SLOVENSKI STANDARD
SIST EN 456:1997

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Barve, laki in sorodni izdelki - Določanje plamenišča - Hitra ravnotežna metoda (ISO 3679:1983, spremenjen)

Paints, varnishes and related products - Determination of flashpoint - Rapid equilibrium method (ISO 3679:1983, modified)

Lacke, Anstrichstoffe und ähnliche Produkte - Bestimmung des Flammpunktes - Schnellverfahren (ISO 3679:1983, modifiziert)

Peintures, vernis et produits assimilés - Détermination du point d'éclair - Méthode rapide a l'équilibre (ISO 3697:1983, modifiée)

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Paints, varnishes and related products - Determination of flashpoint - Rapid equilibrium method (ISO 3679, edition 1983 modified)

Peintures, vernis et produits assimilées - Détermination du point e'éclair - Méthode rapide à l'équilibre (ISO 3679, édition 1983 modifiée)

Lacke, Anstrichstoffe und ähnliche Produkte - Bestimmung des Flammpunktes - Schnellverfahren (ISO 3679, Ausgabe 1983 modifiziert)

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CEN

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Foreword

This European Standard is the endorsement of ISO 3679. Endorsement of ISO 3679 was recommended by CEN/TC 139, Paints and Varnishes, under whose competence this European Standard will henceforth fall.

Natonal Standards identical to this European Standard shall be published at the latest by 92-02-26 and conflicting national standards shall be withdrawn at the latest 92-02-26.

The Standard was approved and in accordance with 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.

Endorsement notice

The text of the International Standard ISO 3679:1983 was approved by CEN as a European Standard with agreed common modifications as given below:

This European Standard is based on ISO 3679:1983. Whereas the scope of ISO 3679 covers paints, varnishes, petroleum and related products, the scope of this European Standard is restricted to paints, varnishes and related products. This European Standard therefore comprises the text of ISO 3679:1983 amended by the deletion of all references to petroleum products.

0 Introduction

This International Standard describes one of two methods for the determination of the flashpoint of paints, varnishes and related products and it should be read in conjunction with ISO 1523 when selecting a method. In ISO 1523, a similar determination is specified, using cups described in various national standards.

In both methods, the test ist carried out only when the product under test and the air/vapour mixture above it in the test vessel are approximately in temperature equilibrium.

The apparatus specified in this International Standard enables a similar result to be determined using a more rapid procedure and with a smaller test portion (2 ml) of material than that given in ISO 1523. In addition, the apparatus can be made portable to the extent of being suitable for on-site testing as well as for normal use in laboratories. Collaborative work[1]) has shown that results obtained by these procedures are comparable.

Nevertheless the interpretation of results obtained form solvent mixture containing halogenated hydrocarbons should be considered with caution as these mixtures can give anomalous results[2]).

NOTE - The flash/no flash test using the same equipment under equilibrium conditions is given in ISO 3680.

1 Scope and field of application

This International Standard specifies a method for determining the flashpoint of a paint, varnish, paint binder, solvent, or a related product when the flashpoint is below 110 °C.

NOTE - Care should be taken in the interpretation of results obtained from solvent mixtures containing halogenated hydrocarbons (see clause 0).

2 References

ISO 1512, Paints and varnishes - Sampling.

ISO 1513, Paints and varnishes - Examination and preparation of samples for testing.

ISO 1523, Paints, varnishes, petroleum and related products - Determination of flashpoint - Closed cup equilibrium method.

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[1]) Bell, L.H. J. Inst. Petrol 57 (556) July 1971.

[2]) Rybicky, J. and Stevens, J.R. J. Coatings Technol. 53 (676) May 1981: 40-42

ISO 3680, Paints, varnishes, petroleum and related products - Flash/no flash test - Rapid equilibrium method.

3 Definition

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 International Standard the flashpoint is corrected to an atmospheric pressure of 101,3 kPa (1 013 mbar).

4 Principle

4.1 Method 1 (For liquids whose expected flashpoint is between ambient temperature and 110 °C)

The test portion is heated in the specified apparatus. The ignition trial is carried out after the test portion has been maintained under equilibrium conditions for 60 s at a temperature approximately 3 °C below the expected flashpoint.

The trial is repeated at other temperatures until a flash is observed at a temrature which is not more than 1 °C above a temperature at which no flash was observed. The temperature at which the flash occurs is recorded as the flashpoint at the atmospheric pressure prevailing during the test and this temperature is then corrected to the standard atmospheric pressure of 101,3 kPa (1 013 mbar).

4.2 Method 2 (For liquids whose expected flashpoint is below ambient temperature)

The test portion is cooled to at least 3 °C below the expected flashpoint and then, in the specified apparatus, an ignition trial is carried out as in 4.1, after the test portion has been maintained under equilibrium conditions for 60 s.

The trial is repeated at other temperatures until a flash is observed at a temperature which is not more than 1 °C above a temperature at which no flash was observed. The temperature at which the flash occurs is recorded as the flashpoint at the atmospheric pressure prevailing during the test and this temperature is then corrected to the standard atmospheric pressure of 101,3 kPa (1 013 mbar).

5 Apparatus

5.1 Flashpoint tester, consisting of a block of aluminium alloy or other suitable corrosion-resistant metal of high thermal conductivity. The block has a cylindrical depression or test portion well, of depth approximately 10 mm and diameter approximately 50 mm, over which is fitted a cover. A thermometer is embedded in the block. A plan diagram is given in figure 1 and the essential dimensions are given in figures 2 to 5.

The cover is fitted with an opening slide and a device capable of inserting a test flame (diameter 3,5 ± 0,5 mm) into the well when the slide is open.

