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Petroleum and related products — Determination of flash and fire points — Cleveland open cup method

Pétrole et produits connexes — Détermination des points d'éclair et de feu — Méthode Cleveland à vase ouvert

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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).

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For an explanation on 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 the following URL: 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*.

This third edition cancels and replaces the second edition (ISO 2592:2000), which has been technically revised and aligned with ASTM D92.

The main technical changes compared to the previous edition are as follows:

a) Annex D on an alternative procedure for handling skin forming products has been added;

b) the temperature measuring device requirements in Annex B has been revised;

c) the flash point reproducibility has been changed from 17 °C to 18 °C, to align with ASTM D92 on the basis of recent precision data;

d) a procedure to determine an approximate flash point of a sample with an unknown expected flash point has been included, to align with ASTM D92.

Petroleum and related products — Determination of flash and fire points — Cleveland open cup method

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 this document, and fulfil statutory and regulatory requirements for this purpose.

1 Scope

This document specifies a procedure for the determination of flash and fire points of petroleum products using the Cleveland open cup apparatus. It is applicable to petroleum products having open cup flash points between 79 °C and 400 °C, except fuel oils which are most commonly tested by the closed cup procedure described in ISO 2719.

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 3170, *Petroleum liquids — Manual sampling*

ISO 3171, *Petroleum liquids — Automatic pipeline sampling*

3 Terms and definitions

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

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

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

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

3.1
flash point
lowest temperature of the test portion, corrected to a standard atmospheric pressure of 101,3 kPa, at which application of a test flame causes the vapour of the test portion to ignite under the specified conditions of test

Note 1 to entry: See 10.10.

3.2
fire point
lowest temperature of the test portion, corrected to a barometric pressure of 101,3 kPa, at which application of a test flame causes the vapour of the test portion to ignite and sustain burning for a minimum of 5 s under the specified conditions of test

4 Principle

The test cup is filled to a specified level with the test portion. The temperature of the test portion may be increased rapidly (5 °C/min to 17 °C/min) at first and then at a slow constant rate (5 °C/min to

6 °C/min) as the flash point is approached. At specified temperature intervals, a small test flame is passed across the test cup.

The lowest temperature at which application of the test flame causes the vapour above the surface of the liquid to ignite is taken as the flash point at ambient barometric pressure. To determine the fire point, the test is continued until the application of the test flame causes the vapour above the test portion to ignite and burn for at least 5 s. The flash point and fire point obtained at ambient barometric pressure are corrected to standard atmospheric pressure using a formula.

5 Chemicals and materials

5.1 Cleaning solvent, for 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 may be used to remove traces of oil, and mixed solvents can be efficacious for the removal of gum type deposits.

5.2 Verification liquids, certified reference material (CRM) or secondary working standards (SWS) (see, for instance, C.2).

5.3 Steel wool, any grade capable of removing carbon deposits without damage to the test cup.

6 Apparatus

6.1 Cleveland open cup apparatus, as specified in Annex A.

If automated equipment is used, ensure that it has been established that the results obtained are within the precision of this document, that the test cup and test flame applicator conform to the key dimensional and mechanical requirements specified in Annex A and the procedures described in Clauses 7, 10 and 11 are followed. If automated testers are used, the user shall ensure that all the manufacturer's instructions for adjusting and operating the instrument are followed.

In cases of dispute, the flash point as determined manually shall be considered the referee test.

6.2 Shield, to cover at least three sides of the test cup.

The apparatus may include a built-in draught shield.

6.3 Temperature measuring device, which shall meet the requirements for accuracy and have the response as specified in Annex B.

6.4 Barometer, reading absolute pressure accurate to 0,5 kPa with a resolution of 0,1 kPa. Barometers precorrected to give sea level readings, such as those used at weather stations and airports, shall not be used.

7 Preparation of apparatus

7.1 Location of apparatus

Place the apparatus (6.1) on a level and steady surface in a draught-free room (see below). Shield the top of the manual apparatus from strong light by any suitable means to permit detection of the flash point.

When draughts cannot be avoided, it is recommended good practice to surround the apparatus with a shield.

When testing samples which produce toxic vapours, the apparatus may be located in a fume hood with an individual control of air flow, adjusted so that vapours are withdrawn without causing air currents over the test cup.

7.2 Cleaning the test cup

Wash the test cup with an appropriate solvent (5.1) to remove any traces of gum or residue remaining from a previous test. Dry the test cup using a stream of clean air to ensure complete removal of the solvent used. If any deposits of carbon are present, remove them by rubbing with steel wool (5.3).

