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ISO 9038

Second edition
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Determination of sustained combustibility of liquids

Détermination de la combustion entretenue des liquides

Reference number
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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. www.iso.org/directives

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The committee responsible for this document is ISO/TC 28, *Petroleum products and lubricants*, (WG 9) in conjunction with Technical Committee ISO/TC 35, *Paints and varnishes*.

This second edition cancels and replaces the first edition (ISO 9038:2002), which has been technically revised.

The main technical changes are the inclusion of 3 reference materials for verification in Annex B.

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, may not. This International Standard 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, gives off sufficient flammable vapour to cause ignition when an ignition source is applied, and continues to generate sufficient vapour to burn when the ignition source is removed.

This test method does not determine the flash point of the product under test but, by means of a pass/fail procedure, merely determines if it sustains combustion (fail) at a selected test temperature, as can be required to comply with laws or regulations relating to the storage, transport and use of flammable products. Before performing this test, it will normally be necessary to determine either the actual flash point of the material or the temperature range in which the flash point is located.

The apparatus specified in this International Standard 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 International Standard may involve hazardous materials, operations or 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 to determine the applicability of regulatory limitations prior to use.

1 Scope

This International Standard specifies a pass/fail procedure, at temperatures up to 100 °C, to determine whether or not a liquid product, that would be classified as "flammable" by virtue of its flash point, has the ability to sustain combustion at the temperature or temperatures specified in the appropriate regulations.

NOTE 1 Many national and international regulations classify liquids as presenting a flammable hazard on the basis of 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 or temperatures.

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

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

NOTE 3 This test method can be used, in addition to test methods for flash point, in assessing the fire hazard of a product.

NOTE 4 Particular care needs to be taken in translating results from this test method to large scale (real life) situations, as liquids in large quantities may not behave in the same way as small samples.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. 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.

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, as measured in the prescribed manner, of a test portion, corrected to a barometric pressure of 101,3 kPa, at which application of an ignition source 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

4 Principle

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

The ability of the product to sustain combustion is assessed on the basis of its ability to ignite, when exposed to an ignition source, and whether it continues to burn after the ignition source has been removed.

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 up to and including 100 °C.

NOTE The combustibility tester, heater and heater control unit can 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.

5.4.1 This shall be suitable for horizontal operation, and of suitable range and dimensions.

5.4.2 Resolution to be able to be read to the nearest 0,5 °C.

5.4.3 Accuracy ± 0,5 °C.

5.4.4 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.

NOTE It is recommended that the accuracy of the temperature measuring device be checked at least every 12 months or when indicated by a user verification check schedule.

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 can 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. The ignition source can be fuelled by natural gas, coal gas, butane or any other gas found to be suitable. The fuel supply to the gas jet shall be 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.

5.9 Barometer, measuring absolute pressure, with an accuracy of 0,5 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

6.1 Do not carry out the test 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 (see Notes 1 and 2). It can be necessary to surround the tester on three sides with a draught shield (5.8) for protection. If a fume hood is used, minimize the exhaust draught.

NOTE 1 The air speed within 50 mm of the top of the test cup should preferably be less than 0,05 m/s.

NOTE 2 Because the combustibility tester has an open test cup, the apparatus should always be used with a draught shield in place.

7 Sampling

7.1 Paints, varnishes and related products

Take a representative sample of the product to be tested, as described in ISO 15528, and 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 freedom from contamination. Use an absorbent paper tissue to wipe clean, if necessary.

8.3 If the sustained-combustibility test is to be carried out at a temperature specified in regulations or specifications, calculate the adjusted temperature using the specification temperature by correcting for the effect of atmospheric pressure (see Clause 11). Use this adjusted temperature for the test.

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.

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. Immediately start the timing device (5.5). Take care not to lose any sample.

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 give a violent ignition.

8.8 After a heating time of 60 s ± 2 s, by which time the test portion is deemed to have reached its equilibrium temperature, and if the test portion has not ignited, 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 the behaviour of the test portion. The ignition source shall remain alight throughout the test.

8.9 Turn off the pilot flame and ignition source 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 instrument.

Carry out the determination in duplicate, using a new test portion for each test. If duplicate determinations do not give the same result, carry out an additional single determination; this determination is the result.

