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

Détermination du point d'éclair — Méthode Pensky-Martens 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.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation on the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see the following URL: www.iso.org/iso/foreword.html.

The committee responsible for this document is ISO/TC 28, *Petroleum products and related products of synthetic or biological origin*, in conjunction with ISO/TC 35, *Paints and varnishes*.

This fourth edition cancels and replaces the third edition (ISO 2719:2002), which has been technically revised.

The main technical updates include:

- 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, it being optional when a permanent ferrule is attached to the thermometer;
- d) revision of procedures regarding sampling and sample handling;
- 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 different from that specified.

ISO/TR 29662^[6] (CEN/TR 15138^[12]) gives useful advice in carrying out flash point tests and interpreting their results.

Determination of flash point — Pensky-Martens closed cup method

WARNING — The use of this International Standard can 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 users of this International Standard to take appropriate measures to ensure the safety and health of personnel prior to application of this International Standard, and fulfil statutory and regulatory requirements for this purpose.

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, kerosene with a flash point above 40 °C can be tested using this International Standard, it is standard practice to test kerosene according to ISO 13736.^[5] Similarly, lubricating oils are normally tested according to ISO 2592^[2].

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^[11] or ASTM D6751^[13].

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^[3]. Liquids containing traces of highly volatile materials can be tested using ISO 1523^[4] 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 test samples*

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 standard 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 a specified formula.

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 devices, meeting the requirements for accuracy and have the response as specified in [Annex C](#).

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 ± 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 setup, 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 flash point 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 flash point apparatus may be located in a fume hood with individual air flow control, 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 cleaning 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.

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. For samples for storage, ensure that the sample container is tightly closed and leak free. 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 Sub-sampling

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 precision of the method when the second test portion is taken with the sample container at least 50 % filled.

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

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 a water-free aliquot 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[3].

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 temperature 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 explained in [Clause 1](#).

Electronic flash point detection shall be used for procedure C, as it is difficult to observe a flash by visual means, and may be used for procedures A and B.

10.1.2 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.

10.1.3 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.

10.1.4 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.

10.1.5 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 reading 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 rotational rate of 90 r/min (1,5 Hz) to 120 r/min (2,0 Hz), 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.

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 detected flash point, the temperature of the test portion read on the temperature measuring device at the time when 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 a flash point is detected on the first application, the test shall be discontinued, the result discarded, and the test repeated with a fresh test portion. The first application of the ignition source with the fresh test portion is recommended to be approximately 23 °C below the temperature at which a flash point was detected on the first application.

10.2.8 When the temperature at which the flash point is detected is less than 18 °C or greater than 28 °C from the temperature of the first application of the ignition source, the result shall be considered approximate. 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.

10.3 Procedure B

10.3.1 Record the laboratory absolute barometric pressure at the time of test (see [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 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 1,0 °C/min to 1,5 °C/min, and maintain this heating rate throughout the test. Stir the test portion at a rotational rate of (250 ±10) r/min, stirring in a downward direction.

10.3.3 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.

10.3.4 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.

10.3.5 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 [10.3.3](#) or [10.3.4](#), as applicable.

10.3.6 Record, as the detected flash point, the temperature of the test portion read on the temperature measuring device at the time when 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.

10.3.7 When a flash point is detected on the first application, the test shall be discontinued, the result discarded, and the test repeated with a fresh test portion. The first application of the ignition source with the fresh test portion is recommended to be approximately 23 °C below the temperature at which a flash point was detected on the first application.

10.3.8 When the temperature at which the flash point is detected is less than 18 °C or greater than 28 °C from the temperature of the first application of the ignition source, the result shall be considered approximate. 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.

10.4 Procedure C

10.4.1 Record the laboratory absolute barometric pressure at the time of test (see [10.2.1](#)).

10.4.2 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 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 2,5 °C/min to 3,5 °C/min, and maintain this heating rate throughout the test. Stir the test portion at a rotational rate of 90 r/min to 120 r/min, stirring in a downward direction.

10.4.3 Use an expected flash point of 100 °C for the first test on the sample.

10.4.4 Make the first application of the ignition source when the temperature of the test portion is approximately 24 °C below the expected flash point and each time thereafter at a temperature reading that is 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.

