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SIST EN 1239:1999

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EUROPEAN STANDARD
NORME EUROPÉENNE
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EN 1239

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Descriptors: adhesives, components, tests, determination, freeze-thaw resistance, viscosity measurements, procedure, test results, classifications

English version

Adhesives - Freeze-thaw stability

Adhésifs - Stabilité au gel-dégel

Klebstoffe - Gefrier-Auftau-Stabilität

This European Standard was approved by CEN on 2 January 1998.

CEN members are bound to comply with the CEN/CENELEC Internal Regulations which stipulate the conditions for giving this European Standard the status of a national standard without any alteration. Up-to-date lists and bibliographical references concerning such national standards may be obtained on application to the Central Secretariat or to any CEN member.

This European Standard exists in three official versions (English, French, German). A version in any other language made by translation under the responsibility of a CEN member into its own language and notified to the Central Secretariat has the same status as the official versions.

CEN members are the national standards bodies of Austria, Belgium, Czech Republic, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and United Kingdom.

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EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
EUROPÄISCHES KOMITEE FÜR NORMUNG

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Foreword

This European Standard 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of an identical text or by endorsement, at the latest by August 1998, and conflicting national standards shall be withdrawn at the latest by August 1998.

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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1 Scope

This 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

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.

EN 923	Adhesives - Terms and definitions
EN 1066	Adhesives - Sampling
EN 1067	Adhesives - Examination and preparation of samples for testing
prEN 12092	Adhesives - Determination of viscosity

3 Definitions

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For the purposes of this standard the definitions in accordance with EN 923 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°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°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 manufacturers 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 Safety

Persons using this standard shall be familiar with normal laboratory practice.

This standard does not purport to address all safety problems, if any, associated with its use.

It is the responsibility of the user to establish safety and health practices and to ensure compliance with any European and national regulatory conditions.

6 Apparatus

6.1 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 $-5\text{ }^{\circ}\text{C}$ or $-10\text{ }^{\circ}\text{C}$ the container can be made of polyethylene; at lower freezing temperatures cylindrical stainless steel container can be used.

6.2 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.

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

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6.4 Viscometer, in accordance with prEN 12092 to determine the viscosity of the adhesive to be tested.

7 Sampling, examination and preparation of samples

A significant sample of the adhesive shall be taken in accordance with EN 1066 and prepared for testing as described in EN 1067.

8 Procedure

8.1 Measure the viscosity of the sample at $23\text{ }^{\circ}\text{C}$ in accordance with prEN 12092 using a suitable viscometer (see 6.4) and note the result.

8.2 Put (200 ± 5) ml of the test sample into the cylindrical container (see 6.1).

8.3 Seal the container airtight and place it for 16 h in the test chamber (see 6.2) set at the selected freezing temperature.

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

8.5 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

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should be exposed to a further complete freeze-thaw cycle.

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

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

8.8 Measure again the viscosity of the sample at 23 °C as described in 8.1.

8.9 Note the test temperature, the condition of the adhesive (see 8.5) after each cycle, the number of cycles completed without formation of visible, indispensable coagulum, the initial viscosity (see 8.1) and the final viscosity (8.8) determined.

9 Expression of results and classification

9.1 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 indispensable coagulum and a change of viscosity not exceeding the manufacturers specification limits.

9.2 Classification

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

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"freeze temperature / number of cycles"
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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.