International Standard 1516

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

Peintures, vernis, produits pétroliers et assimilés — Essai de point d'éclair par tout ou rien — Méthode à l'équilibre en vase clos

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards institutes (ISO member bodies). The work of developing International Standards is carried out through ISO technical committees. Every member body interested in a subject for which a technical committee has been set up 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.

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 1516 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 July 1979.

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

Australia	Hungary	Romania
Austria	India	South Africa, Rep. of
Brazil	Israel	Sweden
Canada	Italy	Switzerland
Chile	Kenya	United Kingdom
China	Korea, Rep. of	USA
Egypt, Arab Rep. of	Netherlands	USSR
France	New Zealand	
Germany, F.R.	Poland	

No member body expressed disapproval of the document.

This second edition cancels and replaces the first edition (i.e. ISO 1516-1973).

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

0 Introduction

This International Standard sets out one of two methods for carrying out the flash/no flash test for paints, varnishes, petroleum and related products, and it should be read in conjunction with ISO 3680 when selecting a method.

This method of test does not determine the flashpoint 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 flashpoint but it is necessary to determine whether or not flashing occurs at a single 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 exactly in temperature equilibrium.

NOTE — The determination of the exact flashpoint using the same equipment is given in ISO 1523.

1 Scope and field of application

This International Standard specifies a method to determine if a flammable material such as a paint, varnish, paint binder, solvent, petroleum or a related product, when maintained at a selected equilibrium temperature and under the conditions of the test, gives off sufficient flammable vapour at this temperature to cause ignition on application of an external source of flame applied in a standard manner.

The method is suitable for use over the temperature range 5 to 65 °C, although some of the apparatus listed in annex A cannot cover all of this range using the thermometer supplied with the apparatus. The procedure also makes allowance for deviations from standard atmospheric pressure.

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.* [1)]

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

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

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

3 Principle

The test portion is heated in a suitably designed closed cup in a suitable water-bath. The ignition trial is carried out after the test portion has been maintained under equilibrium conditions for at least 10 min at the selected equilibrium temperature. This procedure ensures that the air/vapour space above the test portion has attained the saturation concentration of flammable vapour before the ignition trial is performed. The test report records whether the test portion flashed or did not flash.

1) At present at the stage of draft. (Revision of ISO 1523-1973.)

4 Apparatus

4.1 Test cup : A closed cup with an internal level indicator; the closed cups specified in a number of national standards satisfy the necessary requirements (see annex A). 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 tightly fitting cover which carries an opening slide and an ignition device capable, when the slide is open, of inserting an ignition flame (diameter 3,5 ± 0,5 mm); when inserted, the nozzle of the ignition device shall be 1 ± 1 mm above the underside of the cover. 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 doing this is permitted. The source of flame in the ignition device may be any suitable flammable gas.

4.2 Water-bath, capable of being adjusted to the required temperature (see 6.1), and of adequate heat capacity to meet the requirements of 6.3. A bath fitted with a stirrer and thermostat of suitable range is convenient.

4.3 Thermometers.

The test cup shall be fitted with a thermometer of appropriate range and dimensions, which when immersed in the test portion measures its temperature within a maximum error of 0,5 °C. A thermometer having a graduation at each 0,5 °C is recommended.

The water-bath 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.

