INTERNATIONAL STANDARD

ISO 9038

First edition
2002-10-15

Test for sustained combustibility of liquids

Essai de combustion entretenue de liquides

Reference number
ISO 9038:2002(E)

ISO copyright office
Case postale 56 • CH-1211 Geneva 20
Tel. + 41 22 749 01 11
Fax + 41 22 749 09 47
E-mail copyright@iso.ch
Web www.iso.ch

Printed in Switzerland

Contents

Page

Annexes

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 3.

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 International Standard may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

International Standard ISO 9038 was prepared by Technical Committee ISO/TC 35, *Paints and varnishes*, Subcommittee SC 9, *General test methods for paints and varnishes*.

It cancels and replaces ISO/TR 9038:1991, which has been technically revised.

Annexes A and B form a normative part of this International Standard.

Introduction

A product with a flash point within a given range may 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, after ignition under controlled laboratory conditions and subsequent removal of the flame, sustain combustion and those which do not.

The method determines whether a flammable product, when maintained at a selected test temperature and under the conditions of test, gives off sufficient flammable vapour at this temperature to cause ignition when an external source of flame is applied in a standard manner, and continues to generate sufficient vapour to burn when the ignition source is removed.

This method of test 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 at a selected test temperature, as may 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).

Test for sustained combustibility of liquids

WARNING — The use of this International Standard may involve hazardous materials, operations or equipment. This standard does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and to determine the applicability of regulatory limitations prior to use.

1 Scope

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. This International Standard specifies a pass/fail procedure 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 In the United Nations Recommendations on the Transport of Dangerous Goods, temperatures of 60,5 °C and 75,0 °C are specified.

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 2 This test method can be used in addition to the test method for flash point in assessing the fire hazard of a product. Particular care needs to be taken in translating results from small-scale tests to large-scale (real-life) situations, as liquids in large quantities may not behave in the same way as small samples because geometry and the surface-to-volume ratio affect rates of heat transfer and evaporation from the surface.

2 Normative references

The following normative documents contain provisions which, through reference in this text, constitute provisions of this International Standard. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the normative documents indicated below. For undated references, the latest edition of the normative document referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

ISO 1513, *Paints and varnishes — Examination and preparation of samples for testing*

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 International Standard, the following terms and definitions apply.

3.1
sustained combustibility
the behaviour of a material, under specified test conditions, whereby its vapour can be ignited by an external ignition source and, after ignition, sufficient flammable vapour is produced for burning to continue after the source of ignition has been removed

3.2
flash point
the lowest temperature 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, under the specified conditions of test

4 Principle

A specified volume of sample is introduced into a concave depression, the test portion well, in a metal block that is maintained at the test temperature. After a specified time a test flame is applied.

The ability of the product to sustain combustion is assessed on the basis of its ability to ignite, when exposed to a test flame, and whether it continues to burn after the standard flame has been removed, under the specified conditions of test.

5 Apparatus

5.1 Combustibility tester, as specified in annex A.

5.2 Hotplate or other suitable heating device, fitted with a temperature control device and which, in contact with the bottom of the combustibility tester, provides efficient transfer of heat. The heater control is required to maintain the combustibility tester at the equilibrium temperature, as measured on the integral thermometer (5.4), in a draught-free area, to within $\pm$ 0,5 °C for test temperatures up to and including 100 °C.

NOTE 1 The combustibility tester, heater and heater control unit may consist of an integrated apparatus.

NOTE 2 Heating is at or near the base of the aluminium block.

5.3 Gauge, for checking that the height of the centre of the gas jet above the top of the test portion well is $(2{,}2 \pm 0{,}1)$ mm. A calibrated metal strip is suitable.

5.4 Thermometer or other suitable temperature-measuring device. A mercury-in-glass thermometer is recommended. This shall be suitable for horizontal operation, of appropriate range and dimensions, with graduations every 1 °C up to 100 °C and be capable of measuring the temperature of the block to an accuracy of $\pm$ 0,5 °C. If another temperature-measuring device is used, it shall be of equivalent sensitivity, permitting readings at 0,5 °C intervals. When in position in the block, the thermometer bulb or temperature-measuring sensor 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 thermometer or other measuring device be checked, annually or when indicated by a verification check schedule, by an authorized laboratory or by comparison against a calibrated thermometer with a valid calibration certificate, using the stipulated immersion depth.

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

5.6 Syringe, capable of delivering 2 ml to an accuracy of $\pm$ 0,05 ml.

5.7 Test flame and gas supply. The flame may 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 $(4{,}0 \pm 0{,}5)$ 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.

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

5.9 Barometer, readable to, and with an accuracy of, 0,1 kPa (1 mbar). 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

WARNING — Do not carry out the test in a small confined area (for example a glove box) because of the risk of explosions.

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

Position the metal block on the hotplate (5.2) and place the assembly, or the integral unit, on a level, stable surface and away from strong light (to facilitate observation of a flash, flame, etc.). Ensure that the top of the metal block is exactly horizontal.

Use the gauge (5.3) to check that the jet is $(2{,}2 \pm 0{,}1)$ mm above the top of the block (see Figure A.2).

