
**Determination of flash/no flash — Rapid
equilibrium closed cup method**

*Essai de point d'éclair de type passe/ne passe pas — Méthode rapide à
l'équilibre en vase clos*

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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 2.

The main task of technical committees is to prepare International Standards. 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 document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 3680 was prepared jointly by Technical Committees ISO/TC 28, *Petroleum products and lubricants* and ISO/TC 35, *Paints and varnishes*.

This fourth edition cancels and replaces the third edition (ISO 3680:1983), which has been technically revised.

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Introduction

This International Standard describes one of two closed cup equilibrium methods for carrying out a flash/no flash test for paints, varnishes, paint binders, solvents, adhesives, petroleum and related products. When selecting a method, it should therefore be read in conjunction with the second method, ISO 1516 [4]. When used in conjunction with the flash detector (A.1.6) this International Standard is also suitable for the flash/no flash testing of fatty acid methyl esters (FAME).

In both ISO 3680 and ISO 1516, the test is only carried out when the material under test and the air/vapour mixture above the material in the test cup are approximately in temperature equilibrium.

This test method does not determine the flash point of the product under test, but merely its behaviour at the selected test temperature as may be required to comply with laws or regulations relating to the storage, transport and use of flammable products. For this purpose, it is unnecessary to determine the exact flash point, but it is necessary to determine whether or not flashing occurs at a given temperature.

The apparatus specified in this International Standard enables a similar test result to be determined using a more rapid procedure and a smaller test portion (2 ml or 4 ml) than that required in ISO 1516. In addition, the apparatus can be made portable to the extent of being suitable for on-site testing in addition to its more normal use in laboratories.

Collaborative work (see [6] in the Bibliography) has shown that results obtained by these procedures are comparable. 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 (see [7] in the Bibliography).

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 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.

Determination of flash/no flash — Rapid equilibrium closed cup method

WARNING — The use of this International Standard may involve hazardous materials, operations and 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 determine the applicability of regulatory limitations prior to use.

1 Scope

This International Standard specifies a method for the determination of the ability of paints (including water-borne paints), varnishes, paint binders, adhesives, solvents and petroleum and related products, when maintained at a selected test temperature within the range of $-30\text{ }^{\circ}\text{C}$ to $300\text{ }^{\circ}\text{C}$, and under the conditions of test, to yield sufficient flammable vapour at this temperature to cause ignition on the application of a test flame in a standard manner. When used in conjunction with the flash detector (A.1.6), this International Standard is also suitable to carry out a flash/no flash test on fatty acid methyl esters (FAME).

2 Normative references

The following referenced documents are indispensable for the application 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 1513:1992, *Paints and varnishes — Examination and preparation of samples for testing*

ISO 3170:2004, *Petroleum liquids — Manual sampling*

ISO 3171:1988, *Petroleum liquids — Automatic pipeline sampling*

ISO 3679:2004, *Determination of flash point — Rapid equilibrium closed cup method*

ISO 15528:2000, *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

flash/no flash test

application of a test flame at the specified temperature of the test portion (as measured in the prescribed manner), adjusted to a barometric pressure of $101,3\text{ kPa}$, to determine whether the vapours of the test portion ignite momentarily and a flame propagates 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, a test flame is applied and the presence or absence of a flash observed.

5 Reagents and materials

5.1 Cleaning solvent

An appropriate solvent for the removal of traces of the previous test portion from the test cup and cover.

NOTE The choice of solvent will depend 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 such as toluene-acetone-methanol may be effective for the removal of gum-type deposits.

5.2 Verification liquids

A series of certified reference materials (CRM) and/or secondary working standards (SWS) as described in Annex C.

6 Apparatus

6.1 General

The apparatus is described in Annex A, and this includes the details of the test cup and cover assembly, together with dimensions and special requirements. These are shown in Figure A.1 to Figure A.5. Tests for flash/no flash over the whole range of $-30\text{ }^{\circ}\text{C}$ to $300\text{ }^{\circ}\text{C}$ may require more than one instrument.

6.2 Syringes

6.2.1 2 ml syringe, adjusted to deliver $2,00\text{ ml} \pm 0,05\text{ ml}$ and equipped, if required, with a needle suitable for use with the apparatus at test temperatures up to and including $100\text{ }^{\circ}\text{C}$. When testing FAME, a 2 ml test portion is used at all temperatures.

