

Adhesives for leather and footwear materials — Preparation of bonded test pieces by moulding-on processes

ICS 61.060; 83.180

National foreword

This British Standard is the UK implementation of EN 14294:2010. It supersedes BS EN 14294:2004 which is withdrawn.

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A list of organizations represented on this committee can be obtained on request to its secretary.

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Adhesives for leather and footwear materials - Preparation of bonded test pieces by moulding-on processes

Adhésifs pour cuir et matériaux de la chaussure -
Préparation d'éprouvettes collées par moulage direct sur
tige

Klebstoffe für Leder und Schuhwerkstoffe - Herstellung von
Verbund-Proben nach dem Anformverfahren

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Foreword

This document (EN 14294:2010) 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of an identical text or by endorsement, at the latest by August 2010, and conflicting national standards shall be withdrawn at the latest by August 2010.

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This document supersedes EN 14294:2004.

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Introduction

EN 15307 and EN 1392 specify methods of testing and evaluation of adhesives for leather and footwear materials in "stuck-on" assemblies. Most footwear produced in Europe is manufactured using stuck-on processes, but a significant proportion is produced by "moulding-on" processes such as vulcanising, injection moulding and reaction moulding.

This document specifies the preparation of bonded test pieces by moulding-on processes. The form and dimensions of the test pieces produced allows testing according to the test methods specified in EN 1392, and evaluation of the bond strength obtained according to EN 15307.

This document therefore complements the above mentioned series of documents, and together they allow the simulation, testing and evaluation of almost all bonds occurring in common footwear constructions.

1 Scope

This European Standard specifies procedures for the preparation of test pieces comprising adhesive coated leather or other footwear upper material onto which a soling material is moulded directly. The procedures described simulate direct vulcanising of rubber, injection moulding of thermoplastics and reaction moulding of polyurethane.

The prepared test pieces are suitable for the test procedures described in EN 1392, to meet the requirements of EN 15307.

SAFETY STATEMENT— Persons using this document should be familiar with the normal laboratory practice, if applicable. This document does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user to establish appropriate safety and health practices and to ensure compliance with any regulatory conditions.

ENVIRONMENTAL STATEMENT— It is understood that some of the material permitted in this standard may have negative environmental impact. As technological advantages lead to acceptable alternatives for these materials, they will be eliminated from this standard to the extent possible.

At the end of the test, the user of the standard should take care to carry out an appropriate disposal of the wastes, according to local regulation.

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 1392:2006, *Adhesives for leather and footwear materials — Solvent-based and dispersion adhesives — Testing of bond strength under specified conditions*

EN 15307, *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)*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in EN 923:2005 and EN 1392:2006 and the following apply.

3.1

moulding-on process

process for soling footwear in which the soling material is directly moulded onto the bottom of the shoe in a mould closed by the shoe upper or other footwear component

NOTE The other footwear component can be an outsole or midsole onto which another soling layer is to be moulded.

3.2

vulcanising

moulding process in which uncured rubber is inserted or extruded into the mould and is shaped and crosslinked by heat and pressure

3.3 injection moulding

moulding process in which molten thermoplastic material is injected into the mould and sets on cooling

3.4 reaction moulding

moulding process in which reactive chemicals are poured or injected into the mould and polymerise and crosslink by chemical reaction

4 Procedure

The surface of the leather, other footwear upper material or other footwear component material used is treated in a manner specific to the nature of the material. Strips of specified length and width are cut from the treated material.

One or more of these strips are coated with adhesive and attached, with the bonding surface uppermost, to the bottom plate of a mould. Soling material is introduced to fill the mould and allowed to cure or set as appropriate to produce an assembly of soling bonded to upper material.

The moulded assembly, and test pieces cut from it, are stored under specified conditions before testing.

5 Adhesives and materials

5.1 Footwear adhesives

The adhesive used shall be identified in the report, in particular note name and/or designation, manufacturer, date of manufacture or supply and/or lot number, main polymer and colour. For two-part adhesives the nature of the crosslinking agent and the mixing ratio of the components shall be identified.

Reference footwear test adhesives are one- or two-part adhesives with specified properties (e.g. Test adhesive CR 1, Test adhesive CR 2, Test adhesive PU 1, Test adhesive PU 2, Test adhesive PUD 1, Test adhesive PUD 2). If a reference footwear test adhesive in accordance with EN 15307 is used, record its designation in the report.

