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Determination of flash point – Method for flash no-flash and flash point by small scale closed cup tester

Détermination du point d'éclair — Méthode de l'éclair de type passe/ne passe pas et méthode du point d'éclair en vase clos à petite échelle

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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).

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).

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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 and related products, fuels and lubricants from natural or synthetic sources*, 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 fifth edition cancels and replaces the fourth edition (ISO 3679:2015), which has been technically revised.

The main changes are as follows:

— introduction, title and scope have been revised to present a more generic method description;

— terms and definitions in Clause 3 have been added;

— verification clause has been revised;

— new procedure C has been added;

— Clause 13 wording has been revised and precision for procedure C has been included;

— the apparatus description in Annex A has been revised;

— Annex B has been revised and changed to normative;

— the text has been editorially revised in line with the ISO/IEC Directives Part 2, 2021.

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

This document includes three procedures (A, B and C) covering determinations of flash no-flash and flash point. Rapid equilibrium procedures A and B enable the determination of the flash no-flash and flash point, respectively. Non-equilibrium procedure C uses automated test cup temperature control for flash point determination.

ISO 1516 and ISO 1523 are also closed cup equilibrium test methods that can be considered when selecting a method.

The apparatus specified in this document enables a similar test result to be determined using more rapid procedures, A or B, and a smaller test portion (2 ml or 4 ml), than those required in ISO 1516 or ISO 1523. In addition, the apparatus in this document can be made portable so that it is suitable for on-site testing, as well as its regular use in laboratories. Collaborative work[16] has shown that results obtained by these methods are comparable. Procedure C is based on test methods IP 534[18] and ASTM D7236[14].

The interpretation of flash point results obtained on solvent mixtures containing halogenated hydrocarbons should be considered with caution, as these mixtures can give anomalous results[17].

A limited study has indicated that some water borne paints can give an elevated flash point when an electric ignitor is used with this document.

Flash point is 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.

The flash point indicates the presence of highly volatile material(s) in a relatively non-volatile or non-flammable material. Flash point testing is often used as 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. That is, 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.

The flash point is not a constant physical-chemical property of a material tested. It is a function of the apparatus design, the condition of the apparatus used, and the operational procedure carried out. Flash point can therefore only be defined 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 also gives useful advice in carrying out flash point tests and interpreting results.

Determination of flash point – Method for flash no-flash and flash point by small scale closed cup tester

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 describes three procedures (A, B and C) covering determinations of flash no-flash and flash point.

Rapid equilibrium procedures A and B are applicable to flash no-flash and flash point tests of paints, including water-borne paints, varnishes, binders for paints and varnishes, adhesives, solvents, petroleum products including aviation turbine, diesel and kerosene fuels, fatty acid methyl esters and related products over the temperature range –30 °C to 300 °C. The rapid equilibrium procedures are used to determine whether a product will or will not flash at a specified temperature (flash no-flash procedure A) or the flash point of a sample (procedure B). When used in conjunction with the flash detector (A.1.6), this document is also suitable to determine the flash point of fatty acid methyl esters (FAME). The validity of the precision is given in Table 2.

Non-equilibrium procedure C is applicable to petroleum products including aviation turbine, diesel and kerosine fuels, and related petroleum products, over the temperature range –20 °C to 300 °C. The non-equilibrium procedure is automated to determine the flash point. Precision has been determined over the range 40 °C to 135 °C.

For specifications and regulations, procedures A or B are routinely used (see 10.1.1).

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 Guide 35, *Reference materials — Guidance for characterization and assessment of homogeneity and stability*

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*

ISO 17034, *General requirements for the competence of reference material producers*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

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

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

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

3.1
equilibrium
condition in flash point test methods where the vapour above the test portion and the test portion are at the same temperature at the time the ignition source is applied

Note 1 to entry: This condition cannot be fully achieved in practice, since the temperature can be uneven throughout the test portion, and the test cover and shutter on the apparatus can be cooler or warmer.

EXAMPLE Procedures A and B in this document, ISO 1516 and ISO 1523.

3.2
fatty acid methyl ester
FAME
fuel comprising mono-alkyl esters of long chain fatty acids derived from vegetable oil or animal fats, designated B100 or biodiesel (100 %)

Note 1 to entry: FAME is specified in specifications such as EN 14214 and ASTM D6751.

