INTERNATIONAL STANDARD

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Petroleum products and other liquids — Determination of flash point — Abel closed cup method

Produits pétroliers et autres liquides — Détermination du point d'éclair — Méthode Abel en vase clos

Reference number
ISO 13736:1997(E)

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.

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.

International Standard ISO 13736 was prepared by Technical Committee ISO/TC 28, *Petroleum products and lubricants*.

Annexes A to D form an integral part of this International Standard. Annex E is for information only.

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Petroleum products and other liquids — Determination of flash point — Abel closed cup method

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

1 Scope

This International Standard specifies a method for the determination of the closed cup flash point of petroleum products and other liquids having flash points between – 30 °C and 70 °C inclusive. However the precision given for the method is only valid for flash points in the range – 5 °C to 66,5 °C.

This standard is not applicable to water borne paints which may however be tested using ISO 3679.

NOTES

1 This method should be used to measure and describe the properties of materials, products, or assemblies in response to heat and flame under controlled laboratory conditions, and should not be used to describe or appraise the fire hazard or fire risk of materials, products, or assemblies under actual fire conditions. However, results of this test may be used as elements of a fire risk assessment which takes into account all of the factors which are pertinent to an assessment of the fire hazard of a particular end use.

2 Flash point is used in shipping, storage, and 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.

3 Flash point may indicate the possible presence of highly volatile materials in a relatively non-volatile or non-flammable material.

4 Since the presence of small proportions of highly volatile materials need to be detected, this test should be the first determination on a received sample.

5 Liquids containing halogenated compounds may give anomalous results.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

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

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

ISO 3679:1983, *Paints, varnishes, petroleum and related products — Determination of flash point — Rapid equilibrium method.*

3 Definition

For the purposes of this International Standard, the following definition applies.

3.1 flash point: The lowest temperature, 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 under the specified conditions of test.

4 Principle

The test portion is placed in the test cup of an Abel apparatus and heated at specified rates. A small test flame is directed into the test cup at regular intervals and the flash point is taken as the lowest temperature at which application of the test flame causes the vapour above the test portion to ignite with a distinct flash inside the test cup.

NOTE — Separate test procedures are defined for liquids flashing between – 30 °C and 18,5 °C inclusive, and between 19 °C and 70 °C inclusive.

5 Reagents and materials

5.1 Solvent, low volatility aromatic solvent (benzene-free) for removal of traces of sample from the test cup.

NOTE — The choice of solvent will depend upon the previous sample, and the tenacity of the residue. Mixed solvents, such as toluene-acetone-methanol (TAM) may be efficacious for the removal of gum-type deposits.

5.2 Ethanediol (ethylene glycol), corrosion inhibited or **glycerol**.

5.3 Silicone lubricant.

6 Apparatus

6.1 Abel flash point apparatus

Use an Abel petroleum testing apparatus as described in annex A.

If automated testers are used, ensure that all of the manufacturer's instructions for calibrating, adjusting and operating the instrument are followed. In any cases of dispute, the flash point as determined manually shall be considered the refereeing test.

NOTE — Automated equipment may be used provided that it has been established that results obtained will not differ from those obtained by the manual procedure.

6.2 Test cup thermometer

Use a test cup thermometer conforming to the specification given in annex C. It shall be fitted into a collar as described in annex B.

6.3 Heating vessel thermometer

Use a heating vessel thermometer conforming to the specification given in annex C. It shall be fitted into a collar as described in annex B.

6.4 Low temperature thermometer

Use a low temperature thermometer conforming to the specification given in annex C or a thermocouple with similar or better precision.

6.5 Timer

Use one of the following:

a) metronome, that beats at a frequency of 75 beats per min to 80 beats per min;

b) pendulum, of 610 mm effective length, counting one beat from one extremity of the swing to the other;

c) electric/electronic timing device, which can measure intervals of 0,75 s to 0,80 s or 1 s.

6.6 Barometer

Use either a Fortin type or other suitable type of barometer, readable to, and with an accuracy of 1 hPa (0,1 kPa). Do not use aneroid barometers pre-corrected to give sea level readings, such as those used at weather stations and airports.

6.7 Cooling bath

Use either liquid or metal block or a recirculating cooler.

6.8 Test cup thermal insulator

Use either a cover made of foam plastics or woollen material.

7 Sampling

7.1 Obtain samples according to the procedures given in ISO 3170, ISO 3171 or an equivalent National Standard, and place in tightly sealed containers appropriate to the material being sampled.

