

BS EN 1239:2011



BSI Standards Publication

Adhesives — Freeze-thaw stability

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National foreword

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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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ISBN 978 0 580 72243 1

ICS 83.180

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This British Standard was published under the authority of the Standards Policy and Strategy Committee on 31 May 2011.

Amendments issued since publication

Date	Text affected
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English Version

Adhesives - Freeze-thaw stability

Adhésifs - Stabilité au gel-dégel

Klebstoffe - Gefrier-Auftau-Stabilität

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

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Foreword

This document (EN 1239:2011) 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of an identical text or by endorsement, at the latest by October 2011, and conflicting national standards shall be withdrawn at the latest by October 2011.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. CEN [and/or CENELEC] shall not be held responsible for identifying any or all such patent rights.

This document supersedes EN 1239:1998.

The main modifications regarding the previous version are in the Normative References and 8.1.

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1 Scope

This European Standard specifies a method for the evaluation of the freeze-thaw stability of adhesives, their basic constituents and related products.

This test has no significance if the sample does not freeze under the test conditions.

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 12092, *Adhesives — Determination of viscosity*

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 apply.

4 Principle

Measure the viscosity of a sample of the adhesive being tested by placing it in a test chamber for 16 h at a selected temperature of - 5 °C, - 10 °C, - 15 °C or - 20 °C, then holding it for 8 h at (23 ± 1) °C and finally checking the aspect of the adhesive.

If no coagulum is formed, the cycle is repeated, i.e. the sample is stored at the selected temperature for another 16 h and then held at (23 ± 1) °C for 8 h up to a maximum number of three cycles. The final viscosity of the adhesive is measured.

Freeze-thaw cycle stability is indicated by the selected test temperature and the number of cycles endured without visible coagulation at an increase of viscosity not exceeding the manufacturer's specification limits.

NOTE 1 The freezing temperature selected depends on the chemical nature of the adhesive to be tested and the temperature conditions to which the adhesive is exposed in service. If necessary, preliminary tests should be carried out in order to determine the appropriate test temperature.

NOTE 2 The results obtained by this method are not necessarily applicable to larger-volume vessels.

5 Apparatus

5.1 Cylindrical container, 90 mm height, 60 mm inside diameter and 2 mm wall thickness with a large opening on the top and capable of being sealed with an airtight lid.

NOTE At test temperatures of - 5 °C or - 10 °C the container can be made of polyethylene; at lower freezing temperatures cylindrical stainless steel container can be used.

5.2 Test chamber, a cabinet, room or enclosure space large enough to contain the samples to be tested allowing at least 25 mm of air space between the sides of adjacent containers and capable of being controlled at temperatures of $(- 5 \pm 1) ^\circ\text{C}$, $(- 10 \pm 1) ^\circ\text{C}$, $(- 15 \pm 1) ^\circ\text{C}$, and $(- 20 \pm 1) ^\circ\text{C}$, respectively.

5.3 Thermometer, allowing measurement of the temperature inside the test chamber to be $\pm 0,5 ^\circ\text{C}$.

5.4 Viscometer, in accordance with EN 12092 to determine the viscosity of the adhesive to be tested.

6 Sampling, examination and preparation of samples

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 Procedure

7.1 Measure the viscosity of the sample at $(23 \pm 1) ^\circ\text{C}$ in accordance with EN 12092 using a suitable viscometer (see 5.4) and note the result.

7.2 Put (200 ± 5) ml of the test sample into the cylindrical container (see 5.1).

7.3 Seal the container airtight and place it for 16 h in the test chamber (see 5.2) set at the selected freezing temperature.

7.4 Remove the container from the test chamber and let it thaw at $(23 \pm 1) ^\circ\text{C}$ for 8 h.

7.5 Check by insertion of a glass rod the condition of the sample (homogeneous, water layer on its top, thickened, irreversible gelation, e.g. by formation of elastic, rubbery materials) and note the result.

NOTE If visible coagulation is formed that cannot be dispersed by manual stirring, the test is regarded as completed. If this test is not conclusive, the adhesive should be exposed to a further complete freeze-thaw cycle.

7.6 Homogenize the sample, where the sample shows a surface-water layer or is thickened.

7.7 Continue the test until three freeze-thaw cycles are completed without stating visible coagulum.

7.8 Measure again the viscosity of the sample at $(23 \pm 1) ^\circ\text{C}$ as described in 7.1.

7.9 Note the test temperature, the condition of the adhesive (see 7.5) after each cycle, the number of cycles completed without formation of visible, indispensable coagulum, the initial viscosity (see 7.1) and the final viscosity (see 7.8) determined.

8 Expression of results and classification

8.1 Expression of results

The freeze-thaw stability of the adhesive tested at a selected freeze temperature is expressed as the number of cycles endured without the formation of visible and indispersable coagulum and a change of viscosity not exceeding the manufacturer's specification limits.

8.2 Classification

In accordance with the test results obtained, the adhesive shall be classified:

"freeze temperature / number of cycles"

For example, an adhesive belongs to the freeze-thaw stability class "- 10 °C/3 cycles" in accordance with EN 1239 if enduring three cycles without visible coagulation at a freeze temperature of - 10 °C.

9 Test report

The test report shall include:

- a) reference to this European Standard, i.e. EN 1239;
- b) complete identification of the adhesive under test, in particular type, name/designation, manufacturer, and lot number/date of supply;
- c) freeze temperature applied in the test cycles;
- d) initial (see 7.1) and final (see 7.8) viscosities of the adhesive tested;
- e) test results expressed in accordance with 8.1;
- f) classification in accordance with 8.2;
- g) any modification of the procedure described in this standard and any circumstances which may have affected the result;
- h) date of test.

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