SLOVENSKI STANDARD
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Naftni proizvodi in maziva - Določanje plamenišča - Metoda z zaprto posodo po Pensky-Martensu

Petroleum products and lubricants -- Determination of flash point -- Pensky-Martens closed cup method

Produits pétroliers et lubrifiants -- Détermination du point d'éclair -- Méthode Pensky-Martens en vase clos

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МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ

Petroleum products and lubricants — Determination of flash point — Pensky-Martens closed cup method

Produits pétroliers et lubrifiants — Détermination du point d'éclair — Méthode Pensky-Martens en vase clos

Reference number
ISO 2719 : 1988 (E)

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.

Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council. They are approved in accordance with ISO procedures requiring at least 75 % approval by the member bodies voting.

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

This second edition cancels and replaces the first edition (ISO 2719 : 1973), of which it constitutes a technical revision.

Annexes A, B and C form an integral part of this International Standard.

Petroleum products and lubricants — Determination of flash point — Pensky-Martens closed cup method

1 Scope

This International Standard specifies methods, using the Pensky-Martens closed cup apparatus, for determining the flash point of combustible liquids, liquids with suspended solids, lubricating oils, liquids that tend to form a surface film under the test conditions, and other liquids.

Open cup flash and fire points of petroleum products may be determined by the use of ISO 2592 : 1973, *Petroleum products — Determination of flash and fire points — Cleveland open cup method.* Flash points of paints and varnishes and drying oils may be determined by the use of ISO 1523 : 1983, *Paints, varnishes, petroleum and related products — Determination of flashpoint — Closed cup equilibrium method.*

NOTES

1 The method described in this International Standard may be employed for the detection of contamination of lubricating oils by minor amounts of volatile material, which also often occur in heat transfer oils due to partial cracking. However, the lowest temperature at which such a liquid is capable of producing an ignitable atmosphere may be lower than that found by this method. (See also clause 6, second paragraph, and 7.3, note.)

2 This International Standard should be used to measure and describe the properties of materials, products or systems in response to heat and flame under controlled laboratory conditions. Under actual fire conditions the response to heat and flame may be different.

2 Definition

flash point: The lowest temperature, corrected to a barometric pressure of 101,3 kPa, at which application of a test flame causes the vapour of a test portion to ignite under specified conditions of test.

NOTES

1 The sample is deemed to have flashed when a large flame appears and instantaneously propagates itself over the surface of the sample.

2 Occasionally, particularly near the actual flash point, the application of the test flame will cause a blue halo or an enlarged flame; this is not a flash and should be ignored.

3 Principle

The test portion is heated at a slow, constant rate with continual stirring in a cup closed by a lid. A small flame is directed through an opening (kept closed at other times) into the cup at regular temperature intervals with simultaneous interruption of stirring. The flash point is the lowest temperature at which application of the test flame causes the vapour above the test portion to ignite.

4 Apparatus

4.1 Thermometer, partial immersion, conforming to the appropriate specification in annex A:

— low range, for samples giving a flash point between 10 °C and 110 °C;

— medium range, such as ASTM 88C or IP 101C thermometers;

— high range, for samples giving a flash point between 110 °C and 370 °C.

4.2 Pensky-Martens closed cup apparatus, as described in annex B.

Automatic flash point testers are available and in use which may be advantageous in the saving of testing time, in permitting the use of smaller samples, and in other factors which may merit their use. If automatic testers are used, the user shall be sure that all the manufacturer's instructions for calibrating, adjusting and operating the instrument are followed.

In any cases of dispute, the flash point as determined manually shall be considered the referee test.

4.3 Adapter, for use with low-range thermometer (see annex C).

5 Preparation of apparatus

Support the apparatus on a level, steady table. Unless tests are made in a draught-free room or compartment, it is good practice, but not obligatory, to surround the tester on three sides with a shield approximately 400 mm wide and 600 mm high.

6 Preparation of test sample

Test samples shall not be stored in polyethylene, polypropylene or other plastics bottles, because volatile material may diffuse through the walls of the bottle.

If it is suspected that a test sample contains volatile contaminants, the treatment described in 6.1 and 6.2 shall be omitted.

6.1 Test samples of very viscous materials may be warmed until they are reasonably fluid before they are tested. However, no test sample shall be heated more than is absolutely necessary. A test sample shall never be heated above a temperature 17 °C below its expected flash point.

If the temperature difference between the melting point and flash point of a liquid is less than 20 °C, heat the sample to 3 °C ± 1 °C above its melting point and immediately transfer a test portion into the cup and proceed as in clause 8, neglecting minimum temperatures specified in this clause.

