INTERNATIONAL STANDARD

ISO 3679

Fifth edition
2022-11

Determination of flash point – Method for flash no-flash and flash point by small scale closed cup tester

Détermination du point d'éclair — Méthode de l'éclair de type passe/ne passe pas et méthode du point d'éclair en vase clos à petite échelle

Reference number
ISO 3679:2022(E)

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Published in Switzerland

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Foreword

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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

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For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT), see www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 28, *Petroleum and related products, fuels and lubricants from natural or synthetic sources*, in collaboration with the European Committee for Standardization (CEN) Technical Committee CEN/TC 19, *Gaseous and liquid fuels, lubricants and related products of petroleum, synthetic and biological origin,* in accordance with the Agreement on technical cooperation between ISO and CEN (Vienna Agreement).

This fifth edition cancels and replaces the fourth edition (ISO 3679:2015), which has been technically revised.

The main changes are as follows:

— introduction, title and scope have been revised to present a more generic method description;

— terms and definitions in Clause 3 have been added;

— verification clause has been revised;

— new procedure C has been added;

— Clause 13 wording has been revised and precision for procedure C has been included;

— the apparatus description in Annex A has been revised;

— Annex B has been revised and changed to normative;

— the text has been editorially revised in line with the ISO/IEC Directives Part 2, 2021.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Introduction

This document includes three procedures (A, B and C) covering determinations of flash no-flash and flash point. Rapid equilibrium procedures A and B enable the determination of the flash no-flash and flash point, respectively. Non-equilibrium procedure C uses automated test cup temperature control for flash point determination.

ISO 1516 and ISO 1523 are also closed cup equilibrium test methods that can be considered when selecting a method.

The apparatus specified in this document enables a similar test result to be determined using more rapid procedures, A or B, and a smaller test portion (2 ml or 4 ml), than those required in ISO 1516 or ISO 1523. In addition, the apparatus in this document can be made portable so that it is suitable for on-site testing, as well as its regular use in laboratories. Collaborative work[16] has shown that results obtained by these methods are comparable. Procedure C is based on test methods IP 534[18] and ASTM D7236[14].

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[17].

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

Flash point is 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 indicates the presence of highly volatile material(s) in a relatively non-volatile or non-flammable material. Flash point testing is often used as 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. That is, 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 also gives useful advice in carrying out flash point tests and interpreting results.

Determination of flash point – Method for flash no-flash and flash point by small scale closed cup tester

WARNING — The use of this document can involve hazardous materials, operations and equipment. This document does not purport to address all of the safety problems associated with its use. It is the responsibility of users of this document to take appropriate measures to ensure the safety and health of personnel prior to the application of the standard, and to determine the applicability of any other restrictions for this purpose.

1 Scope

This document describes three procedures (A, B and C) covering determinations of flash no-flash and flash point.

Rapid equilibrium procedures A and B are applicable to flash no-flash and flash point tests of paints, including water-borne paints, varnishes, binders for paints and varnishes, adhesives, solvents, petroleum products including aviation turbine, diesel and kerosene fuels, fatty acid methyl esters and related products over the temperature range –30 °C to 300 °C. The rapid equilibrium 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 document is also suitable to determine the flash point of fatty acid methyl esters (FAME). The validity of the precision is given in Table 2.

Non-equilibrium procedure C is applicable to petroleum products including aviation turbine, diesel and kerosine fuels, and related petroleum products, over the temperature range –20 °C to 300 °C. The non-equilibrium procedure is automated to determine the flash point. Precision has been determined over the range 40 °C to 135 °C.

For specifications and regulations, procedures A or B are routinely used (see 10.1.1).

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO Guide 35, *Reference materials — Guidance for characterization and assessment of homogeneity and stability*

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

ISO 3170, *Petroleum liquids — Manual sampling*

ISO 3171, *Petroleum liquids — Automatic pipeline sampling*

ISO 15528, *Paints, varnishes and raw materials for paints and varnishes — Sampling*

ISO 17034, *General requirements for the competence of reference material producers*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

ISO and IEC maintain terminology databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at https://www.electropedia.org/

3.1
equilibrium
condition in flash point test methods where the vapour above the test portion and the test portion are at the same temperature at the time the ignition source is applied

Note 1 to entry: This condition cannot be fully achieved in practice, since the temperature can be uneven throughout the test portion, and the test cover and shutter on the apparatus can be cooler or warmer.

