

BS EN ISO 3679:2015  
BS 2000-523:2015



BSI Standards Publication

# Determination of flash no- flash and flash point — Rapid equilibrium closed cup method

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### **National foreword**

This British Standard is the UK implementation of EN ISO 3679:2015. It supersedes BS EN ISO 3679:2004/BS 2000-523:2004 and BS EN ISO 3680:2004/BS 2000-524:2004, which are withdrawn.

The UK participation in its preparation was entrusted to Technical Committee PTI/13, Petroleum Testing and Terminology.

A list of organizations represented on this committee can be obtained on request to its secretary.

This publication does not purport to include all the necessary provisions of a contract. Users are responsible for its correct application.

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### **Amendments/corrigenda issued since publication**

Date	Text affected
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English Version

## Determination of flash no-flash and flash point - Rapid equilibrium closed cup method (ISO 3679:2015)

Détermination de l'éclair de type passe/ne passe pas et du point d'éclair - Méthode rapide à l'équilibre en vase clos (ISO 3679:2015)

Bestimmung des Flammpunktes mit dem Ja/Nein-Verfahren - Nach dem schnellen Gleichgewichtsverfahren mit geschlossenem Tiegel (ISO 3679:2015)

This European Standard was approved by CEN on 14 January 2015.

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EUROPEAN COMMITTEE FOR STANDARDIZATION  
COMITÉ EUROPÉEN DE NORMALISATION  
EUROPÄISCHES KOMITEE FÜR NORMUNG

**CEN-CENELEC Management Centre: Avenue Marnix 17, B-1000 Brussels**

## Foreword

This document (EN ISO 3679:2015) has been prepared by Technical Committee ISO/TC 28 "Petroleum products and related products of synthetic or biological origin" in collaboration Technical Committee CEN/TC 19 "Gaseous and liquid fuels, lubricants and related products of petroleum, synthetic and biological origin" the secretariat of which is held by NEN.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by September 2015, and conflicting national standards shall be withdrawn at the latest by September 2015.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. CEN [and/or CENELEC] shall not be held responsible for identifying any or all such patent rights.

This document supersedes EN ISO 3679:2004, EN ISO 3680:2004.

According to the CEN-CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Bulgaria, Croatia, Cyprus, Czech Republic, Denmark, Estonia, Finland, Former Yugoslav Republic of Macedonia, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Romania, Slovakia, Slovenia, Spain, Sweden, Switzerland, Turkey and the United Kingdom.

### Endorsement notice

The text of ISO 3679:2015 has been approved by CEN as EN ISO 3679:2015 without any modification.

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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.

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](http://www.iso.org/directives)).

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

For an explanation on the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the WTO principles in the Technical Barriers to Trade (TBT) see the following URL: [Foreword - Supplementary information](#)

The committee responsible for this document is ISO/TC 28, *Petroleum products and lubricants*, working Group 9, in conjunction with Technical Committee ISO/TC 35, *Paints and varnishes*, Technical Committee CEN/TC 19, *Gaseous and liquid fuels, lubricants and related products of petroleum, synthetic and biological origin*, and Technical Committee CEN/TC 139, *Paints and varnishes*.

This fourth edition cancels and replaces ISO 3679:2004 and ISO 3680:2004, which have been technically revised. The main technical changes are:

- incorporation of ISO 3680 flash point technique into the flash/no flash technique as a separate procedure due to the fact that many apparatus on the market combine both tests;
- title change;
- revision of temperature measuring device requirements;
- new precision covering both gas and electric ignition.

## Introduction

This International Standard is a closed cup equilibrium test method for the determination of the flash/no-flash and flash point of paints, varnishes, binders for paints and varnishes, solvents, adhesives, petroleum, and related products. ISO 1516<sup>[1]</sup> and ISO 1523<sup>[2]</sup> are also closed cup equilibrium test methods that are to be considered when selecting a method.

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 and ISO 1523. 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<sup>[3]</sup> has shown that results obtained by these methods 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.<sup>[4]</sup>

A limited study has indicated that some water borne paints can give an elevated flash point when an electric ignitor is used with ISO 3679.

Flash point can be used in shipping, storage, handling, and safety regulations, as a classification property to define “flammable” and “combustible” materials. Precise definition of the classes is given in each particular regulation.

The flash point can indicate the presence of highly volatile material(s) in a relatively non-volatile or non-flammable material and flash point testing can be a preliminary step to other investigations into the composition of unknown materials.

It is not appropriate for flash point determinations to be carried out on potentially unstable, decomposable, or explosive materials, unless it has been previously established that heating the specified quantity of such materials in contact with the metallic components of the flash point apparatus, within the temperature range required for the method, does not induce decomposition, explosion or other adverse effects.

The flash point is not a constant physical-chemical property of a material tested. It is a function of the apparatus design, the condition of the apparatus used, and the operational procedure carried out. Flash point can therefore only be defined in terms of a standard test method, and no general valid correlation can be guaranteed between results obtained by different test methods or with test apparatus different from that specified.

ISO/TR 29662<sup>[5]</sup> (an adoption of CEN/TR 15138<sup>[6]</sup>) gives useful advice in carrying out flash point tests and interpreting results.





# Determination of flash no-flash and flash point — Rapid equilibrium closed cup method

**WARNING** — The use of this International Standard can 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 procedures for flash point tests, within the temperature range of  $-30\text{ }^{\circ}\text{C}$  to  $300\text{ }^{\circ}\text{C}$ , for paints, including water-borne paints, varnishes, binders for paints and varnishes, adhesives, solvents, petroleum, and related products. The procedures are used to determine whether a product will or will not flash at a specified temperature (flash no-flash Procedure A) or the flash point of a sample (Procedure B). When used in conjunction with the flash detector ([A.1.6](#)), this International Standard is also suitable to determine the flash point of fatty acid methyl esters (FAME).