6.2 Test samples containing dissolved or free water may be dehydrated with calcium chloride or by filtering through a qualitative filter paper or a loose plug of dry absorbent cotton. Warming the test sample is permitted, but it shall not be heated for prolonged periods or above a temperature 17 °C below its expected flash point.

On removing water, any water-soluble flammable material present is also likely to be removed.

6.3 For expected flash points below ambient temperature, prepare the apparatus as follows.

6.3.1 Remove the test cup assembly (including lid, thermometer and stirrer) from the apparatus.

6.3.2 Place the test cup assembly in a suitable cooling bath (water or a 1 + 1 mixture of water and ethylene glycol may be used). The bath shall include a stirrer and cover. Provide support for the test cup assembly in the bath so that the lid and upper edge are horizontal and the cup is immersed in direct contact with the bath liquid in such a position that the level of the test portion in the cup is the same as that of the liquid in the water bath.

NOTE — If acetone and dry ice are used to cool the bath, do not use in direct contact with the bath liquid.

6.3.3 When the thermometer, in contact with the test portion, reaches a temperature at least 5 °C below the expected flash point, remove the bath. Apply the test flame every 1 °C as the higher ambient temperature causes the test portion to rise in temperature.

7 Procedure

7.1 Thoroughly clean and dry all parts of the cup and its accessories before starting the test. Ensure that all traces of solvent used to clean the equipment have been completely removed. Fill the cup with the test portion to the level indicated by the filling mark. Place the lid on the cup and set the latter in the heating chamber (or cooling bath in the case of an expected flash point below ambient temperature — see 6.3). Ensure that the cup is properly seated. Insert the thermometer. Light the test flame and adjust it to a diameter of 4 mm ± 0,5 mm. Heat at a rate such that the temperature as indicated by the thermometer increases 5 °C/min to 6 °C/min. Turn the stirrer at 90 r/min to 120 r/min, so that the test portion is made to flow from top to bottom.

7.2 For products whose flash point is expected to be equal to or below 110 °C, apply the flame at each degree up to 110 °C, beginning at a temperature between 18 °C and 28 °C below the expected flash point. Apply the test flame by operating the mechanism on the lid which controls the shutter and test flame burner so that the flame is lowered into the vapour space of the cup in 0,5 s, left in its lowered position for 1 s, and quickly retracted. Do not stir the test portion while applying the test flame. (See also 6.1, second paragraph.)

7.3 If the test portion is expected to have a flash point above 110 °C, apply the test flame in the manner just described at each temperature that is a multiple of 2 °C, beginning at a temperature between 17 °C and 28 °C below the expected flash point.

NOTE — The procedure described in 7.2 and 7.3 is not necessarily applicable when the test is run to determine the possible presence of volatile contaminants.

7.4 Record as the flash point the temperature read on the thermometer at the time the second or subsequent application of the test flame, up to the twentieth application, causes a distinct flash in the interior of the cup. Do not confuse the true flash point with the bluish halo that sometimes surrounds the test flame at applications preceding the one that causes the actual flash.

If a flash is observed on the initial test flame application, or if no flash has been observed by the twentieth application, the procedure shall be started again with a new test portion, this time fixing a lower or a higher expected flash point, respectively. If a flash is observed at the initial test flame application but no flash is observed at a temperature lower than that of the initial test flame application, then the temperature of the initial test flame application is taken as the flash point.

8 Alternative procedure for highly viscous products

8.1 Bring the material to be tested and the tester to a temperature of 15 °C ± 5 °C or 11 °C lower than the expected flash point, whichever is the lower. Turn the stirrer at 250 r/min ± 10 r/min, stirring so that the test portion is made to flow from top to bottom. Raise the temperature throughout the duration of the test at a rate of not less than 1 °C/min and not more than 1,5 °C/min. With the exception of these requirements for rates of stirring and heating, proceed as prescribed in clause 7.

8.2 If the temperature difference between the melting point and flash point of a test material is less than 14 °C, heat the sample to 3 °C ± 1 °C above its melting point and immediately transfer a test portion into the cup and proceed as in clause 7, neglecting minimum temperatures specified in the clause.

8.3 Examples of highly viscous materials are heavy oils, polymeric solutions, adhesives, etc. If the results obtained with such materials, following the directions in clauses 6 and 7, are in doubt, repeat using the alternative procedure given in this clause. The higher flash temperature shall be considered the flash point of the material.

9 Calibration

9.1 Determine the flash point of *p*-xylene following the directions in clauses 6 and 7. When the tester is operating properly, a value of 27,2 °C ± 1,1 °C will be obtained.

