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Determination of flash point — Abel closed-cup method

Détermination du point d'éclair — Méthode Abel en vase clos

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 13736 was prepared by Technical Committee ISO/TC 28, *Petroleum products and lubricants*.

This second edition cancels and replaces the first edition (ISO 13736:1997), which has been technically revised.

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Introduction

Flash-point values can 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 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 that flash-point determinations 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 do 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.

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ISO/TR 29662[6] (CEN/TR 15138[7]) gives useful advice in carrying out flash-point tests and interpreting results.

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Determination of flash point — Abel closed-cup method

CAUTION — The use of this International Standard can involve hazardous materials and equipment. This International Standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this International Standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

1 Scope

This International Standard specifies a method for the determination of the closed-cup flash point of combustible liquids having flash points between − 30,0 °C and 70,0 °C, inclusive. However, the precision given for this method is only valid for flash points in the range − 5,0 °C to 66,5 °C.

This International Standard is not applicable to water-borne paints, which can, however, be tested using ISO 3679[4].

NOTE 1 See 4.1 for the importance of this test to avoid loss of volatile materials.

NOTE 2 Liquids containing halogenated compounds can give anomalous results.

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2 Normative references

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

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

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

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

3 Term and definition

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 a test flame causes the vapour of the test portion to ignite momentarily and the flame to propagate across the surface of the liquid under the specified conditions of test

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4 Principle

4.1 General

Since it is necessary to detect the presence of small proportions of highly volatile materials, this test should be the first determination on a received sample to help avoid the loss of these volatile materials.

4.2 Test principle

The test portion is placed in the test cup of an Abel apparatus and heated to give a constant temperature increase with continuous stirring. A small test flame is directed through an opening in the test cup cover at regular temperature intervals with simultaneous interruption of stirring. The lowest temperature at which application of the test flame 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. The temperature is corrected to standard atmospheric pressure using an equation. Separate test procedures are defined for liquids with expected flash points between – 30,0 °C and 18,5 °C, inclusive, and between 19,0 °C and 70,0 °C, inclusive.

5 Chemicals and materials

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

NOTE The choice of solvent depends on the previous material tested and the tenacity of the residue. Low-volatility aromatic (benzene-free) solvents can be used to remove traces of oil, and mixed solvents can be efficacious for the removal of gum-type deposits.

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5.2 Coolant, ethanediol (ethylene glycol), glycerol or silicone oil (optional), for use in the cooling bath or in the Abel apparatus.

See 10.1.2.

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5.3 Silicone lubricant (optional), to inhibit the formation of ice crystals on the lid and shutter mechanism when carrying out tests at temperatures below 5,0 °C.

See the Note to 10.1.6.

5.4 Verification liquids, as described in Annex D.

6 Apparatus

6.1 Flash-point apparatus, as specified in Annex A.

If automated equipment is used, ensure that it has been established that the results obtained are within the precision of this International Standard, that the test cup and cover assembly conform to the key dimensions specified in Annex A, 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.

6.2 Test cup thermometer, conforming to the specification given in Annex C.

It shall be fitted into a collar as described in Annex B.

6.3 Heating vessel thermometer, conforming to the specification given in Annex C.

It shall be fitted into a collar as described in Annex B.

NOTE Other types of temperature-measuring devices can be used, provided that they meet the requirements for accuracy and have the same response as the thermometers specified in Annex C.

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6.4 Timing device, use one of the following:

a) electric/electronic timing device, which can indicate intervals of 1 s;

b) pendulum, of 610 mm effective length, counting one beat from one extremity of the swing to the other;

c) metronome, that beats at a frequency of 75 beats per minute to 80 beats per minute.

6.5 Barometer, accurate to 0,5 kPa.

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

6.6 Cooling bath, either liquid or metal block, or a recirculating cooler.

See 10.1.4 and 10.2.4.

6.7 Test cup thermal insulating cover (optional), to reduce the formation of ice crystals on the cup and cover assembly during sub-ambient testing.

See 10.1.5.

7 Apparatus preparation

7.1 Location of the apparatus

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Support the Abel 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.

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

7.2 Cleaning the test cup

Wash the test cup 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.

7.3 Apparatus examination

Examine the test cup, the cover and other parts to ensure that they are free from signs of damage and deposits. If any damage is found, either rectify or, if this is not possible, obtain a replacement. If deposits are found, remove them.

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) (see 5.4 and Annex D). 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 test. More frequent verification checks shall be made using secondary working standards (SWS).

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

7.4.2 Do not use the numerical values obtained during the verification check to provide a bias statement, or to make any correction to the flash points subsequently determined using the apparatus.

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8 Sampling

8.1 Obtain samples in accordance with the procedures given in ISO 3170, ISO 3171, ISO 15528 unless otherwise agreed.

8.2 Place sufficient sample volume for testing in a tightly sealed container appropriate to the material being sampled and, for safety purposes, ensure that the sample container is filled only to between 85 % and 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 30,0 °C.

