

INTERNATIONAL STANDARD

ISO 2592

Second edition
2000-09-15

Determination of flash and fire points — Cleveland open cup method

*Détermination des points d'éclair et de feu — Méthode Cleveland à vase
ouvert*



Reference number
ISO 2592:2000(E)

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Printed in Switzerland

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 3.

Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this International Standard may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

International Standard ISO 2592 was prepared by Technical Committee ISO/TC 28, *Petroleum products and lubricants*.

This second edition cancels and replaces the first edition (ISO 2592:1973), which has been technically revised.

Annexes A and B form a normative part of this International Standard. Annex C is for information only.

Determination of flash and fire points — Cleveland open cup method

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

1 Scope

This International Standard specifies a 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 an open cup flash point above 79 °C, except fuel oils, which are most commonly tested by the closed cup procedure described in ISO 2719 [1].

NOTE Flash point and fire point are indications of the ability of a substance to form a flammable mixture with air under controlled conditions, and then to support combustion. They are only two of a number of properties that may contribute towards the assessment of overall flammability and combustibility of a material.

2 Normative references

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

ISO 3170:1988, *Petroleum liquids — Manual sampling*.

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

3 Terms and definitions

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

3.1

flash 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 the flame to propagate across the surface of the liquid, under the specified conditions of test

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 is increased rapidly at first and then at a slow constant rate 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 an equation.

5 Reagents and materials

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

NOTE The choice of solvent will depend upon the previous material tested, and the tenacity of the residue. Low volatility aromatic (benzene free) solvents may be used to remove traces of oil, and mixed solvents such as toluene-acetone-methanol may be efficacious for the removal of gum-type deposits.

5.2 Verification liquids, as described in 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 International Standard and that the test cup and test flame applicator conform to the dimensional and mechanical requirements specified in annex A. 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, approximately 460 mm square and 610 mm high, and having an open front.

6.3 Thermometer, of the partial immersion type, conforming to the specification given in annex B.

NOTE Other types of temperature-measuring devices may be used, provided that they meet the requirements for accuracy and have the same response as the thermometers specified in annex B.

6.4 Barometer, accurate to 0,1 kPa. Barometers pre-corrected 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 notes 1 and 2 below). Shield the top of the apparatus from strong light by any suitable means, to permit detection of the flash point.

NOTE 1 When draughts cannot be avoided, it is good practice to surround the apparatus with a shield.

NOTE 2 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 can be 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

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

7.4 Assembly of apparatus

Support the thermometer in a vertical position with the bottom of the bulb 6 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.

NOTE The immersion line engraved on the thermometer will be 2 mm below the level of the rim of the test cup when the thermometer is properly positioned. An alternative method is to gently lower the thermometer until it contacts the bottom of the test cup, and then raise it 6 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 that more frequent verification checks are made using secondary working standards (SWSs) (5.2).

NOTE 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.

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 to between 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 (see 10.1).

9.2 Samples containing undissolved water

If a sample contains undissolved water, decant an aliquot from the water prior to mixing.

NOTE Flash point results can be affected by the presence of water.

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 semi-solid 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. 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 The results of flash point determinations may be affected if the sample volume falls below 50 % of the container capacity.

10.2 Record the ambient 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 exactly at the filling line. 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 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 whilst maintaining the correct level of test portion in the test cup. If a 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, the size of the comparison bead if one is mounted on the apparatus.

10.5 Apply heat initially so that the rate of temperature rise of the test portion is 14 °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 (23 ± 5) °C before the expected flash point is 5 °C/min to 6 °C/min.

During the test, take care to avoid disturbing the vapours in the test cup by careless movements or breathing near the test cup (see note 2 to 7.1).

10.6 Starting at least (23 ± 5) °C below the expected flash point, apply the test flame when the temperature read on the thermometer (6.3) reaches each successive 2 °C mark. With a smooth continuous motion, taking approximately 1 s, pass the test flame in one direction 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.

If a skin forms over the test portion, carefully move it aside and continue the determination.