## 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 4259, *Petroleum products — Determination and application of precision data in relation to methods of test*

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

#### **flash point**

lowest temperature, as measured in the prescribed manner, of the 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

### 3.2

#### **flash no-flash**

application of an ignition source at the specified temperature of the test portion, as measured in the prescribed manner, adjusted to a barometric pressure of 101,3 kPa, to determine whether the vapours of the test portion ignite momentarily and a flame propagate across the surface of the liquid under the specified conditions of test

### 3.3 fatty acid methyl ester FAME

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

Note 1 to entry: FAME is specified in specifications such as EN 14214<sup>[Z]</sup> and ASTM D6751.<sup>[8]</sup>

## 4 Principle

A test portion of specified volume is introduced into the test cup that is set and maintained at the required test temperature. After a specific time, an ignition source is applied and a determination is made (Procedure A) as to whether or not a flash occurred. In order to determine the actual flash point of the sample, further tests, with fresh test portions at different test cup temperatures, are carried out (Procedure B) until the flash point is determined.

## 5 Reagents and materials

**5.1 Cleaning solvent**, for the removal of traces of sample from the test cup and cover.

The choice of solvent will depend upon the previous material tested and the tenacity of the residue. Low volatility aromatic (benzene-free) solvents can be used to remove traces of oil, and mixed solvents can be effective for the removal of gum-type deposits.

**5.2 Reference materials**, series of certified reference materials (CRM) and/or secondary working standards (SWS) as described in [Annex B](#).

**5.3 Gas for ignitor and pilot flame**, not required if an electric ignitor is used.

Butane, propane, coal gas, or natural gas can be used.

## 6 Apparatus

**6.1 Flash point apparatus**, as specified in [Annex A](#).

**6.2 Barometer**, reading absolute pressure, accurate to  $\pm 0,5$  kPa and with a resolution of 0,1 kPa.

Barometers pre-corrected to give sea level readings, such as those used at weather stations and airports, shall not be used.

**6.3 Heating bath or oven**, for warming the sample, if required, and capable of controlling the temperature to an accuracy of  $\pm 5$  °C.

The oven shall be ventilated and constructed in such a way that it will not cause ignition of any flammable vapours that can be produced when the sample is heated. It is recommended that the oven is of explosion-protected design.

**6.4 Cooling bath or freezer (optional)**, for cooling the samples, if required, and capable of cooling the sample to 10 °C below the expected flash point, and controlling the temperature to an accuracy of  $\pm 5$  °C.

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

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

## 6.6 Cup insert (optional), see [Annex C](#).

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

## 6.7 Syringes

**6.7.1 Syringe**, capable of delivering  $2,00 \text{ ml} \pm 0,05 \text{ ml}$  and equipped with a nozzle suitable for the required test temperature and apparatus.

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

**6.7.2 Syringe**, capable of delivering  $4,00 \text{ ml} \pm 0,10 \text{ ml}$  and equipped with a nozzle suitable for the required test temperature and apparatus.

# 7 Preparation of apparatus

## 7.1 General

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

**7.1.2** The use of a cup insert ([6.6](#)) for potentially adherent materials is described in [Annex C](#).

**7.1.3** When testing FAME use a  $2 \text{ ml} \pm 0,05 \text{ ml}$  test portion and a  $60 \text{ s} \pm 2 \text{ s}$  test time, combined with an electronic thermal flash detector (see [A.1.6](#)).

**7.1.4** For sub-ambient test temperatures use [Annex D](#), unless the apparatus has integral test cup cooling facilities.

## 7.2 Location of apparatus

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

A draught shield ([6.5](#)) is recommended to be used when protection from draughts is not available.

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

## 7.3 Cleaning of the test cup assembly and accessories

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

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

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

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

## 7.4 Apparatus verification

**7.4.1** Verify and correct, if necessary, the reading of the temperature measuring device (see [Annex E](#)) at least every 12 months, according to the manufacturer's instructions.

**7.4.2** Verify the correct functioning of the apparatus at least once a year, by using a certified reference material (CRM) ([5.2](#)) according to Procedure B. The result obtained, after barometric pressure correction ([11.2](#)) 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 for petroleum and related products (see [13.3](#)).

It is recommended that more frequent verification checks are made using secondary working standards (SWSs) ([5.2](#)).

A recommended procedure for apparatus verification using CRMs and SWSs, and the production of SWSs, is described in [Annex B](#).

**7.4.3** The numerical values obtained during the verification check shall not be used to provide a bias statement, nor shall they be used to make any correction to the flash points subsequently determined using the apparatus.

**7.4.4** If the instrument fails the verification test, it is recommended that the operator should follow the manufacturers' instructions and check the following, and then repeat the verification check:

- a) the cover makes a vapour tight seal with the test cup;
- b) the shutter provides a light tight seal;
- c) adequate heat transfer paste surrounds the temperature measuring device inserted in the test cup block;
- d) the correct operation of the ignition source;
- e) the operation of the flash detector ([A.1.6](#)) (if fitted);
- f) the correct reading of the temperature measuring device.

## 8 Sampling

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

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

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

## 9 Sample handling

### 9.1 Petroleum products and fatty acid methyl esters

#### 9.1.1 Subsampling

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

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

#### 9.1.2 Samples liquid at ambient temperature

If sufficiently fluid, mix samples by gentle hand shaking prior to the removal of the test portion, taking care to minimize the loss of volatile components. Ensure that the sample is at least 10 °C below the selected test temperature before opening to remove the test portion. For mobile materials, mix the sample by gentle shaking. If necessary, heat the sample in its container to a temperature such that the sample can be mixed by gentle shaking or to at least 10 °C below the selected test temperature, whichever is lower. Ensure that high pressures do not develop in the container.