7.3 Preparing the test cup

7.3.1 If the alternative procedure referred to in 10.1 is used, follow the instructions in Annex D.

7.3.2 Before use, cool the test cup to at least 56 °C below the expected flash point.

7.4 Assembly of apparatus

Support the liquid in glass thermometer in a vertical position with the bottom of the bulb (6,4 ± 0,5) mm from the bottom of the test cup, and located at a point halfway between the centre and side of the test cup on a diameter perpendicular to the arc (or line) of the sweep of the test flame, and on the side opposite to the test flame applicator. It is not necessary to restrict electronic temperature measuring devices to be mounted vertically, provided their performance is in accordance with the requirements in the test method.

The vertical position of the temperature measuring device may be set by lowering until it contacts the bottom of the test cup, and then raise it by (6,4 ± 0,5) mm.

7.5 Verification of apparatus

7.5.1 Verify the correct functioning of the apparatus at least once a year by testing a certified reference material (CRM) (5.2). The result obtained shall be equal to or less than $R/\sqrt{2}$ from the certified value of the CRM, where R is the reproducibility of the method (see Clause 14).

It is recommended practice during verification of an automated apparatus to visually observe the detected flash point for correct operation.

It is recommended that more frequent verification checks are made using secondary working standards (SWSs) (5.2) or other verification materials with a proven value.

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

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

7.5.3 It is good practice to select a CRM or SWS that has a certified value similar to the flash point of products being tested.

8 Sampling

8.1 Unless otherwise specified, obtain samples for analysis in accordance with the procedures given in ISO 3170, ISO 3171 or an equivalent national standard.

8.2 Place samples in tightly sealed containers, appropriate to the material being sampled, and for safety purposes, ensure that the sample container is only filled 85 % to 95 % of its capacity.

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

9 Sample handling

9.1 Subsampling

Subsample at a temperature at least 56 °C below the expected flash point. If an aliquot of the original sample is to be stored prior to testing, ensure that the container is filled to more than 50 % of its capacity.

NOTE The results of flash point determinations can be affected if the sample volume falls below 50 % of the container capacity.

9.2 Samples containing undissolved water

Flash point results can be affected by the presence of water; if a sample contains undissolved water, decant a water-free aliquot prior to mixing.

NOTE Flash and fire point results can be affected by the presence of water, and splashing can occur.

9.3 Samples that are liquid at ambient temperature

Mix samples by gently shaking by hand prior to the removal of the test portion, taking care to minimize the loss of volatile components, and proceed in accordance with Clause 10.

9.4 Samples that are semisolid or solid at ambient temperature

Heat the sample in its container in a heating bath or oven at a temperature not exceeding 56 °C below the expected flash point. Ensure that high pressures do not develop in the container. Avoid overheating the sample as this could lead to the loss of volatile components. After gentle agitation, proceed in accordance with Clause 10.

10 Procedure for determining flash point

10.1 Samples that can form a skin during testing may be tested by removing the skin formed as described in 10.6 to 10.8.1. An alternative procedure is given in Annex D.

10.2 Record the absolute barometric pressure using a barometer (6.4) in the vicinity of the apparatus at the time of 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.

10.3 Fill the test cup at ambient or elevated temperature (see 9.4) so that the top of the meniscus is level with the filling mark. Position the test cup on the centre of the heating plate. If too much sample has been added to the test cup, remove the excess using a pipette or other suitable device; however, if there is any sample on the outside of the apparatus, empty, clean and refill it. Destroy or remove any air bubbles or foam on the surface of the sample while maintaining the correct level of test portion in the test cup. If foam persists in the final stages of the test, discard the result.

10.4 Light the test flame and adjust it to a diameter between 3,2 mm and 4,8 mm.

As a safety practice, it is strongly advised, when using automated or a manual apparatus, before heating the test cup and test portion, to pass the test flame across the test portion in the test cup to check for the presence of unexpected volatile material. Thereafter, it is recommended to test for a flash every 10 °C until the test portion is within 56 °C of the expected flash point.

10.5 Apply heat initially so that the rate of temperature rise of the test portion is 5 °C/min to 17 °C/min. When the test portion temperature is approximately 56 °C below the expected flash point, decrease the heat so that the rate of temperature rise for the last 28 °C before the expected flash point is 5 °C/min to 6 °C/min.

