International Standard 1523

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Paints, varnishes, petroleum and related products — Determination of flashpoint — Closed cup equilibrium method

Peintures, vernis, pétrole et produits assimilés — Détermination du point d'éclair — Méthode à l'équilibre en vase clos

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Foreword

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Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council.

International Standard ISO 1523 was developed jointly by Technical Committees ISO/TC 35, *Paints and varnishes*, and ISO/TC 28, *Petroleum products and lubricants*, and was circulated to the member bodies in May 1981.

It has been approved by the member bodies of the following countries:

Australia	Israel	Romania
Austria	Italy	South Africa, Rep. of
Belgium	Kenya	Spain
Brazil	Korea, Rep. of	Sri Lanka
Canada	Mexico	Sweden
China	Netherlands	Switzerland
Czechoslovakia	New Zealand	Thailand
Egypt, Arab Rep. of	Norway	United Kingdom
Hungary	Poland	USA
India	Portugal	USSR

The member bodies of the following countries expressed disapproval of the document on technical grounds:

France
Germany, F.R.
Ireland

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Paints, varnishes, petroleum and related products — Determination of flashpoint — Closed cup equilibrium method

0 Introduction

This International Standard describes one of two methods for the determination of the flashpoint of paints, varnishes, petroleum and related products and it should be read in conjunction with ISO 3679 when selecting a method.

By the procedure specified in this International 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.

Nevertheless the interpretation of results obtained from solvent mixtures containing halogenated hydrocarbons should be considered with caution as these mixtures can give anomalous results. [1]

NOTE — The flash/no flash test using the same equipment under equilibrium conditions is given in ISO 1516.

1 Scope and field of application

This International Standard specifies a method for determining the flashpoint of a paint, varnish, paint binder, solvent, petroleum or a related product and makes allowance for deviations from standard atmospheric pressure.

The method is suitable for use over the temperature range 5 to 110 °C, although some of the apparatus listed in annex A cannot cover all of this range using the thermometer supplied with the apparatus.

NOTES

1 In some countries, existing regulations may require the use of other methods over at least a part of the temperature range 5 to 110 °C.

2 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 1516, *Paints, varnishes, petroleum and related products — Flash/no flash test — Closed cup equilibrium method.*

ISO 3170, *Petroleum products — Liquid hydrocarbons — Manual sampling.*

ISO 3171, *Petroleum products — Liquid hydrocarbons — Automatic pipeline sampling.*

ISO 3679, *Paints, varnishes, petroleum and related products — Determination of flashpoint — 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

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).

[1] RYBICKY, J. and STEVENS, J.R., *J. Coatings Technol.* **53** (676) May 1981: 40-42.

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 (1 013 mbar).

NOTES

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, although it is **not** to be operated during an ignition trial.

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; the closed cups specified in a number of national standards (see annex A) satisfy the necessary requirements. If a stirrer is fitted to the test cup used, it may be operated during the heating-up period but shall be stopped during the ignition trial. 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 details of the cover, slide, ignition device and its movement shall be in accordance with the appropriate national standard listed in annex A. 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, in 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.

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 dimensions 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 the figure).

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 annex B.

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 possibility 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. 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 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 about 5 °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 and preferably in subdued light.

7.2.1.2 Adjust the temperature of the liquid in the bath (5.2) to 5 °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 ignition device 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), perform an ignition trial by opening the slide, inserting and removing the nozzle of the ignition device, 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 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 blue 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 never exceeds 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, millibars, or millimetres of mercury.

NOTE — As volatile components are likely to be present in the products to be tested, the total duration of the test should 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. Calculate the mean corrected flashpoint (see clause 8) to the nearest 0,5 °C.

7.2.4.2 For referee tests where a better level of precision is required, the procedure specified in clause 11 should be followed.