7.2 Samples shall not be taken or stored in plastic containers, since volatile materials may diffuse through the walls, and/or react with the container.

8 Apparatus preparation

Support the Abel apparatus on a level steady table. Unless tests are made in a draught-free room or compartment, surround the tester on three sides with a shield, each section of which shall be approximately 450 mm wide and 600 mm high.

9 Apparatus verification

The correct functioning of the apparatus shall be verified in accordance with annex D.

10 Procedure

10.1 Procedure for liquids flashing between – 30 °C and 18,5 °C

10.1.1 Note the ambient pressure of the laboratory at the time of test by recording the barometric pressure and the temperature of the barometer used or its immediate surroundings.

10.1.2 Fill the heating vessel completely and the air chamber which surrounds the test cup to a depth of at least 38 mm with either a mixture of equal volumes of ethanediol (5.2) and water or a higher ratio of glycerol (5.2) and water.

10.1.3 Adjust the temperature of the heating vessel, using either a cooling bath or recirculating cooler (6.7), to – 35 °C, or to at least 9 °C below the expected flash point of the material being tested, whichever is the higher, measuring the temperature with a low temperature thermometer (6.4). Carry out a trial flash point determination if necessary.

While cooling, mix the water-ethanediol or water-glycerol mixture in the heating vessel either by stirring manually or mechanically, or by means of a gentle stream of air introduced into the heating vessel by a tube inserted through the thermometer socket and reaching to the bottom of the heating vessel.

CAUTION — Eye protection shall be worn to guard against the possible risk from drops splashed or liquid thrown off whilst mixing.

10.1.4 Cool the sample in its container, in a cooling bath or refrigerator to below – 35 °C, or to at least 17 °C below the expected flash point, whichever is the higher, before opening. Replace the closure with a vapour tight closure carrying a low temperature thermometer or suitable thermocouple (6.4) to check the temperature. After reaching the required temperature remove the cover carrying the thermometer and replace the original closure. Maintain the sample at this temperature, or lower, until all flash point tests on the sample are completed.

Cool liquids which crystallise on cooling to just above their crystallising points.

10.1.5 Wash the test cup with an appropriate solvent (5.1) to remove any traces of gum or residue remaining from a previous test. Dry using a stream of clean air. Place a low temperature thermometer (6.4) in position in the cover of the test cup. Loosely assemble the cover and test cup. Cover with the thermal insulator (6.8), and cool the assembly in a cooling bath or refrigerator until the thermometer registers – 35 °C or at least 17 °C below the expected flash point, whichever is the higher.

If a liquid cooling bath is used, ensure that neither cooling liquid nor vapour which could affect the flash point of the product under test enters the test cup.

NOTES

1 A low temperature thermometer filled with alcohol or toluene is used when cooling the test cup and cover to avoid the risk of freezing the mercury in the flash point thermometer and the consequent rupture of the thread.

2 Cooling a cover or test cup that is wet with water to below 0 °C can cause sticking due to ice (e.g. sticking of the slide). Wiping the apparatus dry with a duster or a piece of absorbent paper before cooling to below 0 °C is usually sufficient to prevent icing, but, alternatively, icing can be minimized by the use of a thermal insulating cover (6.8) and by lubricating the outer face of the lip of the test cup and the slide with a silicone lubricant (5.3).

10.1.6 Position the heating vessel on a firm level surface. Place the test cup in position in the apparatus (see clause A.2) and replace the low temperature thermometer by a test cup thermometer (6.2). Remove the cover and pour in the test portion without undue agitation, avoiding as far as possible the formation of air bubbles, until the level just reaches the point of the index gauge on the wall of the test cup. Do not move the apparatus after filling. Place the cover on the test cup and push it down into position. Ignite the test flame, adjust its size to approximately 3,8 mm in diameter, and maintain it at that size throughout the test, comparing it frequently with the projecting white bead mounted on the cover of the test cup.

10.1.7 Remove the low temperature thermometer from the heating vessel and insert the heating vessel thermometer (6.3).

10.1.8 Apply heat to the heating vessel in such a manner that the temperature of the test portion in the test cup rises at a rate of 1 °C/min.

Stir the test portion in a clockwise direction (i.e. to give a downward thrust) at approximately 0,5 s^{-1} (30 r/min) or as close to this rate as the viscosity of the material permits. When testing viscous products ensure that the stirring action does not push the test portion above the filling mark. Continue stirring in a steady manner for the duration of the test but do not stir during the application of the test flame.