It is essential that the apparatus is set up in a completely draught-free area (see warning above). It may 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 Because the combustibility tester has an open test portion well, it is recommended that the apparatus is always used with a draught shield in place.

7 Samples and sampling

7.1 Products

7.1.1 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.

7.1.2 Petroleum and related products

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

7.1.3 Other products

For products other than paints and petroleum-based materials, e.g. aqueous solutions of alcohols, exercise suitable care to ensure that a representative sample of the product is removed for testing.

7.2 Procedure

A 2 ml test portion is required for each test. Obtain a sample of at least 50 ml of product and store in a clean, tightly closed container in a cool place to minimize vapour loss or pressure build-up. 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.

Because of the possibility of loss of volatile constituents, the sample shall receive only the minimum treatment to ensure its homogeneity. 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 incomplete, an entirely new sample shall be taken.

If necessary, warm or cool the sample in its container to 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.

WARNING — If it is necessary to warm the sample in its container, take care to ensure that dangerously high pressures do not develop.

8 Procedure

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

NOTE 1 It is not considered necessary to correct the barometric pressure reading from ambient temperature to 0 °C, although some barometers automatically make this correction.

Inspect the test portion well for cleanliness and freedom from contamination. Use an absorbent paper tissue to wipe clean, if necessary.

If the sustained-combustibility test is to be carried out at a temperature specified in regulations or specifications, adjust the specified temperature to correct for the effect of atmospheric pressure (see clause 11). Use this adjusted temperature for the test.

Adjust the heater control so that the combustibility tester is at the specified temperature, adjusted as necessary for atmospheric pressure, and allow the temperature to stabilize.

Open the gas control valve and ignite the test flame with the jet away from the test position (i.e. in the “off” position, away from the test portion well). Adjust the test flame using the flow control valve so that its width conforms to the size of the $(4{,}0 \pm 0{,}5)$ mm gauge ring.

Charge a clean and dry syringe with a $(2{,}0 \pm 0{,}1)$ ml test portion of the sample and discharge this test portion into the test portion well by fully depressing the syringe plunger. Immediately start the timing device (5.5). Take care not to lose any sample.

WARNING — The operator shall take appropriate safety precautions during the transfer of the test portion to the well and the initial application of the test flame to the test portion. Samples containing low-flash-point material may give a violent ignition. The use of safety glasses is recommended.

After a heating time of (60 ± 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 test flame into the test position over the edge of the pool of liquid. Maintain it in this position for (15 ± 1) s and then return it to the “off” position while observing the behaviour of the test portion. The test flame shall remain alight throughout the test.

Turn off the pilot and test flames 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 test portion and clean the instrument.

Carry out the determination in duplicate, using a new test portion for each test. If the results are inconclusive, repeat the determination.

For each test observe and record:

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

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

NOTE 2 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.

If sustained combustion, interpreted in accordance with clause 9, is not found, repeat the procedure with new test portions but with a heating time of (30 ± 1) s.

9 Interpretation of observations

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 occurs with either of the test portions:

a) when the test flame is in the “off” position, the test portion ignites;

b) the test portion ignites while the test flame is in the “test” position, maintained for 15 s, and continues to burn for more than 15 s after the test flame 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 and standardization

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

If the apparatus fails, check that the immediate environment of the test portion well is free of draughts, and that adequate heat-transfer paste surrounds the thermometer bulb and the immersed portion of the thermometer barrel.

11 Calculation of temperature adjustment

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

10 hPa $=$ 1 kPa

10 mbar $=$ 1 kPa

7,5 mmHg $=$ 1 kPa

If the sustained-combustibility test is to be carried out at a temperature specified in regulations or specifications at a specified pressure, normally 101,3 kPa, correct the specified temperature for atmospheric pressure before the test as follows:

$$T_{\mathrm{a}} = T_{\mathrm{t}} - 0{,}25\,(101{,}3 - p)$$

where

T_{a} is the adjusted test temperature;

T_{t} is the specified test temperature at 101,3 kPa;

p is the ambient barometric pressure, in kilopascals.

NOTE For all practical purposes, a 4 kPa change in atmospheric pressure is equivalent to a 1 °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.

12 Precision

Precision data are not quoted for this method, as it is a pass/fail procedure.

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);

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

d) the ambient atmospheric pressure;

e) the temperature or temperatures 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 name of the laboratory;

h) 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 portion well) and a hole (pocket) drilled to take a thermometer (5.4). A small gas jet assembly on a swivel is attached to the block. The handle and gas inlet for the gas jet may 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 portion well is $(2{,}2 \pm 0{,}1)$ mm (see Figure A.2). A gauge ring $(4{,}0 \pm 0{,}5)$ mm in diameter shall be engraved on top of the tester near the test flame (5.7) when it is in the "off" position. The dimensions of the block and test portion well assembly are given in Table A.1 and the dimensions of the gas jet assembly are given in Table A.2.

