INTERNATIONAL STANDARD

ISO 1516

Third edition
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Determination of flash/no flash — Closed cup equilibrium method

Essai de point d'éclair de type passe/ne passe pas — Méthode à l'équilibre en vase clos

Reference number
ISO 1516:2002(E)

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 3.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this International Standard may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 1516 was prepared jointly by Technical Committees ISO/TC 28, *Petroleum products and lubricants* and ISO/TC 35, *Paints and varnishes*.

This third edition cancels and replaces the second edition (ISO 1516:1981), which has been technically revised.

Annex A of this International Standard is for information only.

Introduction

This International Standard describes one of two closed cup equilibrium methods for carrying out a flash/no flash test for paints, varnishes, petroleum and related products, and it should be read in conjunction with the second equilibrium method, ISO 3680 ([2] in the bibliography), when selecting a method.

The determination of the flash point using the same equipment is described in ISO 1523.

This test method does not determine the flash point of the product under test, but merely its behaviour at the selected equilibrium temperature as may be required to comply with laws or regulations relating to the storage, transport and use of flammable products. For this purpose, it is unnecessary to determine the exact flash point, but it is necessary to determine whether or not flashing occurs at a given temperature. By the procedure specified, 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 considered to be in temperature equilibrium.

Determination of flash/no flash — Closed cup equilibrium method

WARNING — The use of this International Standard may involve hazardous materials, operations and equipment. This International Standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this International Standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

1 Scope

This International Standard specifies a method to determine if paints, varnishes, paint binders, solvents, petroleum or related products, when maintained at a selected equilibrium temperature and under the conditions of the test, give off sufficient flammable vapour to cause ignition on application of an external source of flame applied in a standard manner.

This International Standard is not applicable to water-borne paints which may, however, be tested using ISO 3680 ([2] in the bibliography).

The method is suitable for use over the temperature range – 30 °C to 110 °C, depending on the use of different apparatus listed in Table 1.

The interpretation of results obtained from solvent mixtures containing halogenated hydrocarbons should be considered with caution, as these mixtures can give anomalous results.

2 Normative references

The following normative documents contain provisions which, through reference in this text, constitute provisions of this International Standard. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the normative documents indicated below. For undated references, the latest edition of the normative document referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

ISO 1513:1992, *Paints and varnishes — Examination and preparation of samples for testing*

ISO 1523:2002, *Determination of flash point — Closed cup equilibrium method*

ISO 2719:—[1)], *Determination of flash point — Pensky-Martens closed cup method*

ISO 3170:1988, *Petroleum liquids — Manual sampling*

ISO 3171:1988, *Petroleum liquids — Automatic pipeline sampling*

ISO 13736:1997, *Petroleum products and other liquids — Determination of flash point — Abel closed cup method*

ISO 15528:2000, *Paints, varnishes and raw materials for paints and varnishes — Sampling*

1) To be published. (Revision of ISO 2719:1988)

ASTM D56-00, *Standard Test Method for Flash Point by Tag Closed Tester*

DIN 51755:1974, *Testing of mineral oils and other combustible liquids; determination of flash point by the closed tester according to Abel-Pensky*

3 Term and definition

For the purposes of this International Standard, wherein ignition source is recognized as being a flame, the following term and definition applies.

3.1
flash point
lowest temperature of the test portion, corrected to a barometric pressure of 101,3 kPa, at which application of an ignition source causes the vapour of the test portion to ignite and the flame to propagate across the surface of the liquid under the specified conditions of test

4 Principle

The test portion is placed in a suitably designed closed cup mounted in a thermostatically controlled bath. After the test portion has been maintained at the selected equilibrium temperature conditions for at least 10 min, an ignition trial is carried out by directing a small flame into the test cup. Whether or not the test flame causes the vapour above the test portion to ignite, is recorded.

5 Chemicals and materials

5.1 Cleaning solvent, for removal of traces of the previous test portion from the test cup and cover.

NOTE The choice of solvent will depend upon the previous material tested, and the tenacity of the residue. Low volatility aromatic (benzene-free) solvents may be used to remove traces of oil, and mixed solvents such as toluene-acetone-methanol may be effective for the removal of gum-type deposits.

5.2 Verification liquids, see ISO 1523.

6 Apparatus

6.1 Test cup and **lid:** a closed cup with, where specified, an internal level indicator, and lid, as specified in the standards listed in Table 1.