EXAMPLE Procedures A and B in this document, ISO 1516 and ISO 1523.

3.2
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 and ASTM D6751.

3.3
flash no-flash
application of an ignition source at the specified temperature of the test portion, as measured in the prescribed manner, adjusted to account for variations in atmospheric pressure from 101,3 kPa, to determine whether the vapour of the test portion ignites and the flame propagates across the surface of the liquid under the specified conditions of test

3.4
flash point
lowest temperature of the test portion, adjusted to account for variations in atmospheric pressure from 101,3 kPa, at which application of an ignition source causes the vapour of the test portion to ignite and the flame to propagate across the surface of the liquid under the specified conditions of test

3.5
non-equilibrium
condition in flash point test methods where the vapour above the test portion and the test portion are not in temperature equilibrium at the time that the ignition source is applied

Note 1 to entry: This condition is primarily caused by the heating of the test portion at the constant prescribed rate with the vapour temperature lagging behind the test portion temperature.

EXAMPLE Procedure C in this document, ISO 2719 and ISO 13736.

4 Principle

4.1 Rapid equilibrium procedures A and B

A 2 ml or 4 ml test portion is introduced into the test cup that is set and maintained at the required test temperature. After a specific time, when the vapours and test portion are deemed to be in temperature equilibrium, 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. The temperature is adjusted to account for variation in atmospheric pressure from 101,3 kPa, using a formula.

4.2 Non-equilibrium procedure C

A 2 ml test portion is introduced into the test cup that is set and maintained at the required start temperature. The test cup is then heated at a ramp rate of 1,5 °C/min to 2,5 °C/ min and the ignition source is applied at 1 °C temperature intervals until a flash point is detected. The detected flash point temperature is adjusted to account for variation in atmospheric pressure from 101,3 kPa, using a formula.

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 depends 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, for flash point, 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 may be used.

6 Apparatus

6.1 Flash point apparatus, as specified in Annex A.

6.2 Barometer, absolute pressure reading, accurate to ±0,5 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 (optional), for warming the sample, if required.

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

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.

The bath and freezer 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 ml ± 0,05 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 can be used twice.

6.7.2 **Syringe**, capable of delivering 4,00 ml ± 0,10 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 relevant procedure and the expected flash point temperature. Follow the manufacturer's instructions for the correct set-up, verification (see 7.4) and operation of the apparatus, especially the operation and setting of the ignition source.

7.1.2 Procedure C is automated and requires automated temperature ramp control; sub-ambient testing requires integrated cooling (see A.1.5).

7.1.3 The use of a cup insert (6.6) for potentially adherent materials is described in Annex C.

7.1.4 When testing FAME (procedures A and B), use a 2 ml ± 0,05 ml test portion and a 60 s ± 2 s test time, combined with an electronic thermal flash detector (see A.1.6).

7.1.5 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 specified in Annex A on a level and steady surface in a draught-free position.

A draught shield (6.5) should be used when protection from draughts is not available.

WARNING — 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, can 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 Check the temperature measuring devices and barometer at least once a year to ensure that they are in accordance with A.1.4, Annex E and 6.2, respectively.

7.4.2 Ensure the correct operation of ignition sources, in accordance with the manufacturer's instructions and this test method.

7.4.3 Verify the accuracy of the apparatus at least once a year by testing a CRM (see 5.2 and Annex B). It is recommended that more frequent verification checks are made using a reference material (see 5.2 and Annex B).

7.4.4 The result of a single test obtained for a reference material shall be equal to or less than $R/\sqrt{2}$ from the certified value of the CRM or from the accepted reference value (ARV) of the SWS, where R is the reproducibility of the test procedure.

NOTE These reference materials (RM) and in-house quality control samples can also be used to monitor stability and establish statistical control limits, according to ISO 4259-4 or equivalent standard, if required.

7.4.5 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.

7.4.6 If the instrument fails the verification test, it is recommended that the operator follow the manufacturer's 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 ignition source operates correctly;

e) the flash detector (A.1.6) operates correctly (if fitted);

f) the temperature measuring device reads correctly.

