
**Determination of flash point — Rapid
equilibrium closed cup method**

*Détermination du point d'éclair — Méthode rapide à l'équilibre en vase
clos*

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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 2.

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 document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 3679 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 3679:1983), which has been technically revised.

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Introduction

This International Standard describes one of two closed cup equilibrium methods for the determination of the flash point of paints, varnishes, paint binders, solvents, adhesives, petroleum and related products. When selecting a method, it should therefore be read in conjunction with the second method, ISO 1523 [4]. When used in conjunction with the flash detector (A.1.6), this International Standard is also suitable for the determination of the flash point of fatty acid methyl esters (FAME).

In both ISO 3679 and ISO 1523, the test is only carried out when the material under test and the air/vapour mixture above the material in the test cup are approximately in temperature equilibrium.

The apparatus specified in this International Standard enables a similar test result to be determined using a more rapid procedure and a smaller test portion (2 ml or 4 ml) than that required in ISO 1523. In addition, the apparatus can be made portable to the extent of being suitable for on-site testing in addition to its more normal use in laboratories.

Collaborative work (see [6] in the Bibliography) has shown that results obtained by these procedures are comparable. The interpretation of flash point results obtained on solvent mixtures containing halogenated hydrocarbons should be considered with caution, as these mixtures can give anomalous results (see [7] in the Bibliography).

Flash point values are not a constant physical-chemical property of materials tested. They are a function of the apparatus design, the condition of the apparatus used, and the operational procedure carried out. Flash point can therefore only be defined in terms of a standard test method, and no general valid correlation can be guaranteed between results obtained by different test methods or with test apparatus different from that specified.

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Determination of flash point — Rapid equilibrium closed cup 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 for the determination of the closed cup flash point of paints (including water-borne paints), varnishes, paint binders, adhesives, solvents, petroleum, and related products having closed cup flash points within the range of $-30\text{ }^{\circ}\text{C}$ to $300\text{ }^{\circ}\text{C}$. When used in conjunction with the flash detector (A.1.6), this International Standard is also suitable for the determination of the flash point of fatty acid methyl esters (FAME).

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2 Normative references (standards.iteh.ai)

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

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

ISO 3170:2004, *Petroleum liquids — Manual sampling*

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

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

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

3.1

flash point

lowest temperature of the test portion (as measured in the prescribed manner), corrected to a barometric pressure of 101,3 kPa, at which application of a test flame causes the vapour of the test portion to ignite momentarily and the flame to propagate across the surface of the liquid under the specified conditions of test

4 Principle

A test portion of specified volume is introduced into the test cup, which is maintained at the temperature of the estimated flash point of the material under test. After a specified time, a test flame is applied and the presence or absence of a flash observed. Further tests, with fresh test portions at different temperatures, are carried out until the flash point is determined to the sensitivity specified.

5 Reagents and materials

5.1 Cleaning solvent

An appropriate solvent for the 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

A series of certified reference materials (CRM) and/or secondary working standards (SWS) as described in Annex C.

6 Apparatus

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6.1 General

The apparatus is described in Annex A, and this includes the details of the test cup and cover assembly, together with dimensions and special requirements. These are shown in Figure A.1 to Figure A.5. The measurement of flash points over the whole range of $-30\text{ }^{\circ}\text{C}$ to $300\text{ }^{\circ}\text{C}$ may require more than one instrument.

6.2 Syringes

6.2.1 2 ml syringe, adjusted to deliver $2,00\text{ ml} \pm 0,05\text{ ml}$ and equipped, if required, with a needle suitable for use with the apparatus at test temperatures up to and including $100\text{ }^{\circ}\text{C}$. When testing FAME, a 2 ml sample is used at all temperatures.

6.2.2 5 ml syringe, adjusted to deliver $4,00\text{ ml} \pm 0,10\text{ ml}$ and equipped, if required, with a needle suitable for use with the apparatus at test temperatures above $100\text{ }^{\circ}\text{C}$. A 5 ml syringe is not required when testing FAME.

6.3 Barometer, accurate to $0,1\text{ kPa}$. Barometers precorrected to give sea-level readings, such as those used at weather stations and airports, shall not be used.