The test cup shall be fitted with a tightly fitting cover which carries an opening slide and an ignition device capable, when the slide is open, of positioning an ignition flame, with a diameter of between 3 mm and 4 mm, at the approximate centre of the opening in the lid. When positioned, the tip of the ignition device shall be between the planes of the lower and upper surfaces of the lid at a point on a radius passing through the centre of the opening. The apparatus shall be designed such that an ignition trial can be performed by opening the slide, positioning and removing the nozzle of the ignition device, and closing the slide again in a period of between 2 s and 3 s. A mechanically driven device for carrying out this operation is permitted provided that it can be shown that it meets the specification.

NOTE The source of flame in the ignition device may be any suitable flammable gas.

6.2 Test cup thermometer, as specified for use with the test cup in the standards listed in Table 1.

NOTE Other types of temperature measuring devices may be used, provided that they meet the requirements for accuracy and have the same response as the thermometers specified in the standards listed in Table 1.

Table 1 — Applicable temperature range

Standard test method	Temperature range °C
ISO 2719 Pensky-Martens	10 to 110
ISO 13736 Abel	– 30 to 80
ASTM D56 Tag	up to 93
DIN 51755 Abel-Pensky	– 30 to 65

6.3 Thermostated bath, containing a suitable liquid, thermostated and capable of being adjusted to, and maintained at, the required temperature ± 0,5 °C.

6.4 Thermostated bath thermometer, capable of measuring the bath temperature with the same accuracy as that of the test cup thermometer (6.2).

6.5 Support, designed such that the test cup is immersed in direct contact with the liquid in the thermostated bath, in such a position that the level of the test portion in the cup is the same as that of the liquid and that the cover and upper edge are horizontal. For an example, see Figure 1, which illustrates the use of the Abel test cup.

6.6 Barometer, accurate to 0,1 kPa. Barometers precorrected to give sea-level readings, such as those used at weather stations and airports, shall not be used.

6.7 Heating bath or **oven** (if required)**,** capable of meeting the requirements for the pretreatment of samples that are semi-solid or solid at ambient temperature. See 9.1.4.

6.8 Cooling bath or **refrigeration unit** (if required)**,** capable of cooling the sample to at least 10 °C below the test temperature. See 9.3.

7 Apparatus preparation

7.1 Location of the apparatus

Set up the apparatus in a draught-free position and preferably in subdued light.

7.2 Preparation of the thermostated bath

Bring the temperature of the thermostated bath (6.3) to, and maintain it at, the selected equilibrium test temperature ± 0,5 °C (see 10.3).

7.3 Preparation of the flash point apparatus

Carefully clean and dry the test cup (6.1), its cover and thermometer (6.2). Bring them to a temperature at least 2 °C below the selected equilibrium test temperature (see 10.3).

7.4 Apparatus verification

Verify the correct functioning of the apparatus at least once a year by testing a certified reference material (CRM) (see 5.2) in accordance with the procedures for apparatus verification given in ISO 1523.

It is recommended that more frequent verification checks are made using secondary working standards (SWSs).

8 Sampling

8.1 Unless otherwise specified, obtain samples for analysis in accordance with the procedures given in ISO 15528, ISO 3170, ISO 3171 or an equivalent National Standard.

8.2 Place samples in tightly sealed containers, appropriate to the material being sampled. **For safety purposes, ensure that the sample container is only filled to between 85 % and 95 % of its capacity.**

8.3 Store the samples in conditions to minimize vapour loss and pressure build-up. Avoid storing the samples at temperatures in excess of 30 °C.

9 Sample handling

9.1 Petroleum products

9.1.1 Subsampling

If an aliquot of the original sample is to be stored prior to testing, ensure that the container is filled to at least 50 % of its capacity.

NOTE Results of flash point determinations may be affected if the sample volume falls below 50 % of the container capacity.

9.1.2 Samples containing undissolved water

If a sample contains undissolved water, decant an aliquot from the water prior to mixing.

Flash point results can be affected by the presence of water. For certain fuel oils and lubricants, it may not always be possible to decant the sample from the free water. In such cases, the water should be separated from the aliquot physically, prior to mixing, or, if this is not possible, the material should be tested according to ISO 3679 ([1] in the bibliography).

9.1.3 Samples that are liquid at ambient temperature

Mix samples by gently shaking by hand prior to the removal of the test portion, taking care to minimize the loss of volatile components, and proceed in accordance with clauses 10 and 11.

9.1.4 Samples that are semi-solid or solid at ambient temperature

Heat the sample in its container in a heating bath or oven (6.7) at a temperature of 30 °C ± 5 °C or a higher temperature not exceeding 28 °C below the expected flash point, whichever is the greater, for 30 min. If after 30 min the sample is still not completely liquefied, extend the preheating for further 30 min periods as required. Avoid overheating the sample, as this could lead to loss of volatile components. After gentle agitation, proceed in accordance with clauses 10 and 11.