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 must be stored prior to testing, the container shall be filled 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 can 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 Procedures

10.1 General

10.1.1 For specifications and regulations use procedures A or B, unless procedure C has been specified.

10.1.2 Follow the manufacturer's instructions for setting the test temperature.

10.1.3 When testing fatty acid methyl esters (FAME), a flash detector (A.1.6) shall be used.

10.1.4 Use a new test portion of the sample for each test. 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.

10.1.5 Do not confuse the true flash point with the bluish halo that sometimes surrounds the test flame at applications preceding the one 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.

10.1.6 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.

10.1.7 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.

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

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

10.2.4 When the test cup is at the test temperature, fill the appropriate syringe (6.7) with the sample to be tested. Close the sample container immediately after withdrawal to minimize any loss of volatile components. 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. Do not apply the ignition source to the test portion more than once.

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.

10.3 Procedure B — Flash point determination

10.3.1 This procedure repeats the flash no-flash test described in procedure A (10.2.4 to 10.2.7) a number of times. Each flash no-flash test uses a fresh test portion and a different test temperature.

Testing at different temperatures allows the determination of two test temperatures that are 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.

If the expected flash point is unknown, then procedure C can be used to obtain an initial test temperature for materials compliant with the scope of procedure C.

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 or 6.7.2) with the sample to be tested.

Close the sample container immediately after withdrawal to minimize any loss of volatile components.

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. Do not apply the ignition source to the test portion more than once.

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. Calculate the corrected flash point using Formula (2).

NOTE Results obtained directly from 10.3.12 without following the more accurate procedure in 10.3.13 are permitted but the precision can be affected.

10.4 Procedure C — Flash point determination

10.4.1 Set the test cup temperature to 15,0 °C ± 5,0 °C below the expected flash point temperature.

10.4.2 When the test cup is at the required temperature, fill the syringe (6.7.1) with 2 ml of the sample to be tested. Close the sample container immediately after withdrawal to minimize any loss of volatile components. 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.

NOTE A 2 ml sample is used for all test temperatures in procedure C.

10.4.3 Light the pilot light and adjust the test flame (if used) to conform to the 4 mm gauge.

10.4.4 Heat the test cup at a rate such that the temperature, as indicated by the thermometer, increases by 1,5 °C / min to 2,5 °C / min. Dip the test flame into the test cup at 1 °C intervals for temperatures up to and including 100 °C and thereafter every 2 °C for temperatures over 100 °C.

10.4.5 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, repeat the test with a lower expected flash point using a fresh test portion.

10.4.6 When the temperature at which the flash is detected is less than 10 °C or greater than 30 °C from the first application of the of the test flame, the result is not valid. Repeat the test using a fresh test portion adjusting the temperature of the first application of the test flame until a valid determination is made.

10.4.7 Record the temperature read on the temperature sensor when the application of the test flame causes a flash to be detected. Calculate the corrected flash point using Formula (2).

11 Calculation

11.1 Conversion of barometric pressure reading

If the absolute barometric pressure reading, taken in accordance with 10.1.6, is in a unit other than kilopascals, convert to kilopascals using one of the following conversions:

— Reading in hPa × 0,1 = kPa;

— Reading in mbar × 0,1 = kPa;

— Reading in mmHg × 0,133 = kPa.

11.2 Correction of the detected flash point

This adjustment, to account for variations in atmospheric pressure from 101,3 kPa, is only required for procedures B and C. Calculate the corrected flash point, t_c, using Formula (2):

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

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 Formula (2) has been proven for barometric pressures down to 82,0 kPa[18] and is strictly correct only up to 104,7 kPa.

NOTE 2 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

For procedure A, record the flash no-flash temperature, corrected to standard atmospheric pressure, rounded to the nearest 0,5 °C.

For procedures B and C, record 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 inter-laboratory 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.

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[1)]. The precision for paints and related products is not known to have been developed in accordance with ISO 4259[1)].

13.2 Repeatability, *r*

The difference between two independent results obtained using this method for test material considered to be the same in the same laboratory, by the same operator using the same equipment within short intervals of time, in the normal and correct operation of the method that is expected to be exceeded with a probability of 5 % due to random variation, is given in Tables 2 and 3.