10.4.5 Record as the detected flash point the temperature of the test portion read on the temperature measuring device at the time when the ignition source application causes a distinct flash in the interior of the test cup.

10.4.6 When a flash point is detected on the first application, the test shall be discontinued, the result discarded, and the test repeated with a fresh test portion. The first application of the ignition source with the fresh test portion is recommended to be approximately 24 °C below the temperature at which a flash point was detected on the first application.

10.4.7 When the temperature at which the flash point is detected is less than 16 °C or greater than 30 °C from the temperature of the first application of the ignition source, the result shall be considered approximate. 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 16 °C to 30 °C above the temperature of the first application of the ignition source.

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 conversions:

- Reading in hPa $\times 0,1 =$ kPa,
- Reading in mbar $\times 0,1 =$ kPa, or
- Reading in mmHg $\times 0,133\ 3 =$ kPa.

11.2 Correction of observed flash point to standard atmospheric pressure

Calculate the corrected flash point, T_c , using [Formula \(1\)](#):

$$T_c = T_d + 0,25 \times (101,3 - p) \quad (1)$$

where

- T_d is the detected flash point, expressed in °C;
- p is the absolute barometric pressure, expressed in kPa;
- 0,25 is a constant, expressed in °C/kPa;
- 101,3 is used as the standard atmospheric pressure, expressed in kPa.

NOTE The above formula has been proven for barometric pressures down to 82,0 kPa[[17]] and is strictly correct only up to 104,7 kPa.

12 Expression of results

Report the flash point, corrected to standard atmospheric pressure, rounded to the nearest 0,5 °C.

13 Precision

13.1 General

The precision, as determined by statistical examination according to ISO 4259[4] of interlaboratory test results, is given in [13.2](#) and [13.3](#).

NOTE The precision for in-process lubricating oils, such as those tested using Procedure A as part of a lubricant monitoring scheme, has not been determined.

13.2 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 and in normal and correct operation of the test method, exceed the values given in [Tables 1, 2 and 3](#) in only one case in 20.

Table 1 — Repeatability values for procedure A

Material	Flash point range °C	Repeatability, <i>r</i> °C
Paints and varnishes	—	1,5
Distillates and unused lubricating oils	40 to 250	0,029 <i>X</i> ^a

^a *X* is the average of the test results being compared. The precision data was developed in 1991 by ASTM D02[18] and in 1994 by the Energy Institute[19].

Table 2 — Repeatability values for procedure B

Material	Flash point range °C	Repeatability, <i>r</i> °C
Residual fuels and cutback bitumen	40 to 110	2 ^a
Used lubricating oils	170 to 210	5 ^b
Liquids that tend to form a surface film, liquids with suspensions of solids, highly viscous materials	—	5 ^c

^a The precision data was determined by the Energy Institute[19].
^b Data generated from one sample of used diesel fuel engine oil by 20 laboratories.
^c The precision data was determined by ASTM Committee D01.

Table 3 — Repeatability values for procedure C

Material	Flash point range °C	Repeatability, <i>r</i> °C
FAME (B100)	60 to 190	8,4

NOTE The precision data was determined by ASTM Committee D02[20].

13.3 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 values given in Tables 4, 5 and 6 in only one case in 20.

Table 4 — Reproducibility values for Procedure A

Material	Flash point range °C	Reproducibility, <i>R</i> °C
Paints and varnishes	—	No reproducibility data available
Distillates and unused lubricating oils	40 to 250	0,071 <i>X</i> ^a

^a *X* is the average of the test results being compared. The precision data was developed in 1991 by ASTM D02[18] and in 1994 by the Energy Institute[19].

Table 5 — Reproducibility values for Procedure B

Material	Flash point range °C	Reproducibility, <i>R</i> °C
Residual fuels and cutback bitumen	40 to 110	6 ^a
Used lubricating oils	170 to 210	16 ^b
Liquids that tend to form a surface film, liquids with suspensions of solids, highly viscous materials	—	10 ^c
^a The precision data was determined by the Energy Institute ^[19] . ^b Data generated from one sample of used diesel fuel engine oil by 20 laboratories. ^c The precision data was determined by ASTM Committee D01.		