6.2.2 5 ml syringe, adjusted to deliver $4,00\text{ ml} \pm 0,10\text{ ml}$ and equipped, if required, with a needle suitable for use with the apparatus at test temperatures above $100\text{ }^{\circ}\text{C}$. A 5 ml syringe is not required when testing FAME.

6.3 Barometer, accurate to $0,1\text{ kPa}$. Barometers precorrected to give sea-level readings, such as those used at weather stations and airports, shall not be used.

6.4 Heating bath or oven (optional), for warming the samples, if required, and capable of controlling the temperature to $\pm 5\text{ }^{\circ}\text{C}$. If an oven is used, it shall be intrinsically safe for hydrocarbon vapours.

It is recommended that the oven be of explosion-protected design.

6.5 Cooling bath or freezer (optional), for cooling the samples, if required, and capable of cooling the sample to $10\text{ }^{\circ}\text{C}$ below the selected test temperature, and controlling the temperature of the sample to $\pm 5\text{ }^{\circ}\text{C}$. If a freezer is used, it shall be of an explosion-protected design.

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

NOTE A shield, 350 mm high, 480 mm wide and 240 mm deep, is suitable.

6.7 Cup insert (optional). See Annex D.

NOTE For samples that are difficult to remove, a thin metal cup insert can be used.

7 Apparatus preparation

7.1 General

Select the appropriate instrument for the required flash/no flash temperature. If a flash/no flash test is to be carried out at a temperature specified in regulations or specifications, the specified temperature shall be adjusted for atmospheric barometric pressure before the test (see 11.2). Follow the manufacturer's instructions for the correct set-up and operation of the apparatus. The use of a cup insert (6.7) for potentially adherent materials is described in Annex D.

7.2 Location of apparatus

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

Use of a draught shield (6.6) is recommended when protection from draughts is not available.

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

7.3 Cleaning of the test cup 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. Follow the manufacturer's instructions for the care and servicing of the instrument.

NOTE 1 A stream of clean dry air may be used to remove the last traces of solvent used.

NOTE 2 The filler orifice may be conveniently cleaned with a pipe cleaner.

7.4 Apparatus verification

7.4.1 Verify the correct functioning of the apparatus at least once a year by testing a certified reference material (CRM) (5.2) in accordance with ISO 3679. The result obtained shall be equal to or less than $R/\sqrt{2}$ from the certified value of the CRM, where R is the reproducibility of the method (see 13.3).

It is recommended that more frequent verification checks be made using secondary working standards (SWs) (5.2).

NOTE A recommended procedure for apparatus verification using CRMs and SWs, and the production of SWs, is given in Annex C.

7.4.2 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.

If the instrument fails the verification test, it is recommended that the operator should check that:

- 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 thermometer bulb and immersed portion of the thermometer barrel.

8 Sampling

8.1 Unless otherwise specified, obtain samples in accordance with the procedures given in 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 storing the 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 in a cooling bath or freezer (6.5), 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 is to be stored prior to testing, ensure that the container is still filled to at least 85 % 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/no flash tests may be affected if the sample volume falls below 85 % of the container capacity.

9.1.2 Samples that are liquid at ambient temperature. If sufficiently fluid, mix samples gently shaking by hand prior to the removal of the test portion, taking care to minimize the loss of volatile components. If the sample is too viscous at ambient temperature, heat the sample in its container using a heating bath or oven (6.4) to a temperature not warmer than 10 °C below the test temperature, such that the sample can be mixed by gentle shaking.

9.1.3 Solid or semi-solid samples. 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 whilst the cover is open.

9.2 Paints, varnishes and related materials

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

10 Procedure

10.1 General

10.1.1 Use the equation in 11.2 to determine the test temperature. Follow the manufacturer's instructions for setting the required test temperature.

10.1.2 When testing fatty acid methyl esters (FAME), the flash point detector (A.1.6) shall be used.

10.1.3 Do not apply the test flame to the test portion more than once. Use a new test portion of the sample for each test. After each test, turn off the pilot and test flames using the gas control valve, and when the test cup temperature falls to a safe level, remove the test portion and clean the instrument.