8 Calculation

Calculate the flashpoint, in degrees Celsius, corrected to standard atmospheric pressure of 101,3 kPa (1 013 mbar or 760 mmHg), by adding algebraically to the observed temperature the correction from one of the following equations

$$C = \frac{101{,}3 - p_0}{4} \text{ or } \frac{1013 - p_1}{40} \text{ or } \frac{760 - p_2}{30}$$

where

C is the correction, in degrees Celsius;

p_0 is the atmospheric pressure, expressed in kilopascals;

p_1 is the atmospheric pressure, expressed in millibars;

p_2 is the atmospheric pressure, expressed in millimetres of mercury.

Record the mean corrected flashpoint to the nearest 0,5 °C.

NOTE — Whilst these formulae are strictly correct only within the barometric pressure range from 98,0 to 104,7 kPa, for pressures outside this range the error is sufficiently small to be ignored.

9 Precision

9.1 Repeatability (*r*)

The value below which the absolute difference between two single test results obtained on identical material by one operator in one laboratory using the same equipment within a short interval of time using the standardized test method, may be expected to lie with a 95 % probability, is 2 °C.

9.2 Reproducibility (R)

The value below which the absolute difference between the mean of two single test results obtained on identical material by operators in different laboratories, using the standardized test method, may be expected to lie with a 95 % probability, is 3 °C.

10 Test report

The test report shall include at least the following information:

a) the type and identification of the product tested;

b) a reference to this International Standard (ISO 1523);

c) a reference to the standard describing the test cup used, and details of any modifications made;

d) the mean corrected flashpoint, in degrees Celsius, calculated as in clause 8;

e) any deviation, by agreement or otherwise, from the test procedure specified;

f) the date of the test.

11 Tests for referee purposes

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

11.2 If the difference between the results of two individual tests, calculated in accordance with clause 8, does not exceed 1,0 °C, report the mean value to the nearest 0,5 °C as the flashpoint.

11.3 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.

11.4 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.

11.5 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.