#### 9.1.3 Samples solid or semi-solid at ambient temperature

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

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

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

### 9.2 Paints, varnishes, and related materials

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

## 10 Procedure

### 10.1 General

**10.1.1** Follow the manufacturer's instructions for setting the test temperature.

**10.1.2** When testing fatty acid methyl esters (FAME), a flash detector ([A.1.6](#)) shall be used.

**10.1.3** Use a new test portion of the sample for each test. Do not apply the ignition source to the test portion more than once. After each test, turn off the pilot and test flames (if used) using the gas control valves, and when the test cup temperature falls to a safe level, remove the test portion and clean the instrument.

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

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

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

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

## 10.2 Procedure A — Flash no-flash test

**10.2.1** Inspect the test cup and cover for cleanliness and correct operation, especially with regard to tightness of the cover “O” ring (A.1.1.3), the action of the shutter, the size or intensity of the ignition source, and the position of the ignition source (A.1.2). Clean if necessary (7.3). Put the cover in place and close securely.

**10.2.2** Correct the required test temperature for the flash no-flash test according to the absolute barometric pressure as shown in Formula (1). Allow for any known thermometer correction, and then round to the nearest 0,5 °C.

$$t_t = t_s - 0,25(101,3 - p) \quad (1)$$

where

$t_t$  is the actual test temperature, in degrees Celsius;

$t_s$  is the specification or uncorrected target test temperature, in degrees Celsius;

$p$  is the absolute barometric pressure, in kilopascals;

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

101,3 is the standard pressure, in kilopascals.

**10.2.3** Follow the manufacturer’s instructions to set the test temperature and the test time, and select the test portion volume and test time in accordance with Table 1.

**Table 1 — Test Conditions**

Sample	Test temperatures	Test portion volume	Test time
All except FAME	≤100 °C	2 ml	60 s
All except FAME	>100 °C	4 ml	120 s
FAME	≤300 °C	2 ml	60 s

**10.2.4** When the test cup is at the test temperature, fill the appropriate syringe (6.7) with the sample to be tested. Transfer the syringe to the filling orifice, taking care not to lose any sample. Discharge the test portion into the test cup by fully depressing the syringe plunger. Remove the syringe.

**10.2.5** Start the test timer. Light the pilot light and adjust the test flame (if used) to conform to the 4 mm gauge.

**10.2.6** When the end of the test time is indicated, apply the ignition source by slowly and uniformly opening the shutter and closing it completely over a period of 2 s to 3 s. Watch closely for a flash at the test cup openings if a flash detector is not being used.

The test portion is deemed to have flashed when a flame appears and instantaneously propagates itself over the surface of the test portion. Occasionally, particularly near the actual flash point, application of the test flame can cause a blue halo or an enlarged test flame; this is not a flash and should be ignored.

**10.2.7** Record the test result as “flash” or “no flash”, and the test temperature.

**10.2.8** Turn off the pilot and test flames, if used. When the cup temperature falls to a safe level, remove the used test portion and clean the cup and cover.

### **10.3 Procedure B — Flash point determination**

**10.3.1** This procedure repeats the flash no-flash test described in Procedure A ([10.2.4](#) to [10.2.8](#)) a number of times. Each flash no-flash test uses a fresh test portion and a different test temperature. The testing at different temperatures allows the determination of two test temperatures 1 °C or 0,5 °C apart for which the lower temperature did not result in a flash while the higher temperature resulted in a flash (the flash point).

**10.3.2** Select the expected flash point of the sample as the initial test temperature.

**10.3.3** Follow the manufacturer’s instructions to set the test temperature and the test time, and select the test portion volume and test time in accordance with [Table 1](#).

**10.3.4** When the test cup is at the test temperature, fill the appropriate syringe ([6.7.1](#) and [6.7.2](#)) with the sample to be tested. Transfer the syringe to the filling orifice, taking care not to lose any sample. Discharge the test portion into the test cup by fully depressing the syringe plunger. Remove the syringe.

**10.3.5** Start the test timer. Light the pilot light and adjust the test flame (if used) to conform to the 4 mm gauge.

**10.3.6** When the end of the test time is indicated, apply the ignition source by slowly and uniformly opening the shutter and closing it completely over a period of 2 s to 3 s. Watch closely for a flash at the test cup openings if a flash detector is not being used.

**10.3.7** The test portion is deemed to have flashed when a flame appears and momentarily propagates itself over the surface of the test portion. Occasionally, particularly near the actual flash point, application of the test flame can cause a blue halo or an enlarged test flame. This is not a flash and should be ignored.

**10.3.8** Record the test result as “flash” or “no flash” and the test temperature.

**10.3.9** Turn off the pilot and test flames (if used). Remove the used test portion. Take appropriate measures if the used test portion is hot.

**10.3.10** If a flash is detected, repeat the procedure given in [10.3.3](#) to [10.3.9](#) testing a fresh test portion at a temperature 5 °C lower each time until no flash is detected. Then proceed to [10.3.12](#).

**10.3.11** When no flash is detected repeat the procedure given in [10.3.3](#) to [10.3.9](#) testing a fresh test portion at a temperature 5 °C higher each time until a flash is detected.

**10.3.12** Having established a flash within two temperatures 5 °C apart, repeat the procedure given in [10.3.3](#) to [10.3.9](#) at 1 °C intervals from the lower of the two temperatures until a flash is detected.