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

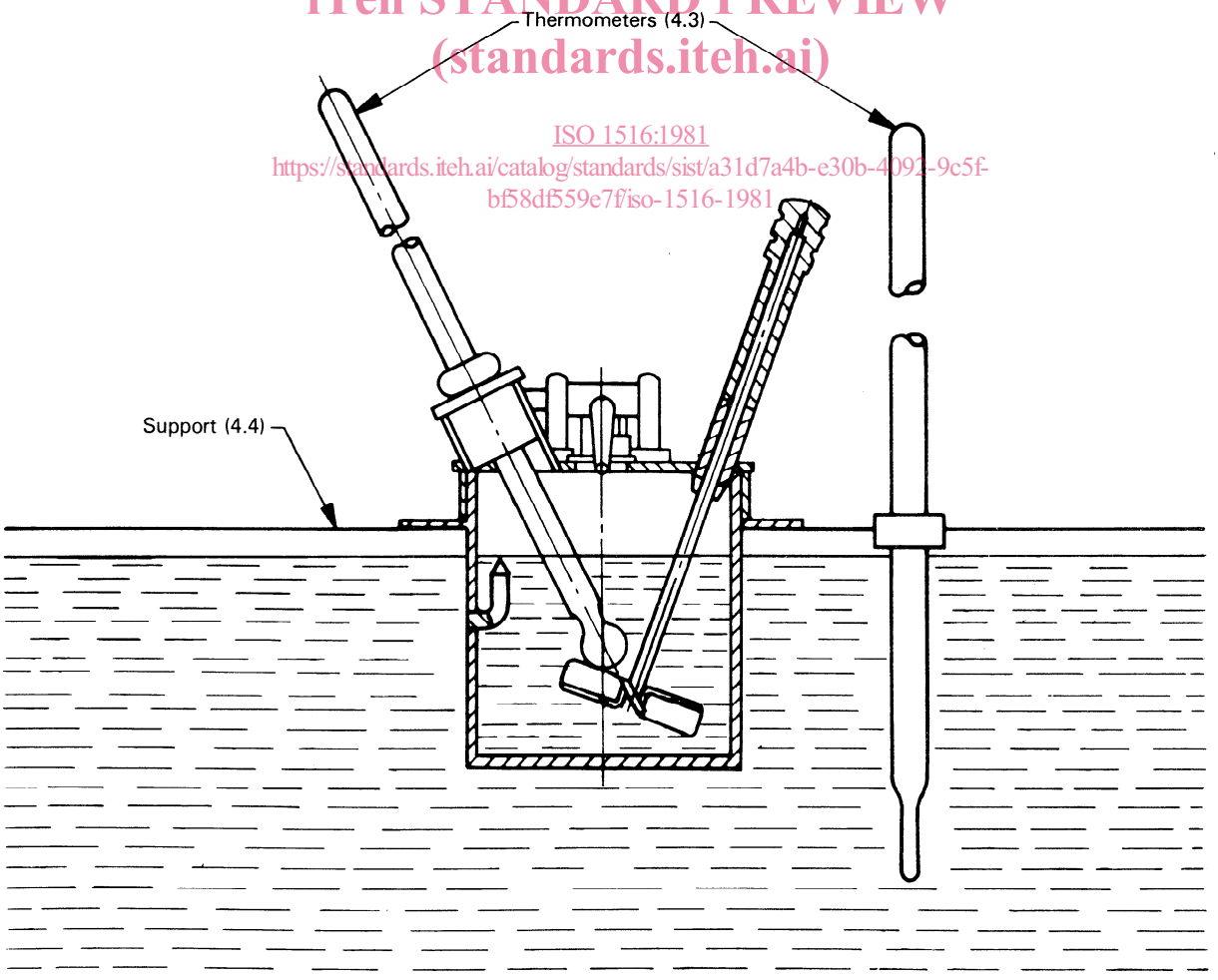

Figure — Closed cup, with fitted stirrer (see 4.1), immersed in the water-bath
(The stirrer for the water-bath is not shown.)

5 Sampling and sample treatment

5.1 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 air-tight 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.

NOTE — Samples should not be stored in plastics (polyethylene, polypropylene, etc.) bottles since volatile material may diffuse through the walls of the bottle.

5.2 Because of the possibility of loss of volatile constituents, the sample container shall be cooled to at least 10 °C below the selected equilibrium temperature 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 flammable components from the container is minimized. If this is not carried out, the product sample shall be deemed unsuitable for further testing.

6 Procedure

6.1 Preparation of apparatus

6.1.1 Adjust the temperature of the bath (4.2) to, and maintain it at, the selected equilibrium temperature (subject to a tolerance of $^{+0,5}_{\ 0}$ °C).

6.1.2 The selected equilibrium temperature shall be corrected for variation from an atmospheric pressure of 101,3 kPa (1 013 mbar or 760 mmHg), by raising the value for a higher pressure or lowering the value for a lower pressure at the rate of 1 °C for each 4 kPa (40 mbar or 30 mmHg) difference.

NOTE — Although this correction is only strictly valid within the atmospheric pressure range 98,0 to 104,7 kPa, for pressures outside this range the error is sufficiently small to be ignored.

6.1.3 Carefully clean and dry the test cup (4.1) its cover and thermometer (4.3). Bring them to a temperature at least 2 °C below the selected equilibrium temperature.

6.2 Test portion

6.2.1 Obtain and prepare the test sample in accordance with clause 5, and ensure that at all times during this preparation its temperature is at least 10 °C below the selected equilibrium temperature.

6.2.2 Fill the test cup with the test sample until the internal level indicator just disappears under the surface of the liquid. Take care to avoid 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 6.1.3 and fill it with a fresh test portion.

6.3 Determination

6.3.1 Immediately after filling the test cup, place the cover and thermometer in position and support the cup in the bath so that the cover is horizontal and the cup is immersed in direct contact with the water, and with the surface of the test portion at the same level as the water in the bath.

6.3.2 Light the flame of the ignition device and adjust it to the size of a bead of diameter 3,5 ± 0,5 mm.

6.3.3 Allow the temperature of the test portion to rise within 0,5 °C of the selected equilibrium temperature. Maintain these conditions for 10 min, or such longer time interval as is necessary, to permit the temperature of the test portion to reach the selected equilibrium temperature. Then perform the 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.

6.3.4 Record whether a flash has occurred.

6.3.5 If no flash was observed, maintain the test portion at the test temperature for 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 temperature.

NOTES

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. 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.

2 If a continuous luminous flame burns in the orifice when the slide is opened and the ignition flame is introduced, then the flashpoint lies considerably below the selected equilibrium temperature.

7 Precision

Precision data are not quoted for this method. However, when selecting a temperature to be specified for this test method, a knowledge of the precision of the procedure can be useful. An indication of the repeatability and reproducibility of results close to a selected equilibrium temperature is obtainable from the precision data given in ISO 1523, which is a similar method used for the determination of the flashpoint temperature.

8 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;

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

d) the selected equilibrium temperature used, in degrees Celsius, the correction applied, the atmospheric pressure and whether the product flashed or did not flash;

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

f) the date of the test.

Annex A

Suitable closed cups specified in standards

The cups listed below of closed-cup apparatus described in standards are known to satisfy the necessary requirements of this method for carrying out the flash/no flash test. The method requires immersion of the cup in a water-bath and if a suitable bath is not included in the standard apparatus one must be provided (see 4.2).

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	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 specified in ISO 1512 and examine and prepare it for testing as specified in ISO 1513. In addition observe the precautions of clause 5 and 6.2.1

B.2 Petroleum and related products

Take a representative sample of the product to be tested as specified in ISO 3170 or ISO 3171 as appropriate. In addition observe the precautions of clause 5 and 6.2.1

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