10.1.9 When the temperature of the test portion reaches – 35 °C or at least 9 °C below the expected flash point, start the timer (6.5), apply the test flame by slowly and uniformly opening the slide in the cover while the timer beats three times, and closing it during the fourth beat. If an electric/electronic timing device calibrated in seconds is used then the application of the test flame shall be made by slowly and uniformly opening the slide over a period of 2 s and then closing it over a period of 1 s.

If a flash occurs, discontinue the test, discard the test portion and proceed in acordance with 10.1.3, commencing the test at – 35 °C or at least 17 °C below the previous starting temperature, whichever is the higher. If no flash occurs proceed in accordance with 10.1.10. If a flash occurs at a temperature below – 30 °C record and report this fact and discontinue the test.

10.1.10 Apply the test flame in this manner every 0,5 °C rise in temperature until a distinct flash occurs in the interior of the test cup, or until a temperature corresponding to a corrected temperature of 18,5 °C is reached. Record the temperature of the test portion when the flash occurs.

NOTE — The test portion is deemed to have flashed when a large flame appears and instantaneously propagates itself over its surface.

Do not confuse the true flash point with the bluish halo that sometimes surrounds the test flame or an enlarged flame at applications preceding the one that causes the actual flash.

10.1.11 Record as observed flash point the temperature read on the thermometer at the time the test flame application caused a distinct flash in the interior of the test cup.

10.2 Procedure for liquids flashing between 19 °C and 70 °C

10.2.1 Note the ambient pressure of the laboratory at the time of test by recording the barometric pressure and the temperature of the barometer (6.6) used or its immediate surroundings.

10.2.2 Fill the heating vessel completely and the air chamber which surrounds the test cup to a depth of at least 38 mm with water.

10.2.3 Adjust the temperature of the heating vessel, using either a cooling bath or a recirculating cooler (6.7) or a heater, to at least 9 °C below the expected flash point of the material being tested, or to 10 °C, whichever is the higher. Carry out a trial flash point determination if necessary.

10.2.4 Bring the sample in its container, if necessary in a cooling bath or refrigerator, to 2 °C or at least 17 °C below the expected flash point, whichever is the higher, before opening. Maintain the sample at this temperature or lower until all flash point tests on the sample are completed.

10.2.5 Wash the test cup with an appropriate solvent (5.1) to remove any traces of gum or residue remaining from a previous test. Dry, using a stream of clean air. Place a test cup thermometer (6.2) in position in the cover of the test cup. Loosely assemble the cover and test cup, and cool in a refrigerator or a cooling bath until the thermometer registers 2 °C or at least 17 °C below the expected flash point, whichever is the higher.

If a liquid cooling bath is used, ensure that neither cooling liquid nor vapour enters the test cup, which could affect the flash point of the product under test.

10.2.6 Position the heating vessel on a firm level surface. Place the test cup in position in the apparatus (see clause A.2). Remove the cover and pour in the test portion without undue agitation, avoiding as far as possible the formation of air bubbles, until the level just reaches the point of the index gauge on the wall of the test cup. Do not move the apparatus after filling. Place the cover on the test cup and push it down into position. Ignite the test flame, adjust its size to approximately 3,8 mm in diameter, and maintain it at that size throughout the test, comparing it frequently with the projecting white bead mounted on the cover of the test cup.

10.2.7 Apply heat to the heating vessel in such a manner that the temperature of the test portion in the test cup rises at a rate of 1 °C/min.

Stir in a clockwise direction (i.e. to give a downward thrust) at approximately 0,5 s^{-1} (30 r/min) or as close to this rate as the viscosity of the material permits. When testing viscous products, ensure that the stirring action does not push the test portion above the filling mark. Continue stirring in a steady manner for the duration of the test but do not stir during the application of the test flame.

10.2.8 When the temperature of the test portion reaches 10 °C or at least 9 °C below the expected flash point, start the timer (6.5), apply the test flame by slowly and uniformly opening the slide in the cover while the timer beats three times, and closing it during the fourth beat. If an electric/electronic timing device calibrated in seconds is used then the application of the test flame shall be made by slowly and uniformly opening the slide over a period of 2 s and then closing it over a period of 1 s.

If a flash occurs, discontinue the test, discard the test portion and proceed in accordance with 10.1.2 or 10.2.3, as appropriate, commencing the test at least 17 °C below the previous starting temperature. If no flash occurs proceed in accordance with 10.2.9.

10.2.9 Apply the test flame in this manner every 0,5 °C rise in temperature until a distinct flash occurs in the interior of the test cup, or until a temperature corresponding to a corrected temperature of 70 °C is reached. Record the temperature of the test portion when the flash occurs. (See note to 10.1.10.)