10.7 Record as the observed flash point, the temperature of the test portion, read on the thermometer, when application of the test flame causes the vapour of the test portion to ignite and propagate across the surface of the liquid. Do not confuse the true flash point with the bluish halo that sometimes surrounds the test flame.

10.8 When the temperature at which the flash point is observed is less than 18 °C from the temperature of the first application 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 obtained when the flash point is 18 °C above the temperature of the first application of the test flame.

11 Procedure for determining fire point

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 observed 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 Conversion of barometric pressure reading

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

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

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

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

12.2 Correction of observed flash point or fire point to standard atmospheric pressure

Calculate the flash point or fire point corrected to a standard atmospheric pressure of 101,3 kPa, T_C , using the following equation:

$$T_C = T_0 + 0,25(101,3 - p)$$

where

T_0 is the flash point or fire point at ambient barometric pressure, in degrees Celsius;

p is the ambient barometric pressure, in kilopascals.

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

13 Expression of results

Report the corrected flash point or fire point, rounded to the nearest even number, in degrees Celsius.

14 Precision

14.1 General

The precision, as determined by statistical examination by ISO 4259 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 twenty.

Flash point, $r = 8\text{ °C}$

Fire point, $r = 8\text{ °C}$

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 twenty.

Flash point, $R = 17\text{ °C}$

Fire point, $R = 14\text{ °C}$

15 Test report

The test report shall contain at least the following information:

- a) a reference to this International Standard;
- b) the type and complete identification of the product tested;
- c) the result of the test (see clause 13);
- d) any deviation, by agreement or otherwise, from the procedure specified;
- e) the date of the test.

Annex A (normative)

Cleveland open cup apparatus

A.1 Test cup

Manufactured from brass, or other non-rusting metal of equivalent heat conductivity, conforming to the dimensional requirements shown in Figure A.1.

NOTE The test cup may be equipped with a handle.

A.2 Heating plate

A brass, cast iron, wrought iron or steel plate, with a centrehole surrounded by an area of plane depression, and a sheet of hard heat-resistant board (not containing asbestos) which covers the metal plate except over the area of plane depression in which the test cup is supported. The heating plate shall conform to the dimensions shown in Figure A.1.

NOTE The heating plate may be square instead of round, and the metal plate may have suitable extensions for mounting the test flame applicator and the thermometer support. Also, a metal bead, as mentioned in A.3, may be mounted on the plate so that it extends through and slightly above a suitable small hole in the heat-resistant board.

A.3 Test flame applicator

The device for applying the flame may be of any suitable type, but it is suggested that the tip is approximately 1,6 mm in diameter at the end, and that the orifice is 0,8 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 centre of the orifice being supported so that it swings in a plane not more than 2 mm above the plane of the rim of the test cup.

It is desirable that a metal bead, having a diameter of 3,2 mm to 4,8 mm, be mounted in a convenient position on the apparatus so that the size of the test flame can be compared to it.

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 it does not come into direct contact with the test cup.

