SLOVENSKI STANDARD
SIST EN 924:1998

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Lepila - Raztopinska lepila in lepila brez topil - Določanje plamenišča

Adhesives - Solvent-borne and solvent-free adhesives - Determination of flashpoint

Klebstoffe - Lösemittelhaltige und lösemittelfreie Klebstoffe - Bestimmung des Flammpunktes

Adhésifs - Adhésifs a base de solvant et exempts de solvant - Détermination du point d'éclair

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English version

Adhesives - Solvent-borne and solvent-free adhesives - Determination of flashpoint

Adhésifs - Adhésifs à base de solvant et exempts de solvant - Détermination du point d'éclair

Klebstoffe - Lösemittelhaltige und lösemittelfreie Klebstoffe - Bestimmung des Flammpunktes

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CEN

European Committee for Standardization
Comité Européen de Normalisation
Europäisches Komitee für Normung

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FOREWORD

This European Standard has been prepared by the Technical Committee CEN/TC 193 "Adhesives", the secretariat of which is held by AFNOR.

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of an identical text or by endorsement, at the latest by May 1995, and conflicting national standards shall be withdrawn at the latest by May 1995.

The annex A is informative and contains a "Bibliography".

According to the CEN/CENELEC Internal Regulations, the following countries are bound to implement this European Standard: Austria, Belgium, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland, United Kingdom.

0. INTRODUCTION

This European standard describes one method for the determination of the flashpoint of adhesives containing volatile organic compounds. It is generally equivalent to ISO 1523, Paints, varnishes, petroleum and related products - Determination of flashpoint - Closed cup equilibrium method. By the procedure specified in this European Standard, differences between test apparatus of various standard designs are minimized by ensuring that the test is carried out only when the product under test and the air/vapour mixture above it in the test vessel are approximately in temperature equilibrium.

1. SCOPE

This European standard specifies a method for determining the flashpoint of adhesives containing volatile organic compounds and makes allowance for deviations from standard atmospheric pressure. It applies to solvent-borne and solvent-free adhesives but not to water-borne adhesives containing less than 10% of solvents.

NOTE: Care should be taken in the interpretation of results obtained from solvent mixtures containing halogenated hydrocarbons as such mixtures can give anomalous results (see Annex A).

2. NORMATIVE REFERENCES

This European standard incorporates by dated references, provisions from other publications. These normative references are cited at the appropriate places in the text and the publications are listed hereafter. For these 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.

EN 1066 [1] : Adhesives - Sampling.

EN 1067 [1]: Adhesives - Examination and preparation of samples for testing.

3. DEFINITION

For the purposes of this standard, the following definition applies: Flashpoint (closed cup): Minimum temperature to which a product, confined in a closed cup, must be heated for the vapours emitted to ignite momentarily in the presence of a flame, when operating under standardized conditions.

NOTE : in this European Standard, the flashpoint is corrected to an atmospheric pressure of 101,3 kPa.

4. PRINCIPLE

The test portion is heated in a suitably designed closed cup by immersing it to the required level in a suitable bath.
The temperature of the bath is slowly raised at such a rate that the difference in temperature between the liquid in the bath and the test portion in the cup never exceeds 2°C, and the heating procedure ensures that the temperature of the test portion does not rise more quickly than about 0,5°C in 1,5 min (see note 1).
During the heating-up period, ignition trials are carried out at intervals of not less than 1,5 min (see note 2).
The lowest temperature at which a flash occurs is noted and from this and a duplicate determination the flashpoint of the test product is calculated, corrected to the standard atmospheric pressure of 101,3 kPa.

NOTE 1: To ensure that the test is carried out under approximately equilibrium conditions, a slow rate of heating is necessary because of the low thermal conductivity of some products and also because heat transfer by convection is hindered by the high viscosity of many products. Uniformity of temperature throughout the product under test may be assisted by use of a stirring device, which is not operated during an ignition trial.

