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

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

ICS: 75.080

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### ISO/CEN PARALLEL PROCESSING

This draft has been developed within the International Organization for Standardization (ISO), and processed under the **ISO lead** mode of collaboration as defined in the Vienna Agreement.

This draft is hereby submitted to the ISO member bodies and to the CEN member bodies for a parallel five month enquiry.

Should this draft be accepted, a final draft, established on the basis of comments received, will be submitted to a parallel two-month approval vote in ISO and formal vote in CEN.

To expedite distribution, this document is circulated as received from the committee secretariat. ISO Central Secretariat work of editing and text composition will be undertaken at publication stage.

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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 2719 was prepared by Technical Committee ISO/TC 28, *Petroleum products and related products of synthetic or biological origin*, Working Group 9, in conjunction with Technical Committee ISO/TC 35, *Paints and varnishes*, Technical Committee CEN/TC 19, *Gaseous and liquid fuels, lubricants and related products of petroleum, synthetic and biological origin*, and Technical Committee CEN/TC 139, *Paints and varnishes*.

This third edition cancels and replaces the second edition (ISO 2719:2002).

The main technical updates are:

- a) Introduction of procedure C for FAME products,
- b) Revision of temperature measuring device requirements, allowing alternatives for Hg-containing thermometers,
- c) Removal of the original Annex D on the adaptor for the low-range thermometer,
- d) Revision of procedures regarding sampling and sample handling, and
- e) Inclusion, for automated apparatus, manufactured after 1 January 2017, of a device to automatically dispense an inert gas or vapour over the test cup in the event of a test cup fire.

## Introduction

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

It is not appropriate for flash point determinations to 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, does not induce decomposition, explosion or other adverse effects.

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 be defined only 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 differ rent from that specified.

ISO TR 29662<sup>[1]</sup> (CEN/TR 15138<sup>[2]</sup>) gives useful advice in carrying out flash point tests and interpreting their 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 three procedures, A, B and C, 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, biodiesel and other liquids in the temperature range of 40 °C to 370 °C.

**CAUTION** — For certain mixtures no flash point, as defined, is observed; instead a significant enlargement of the test flame (not halo effect) and a change in colour of the test flame from blue to yellowish-orange can occur. Continued heating can result in significant burning of vapours outside the test cup, and can be a potential fire hazard.

NOTE 1 Although, technically, kerosenes with a flash point above 40 °C can be tested using this International Standard, it is standard practice to test kerosenes according to ISO 13736<sup>[3]</sup>. Similarly, lubricating oils are normally tested according to ISO 2592<sup>[4]</sup>.

Procedure A is applicable to distillate fuels (diesel, biodiesel blends, heating oil and turbine fuels), new and in-use lubricating oils, paints and varnishes, and other homogeneous liquids not included in the scope of Procedures B or C.

Procedure B is applicable to residual fuel oils, cutback residua, used lubricating oils, mixtures of liquids with solids, liquids that tend to form a surface film under test conditions or are of such kinematic viscosity that they are not uniformly heated under the stirring and heating conditions of Procedure A.

Procedure C is applicable to fatty acid methyl esters (FAME) as specified in specifications such as EN 14214<sup>[5]</sup> or ASTM D 6751<sup>[6]</sup>.

This International Standard is not applicable to water-borne paints and varnishes.

NOTE 2 Water-borne paints and varnishes can be tested using ISO 3679<sup>[7]</sup>. Liquids containing traces of highly volatile materials can be tested using ISO 1523<sup>[8]</sup> or ISO 3679.

## 2 Normative references

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

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

ISO 3170, *Petroleum liquids — Manual sampling*.

ISO 3171, *Petroleum liquids — Automatic pipeline sampling*.

ISO 15528, *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, 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 into 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 a flame propagate over the surface of the liquid is recorded as the flash point at the absolute barometric pressure. This temperature is corrected to standard atmospheric pressure using the specified equation.

### 5 Chemicals and materials

**5.1 Cleaning solvent**, for removal of traces of sample from the test cup and cover.

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 can be efficacious for the removal of gum-type deposits.

**5.2 Liquids for verification**, see Annex A.

### 6 Apparatus

**6.1 Flash point apparatus**, as described in Annex B.

If automated equipment is used, ensure that the test cup and cover assembly conform to the key dimensions specified in Annex B, and the procedure described in Clause 10 is followed. 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 can give different results to those obtained when using a flame ignition source.

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

**6.2 Temperature measuring device**, meeting the requirements for accuracy and have the response as specified in Annex C.

The aperture in the cover assembly and the fitting required to hold the temperature measuring device may be modified to closely fit the temperature measuring device utilized.

**6.3 Barometer, absolute pressure reading**, accuracy of  $\pm 0,5$  kPa, and with a resolution of 0,1 kPa.

Barometers pre-corrected to give sea level readings, such as those used at weather stations and airports, shall not be used.



NOTE Some automated apparatus include an integral barometer that automatically measures and records the absolute barometric pressure, and makes the required corrections to the detected flash point.

**6.4 Heating bath or oven, capable of controlling the temperature to  $\pm 5$  °C**, for warming the sample if required.