13.3 Reproducibility, *R*

The difference between two independent results obtained using this method for test material considered to be the same in different laboratories, where different laboratory means a different operator, different equipment, different geographic location, and under different supervisory control, in the normal and correct operation of the method that is expected to be exceeded with a probability of 5 % due to random variation, is given in Tables 2 and 3.

1) Withdrawn.

Table 2 — Precision values

Procedure	Range	Temperature range °C	Ignitor[a]	Repeatability °C	Reproducibility °C
B	Petroleum and related products[19]	20 to 220	G/E	0,015 20 (X + 110)[b]	0,025 61 (X + 110)[b]
—	—	—	—	—	—
B	Paints and related products with kinematic viscosity[20]	—	—	—	—
	≤5,8 mm^2/s at 37,8 °C	4 to 82	G	1,7	3,3
	>5,8 mm^2/s at 37,8 °C	17 to 52	G	3,3	5,0
—	—	—	—	—	—
B	Fatty acid methyl esters (FAME)[11]	85 to 175	G	1,9	15,0
—	—	—	—	—	—
C	Petroleum and related petroleum products[18]	40 to 135	G	0,012 85 (X + 40)[b]	0,024 82 (X + 40)[b]

[a] E refers to electric ignition and G refers to gas ignition.

[b] Where X is the average of the results being compared.

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

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

13.4 Relative bias between the procedures C and B

The relative bias was examined according to a t-test at a 99 % confidence level as there were insufficient samples to be able to perform a D6708[12] analysis.

The t-test outcome was that procedure C gave results 0,8 °C higher than procedure B on average.

14 Test report

The test report shall contain at least the following information:

a) a reference to this document (i.e. ISO 3679:2022);

b) the type and complete identification of the product tested;

c) the result of the test (see Clause 12);

d) ignitor type (gas or electric);

e) laboratory absolute barometric pressure;

f) the procedure used, i.e. procedure A, B or C;

g) any deviation, by agreement or otherwise, from the procedures specified;

h) the date of the test.

Annex A
(normative)

Flash point apparatus

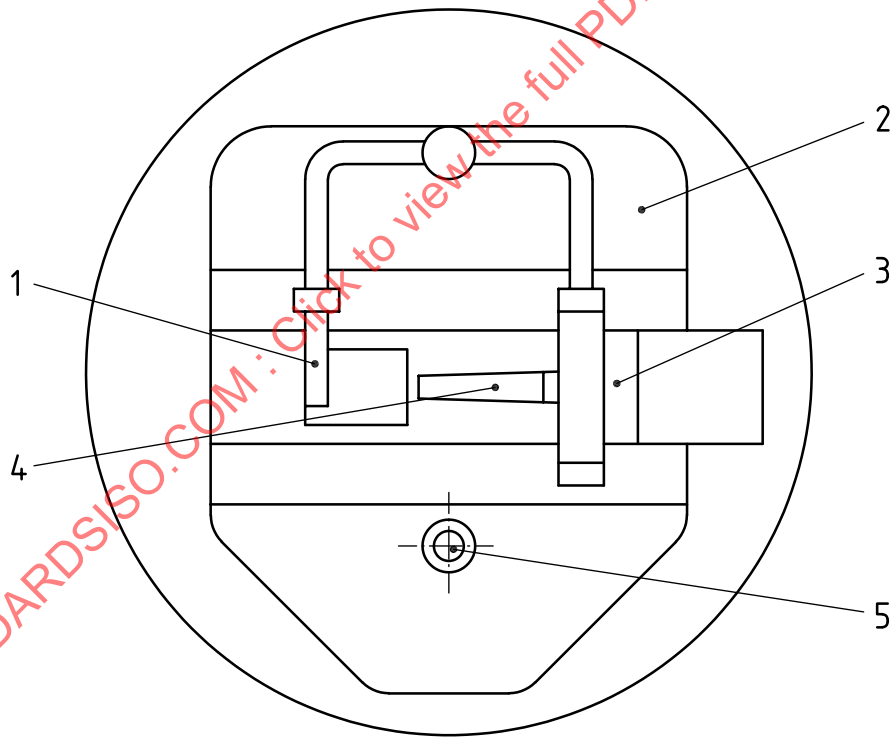
A.1 Apparatus

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 Figures A.1 to A.5.