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1) In course of preparation.

NOTE 2: A minimum time interval of 1,5 min is necessary to ensure that a saturation concentration of vapour in the air space above the test portion is re-established after each ignition trial.

5. APPARATUS

5.1 Test cup

A closed cup with an internal level indicator (see figure 1).
Examples of suitable closed cups include Abel, Abel-Pensky and Pensky-Martens.
If the adhesive is liable to be affected by the cup material, usually copper, an alternative more appropriate cup material,e.g. stainless steel, shall be used.
The cup may be fitted with a stirrer to improve uniformity of temperature.
If a stirrer originally fitted to the test cup is removed, the aperture in the cover shall be securely plugged before starting the test.
Essentially, the test cup shall be fitted with a cover which carries an opening slide and an ignition device which is inserted to a prescribed level into one of the openings in the cover when a test is made.
The equipment is such that an ignition trial can be performed by opening the slide, inserting and removing the nozzle of the ignition device, and closing the slide again, within a period of (2,5 ±0,5) s.
A mechanically driven device for the opening of the slide and applying the ignition device to the air vapour mixture in the cup is permissible and the source of flame for the ignition device may be any suitable flammable gas.
Alternatively, an electrical ignition device can be used instead of a flame.

5.2 Bath, containing a suitable liquid, capable of being adjusted to the required temperature (see 7.2.1.2.) and of adequate heat capacity to meet the requirements of 7.2.3.6.
A bath fitted with a stirrer and thermostat of suitable range is convenient.
Other procedures for heating the closed cup may be used if the rate of heating complies with the requirements given in clause 4.

5.3 Thermometers.

The test cup (5.1) shall be fitted with a thermometer of appropriate range and dimension that, when immersed in the test portion, measures its temperature with an error no greater than 0,5°C.
A thermometer having a graduation at each 0,5°C is recommended.
The bath (5.2) shall be fitted with a thermometer of equal precision.
When required, the accuracy of the thermometers shall be checked against a reference standard by an authorized laboratory using the stipulated immersion.

5.4 Support, for holding the test cup in the bath (5.2) so that the cover and upper edge are horizontal.
The cup is immersed in direct contact with the liquid in the bath in such a position that the level of the test portion in the cup is the same as that of the liquid in the bath (see figure 1).

6. SAMPLING AND SAMPLE TREATMENT

6.1 Take a representative sample of the product to be tested as described in EN 1066 and examine and prepare it for testing as described in EN 1067.
Samples shall not be stored in plastic (polyethylene, polypropylene, etc,) bottles.

6.2 Because of the possibility of loss of volatile constituents, cool the sample container to at least 10°C below the expected flashpoint before opening it to remove the test portion.
The sample shall receive only the minimum mixing treatment to ensure uniformity.
Immediately after removal of the test portion, tightly close the sample container to ensure that loss of volatile components from the container is minimized.
If this is not carried out, the product sample shall be deemed unsuitable for further testing.

7. PROCEDURE

7.1 Preliminary test

Determine the approximate flashpoint of the sample by one or more preliminary tests.
This determines the starting temperature for the definitive test, which shall be (5 ± 1) °C below the expected value.

7.2 Definitive test

7.2.1 Preparation of apparatus

7.2.1.1 Set up the apparatus in a draught-free position,e.g. in a hood, and preferably in subdued light.

7.2.1.2 Adjust the temperature of the liquid in the bath (5.2) to (5 ± 1)°C below the approximate flashpoint as determined according to 7.1.

7.2.1.3 Carefully clean and dry the test cup (5.1), its cover and thermometer (5.3). Bring them to approximately the same temperature as the bath in 7.2.1.2.

7.2.2 Test portion

7.2.2.1 Obtain and prepare the test portion in accordance with clause 6, and ensure that at all times during this preparation its temperature is at least 10°C below the expected flashpoint temperature.

