BS EN 1238:2011

BSI Standards Publication

Adhesives — Determination of the softening point of thermoplastic adhesives (ring and ball)

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National foreword

This British Standard is the UK implementation of EN 1238:2011. It supersedes BS EN 1238:1999 which is withdrawn.

The UK participation in its preparation was entrusted to Technical Committee PRI/52, Adhesives.

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

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English Version

Adhesives - Determination of the softening point of thermoplastic adhesives (ring and ball)

Adhésifs - Détermination du point de ramollissement des adhésifs thermoplastiques (méthode bille et anneau)

Klebstoffe - Bestimmung des Erweichungspunktes von thermoplastischen Klebstoffen (Ring und Kugel)

This European Standard was approved by CEN on 10 March 2011.

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Foreword

This document (EN 1238:2011) 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of an identical text or by endorsement, at the latest by October 2011, and conflicting national standards shall be withdrawn at the latest by October 2011.

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This document supersedes EN 1238:1999.

The main modifications regarding the previous version are in the Foreword, Normative References and Note in 5.8.

This European Standard includes Annex A (normative) "Thermometer specification".

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.

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Introduction

Thermoplastic adhesives do not change from the solid to the liquid state at a fixed temperature, but their viscosity decreases progressively as the temperature rises. For this reason, the determination of the softening point shall be carried out by defined methods to obtain comparable results.

1 Scope

This European Standard specifies a method for the determination of the softening point of hot-melt adhesives.

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+A1:2008, *Adhesives — Terms and definitions*

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

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

3 Terms and definitions

For the purposes of this document, the terms and definitions given in EN 923:2005+A1:2008 and the following apply.

3.1
softening point
temperature at which the adhesive under test attains a degree of softness under the specified conditions

4 Principle

A steel ball of a specified mass is placed on a sample of adhesive contained on a metal ring of specified dimensions. The apparatus is heated at a constant defined rate. The temperature at which the sample is sufficiently soft to allow the ball to pass through the ring by a fixed distance is taken as the "softening point".

5 Apparatus

5.1 **Ball**, two steel balls, (9,53 ± 0,02) mm in diameter, each having a mass of (3,50 ± 0,05) g.

5.2 **Ring,** tapered brass ring to the dimensions of Figure 1.

NOTE As alternative the following can be used:

a) shouldered brass ring to the dimensions of Figure 2;

b) a straight-sided cylindrical ring with the following:

   1) interior diameter (15,9 ± 0,1) mm;

   2) depth (6,4 ± 0,1) mm.

Dimensions in millimetres

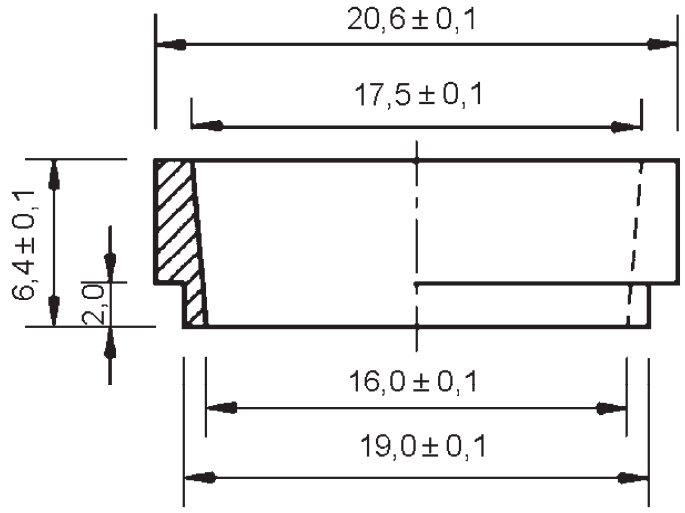


Figure 1 — Tapered brass ring

Dimensions in millimetres

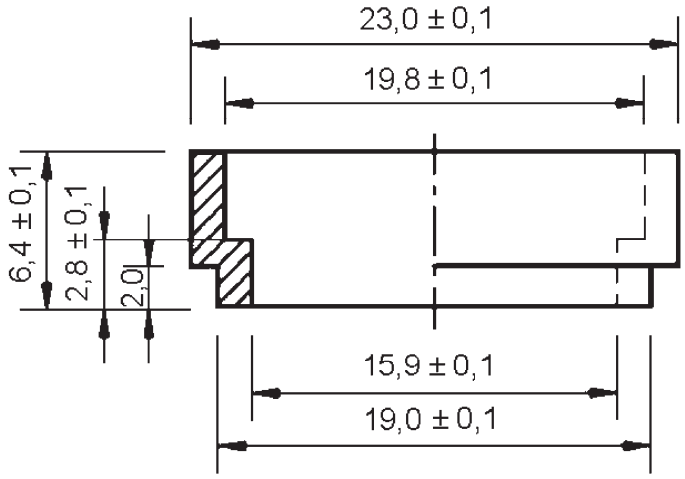


Figure 2 — Shouldered brass ring

To ensure the correct positions of the ring in its support, the exterior diameter shall be reduced at the lower part to (19,0 ± 0,1) mm for a depth of 2 mm.

