unless otherwise specified and recorded in the test report, store for these purposes the halogenated surfaces open for at least (30 ± 5) min at a controlled temperature of (23 ± 2) °C and a relative humidity of less than 70 %.



Key

1 Finish
2 Grain
3 Corium
4 Rough to here
5 LEATHER UPPERS
6 Rough to here
7 POROMERIC UPPERS
8 Woven or non-woven interlayer
9 Rough to here
10 POROMERIC UPPERS WITH INTERLAYER
11 PU coating
12 Raised fabric
13 Rough to here
14 POLYURETHANE TRANSFER COATED FABRICS
15 PU coating
16 Coagulated PU
17 Rough to here
18 COAGULATED POLYURETHANE COATED FABRICS
19 PU coating
20 Cellular PU
21 Rough to here
22 CELLULAR POLYURETHANE COATED FABRICS

Figure 2 — Roughing and scouring of different upper materials

5.6.4 Preparation of the adhesive

Take according to EN ISO 15605 a significant sample of the adhesive to be tested and examine and prepare it according to EN 1067. Store the sample in a closed container at (23 ± 2) °C until it has completely reached this temperature.

The same requirement applies to all parts of a two or multi-part adhesive. In case of a two or multi-part adhesive then mix all parts of the adhesive before use in the specified ratio by vigorous stirring until the mixture is fully homogeneous.

5.6.5 Application of the adhesive

Unless otherwise specified and recorded in the test report:

Before applying the adhesive store the material strips for the times specified in 5.6.3.5 or otherwise for (30 ± 5) min after the preparation of the surface to be bonded (see 5.6.3) at a controlled temperature of (23 ± 2) °C and a relative humidity of less than 70 %. Depending on the absorptive capacity of the material apply one or more coats of adhesive in the length direction of the strips, using an appropriate device (5.3.12).

When assembling the material strips to form test pieces for peel and creep tests insert strips of paper so that after pressing, the ends of the strips can be bent apart for insertion into the tensile testing machine. Mark on the test pieces the first and the last 5 mm of the bond to be separated.

If several adhesive coats are applied, for example on absorbent materials, a drying interval of (30 ± 5) min shall be allowed, unless otherwise specified by the adhesive manufacturer.

Record the number of coats, and the time between adhesive application and bonding in the test report.

5.6.6 Drying for cold contact bonding

Unless otherwise specified and recorded in the test report:

Apply in contact bonding between the application of the adhesive and the assembling of the bond a drying time of (30 ± 5) min at a controlled temperature of (23 ± 2) °C and a relative humidity of less than 70 % to allow the solvents to evaporate.

5.6.7 Heat activation

Unless otherwise specified and recorded in the test report:

Store the bonding surfaces coated with the adhesive to be tested for (30 ± 5) min at a controlled temperature of (23 ± 2) °C and a relative humidity of less than 70 % until the solvent has evaporated.

Activate the adhesive coats on strips of both materials to be tested using a heat activator (5.3.13). Ensure that the required optimum activation temperature for the adhesive coats, usually (85 ± 5) °C, is reached within 15 s.

If the optimum activation temperature which ensures a full coalescence of the adhesive coat within the pressing time is unknown, preliminary tests shall be carried out according to EN 12961.

Determine the surface temperature of the adhesive coat on a spare strip using any of the items in 5.3.14. Record the activation temperature measured in the test report.

5.6.8 Assembling and pressing of test pieces

Unless otherwise specified and recorded in the test report:

For contact bonding (see 5.6.6) assemble both coats after drying as specified. Activated coats have to be assembled immediately after removal from the heat activator. Carry out all operations from the end of activation to attainment of maximum pressure within 10 s.

Then hold the test piece under maximum pressure for 15 s. Ensure that the pressure is precise and evenly distributed over the whole surface. The effective value of pressure in the adhesive joint shall depend on the material under test as follows:

for outsole leather	(0,6 ± 0,02) MPa;
for all other soling materials	(0,4 ± 0,02) MPa.

NOTE For soling materials with less than Shore A hardness 75, depending on hardness, a lower pressure may be applied but at least (0,2 ± 0,02) MPa.

5.7 Storage of test pieces

Carry out as specified immediately or after storing of the test pieces under specified conditions (e.g for a specified time in the standard atmosphere of 23/50 according to ISO 554) the separation tests described in 5.8.