The oven shall be ventilated and constructed in such a way that it will not cause ignition of any flammable vapours that can be produced when the sample is heated.

It is recommended that the oven is an explosion-protected type.

## 7 Apparatus preparation

### 7.1 General

Follow the manufacturer's instructions for the correct set-up, calibration, verification (7.5) and operation of the apparatus including the integral barometer (if fitted) and temperature measuring device (see Annex C), especially the operation and setting of the ignition source,

### 7.2 Location of the apparatus

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

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

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.

### 7.3 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.

### 7.4 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.

### 7.5 Apparatus verification

**7.5.1** 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 as shown in Table 4.

**7.5.1.1** Use the reproducibility for Procedure A (Table 4) to calculate the verification tolerance for verification materials certified for Procedures A, B or C.

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

A recommended procedure for apparatus verification using CRMs and SWSS, and the production of SWSS, is described in Annex A.

**7.5.2** 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 representative samples for analysis in accordance with the procedures given in ISO 3170, ISO 3171, ISO 15528 or an equivalent National Standard.

**8.2** When obtaining a sample of residual fuel oil, the sample container shall be from 85 % to 95 % full. For FAME samples, a typical one litre container filled to 85 % volume is recommended. For other types of samples, the size of the container shall be chosen such that the container is not more than 85 % full or less than 50 % full prior to any sample aliquot being taken.

**8.3** Erroneously high flash points can be obtained if precautions are not taken to avoid the loss of volatile material. Do not open containers unnecessarily, to prevent loss of volatile material or possible introduction of moisture. Avoid storage of samples at temperatures in excess of 30 °C. Samples for storage shall be capped tightly with inner seals. Do not make a transfer unless the sample temperature is at least the equivalent of 18 °C below the expected flash point.

**8.4** Do not store samples in gas-permeable containers, since volatile material may diffuse through the walls of the enclosure. Samples in leaky containers are suspect and not a source of valid results.

## 9 Sample handling

### 9.1 Petroleum products

#### 9.1.1 Subsampling

**9.1.1.1** Sub sample at a temperature at least 18 °C below the expected flash point.

**9.1.1.2** Successive test portions may be taken from the same sample container. Repeat tests have been shown to be within the precisions of the method when the second test portion is taken with the sample container at least 50 % filled (See Note 1 to 10.1.1)

#### 9.1.2 Samples containing undissolved water

**9.1.2.1** Flash point results can be affected by the presence of water, if a sample contains undissolved water, decant an aliquot from the water prior to mixing.

**9.1.2.2.** For certain fuel oils and lubricants it is not always possible to decant the sample from the free water. In such cases, separate the water from the aliquot physically, prior to mixing, or, if this is not possible, test the material in accordance with ISO 3679 <sup>[7]</sup>.

#### 9.1.3 Samples that are liquid at ambient temperature

Mix samples by gentle manual shaking prior to 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 very viscous, semi- solid or solid at ambient temperature.

Samples shall be heated in their containers, with lid/cap slightly loosened to avoid build-up of dangerous pressure, at the lowest temperature adequate to liquefy any solids, not exceeding 18 °C below the expected flash point, for 30 min. If the sample is then not completely liquefied, extend the heating period for additional 30 min periods as necessary. Then gently agitate the sample to provide mixing, such as orbiting the container horizontally, before transferring to the test cup. No sample shall be heated and transferred unless its temperatures is more than 18 °C below its expected flash point.

**IMPORTANT — Volatile vapours can escape during heating when the sample container lid/cap is too loose.**

## 9.2 Paints and varnishes

Prepare the samples in accordance with ISO 1513.

## 10 Procedure

### 10.1 General

**10.1.1** The applicability of the three procedures A, B and C is given in the Scope.

Electronic flash point detection shall be used for this procedure as it is difficult to observe a flash by visual means.

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

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

**10.1.2** As a safety practice it is strongly advised to apply the ignition source to the test portion in the cup before heating the test cup and test portion, to check for the presence of unexpected volatile material.

**10.1.3** As a safety practice, it is strongly advised that, for an expected flash point above 130 °C, to dip the ignition source every 10 °C throughout the test until the sample temperature reaches 28 °C below the expected flash point and then follow the prescribed dipping procedure. This practice has been shown to reduce the possibility of a fire, and not to significantly affect the result.

**10.1.4** At the end of a test, when the apparatus has cooled down to a safe handling temperature, remove the test cover and the test cup and clean the apparatus as recommended by the manufacturer.

### 10.2 Procedure A

**10.2.1** Record the laboratory absolute barometric pressure at the time of test.(see 6.3)

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 them in the heating chamber. Ensure that the locating or locking device is properly engaged and insert the temperature measuring device (6.2). Light the test flame and adjust to a diameter of 3,2 mm to 4,8 mm or enable the alternative ignition source. Supply heat at such a rate that the temperature of the test portion, as indicated by the temperature measuring device, increases at 5,0 °C/min to 6,0 °C/min, and maintain this heating rate throughout the test. Stir the test portion at a rate of plus minus 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, that 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 that are a multiple of 2 °C. Cease stirring and apply the ignition source by operating the mechanism on the cover, that 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.