9 Sample handling

9.1 Storage prior to testing

If an aliquot of the original sample is stored prior to testing, ensure that the container is filled to more than 50 % of its capacity.

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

9.2 Liquids with expected flash points between – 30,0 °C and 18,5 °C

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9.2.1 Cool the sample to a temperature of – 35 °C or to at least 17,0 °C below the expected flash point, whichever is the higher, before opening the container.

9.2.2 Cool the liquids that crystallize on cooling to just above their melting points.

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9.3 Liquids with expected flash points between 19,0 °C and 70,0 °C

Cool the sample to a temperature of 2 °C or to at least 17,0 °C below the expected flash point, whichever is the higher, before opening the container.

9.4 Samples containing water as a separate phase

If a sample contains water as a separate phase, decant an aliquot from the water prior to mixing.

Flash-point results can be affected by the presence of water. For certain fuels, it might 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[4].

9.5 Sample mixing

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.

10 Procedure

10.1 Liquids with expected flash points between – 30,0 °C and 18,5 °C

10.1.1 Using a barometer (6.5), record the ambient pressure in the vicinity of the apparatus at the time of test.

NOTE It is not necessary to correct the barometric pressure for ambient temperature, although some barometers are designed to make this correction automatically.

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10.1.2 Use either a mixture of equal volumes of ethanediol (5.2) and water, or glycerol (5.2) and water, or silicone oil (5.2) to completely fill the heating vessel (Clause A.5) and to fill the inner air chamber, which surrounds the test cup (Clause A.2), to a depth of at least 38 mm.

10.1.3 Insert the heating vessel thermometer (6.3).

10.1.4 Adjust the temperature of the heating vessel (Clause A.5), using a cooling bath (6.6) connected via the funnel aperture and overflow pipe, to − 35 °C or to at least 9,0 °C below the expected flash point of the material being tested, whichever is the higher. Carry out a trial flash-point determination if necessary.

10.1.5 Loosely assemble the cover (Clause A.3) and test cup (Clause A.2). Cover with the thermal insulator (6.7), and cool the assembly to − 35 °C or to at least 17,0 °C below the expected flash point, whichever is the higher.

10.1.6 Ensure that neither cooling liquid nor vapour from the cooling bath, which could affect the flash point of the product under test, enters the test cup.

NOTE Cooling a cover or test cup that is wet with water to below 0 °C can cause sticking due to ice (e.g. sticking of the slide). Wiping the apparatus dry with a duster or a piece of absorbent paper before cooling to below 0 °C is usually sufficient to prevent icing, but, alternatively, icing can be minimized by the use of a thermal insulating cover (6.7) and by lubricating the outer face of the lip of the test cup and the slide with a silicone lubricant (5.3).

10.1.7 Place the test cup in position in the apparatus (see Clause A.3) and insert the test cup thermometer (6.2). Remove the cover and pour in the test portion without undue agitation, avoiding as far as possible the formation of air bubbles, until the level just reaches the point of the index gauge on the wall of the test cup. Do not move the apparatus after filling. Place the cover on the test cup and push it down into position. Ignite the test flame, adjust its size to conform to the size of the reference bead mounted on the cover of the test cup, and maintain it at that size throughout the test.

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10.1.8 Apply heat to the heating vessel (Clause A.5) in such a manner that the temperature of the test portion in the test cup rises at a rate of 1 °C/min from the first application of the test flame to the end of the test.

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10.1.9 Using the stirrer (Clause A.4), stir the test portion in a clockwise direction (i.e. to give a downward thrust) at 30 r/min ± 5 r/min. Continue stirring in a steady manner for the duration of the test but do not stir during the application of the test flame.

10.1.10 When the temperature of the test portion reaches − 35 °C or at least 9,0 °C below the expected flash point, start the timing device (6.4), apply the test flame by slowly and uniformly opening the slide in the cover while the timer beats three times and closing it during the fourth beat. If an electric/electronic timing device calibrated in seconds is used, the application of the test flame shall be made by slowly and uniformly opening the slide over a period of 2 s and then closing it over a period of 1 s.

10.1.11 If a flash occurs, discontinue the test, discard the test portion and proceed in accordance with 10.1.3, commencing the test at a lower expected flash-point temperature. If no flash occurs, proceed in accordance with 10.1.12. If a flash occurs at a temperature below − 30,0 °C, record and report this fact and discontinue the test.

10.1.12 Apply the test flame in this manner every 0,5 °C rise in temperature until a distinct flash occurs in the interior of the test cup or until a temperature corresponding to a corrected temperature of 18,5 °C is reached. Record the temperature of the test portion when the flash occurs.

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

10.1.14 Record as the observed flash point the temperature read on the test cup thermometer at the time the test flame application caused a distinct flash in the interior of the test cup.

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