NOTE 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 Thermometer support

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

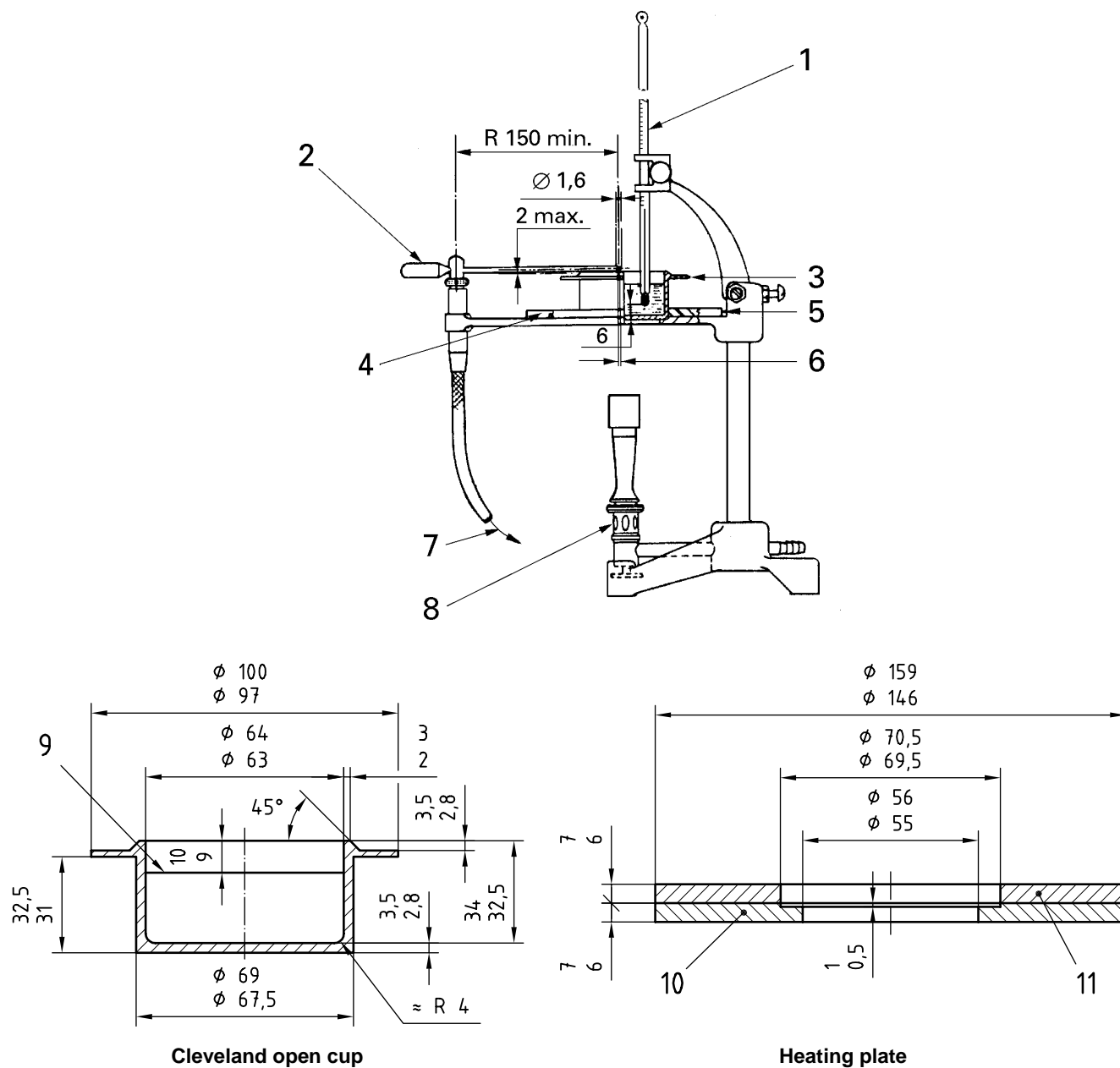


Figure A.1 — Cleveland open cup apparatus

Key

- | | | | |
|---|-------------------------|----|--|
| 1 | Thermometer | 7 | To gas supply |
| 2 | Test-flame applicator | 8 | Heater (flame or electric resistance type) |
| 3 | Test cup | 9 | Filling mark |
| 4 | Metal bead Ø 3,2 to 4,8 | 10 | Metal |
| 5 | Heating plate | 11 | Heat-resistant material |
| 6 | Orifice Ø 0,8 | | |