NOTE For direct vulcanising processes, and injection moulding processes with styrene-butadiene-styrene thermoplastic rubber (SBSR), special types of adhesive are required.

5.2 Footwear materials

5.2.1 General

Identify all footwear materials used in the report.

5.2.2 Upper materials (or other footwear materials)

Record the name and/or designation, manufacturer, date of manufacture or supply and type of leather, other footwear upper material (or other footwear material) used. For leathers list colour, thickness and type of tannage (if known). If reference test material Leather 1 is used in accordance with EN 15307, record its designation.

5.2.3 Soling moulding materials

5.2.3.1 General

The soling moulding material required depends on the moulding-on process to be simulated.

5.2.3.2 Direct vulcanising materials

Record the name and/or designation, manufacturer, date of manufacture or supply, colour, polymer base and Shore hardness in accordance with EN ISO 868 of the material used.

NOTE Uncured compounds of acrylonitrile-butadiene (nitrile) rubber (NBR) or styrene-butadiene rubber (SBR) are mainly used.

5.2.3.3 Injection moulding materials

Record the name and/or designation, manufacturer, date of manufacture or supply, colour, polymer base and Shore hardness in accordance with EN ISO 868 of the material used.

NOTE PVC compounds or styrene-butadiene-styrene thermoplastic rubber (SBSR) compounds are mainly used.

5.2.3.4 Reaction moulding materials

Record the name and/or designation, manufacturer, date of manufacture or supply, colour, chemical nature of the reactive constituents and the catalysts used, if any.

NOTE Polyurethane chemicals are mainly used, consisting of a prepolymer and a resin which are mixed in the appropriate proportions before moulding. If a catalyst is also required, this can be added to the resin prior to the weighing out of the constituents.

6 Apparatus

6.1 General

The items required depend on the types of material used and the type of moulding-on process.

6.2 Cutting knife, sharp for cutting test pieces in accordance with 7.1. The angle between the inner and outer cutting surface shall be approximately 20°.

6.3 Adhesive applicator, brush, roller or coating machine for applying a uniform coat of the adhesive under test.

6.4 Platen press, a heated platen press suitable for direct vulcanisation of rubber, capable of:

- a) maintaining platen temperatures of (105 ± 5) °C for the bottom platen and (180 ± 5) °C for the top platen;
- b) applying a pressure up to 5 MPa to the mould.

6.5 Rectangular mould, of approximate dimensions 150 mm × 100 mm × 5 mm for direct vulcanisation. The wall of the mould base should include a 10 mm wide shelf all round, half way up, to accept excess rubber displaced from the cavity.

6.6 Surface pyrometer, suitable for measuring the operating temperatures of the press platens.

6.7 Oven, capable of maintaining a temperature of (75 ± 5) °C to pre-heat the rubber prior to moulding.

6.8 Gloves, heat resistant, suitable for handling the hot mould.

6.9 Injection moulding machine, fitted with a mould of approximate dimensions 150 mm × 100 mm × 5 mm for the preparation of flat test plaques. A commercial single station low pressure screw injection moulder is suitable.

6.10 Mould, for polyurethane reaction moulding, having:

- a) flat rigid base with a rectangular cavity of approximate dimensions 300 mm × 200 mm × 6,5 mm;

- b) rigid hinged lid which can be held in place, for example by two wing nuts;
- c) rebate a few millimetres deep across the two corners of the base away from the hinge. This provides a gap which can assist in opening of the mould.

6.11 Oven, capable of maintaining a temperature of (50 ± 5) °C to heat the mould prior to moulding.

6.12 Mould release agent, suitable for polyurethanes.

6.13 Solvent, such as 2-butanone (MEK), suitable for removing mould release agent.

6.14 General purpose balance, reading up to 300 g with an accuracy of 0,1 g.

6.15 Metal mixing cans, of approximate volume 400 cm³ capable of holding about 300 g of the polyurethane constituents in liquid form.

6.16 Paper weighing cups, of approximate volume 200 cm³, to weigh the polyurethane prepolymer prior to mixing.

6.17 High speed stirrer, suitable for mixing the viscous polyurethane constituents in a metal can. A stirrer with a mixing head comprising a horizontal spoked metal wheel with rectangular teeth around the edge is suitable.

6.18 Polyethylene gloves.

6.19 Safety goggles.

6.20 Adhesive tape, double side adhesive tape.

7 Test pieces

7.1 General

The form and dimensions of the test pieces prepared from the moulded-on assemblies shall comply with the requirements for test pieces for peel tests at (23 ± 2) °C, and test pieces for peel tests under constant load and at a constant elevated temperature ("creep test") in EN 1392.