Table 6 — Reproducibility values for Procedure C

Material	Flash point range °C	Reproducibility, <i>R</i> °C
FAME (B100)	60 to 190	14,7
NOTE The precision data was determined by ASTM Committee D02 ^[20] .		

14 Test report

The test report shall contain at least the following information:

- a) a reference to this International Standard, i.e. ISO 2719, and the procedure used;
- b) the type and complete identification of the sample tested;
- c) the pre-heat temperature and pre-heat time, if applicable (see [9.1.4](#));
- d) the ignitor type (gas or electric);
- e) the laboratory absolute barometric pressure (see [10.2.1](#), [10.3.1](#) and [10.4.1](#));
- f) the result of the test (see [Clause 12](#));
- g) any deviation, by agreement or otherwise, from the procedure specified;
- h) the date of the test.

Annex A (informative)

Apparatus verification

A.1 General

This Annex describes the procedure for conducting verification checks using either a secondary working standard (SWS) or a certified reference material (CRM) and includes a procedure for producing a secondary working standard.

The performance of the apparatus (manual or automated) should be verified on a regular basis using either a CRM produced in accordance with ISO Guide 34^[8] and ISO Guide 35,^[9] or an in-house reference material/SWS prepared in accordance with one of the procedures given in [A.2.2](#). Further guidance is given in ISO Guide 33^[7] and ISO 4259^[4].

The evaluation of the test result assumes a 95 % confidence limit for the trueness of the result.

A.2 Verification check standards

A.2.1 Certified reference material (CRM), comprising of a stable single hydrocarbon or other stable substance with a flash point determined in accordance with ISO Guide 34 and ISO Guide 35, using a method-specific inter-laboratory study to produce a method-specific certified value.

A.2.2 Secondary working standard (SWS), comprising a stable petroleum product or a single hydrocarbon or other stable substance with a flash point determined either by

- a) testing representative sub-samples at least three times using an instrument previously verified using a CRM, statistically analysing the results and, after the removal of any outliers, calculating the arithmetic mean of at least three results, or
- b) conducting an inter-laboratory method-specific test program utilizing at least three laboratories testing representative samples in duplicate. The assigned value of the flash point should be calculated after statistically analysing the inter-laboratory data.

Store SWSs in containers that will retain the integrity of the SWS, out of direct sunlight and at a temperature not exceeding 10 °C.

A.3 Procedure

A.3.1 Choose a CRM or SWS that falls within the range of flash points to be determined with the apparatus.

See [Table A.1](#) for approximate flash point values (acceptance limits for a single result). These are not the certified values nor the values that should be obtained and are given only for guidance.

It is recommended that two CRMs or SWSs are used in order to cover as wide a range as possible. In addition, it is also recommended that replicate tests are carried out on aliquots of the CRM or SWS.

A.3.2 For new apparatus, and at least once a year for working apparatus, conduct a verification check using a CRM ([A.2.1](#)) tested in accordance with [10.2](#), [10.3](#) or [10.4](#).

Table A.1 — Expected flash point values and tolerances for hydrocarbons (Procedure A)

Substance	Nominal flash point °C	Tolerances (see 7.5.1) $R / \sqrt{2}$	Source of expected flash point values
Decane	52,8	±2,6	a
Undecane	68,7	±3,2	a
Dodecane	84	±4,2	b
Tetradecane	109,3	±5,5	a
Hexadecane	133,9	±6,7	a

NOTE None of the flash point values are certified.

a Mean values from an interlaboratory test programme[[21]] and are based on materials with a >99 % purity.

b Historical figure.

A.3.3 For intermediate verification, conduct a verification check using an SWS (A.2.2) tested in accordance with 10.2, 10.3 or 10.4.

A.3.4 Correct the result for barometric pressure in accordance with 11.2. Record the corrected result, to the nearest 0,1 °C, in a permanent record.

A.4 Evaluation of test result

A.4.1 General

Compare the corrected test result(s) with the certified value of the CRM or the assigned value of the SWS.