10.6 Avoid disturbing the vapours in the test cup by careless movements or breathing near the test cup (see 7.1).

10.7 Apply the test flame when the temperature of the test portion is 28 °C below the expected flash point. The test flame is applied in one direction with a smooth continuous motion, taking (1 ± 0,1) s, to pass across the centre of the test cup, at right angles to the diameter which passes through the thermometer, either in a straight line or along the circumference of a circle having a radius of at least 150 mm. The centre of the test flame shall move in a horizontal plane not more than 2 mm above the plane of the upper edge of the test cup. For the next test flame application, pass the flame in the opposite direction.

NOTE Some automated apparatus pass the test flame in one single direction.

10.8 If a flash is detected on this application of the test flame or during a preliminary application (see 10.4), discontinue the test and repeat the test using a fresh test portion with an expected flash point of at least 28 °C below the previous test value.

10.8.1 If a skin forms over the test portion, carefully move it aside with a spatula or comb and continue the determination.

10.8.2 If a flash has not been detected, continue applying the test flame each time thereafter at a temperature reading that is a multiple of 2 °C.

NOTE Higher flash points have been detected when skins formed on the surfaces of test portions have not been removed.

10.9 When testing a sample whose expected flash point temperature is not known, bring the test portion in the test cup to a temperature no greater than 50 °C, or if the sample required heating to be transferred into the test cup, bring the test portion in the test cup to that temperature. Apply the test flame, in the manner described in 10.7, beginning at least 5 °C above the starting temperature. Continue heating the test specimen at 5 °C/min to 6 °C/min and testing the test specimen every 2 °C as described in 10.7 until the flash point is obtained.

This value may be used as the expected flash point when a fresh test portion is tested in the standard mode of operation. Flash point results determined in an unknown expected flash point mode should be considered approximate.

10.10 Record as the detected flash point, the temperature of the test portion, read on the thermometer, when application of the test flame causes the vapours of the test portion to ignite at any point on the surface of the test portion and a large flame propagate over the surface of the test portion, under the specified conditions of test.

Do not confuse the true flash point with the bluish halo that sometimes surrounds the test flame.

11 Procedure for determining fire point

WARNING — This test can involve very high test portion and heating plate temperatures at which the test portion can spit, bubble, foam or smoke, and when the fire point is reached the resultant flame can be very large and difficult to extinguish, and can alarm personnel.

To determine the fire point, after carrying out the procedure specified in Clause 10, continue heating so that the test portion temperature increases at a rate of 5 °C/min to 6 °C/min. Continue the application of the test flame at 2 °C intervals until the vapour of the test portion ignites and continues to burn for at least 5 s. Record the temperature at this point as the detected fire point of the sample.

If the fire persists for more than 5 s, extinguish it with a cover made of metal or other fire-resistant material fitted with a handle. An example of such a cover is given in Figure A.2.

12 Calculation

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

Reading in hPa × 0,1 = kPa

Reading in mbar × 0,1 = kPa

Reading in mmHg × 0,133 3 = kPa

12.2 Calculate the corrected flash point or fire point, t_c, using Formula (1):

$$t_c = t_d + 0{,}25(101{,}3 - p) \quad (1)$$

where

t_d is the detected flash point or fire point at ambient barometric pressure, in °C;

p is the absolute barometric pressure, in kPa;

0,25 is a constant with dimensions °C/kPa;

101,3 is used as the standard atmospheric pressure in kPa.

NOTE 1 Formula (1) has been proven for barometric pressures down to 82,0 kPa[13] and is strictly correct only up to 104,7 kPa.

NOTE 2 For practical purposes, 4 kPa is equivalent to a flash or fire point temperature change of 1 °C.

13 Expression of results

Record the following:

a) the flash point, corrected to standard atmospheric pressure, rounded to the nearest 1 °C, and, if required,

b) the fire point, corrected to standard atmospheric pressure, rounded to the nearest 1 °C.

14 Precision

14.1 General

The precision[14], as determined by statistical examination by ISO 4259 (flash point only) of interlaboratory test results, is given in 14.2 and 14.3.

14.2 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 following values in only one case in 20.

Flash point, $r = 8$ °C

Fire point, $r = 8$ °C

14.3 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 following values in only one case in 20.

Flash point, $R = 18$ °C

Fire point, $R = 14$ °C

15 Test report

The test report shall contain at least the following information:

a) a reference to this document;

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

c) the result of the test (see Clause 13);

d) whether the procedure in Annex D was used or not;

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

f) the date of the test.