7.2 Number of test pieces

Prepare sets of test pieces in accordance with EN 1392.

7.3 Preparation of test strips

7.3.1 General

Condition the footwear upper material in accordance with EN 1392.

7.3.2 Cutting of material strips

Cut strips of the required size as indicated in EN 1392 from the upper material. Alternatively, if the material does not allow the cutting of strips of the required dimensions, smaller and/or shorter strips can be used, subject to their dimensions and bonding length being recorded in the report.

7.3.3 Preparation of the bonding surface

Prepare the surface of the upper material in accordance with EN 1392.

In some cases upper materials are solvent wiped immediately before moulding on the sole material (see 8.2.3 and 8.3.4).

7.4 Preparation of the adhesive

Prepare the adhesive in accordance with EN 1392.

7.5 Application of the adhesive

Unless otherwise specified the application method of the adhesive shall be recorded in the report.

Before applying the adhesive, the upper material test strips shall be stored for (30 ± 5) min after the preparation of the surface to be bonded (see 7.3.3) at a controlled temperature of (23 ± 5) °C and a relative humidity of less than 70 %.

Apply self adhesive tape, somewhat wider than the strip, to each upper material test strip to define the non-bonded length. Wrap excess tape round the edges of the strip and stick it to the back. In the case of test strips for direct vulcanising assemblies use paper, attached by staples, instead of adhesive tape. Mark the first and last 5 mm of the bonded length to be separated.

Depending on the absorptive capacity of the material one or more coats of adhesive shall be applied in the length direction of the strips, using an appropriate device (6.3). 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.

Unless otherwise specified store the bonding surfaces coated with adhesive for (30 ± 5) min at a controlled temperature of (23 ± 5) °C and a relative humidity of less than 70 % until the solvent has evaporated.

NOTE For thermoplastic injection moulding process a shorter drying time is often used, for instance 2 min or 5 min.

The number of adhesive coats, and the time between adhesive application and sole moulding, shall be recorded in the report.

8 Preparation of moulded-on assemblies

8.1 Rubber direct vulcanised assemblies

8.1.1 General

The procedure described in this section simulates the method most commonly used in the shoe factory for the production of footwear with solid rubber vulcanised moulded-on soles. The rubber, initially in the form of a flat "blank", is vulcanised onto upper material test strips, usually coated with adhesive, in a flat mould (6.5) inserted between the heated platens of a laboratory press (6.4).

NOTE Stocks of unvulcanised rubber sheet should always be stored in a refrigerator at a temperature below 10 °C to prevent premature curing.

8.1.2 Preparation of rubber

Remove some of the unvulcanised rubber sheet (5.2.3.2) from the refrigerator and allow it to warm to room temperature, for at least 30 min.

Cut from it a piece or pieces that will be sufficient to completely fill the mould cavity (6.5) after moulding, normally about 120 g for a cavity of 75 cm³.

Ensure that the cut pieces of rubber can be fitted easily into the base part of the mould (6.5) and that each of the upper material test strips can be covered with approximately equal amounts of rubber.

8.1.3 Preparation of press and mould

Set the thermostat for the bottom platen of the press (6.4) to (105 ± 5) °C and the thermostat for the top platen to (180 ± 5) °C, unless different temperatures are specified by the rubber manufacturer or are appropriate to the tests being done.

Place the lid on the empty mould base and insert the mould (6.5) into the press. Close the press until its upper platen just touches the lid of the mould.

Heat the press until its two platens reach the required temperatures, normally about 45 min when heated from room temperature. Check that the correct temperatures have been achieved using the surface pyrometer (6.6) with the probe held in contact with the surface of the platens adjacent to the mould (6.5).

When the press is at the required temperatures, heat a cut and weighed sample of unvulcanised rubber in the oven (5.7) at (75 ± 5) °C for 10 min, unless a different temperature has been recommended by the rubber supplier. Remove the heated mould from the press wearing the heat resistant gloves (6.8) and lift off its lid.

8.1.4 Moulding

Place one or more upper material test strips symmetrically in the base of the mould (6.5) so that the paper strips previously applied to each are uppermost. Cover the upper material test specimens with the weighed and heated pieces of the rubber and replace the mould lid. Reinsert the mould into the press.

NOTE The loading of the moulds should be carried out as quickly as possible to minimise cooling.