Some versions of the apparatus can automate, temperature control down to sub-ambient temperatures, timing and dipping, include hot wire ignition, automatically detect a valid flash point and measure barometric pressure.



Key

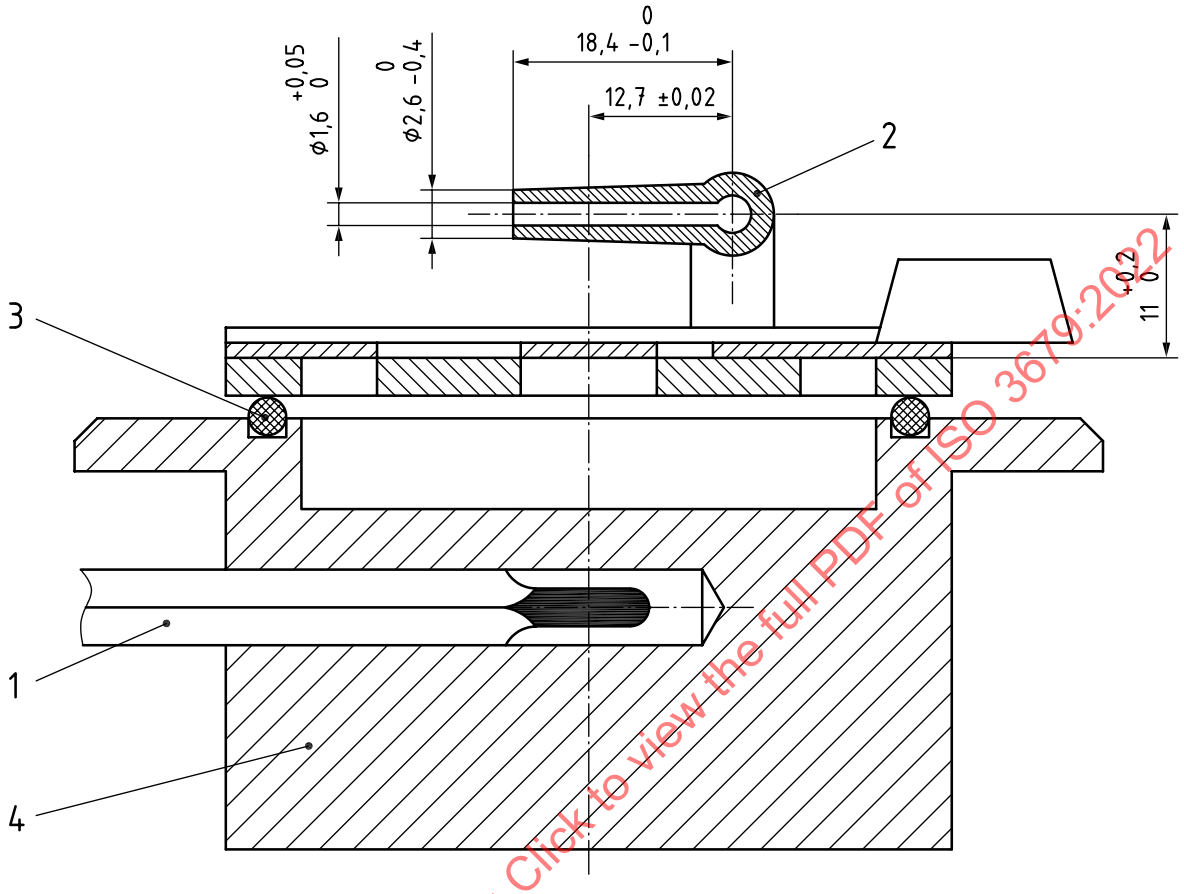
1 nozzle of pilot flame
2 cover
3 slide
4 nozzle of test flame
5 filler orifice

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 ± 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 ±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 ignitor is automated on some instruments.