Annex A
(normative)

Cleveland open cup apparatus

A.1 Test cup.

The test cup shall be manufactured from brass, or other non-rusting metal of equivalent heat conductivity, conforming to the dimensional requirements as shown in Figure A.1 (left). The test cup may be equipped with a handle.

A.2 Heating plate.

The heating plate shall be of sufficient dimensions and materials to ensure that thermal heat to the test cup is only applied to the bottom of the test cup and that extraneous heat to the test cup is minimized. Figure A.1 (right) shows a typical configuration when using a gas burner.

A.3 Test flame applicator.

The device for applying the flame may be of any suitable type, but it is recommended that the outside diameter of the tube has a maximum of 2 mm and the tip is approximately (1,6 ± 0,05) mm in diameter at the end, and that the orifice at the end is approximately (0,8 ± 0,05) mm in diameter. The device for operating the test flame may be mounted in such a manner as to permit automatic duplication of the sweep of the test flame, the radius of swing being not less than 150 mm, and the position of the centre of the test flame being supported so that it swings in a plane not more than 2 mm above the plane of the rim of the test cup.

A metal bead or ring, having a diameter of 3,2 mm to 4,8 mm, mounted in a convenient position on the apparatus can help ensure that the test flame is the specified size. In Figure A.1, it is pictured on the heating plate.

A.4 Heater.

Use either a controlled electric heater, or a gas burner or alcohol lamp, although under no circumstances allow the products of combustion or free flame to come up around the test cup. Centre the source of heat under the opening of the heating plate so that there is no local superheating. If an electric heater is used, ensure that the electrical element does not come into direct contact with the test cup.

Flame-type heaters may be protected from draughts or excessive radiation by any suitable type of shield that does not project above the level of the upper surface of the heat-resistant barrier.

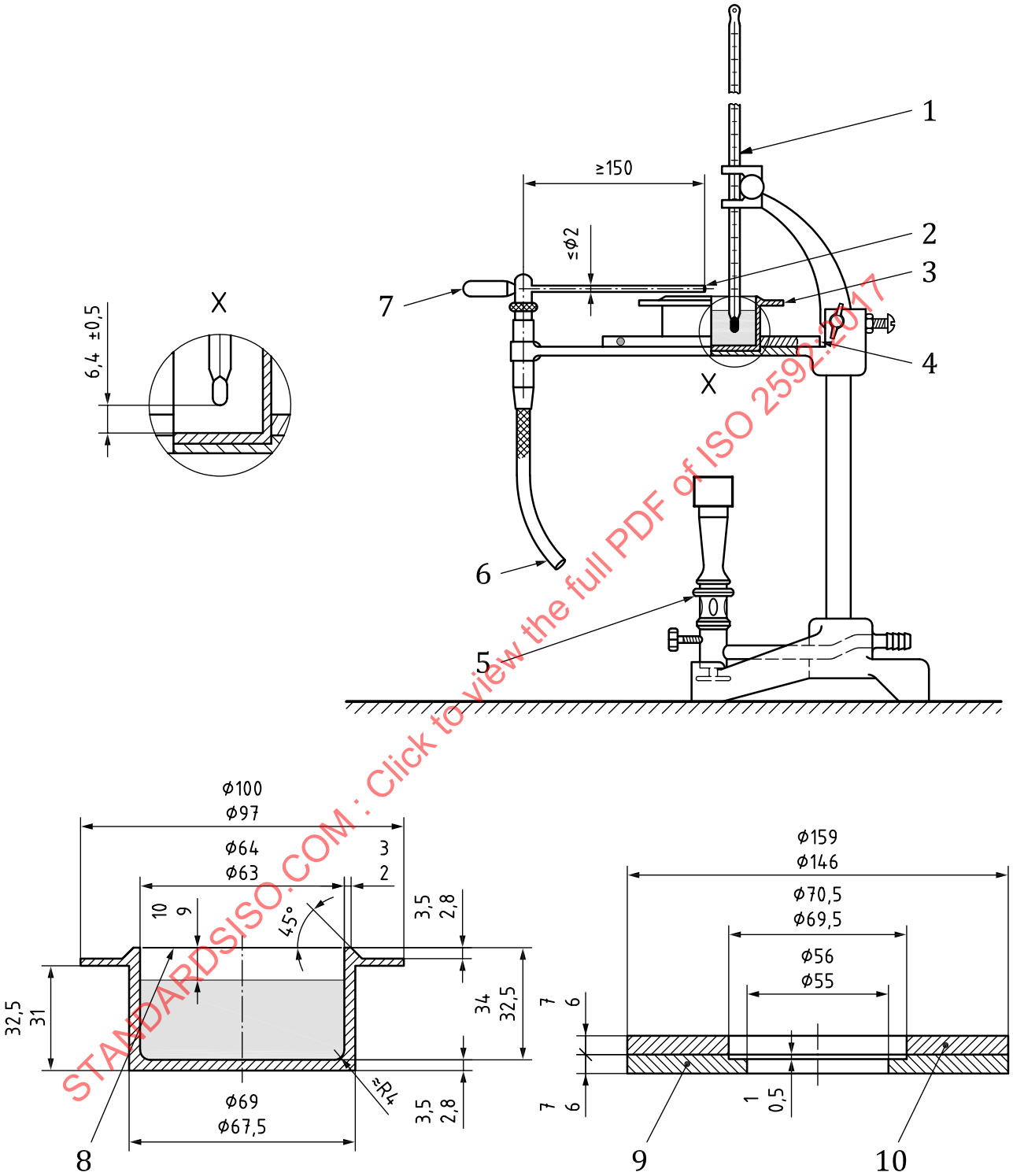
A.5 Temperature measuring device support.