9.2 Paints and varnishes

Prepare the samples in accordance with ISO 1513.

9.3 Samples to be tested at a temperature below ambient

Cool the sample in a cooling bath or refrigeration unit (6.8) to at least 10 °C below the test temperature.

10 Test temperature

10.1 Barometric pressure

Measure and record the ambient barometric pressure using a barometer (6.6) in the vicinity of the apparatus at the time of test.

NOTE It is not considered necessary to correct the barometric pressure to 0 °C, although some barometers are designed to make this correction automatically.

10.2 Conversion of barometric pressure reading

If the barometric pressure reading is measured in a unit other than kilopascals, convert to kilopascals using one of the following equations:

Reading in hPa × 0,1 = kPa

Reading in mbar × 0,1 = kPa

Reading in mmHg × 0,133 3 = kPa

10.3 Calculation of test temperature corrected to standard atmospheric pressure

If it is necessary to correct the test temperature to standard atmospheric pressure of 101,3 kPa, calculate the test temperature, T_T, using the following equation.

$$T_T = T_S - 0{,}25\,(101{,}3 - p)$$

where

T_S is the selected test temperature, expressed in degrees Celsius;

p is the ambient barometric pressure, expressed in kilopascals.

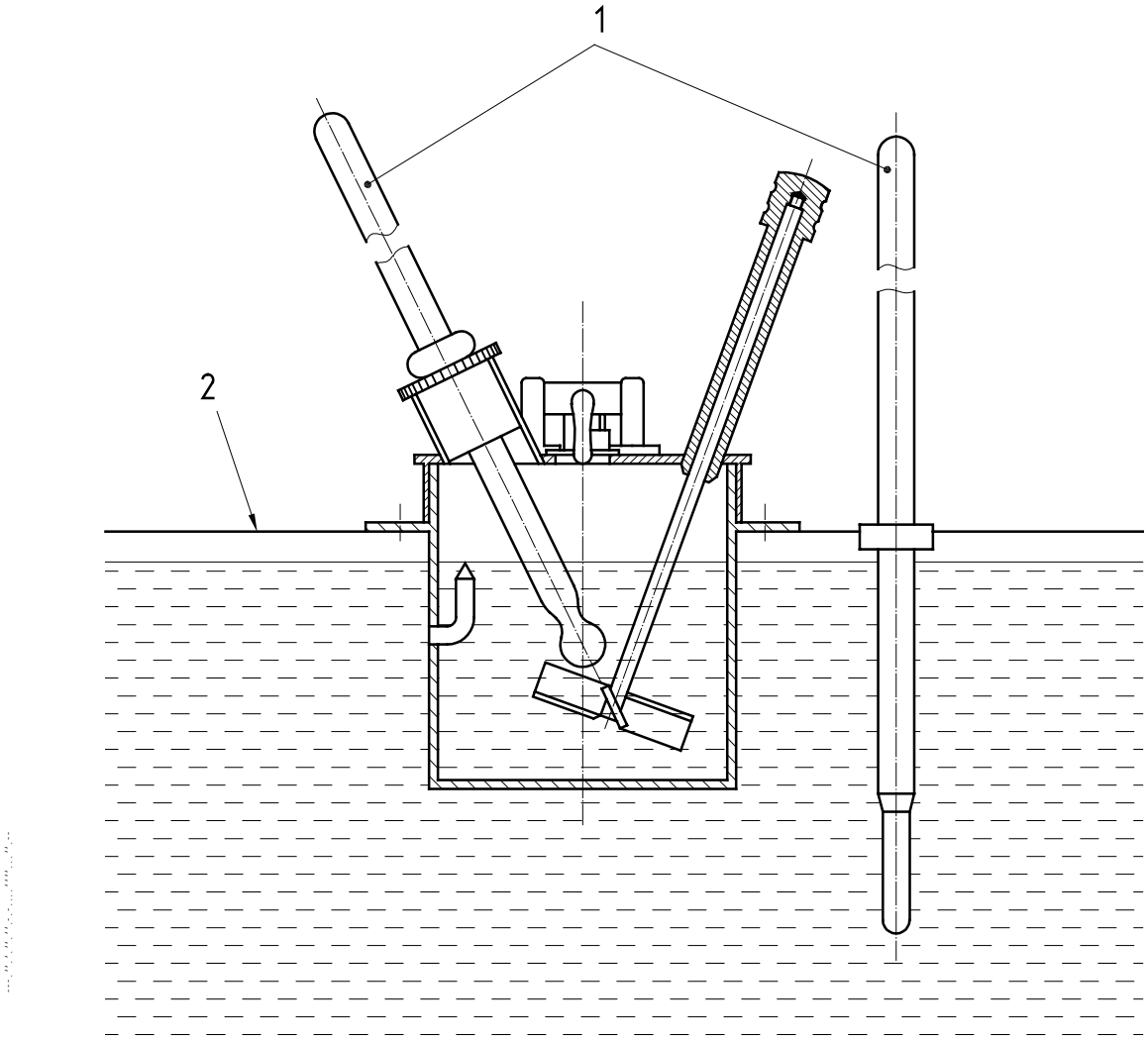
NOTE This equation is strictly correct only within the barometric pressure range from 98,0 kPa to 104,7 kPa.