Apply a force to the mould sufficient to give a pressure of 3,5 MPa to 4,2 MPa. After a few seconds the rubber will start to compress, which with hydraulic and mechanical presses will cause the force to drop. When this happens increase the force, as necessary, to keep it in the required range. Maintain the force for 10 min, unless otherwise recommended by the rubber manufacturer.

If further assemblies are to be moulded, place the next cut and weighed sample of unvulcanised rubber in the oven (6.7) at (75 ± 5) °C as soon as the previous sample is in the moulding press.

Release the pressure and remove the mould from the press wearing the heat resistant gloves (6.8). Lever off the mould lid and remove the moulded rubber without waiting for it to cool. This is best achieved by inserting a blunt point under the material in the flash channel at one end so that it can be gripped, and pulling the moulding out of the base mould. This shall be done very carefully to avoid damaging the bond by disturbing the adhesive.

Repeat the procedure for any remaining test strips.

When moulding has been completed, switch off the press platens and allow them to cool.

8.1.5 Storage of moulded assemblies

Leave the moulded assemblies for at least two days before cutting the bonded test pieces from them.

8.2 Thermoplastics injection-moulded assemblies

8.2.1 General

The procedure described in this section simulates the process used in the shoe factory for the production of footwear with thermoplastic moulded-on soles. The soling material is injected moulded onto the upper material test strips using an injection moulding machine. The upper material is usually adhesive coated, but to simulate some shoemaking processes test assemblies may need to be prepared with materials which are not adhesive coated,

although they may be solvent wiped immediately before moulding. In this case the moulded-on material acts as an adhesive.

8.2.2 Preparation of moulding machine

Set the temperature controls on the injection moulding machine (6.9) to the correct temperatures for the compound to be moulded.

Set the "in-mould" cooling time after injection to approximately 90 s.

Before the test assemblies can be prepared, the moulding machine shall be purged of the remains of the old compound in it, by moulding a number of "blanks" with the compound (5.2.3.3) that is to be used for the test assemblies. Operate the machine and commence the moulding of the blanks. It is normally necessary to mould about five blanks to purge the machine if the compound is the same as previously moulded, or up to about 15 blanks if the compound is different from that previously moulded. Once the mouldings are satisfactory – of uniform appearance and not containing any visible remains of the previous compound – injecting onto the prepared upper material can begin.

8.2.3 Moulding

Withdraw the bottom plate of the mould (6.5) from its moulding position. Attach one or more upper material test strips to the surface of the bottom plate with double-sided adhesive tape, so that the side to be bonded is uppermost.

The test strips should be positioned so that the tape strip previously applied to define the non-bonding length is towards the injection point, the nearest test strip is at least 30 mm from the injection point and the test specimens are at least 10 mm apart.

It is important to ensure that the upper material test strips are stuck to the base plate right up to their edges to prevent the compound getting underneath them.

When a material, such as a PVC coated fabric, is to be solvent wiped and then bonded after a short drying time without the application of any adhesive, attach it to the bottom plate of the mould. Then solvent wipe the surface and leave for the required time, for example 30 s, before injecting on the soling.

Return the bottom plate of the mould to the moulding position and inject the soling compound onto the upper material. When the mould opens, withdraw the bottom plate and remove the moulding. This shall be done very carefully to avoid damaging the bond, which will still be warm.

Repeat the procedure for any remaining test strips.

When moulding has been completed, purge and shut down the moulding machine using the procedure recommended by the material or machine manufacturer.

8.2.4 Storage of moulded assemblies

Leave the moulded assemblies for at least two days before cutting the bonded test pieces from them.

8.3 Polyurethane reaction moulded assemblies

8.3.1 General

The procedure described in this section simulates the method most commonly used in the shoe factory for the production of footwear with reaction moulded-on polyurethane solings. A premixed polyurethane compound is poured into a heated mould (6.10) containing the upper material test specimens. The upper material is usually not adhesive coated, but to simulate some shoemaking processes test assemblies may need to be prepared with materials which are adhesive coated, or simply solvent wiped immediately before moulding. The mould is then kept closed until the reaction is completed and the cured soling has bonded to the upper material.

Alternatively commercial equipment can be used for the reaction moulding of the polyurethane soling. If this is done it will be necessary to tape together the samples to conform to the mould in the commercial equipment. Alternatively a large piece of material can be used, of sufficient size to cover the area of the mould.