Dimensions in millimetres

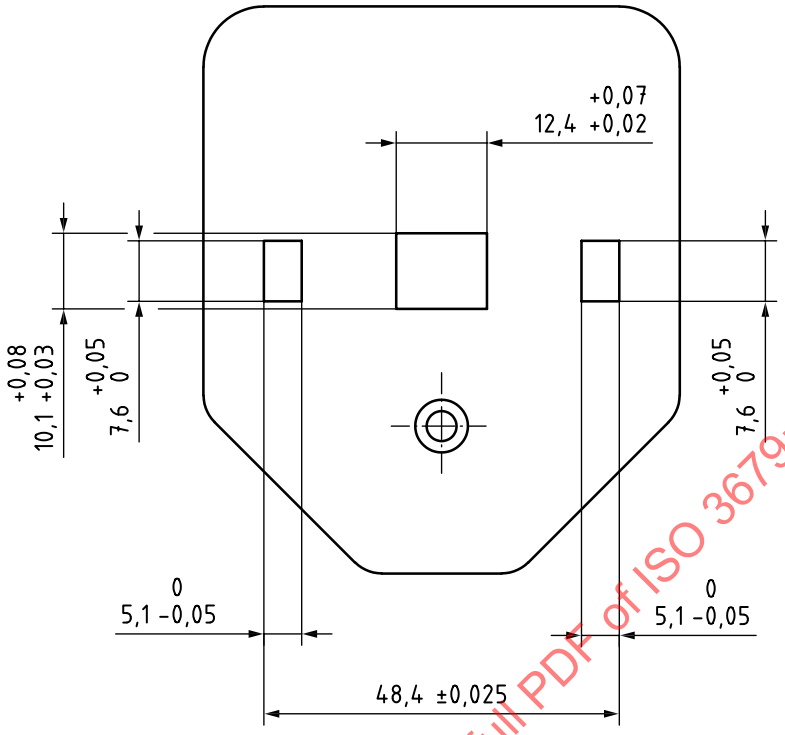


Figure A.3 — Cover

Dimensions in millimetres

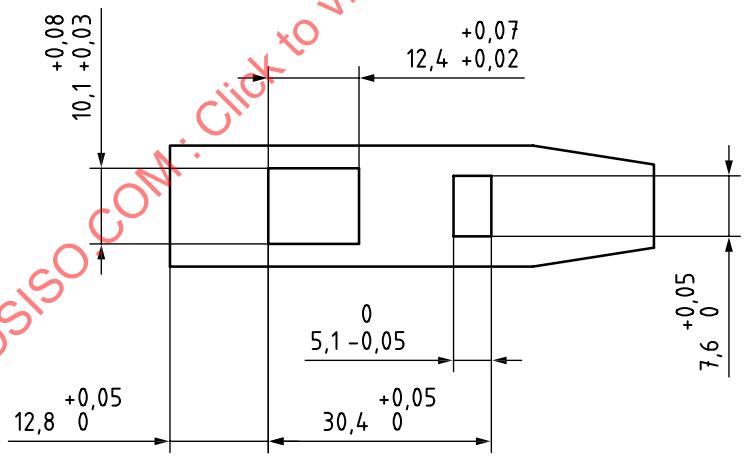


Figure A.4 — Slide

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 are as follows:

— For procedures A and B: within ±0,5 °C for test temperatures up to and including 100 °C, and within ±2,0 °C for test temperatures above 100 °C during the test.

— For procedure C: within the ramp rate of 1,5 °C/ min to 2,5 °C/min with an accuracy of ± 0,5 °C.