Do not confuse the true flash point with the bluish halo that sometimes surrounds the test flame or an enlarged flame at applications preceding the one that causes the actual flash.

10.2.10 Record as the observed flash point the temperature read on the thermometer at the time the test flame application causes a distinct flash in the interior of the test cup.

11 Calculation

11.1 If the barometric pressure reading, taken in accordance with 10.1.1 or 10.2.1, is in a unit other than kilopascals, convert to kilopascals using the following equations

$$kPa = hPa \times 10^{-1}$$

$$kPa = mbar \times 10^{-1}$$

$$kPa = mmHg \times 0{,}133\,322$$

NOTE — For the purposes of correcting flash point values to standard barometric pressure it is not considered necessary to correct the barometer readings to 0 °C. However some barometers are designed to automatically correct the barometric pressure to 0 °C.

11.2 Calculate the corrected flash point, T_c, using the following equation.

$$T_c = T_o + 0{,}25(101{,}3 - P)$$

where

T_o is the observed flash point, in degrees Celsius;

P is the barometric pressure at 0 °C, in kilopascals.

NOTE — For practical purposes 4 kPa is equivalent to a flash point temperature change of 1 °C.

12 Expression of results

Report the corrected result, to the nearest 0,5 °C.

13 Precision

13.1 Repeatability r

The difference between successive test results obtained by the same operator with the same apparatus under constant operating conditions on identical test material, in the normal and correct operation of the test method, would exceed the value below only in one case in twenty.

r = 1,0 °C

13.2 Reproducibility R

The difference between two test results independently obtained by different operators operating in different laboratories on nominally identical test material, in the normal and correct operation of the test method, would exceed the value below only in one case in twenty.

R = 1,5 °C

NOTE — The precision data quoted in 13.1 and 13.2 apply over the range – 5 °C to 66,5 °C.

14 Test report

The test report shall contain at least the following information:

a) a reference to this International Standard;

b) all details necessary for the complete identification of the sample tested;

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

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

e) the date of the test.

Annex A
(normative)

Abel flash point apparatus

The apparatus shall consist of a test cup, cover assembly and heating vessel as described below.

A.1 Test cup

The test cup shall be made of brass and conform to the form and dimensions shown in figure A.1.

A gauge consisting of a rod bent upwards and terminating in a point, shall be fixed within the test cup through the wall, and silver soldered or brazed in place.

A.2 Test cup cover assembly

The test cup shall be provided with a close fitting cover made of brass and conform to the form and dimensions shown in figure A.1. A downward projecting rim barely reaching the flange on the test cup shall either be made solid with the top or silver soldered or brazed in place.

Upon the cover shall be mounted a thermometer socket, a bush for the stirrer, trunnions to support a test gas jet, a pair of guides in which a slide moves, and a white bead. The top of the cover shall be pierced by three rectangular holes symmetrically placed on a diameter, one in the centre and the other two as close as practicable to the inner sides of the rim and opposite each other.

These three holes shall be covered or uncovered by means of a slide moving in suitably disposed guides. The slide shall have two perforations, one corresponding in all particulars to the centre hole in the cover and the other to one of the holes at the side. The movement of the slide shall be restricted by suitable stops, and its length and the disposition of the holes shall be such, that at the outer extremity of the movement of the slide the holes in the cover are just completely opened, and at the inner extremity of the movement of the slide they are completely closed.

The trunnions supporting the test gas jet shall be fixed to the top of the guides and the gas jet shall be mounted in the trunnions so that it is free to oscillate. The test gas jet shall be arranged so that when the slide is moved so as to uncover the holes, the oscillating test gas jet is caught by a pin fixed in the slide and tilted over the central hole in such a way that the lower edge of the cover bisects the circle formed by the bore of the jet when in the lowest position. The flame shall then occupy a central position within the hole in both directions.

The thermometer socket shall be in the form of a split tube, mounted on a diameter at right angles to the diameter through the centres of the holes, and fitted at such an angle as to bring the bulb of the thermometer when in place, vertically below the centre of the cover and at the correct distance from it.

A bush for the stirrer shall be mounted on the cover in a position diametrically opposite the thermometer mounting. Its length and the angle at which it is set shall be such that the stirrer rod clears the oil level gauge and the blades operate below the level of and without fouling the thermometer bulb. The bush shall be placed as near as practicable to the outer edge of the cover.