6.4 Heating bath or oven (optional), for warming the samples, if required, and capable of controlling the temperature to $\pm 5\text{ }^{\circ}\text{C}$. If an oven is used, it shall be intrinsically safe for hydrocarbon vapours.

It is recommended that the oven be of explosion-protected design.

6.5 Cooling bath or freezer (optional), for cooling the samples, if required, and capable of cooling the sample to $10\text{ }^{\circ}\text{C}$ below the expected flash point, and controlling the temperature to $\pm 5\text{ }^{\circ}\text{C}$. If a freezer is used, it shall be of an explosion-protected design.

6.6 Draught shield (optional), if required to minimize draughts, fitted at the back and on two sides of the instrument.

NOTE A shield, 350 mm high, 480 mm wide and 240 mm deep, is suitable.

6.7 Cup insert (optional). See Annex D.

NOTE For samples that are difficult to remove, a thin metal cup insert can be used.

7 Apparatus preparation

7.1 General

Select the appropriate instrument for the expected flash point temperature. Follow the manufacturer's instructions for the correct set-up and operation of the apparatus. The use of a cup insert (6.7) for potentially adherent materials is described in Annex D.

7.2 Location of apparatus

Support the apparatus (Annex A) on a level and steady surface in a draught-free position.

Use of a draught shield (6.6) is recommended when protection from draughts is not available.

NOTE When testing materials which may produce toxic vapours, the apparatus may be located in a fume hood with an individual control of air flow, adjusted such that the vapours can be withdrawn without causing air currents around the test cup during the test.

7.3 Cleaning of the test cup and accessories

Clean the test cup, cover and its accessories with an appropriate solvent (5.1) to remove traces of gum or residue from the previous test. Follow the manufacturer's instructions for the care and servicing of the instrument.

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NOTE 1 A stream of clean dry air may be used to remove the last traces of solvent used.

NOTE 2 The filler orifice may be conveniently cleaned with a pipe cleaner.

7.4 Apparatus verification

7.4.1 Verify the correct functioning of the apparatus at least once a year by testing a certified reference material (CRM) (5.2). The result obtained shall be equal to or less than $R/\sqrt{2}$ from the certified value of the CRM, where R is the reproducibility of the method (see 13.3).

It is recommended that more frequent verification checks be made using secondary working standards (SWSs) (5.2).

NOTE A recommended procedure for apparatus verification using CRMs and SWSs, and the production of SWSs, is given in Annex C.

7.4.2 The numerical values obtained during the verification check shall not be used to provide a bias statement, nor shall they be used to make any correction to the flash points subsequently determined using the apparatus.

If the instrument fails the verification test, it is recommended that the operator should check that:

- a) the cover makes a vapour-tight seal with the test cup;
- b) the shutter provides a light-tight seal;
- c) adequate heat-transfer paste surrounds the thermometer bulb and immersed portion of the thermometer barrel.

8 Sampling

8.1 Unless otherwise specified, obtain samples in accordance with the procedures given in ISO 15528, ISO 3170 or ISO 3171 or an equivalent national standard.

8.2 Place sufficient sample volume for testing in a tightly sealed container made of material appropriate to the liquid being sampled, and for safety purposes, ensure that the sample container is only filled to between 85 % and 95 % of its capacity.

If more than one determination is anticipated, the volume of the subsample should be selected so that the conditions of 9.1.1 apply.

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 and fatty acid methyl esters

9.1.1 Subsampling. Cool in a cooling bath or freezer (6.5), or adjust the temperature of the sample and its container to at least 10 °C below the first selected test temperature before opening to remove the test portion. If an aliquot of the original sample is to be stored prior to testing, ensure that the container is still filled to at least 85 % of its capacity. Gently mix the subsample to ensure uniformity, so that the loss of volatile components and light ends is minimized.

NOTE Results of flash point tests may be affected if the sample volume falls below 85 % of the container capacity.

9.1.2 Samples that are liquid at ambient temperature. If sufficiently fluid, mix samples gently shaking by hand prior to the removal of the test portion, taking care to minimize the loss of volatile components. If the sample is too viscous at ambient temperatures, heat the sample in its container using a heating bath or oven (6.4) to a temperature not warmer than 10 °C below the test temperature, such that the sample can be mixed by gentle shaking.