Dimensions in millimetres

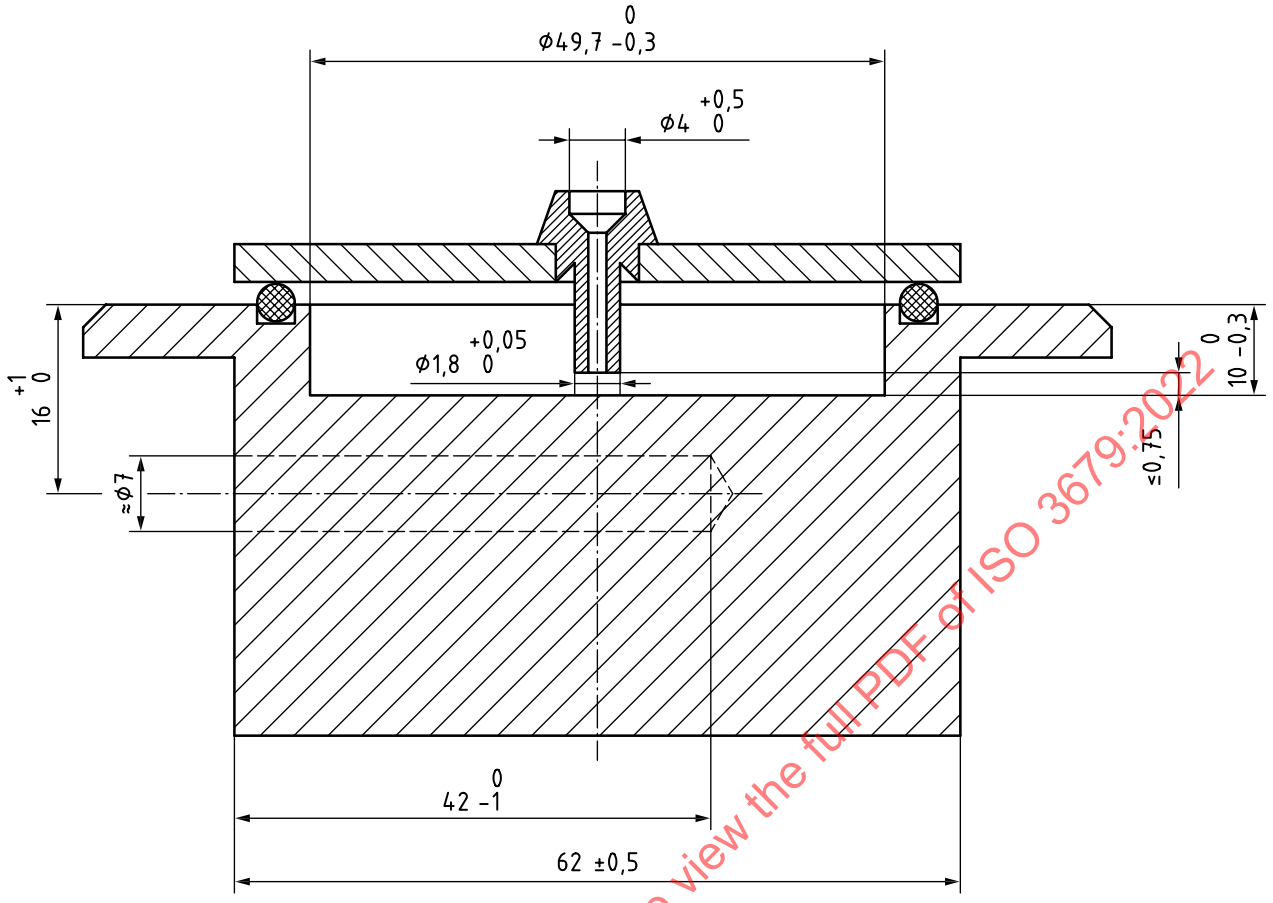


Figure A.5 — Section of test cup block through filler orifice

A.1.2 Ignition source

A.1.2.1 Test and pilot 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.

In cases of dispute, unless explicitly agreed otherwise, the determination of the flash point, using a flame ignition source, shall be considered the referee test.

A.1.3 Timing device

The timing device shall be capable of emitting an audible signal after (60 ± 2) s and (120 ± 4) s, for procedures A and B, and every 1 °C during the test for procedure C, to indicate when the ignition source shall 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)

The test cup cooler is an electronic Peltier or another suitable cooling device.

A.1.6 Flash detector (optional)

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

A.1.7 Electric ignitor screen

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

Annex B
(normative)

Verification of apparatus

B.1 General

This annex describes a procedure for verifying the correct performance of a flash point apparatus, using reference materials (RM).

The accuracy of the apparatus (manual or automated) shall be verified at least every 12 months using a certified reference material (CRM) produced in accordance with the principles of ISO 17034 and ISO Guide 35 or equivalent standards. Further accuracy verifications on a more frequent basis, between the mandated yearly CRM verifications, can use either a CRM, or a secondary working standard (SWS) in accordance with B.2.3. Further guidance is given in ISO Guide 33 and ISO/IEC 17043.

Reference materials associated with proficiency testing programmes [B.2.3 a)] are the preferred materials for SWS verifications and should be used when available.