If a shouldered or straight-sided ring is used, this shall be noted in the test report.

5.3 Guide, to center the ball, the arrangement in Figure 3 is recommended to be used.

Dimensions in millimetres

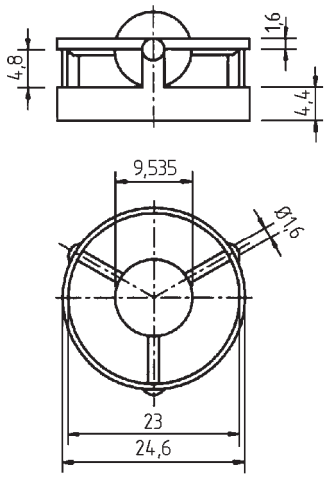
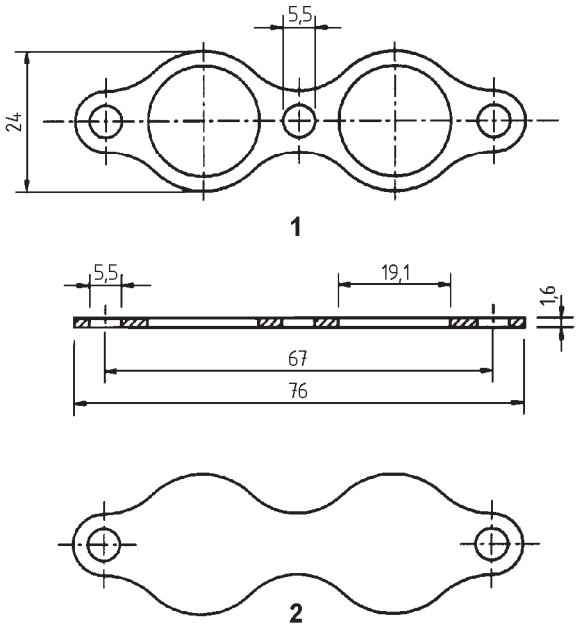


Figure 3 — Ball centering guide

5.4 Ring supports

The support described in Figure 4 shall be used. The distance between the two supports plates is (25 ± 1) mm. The same distance separates the ring holder from the bottom plate.

Dimensions in millimetres



Key

1 Ring holder

2 Base

Figure 4 — Ring holder and base

5.5 Thermometer, conforming to the specification of Annex A.

5.6 Bath, of heat resistant glass, to the dimensions of Figure 5, with the rings supported horizontally as shown.

The bottom of the bulb of the thermometer shall be level with the bottom of the rings and within 10 mm of them, but not touching them.

5.7 Stirrer, to ensure uniform heat distribution throughout the bath a mechanical stirrer which operates smoothly shall be used.

It shall be so placed that the samples are not disturbed when it is in operation.

5.8 Bath liquid

Water may be used to 80 °C. Glycerol may be used up to 200 °C. Above 200 °C a liquid heat-transfer medium shall be chosen which is stable at the temperatures likely to be used; mineral or silicone oils are suitable.

The liquid used shall not affect the specimen at the test temperature.

NOTE Automatic apparatus for measuring the softening point of thermoplastics adhesives is commercially available. Differences in the softening point of some hot melt adhesives (e.g. poly(ethylene-vinylacetate) based adhesives) measured by automatic or manual equipment can occur. In such case comparative tests should be carried out to determine whether it is necessary to apply a correction factor.

6 Sampling

A significant sample of the adhesive shall be taken in accordance with EN ISO 15605 and prepared for testing as described in EN 1067.

7 Preparation of sample

7.1 Preparation of test specimens in duplicate

Heat a portion of the sample in air or a liquid bath, to obtain an approximate value of the softening point. When this is noted continue until the temperature of the sample is between 25 °C and 50 °C above the softening point noted. Stir the sample during heating until it is completely fluid, homogeneous, and free from air bubbles. If necessary, pour the fluid sample through a 315 µm sieve, to remove solid impurities.

Heat the ring to the same temperature as the sample under test on a metal, porcelain or heat resistant glass plate, coating lightly with oil or silicone grease to prevent adhesive sticking.

Fill the ring with an adequate amount of fluid adhesive, so that a slight excess remains after cooling. Cool in air for 30 min removing excess adhesive with a heated knife.

