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Determination of sustained combustibility of liquids

Essai de combustion entretenue de liquides

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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).

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

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 139, *Paints and varnishes*, in accordance with the Agreement on technical cooperation between ISO and CEN (Vienna Agreement).

This third edition cancels and replaces the second edition (ISO 9038:2013), which has been technically revised.

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

— the test method has been aligned with the requirements of UN Test L.2[1][2]. In particular:

  — the requirement for triplicate tests instead of duplicate tests has been specified;

  — the standard test temperature has been changed to 60,5 °C;

  — the criteria for sustained combustion have been revised.

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

A product with a flash point within a given range can continue to burn after initial ignition, while a similar product, although it has a similar flash point, does not continue to burn. This document describes a method for discriminating between those products that sustain combustion and those that do not.

The method determines whether a flammable product, when maintained at a selected test temperature, generates sufficient flammable vapour to cause ignition when an ignition source is applied and then continues to generate sufficient vapour to burn when the ignition source is moved to the "off" position.

This test method does not determine the flash point of the product under test but, by means of a test procedure, merely determines if it sustains combustion at a selected test temperature; this criterion can be required to comply with laws or regulations relating to the storage, transport and use of flammable products. Before performing this test, for safety and test optimization reasons, it is usual to determine either the actual flash point of the material or know the temperature range in which the flash point is located.

The apparatus specified in this document enables a result to be determined by a rapid procedure using a small test portion (2 ml).

Determination of sustained combustibility of liquids

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 this document, and to determine the applicability of any other restrictions for this purpose.

1 Scope

This document specifies a procedure, at temperatures up to 100 °C, to determine whether a liquid product, that would be classified as "flammable" by virtue of its flash point, sustains combustion at the temperature(s) specified e.g. in regulations.

NOTE Many national and international regulations classify liquids as presenting a flammable hazard based on their flash point, as determined by a recognized method. Some of these regulations allow a derogation if the substance cannot "sustain combustion" at some specified temperature(s).

The procedure is applicable to paints (including water-borne paints), varnishes, paint binders, solvents, petroleum or related products and adhesives, that have a flash point. It is not applicable to painted surfaces in respect of assessing their potential fire hazards.

This test method is applicable, in addition to test methods for flash point, for assessing the fire hazard of a product.

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 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.

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
sustained combustibility
behaviour of a material, under specified test conditions, whereby its vapour can be ignited by an ignition source and, after ignition, sufficient flammable vapour is produced for burning to continue for at least 15 s after the source of ignition has been removed

3.2
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

A test portion of specified volume is introduced into a test cup, which is maintained at the test temperature. After a specified time, an ignition source is applied.

In connection with the United Nations recommendations on the Transport of Dangerous Goods[2] as well as with the Globally Harmonized System of Classification and Labelling of Chemicals[1], and also with derived national/EC regulations, temperatures of 60,5 °C and 75,0 °C are specified for this test.

The property of the product to sustain combustion is assessed based on the ignition of its vapours or components, when exposed to an ignition source, and whether it continues to burn after the ignition source has been moved to the “off” position. The “off” and “test” positions of the ignitor are shown in Figure A.1.

5 Apparatus

5.1 Combustibility tester, as specified in Annex A.

5.2 Electrical heater, attached to the bottom of the test cup in a manner that provides efficient transfer of heat. The heater control shall be capable of maintaining the test cup temperature, as measured on the temperature measuring device, and in a draught-free area, within ± 0,5 °C for test temperatures ≤ 100 °C.

The combustibility tester, heater and heater control unit may consist of an integrated apparatus.

5.3 Gauge, for checking that the height of the centre of the gas jet above the top of the test cup is 2,2 mm ± 0,1 mm. A calibrated metal strip is suitable.

5.4 Temperature measuring device, suitable for horizontal operation, and of suitable range and dimensions, with a resolution 0,5 °C or better. A resolution of 0,1 °C is recommended as it simplifies verification and calibration.

It shall have an accuracy ± 0,5 °C.

When in position, in the block, the temperature measuring device shall be surrounded with heat transfer paste to ensure good heat transfer between the block and the measuring device.

5.5 Stopwatch or **other suitable timing device,** capable of measuring 15 s ± 1 s, 30 s ± 1 s and 60 s ± 2 s. The timing device may be fitted with a means of producing an audible signal.

5.6 Syringe or **pipette,** capable of delivering 2,00 ml to an accuracy of ± 0,05 ml.

5.7 Ignition source and **gas supply,** fuelled by natural gas, coal gas, butane or any other gas found to be suitable and with gas jet fitted with a suitable regulator, or other means of regulating the gas flow, such that the width of the flame can be adjusted to 4,0 mm ± 0,5 mm.

5.8 Draught shield, to minimize draughts, fitted at the back and two sides of the instrument. A shield 350 mm high, 480 mm wide and 240 mm deep is suitable. The shield shall not be placed too close to the apparatus to avoid air turbulences over the test portion.

5.9 Barometer, measuring absolute pressure, with an accuracy of 0,5 kPa and resolution of 0,1 kPa. Do not use aneroid barometers pre-corrected to give sea level readings, such as those used at weather stations and airports.

6 Preparation of apparatus and verification

6.1 The test shall not be carried out in a small confined area because of the risk of explosion.

6.2 Thoroughly clean and dry the test cup and assembly before use, taking care not to damage the surface of the test cup.

6.3 Position the combustibility tester on a level, stable surface and away from strong light (to facilitate observation of a flash or flame). Ensure that the top of the metal block is horizontal.

6.4 Use the gauge (5.3) to check that the jet is 2,2 mm ± 0,1 mm above the top of the block (see Figure A.2).

6.5 It is essential that the apparatus is set up in a draught free area.

NOTE An air speed of greater than 0,05 m/s within 50 mm of the top of the test cup can affect the result.