The number of data sets to characterize the reference material shall be large enough to provide an adequately small uncertainty for the certified or accepted reference value.

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

RM should be stored in containers out of direct sunlight and at such a temperature as to retain the integrity of the flash point of the sample.

B.2 Reference materials

B.2.1 An RM for flash point is a CRM or SWS, that is sufficiently homogeneous and stable with respect to flash point measurements, which has been established to be fit for its intended use in a measurement process.

B.2.2 A CRM for flash point shall be a stable single hydrocarbon or other stable substance with a flash point determined using a method specific inter-laboratory study (ILS) and be accompanied by an RM certificate that provides the certified value of the flash point, its associated uncertainty, and a statement of traceability.

B.2.3 A SWS for flash point shall be a stable petroleum product or a single hydrocarbon or other stable substance with a flash point accepted reference value (ARV) for a specific test method. The ARV is obtained from either of the following ways:

a) a proficiency testing programme where the instrument being validated participated in the testing, or by obtaining sample from a proficiency scheme after the ARV had been established according to ISO 4259-3, ISO/IEC 17043 or equivalent statistical requirements.

b) Table B.1 shows the ARVs for a number of pure materials (>99 % purity). The ARVs were derived from official ILS. The ARV can be used for other batches of the same material provided that the purity requirement is maintained. These ARV are uncertified and can be affected by contaminants.

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 the ARV of selected chemicals.

NOTE Annex F presents an aid for the selection of materials for in-house quality standards.

A flash point value is supplied with each CRM or SWS. It is recommended that two CRMs or SWSs be used in order to cover the flash point range of products usually tested. 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 (B.2.2) tested in accordance with Clause 10.

B.3.3 For intermediate verification, conduct a verification check using an SWS (B.2.3) tested in accordance with Clause 10.

B.3.4 Correct the result for barometric pressure in accordance with Clause 11. Note the corrected result, to the nearest 0,1 °C, in a permanent record.

NOTE Verification test results are not rounded to the nearest 0,5 °C, as CRM and SWS are recorded to the nearest 0,1 °C.

Table B.1 — Flash point ARV and tolerances (acceptance limits for a single result)

Procedure	Substance	Flash point °C	Tolerances (see 7.4.2) $R/\sqrt{2}$ °C	Source of expected flash point values
B	2-Butanol	20,7	±2,3	a
B	p-Xylene (1,4-dimethylbenzene)	26,1	±2,4	a
B	n-Butanol	36,5	±2,6	a
B	Decane	49,7	±2,9	b
B	Undecane	65,9	±3,2	b
B	Diethylene Glycol	142,2	±4,5	a
-	-	-	-	-
C	Hexadecane	134,8	±3,0	c

NOTE None of the flash point values are certified.

a Mean values from an interlaboratory test programme[19].

b Mean values from an interlaboratory test programme[21].

c Mean value from an interlaboratory test programme[20]

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 ARV of the SWS.

In Formulae (B.1), (B2) and (B.3) [see also ISO 4259-2:2017, 4.2.3] it is assumed that:

— reproducibility has been estimated in accordance with ISO 4259-1 or other equivalent statistical techniques;

— the certified value of the CRM, or the ARV of the SWS, has been obtained according to the requirements of Clauses B.1 and B.2, and that its uncertainty is small in comparison with the reproducibility of the test method, R.

B.4.2 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 ARV of the SWS should be within the following tolerance:

$$|x - \mu| \leq R/\sqrt{2} \tag{B.1}$$

where

x is the result of the test;

μ is the certified value of the CRM or the assigned value of the SWS;

R is the reproducibility of the test method.

B.4.3 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 ARV of the SWS, should be within the following tolerance:

$$|\bar{x} - \mu| \leq R/\sqrt{2} \tag{B.2}$$

where

$\bar{x}$ is the mean of the test results;

μ is the certified value of the CRM or the assigned value of the SWS;

R_1 is equal to the expression given by Formula (B.3).

$$\sqrt{R^2 - r^2[1-(1/n)]} \tag{B.3}$$

where

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.4.4 Verification validated

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

B.4.5 Verification not validated

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.

If further verification tests do not conform to the tolerance requirements, check the validity of the RM and that the requirements of Clause 7 are being met. 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 consult the manufacturer of the apparatus.