This shall hold the temperature measuring device in the specified position during a test and permit easy removal from the test cup upon completion of a test.

A.6 Heating plate support.

This shall hold the heating plate level and steady.

Dimensions in millimetres



Key

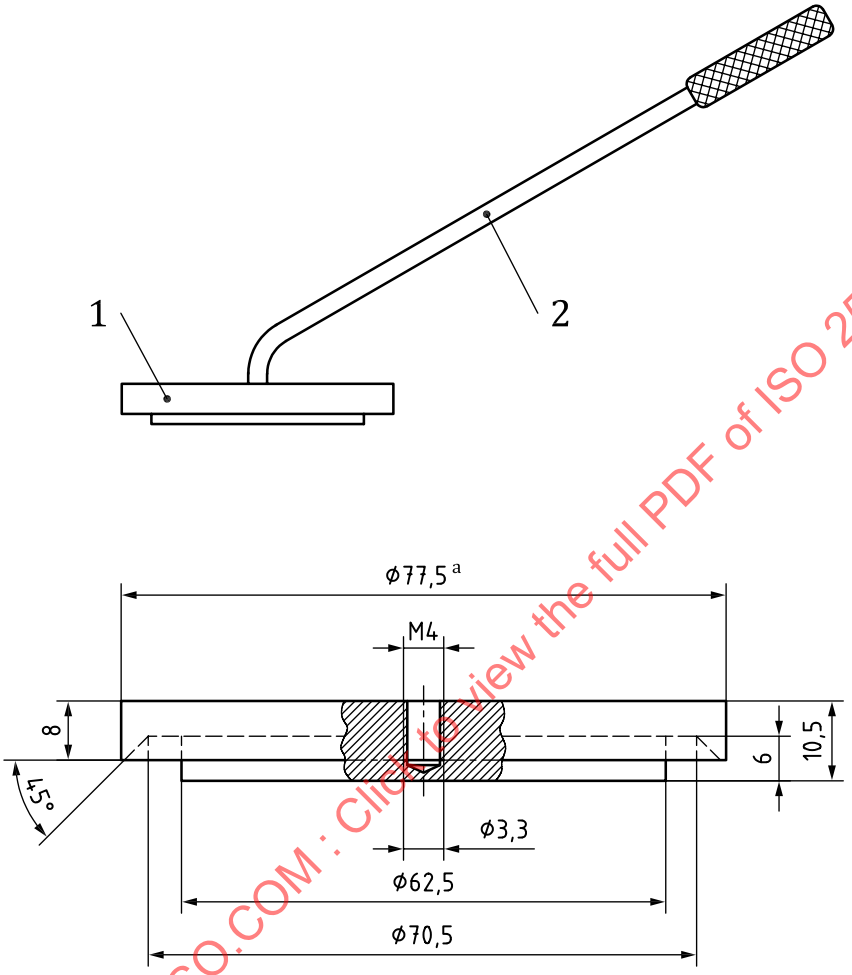
1	thermometer	6	to gas supply
2	test flame orifice (see A.3)	7	test-flame applicator
3	test cup (dimensions as in the figure on the left)	8	filling mark
4	heating plate	9	metal
5	heater (flame or electric resistance type)	10	heat-resistant material

Figure A.1 — Cleveland open cup apparatus

A.7 Flame extinguisher (optional).