**10.3.13** Test a fresh test portion at a temperature 0,5 °C below that at which the flash was detected in [10.3.12](#). If no flash is detected, the temperature recorded in [10.3.12](#) is the flash point to the nearest 0,5 °C. If a flash is detected at the lower temperature, record this latter (lower) temperature as the detected flash point allowing for any known thermometer correction.

NOTE Results obtained directly from [10.3.12](#) without following the more accurate procedure in [10.3.13](#) are permitted but the precision could be affected.

**10.3.14** Turn off the pilot and test flames (if used). When the cup temperature falls to a safe level, remove the used test portion and clean the cup and cover.

## 11 Calculation

### 11.1 Conversion of barometric pressure reading

If the absolute barometric pressure reading, taken in accordance with [10.1.5](#) is in a unit other than kilopascals, convert to kilopascals using one of the following conversions:

- Reading in hPa  $\times 0,1 =$  kPa;
- Reading in mbar  $\times 0,1 =$  kPa;
- Reading in mmHg  $\times 0,133 =$  kPa.

### 11.2 Correction of the detected flash point to standard atmospheric pressure

Calculate the corrected flash point,  $t_c$ , using Formula (2):

$$t_c = t_d + 0,25(101,3 - p) \quad (2)$$

where

- $t_d$  is the detected flash point, in degrees Celsius (see [10.3.13](#));
- $p$  is the absolute barometric pressure, in kilopascals;
- 0,25 is a constant with dimensions degrees Celsius per kilopascal;
- 101,3 is the standard pressure, in kilopascals.

NOTE 1 This correction is only required for Procedure B.

NOTE 2 Formula (2) has been proven for barometric pressures down to 82,0 kPa<sup>[Z]</sup> and is strictly correct only up to 104,7 kPa.

NOTE 3 According to Formula (2), a 4 kPa difference in atmospheric pressure is equivalent to a flash point temperature change of 1 °C.

## 12 Expression of results

Record

- a) the flash no-flash temperature, corrected to standard atmospheric pressure, rounded to the nearest 0,5 °C, or
- b) the corrected flash point,  $t_c$ , rounded to the nearest 0,5 °C.



## 13 Precision

### 13.1 General

The precision given in [13.2](#), [13.3](#), and [Table 2](#) was derived from statistical examination of interlaboratory test results on petroleum and related products, paint and related products, and FAME products.

[Table 3](#) gives examples of precision calculated for petroleum and related products (excluding paints and varnishes).

NOTE 1 Precision has not been determined for samples that are solid at ambient temperature.

NOTE 2 The precision was developed using the ignitors shown in [Table 2](#). If alternative ignitors are used, the precision can be different.

Sample specific bias between results from gas and electric ignitors, as defined by Practice ASTM D6708,<sup>[9]</sup> was observed for some petroleum and related samples. However it is not practicable to include such a bias in the method and hence the precision combines gas and electric ignitors.

Precision data are not quoted for Procedure A of this test method. However, when selecting a test temperature to be specified for this method, 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 Procedure B of this method at the test temperature.

Precision for petroleum and related products, and FAME was developed in accordance with ISO 4259. The precision for paints and related products is not known to have been developed in accordance with ISO 4259.

### 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 2](#) 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 2](#) in only one case in 20.

**Table 2 — Precision values**

Range	Temperature range °C	Ignitor <sup>a</sup>	Repeatability °C	Reproducibility °C
Petroleum and related products <sup>[10]</sup>	20 to 220	G/E	0,015 20 (X + 110) <sup>b</sup>	0,025 61 (X + 110) <sup>b</sup>
Paints and related products with kinematic viscosity <sup>[11]</sup>	≤5,8 mm <sup>2</sup> /s at 37,8 °C	G	1,7	3,3
	>5,8 mm <sup>2</sup> /s at 37,8 °C	G	3,3	5,0
Fatty acid methyl esters (FAME) <sup>[12]</sup>	85 to 175	G	1,9	15,0

<sup>a</sup> E refers to electric ignition and G refers to gas ignition.

<sup>b</sup> Where *X* is the average of the results being compared.

**Table 3 — Calculated repeatability and reproducibility for petroleum and related products, excluding paints, and varnishes**

Temperature, °C	20	40	60	80	100	120	140	160	180	200	220
Repeatability, °C	2,0	2,3	2,6	2,9	3,2	3,5	3,8	4,1	4,4	4,7	5,0
Reproducibility, °C	3,3	3,8	4,4	4,9	5,4	5,9	6,4	6,9	7,4	7,9	8,5

## 14 Test report

The test report shall contain at least the following information:

- a) a reference to this International Standard (i.e. ISO 3679) and Procedure A or B;
- b) the type and complete identification of the product tested;
- c) the result of the test (see [Clause 12](#));
- d) any deviation, by agreement or otherwise, from the procedures specified;
- e) date of the test.