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

Dimensions in millimetres

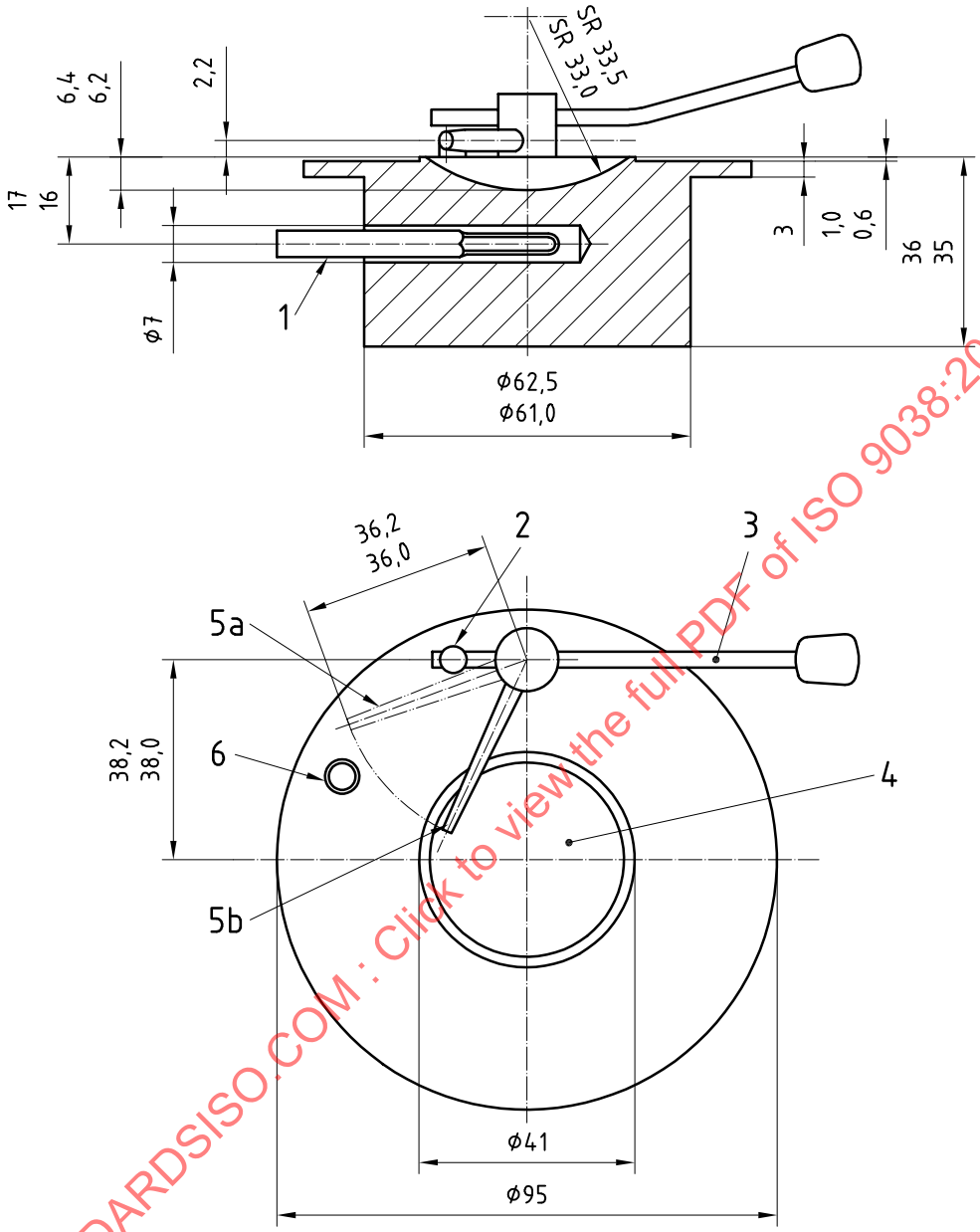
Diameter of block	61,0 to 62,5
Height of block	35,0 to 36,0
Diameter of flange	95,0 (approx.)
Thickness of flange	3,0 (approx.)
Height of (test portion) well lip above flange	0,6 to 1,0
Outside diameter of (test portion) well lip	41,0 (approx.)
Spherical radius of (test portion) well	33,0 to 33,5
Depth of (test portion) well	6,2 to 6,4
Distance from top of block to thermometer hole (centre)	16,0 to 17,0
Diameter of flame gauge ring	$4{,}0 \pm 0{,}5$
Diameter of thermometer pocket	7,0 (approx.)
Angle subtended by jet from stop ("off" position) to "test" position	45°

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

Dimensions in millimetres

Outside diameter of jet	3,0 to 4,0
Jet end taper to	1,7 to 2,3
Bore of jet	0,6 to 0,8
Length of jet from centre of pivot to tip	36,0 to 36,2
Distance of axis of pivot from centre of well	38,0 to 38,2
Height of centreline of jet above top of (test portion) well lip	$2{,}2 \pm 0{,}1$

Dimensions in millimetres



Key

1	Thermometer
2	Stop
3	Handle
4	Test portion well
5a	Test flame in “off” position
5b	Test flame in “test” position
6	Gauge ring

Figure A.1 — Block and test portion well assembly