When inserted, the extremity of the nozzle of the ignition device shall just intersect the plane of the underside of the cover with a tolerance of ± 0,1 mm. The cover is also provided with an orifice extending into the well for insertion of the test portion and with a suitable clamping device for securing the cover tightly to the metal block so that the three openings in the cover are within the diameter of the well.

It is important that, when the slide is in the open position, the two openings in the slide coincide exactly with the two corresponding openings in the cover. It is also important that, when the slide is in the shut position, all three openings in the cover are closed by the slide.

5.2 Thermometer, of appropriate range and dimensions which measures the temperature of the block within an error no greater than 0,5 °C. A thermometer having a graduation at each 0,5 °C is recommended. When required, the accuracy of the thermometer shall be checked against a reference standard by an authorized laboratory, using the stipulated immersion.

5.3 Heating device, fitted with a temperature controller such that the temperature of the metal block can be maintained within ± 0,2 °C of the required temperature. A signal light is necessary to indicate when heating is on. If the apparatus is intended to be portable, the heating device shall be electrical and shall be part of the complete appartus.

The heating device shall be capable of controlling the rate of increase in the temperature of the flashpoint tester to within 0,5 °C in 30 s.

5.4 Means of cooling the well: Ice, solid carbon dioxide (CO_2), or a Peltier or other suitable cooling device.

If a continuously operating cooling device is used in method 2 (8.2) to control the well temperature, it should be possible to stabilize the temperature to within ± 0,2 °C of the expected flashpoint for a period of 60 s after the test portion has been discharged into the well. This ensures that equilibrium conditions are attained.

5.5 Syringe, capable of delivering 2 ml to an accuracy of ± 0,1 ml or, for use with highly viscous products, a micropipette or spatula (see the note in 8.1.3). 8.1.3).

5.6 Fuel source for the ignition device: flammable gas, for example butane.

5.7 Suitable timing device.

6 Sampling and sample treatment

6.1 Take a representative sample of the product to be tested using the appropriate sampling procedure for the product concerned. References to sampling procedures for various products are given in the annex.

The sample shall be kept in an airtight container until it is to be tested. The ullage, i.e. the air space above the contents of the container, shall not be more than 10 % of the total capacity of the container.

Samples shall not be stored in plastics (polyethylene, polypropylene, etc.) bottles.

6.2 Because of the possiblity of loss of volatile constituents, the sample container shall be cooled to at least 10 °C below the expected flashpoint before opening it to remove the test portion, except when method 2 (see 8.2) is used. In this case, the sample shall be cooled to 3 to 5 °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. After removal of the test portion, the sample container shall immediately be tightly closed 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 Preparation of apparatus

Place the test apparatus in a position where it is not exposed to draughts, and preferably in subdued light.

8 Procedure

8.1 Method 1 (Determination of flashpoint when the expected flashpoint is between ambient temperature and 110 °C)

NOTE - When the expected flashpoint is close to ambient temperature, it may be more appropriate to use method 2.

8.1.1 Ensure that the well and cover slide are clean and free from contamination, using a paper tissue if necessary. Close the cover and ensure that the slide is in the closed position.

8.1.2 Turn on the heating device (5.3). When the thermometer (5.2) reads approximately 3 °C below the expected flashpoint of the product to be tested, slowly adjust the controller of the heating device to the point at which the signal light is just extinguished. Allow the temperature of the well to stabilize, as indicated by the signal light cycling ON/OFF.

8.1.3 Ensure that the syringe (5.5) is clean and dry. Charge the syringe with 2 ml of the cooled sample (6.2) and transfer the syringe to the filling orifice, taking care not to lose any of the contents. Quickly discharge the test portion into the well, remove the syringe, and immediately start the timing device (5.7). Check that the slide of the cover is still in the closed position.

NOTE - If the viscosity of the product under test is so high as to prevent discharge through the orifice, a test portion of 2 to 3 ml may be transferred with a micropipette or a spatula into the well while the cover is open.

Immediately after filling the well, close the cover tightly.

8.1.4 Open the gas control valve and light the pilot and test flames. Adjust the test flame size to an approximately spherical shape of diameter 3,5 ± 0,5 mm.

8.1.5 When 60 s have elapsed, by which time the test portion is deemed to have reached the test temperature, perform the ignition trial by opening the slide, inserting and removing the nozzle, and closing the slide again over a period of 2,5 ± 0,5 s. Watch for a flash between opening and closing the slide.

8.1.6 Record whether a flash has occurred.

NOTE 1 - 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 blue flame appears and propagates itself over the surface of the liquid.

NOTE 2 - If a continous luminous flame burns in the orifice when the slide is opened and the ignition flame is introduced, then the flashpoint lies considerably below the test temperature.

8.1.7 Close the gas control valve and clean the apparatus.

8.1.8 If no flash is observed, repeat the test at 5 °C higher intervals, using a fresh test portion in each test, until a flash is observed.

If a flash is observed, repeat the test at 5 °C lower intervals, using a fresh test portion in each test, until no flash is observed.

CAUTION - Once a test flame has been applied to the test portion, the test is terminated and fresh test portions shall be used for each successive test.

8.1.9 Having established a flash between two temperatures 5 °C apart, repeat, using fresh test portions the procedures in 8.1.1 to 8.1.7 at 1 °C intervals above the lower of the two temperatures until a flash is observed. Read to the nearest 0,5 °C the temperature indicated by the thermometer when this flash occurs, correct this reading for any known thermometer correction, and record the result as the flashpoint at the atmospheric pressure prevailing during the test (see clause 9). Record also the atmospheric pressure in kilopascals, millibars, or millimetres of mercury.

8.1.10 Repeat the determination (8.1.1 to 8.1.9) and calculate the mean corrected flashpoint (see clause 9) to the nearest 0,5 °C.