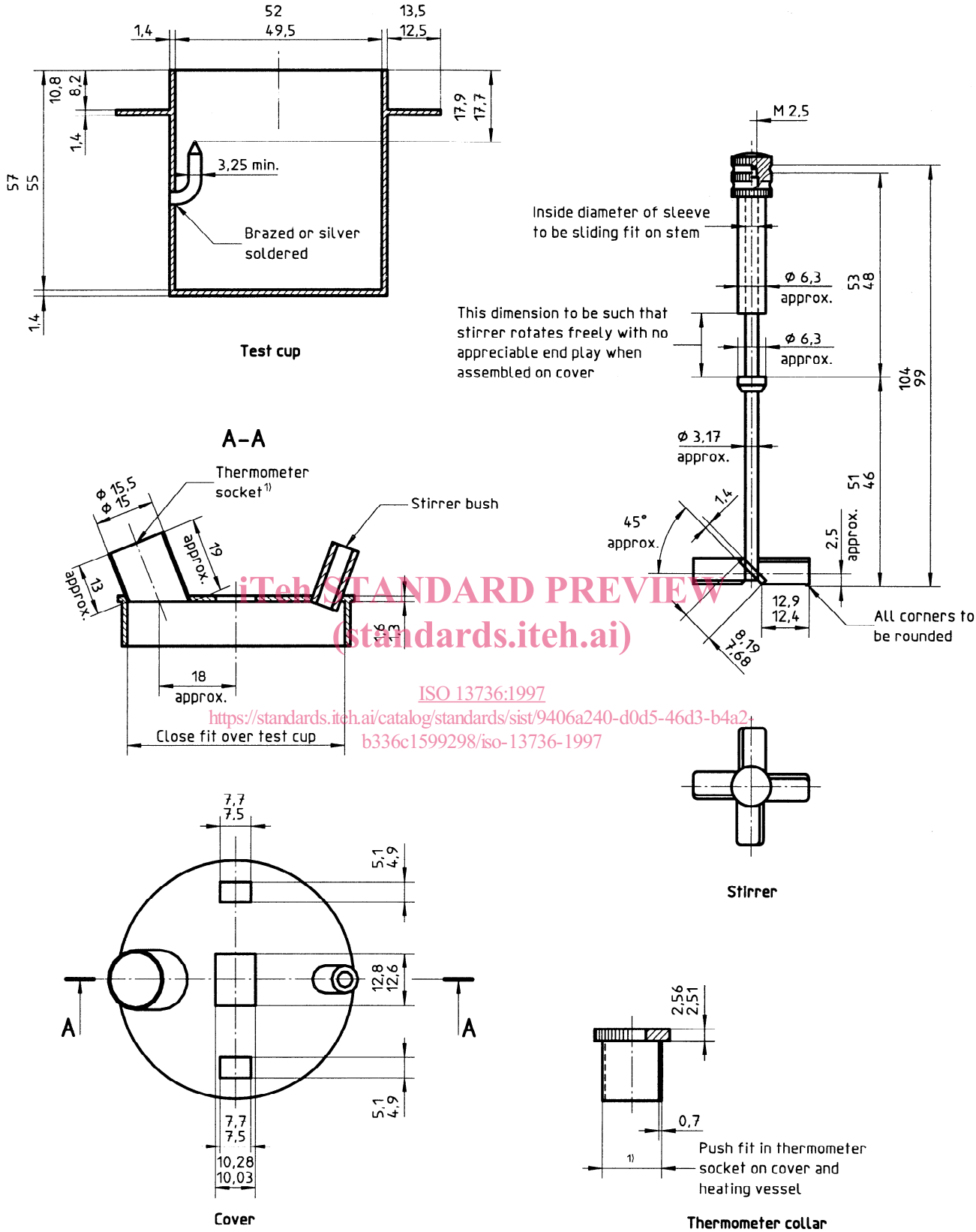
A white bead made of suitable material, the dimensions of which represent the size of test flame to be used, shall be mounted in a visible position on the cover.

A.3 Stirrer

This shall be made of brass and conform to the form and dimensions given in figure A.1.

It shall consist of a round stem having four blades or vanes silver soldered in place at one end. The blades of the stirrer shall be set so that the liquid is thrust in a downward direction when the stirrer is rotated clockwise.

Dimensions in millimetres



NOTE — All items of the apparatus shown are made of brass.

1) It is recommended that in order to achieve interchangeability the internal diameter of the thermometer socket should be between 15,235 and 15,253 and that the external diameter of the thermometer collar be between 15,222 and 15,232.

Figure A.1 — Abel flash point apparatus — Test cup, cover, stirrer and thermometer collar