5.8 Procedures and evaluation

5.8.1 Peel resistance at (23 ± 2) °C

For measuring the peel resistance bend the unbonded ends of the test pieces over to either side and clamp them (see Figure 3) for a distance of (20 ± 2) mm in the jaws of the tensile testing machine (5.3.16).

Dimensions in mm

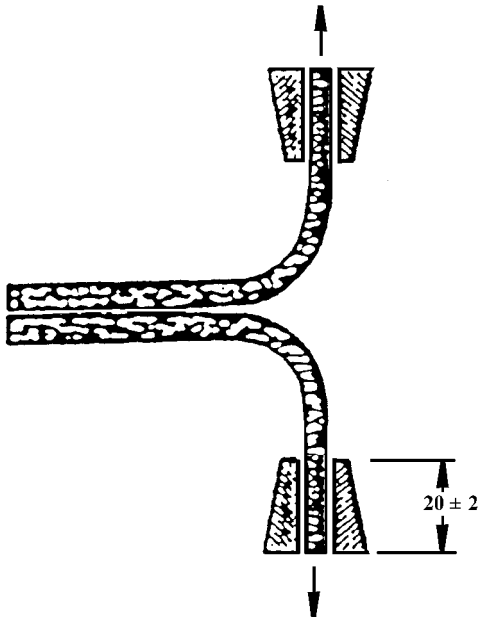


Figure 3 — Clamping of test pieces for the peel tests

Then pull the ends apart at a speed of (100 ± 10) mm/min. The temperature of the test piece during testing shall be (23 ± 2) °C.

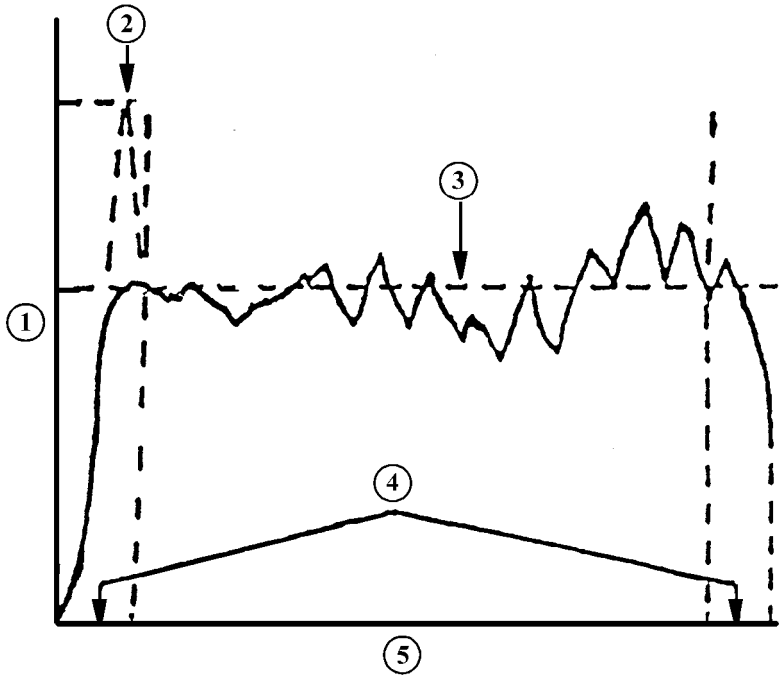
When determining the initial peel strength start the tensile testing machine so that also effective values immediately after assembling can be read.

NOTE For best accuracy choose a force range, if possible, such that the separation force is in the middle of the range.

Peel resistance is the mean peel force per unit width, in N/mm, calculated from the trace over the course of separation.

$$\text{Peel resistence} = \frac{\text{mean peel force in newtons during separation}}{\text{width of the test piece in millimeters}}$$

Obtain the mean peel force by means of a computer integrated in or connected with the tensile testing machine calculating the mean peel force in conformity with the following graphical method, which may be, used instead of a computer aided calculation, if necessary: lay a transparent plate with the length axis drawn on it, onto the curve in such a way that the straight line is at right angles to the axis of the peel force and move it till the areas above and below the straight line are equal (see Figure 4).



Key

1 peel force, in newtons

2 initial peak value of peel force

3 mean peel force value after initial peak (if any)

4 first and last 5 mm are ignored

5 peel length of bond, in millimetres

Figure 4 — Determination of mean peel force

In determining the mean peel force ignore the first and the last 5 mm of the peel length. A statement about the regularity of the peel trace is necessary for the correct assessment of the bond, with corresponding explanations in the case of irregular curves. Test the other four test pieces prepared for this test in the same way.