An example of a suitable device is shown in Figure A.2. In order for it to be used, one should allow the thermometer (A.5) mounting to be flexible as it should be hinged or pushed out of the way or pulled out of the way in order for the extinguisher to stop some of the flames.

Dimensions in millimetres



Key

1 cover of metal or other fire-resistant material
2 handle
a Reference size.

Figure A.2 — Example of a flame extinguisher

Annex B
(normative)

Temperature measuring device specification

B.1 Electronic

B.1.1 Temperature range, at least ambient to 400 °C.

B.1.2 Display resolution, better than 0,5 °C.

B.1.3 Accuracy (after calibration), 2,0 °C for temperatures up to 260 °C and 4,0 °C for temperatures over 260 °C.

B.1.4 Immersion depth, less than 25 mm.

B.1.5 Thermal response time (63,2 % according to ASTM E1137), 0 s to 5 s.

NOTE 1 Guidelines for digital temperature measuring devices are given in ASTM E1137[11] and IEC 60751[8].

NOTE 2 The 63,2 % thermal response time is the time for the display to indicate 63,2 % of a step change in temperature from a nominal ambient of 20 °C in air to a nominal 77 °C in stirred water.

B.2 Liquid in glass thermometers

The thermometer shall be in line with the specifications in Table B.1.

Table B.1 — Liquid in glass thermometer specification

Feature	Specification
Temperature range, °C	−6 to 400
Immersion, mm	25
Scale marks:	
Subdivision, °C	2
Long lines at each °C	10
Numbered at each °C	20
Scale error, maximum, °C	2 up to 260 4 over 260
Expansion chamber:	
Permit heating to °C	400
Total length, mm	(310 ± 5)
Stem OD, mm	(7,0 ± 1,0)
Bulb length, mm	(5,25 ± 0,75)
Scale location:	
Bottom of bulb to line at °C	0
Distance, mm	(50 ± 5)
Length of scale range, mm	(225 ± 15)
NOTE Guidance on liquid in glass thermometers with low hazard precision liquids is given in ASTM E2251[12].	

Annex C
(informative)

Verification of apparatus

C.1 General

This annex describes a procedure for conducting verification checks 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 (manual or automated) should be verified on a regular basis using either a CRM produced in accordance with ISO Guide 34 and ISO Guide 35 or an in-house reference material/SWS prepared in accordance with one of the procedures given in C.2.2. Further guidance is given in ISO Guide 33 and ISO 4259.

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

C.2 Verification check standards

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

C.2.2 Secondary working standard (SWS), comprising a stable petroleum product or a 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 at least three 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 that will retain the integrity of the SWS out of direct sunlight and at a temperature not exceeding 10 °C.

C.3 Procedure

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

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

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

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

C.3.4 Correct the result for barometric pressure in accordance with Clause 12. Record the corrected result, to the nearest 0,1 °C, in a permanent record.

Table C.1 — Approximate values of the Cleveland open cup flash points of hydrocarbons

Hydrocarbon	Nominal flash point °C
Tetradecane	116
Hexadecane	139

C.4 Evaluation of the test result

C.4.1 Comparison

C.4.1.1 General

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

In the relations given in C.4.1.2 and C.4.1.3, 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 set out in ISO Guide 35, and that its uncertainty is small in comparison with the standard deviation of the test method and thus small compared with the reproducibility of the test method, *R*.

C.4.1.2 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 assigned value of the SWS should be within the tolerance given in Formula (C.1):

$$|x-\mu| \leq \frac{R}{\sqrt{2}} \tag{C.1}$$

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.

C.4.1.3 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 assigned value of the SWS, should be within the tolerance given in Formula (C.2):

$$\left|\bar{x}-\mu\right| \leq \frac{R_1}{\sqrt{2}} \tag{C.2}$$

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 $\sqrt{R^2 - r^2\left[1-(1/n)\right]}$

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.

C.4.2 Conformity

C.4.2.1 If the test result conforms to the tolerance requirements, record this fact.

C.4.2.2 If the result does not conform to the tolerance requirements and a 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.

C.4.2.3 If the test result still does not conform to the tolerance requirements, examine the apparatus and check that it conforms to 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.