9.2 If the flash point obtained with *p*-xylene is not within the limits stated in 9.1, check the condition and operation of the apparatus to ensure conformity with the details listed in annex B, especially with regard to the tightness of the lid, the action of the shutter and the position of the test flame. After adjustment, if necessary, repeat the test. *p*-Xylene having a flash point of 27,2 °C ± 1,1 °C is not a suitable reference material in the high-temperature range of the Pensky-Martens Closed Tester, which may be as high as 370 °C.

9.3 The *p*-xylene shall conform to the following requirements:

relative density at 15,56 °C / 15,56 °C..... 0,860 min./ 0,866 max.

boiling range..... 2 °C from start to dry (the range shall include the boiling point of pure *p*-xylene, which is 138,35 °C)

purity..... 95 % min. (freezing point 11,23 °C min.)

10 Expression of results

10.1 Correction for barometric pressure

Observe and record the ambient barometric pressure at the time of the test. When the pressure differs from 101,3 kPa, correct the flash point using the following formula:

$$\text{Corrected flash point} = C + 0{,}25(101{,}3 - p)$$

where

C is the observed flash point, in degrees Celsius;

p is the ambient barometric pressure, in kilopascals.

The barometric pressure used in this calculation shall be the ambient pressure in the laboratory at the time of test. Many aneroid barometers, such as those used at weather stations and airports, are precorrected to give sea-level readings. These shall not be used.

NOTE — If the pressure is measured in millimetres of mercury, use the formula

$$\text{corrected flash point} = C + 0{,}033\,(760 - p')$$

where p' is the pressure in millimetres of mercury.

10.2 Record the corrected flash point to the nearest 0,5 °C.

10.3 Precision

10.3.1 Basic procedure

The precision of this method, as obtained by statistical examination of interlaboratory test results, is as follows.

10.3.1.1 *Repeatability*

The difference between successive test results, obtained by the same operator with the same apparatus under constant operating conditions on identical test material, would, in the long run, in the normal and correct operation of the test method, exceed the values shown in table 1 only in one case in 20.

Table 1 — Repeatability

Material	Flash point range °C	Repeatability °C
Suspension of solids	35 to 43,5	2
All others	104 and under Above 104	2 6

10.3.1.2 *Reproducibility*

The difference between two single and independent 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 shown in table 2 only in one case in 20.

Table 2 — Reproducibility

Material	Flash point range °C	Reproducibility °C
Suspension of solids	35 to 43,5	3,5
All others	104 and under Above 104	3,5 8,5

10.3.2 Alternative procedure

The following criteria shall be used for judging the acceptability (95 % confidence) of results obtained on viscous materials, which tend to form a surface film.

10.3.2.1 *Repeatability*

The average of two results obtained on the same sample on the same day by the same operator and that of two results on a different day should be considered suspect if they differ by more than 5 °C.

10.3.2.2 *Reproducibility*

The average of two results obtained on the same sample on the same day by one operator compared with the average of two results on the same sample by a different operator in a different laboratory on any one day should be considered suspect if they differ by more than 10 °C.

NOTE — The definitions of repeatability and reproducibility for this procedure represent different parameters of the variance from those corresponding to the definitions in 10.3.1.

11 Test report

The test report shall contain at least the following information:

a) the type and identification of the product tested;

b) a reference to this International Standard;

c) the result of the test (see 10.2);

d) any deviation, by agreement or otherwise, from the procedures specified;

e) the date of the test.

Annex A
(normative)

Thermometer specifications

	Low range	Medium range	High range
Range	−5 °C to +110 °C	10 °C to 200 °C	90 °C to 370 °C
Immersion, mm	57	57	57
Graduations:			
Subdivisions	0,5 °C	0,5 °C	2 °C
Long lines at each	1 °C and 5 °C	1 °C and 5 °C	10 °C
Numbers at each	5 °C	5 °C	20 °C
Scale error, max.	0,5 °C	0,5 °C	1 °C to 260 °C 2 °C over 260 °C
Expansion chamber:			
Permits heating to	160 °C	205 °C	370 °C
Total length, mm	285 to 295	285 to 295	285 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	greater than 4,5 and less than stem	not less than 5,5 and not greater than stem
Distance from bottom of bulb to line at	0 °C: 85 to 95 mm	20 °C: 80 to 90 mm	90 °C: 80 to 90 mm
Length of graduated portion, mm	140 to 175	145 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

NOTE — Though established thermometers ASTM 9C, 10C, 88C and IP 15C, 16C and 101C do not meet all requirements, their use is permitted.