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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

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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).

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For an explanation of the voluntary nature of standards, 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 www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 28, *Petroleum products and lubricants,* in collaboration with the European Committee for Standardization (CEN) Technical Committee CEN/TC 19, *Gaseous and liquid fuels, lubricants and related products of petroleum, synthetic and biological origin* in accordance with the Agreement on technical cooperation between ISO and CEN (Vienna Agreement).

This fourth edition cancels and replaces the third edition (ISO 13736:2013), which has been technically revised.

The main changes compared to the previous edition are as follows:

— the Subclause 7.5 has been further elaborated;

— under 13.2 and 13.3, the precision definitions have been updated in line with ISO 4259-1[3];

— in Annex C the digital contact thermometers have been introduced and furthermore explanation on the generic liquid-in-glass thermometers has been introduced;

— Annex D has been revised (especially the evaluation subclause) and changed to normative status;

— a new Annex E on flash point values of chemicals has been introduced.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

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.

Flash point determinations are not appropriate for potentially unstable, decomposable, or explosive materials, unless 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[7] gives useful advice on carrying out flash point tests and interpreting results.

Determination of flash point — Abel closed-cup method

WARNING — The use of this document can involve hazardous materials, operations and equipment. This document does not purport to address all of the safety problems associated with its use. It is the responsibility of users of this document to take appropriate measures to ensure the safety and health of personnel prior to the application of the standard, and to determine the applicability of any other restrictions for this purpose.

1 Scope

This document specifies a method for the determination of the manual and automated closed cup flash point of combustible liquids having flash points between –30,0 °C to 75,0 °C. However, the precision given for this method is only valid for flash points in the range –8,5 °C to 75,0 °C.

This document is not applicable to water-borne paints.

NOTE 1 Water borne paints can be tested using ISO 3679[1].

NOTE 2 See 9.1 for the importance of this test in avoiding loss of volatile materials.

NOTE 3 Liquids containing halogenated compounds can give anomalous results.

NOTE 4 The thermometer specified for the manual apparatus limits the upper test temperature to 70,0 °C.

NOTE 5 See 13.1 for more specific information related to precision.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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, *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.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at http://www.electropedia.org/

3.1
flash point
lowest temperature of the test portion, adjusted to account for variations in atmospheric pressure from 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 in the test cup of an Abel apparatus and heated to give a constant temperature increase with continuous stirring. An ignition source 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 ignition source causes the vapours 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 adjusted to account for variation in atmospheric pressure from 101,3 kPa, using a formula.

5 Chemicals and materials

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

The choice of solvent depends 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 can be efficacious for the removal of gum-type deposits.

5.2 Coolant, mixture of equal volumes of 1,2-ethanediol (ethylene glycol) and water, or mixture of equal volumes of glycerol and water, or silicone oil (optional), for use in an external cooling bath (6.5) or in the Abel apparatus (6.1).

5.3 Lubricant (optional), to reduce the formation of ice crystals on the cover and shutter mechanism when carrying out tests at temperatures below 5,0 °C (7.4.3, Note 1).

5.4 Verification liquids, certified reference materials (CRM) and secondary working standards (SWS) as described in Annex D.

5.5 Ignitor and **pilot light gas**, which may be propane, butane or natural gas (not required if an electric ignitor is used).

6 Apparatus

6.1 Flash point apparatus, as specified in Annex A.

If automated equipment is used, ensure that the test cup and cover assembly conform to the key dimensions specified in A.2 and that 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.

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

6.2 Thermometers

6.2.1 Test cup thermometer, installed as in Annex B and conforming to the specification given in Annex C.

6.2.2 Heating vessel thermometer, installed as in Annex B and conforming to the specification given in Annex C.

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

6.3 Timing device, stopwatch or electronic timer with an accuracy better than 5 %.

6.4 Barometer, absolute pressure reading, 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.

6.5 External cooling bath (optional), for assisting in the cooling of the Abel apparatus and test sample (7.4.1 and 7.4.2).

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

7 Apparatus preparation

7.1 Location of the apparatus

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.

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 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 or other proven procedure to ensure complete removal of the solvent used.

WARNING — Any remaining cleaning solvent can significantly affect the measured flash point of a sample.

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 the problem or, if this is not possible, obtain a replacement. If deposits are found remove them.

7.4 Heating and cooling

7.4.1 Liquid baths

Use water or, for less than or near 0 °C bath temperatures, a coolant (5.2), to completely fill the heating vessel and to fill the inner air chamber that surrounds the test cup to a depth of at least 38 mm.

Adjust the temperature of the heating vessel using an external cooling bath (6.5) if required, to at least 9,0 °C below the expected flash or to –35,0 °C, whichever is the higher.

7.4.2 Solid metal baths

Follow the manufacturers' instructions to adjust the temperature of the bath to at least 9,0 °C below the expected flash point or to –35,0 °C, whichever is the higher.

7.4.3 Test cup and cover

Loosely assemble the cover and test cup. Adjust their temperature, using an external cooling bath (6.5) or refrigerator if required, to at least 17,0 °C below the expected flash point or to –35,0 °C, whichever is the higher.

Use the thermal insulating cap (6.6) at lower temperatures.

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

NOTE 1 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 cap (6.6) and by lubricating the outer face of the lip of the test cup and the slide with a lubricant (5.3).