11 Procedure

11.1 Fill the test cup (6.1) with the test portion either until the internal level indicator just disappears under the surface of the liquid, or with the required volume of test portion (see the note below). Take care to avoid the formation of bubbles and contact between the test portion and the test cup wall above the level indicator. If either of these conditions occurs to a significant extent, empty the test cup and prepare it in accordance with 7.3 before refilling it with a fresh test portion.

NOTE For the Tag closed tester, 50 ml ± 0,5 ml are used for the test.

11.2 Immediately after filling the test cup, place the cover and thermometer (6.2) in position. Support the test cup in the bath (6.3) so that the cover is horizontal and the test 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 the liquid in the bath. For an example, see Figure 1, which illustrates the use of the Abel test cup.



Key

1 Thermometers

2 Support

NOTE The stirrer for the thermostated bath is not shown.

Figure 1 — Abel closed cup, with fitted stirrer, correctly positioned in the thermostated bath

11.3 Light the flame of the ignition device and adjust it to an approximately spherical shape with a diameter of between 3 mm and 4 mm.

11.4 If the apparatus is fitted with a stirrer, use it in accordance with the procedure given in the test method appropriate to the test cup being used.

11.5 Allow the temperature of the test portion to rise to the selected equilibrium test temperature (see 10.3) and maintain it at this temperature ± 0,5 °C for 10 min.

11.6 When appropriate, cease stirring the test portion and perform an ignition trial by opening the slide, lowering and raising the ignition device, and closing the slide again, over a period of 2 s to 3 s. Record whether or not a flash occurs.

11.7 If no flash is observed, maintain the test portion at the test temperature for a further 10 min and repeat the test. If the second test results in a flash, the product shall be considered to have flashed at the selected equilibrium test temperature. If no flash is observed during the second test, record that no flash has occurred.

11.8 If the vapour mixture under test is near the flash point, 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. In case of doubt, the test shall be repeated with a fresh test portion and, if the doubt is unresolved by the second test, the product shall be regarded as having flashed.

11.9 If a continuous luminous flame burns in the orifice when the slide is opened and the ignition flame is introduced, the flash point lies below the selected equilibrium temperature.

12 Expression of results

Report whether or not the test portion flashed at the selected test temperature, corrected to standard atmospheric pressure.

13 Precision

Precision data are not available for this method; however, an indication of the repeatability and reproducibility of results at a selected equilibrium temperature may be obtained from the precision data given in ISO 1523, see annex A.

14 Test report

The test report shall include at least the following information:

a) a reference to this International Standard;

b) the type and complete identification of the sample tested;

c) a reference to the standard describing the test cup used (see Table 1);

d) the ambient barometric pressure in the vicinity of the apparatus at the time of the test (see 10.1);

e) the test temperature (see clause 10);

f) whether or not the test portion flashed at the test temperature;

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

h) the date of the test.

Annex A
(informative)

Precision data from ISO 1523

A.1 Repeatability, r

The difference between two test results obtained by the same operator with the same apparatus under constant operating conditions on identical test material would in the long run, in the normal and correct operation of the test method, exceed the value given below in only one case in twenty:

r = 2 °C

A.2 Reproducibility, R

The difference between two single and independent test results obtained by different operators working in different laboratories on identical test material would in the long run, in the normal and correct operation of the test method, exceed the value given below in only one case in twenty:

R = 3 °C

Bibliography

[1] ISO 3679:—[2], *Determination of flash point — Rapid equilibrium closed cup method*

[2] ISO 3680:—[3], *Determination of flash/no flash — Rapid equilibrium closed cup method*

2) To be published. (Revision of ISO 3679:1983)

3) To be published. (Revision of ISO 3680:1983)

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