8.3.2 Preparation of chemicals

According to the manufacturer's instructions concerning the proportions of prepolymer and resin to be used, containing added catalyst if required, calculate the weights of each that will be required to fill the mould (6.10).

Because it is not possible to pour all of the prepolymer from its weighing cup (6.16) into the mixing can (6.15), an allowance shall be made for the quantity of prepolymer that will remain in the weighing cap. This amount shall be determined by weighing a weighing cap before and after pouring the prepolymer from it.

Place the caps of polyurethane prepolymer and resin in the oven (6.11) at (50 ± 5) °C to melt overnight. If the resin does not already contain the catalyst, add the appropriate quantity of catalyst to the melted resin and mix it in thoroughly.

8.3.3 Preparation of mould

Spray liberally the upper surface of the mould base and the lower surface of the mould lid with mould release agent (6.12). Place the mould in the oven at (50 ± 5) °C and allow it to heat up for at least 2 h.

8.3.4 Moulding

Cover the reverse side of one or more upper material test strips with double-sided adhesive tape. The test strips should be positioned at least 10 mm apart and the double sided adhesive tape (6.20) should fill all the gaps between the test strips and extend beyond their outer edges, to prevent compound getting underneath them during moulding.

Remove the heated mould from the oven (6.11), and using the solvent (6.13), clean the mould release agent (6.12) from an area on the mould base which is the same size as the array of test strips including the double sided tape.

Firmly attach the test strips to the cleaned area of the mould base.

When a material, such as a coated fabric, is to be solvent wiped and then bonded after a short drying time without the application of any adhesive, solvent wipe it after attaching to the base of the mould. Then leave it for the required time (for example 30 s) before moulding on the soling.

Wearing polyethylene gloves (6.18) and safety goggles (6.19), use the balance (6.14) to weigh the required amount of resin (5.2.3.4) into a mixing can (6.15) and the required amount of prepolymer (5.2.3.4), including the allowance for pouring, into a weighing cup (6.16).

Without delay pour the prepolymer from the weighing cap into the resin in the mixing can and allow it to drain in for a total time of 5 s. Leave in the weighing cap any prepolymer that has not run out after this time.

Wearing heat resistant gloves (6.8) and safety goggles (6.19), immediately insert the mixing head of the stirrer (6.17) into the contents of the mixing can, switch on and mix thoroughly for 8 s.

NOTE 1 It is easier to do this by having the stirrer fixed on a stand and holding and moving the can by hand so that the mixer goes into all parts of the can bottom.

Immediately pour as much as possible of the contents of the can over the test strips in the mould so that they are largely covered. This should take about 3 s or 4 s but do not allow the mixture to drain for longer. Close the mould lid and tighten the wing nuts or other fastening fully. As the reaction proceeds a little excess material can be forced out between the mould base and the lid.

After at least 10 min open the mould. If the lid sticks because of excess material, carefully lever it open by inserting a screwdriver into the two gaps between it and the base at the corners. Remove the moulding from the base. Usually it is easiest to start at a corner, either levering it out with a screwdriver or pulling on sprue from between the base and the lid.

NOTE 2 There can be some sticking of the polyurethane to the base round the test specimen assembly but it does not matter if a little of the polyurethane here tears away and remains on the base. It should however be cleaned off fairly quickly either by rubbing with a finger or a piece of crepe rubber, or by using dichloromethane solvent.

Reweigh the weighing cap with the remains of the prepolymer that did not drain out. Calculate and record the amounts of resin and prepolymer actually mixed together and the amount of prepolymer left in the weighing cap. If it is found in practice that the weight of residue differs from the allowance made for wastage in the initial weighing, then change the allowance to a more appropriate value.

Repeat the procedure for any further test strips.

8.3.5 Storage of moulded assemblies

Store the moulded assemblies for at least two days at room temperature before cutting the bonded test pieces from them.

9 Test report

The report on the prepared test pieces shall include, at least, the following:

- a) reference to this document;
- b) complete identification of the adhesive used or the designation of a reference test adhesive in accordance with 5.1;
- c) complete identification of the upper material used for the preparation of the test pieces in accordance with 5.2.2;
- d) preparation of the bonding surface of the upper material used in accordance with 7.3.3;
- e) complete identification of the sole moulding material used in accordance with 5.2.3;
- f) type and manufacturer of moulding machine (6.9), where used;
- g) moulding temperature and, for vulcanising, the moulding pressure;
- h) any modification of the procedures described, and any incident which may have affected the results;
- i) date of moulding.

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