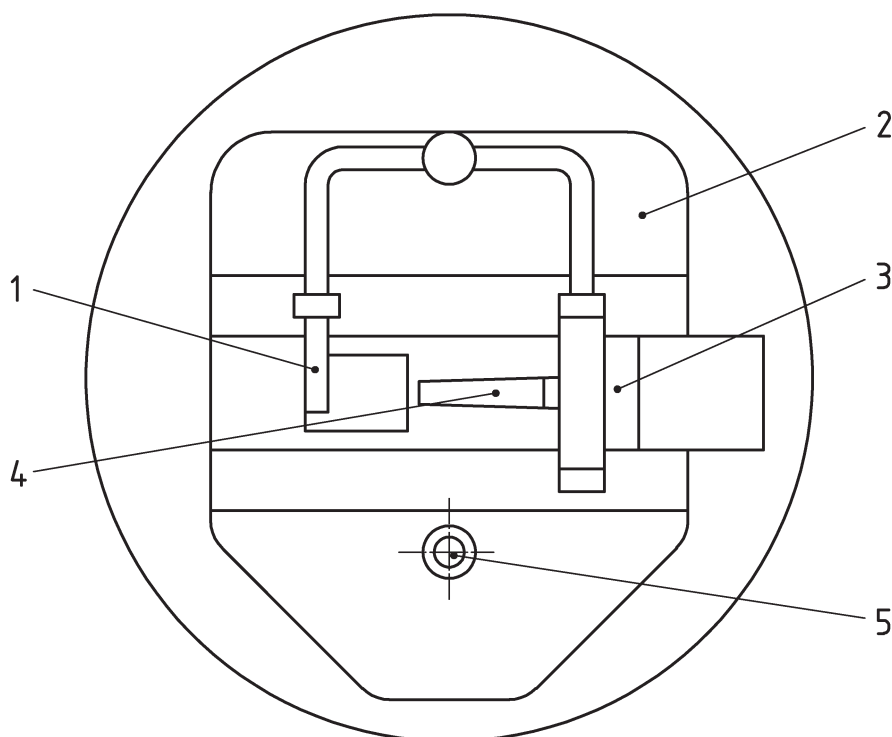
## Annex A (normative)

### Flash point apparatus

#### A.1 Apparatus

##### A.1.1 Test cup and cover assembly

**A.1.1.1 General.** The plan and details of the test cup and cover assembly are illustrated in [Figure A.1](#) to [Figure A.5](#). Some versions of the apparatus do not cover the full  $-30\text{ }^{\circ}\text{C}$  to  $300\text{ }^{\circ}\text{C}$  flash point range.



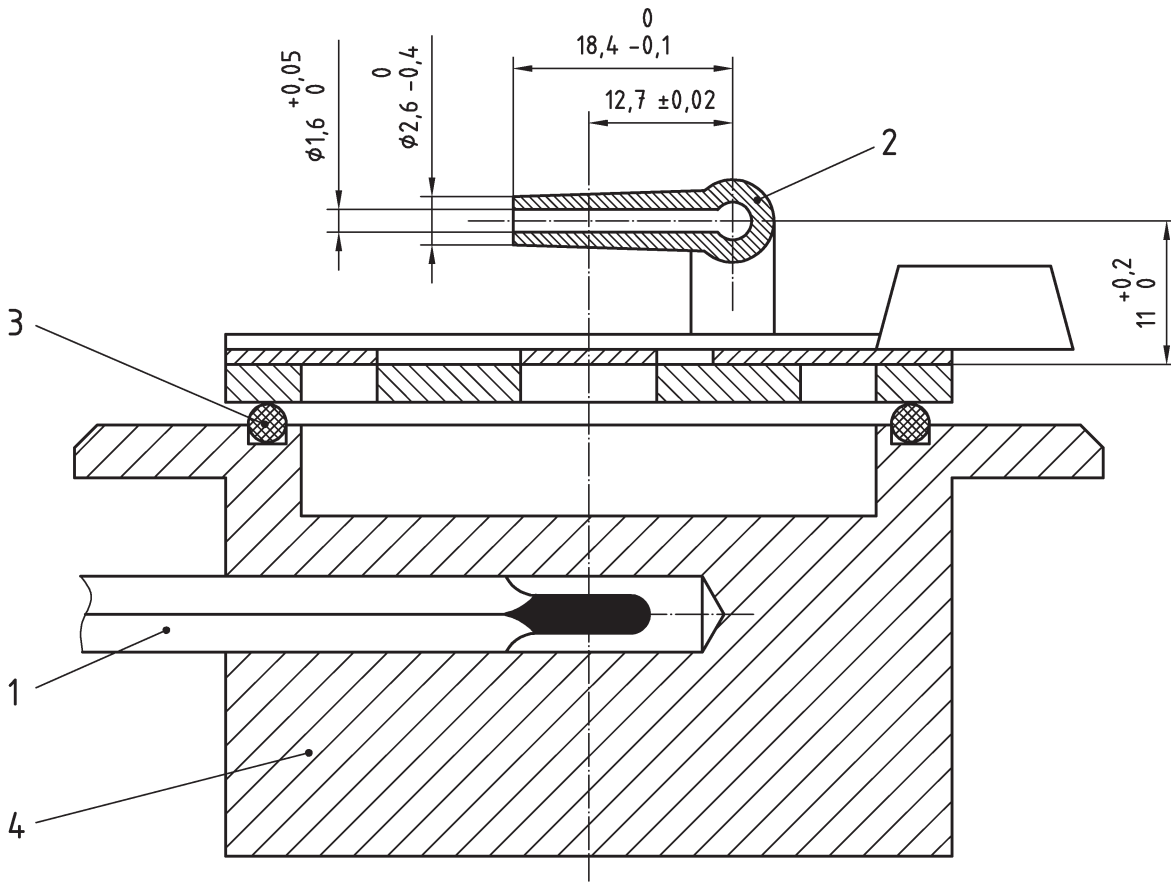
#### Key

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

**Figure A.1 — Cover assembly**

**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 well in the side to accommodate a temperature measuring device ([A.1.4](#)). The diameter of the well can be reduced to accommodate an electronic temperature measuring device. The sensing element of the temperature measuring device shall be located centrally under the test cup as shown in [Figure A.2](#).