Record in the test report the individual values of peel resistance and their mean in accordance with ISO 2602 and the main failure pattern in accordance with EN ISO 10365.

If the test piece consisted of two different materials, record in case of material cohesion failure (CSF) the material which failed.

5.8.2 Peel test under constant load and at a constant elevated temperature ("creep test")

5.8.2.1 Test procedure with specified test conditions (e.g. for quality control or determination of specified minimum requirements)

Heat the warm air cabinet (5.3.18) to the temperature specified.

Carefully bend apart the unbonded ends of five test pieces, mark the beginning of the bonds, and insert the ends in the clamps of the cabinet. Then heat the test pieces in the cabinet for 1 h to allow them to reach the temperature specified.

After this heating up period load each of the five test pieces for 10 min with the specified constant weight. Finally open the warm air cabinet and mark the separations of the bonds while still loaded. Remove the weights, take out the test pieces from the cabinet, and measure the length of separation to the nearest 1 mm. In the case of a complete separation of 50 mm and more also determine the time (in minutes) from fixing the weights to complete separation.

Record in the test report the individual values and the mean value of the separation length. In calculating the mean of the separation lengths of a bond ignore the highest and the lowest value. In the case of a separation length of 50 mm or more note in the test report after the statement "50 mm" the period of time (in minutes) elapsed between loading with the weight and the separation of 50 mm. In addition record in the test report the main failure pattern in accordance with EN ISO 10365.

5.8.2.2 Test procedure with non-specified test conditions (e.g. for development work)

If a test temperature is not specified a suitable temperature giving a satisfactory separation length of test pieces in the creep test has to be determined by preliminary tests.

After a preheating period of 1 h at a medium test temperature (e.g. 50 °C) load at the same time five test pieces for 10 min with different weights, i.e. with 0,5 kg; 1,0 kg; 1,5 kg; 2,0 kg and 2,5 kg.

If all bonds separate at this temperature by more than 50 mm, repeat the same test at a temperature 10 °C lower and note whether a separation of less than 50 mm is obtained with one or more weights.

If however at the initially selected temperature no separation of the bond, or a separation of less than 2 mm is obtained continue the test series at higher temperatures, by 10 °C in each case, until a separation length of the bond of more than 2 mm but less than 50 mm results with one or more weights.

It is useful to list all test values as shown in Table 1.

Table 1 — Evaluation of creep test.

Temperature °C	Weight kg	Test piece 1	Test piece 2	Test piece 3	Test piece 4	Test piece 5	Mean value mm
40	0,5 1,0 1,5 2,0 2,5						
50	0,5 1,0 1,5 2,0 2,5						
60	0,5 1,0 1,5 2,0 2,5						
70	0,5 1,0 1,5 2,0 2,5						etc.

For the final determination of separation length of the bond at the selected test temperature and load carry out test as described in Clause 5.8.2.1 using five test pieces in each case.

Record in the test report the individual values and the mean value of the separation length. In calculating the mean of the separation lengths of a bond ignore the highest and the lowest value. In the case of a separation length of 50 mm or more note in the test report after the statement "50 mm" the period of time (in minutes) elapsed between loading with the weight and the separation of 50 mm. In addition record in the test report the main failure pattern in accordance with EN ISO 10365.

5.9 Test report

The test report shall include:

a) reference to this European Standard;

b) complete identification of the adhesive used or the designation of the footwear test adhesive according to 5.2.1. Record the designation of reference test adhesive, if used, and its source and date of supply;

c) complete identification of the leather and footwear material(s) used for the preparation of the test pieces or the designation of the footwear test material according to 5.2.2. Record the designation of reference footwear test material(s), if used, and its (their) source and date of supply;

d) preparation of the bonding surfaces of the test material(s), according to 5.6, identify halogenation agent, if used (1- or 2-part and solvent, if known);

e) complete identification of the bonding procedure applied (number of adhesive coats applied, length of intermediate drying periods, contact or heat activation bonding, activation temperature of the adhesive coats etc.) according to 5.6;

f) time and condition of storage of the test pieces according to 5.7;

g) types of tests carried out according to 5.8.1 and 5.8.2;

h) test results, individual and mean values and mode of failure according to 5.8.1 and 5.8.2;

i) any modification of the procedures described, and any incident which may have affected the results;

j) date of the test.

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