
**Determination of flash point — Pensky-
Martens closed cup method**

Détermination du point d'éclair — Méthode Pensky-Martens en vase clos

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Reference number
ISO 2719:2002(E)

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Printed in Switzerland

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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.

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.

International Standard ISO 2719 was prepared by Technical Committee ISO/TC 28, *Petroleum products and lubricants*.

This third edition cancels and replaces the second edition (ISO 2719:1988), which has been technically revised.

Annexes B and C form a normative part of this International Standard. Annexes A and D are for information only.

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Introduction

Flash point values may be used in shipping, storage, handling and safety regulations, as a classification property to define “flammable” and “combustible” materials. Precise definition of the classes is given in each particular regulation.

A flash point value may indicate the presence of highly volatile material(s) in a relatively non-volatile or non-flammable material and flash point testing may be a preliminary step to other investigations into the composition of unknown materials.

Flash point determinations should not be carried out on potentially unstable, decomposable, or explosive materials, unless it has been previously established that heating the specified quantity of such materials in contact with the metallic components of the flash point apparatus within the temperature range required for the method will not induce decomposition, explosion or other adverse effects.

The interpretation of flash point results obtained on material containing halogenated hydrocarbons should be considered with caution, as these mixtures can give anomalous results.

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Determination of flash point — Pensky-Martens 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 describes two procedures, A and B, using the Pensky-Martens closed cup tester, for determining the flash point of combustible liquids, liquids with suspended solids, liquids that tend to form a surface film under the test conditions and other liquids. It is applicable for liquids with a flash point above 40 °C.

NOTE 1 Although technically kerosines with a flash point above 40 °C may be tested using this International Standard, it is standard practice to test kerosines according to ISO 13736^[8]. Similarly, unused lubricating oils are normally tested according to ISO 2592^[5].

Procedure A is used for the determination of the flash point of paints and varnishes that do not form a surface film, unused lubricating oils and other petroleum products not covered by Procedure B.

Procedure B is used for the determination of the flash point of residual fuel oils, cutback bitumens, used lubricating oils, liquids that tend to form a surface film, liquids with suspensions of solids and highly viscous materials such as polymeric solutions and adhesives.

NOTE 2 For the comparison of the flash points of used and unused lubricating oils, such as in a lubricant monitoring scheme, used lubricating oils may be tested using Procedure A. However, the precision data for these materials is only valid for Procedure B.

This International Standard is not applicable to water-borne paints or liquids contaminated by traces of highly volatile materials.

NOTE 3 Water-borne paints may be tested using ISO 3679^[6]. Liquids contaminated by traces of highly volatile materials may be tested using ISO 1523^[4] or ISO 3679.

NOTE 4 Precision data is only valid for the flash point ranges given in clause 13.

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 3170:—¹⁾, *Petroleum liquids — Manual sampling*

1) To be published. (Revision of ISO 3170:1988)

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

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

3 Term and definition

For the purposes of this International Standard, 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 the 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 the test cup of a Pensky-Martens apparatus and heated to give a constant temperature increase with continuous stirring. An ignition source is directed through an opening in the test cup lid at regular temperature intervals with simultaneous interruption of stirring. The lowest temperature at which the application of the ignition source causes the vapour of the test portion to ignite and propagate over the surface of the liquid is recorded as the flash point at the ambient barometric pressure. This temperature is corrected to standard atmospheric pressure using an equation.

5 Chemicals and materials

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5.1 Cleaning solvent, for the removal of traces of sample 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 efficacious for the removal of gum-type deposits.

5.2 Verification liquids, as described in annex A.

6 Apparatus

6.1 Flash point apparatus, Pensky-Martens closed cup test apparatus as specified in annex B.

If automated equipment is used, ensure that it has been established that the results obtained are within the precision of this International Standard and that the test cup and cover assembly conform to the dimensional and mechanical requirements as specified in annex B. If automated testers are used, the user shall ensure that all of the manufacturer's instructions for adjusting and operating the instrument are followed.

NOTE Under certain circumstances, the use of electric ignition sources may give different results to those obtained when using a flame ignition source. In addition, electric ignition sources may give variable results.

In cases of dispute, unless explicitly agreed otherwise, the manual determination of the flash point using a flame ignition source shall be considered as the referee test.

6.2 Thermometers, low, medium and high range, conforming to annex C. The initial choice of thermometer shall be based on the expected flash point.

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 annex C.

6.3 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.

6.4 Heating bath or oven, for warming the sample if required, capable of controlling the temperature to ± 5 °C. The oven shall be ventilated and constructed in such a way that it will not cause ignition of any flammable vapours that may be produced when the sample is heated.

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

7 Apparatus preparation

7.1 Location of the apparatus

Support the apparatus (6.1) on a level and steady surface in a draught-free position (see the notes below).

NOTE 1 When draughts cannot be avoided, it is good practice to surround the apparatus with a shield.

NOTE 2 When testing samples which produce toxic vapours, the apparatus may be located in a fume hood with an individual control of air flow, adjusted so that vapours can be withdrawn without causing air currents around the test cup during the test.