The formulae given in A.4.2 and A.4.3 assume that reproducibility has been estimated in accordance with ISO 4259[4] and the certified value of the CRM, or the assigned value of the SWS, has been obtained by the procedures set out in A.2.1 and A.2.2, respectively, and that its uncertainty is small in comparison with the standard deviation of the test method and thus small compared with the reproducibility of the test method, R .

A.4.2 Single test

For a single test made on a CRM or SWS, the difference between a single result and the certified value of the CRM or the assigned value of the SWS should be within the following tolerance:

$$|x - \mu| \leq R / \sqrt{2} \tag{A.1}$$

where

x is the result of the test;

μ is the certified value of the CRM or the assigned value of the SWS;

R is the reproducibility of the test method.

A.4.3 Multiple tests

If a number of replicate tests, n , are made on a CRM or SWS, the difference between the mean of the n results and the certified value of the CRM or the assigned value of the SWS should be within the following tolerance:

$$|\bar{x} - \mu| \leq R_1 / \sqrt{2} \quad (\text{A.2})$$

where

\bar{x} is the mean of the test results;

μ is the certified value of the CRM or the assigned value of the SWS;

R_1 is equal to $\sqrt{\left\{R^2 - r^2 \left[1 - \left(1/n\right)\right]\right\}}$;

R is the reproducibility of the test method;

r is the repeatability of the test method;

n is the number of replicate tests carried out on the CRM or SWS.

A.4.4 Test conformance

If the test result conforms to the tolerance requirements, record this fact.

A.4.5 Test non-conformance

If the result does not conform to the tolerance requirements and an SWS has been used for the verification check, repeat using a CRM. If the result conforms to the tolerance requirements, record this fact and dispose of the SWS.

A.4.6 Troubleshooting

If the test result still does not conform to the tolerance requirements, examine the apparatus and check that it conforms to the apparatus specification requirements.

In particular, referring to the specific requirements of this test method and automated instrument manufacturers' instructions, check the size of the gas ignition source flame or temperature/setting of the electric ignition source, alignment of ignition source, flash detector (if fitted), test cup temperature measuring device calibration and immersion depth, test portion heating rate and correct operation of the shutter and dipping assembly.

If there is no obvious non-conformity, conduct a further verification check using a different CRM. If the result conforms to the tolerance requirements, record this fact. If it is still not within the required tolerances, contact the manufacturer.

Annex B (normative)

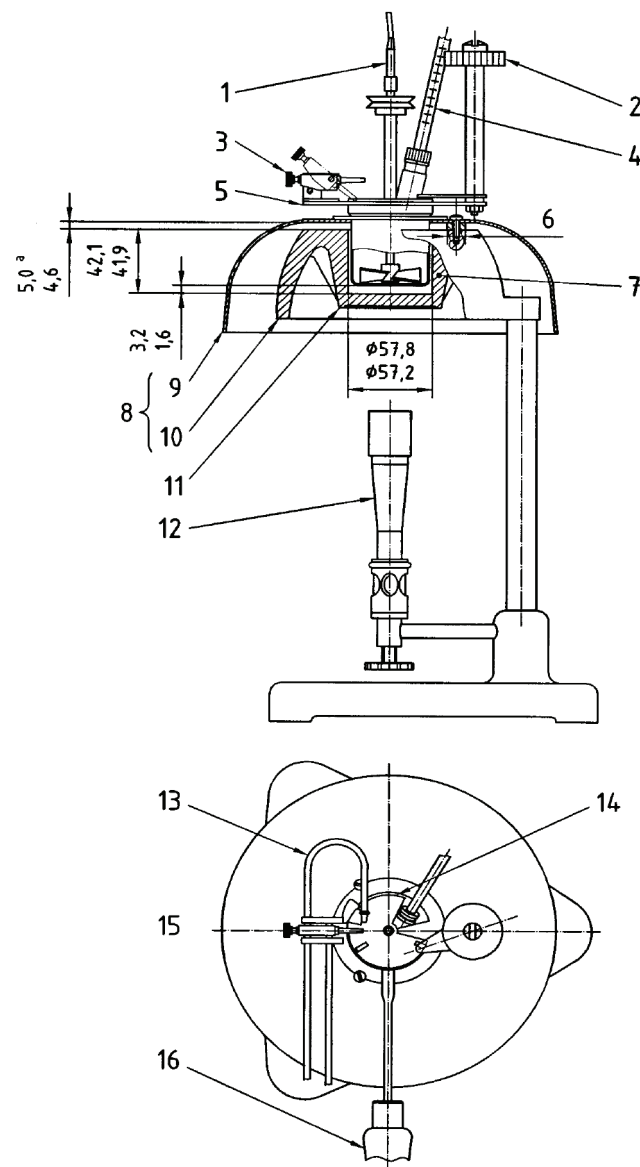
Pensky-Martens closed cup test apparatus