Dimensions in millimetres



**Key**

- 1 temperature measuring device
- 2 nozzle of test flame
- 3 O-ring seal
- 4 test cup block

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

**A.1.1.3 Cover**, fitted with an opening slide and a device which permits insertion of a test flame of diameter  $(4,0 \pm 0,5)$  mm into the test cup when the slide is open. When inserted, the nozzle of the test flame (see [A.1.2.1](#)) shall intersect the plane of the underside of the cover within  $\pm 0,1$  mm. The cover shall be provided with an orifice extending into the test cup for insertion of the test portion and 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 seal or gasket, which provides a tight seal when the cover is shut, should be made of a heat-resistant material capable of withstanding the test temperatures and the materials being tested.

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

Dimensions in millimetres

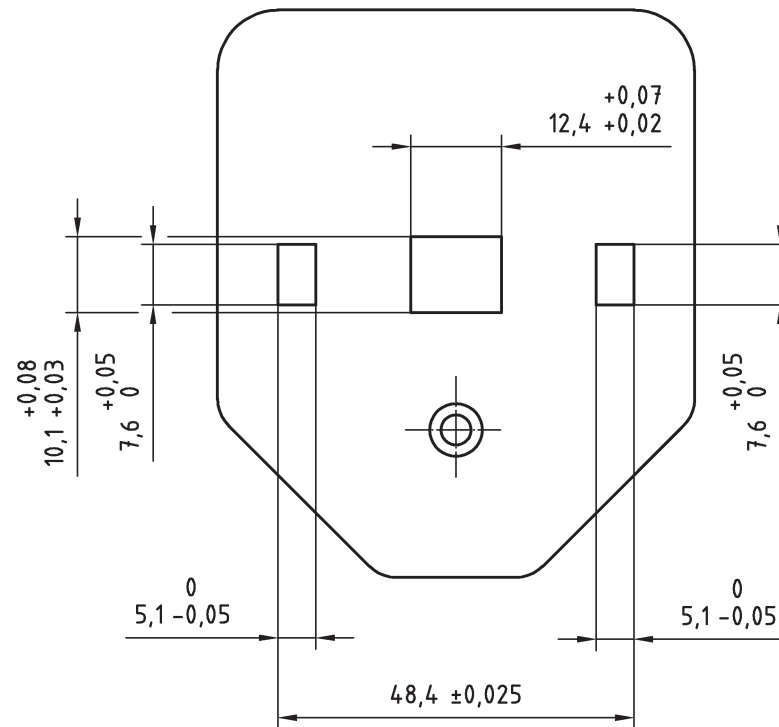


Figure A.3 — Cover

Dimensions in millimetres

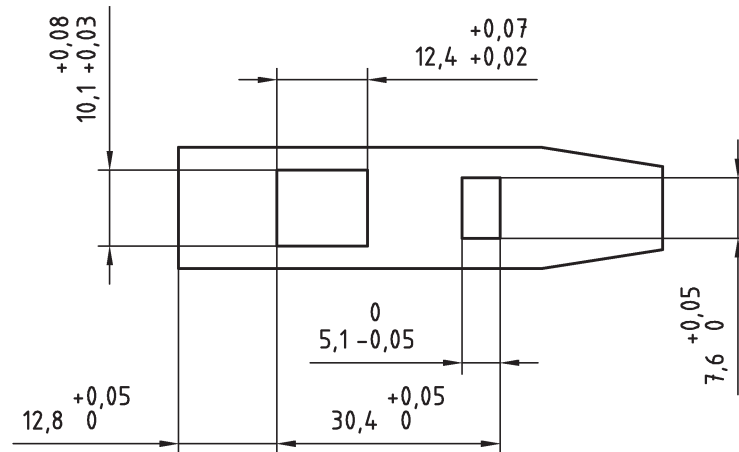


Figure A.4 — Slide

**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  $\pm 0,5$  °C for test temperatures up to and including 100 °C, and within  $\pm 2,0$  °C for test temperatures above 100 °C during the test.