3.3
flash no-flash
application of an ignition source at the specified temperature of the test portion, as measured in the prescribed manner, adjusted to account for variations in atmospheric pressure from 101,3 kPa, to determine whether the vapour of the test portion ignites and the flame propagates across the surface of the liquid under the specified conditions of test

3.4
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

3.5
non-equilibrium
condition in flash point test methods where the vapour above the test portion and the test portion are not in temperature equilibrium at the time that the ignition source is applied

Note 1 to entry: This condition is primarily caused by the heating of the test portion at the constant prescribed rate with the vapour temperature lagging behind the test portion temperature.

EXAMPLE Procedure C in this document, ISO 2719 and ISO 13736.

4 Principle

4.1 Rapid equilibrium procedures A and B

A 2 ml or 4 ml test portion is introduced into the test cup that is set and maintained at the required test temperature. After a specific time, when the vapours and test portion are deemed to be in temperature equilibrium, an ignition source is applied and a determination is made (procedure A) as to whether or not a flash occurred. In order to determine the actual flash point of the sample, further tests, with fresh test portions at different test cup temperatures, are carried out (procedure B) until the flash point is determined. The temperature is adjusted to account for variation in atmospheric pressure from 101,3 kPa, using a formula.

4.2 Non-equilibrium procedure C

A 2 ml test portion is introduced into the test cup that is set and maintained at the required start temperature. The test cup is then heated at a ramp rate of 1,5 °C/min to 2,5 °C/ min and the ignition source is applied at 1 °C temperature intervals until a flash point is detected. The detected flash point temperature is adjusted to account for variation in atmospheric pressure from 101,3 kPa, using a formula.

5 Reagents 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 can be used to remove traces of oil, and mixed solvents can be effective for the removal of gum-type deposits.

5.2 Reference materials, for flash point, certified reference materials (CRM) and/or secondary working standards (SWS), as described in Annex B.

5.3 Gas for ignitor and pilot flame, not required if an electric ignitor is used. Butane, propane, coal gas, or natural gas may be used.

6 Apparatus

6.1 Flash point apparatus, as specified in Annex A.

6.2 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.

6.3 Heating bath or oven (optional), for warming the sample, if required.

The bath and oven shall be suitable for use with volatile and flammable materials.

6.4 Cooling bath or freezer (optional), for cooling the samples, if required, and capable of cooling the sample to 10 °C below the expected flash point.

The bath and freezer shall be suitable for use with volatile and flammable materials.

6.5 Draught shield (optional), if required to minimize draughts, a shield fitted at the back and on two sides of the instrument.

6.6 Cup insert (optional), see Annex C.

For samples that are difficult to remove, a thin metal cup insert can be used but the precision has not been determined.

6.7 Syringes

6.7.1 Syringe, capable of delivering 2,00 ml ± 0,05 ml and equipped with a nozzle suitable for the required test temperature and apparatus.

To enable a 4 ml test portion to be used, this syringe can be used twice.

6.7.2 **Syringe**, capable of delivering 4,00 ml ± 0,10 ml and equipped with a nozzle suitable for the required test temperature and apparatus.

7 Preparation of apparatus

7.1 General

7.1.1 Select the appropriate instrument for the relevant procedure and the expected flash point temperature. Follow the manufacturer's instructions for the correct set-up, verification (see 7.4) and operation of the apparatus, especially the operation and setting of the ignition source.

7.1.2 Procedure C is automated and requires automated temperature ramp control; sub-ambient testing requires integrated cooling (see A.1.5).

7.1.3 The use of a cup insert (6.6) for potentially adherent materials is described in Annex C.

7.1.4 When testing FAME (procedures A and B), use a 2 ml ± 0,05 ml test portion and a 60 s ± 2 s test time, combined with an electronic thermal flash detector (see A.1.6).

7.1.5 For sub-ambient test temperatures, use Annex D, unless the apparatus has integral test cup cooling facilities.

7.2 Location of apparatus

Support the apparatus specified in Annex A on a level and steady surface in a draught-free position.

A draught shield (6.5) should be used when protection from draughts is not available.

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

7.3 Cleaning of the test cup assembly and accessories

Clean the test cup cover and its accessories with an appropriate solvent (5.1) to remove traces of gum or residue from the previous test. Wipe dry to remove all traces of solvent.

Follow the manufacturer's instructions for the care and servicing of the instrument, especially regarding electronic ignitors and flash detectors which can be fragile.

A stream of clean dry air, such as compressed air, can be used to remove the last traces of solvent used.

The filler orifice can be cleaned using a suitable cleaning device such as a small brush.