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

NOTE 1 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.

NOTE 2 If a continuous luminous flame burns in the orifice when the slide is opened and the test flame is introduced, then the flash point lies considerably below the test temperature. In such cases, ISO 3679 may be used to determine the actual flash point.

10.1.5 Record the ambient barometric pressure using a barometer (6.3) 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.

10.2 Procedure for flash/no flash tests up to and including 100 °C and for FAME at all temperatures

10.2.1 Charge a clean and dry syringe (6.2.1), adjusted to a temperature at least 10 °C below the expected flash point, with a 2 ml test portion. Close the sample container immediately after withdrawal to minimize the loss of volatile components.

10.2.2 Carefully transfer the syringe to the filler orifice and discharge the test portion into the test cup by fully depressing the syringe plunger. Remove the syringe.

10.2.3 For solid or semi-solid samples, transfer a mass equivalent to approximately 2 ml directly into the test cup and spread it over the bottom of the cup as evenly as possible.

10.2.4 Start the 1 min timing device (A.1.3). Open the gas control valve and ignite the pilot and test flames. Adjust the test flame to conform to the size of the 4 mm gauge ring. Reset the flash detector (A.1.6) if fitted.

10.2.5 When the audible time signal sounds, apply the test flame by slowly and uniformly opening and then closing the shutter over a period of 2 s to 3 s. Check for a flash (see 10.1.4 and note 2 to 10.1.4) and record the result.

10.3 Procedure for flash/no flash tests above 100 °C [except when testing FAME (see 10.2)]

10.3.1 Charge a clean and dry syringe (6.2.2) with a 4 ml test portion. Close the sample container immediately after withdrawal to minimize the loss of volatile components.

10.3.2 Carefully transfer the syringe to the filling orifice and discharge the test portion into the test cup by fully depressing the syringe plunger. Remove the syringe.

10.3.3 For solid or semi-solid samples, transfer a mass equivalent to approximately 4 ml directly into the test cup and spread it over the bottom of the cup as evenly as possible.

10.3.4 Start the 2 min timing device (A.1.3). Open the gas control valve and ignite the pilot and test flames. Adjust the test flame with the pinch valve to conform to the size of the 4 mm gauge ring. Reset the flash detector (A.1.6) if fitted.

10.3.5 When the audible time signal sounds, apply the test flame by slowly and uniformly opening and then closing the shutter over a period of 2 s to 3 s. Check for a flash (see 10.1.4 and note 2 to 10.1.4) and record the result.

11 Calculation

11.1 Conversion of barometric pressure reading

If the barometric pressure reading is measured in a unit other than kilopascals, convert to kilopascals using one of the following equations:

$$\text{Reading in hPa} \times 0,1 = \text{kPa}$$

Reading in mbar $\times 0,1 = \text{kPa}$

Reading in mmHg $\times 0,133\ 3 = \text{kPa}$

11.2 Adjustment of specified temperature

If a flash/no flash test is to be carried out at a temperature specified in regulations or specifications, the specified temperature shall be adjusted for ambient barometric pressure before the test, and this adjusted temperature, rounded to the nearest $0,5\text{ }^{\circ}\text{C}$, used as the test temperature. Determine the required test temperature, T_T , from the following equation:

$$T_T = T_S - 0,25(101,3 - p)$$

where

T_S is the specified temperature, expressed in degrees Celsius;

p is the ambient barometric pressure, expressed in kilopascals.

NOTE This equation is strictly correct only within the barometric pressure range from $98,0\text{ kPa}$ to $104,7\text{ kPa}$.

12 Expression of results

Report the specified flash/no flash temperature T_S as shown in 11.2, or the flash/no flash test temperature T_T corrected to the standard atmospheric pressure of $101,3\text{ kPa}$, rounded to the nearest $0,5\text{ }^{\circ}\text{C}$, and whether there was a flash or no flash.

Report the ambient barometric pressure in the vicinity of the apparatus (see 10.1.5).

Report if a continuous luminous flame is present in the orifice.