7.2 Cleaning the test cup

Wash the test cup, cover and its accessories with an appropriate solvent (5.1) to remove any traces of gum or residue remaining from a previous test. Dry using a stream of clean air to ensure complete removal of the solvent used.

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7.3 Apparatus assembly

Examine the test cup, the cover and other parts to ensure that they are free from signs of damage and deposits. Assemble the apparatus in accordance with annex B.

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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) using Procedure A. 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 Table 3).

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

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

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.

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 % to 95 % of its capacity.

8.3 Store the samples in conditions that 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

Subsample at a temperature at least 28 °C below the expected flash point. If an aliquot of the original sample is to be stored prior to testing, ensure that the container is filled to more than 50 % of its capacity (see note to 10.1).

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 in accordance with ISO 3679^[6].

9.1.3 Samples that are liquid at ambient temperature

Mix samples by gentle manual shaking prior to the removal of the test portion, taking care to minimize the loss of volatile components, and proceed in accordance with clause 10.

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 (6.4) to 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 the loss of volatile components. After gentle agitation, proceed in accordance with clause 10.

9.2 Paints and varnishes

Prepare the samples in accordance with ISO 1513.

10 Procedure

10.1 General

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

Care should be taken when testing samples of residual fuel oil which contain significant amounts of water, as heating these samples may cause them to foam and eject from the test cup.

10.2 Procedure A

10.2.1 Using a barometer (6.3), record the ambient barometric pressure in the vicinity of the apparatus at the time of test.

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

10.2.2 Fill the test cup (see 7.3) with the test portion to the level indicated by the filling mark. Place the lid on the test cup and put it in the heating chamber. Ensure that the locating or locking device is properly engaged and insert the

thermometer (6.2). Light the test flame and adjust to a diameter of 3 mm to 4 mm, or switch on the alternative ignition source. Light the heater flame or switch on the electric heater and supply heat at such a rate that the temperature of the test portion as indicated by the thermometer increases at 5 °C/min to 6 °C/min, and maintain this heating rate throughout the test. Stir the test portion at a rate of 90 r/min to 120 r/min, stirring in a downward direction.

10.2.3 When the test portion is expected to have a flash point of 110 °C or below, make the first application of the ignition source when the temperature of the test portion is 23 °C ± 5 °C below the expected flash point, and thereafter at 1 °C temperature intervals. Cease stirring and apply the ignition source by operating the mechanism on the cover, which controls the shutter and ignition source, so that the source is lowered into the vapour space of the test cup in 0,5 s, left in its lowered position for 1 s, and quickly raised to its high position.

10.2.4 When the test portion is expected to have a flash point of above 110 °C, make the first application of the ignition source when the temperature of the test portion is 23 °C ± 5 °C below the expected flash point, and thereafter at temperatures which are a multiple of 2 °C. Cease stirring and apply the ignition source by operating the mechanism on the cover, which controls the shutter and ignition source, so that the source is lowered into the vapour space of the test cup in 0,5 s, left in its lowered position for 1 s, and quickly raised to its high position.

10.2.5 When testing a material of unknown flash point, conduct a preliminary test at a suitable starting temperature. Make the first ignition-source application at 5 °C above the starting temperature and follow the procedure given in 10.2.3 or 10.2.4, as applicable.

10.2.6 Record, as the observed flash point, the temperature of the test portion read on the thermometer at the time when the ignition-source application causes a distinct flash in the interior of the test cup. Do not confuse the true flash point with the bluish halo that sometimes surrounds the ignition source at applications preceding the actual flash point.

10.2.7 When the temperature at which the flash point is observed is less than 18 °C, or greater than 28 °C, from the temperature of the first application of the ignition source, the result is not valid. Repeat the test using a fresh test portion, adjusting the temperature of the first application of the ignition source until a valid determination is obtained, that is where the flash point is 18 °C to 28 °C above the temperature of the first application of the ignition source.

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10.3 Procedure B

10.3.1 Using a barometer (6.3), record the ambient barometric pressure in the vicinity of the apparatus at the time of test (see the note to 10.2.1).

10.3.2 Fill the test cup (see 7.3) with the test portion to the level indicated by the filling mark. Place the lid on the test cup and put it in the heating chamber. Ensure that the locating or locking device is properly engaged and insert the thermometer (6.2). Light the test flame and adjust to a diameter of 3 mm to 4 mm, or switch on the alternative ignition source. Light the heater flame or switch on the electric heater and supply heat at such a rate that the temperature of the test portion as indicated by the thermometer increases at 1,0 °C/min to 1,5 °C/min, and maintain this heating rate throughout the test. Stir the test portion at a rate of 250 r/min ± 10 r/min, stirring in a downward direction.

10.3.3 With the exception of the requirements given in 10.3.2 for rate of heating and stirring, proceed in accordance with 10.2.3 to 10.2.7.

11 Calculation

11.1 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 expressions:

$$\text{Reading in hPa} \times 0,1 = \text{kPa}$$

$$\text{Reading in mbar} \times 0,1 = \text{kPa}$$

$$\text{Reading in mmHg} \times 0,1333 = \text{kPa}$$