For each determination observe and record:

a) whether or not there is ignition and sustained burning or flashing of the test portion before the ignition source is moved into the test position.

b) whether the test portion ignites while the ignition source is in the test position and, if so, how long combustion is sustained after the ignition source is returned to the "off" position.

NOTE If combustion is sustained beyond 20 s, it is permissible to extinguish the flame safely rather than wait for the flame to extinguish itself naturally.

8.10 If the result is that sustained combustion is not found, repeat the procedure with new test portions but with a heating time of 30 s ± 1 s.

NOTE For materials containing low concentrations of volatile combustible compounds a 30 s heating time will minimize the loss of these volatile compounds.

9 Assessment of results

Assess the product either as not sustaining combustion (pass) or as sustaining combustion (fail). Report sustained combustion at either of the heating times if one of the following has occurred:

a) when the ignition source is in the “off” position, the test portion ignites.

b) the test portion ignites while the ignition source is in the “test” position, maintained for 15 s, and continues to burn for more than 15 s after the ignition source has been returned to the “off” position.

Intermittent combustion, for less than 15 s, 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 and hence to fail.

10 Verification

Verify the correct functioning of the apparatus in accordance with Annex B.

11 Calculation of temperature adjustment

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:

Reading in hPa × 0,1 = kPa

Reading in mbar × 0,1 = kPa

Reading in mmHg × 0,133 322 = kPa

Correct the specified test temperature (for example 60,0 °C or 75,0 °C) as follows:

$$t_a = t_s - 0{,}25(101{,}3 - p)$$

where

t_a is the adjusted test temperature in degrees Celsius;

t_s is the specified test temperature in degrees Celsius at 101,3 kPa;

p is the ambient barometric pressure, in kilopascals;

0,25 is a constant with dimensions degrees Celsius per kilopascal;

101,3is the standard pressure in kilopascals.

NOTE 1 For all practical purposes, a 4 kPa change in atmospheric pressure is equivalent to a 1,0 °C change in the test temperature. The temperature correction is positive for pressures above 101,3 kPa, and negative for pressures below 101,3 kPa.

NOTE 2 The above equation has been proven for barometric pressures down to 82,0 kPa and is strictly correct only up to 104,7 kPa. Atmospheric pressures corrections at pressures down to 82,0 kPa have been shown to be valid in an ISO 3679 study.[3]

12 Precision

Precision data are not yet available.

13 Test report

The test report shall contain at least the following information:

a) all details necessary for complete identification of the product tested;

b) a reference to this International Standard (ISO 9038:2013);

c) any deviation, by agreement or otherwise, from the test procedure specified;

d) the ambient atmospheric pressure;

e) the temperature of the final result at which the test was carried out, corrected for barometric pressure;

f) whether or not the product sustained combustion and, if combustion was sustained, the heating time (30 s or 60 s);

g) the date of the test.

Annex A
(normative)

Combustibility tester

A combustibility tester consists of a block of aluminium alloy or other corrosion-resistant metal of high thermal conductivity (see Figure A.1). The block has a concave depression (test cup) and a well drilled to take a temperature measuring device (5.4). A small gas jet assembly on a swivel is attached to the block. The handle and gas inlet for the gas jet can be fitted at any convenient angle to the gas jet. The height of the centre of the gas jet above the top of the test cup is 2,2 mm ± 0,1 mm (see Figure A.2). A gauge ring 4,0 mm ± 0,5 mm in diameter shall be engraved on top of the tester near the ignition source (5.7) when it is in the "off" position. The dimensions of the block and test cup assembly are given in Table A.1 and the dimensions of the gas jet assembly are given in Table A.2. The sensing element of the temperature measuring device shall be located centrally under the test cup as shown in Figure A.1.