13 Precision

13.1 General

Precision data are not quoted for this test method. However, when selecting a test temperature to be specified for this method, a knowledge of the precision of the procedure is required to estimate margins and the general reliability of the result. This estimate is obtained from the precision of ISO 3679 at the test temperature, an analogous method to determine the actual flash point using the same apparatus. The precision of ISO 3679, as determined by statistical examination of interlaboratory test results, is given in 13.2, 13.3 and Table 1.

13.2 Repeatability, r

The difference between two test results, obtained by the same operator with the same apparatus under constant operating conditions on identical test material, would in the long run, in the normal and correct operation of the test method, exceed the values given in Table 1 in only one case in 20.

13.3 Reproducibility, R

The difference between two single and independent test results, obtained by different operators working in different laboratories on identical test material, would in the long run, in the normal and correct operation of the test method, exceed the values given in Table 1 in only one case in 20.

Table 1 — Precision values

Range	Repeatability °C	Reproducibility °C
Petroleum and related products		
20 °C to 70 °C	0,5	$0,03(X + 29)$
Above 70 °C	$0,022 X^{0,9}$	$0,083 X^{0,9}$
Paints, enamels, lacquers and varnishes		
5,8 mm ² /s at 37,8 °C and below	1,7	3,3
Above 5,8 mm ² /s at 37,8 °C	3,3	5,0
Fatty acid methyl esters (FAME)	1,9	15,0
NOTE where X is the average of the results being compared.		

NOTE The following values have been calculated from the precision values given in Table 1 for petroleum and related products.

Temperature °C	Repeatability °C	Reproducibility °C
20	0,5	1,4
70	0,5	2,9
93	1,3	4,9
150	2,0	7,5
200	2,6	9,9
260	3,3	12,4

14 Test report

The test report shall contain at least the following information:

- a reference to this International Standard;
- the type and complete identification of the product tested;
- the result of the test (see Clause 12);
- any deviation, by agreement or otherwise, from the procedures specified;
- the date of the test.

Annex A (normative)

Flash point test apparatus

A.1 Apparatus

A.1.1 Test cup assembly

A.1.1.1 General. The plan and details of the test cup assembly are illustrated in Figure A.1 to Figure A.5.

A.1.1.2 Metal block, made of aluminium alloy or non-rusting metal of similar thermal conductivity, with a cylindrical depression (test cup), and with a hole in the side to accommodate a temperature sensor (A.1.4). When in position, the temperature sensor (thermometer bulb) shall be surrounded with a suitable thermal-conducting thermoplastic compound (see the note below).

NOTE Silicone heat-sink compounds have been found to be suitable.

A.1.1.3 Cover, fitted with an opening slide and a device which permits insertion of a test flame of diameter $4 \text{ mm} \pm 0,5 \text{ mm}$ into the test cup when the slide is open. When inserted, the nozzle of the test flame shall intersect the plane of the underside of the cover within $\pm 0,1 \text{ mm}$. The cover shall be provided with an orifice extending into the test cup for insertion of the test portion, and also a suitable clamping device for securing the cover tightly to the metal block. The three openings in the cover shall be within the diameter of the test cup. The slide shall be fitted with a spring or other device to ensure that it stays in the fully closed position when shut. When the slide is in the open position, the two openings in the slide shall coincide with the two corresponding openings in the cover. The O-ring shall be made of heat-resistant material and provide a tight seal when the cover is shut.

NOTE The dipping of the test flame may be automated on some instruments.

A.1.1.4 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 integral thermometer, and in a draught-free area, within $\pm 0,5 \text{ }^\circ\text{C}$ for test temperatures up to and including $100 \text{ }^\circ\text{C}$, and within $\pm 2,0 \text{ }^\circ\text{C}$ for test temperatures above $100 \text{ }^\circ\text{C}$ during the test.

A.1.2 Test flame and pilot flame

The flames shall be fuelled by any suitable flammable gas (for example, natural gas, reticulated town gas or liquefied petroleum gases). A gauge ring 4 mm in diameter shall be engraved on the cover near the test flame.

A.1.3 Timing device

The timing device is capable of emitting an audible signal after $60 \text{ s} \pm 2 \text{ s}$ and $120 \text{ s} \pm 4 \text{ s}$.