B.1 General

This Annex describes the manual, gas-/electrically heated, flame ignition device apparatus. This shall consist of a test cup, lid assembly and heating chamber as described below. A typical assembly of the apparatus, gas heated, is shown in [Figure B.1](#).

Automated equipment manufactured after 1 January 2017 shall be equipped with a device to automatically dispense an inert gas or vapour over the test cup in the event of a test cup fire.

Dimensions in millimetres



Key

- | | |
|---|---|
| 1 flexible shaft | 9 top plate |
| 2 shutter-operating knob | 10 air bath |
| 3 flame-ignition device | 11 6,5 mm minimum thickness over cup area, i.e. metal surrounding cup |
| 4 thermometer | 12 heater, flame-type or electric-resistance type (flame-type shown) |
| 5 lid | 13 pilot |
| 6 distance piece, $\varnothing 9,5$ mm max. | 14 shutter |
| 7 test cup | 15 front |
| 8 heating chamber | 16 handle (optional) |
| a Air gap. | |

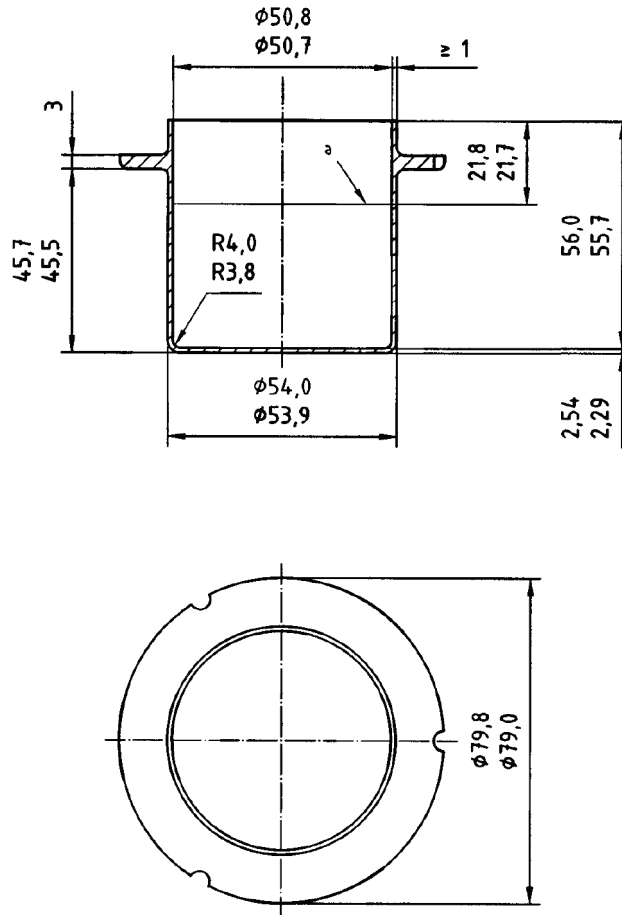
NOTE The lid assembly may be positioned either right-or left-handed.

Figure B.1 — Pensky-Martens closed cup apparatus

B.2 Test cup

The test cup shall be made of brass or other non-rusting metal of equivalent heat conductivity and shall conform to the dimensional requirements shown in [Figure B.2](#). The flange shall be equipped with devices for positioning the test cup in the heating chamber. A handle attached to the flange of the test cup is a desirable accessory, but shall not be so heavy as to tip over the empty test cup.

Dimensions in millimetres



Key

a Filling mark.

Figure B.2 — Test cup

B.3 Cover assembly

The cover assembly shall comprise the following items.