NOTE 2 A low humidity laboratory environment helps minimize the formation of ice crystals at test temperatures of below 5 °C.

7.5 Apparatus verification

7.5.1 Check the temperature measuring devices and barometer at least once a year to ensure that they are in accordance with 6.2 and 6.4 requirements respectively.

7.5.2 Ensure the correct operation of ignition sources, in accordance with the manufacturers' instructions and this test method.

7.5.3 Verify the accuracy of the apparatus at least once a year by testing a certified reference material (CRM) (see 5.4 and Annex D). It is recommended that more frequent verification checks are made using CRM or SWS.

The result of a single test obtained for either CRM or SWS shall be equal to or less than $R/\sqrt{2}$ from the certified value of the CRM or from the accepted reference value (ARV) of the SWS, where R is the reproducibility of the test method.

NOTE These reference materials and in-house quality control samples can also be used to monitor stability and establish statistical control limits, in accordance with ASTM D6299[13] or equivalent standard, if required.

7.5.4 Do not use the numerical values obtained during verification checks to correct subsequent flash point results or provide a bias statement.

7.5.5 When the flash point is not within the required limits, check the condition and operation of the apparatus to ensure conformity with the details listed in A.1 especially with regard to tightness of the lid, the action of the shutter, the position and operation of the ignition source, and the angle and position of the temperature measuring device. After any adjustment, repeat the test in Annex D using a fresh test portion, with special attention to the procedural details prescribed in this test method.

8 Sampling

8.1 Obtain samples in accordance with the procedures given in ISO 3170, ISO 3171, ISO 15528 or an equivalent national standard, unless otherwise agreed.

8.2 Place sufficient sample volume for testing in a tightly sealed container appropriate to the material being sampled and, for sample integrity and safety purposes, ensure that the sample container is filled to between 85 % and 95 % of its capacity.

8.3 Store the samples in conditions that minimize vapour loss and pressure build-up.

IMPORTANT — 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.

8.4 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 17 °C below the expected flash point or to −35,0 °C, whichever is the higher, before opening the container.

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

9 Sampling handling

9.1 General

Since the presence of small proportions of highly volatile materials needs to be detected, this test should be the first determination on a received sample to reduce the loss of these volatile materials.

9.2 Subsampling

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

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

9.2.2 Successive test portions may be taken from the same sample container when the second test portion is taken with the sample container at least 50 % filled.

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

9.3 Samples containing undissolved water

Flash point results can be affected by the presence of water. If a sample contains water as a separate phase, decant an aliquot from the water prior to mixing.

For certain fuels, it is not always 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.

9.4 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 Using a barometer (6.4), 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.

10.2 Follow apparatus preparation (see Clause 7) and sample handling (see Clause 9) to adjust the temperature of the Abel bath, cup and cover respectively.

10.3 Place the test cup in position in the apparatus and insert the test cup thermometer (6.2.1). 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.

The sample may be poured into the test cup before it is placed in position in the apparatus. Place the cover on the test cup and push it down into position. Make any necessary mechanical or electrical connections to the cover and, if a gas ignition source is used, ignite the ignition source 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.

A pre-test dip of the ignition source is strongly recommended, before commencing heating of the test portion, as this could indicate the presence of low flash point components. If a flash is detected, discontinue the test, discard the test portion and proceed in accordance with 10.2, commencing the test with a lower expected flash point temperature.

10.4 At the start of the test**,** apply heat to the heating vessel in such a manner that the temperature of the test portion in the test cup rises at a rate of approximately 1 °C/min from the first application of the ignition source, as described in 10.6, to the end of the test. This rate of heating is not reached immediately at the start of the test. See A.2.5 for specific requirements for automated heating vessels.

10.5 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 ignition source.

10.6 When the temperature of the test portion reaches at least 9,0 °C below the expected flash point or −35,0 °C, whichever is the higher, apply the ignition source by slowly and uniformly opening the slide over a period of approximately 2 s and then closing it over a period of approximately 1 s.

10.7 If a flash is detected on this first application of the ignition source, discontinue the test, discard the test portion and proceed in accordance with 10.2, commencing the test at a lower expected flash point temperature. If no flash occurs, proceed in accordance with 10.8. If a flash occurs at a temperature below −30,0 °C, record and report this fact and discontinue the test.

10.8 Apply the ignition source in this manner at every 0,5 °C rise in temperature until a distinct flash is detected in the interior of the test cup.

10.9 Record, as the detected flash point, the temperature read by the test cup thermometer at the time when the ignition source application causes a distinct flash in the interior of the test cup.

10.10 Do not confuse the true flash point with the bluish halo that sometimes surrounds the ignition source flame at applications preceding the actual flash point.

11 Calculation

11.1 If the barometric pressure reading taken in accordance with 10.1 is in a unit other than kilopascals, convert to kilopascals using the following formulae as appropriate:

— reading in hPa × 0,1 = kPa;

— reading in mbar × 0,1 = kPa;

— reading in mmHg × 0,133 322 = kPa.

NOTE For the purposes of correcting flash point values to account for variations in atmospheric pressure from 101,3 kPa, it is not considered necessary to correct the barometer readings for ambient temperature. However, some barometers are designed to automatically correct the barometric pressure for ambient temperature.