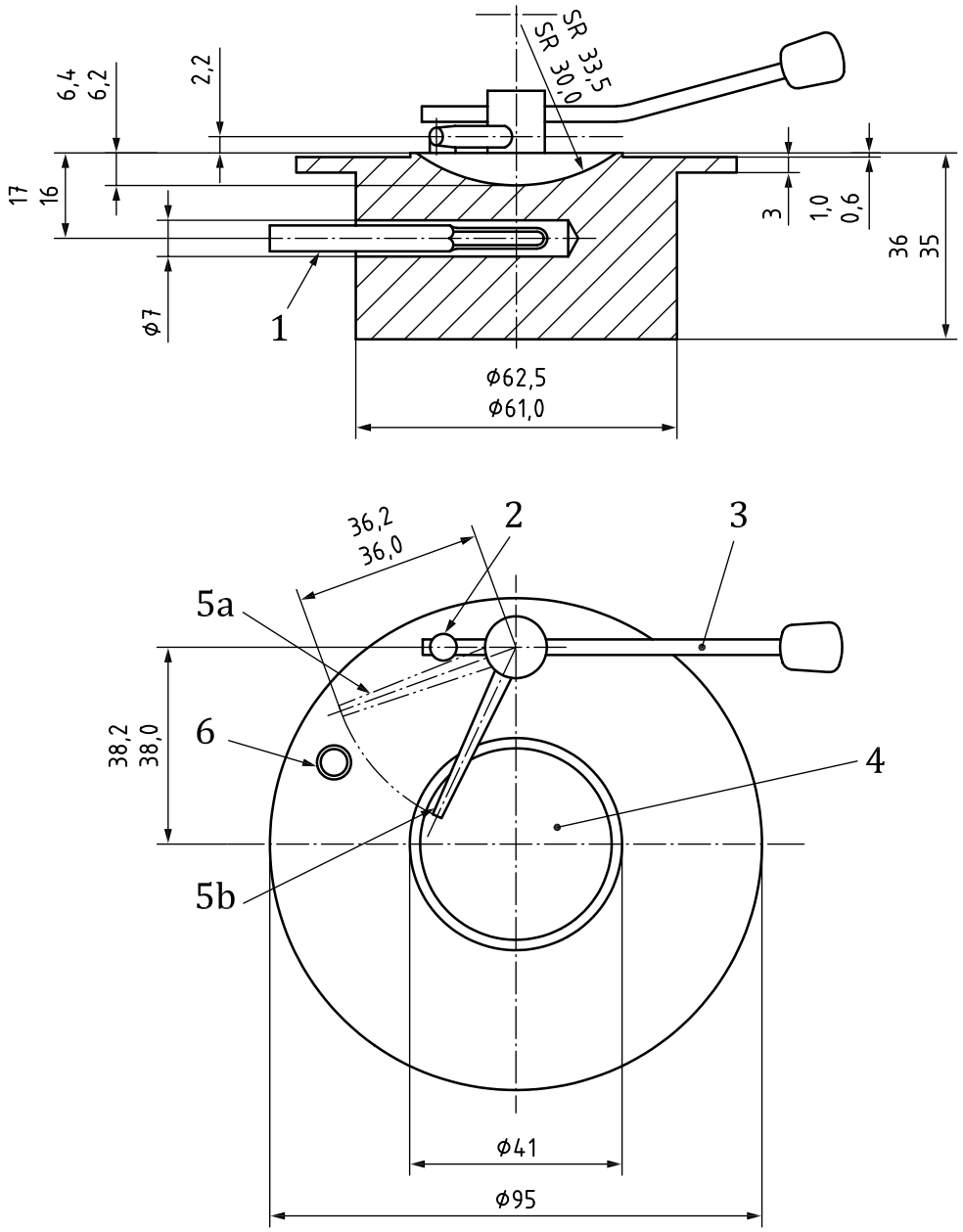
Table A.1 — Dimensions of block and test cup assembly (see Figure A.1)

Property	Dimension
Diameter of block mm	61,0 to 62,5
Height of block mm	35,0 to 36,0
Diameter of flange mm	approx. 95,0
Thickness of flange mm	approx. 3,0
Height of test cup lip above flange mm	0,6 to 1,0
Outside diameter of test cup lip mm	approx. 41,0
Spherical radius of test cup mm	33,0 to 33,5
Depth of test cup mm	6,2 to 6,4
Distance from top of block to temperature measuring device well (centre) mm	16,0 to 17,0
Diameter of flame gauge ring mm	4,0 ± 0,5
Diameter of temperature measuring device well mm	approx. 7,0[a]
Angle subtended by jet from stop ("off" position) to "test" position	45°
[a] The diameter of the temperature measuring device well can be reduced to fit the device used.	