B.3.1 Lid, made of brass or other non-rusting metal of equivalent heat conductivity, and with a rim projecting downward almost to the flange of the test cup, as shown in [Figure B.3](#). The rim shall fit the outside of the test cup with a clearance not exceeding 0,36 mm on the diameter. There shall be a locating or locking device, or both, engaging with a corresponding device on the test cup. Three openings in the lid, A, B and C, are shown in [Figure B.3](#). The upper edge of the test cup shall be in close contact with the inner face of the lid round the whole of its circumference.

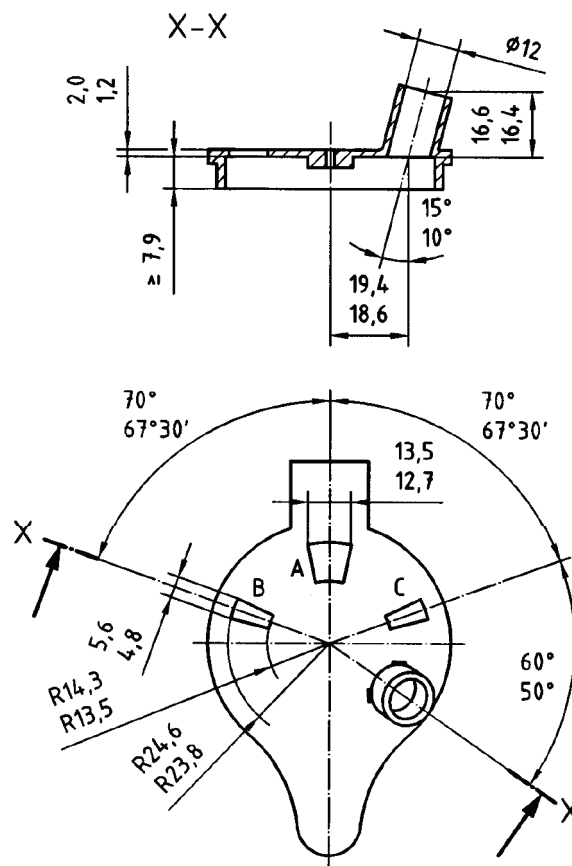


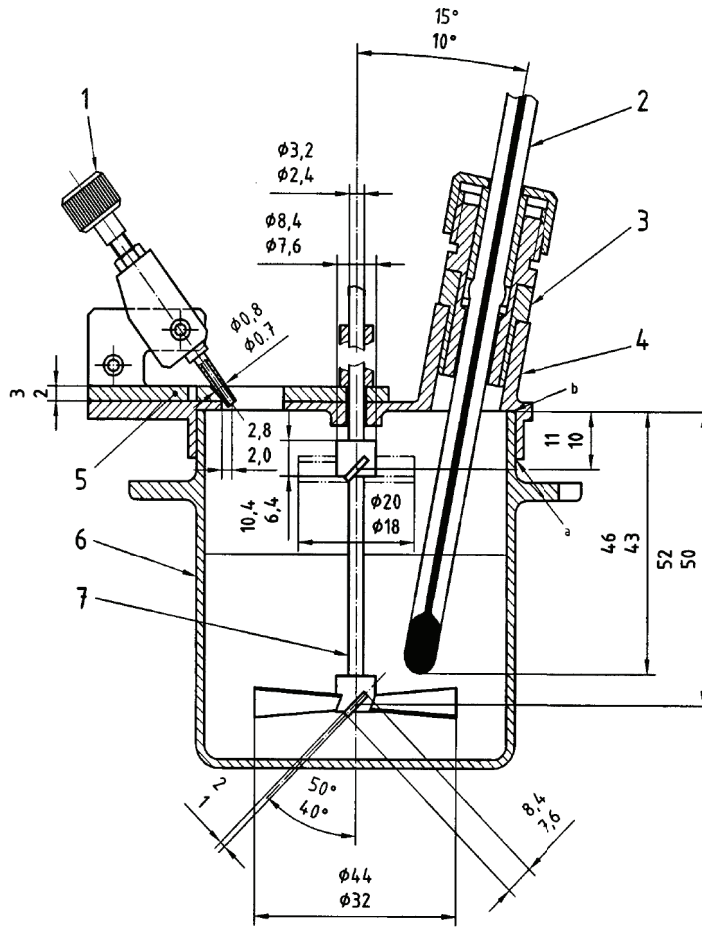
Figure B.3 — Lid

B.3.2 Shutter, made of brass, approximately 2,4 mm thick, operating in the plane of the upper surface of the lid, as shown in [Figure B.4](#). The shutter shall be shaped and mounted so that it rotates about the axis of the horizontal centre of the cover between two stops, placed so that when in one extreme position, the openings A, B and C in the lid are completely closed, and when in the other extreme position, these openings are completely opened. The mechanism operating the shutter shall be of the spring type and constructed so that when at rest the shutter exactly closes the three openings. When the mechanism is operated to the other extreme, the three cover openings shall be exactly open and the tip of the flame-ignition device (see [B.3.3](#)) shall be fully depressed.

B.3.3 Flame-ignition device, having a tip with an opening 0,7 mm to 0,8 mm in diameter, as shown in [Figure B.4](#). This tip shall be made of stainless steel or another suitable material. The flame-ignition device shall be equipped with an operating mechanism that, when the shutter is in the “open” position, depresses the tip so that the centre of the orifice is between the planes of the lower and upper surfaces of the lid at a point on a radius passing through the centre of the largest opening A (see [Figure B.3](#)).

A 4,0 mm bead made of suitable material, of dimensions that indicate the target size of the test flame may be mounted in a visible position on the lid.

B.3.4 Pilot flame for automatic relighting of the ignition-flame. The tip of the pilot flame jet shall have an opening 0,7 mm to 0,8 mm in diameter.



Key

- | | | | |
|---|--|---|----------|
| 1 | flame-ignition device | 5 | shutter |
| 2 | thermometer | 6 | test cup |
| 3 | thermometer adaptor | 7 | stirrer |
| 4 | lid | | |
| a | Clearance, 0,36 mm max. | | |
| b | Rim of cup in contact with the inner face of the lid throughout its circumference. | | |