A.1.4 Temperature sensor

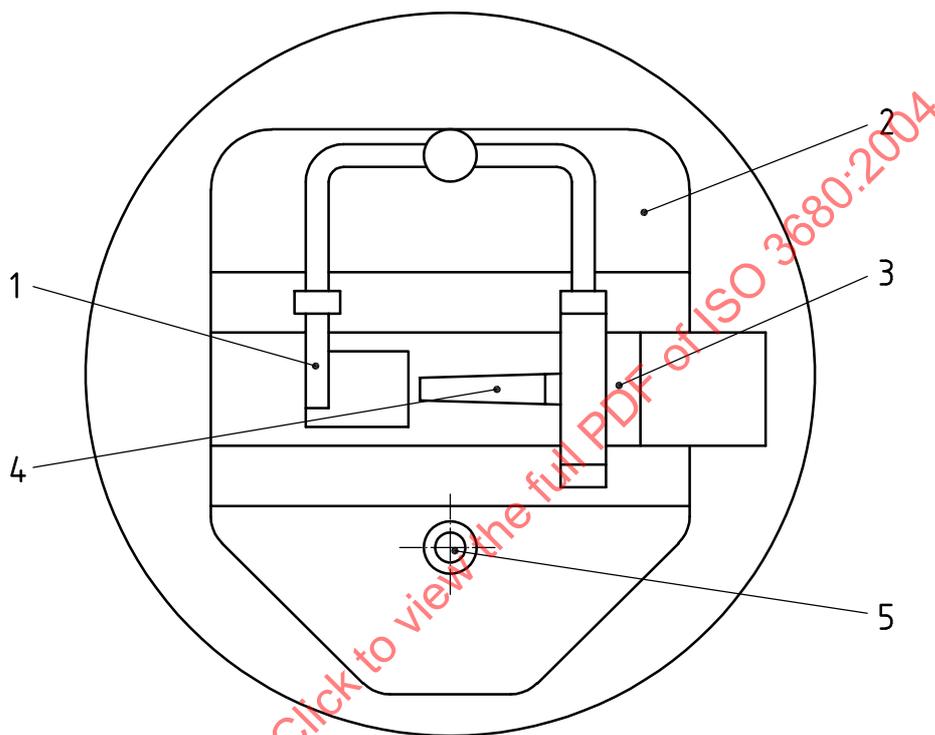
A liquid-in-glass thermometer conforming to the requirements specified in Annex B, or an alternative temperature measuring device and/or system of equivalent accuracy. The initial choice of temperature sensor shall be based on the expected flash point of the material under test.

A.1.5 Test cup cooler (optional)

An electronic Peltier or other suitable cooling device.

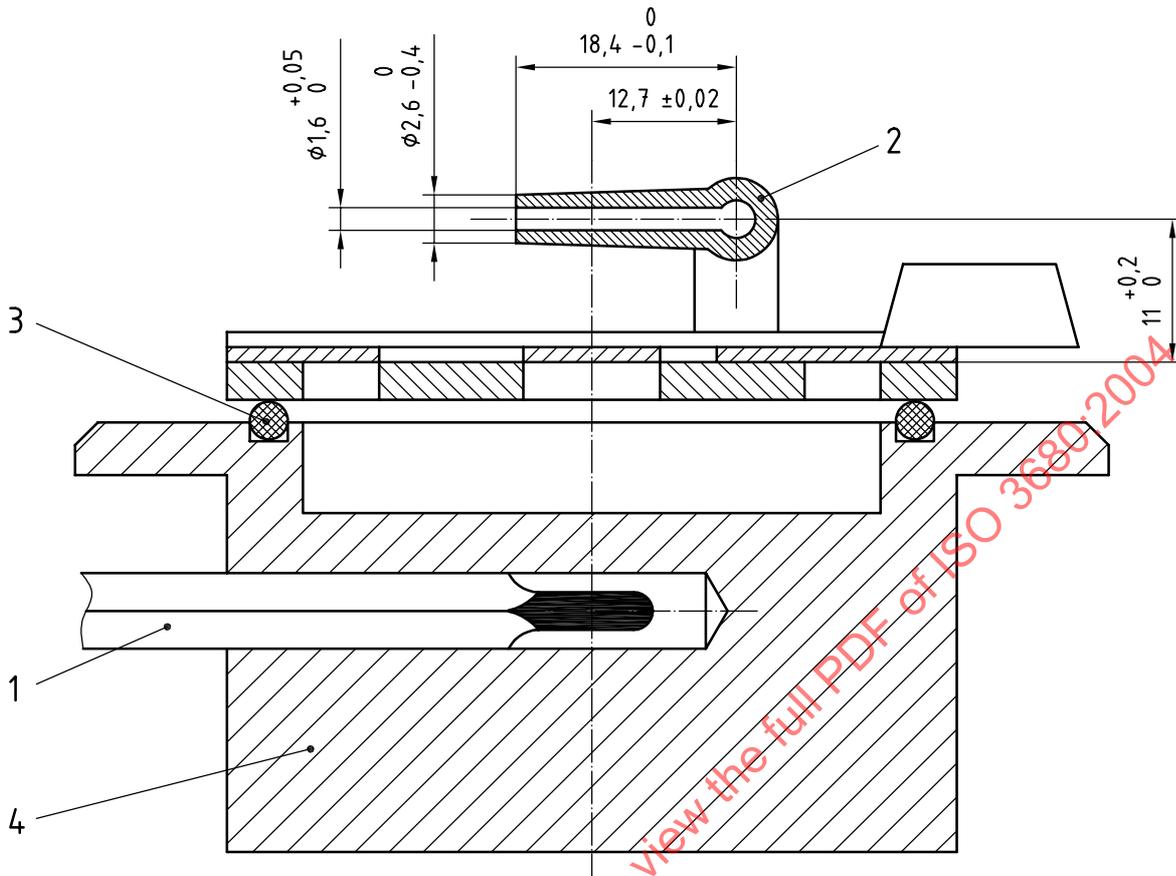
A.1.6 Flash detector (optional)