Table A.2 — Dimensions of gas jet assembly (see Figure A.2)

Property	Dimension
Outside diameter of jet mm	3,0 to 4,0
Jet end taper to mm	1,7 to 2,3
Bore of jet mm	0,6 to 0,8
Length of jet from centre of pivot to tip mm	36,0 to 36,2
Distance of axis of pivot from centre of test cup mm	38,0 to 38,2
Height of centreline of jet above top of test cup lip mm	2,2 ± 0,1

Dimension in millimetres

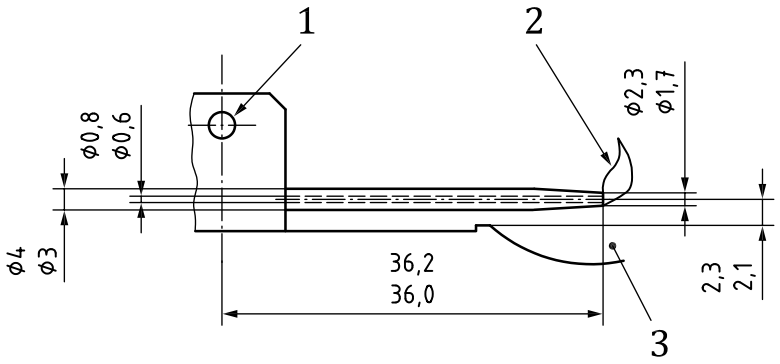


Key

1 Temperature measuring device
2 Stop
3 Handle
4 Test cup
5a Ignition source in “off” position
5b Ignition source in “test” position
6 Gauge ring

Figure A.1 — Block and test cup assembly

Dimension in millimetres



Key

1 Gas inlet
2 Ignition source
3 Test cup

Figure A.2 — Gas jet assembly

Annex B
(normative)

Apparatus verification

B.1 General

This verification procedure shall be used to check the performance of the apparatus. Apparatus shall be checked at least every 12 months or as indicated by a user verification check schedule.

B.2 Reference materials

B.2.1 n-Decane, purity > 99 % (mass fraction).

B.2.2 n-Undecane, purity > 99 % (mass fraction).

B.2.3 n-Dodecane, purity > 99 % (mass fraction).

Table B.1 — Results for verification of the apparatus

Reference material	Test temperature	
	60 °C (adjusted; see Clause 11)	75 °C (adjusted; see Clause 11)
n-Decane	fail	fail
n-Undecane	pass	fail
n-Dodecane	pass	pass

The pass fail criteria were obtained from an interlaboratory study[4]

B.3 Procedure

Use only a 60 s heating time to verify the performance of the apparatus. Use the procedure given in Clauses 6, 7 and 8 with all three reference materials listed in Clause B.2. Use test temperatures of 60,0 °C and 75,0 °C.

B.4 Expression of results

Verification will be confirmed if the results presented in Table B.1 are reproduced, where:

a) “fail” denotes the test portion supports combustion by the criteria described in Clause 9;

b) “pass” denotes that the test portion does not support combustion by the criteria described in Clause 9.

If the results given by the test apparatus meet the required verification performance, record this fact in a permanent record.

If the results given by the test apparatus do not meet the required verification performance, check that the immediate environment of the test cup is free of draughts, and that adequate heat-transfer paste surrounds the temperature measuring device.

If the apparatus continues to give results which do not meet the required responses in Table B.1, recheck the apparatus for conformity with the apparatus specification and the purity of the reference material. If no obvious nonconformity can be identified, contact the manufacturer.

Bibliography

[1] GLOBALLY HARMONIZED SYSTEM OF CLASSIFICATION AND LABELLING OF CHEMICALS (GHS), Fourth revised edition, UNITED NATIONS, New York and Geneva, CH

[2] Recommendations on the Transport of Dangerous Goods, Model Regulations 17. Revised edition, UNITED NATIONS, New York and Geneva, CH

[3] Research Report Round Robin/Interlaboratory Study ISO 3679 Determination of flash point – rapid equilibrium closed cup apparatus. Energy Institute, 61 New Cavendish Street, London. W1G 7AR. UK

[4] Report on the round robin tests on sustained combustibility according to ISO 9038, Physikalisch-Technische Bundesanstalt (PTB), Bundesallee 100, D-38116 Braunschweig, Germany

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