9.1.3 Solid or semi-solid samples. If the material under test cannot be made sufficiently fluid to be introduced into the test cup through the orifice by heating in accordance with 9.1.2, transfer the test portion with a solids dispenser or spatula into the test cup whilst the cover is open.

9.2 Paints, varnishes and related materials

Prepare the samples in accordance with the procedures described in ISO 1513.

10 Procedure

10.1 General

10.1.1 Follow the manufacturer's instructions for setting the test temperature to the required flash point temperature.

10.1.2 When testing fatty acid methyl esters (FAME), the flash point detector (A.1.6) shall be used.

10.1.3 Do not apply the test flame to the test portion more than once. Use a new test portion of the sample for each test. After each test, turn off the pilot and test flames using the gas control valve, and when the test cup temperature falls to a safe level, remove the test portion and clean the instrument.

10.1.4 Do not confuse the true flash point with the bluish halo that sometimes surrounds the test flame at applications preceding that which causes the actual flash.

NOTE The optional flash detector (A.1.6) is not affected by the halo, and does not require the operator to closely observe the flash point test.

10.1.5 Record the ambient barometric pressure using a barometer (6.3) in the vicinity of the apparatus at the time of the test.

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

10.2 Procedure for flash points up to and including 100 °C and for FAME at all temperatures

10.2.1 Charge a clean and dry syringe (6.2.1), adjusted to a temperature at least 10 °C below the expected flash point, with a 2 ml test portion. Close the sample container immediately after withdrawal to minimize the loss of volatile components.

10.2.2 Carefully transfer the syringe to the filler orifice and discharge the test portion into the test cup by fully depressing the syringe plunger. Remove the syringe.

10.2.3 For solid or semi-solid samples, transfer a mass equivalent to approximately 2 ml directly into the test cup and spread it over the bottom of the cup as evenly as possible.

10.2.4 Start the 1 min timing device (A.1.3). Open the gas control valve and ignite the pilot and test flames. Adjust the test flame to conform to the size of the 4 mm gauge ring. Reset the flash detector (A.1.6) if fitted.

10.2.5 When the audible time signal sounds, apply the test flame by slowly and uniformly opening and then closing the shutter over a period of 2 s to 3 s. Check for a flash (see 10.1.4).

If a continuous luminous flame burns in the orifice when the slide is opened and the test flame is introduced, then the flash point lies considerably below the test temperature. In such cases, a reduction of the test temperature by 10 °C is recommended.

10.2.6 If a flash is observed, repeat the procedure given in 10.2.1 to 10.2.5 with a fresh test portion, starting at a temperature 5 °C lower than that at which the flash was observed. If a flash is still observed at this lower temperature, lower the temperature a further 5 °C and repeat again. Repeat until no flash is observed.

10.2.7 If no flash is observed, repeat the procedure given in 10.2.1 to 10.2.5 with a fresh test portion, starting at a temperature 5 °C higher than that at which the last test portion was tested. Repeat tests at 5 °C higher intervals until a flash is observed.

10.2.8 Repeat the procedure given in 10.2.1 to 10.2.5, using a fresh test portion for each test, at 1 °C intervals, starting at the lower of the intervals 5 °C apart established in 10.2.6 and 10.2.7, until a flash is observed. Record the temperature on the thermometer as the observed flash point using 1 °C intervals. If improved sensitivity is required, test a fresh test portion at a temperature 0,5 °C below that at which a flash was observed using 1 °C intervals. If no flash is observed, then the temperature recorded using 1 °C intervals is the observed flash point to the nearest 0,5 °C. If a flash is observed, then this new reading is the observed flash point.

10.3 Procedure for flash points above 100 °C [except when testing FAME (see 10.2)]

10.3.1 Charge a clean and dry syringe (6.2.2) with a 4 ml test portion. Close the sample container immediately after withdrawal to minimize the loss of volatile components.

10.3.2 Carefully transfer the syringe to the filling orifice and discharge the test portion into the test cup by fully depressing the syringe plunger. Remove the syringe.