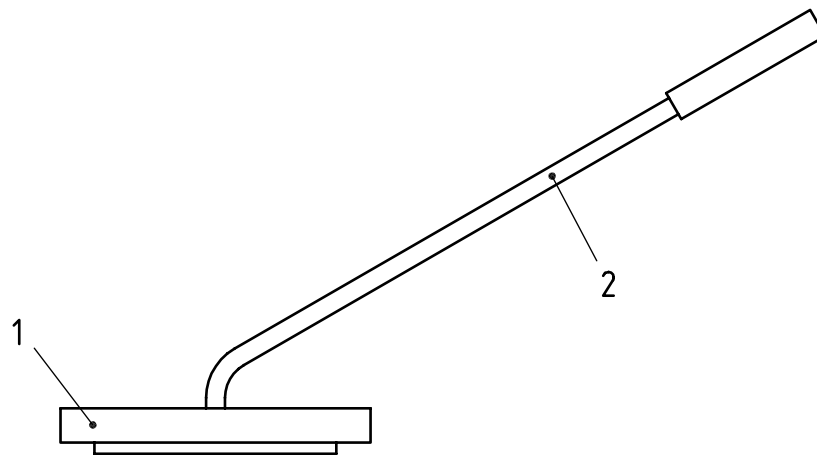
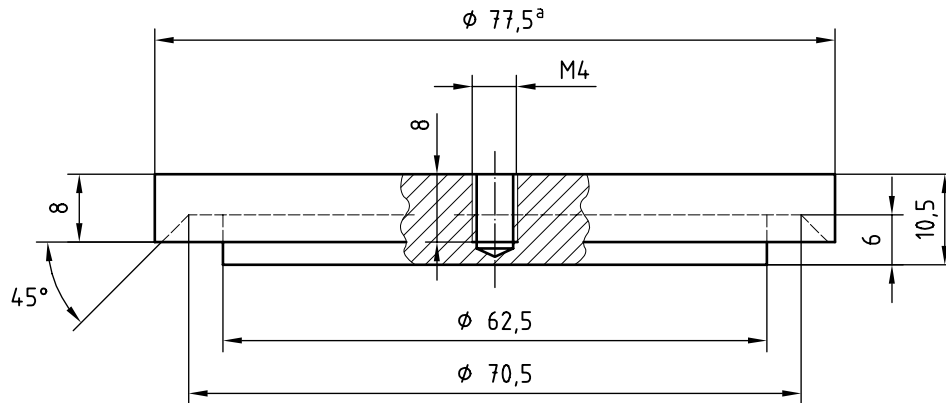
A.6 Heating plate support

This shall hold the heating plate level and steady.

A.7 Flame extinguisher (optional)

An example of a suitable device is shown in Figure A.2.

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)

Thermometer specification

Table B.1 — 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	An IP 28C/ASTM 11C thermometer meets the above specification.

Annex C (informative)

Verification of apparatus

C.1 General

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

The performance of the apparatus (manual or automated) should be verified on a regular basis using either a CRM produced in accordance with ISO Guide 34^[4] and ISO Guide 35^[5], or an in-house reference material/SWS prepared in accordance with one of the procedures given in C.2.2. The performance of the apparatus should be assessed in accordance with the guidance given in ISO Guide 33^[3] and ISO 4259^[2].

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 inter-laboratory 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 the results, or
- b) conducting an interlaboratory method-specific test programme utilizing at least three laboratories testing representative samples in duplicate. The assigned value of the flash point should be calculated after statistically analysing the interlaboratory data.

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

C.3 Procedure

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

It is recommended that 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.

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

Hydrocarbon	Nominal flash point °C
Tetradecane	116
Hexadecane	139

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

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

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.

C.4 Evaluation of the test result

C.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 relations given in C.4.1.1 and C.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 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.4.1.1 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:

$$|x - \mu| \leq \frac{R}{\sqrt{2}}$$

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.4.1.2 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:

$$|\bar{x} - \mu| \leq \frac{R_1}{\sqrt{2}}$$

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 $\sqrt{R^2 - r^2 \{1 - 1/n\}}$;

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.

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

C.4.3 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.4.4 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.

Bibliography

- [1] ISO 2719:1988, *Petroleum products and lubricants — Determination of flash point — Pensky-Martens closed cup method.*
- [2] ISO 4259:1992, *Petroleum products — Determination and application of precision data in relation to methods of test.*
- [3] ISO Guide 33:1989, *Uses of certified reference materials.*
- [4] ISO Guide 34:2000, *General requirements for the competence of reference material producers.*
- [5] ISO Guide 35:1989, *Certification of reference materials — General and statistical principles.*

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