A low-mass thermocouple device for the detection of the flash point flame. A flash is indicated if a temperature rise of 6,0 °C is detected within 100 ms.

**Key**

- 1 nozzle of pilot flame
- 2 cover
- 3 slide
- 4 nozzle of test flame
- 5 filler orifice

Figure A.1 — Plan diagram of test cup assembly



Key

- 1 temperature sensor
- 2 nozzle of test flame
- 3 "O" seal
- 4 sample block

Figure A.2 — Section of block through nozzle of test flame

Dimensions in millimetres

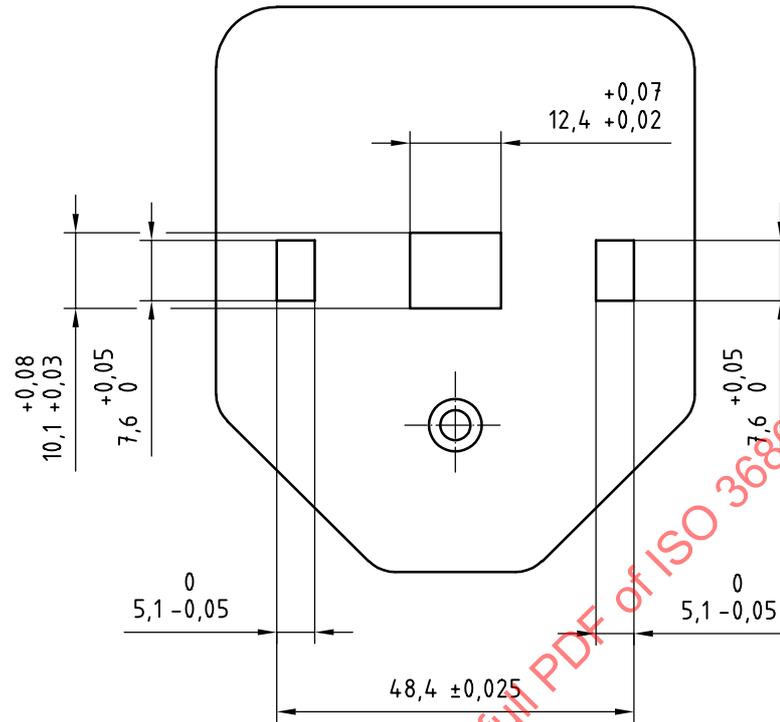


Figure A.3 — Cover

Dimensions in millimetres

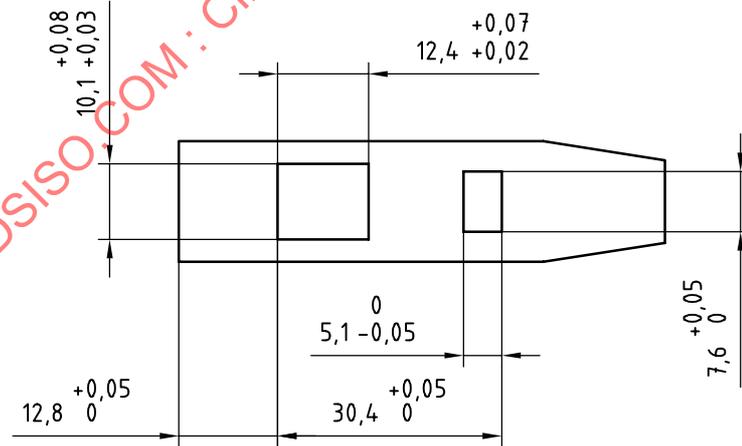


Figure A.4 — Slide

Dimensions in millimetres

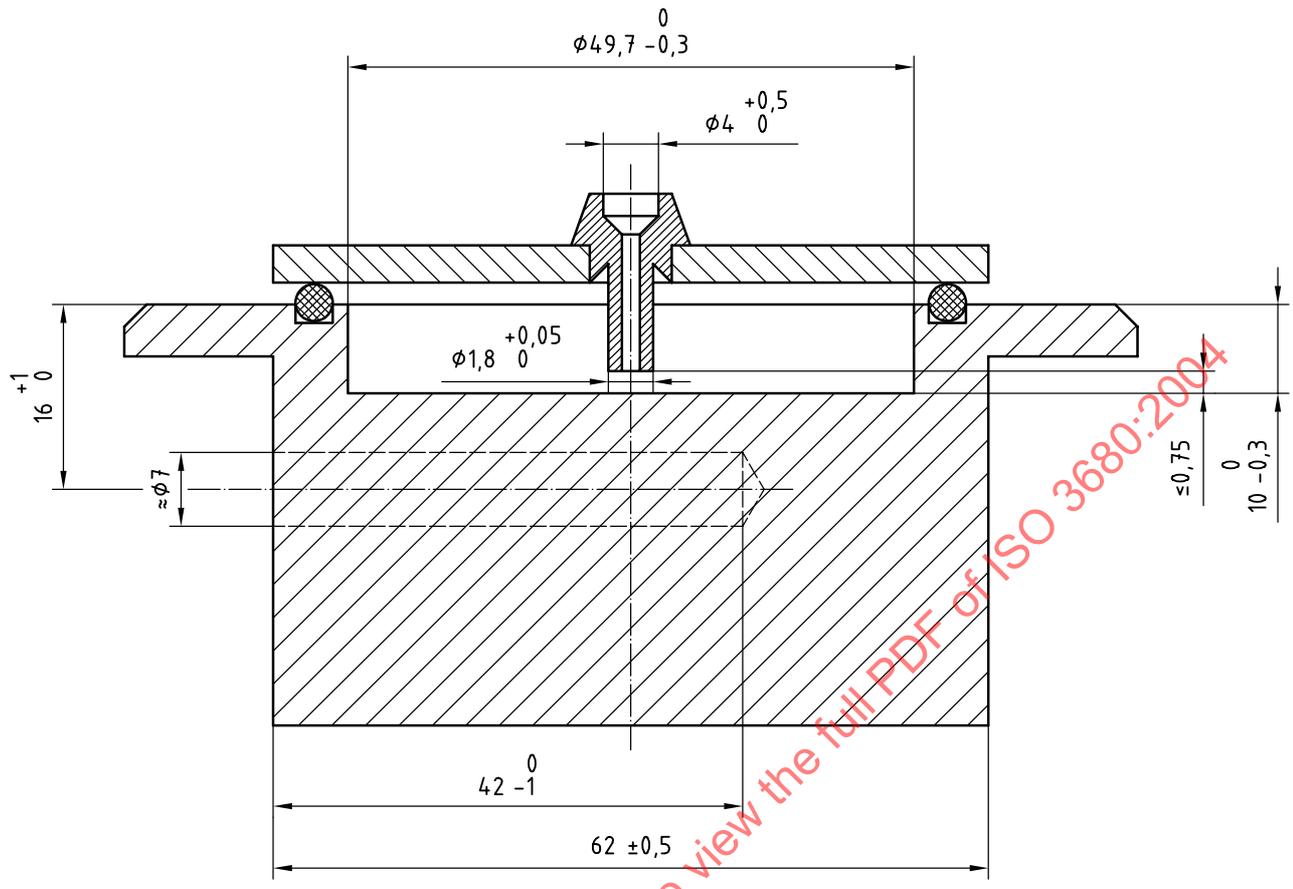


Figure A.5 — Section of test block through filler orifice

Annex B (normative)

Thermometer specifications

B.1 Liquid-in-glass thermometers

Table B.1 gives the specifications for liquid-in-glass thermometers specified in A.1.4. Alternative temperature measurement devices and/or systems shall meet the requirements for scale accuracy, bearing in mind the partial immersion nature of these thermometers.

Table B.1 — Thermometer specifications

Type	Sub-zero	Low range	High range
Temperature range, °C	– 30 to 100	0 to 110	100 to 300
Immersion, mm	44	44	44
Scale marks, °C:			
Subdivisions	1	1	2
Long lines at each	5	5	10
Numbers at each	10	10	10
Maximum line width, mm	0,15	0,15	0,15
Scale error, °C, maximum	0,5	0,5	2,0
Expansion chamber	Required	Required	Required
Total length, mm	195 to 200	195 to 200	195 to 200