NOTE 1 The lid assembly may be positioned either right- or left-handed.

NOTE 2 A thermometer adaptor is optional for use when a permanent ferrule is attached to the thermometer.

Figure B.4 — Test cup and lid assembly

B.3.5 Stirring device, mounted in the centre of the lid (see [Figure B.4](#)) and carrying 2 two-bladed metal propellers. The lower propeller shall measure approximately 38 mm from tip to tip, each of its two blades being 8 mm in width and having a pitch of 45°. The upper propeller shall measure approximately 19 mm from tip to tip, each of its two blades also being 8 mm in width and having a pitch of 45°. Both propellers shall be located on the stirrer shaft in such a manner that, when viewed from underneath the stirrer, the blades of one propeller are at 0° and 180° while the blades of the other propeller are at 90° and 270°.

The stirrer shaft may be coupled to a motor, stirring in a downward direction, by means a flexible shaft or a suitable arrangement of pulleys.

B.4 Heating chamber and top plate

Heat shall be supplied to the test cup by means of a properly designed heating chamber that is equivalent to an air bath. The heating chamber shall consist of an air bath and a top plate on which the flange of the test cup rests.

The air bath shall have a cylindrical interior and conform to the dimensional requirements shown in [Figure B.1](#). It shall be either a flame-heated or an electrically-heated metal casting or have an internal electrical-resistance element. In either case, the air bath shall be suitable for use without deformation at the temperatures to which it will be subjected.

If the air bath is flame-heated, it shall be designed so that the temperatures of the bottom and the walls are approximately the same. On this account, it shall be not less than 6 mm thick. The casting shall be designed so that the products of combustion of the flame cannot pass up and come into contact with the test cup.

If the heater is of the electric resistance type, it shall be constructed so that all parts of the interior surface are heated uniformly. The wall and bottom of the air bath shall not be less than 6,4 mm in thickness unless the resistance heating elements are distributed over at least 80 % of the wall and all the bottom of the air bath. A heater having such a distribution shall have heating elements positioned at least 4,0 mm away from the internal surface of the air bath in conjunction with a minimum thickness of 1,58 mm for the wall and bottom of the air bath.