Dimensions in millimetres

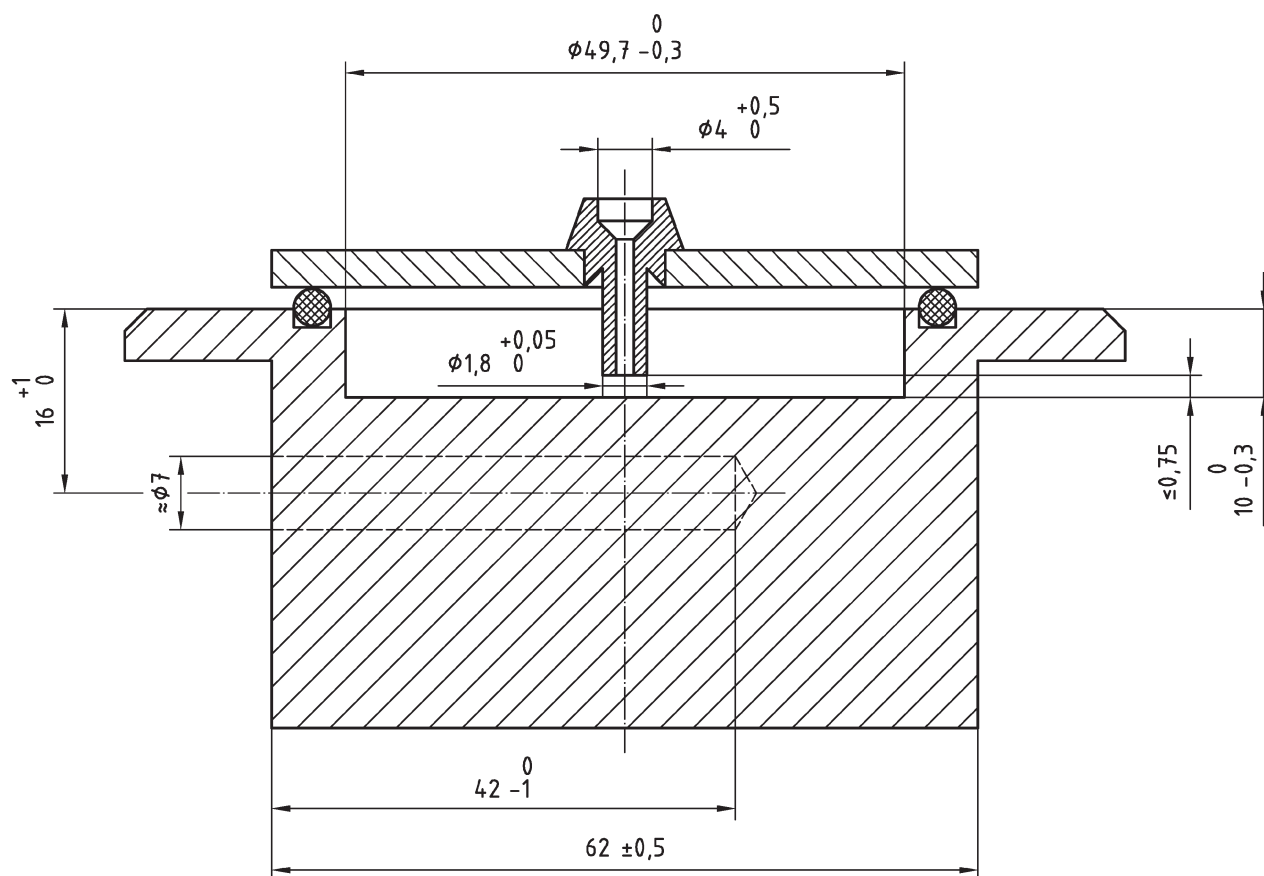


Figure A.5 — Section of test cup block through filler orifice

## A.1.2 Test flame and pilot flame

**A.1.2.1** The flames shall be fuelled by any suitable flammable gas (5.3). A gauge ring 4 mm in diameter shall be engraved on the cover near to the test flame.

**A.1.2.2** An electric ignitor together with a suitable dipping mechanism and electric ignitor screen (A.1.7) is also permitted when testing petroleum, solvents and related products. The electric ignitor shall be of the electric resistance (hot wire) type and shall position the heated section horizontally and intersect the underside of the cover.

Other ignitors can be used but the precision might not apply. Follow the manufacturer's instructions for ensuring the correct operation of the ignitor. In the event of a dispute the gas ignition source is the referee.

## A.1.3 Timing device

Capable of emitting an audible signal after  $(60 \pm 2)$  s and  $(120 \pm 4)$  s to indicate when the ignition source is to be dipped into the cup.

## A.1.4 Temperature measuring device

Annex E specifies the requirements for suitable liquid in glass and digital temperature measuring devices.

#### **A.1.5 Test cup cooler (optional)**

An electronic Peltier or other suitable cooling device.

#### **A.1.6 Flash detector (optional)**

Low mass thermocouple device for the detection of the flash point flame. The flash detector can be used for testing all products and shall be used when testing FAME.

#### **A.1.7 Electric ignitor screen**

A metal screen to optically screen the ignitor from the operator. This is only required when an electric ignitor is used.

## Annex B (informative)

### Verification of apparatus

#### B.1 General

This Annex describes a procedure for the verification of the apparatus using either a secondary working standard (SWS) or a certified reference material (CRM), and includes a procedure for producing a secondary working standard (SWS).

The performance of the apparatus should be verified on a regular basis using either a CRM produced in accordance with ISO Guide 34<sup>[13]</sup> and ISO Guide 35,<sup>[14]</sup> or an in-house reference material/SWS prepared in accordance with one of the procedures given in [B.2.2](#). The performance of the apparatus should be assessed in accordance with the guidance given in ISO Guide 33<sup>[15]</sup> and ISO 4259.

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

#### B.2 Verification standards

**B.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.

**B.2.2 Secondary working standard (SWS)**, comprising of 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, and at a temperature not exceeding 10 °C.

#### B.3 Procedure

**B.3.1** Choose a CRM or SWS which falls within the range of flash points to be determined with the apparatus. See [Table B.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.

**B.3.2** For new apparatus, and at least once a year for working apparatus, conduct a verification check using a CRM specific for this International Standard ([B.2.1](#)) tested in accordance with [Clause 10](#).

**B.3.3** For intermediate verification, conduct a verification check using an SWS specific for this International Standard ([B.2.2](#)) tested in accordance with [Clause 10](#).



**B.3.4** Correct the result for barometric pressure in accordance with [Clause 11](#). Record the corrected result, to the nearest 0,1 °C, in a permanent record.

**Table B.1 — Expected flash point values and tolerances (acceptance limits for a single result)**

Substance	Flash point °C	Tolerances (see <a href="#">7.4.2</a> ) R/√2 °C	Source of expected flash point values
2-Butanol	20,7	±2,3	a
p-Xylene (1,4-dimethylbenzene)	26,1	±2,4	a
Nonane	32	±2,5	c
n-Butanol	36,5	±2,6	a
Decane	49,7	±2,9	b
Undecane	65,9	±3,2	b
Dodecane	81	±3,4	c
Tetradecane	109	±3,9	c
Diethylene Glycol	142,2	±4,5	a
Hexadecane	134	±4,4	c

NOTE None of the flash point values are certified.

a Mean values from an interlaboratory test programme.[\[10\]](#)

b Mean values from an interlaboratory test programme.[\[16\]](#)

c Historical figures.