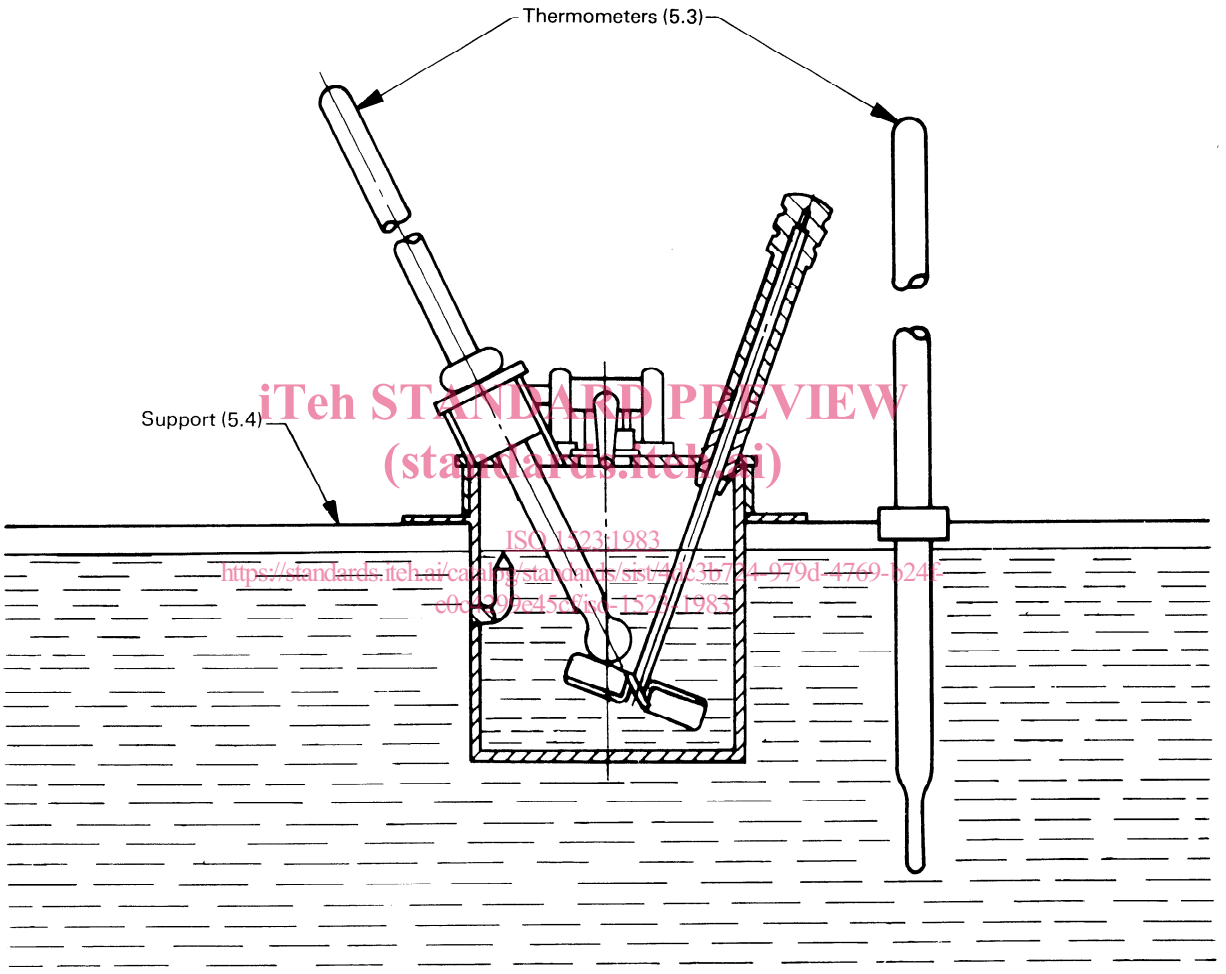

Figure — Closed cup, with fitted stirrer (see 5.1), immersed in the bath
(The stirrer for the bath is not shown.)

Annex A

Suitable closed cups specified in national standards

The cups listed below of closed-cup apparatus described in standards are known to satisfy the necessary requirements of this method of test for the determination of flashpoint. The method requires immersion of the cup in a bath and if a suitable bath is not included in the standard apparatus one must be provided (see 5.2).

Abel cup	French Standard	NF T 66-009
Abel cup	French Standard	NF M 07-011
Abel cup	British Standard	BS 3442 (and IP 33 and IP 170)
Abel-Pensky cup	Czechoslovak Standard	CSN 67 3015
Abel-Pensky cup	French Standard	NF M 07-036
Abel-Pensky cup	German Standard	DIN 51 755
Abel-Pensky cup (+ stirrer)	Swedish Standard	SIS 02 18 11
Abel-Pensky cup (modified according to Bleisch)	German Standard	DIN 53 213 Teil 1
Pensky-Martens cup	International Standard	ISO 2719
Pensky-Martens cup	British Standard	BS 2839 (and IP 34)
Pensky-Martens cup	French Standard	NF M 07-019
Pensky-Martens cup	German Standard	DIN 51 758
Pensky-Martens cup	Netherlands Standard	NEN-ISO 2719
Pensky-Martens cup	Swedish Standard	SIS 02 18 12
Pensky-Martens cup	USA Standard	Z 11.7 (and ASTM D 93)
Tag cup	USA Standard	Z 11.24 (and ASTM D 56)

Annex B

Sampling procedures

B.1 Paints, varnishes and related products

Take a representative sample of the product to be tested as described in ISO 1512 and examine and prepare it for testing as described in ISO 1513. In addition observe the precautions of clause 6 and 7.2.2.1.

B.2 Petroleum and related products

Take a representative sample of the product to be tested as described in ISO 3170 or ISO 3171 as appropriate. In addition observe the precautions of clause 6 and 7.2.2.1.