The top plate shall be made of metal and mounted with an air gap between it and the air bath. It shall be attached to the air bath by means of three screws and spacing bushings. The bushings shall be of sufficient thickness to define an air gap of 4,8 mm \pm 0,2 mm, and they shall be not more than 9,5 mm in diameter.

Annex C (normative)

Temperature measuring device specification

C.1 Electronic

C.1.1 Temperature range, at least ambient to 370 °C.

C.1.2 Display resolution, better than 0,5 °C.

C.1.3 Accuracy (after calibration), 0,5 °C for temperatures up to 110 °C and 1,0 °C for temperatures over 110 °C.

C.1.4 Immersion depth, less than 51 mm.

C.1.5 Thermal response time, 4 s \pm 2 s (63,2 % according to the principles of ASTM E1137[14]).

NOTE 1 Guidelines for digital temperature measuring devices are given in ASTM E1137, IEC 60751[10] and ASTM E2877[15].

The 63,2 % response time is the time for the display to indicate 63,2 % of a step change in temperature from a nominal ambient of 20 °C in air to a nominal 77 °C in stirred water.

The 63,2 % thermal response time may be measured for different temperature ranges provided that any, non-ambient, temperature sources are stirred and are less than 1 metre apart.

The 63,2 % thermal response time measuring procedure may be used for liquid-in-glass thermometers.

NOTE 2 A limited multi-laboratory test programme indicated that the average 63,2 % thermal response for Pensky-Martens temperature measuring devices was 3,5 s to 5 s for mercury in glass thermometers and 4,3 s for electronic temperature measuring devices employed in existing automated Pensky-Martens apparatus.

C.2 Liquid-in-glass thermometers

Liquid-in-glass thermometers using alternative low hazard precision liquids may be used providing they conform to the principles given in specification ASTM E2251,[16] meet the scale error, immersion depth, graduations and diameter criteria given in [Table C.1](#) and have a thermal response time of less than 9 s (see [C.1](#)).

Table C.1 — Liquid-in-glass thermometer specification

	Low range	Medium range	High range
Range	-5 °C to +110 °C	20 °C to 150 °C	90 °C to 370 °C
Immersion, mm	57	57	57
Graduations:			
Subdivisions	0,5 °C	1 °C	2 °C
Long lines at each	1 °C and 5 °C	5 °C	10 °C
Numbers at each	5 °C	5 °C	20 °C

Table C.1 (continued)

	Low range	Medium range	High range
Scale error, maximum	0,5 °C	1 °C	1 °C to 260 °C 2 °C to >260 °C
Expansion chamber: permits heating to	160 °C	200 °C	370 °C
Total length, mm	282 to 295	282 to 295	282 to 295
Stem diameter, mm	6,0 to 7,0	6,0 to 7,0	6,0 to 7,0
Bulb length, mm	9 to 13	9 to 13	7 to 10
Bulb diameter, mm	Not less than 5,5 and not greater than stem	Not less than 5,5 and not greater than stem	Not less than 5,5 and not greater than stem
Distance from bottom of bulb to line at	0 °C: 85 mm to 95 mm	20 °C: 85 mm to 95 mm	90 °C: 80 mm to 90 mm
Length of graduated portion, mm	140 to 175	140 to 180	145 to 180
Stem enlargement:			
Diameter, mm	7,5 to 8,5	7,5 to 8,5	7,5 to 8,5
Length, mm	2,5 to 5,0	2,5 to 5,0	2,5 to 5,0
Distance to bottom, mm	64 to 66	64 to 66	64 to 66

Bibliography

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- [2] ISO 2592, *Determination of flash and fire points — Cleveland open cup method*
- [3] ISO 3679, *Determination of flash no-flash and flash point — Rapid equilibrium closed cup method*
- [4] ISO 4259, *Petroleum products — Determination and application of precision data in relation to methods of test*
- [5] ISO 13736, *Determination of flash point — Abel closed-cup method*
- [6] ISO/TR 29662, *Petroleum products and other liquids — Guidance for flash point testing*
- [7] ISO Guide 33, *Reference materials — Good practice in using reference materials*
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