Stem OD ^a , mm	6 to 7	6 to 7	6 to 7
Bulb length, mm	10 to 14	10 to 14	10 to 14
Bulb OD ^a , mm	4 to 6	4 to 6	4 to 6
Scale location:			
Bottom of bulb to line at, °C	– 30	0	100
Distance, mm	57 to 61	48 to 52	48 to 52
Length of scale range, mm	115 to 135	115 to 135	115 to 135
NOTE Thermometers IP 91C (low range) and IP 98C (high range) meet the above requirements. There is currently no numbered thermometer for the sub-zero range.			
^a OD: outer diameter.			

Annex C (informative)

Verification of apparatus

C.1 General

This annex describes a procedure for producing a secondary working standard (SWS) and conducting verification checks using a SWS and a certified reference material (CRM).

The performance of the apparatus should be verified on a regular basis using either a CRM produced in accordance with ISO Guide 34 [2] and ISO Guide 35 [3], or an in-house reference material/SWS prepared in accordance with one of the procedures given in C.2.2. The performance of the apparatus should be assessed in accordance with the guidance given in ISO Guide 33 [1] and ISO 4259 [5].

The evaluation of the test results assumes a 95 % confidence limit for the trueness of the result.

C.2 Verification check standards

C.2.1 Certified reference material (CRM), comprising a stable single hydrocarbon or other stable substance with a flash point determined in accordance with ISO Guide 34 and ISO Guide 35, using a method-specific interlaboratory study to produce a method-specific certified value.

C.2.2 Secondary working standard (SWS), comprising a stable petroleum product or single hydrocarbon or other stable substance with a flash point determined either by

- a) testing representative subsamples at least three times using an instrument previously verified using a CRM, statistically analysing the results and, after the removal of any outliers, calculating the arithmetic mean of the results, or
- b) conducting an interlaboratory method-specific test programme utilizing at least three laboratories testing representative samples in duplicate. The assigned value of the flash point should be calculated after statistically analysing the interlaboratory data.

Store SWSs in containers which will retain the integrity of the SWS, out of direct sunlight, at a temperature not exceeding 10 °C.

C.3 Procedure

C.3.1 Choose a CRM or SWS which falls within the range of flash points to be determined with the apparatus. See Table C.1 for approximate flash point values.

It is recommended that at least two CRMs or SWSs are used in order to cover as wide a range as possible. In addition, it is also recommended that replicate tests be carried out on aliquots of the CRM or SWS.

C.3.2 For new apparatus, and at least once a year for working apparatus, conduct a verification check using a CRM (C.2.1) tested in accordance with Clause 10.

C.3.3 For intermediate verification, conduct a verification check using a SWS (C.2.2) tested in accordance with Clause 10.