7.2 For heat sensitive adhesives

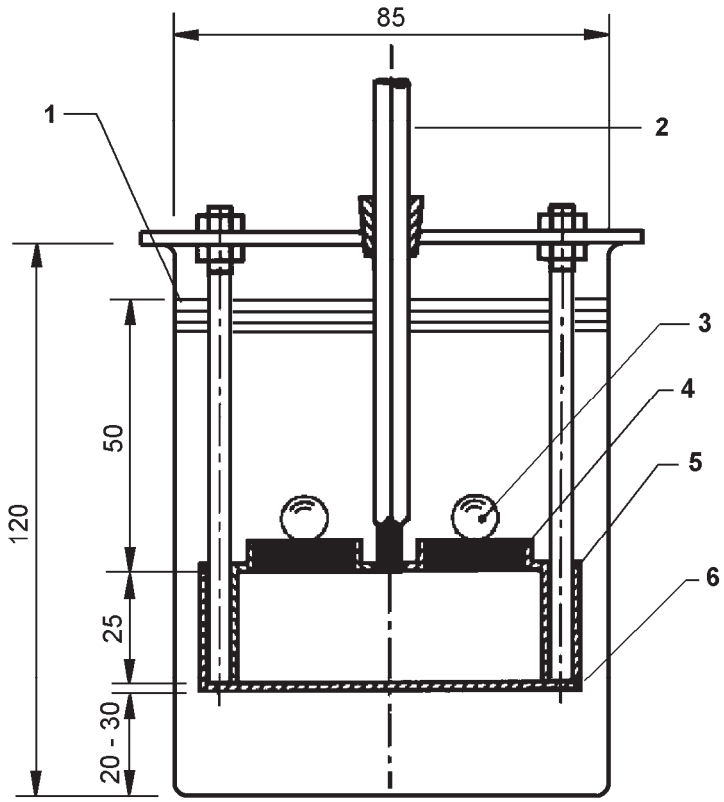
Carry out the above method in an inert atmosphere, or by another method agreed by the parties concerned.

NOTE If guides (see Figure 3) are not being used, a mark for the balls should be made in the middle of the prepared specimens, just deep enough to ensure that they cannot move and are centred in the rings.

8 Procedure

8.1 Prepare the apparatus as indicated in Figure 5, ensuring the rings are horizontal.

Dimensions in millimetres



Key

1	Liquid level	4	Shouldering ring, see Figure 2
2	Thermometer	5	Ring holder, see Figure 4
3	Steel ball	6	Base, see Figure 4

Figure 5 — Complete test assembly

8.2 Sample softening points expected to be below 80 °C.

a) Start from a temperature at least 10 °C below the expected softening point, using the liquid maintained at that temperature in a thermostat bath for at least 15 min. Using forceps, place the balls, previously kept at the same temperature in the same thermostat, into the guides.

b) Heat the apparatus and stir the liquid so that the temperature rises at a uniform rate of (5 ± 1) °C/min until the balls fall through the rings. Do not average the rate of temperature rise over the test period.

Reject any determination in which the rate of temperature rise does not fall within the specified limits after the first 3 min.

Record for each ring and ball the temperature shown by the thermometer at the instant the sample surrounding the ball touches the bottom plate.

8.3 Sample softening points to be above 80 °C.

Fill the bath with glycerol or oil as indicated in 5.8. Maintain the bath at 35 °C for 15 min and using forceps, place the balls, previously brought to 35 °C, in each ball guide.

Follow the procedure of 8.2, b).

9 Expression of results

As softening point report the mean of the temperatures recorded in duplicate determinations, without correction of the emergent stem of the thermometer, rounded to the nearest 0,5 °C if the recorded temperature is 100 °C or below; 1 °C if the recorded temperature is between 100 °C and 260 °C, and 2 °C if the recorded temperature is above 260 °C.

10 Test report

The test report shall include:

a) reference to this European Standard, i.e EN 1238;

b) type and designation of the adhesive tested;

c) type of ring used, if the tapered one (see Figure 1) is not used;

d) type of ring heating fluid used;

e) use or not of inert atmosphere;

f) test temperature;

g) result of the test expressed in accordance with Clause 9;

h) any circumstances which may have affected the result;

i) date of the test.

Annex A
(normative)

Thermometer specification

Table A.1 — Thermometer specification

Characteristics	Low range	Medium range	High range
Immersion	Partial	Partial	Partial
Temperature scale	- 1 °C to 101 °C	- 10 °C to 260 °C	- 10 °C to 400 °C
Subdivisions	0,1 °C	1 °C	2 °C
Max. error (at all points of the scale)	1 °C	1 °C to 100 °C 1,5 °C above 100 °C	2,5 °C to 300 °C 5 °C above 300 °C
Min. scale (mm length)	500	250	250
Diameter (mm)	5,5 to 8	5,5 to 8	5,5 to 8

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