7.2.2.2 Fill the cup with the test portion until the internal level indicator just disappears under the surface of the liquid.
Take care to avoid both the formation of bubbles and contact between the sample and the cup wall above the level indicator.
If either of these conditions occurs to a significant extent, empty the cup, prepare it again according to 7.2.1.3, and fill it with a fresh test portion.

7.2.3 Determination

7.2.3.1 Immediately after filling the cup, place the cover and thermometer in position.
Support the cup in the bath so that the cover is horizontal and the cup is immersed in direct contact with the liquid in the bath and with the surface of the test portion at the same level as that of the liquid in the bath.
Confirm that the bath is at the required temperature as defined in 7.2.1.2.

7.2.3.2 Light the flame of the gas ignition device (if used) and adjust it to an approximately spherical shape of diameter (3,5 ± 0,5) mm.

7.2.3.3 As soon as the test portion has attained the same temperature as the liquid in the bath (i.e. the starting temperature of the definitive test), switch off the stirrer, if used, and perform an ignition trial by opening the slide, inserting and removing the nozzle of the gas ignition device (or operating the electrical ignition device, if used), and closing the slide again, over a period of (2,5 ± 0,5) s.
Watch for a flash between opening and closing the slide.

7.2.3.4 Record whether or not a flash has occurred.

> NOTE: When the vapour mixture under test is near the flashpoint, application of the ignition flame may give rise to a halo; however, the product is only deemed to have flashed if a comparatively large flame appears and propagates itself over the surface of the liquid.
> If a large blue flame does not appear as a flash, but instead a continuous luminous flame burns in the orifice when the slide is opened and the ignition flame is introduced, then the flashpoint of the product lies considerably below the test temperature and therefore the preliminary test (7.1) should be repeated at a temperature of at least 10°C below the expected flashpoint.

7.2.3.5 If a flash occurs (see the note to 7.2.3.4), carry out the procedure (7.2.3) again with a fresh test portion but starting the test at a temperature about 5°C lower than that selected previously.

7.2.3.6 If no flash occurs (see the note to 7.2.3.4), heat the bath at a rate such that the difference in temperature between the bath and the test portion does not exceed 2°C.
When the test portion has increased in temperature by 0,5°C (i.e. after not less than 1,5 min), repeat the ignition test and if no flash is observed repeat the procedure until a temperature is reached at which a flash occurs (see note).
Read to the nearest 0,5°C the temperature indicated by the cup thermometer, correct this reading for any known thermometer correction, and record the result as the flashpoint at the atmospheric pressure prevailing during the test.
Record also the atmospheric pressure in kilopascals.
As volatile components are likely to be present in the products to be tested the total duration of the test shall not exceed 1 h.

7.2.4 Replicate determination

7.2.4.1 Clean the cup and carry out a second determination using a fresh test portion and repeating the procedures described in 7.2.1 to 7.2.3.

7.2.4.2 For referee test where a better level of precision is required, the procedure specified in clause 10 shall be followed.

8. TESTS FOR REFEREE PURPOSES

8.1 For referee tests, more than two individual measurements may be required to achieve the necessary agreement.

8.2 If the difference between the results of two individual tests, calculated in accordance with clause 9, does not exceed 1,0°C, report the mean value to the nearest 0,5°C as the flashpoint.

8.3 If the difference between the first two results exceeds 1,0°C a third test shall be made.
If the greatest difference between the three results does not exceed 1,5°C, report the mean value to the nearest 0,5°C as the flashpoint.

8.4 If the greatest difference between the first three results exceeds 1,5°C, two further tests shall be made. If only one of the five results differs by more than 1,5°C from the mean value, reject this result and report the mean value of the other four results to the nearest 0,5°C as the flashpoint.

8.5 If more than one of the five results differ by more than 1,5°C from the mean value, report this mean value to the nearest 0,5°C as the flashpoint but state also the individual values and add a note to the test report on the irregular flashing of the product tested.