## B.4 Evaluation of the test result

### B.4.1 General

Compare the corrected test result(s) with the certified value of the CRM or the assigned value of the SWS.

In the formula given in [B.4.1.1](#) and [B.4.1.2](#) it is assumed that the reproducibility has been estimated in accordance with ISO 4259 and that the certified value of the CRM, or the assigned value of the SWS, has been obtained by the procedures as set out in ISO Guide 35.

#### B.4.1.1 Single test

For a single test made on a CRM or SWS, the difference between a single result and the certified value of the CRM or the assigned value of the SWS should be within the following tolerance:

$$|x - \mu| \leq \frac{R_1}{\sqrt{2}} \quad (\text{B.1})$$

where

$x$  is the result of the test;

$\mu$  is the certified value of the CRM or the assigned value of the SWS;

$R$  is the reproducibility of the test method.

#### B.4.1.2 Multiple tests

If a number of replicate tests,  $n$ , are made on a CRM or SWS, the difference between the mean of the  $n$  results and the certified value of the CRM or the assigned value of the SWS, should be within the following tolerance:

$$|\bar{x} - \mu| \leq \frac{R_1}{\sqrt{2}} \quad (\text{B.2})$$

where

- $\bar{x}$  is the mean of the test results;
- $\mu$  is the certified value of the CRM or the assigned value of the SWS;
- $R_1 = \sqrt{[R^2 - r^2(1 - 1/n)]}$ ;
- $R$  is the reproducibility of the test method;
- $r$  is the repeatability of the test method;
- $n$  is the number of replicate tests carried out on the CRM or SWS.

**B.4.2** If the test result conforms with the tolerance requirements, record this fact.

**B.4.3** If the result does not conform to the tolerance requirements and an SWS has been used for the verification check, repeat using a CRM. If the result conforms to the tolerance requirements, record this fact and dispose of the SWS.

**B.4.4** If the test result still does not conform to the tolerance requirements, examine the apparatus and check that it conforms with the apparatus specification requirements. If there is no obvious nonconformity, conduct a further verification check using a different CRM. If the result conforms to the tolerance requirements, record this fact. If it is still not within the required tolerances, send the apparatus to the manufacturer for a detailed examination.

## **Annex C** (informative)

### **The use of a cup insert**

#### **C.1 General**

Certain samples are very difficult and time-consuming to remove completely from the test cup at the end of the test. The use of a disposable thin metal cup, inserted into the test cup, removes this problem.

#### **C.2 Cup insert material**

Any metal foil, approximately 0,05 mm thick, can be used provided that it can be shaped to closely fit the test cup.

#### **C.3 Procedure**

Form the foil by pressing it into the test cup using a solid metal block or a proprietary tool. The cup insert formed shall fit the test cup closely.

#### **C.4 Verification**

Verify the cup insert material and cup forming procedure using a CRM or SWS (see [7.4](#)).

## Annex D (normative)

### Sub-ambient testing for manual apparatus without integral test cup cooling facilities

#### D.1 Apparatus preparation

**D.1.1** Apparatus using a liquid in glass thermometer: switch off the power to the test cup heater.

**D.1.2** Manual apparatus using an electronic thermometer: follow the apparatus manufacturer's instructions for setting the required sub-ambient test temperature.

**D.1.3** Follow the manufacturer's instructions to fill a refrigerant-charged cooling block with a suitable material or for alternative methods of cooling.

**D.1.4** When the target or specification temperature is not less than 5 °C, crushed ice and water can be used as a charging (cooling) fluid.

NOTE If the temperature is below 5 °C, a suitable charging (cooling) fluid is solid carbon dioxide (dry ice) and acetone.

#### D.2 Procedure

**D.2.1** Raise the cover, and position the base of the block in the sample cup, being careful not to damage the test cup.

**D.2.2** When the thermometer reads approximately 10 °C below the target temperature, remove the cooling block and quickly dry the test cup and underside of the cover with a paper tissue to remove any moisture.

**D.2.3** Immediately close the cover and secure.

**WARNING — If a liquid in glass thermometer is being used do not cool the sample block below the freezing point of the liquid in the thermometer.**

**D.2.4** Prepare to introduce the test portion into the test cup using the syringe, both of which have been precooled to a temperature at least 10 °C below the required test temperature.

**D.2.5** Adjust the test flame and allow the test cup temperature to rise under ambient conditions until the required test temperature is reached. Immediately apply the test flame. Watch closely for a flash at the cover openings.

**D.2.6** To determine the flash point of any materials, carry out repeat tests with a new test portion each time, in accordance with the steps given in [10.3.2](#) to [10.3.14](#).

## Annex E (normative)

### Temperature measuring device specifications

#### E.1 Digital

**E.1.1** Temperature range,  $-30\text{ °C}$  to  $300\text{ °C}$ .

**E.1.2** Display resolution, better than  $0,5\text{ °C}$ .

**E.1.3** Accuracy (after calibration) to meet the requirements of [A.1.1.4](#).

**E.1.4** 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 1 Silicone heat-sink compounds have been found to be suitable.

NOTE 2 Guidelines for digital temperature measuring devices are given in IEC 60751[17] and ASTM E1137.[18]

#### E.2 Liquid-in-glass thermometers

**Table E.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 mark	°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	max. °C	0,5	0,5	2,0
Expansion chamber		Required	Required	Required
Total length	mm	195 to 200	195 to 200	195 to 200
Stem outer diameter	mm	6 to 7	6 to 7	6 to 7
Bulb length	mm	10 to 14	10 to 14	10 to 14
Bulb outer diameter	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

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