7.4 Apparatus verification

7.4.1 Check the temperature measuring devices and barometer at least once a year to ensure that they are in accordance with A.1.4, Annex E and 6.2, respectively.

7.4.2 Ensure the correct operation of ignition sources, in accordance with the manufacturer's instructions and this test method.

7.4.3 Verify the accuracy of the apparatus at least once a year by testing a CRM (see 5.2 and Annex B). It is recommended that more frequent verification checks are made using a reference material (see 5.2 and Annex B).

7.4.4 The result of a single test obtained for a reference material 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 procedure.

NOTE These reference materials (RM) and in-house quality control samples can also be used to monitor stability and establish statistical control limits, according to ISO 4259-4 or equivalent standard, if required.

7.4.5 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.

7.4.6 If the instrument fails the verification test, it is recommended that the operator follow the manufacturer's instructions and check the following, and then repeat the verification check:

a) the cover makes a vapour tight seal with the test cup;

b) the shutter provides a light tight seal;

c) adequate heat transfer paste surrounds the temperature measuring device inserted in the test cup block;

d) the ignition source operates correctly;

e) the flash detector (A.1.6) operates correctly (if fitted);

f) the temperature measuring device reads correctly.

8 Sampling

8.1 Unless otherwise specified, obtain samples in accordance with the procedures given in ISO 1513, ISO 15528, ISO 3170, or ISO 3171 or an equivalent national standard.

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

8.3 Store the samples in conditions to minimize vapour loss and pressure build up. Avoid storage of samples at temperatures in excess of 30 °C.

9 Sample handling

9.1 Petroleum products and fatty acid methyl esters

9.1.1 Subsampling

Cool or adjust the temperature of the sample and its container to at least 10 °C below the first selected test temperature before opening to remove the test portion. If an aliquot of the original sample must be stored prior to testing, the container shall be filled to between 85 % and 95 % of its capacity. Gently mix the subsample to ensure uniformity, so that the loss of volatile components and light ends is minimized.

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

9.1.2 Samples liquid at ambient temperature

If sufficiently fluid, mix samples by gentle hand shaking prior to the removal of the test portion, taking care to minimize the loss of volatile components. Ensure that the sample is at least 10 °C below the

selected test temperature before opening to remove the test portion. For mobile materials, mix the sample by gentle shaking. If necessary, heat the sample in its container to a temperature such that the sample can be mixed by gentle shaking or to at least 10 °C below the selected test temperature, whichever is lower. Ensure that high pressures do not develop in the container.

9.1.3 Samples solid or semi-solid at ambient temperature

If the material under test cannot be made sufficiently fluid to be introduced into the test cup through the orifice by heating in accordance with 9.1.2, transfer the test portion with a solids dispenser or spatula into the test cup while the cover is open. The test portion size can be the mass equivalent of the required volume and the test portion should be spread over the bottom of the test cup as evenly as possible.

9.1.4 Samples containing dissolved or free water that is not part of the product

If the sample does not contain volatile, low flash point components, the water can be decanted or the sample dehydrated with calcium chloride.

9.2 Paints, varnishes, and related materials

Prepare the samples in accordance with the procedures described in ISO 1513.

10 Procedures

10.1 General

10.1.1 For specifications and regulations use procedures A or B, unless procedure C has been specified.

10.1.2 Follow the manufacturer's instructions for setting the test temperature.

10.1.3 When testing fatty acid methyl esters (FAME), a flash detector (A.1.6) shall be used.

10.1.4 Use a new test portion of the sample for each test. After each test, turn off the pilot and test flames (if used) using the gas control valves, and when the test cup temperature falls to a safe level, remove the test portion and clean the instrument.

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

NOTE The optional flash detector (A.1.6) is not affected by the halo, and does not require the operator to closely observe the flash point test.

10.1.6 Record the absolute barometric pressure using a barometer (6.2) in the vicinity of the apparatus at the time of the test.

NOTE It is not considered necessary to correct the barometric pressure reading to 0 °C, although some barometers are designed to make this correction automatically.

10.1.7 Ensure that the test flame size or setting of an electric ignitor is set correctly, as an incorrect setting can significantly affect the test result.

10.2 Procedure A — Flash no-flash test

10.2.1 Inspect the test cup and cover for cleanliness and correct operation, especially with regard to tightness of the cover "O" ring (A.1.1.3), the action of the shutter, the size or intensity of the ignition