Surround the tester on three sides with a draught shield (5.8) for protection. If a fume hood is used, minimize the draught around the test cup.

6.6 Verify the accuracy of the temperature measuring device (5.4) and the barometer (5.9), at least every 12 months or more frequently as indicated by a user verification schedule.

6.7 Verify the performance of the apparatus, using reference materials in accordance with Annex B at least every 12 months or more frequently as indicated by a user verification schedule.

7 Sampling

7.1 Paints, varnishes and related products

Take a representative sample of the product to be tested, as described in ISO 15528. Examine and prepare it for testing, as described in ISO 1513.

7.2 Petroleum and related products

7.2.1 Sampling procedure

Take a representative sample of the product to be tested, as described in ISO 3170 or ISO 3171, as appropriate.

The container shall be made of a material appropriate to the product being sampled and be filled to between 85 % and 95 % of its capacity.

7.2.2 Sample handling

7.2.2.1 Obtain a representative sample of at least 50 ml and store in a clean, tightly closed container in a cool place to minimize vapour loss or pressure build-up.

7.2.2.2 The sample shall receive only the minimum treatment to ensure homogeneity, to minimize the possible loss of volatile constituents. After removing each test portion, immediately close the sample

container tightly to ensure that no volatile components escape from the container. If this closure is not secure, obtain a new sample.

7.2.2.3 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. For viscous samples, 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.

8 Procedure

8.1 Record the absolute barometric pressure of the laboratory at the time of the test.

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

8.2 Inspect the test cup for cleanliness and absence of contamination. Use an absorbent paper tissue to wipe clean, if necessary.

8.3 This test method may be used at test temperatures up to 100 °C. However, if no test temperature information is given then test at 60,5 °C and 75,0 °C.

The test is essentially made up of three steps.

a) An equilibrium time of 30 s or 60 s while the sample is heated in the test cup to the test temperature.

b) A 15 s period after the ignition source has been put, and remains, in the “test” position, after which the ignition source is returned into the "off" position.

c) A further 15 s period after the ignition source has been returned to the “off “position.

If the sustained-combustibility test is to be reported at a specified temperature (e.g. given in regulations or specifications), calculate the adjusted test temperature using the specified temperature by correcting for the effect of atmospheric pressure (see Clause 10). Use this adjusted temperature for setting the test cup temperature.

8.4 Set the heater control so that the combustibility tester is at the required and stable temperature.

8.5 Open the gas control valve and ignite the ignition source with the jet away from the test position (i.e. in the “off” position, away from the test cup). Adjust the ignition source using the flow control valve so that its width conforms to the size of the flame gauge ring (see Annex A).

8.6 Charge a clean and dry syringe or pipette with a 2,0 ml ± 0,1 ml test portion of the sample and completely discharge this test portion into the test cup. Take care not to lose any sample. Immediately start the timing device (5.5).

8.7 The operator shall take appropriate safety precautions during the transfer of the test portion to the test cup and the initial application of the ignition source to the test portion. Samples containing low flash point material can ignite violently.

8.8 Test criteria are as defined in 8.8.1 to 8.8.3.

8.8.1 After a heating time of 60 s ± 2 s, the test portion is deemed to have reached its equilibrium temperature.

If the test portion vapour ignites and burns for more than 15 s, while the ignition source is in the “off” position the sample sustains combustion and no further testing is required: proceed to 8.9.

Carefully move the ignition source into the "test" position over the edge of the test cup. Maintain it in this position for 15 s ± 1 s and then return it to the “off” position while observing and recording the behaviour (see 8.8.2 and 8.8.3) of the test portion.

The ignition source shall remain alight throughout each test. See Clause 4. for an overview of the test sequence.

8.8.2 If the test portion vapour ignites during the 15 s the ignitor is in the “test” position and the test portion vapour continues to burn, after the ignition source has been returned to the “off” position, for at least 15 s, the sample sustains combustion and no further testing is required.

8.8.3 Intermittent flashing shall not be interpreted as sustained combustion. Normally, at the end of 15 s, the combustion has either clearly ceased or continues. In cases of doubt, the product shall be deemed to sustain combustion.

8.9 Turn off the ignition source at the end of each test using the gas control valve, and if necessary, the power to the heater. When the temperature of the metal block of the combustibility tester reaches a safe level, remove the used test portion and clean the test cup.

8.10 If the previous test did not sustain combustion repeat the test twice (two more tests), using a new test portion for each test.

If combustion is sustained beyond 20 s the flame may be safely extinguished rather than waiting for the flame to extinguish itself naturally.

8.11 If the result is that sustained combustion is not found when using a heating time of 60 s, repeat the procedure in 8.4 to 8.10 with new test portions but with a heating time of 30 s ± 1 s.

8.12 If tests at the default temperature of 60,5 °C have not resulted in sustained combustion then repeat the procedure in 8.4 to 8.10 at a test temperature of 75,0 °C.

NOTE For materials containing volatile combustible compounds, a 30 s heating time minimizes the loss of these volatile compounds.

9 Assessment of results

Particular care needs to be taken in translating results from this test method to large scale (real life) situations, as liquids in large quantities can behave in different ways to small samples.

If, during any of the tests, on test samples, carried out at different equilibrium times and different test temperatures, the vapour of the test portion continues to burn for at least 15 s with the test flame in the “off” position, the sample is assessed as sustaining combustion and no further testing is required.

When verifying the apparatus using reference materials, as described in Annex B, tests are always carried out at 60,5 °C and 75,0 °C, as both results are required.

10 Calculation of the adjusted test temperature

If the absolute barometric pressure reading taken in Clause 8 is in a unit other than kilopascals, convert the reading to kilopascals using the following: