

มาตรฐานผลิตภัณฑ์อุตสาหกรรม

THAI INDUSTRIAL STANDARD

มอก. 1798— 2548

ISO 2719 : 2002

การทดสอบหาจุดวาบไฟ – วิธีถ้วยปิดเพนสกี – มาร์เทนส์

DETERMINATION OF FLASH POINT – PENSKEY – MARTENS CLOSED CUP
METHOD

สำนักงานมาตรฐานผลิตภัณฑ์อุตสาหกรรม

กระทรวงอุตสาหกรรม

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มาตรฐานผลิตภัณฑ์อุตสาหกรรม
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มอก. 1798 – 2548

สำนักงานมาตรฐานผลิตภัณฑ์อุตสาหกรรม
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ประกาศในราชกิจจานุเบกษา ฉบับประกาศและงานทั่วไป เล่ม 122 ตอนที่ 117ง
วันที่ 15 ธันวาคม พุทธศักราช 2548

เพื่อให้การกำหนดหาจุดวาบไฟของหน่วยทดสอบ ผู้ทำ และผู้ที่เกี่ยวข้องเป็นมาตรฐานเดียวกัน จึงกำหนดมาตรฐานผลิตภัณฑ์อุตสาหกรรม การกำหนดหาจุดวาบไฟ – วิธีถ้วยปิดเพนสกี – มาร์เทนส์ ขึ้น

กระทรวงอุตสาหกรรมเคยประกาศใช้มาตรฐานผลิตภัณฑ์อุตสาหกรรมนี้มาแล้ว โดยรับมาตรฐาน ISO 2719 ฉบับปี 1988 มาใช้ในระดับเหมือนกันทุกประการ (identical) ทั้งเนื้อหาและรูปแบบ เป็นมาตรฐานเลขที่ มอก. 1798-2542 โดยประกาศในราชกิจจานุเบกษา ฉบับประกาศทั่วไป เล่ม 118 ตอนที่ 102ง วันที่ 20 ธันวาคม พุทธศักราช 2544 เนื่องจาก ISO ได้แก้ไขปรับปรุงมาตรฐาน ISO 2719 : 1996 เป็น ISO 2719 : 2002 จึงได้ยกเลิกมาตรฐานฉบับเดิมข้างต้นและกำหนดมาตรฐานเลขที่ มอก.1798-2548 ขึ้นใหม่ โดยรับ ISO 2719 : 2002 Determination of flash point – Pensky – Martens closed cup method มาใช้ในระดับเหมือนกันทุกประการ (identical) โดยใช้ ISO ฉบับภาษาอังกฤษเป็นหลัก

มาตรฐานผลิตภัณฑ์อุตสาหกรรมนี้กำหนดขึ้นเพื่อให้ทันกับความต้องการของผู้ใช้ และจักได้แปลเป็นภาษาไทยในโอกาสอันสมควรหากมีข้อสงสัยโปรดติดต่อสอบถามที่สำนักงานมาตรฐานผลิตภัณฑ์อุตสาหกรรม

คณะกรรมการมาตรฐานผลิตภัณฑ์อุตสาหกรรมได้พิจารณามาตรฐานนี้แล้ว เห็นสมควรเสนอรัฐมนตรีประกาศตาม มาตรา 15 แห่งพระราชบัญญัติมาตรฐานผลิตภัณฑ์อุตสาหกรรม พ.ศ. 2511



ประกาศกระทรวงอุตสาหกรรม

ฉบับที่ 3401 (พ.ศ. 2548)

ออกตามความในพระราชบัญญัติมาตรฐานผลิตภัณฑ์อุตสาหกรรม

พ.ศ. 2511

เรื่อง ยกเลิกมาตรฐานผลิตภัณฑ์อุตสาหกรรม

ผลิตภัณฑ์ปิโตรเลียมและสารหล่อลื่น – วิธีทดสอบจุดวาบไฟ – วิธีถ้วยปิดเพนสกี – มาร์เทนส์

และกำหนดมาตรฐานผลิตภัณฑ์อุตสาหกรรม

การทดสอบหาจุดวาบไฟ – วิธีถ้วยปิดเพนสกี – มาร์เทนส์

โดยที่เป็นการสมควรปรับปรุงมาตรฐานผลิตภัณฑ์อุตสาหกรรม ผลิตภัณฑ์ปิโตรเลียมและสารหล่อลื่น – วิธีทดสอบจุดวาบไฟ – วิธีถ้วยปิดเพนสกี – มาร์เทนส์ มาตรฐานเลขที่ มอก.1798-2542

อาศัยอำนาจตามความในมาตรา 15 แห่งพระราชบัญญัติมาตรฐานผลิตภัณฑ์อุตสาหกรรม พ.ศ. 2511 รัฐมนตรีว่าการกระทรวงอุตสาหกรรม ออกประกาศยกเลิกประกาศกระทรวงอุตสาหกรรมฉบับที่ 2951 (พ.ศ. 2544) ออกตามความในพระราชบัญญัติมาตรฐานผลิตภัณฑ์อุตสาหกรรม พ.ศ. 2511 เรื่อง กำหนดมาตรฐานผลิตภัณฑ์อุตสาหกรรมผลิตภัณฑ์ปิโตรเลียมและสารหล่อลื่น – วิธีทดสอบจุดวาบไฟ – วิธีถ้วยปิดเพนสกี – มาร์เทนส์ ลงวันที่ 14 กันยายน พ.ศ. 2544 และออกประกาศกำหนดมาตรฐานผลิตภัณฑ์อุตสาหกรรม การทดสอบหาจุดวาบไฟ – วิธีถ้วยปิดเพนสกี – มาร์เทนส์ มาตรฐานเลขที่ มอก. 1798-2548 ขึ้นใหม่ ดังมีรายการละเอียดต่อท้ายประกาศนี้

ทั้งนี้ ตั้งแต่วันที่ประกาศในราชกิจจานุเบกษาเป็นต้นไป

ประกาศ ณ วันที่ 31 สิงหาคม พ.ศ. 2548

นายสุริยะ จึงรุ่งเรืองกิจ

รัฐมนตรีว่าการกระทรวงอุตสาหกรรม

มาตรฐานผลิตภัณฑ์อุตสาหกรรม

การทดสอบหาจุดวาบไฟ – วิธีถ้วยปิดเพนสกี

– มาร์เทนส์

บทนำ

มาตรฐานผลิตภัณฑ์อุตสาหกรรมนี้กำหนดขึ้นโดยรับ ISO 2719 : 2002 Determination of flash point – Pensky – Martens closed cup method มาใช้ในระดับเหมือนกันทุกประการ (identical) โดยใช้ ISO ฉบับภาษาอังกฤษเป็นหลัก

ขอบข่าย

มาตรฐานผลิตภัณฑ์อุตสาหกรรมนี้กำหนดวิธี A และวิธี B โดยใช้อุปกรณ์ถ้วยปิดเพนสกี-มาร์เทนส์ในการหาจุดวาบไฟของของเหลวที่เผาได้ ของเหลวกับสารแขวนลอย ของเหลวที่มีแนวโน้มที่จะเป็นฟิล์มที่ผิวภายใต้ภาวะทดสอบ และของเหลวอื่นๆ วิธีนี้เหมาะสำหรับของเหลวที่มีจุดวาบไฟมากกว่า 40 องศาเซลเซียส

จุดวาบไฟและจุดติดไฟของผลิตภัณฑ์ปิโตรเลียมอาจหาจากวิธีถ้วยเปิดตามที่กำหนดใน ISO 2592 : 1973 Petroleum products – Determination of flash and fire points – Cleveland open cup method

จุดวาบไฟของสีและวาร์นิชและน้ำมันชักแห้งอาจหาจากวิธีที่กำหนดใน ISO 1523 : 1983 Paint varnishes, petroleum and related products – Determination of flashpoint – Closed cup equilibrium method

วิธี A ใช้สำหรับหาจุดวาบไฟของสีและวาร์นิชที่ไม่เกิดฟิล์มที่ผิวน้ำมันเครื่องที่ยังไม่ผ่านการใช้ และผลิตภัณฑ์ปิโตรเลียมอื่นๆ ที่ไม่ได้กล่าวถึงในวิธี B

วิธี B ใช้สำหรับหาจุดวาบไฟของน้ำมันก๊าด หอย งามะตอยเหลว น้ำมันเครื่องที่ผ่านการใช้แล้ว ของเหลวที่มีแนวโน้มที่จะเป็นฟิล์มที่ผิว ของเหลวที่มีของแข็งแขวนลอย และวัสดุที่มีความหนืดสูง เช่น สารละลายพอลิเมอร์และสารยึดติด วิธีทดสอบนี้ไม่ครอบคลุมสีที่มีน้ำผสมหรือของเหลวที่ปนเปื้อนด้วยวัสดุที่ระเหยง่าย

เอกสารอ้างอิง

ISO 1513 : 1992, Paints and varnishes – Examination and preparation of samples for testing

ISO 3170 : –¹⁾, Petroleum liquids – Manual sampling

ISO 3171 : 1988, Petroleum liquids – Automatic pipeline sampling

ISO 15528 : 2000, Paints, varnishes and raw materials for paints and varnishes – Sampling

หมายเหตุ ¹⁾ Tobe published. (Revision of ISO 3170 : 1988)

บทนิยาม

ความหมายของคำที่ใช้ในมาตรฐานผลิตภัณฑ์อุตสาหกรรมนี้ให้เป็นไปตามที่ระบุไว้ใน ISO 2719 : 2002 ข้อ 3

หลักการ

กำหนดให้ใส่ตัวอย่างลงในถ้วยของเครื่องมือเพนสกี – มาร์เทนส์ แล้วให้ความร้อนโดยตรงที่ถ้วย โดยเพิ่มอุณหภูมิอย่างคงที่พร้อมกับคนอย่างต่อเนื่อง จุติวบไฟเป็นอุณหภูมิต่ำสุดที่ไอระเหยบนผิวหน้าของตัวอย่างเกิดการติดไฟที่ความดันบรรยากาศปกติ แล้วคำนวณเป็นอุณหภูมิที่ความดันบรรยากาศมาตรฐาน

สารเคมีและวัสดุ

สารเคมีและวัสดุที่ใช้ในมาตรฐานผลิตภัณฑ์อุตสาหกรรมนี้ให้เป็นไปตามที่ระบุไว้ในข้อ ISO 2719 : 2002 ข้อ 5

เครื่องมือ

เครื่องมือที่ใช้ในมาตรฐานผลิตภัณฑ์อุตสาหกรรมนี้ให้เป็นไปตามที่ระบุไว้ใน ISO 2719 : 2002 ข้อ 6

การเตรียมเครื่องมือ

การเตรียมเครื่องมือในมาตรฐานผลิตภัณฑ์อุตสาหกรรมนี้ให้เป็นไปตามที่ระบุไว้ใน ISO 2719 : 2002 ข้อ 7

การชักตัวอย่าง

การชักตัวอย่างที่ใช้ในมาตรฐานผลิตภัณฑ์อุตสาหกรรมนี้ให้เป็นไปตามที่ระบุไว้ใน ISO 2719 : 2002 ข้อ 8

การดำเนินการเก็บตัวอย่าง

การดำเนินการเก็บตัวอย่างในมาตรฐานผลิตภัณฑ์อุตสาหกรรมนี้ให้เป็นไปตามที่ระบุไว้ในข้อ ISO 2719 : 2002 ข้อ 9

การทดสอบ

การทดสอบในมาตรฐานผลิตภัณฑ์อุตสาหกรรมนี้ให้เป็นไปตามที่ระบุไว้ใน ISO 2719 : 2002 ข้อ 10

การคำนวณ

การคำนวณในมาตรฐานผลิตภัณฑ์อุตสาหกรรมนี้ให้เป็นไปตามที่ระบุไว้ในข้อ ISO 2719 : 2002 ข้อ 11

การแสดงผลลัพธ์

การแสดงผลลัพธ์ในมาตรฐานผลิตภัณฑ์อุตสาหกรรมนี้ให้เป็นไปตามที่ระบุไว้ใน ISO 2719 : 2002 ข้อ 12

ความเที่ยง

ความเที่ยงในมาตรฐานผลิตภัณฑ์อุตสาหกรรมนี้ให้เป็นไปตามที่ระบุไว้ใน ISO 2719 : 2002 ข้อ 13

การรายงานผลทดสอบ

การรายงานผลทดสอบในมาตรฐานผลิตภัณฑ์อุตสาหกรรมนี้ให้เป็นไปตามที่ระบุไว้ใน ISO 2719 : 2002 ข้อ 14

Determination of flash point — Pensky-Martens closed 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 describes two procedures, A and B, using the Pensky-Martens closed cup tester, for determining the flash point of combustible liquids, liquids with suspended solids, liquids that tend to form a surface film under the test conditions and other liquids. It is applicable for liquids with a flash point above 40 °C.

NOTE 1 Although technically kerosines with a flash point above 40 °C may be tested using this International Standard, it is standard practice to test kerosines according to ISO 13736^[8]. Similarly, unused lubricating oils are normally tested according to ISO 2592^[5].

Procedure A is used for the determination of the flash point of paints and varnishes that do not form a surface film, unused lubricating oils and other petroleum products not covered by Procedure B.

Procedure B is used for the determination of the flash point of residual fuel oils, cutback bitumens, used lubricating oils, liquids that tend to form a surface film, liquids with suspensions of solids and highly viscous materials such as polymeric solutions and adhesives.

NOTE 2 For the comparison of the flash points of used and unused lubricating oils, such as in a lubricant monitoring scheme, used lubricating oils may be tested using Procedure A. However, the precision data for these materials is only valid for Procedure B.

This International Standard is not applicable to water-borne paints or liquids contaminated by traces of highly volatile materials.

NOTE 3 Water-borne paints may be tested using ISO 3679^[6]. Liquids contaminated by traces of highly volatile materials may be tested using ISO 1523^[4] or ISO 3679.

NOTE 4 Precision data is only valid for the flash point ranges given in clause 13.

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.

3 Term and definition

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

3.1

flash point

lowest temperature of the test portion, corrected to a barometric pressure of 101,3 kPa, at which the 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

4 Principle

The test portion is placed in the test cup of a Pensky-Martens apparatus and heated to give a constant temperature increase with continuous stirring. An ignition source is directed through an opening in the test cup lid at regular temperature intervals with simultaneous interruption of stirring. The lowest temperature at which the application of the ignition source causes the vapour of the test portion to ignite and propagate over the surface of the liquid is recorded as the flash point at the ambient barometric pressure. This temperature is corrected to standard atmospheric pressure using an equation.

5 Chemicals and materials

5.1 Cleaning solvent, for the 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 annex A.

6 Apparatus

6.1 Flash point apparatus, Pensky-Martens closed cup test apparatus as specified in annex B.

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 cover assembly conform to the dimensional and mechanical requirements as specified in annex B. If automated testers are used, the user shall ensure that all of the manufacturer's instructions for adjusting and operating the instrument are followed.

NOTE Under certain circumstances, the use of electric ignition sources may give different results to those obtained when using a flame ignition source. In addition, electric ignition sources may give variable results.

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

6.2 Thermometers, low, medium and high range, conforming to annex C. The initial choice of thermometer shall be based on the expected flash point.

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 C.

6.3 Barometer, accurate to 0,1 kPa. Barometers precorrected to give sea-level readings, such as those used at weather stations and airports, shall not be used.

6.4 Heating bath or oven, for warming the sample if required, capable of controlling the temperature to $\pm 5^{\circ}\text{C}$. The oven shall be ventilated and constructed in such a way that it will not cause ignition of any flammable vapours that may be produced when the sample is heated.

It is recommended that the oven is of explosion-protected design.

7 Apparatus preparation

7.1 Location of the apparatus

Support the apparatus (6.1) on a level and steady surface in a draught-free position (see the notes below).

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 around the test cup during the test.

7.2 Cleaning the test cup

Wash the test cup, cover and its accessories 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 to ensure complete removal of the solvent used.

7.3 Apparatus assembly

Examine the test cup, the cover and other parts to ensure that they are free from signs of damage and deposits. Assemble the apparatus in accordance with annex B.

7.4 Apparatus verification

7.4.1 Verify the correct functioning of the apparatus at least once a year by testing a certified reference material (CRM) (5.2) using Procedure A. 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 Table 3).

It is recommended that more frequent verification checks be made using secondary working standards (SWSs) (5.2).

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

7.4.2 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 15528, ISO 3170, ISO 3171 or an equivalent National Standard.

8.2 Place samples in tightly sealed containers, appropriate to the material being sampled. 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 that minimize vapour loss and pressure build-up. Avoid storing the samples at temperatures in excess of 30°C .

9 Sample handling

9.1 Petroleum products

9.1.1 Subsampling

Subsample at a temperature at least 28 °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 note to 10.1).

9.1.2 Samples containing undissolved water

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

Flash point results can be affected by the presence of water. For certain fuel oils and lubricants, it may not always be possible to decant the sample from the free water. In such cases, the water should be separated from the aliquot physically, prior to mixing, or, if this is not possible, the material should be tested in accordance with ISO 3679^[6].

9.1.3 Samples that are liquid at ambient temperature

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

9.1.4 Samples that are semi-solid or solid at ambient temperature

Heat the sample in its container in a heating bath or oven (6.4) to a temperature of 30 °C ± 5 °C, or a higher temperature not exceeding 28 °C below the expected flash point, whichever is the greater, for 30 min. If after 30 min the sample is still not completely liquefied, extend the preheating for further 30 min periods as required. Avoid overheating the sample, as this could lead to the loss of volatile components. After gentle agitation, proceed in accordance with clause 10.

9.2 Paints and varnishes

Prepare the samples in accordance with ISO 1513.

10 Procedure

10.1 General

NOTE Results of flash point determinations may be affected if the sample volume falls below 50 % of the container capacity.

Care should be taken when testing samples of residual fuel oil which contain significant amounts of water, as heating these samples may cause them to foam and eject from the test cup.

10.2 Procedure A

10.2.1 Using a barometer (6.3), record the ambient barometric pressure in the vicinity of the apparatus at the time of test.

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

10.2.2 Fill the test cup (see 7.3) with the test portion to the level indicated by the filling mark. Place the lid on the test cup and put it in the heating chamber. Ensure that the locating or locking device is properly engaged and insert the

thermometer (6.2). Light the test flame and adjust to a diameter of 3 mm to 4 mm, or switch on the alternative ignition source. Light the heater flame or switch on the electric heater and supply heat at such a rate that the temperature of the test portion as indicated by the thermometer increases at 5 °C/min to 6 °C/min, and maintain this heating rate throughout the test. Stir the test portion at a rate of 90 r/min to 120 r/min, stirring in a downward direction.

10.2.3 When the test portion is expected to have a flash point of 110 °C or below, make the first application of the ignition source when the temperature of the test portion is 23 °C ± 5 °C below the expected flash point, and thereafter at 1 °C temperature intervals. Cease stirring and apply the ignition source by operating the mechanism on the cover, which controls the shutter and ignition source, so that the source is lowered into the vapour space of the test cup in 0,5 s, left in its lowered position for 1 s, and quickly raised to its high position.

10.2.4 When the test portion is expected to have a flash point of above 110 °C, make the first application of the ignition source when the temperature of the test portion is 23 °C ± 5 °C below the expected flash point, and thereafter at temperatures which are a multiple of 2 °C. Cease stirring and apply the ignition source by operating the mechanism on the cover, which controls the shutter and ignition source, so that the source is lowered into the vapour space of the test cup in 0,5 s, left in its lowered position for 1 s, and quickly raised to its high position.

10.2.5 When testing a material of unknown flash point, conduct a preliminary test at a suitable starting temperature. Make the first ignition-source application at 5 °C above the starting temperature and follow the procedure given in 10.2.3 or 10.2.4, as applicable.

10.2.6 Record, as the observed flash point, the temperature of the test portion read on the thermometer at the time when the ignition-source application causes a distinct flash in the interior of the test cup. Do not confuse the true flash point with the bluish halo that sometimes surrounds the ignition source at applications preceding the actual flash point.

10.2.7 When the temperature at which the flash point is observed is less than 18 °C, or greater than 28 °C, from the temperature of the first application of the ignition source, the result is not valid. Repeat the test using a fresh test portion, adjusting the temperature of the first application of the ignition source until a valid determination is obtained, that is where the flash point is 18 °C to 28 °C above the temperature of the first application of the ignition source.

10.3 Procedure B

10.3.1 Using a barometer (6.3), record the ambient barometric pressure in the vicinity of the apparatus at the time of test (see the note to 10.2.1).

10.3.2 Fill the test cup (see 7.3) with the test portion to the level indicated by the filling mark. Place the lid on the test cup and put it in the heating chamber. Ensure that the locating or locking device is properly engaged and insert the thermometer (6.2). Light the test flame and adjust to a diameter of 3 mm to 4 mm, or switch on the alternative ignition source. Light the heater flame or switch on the electric heater and supply heat at such a rate that the temperature of the test portion as indicated by the thermometer increases at 1,0 °C/min to 1,5 °C/min, and maintain this heating rate throughout the test. Stir the test portion at a rate of 250 r/min ± 10 r/min, stirring in a downward direction.

10.3.3 With the exception of the requirements given in 10.3.2 for rate of heating and stirring, proceed in accordance with 10.2.3 to 10.2.7.

11 Calculation

11.1 Conversion of barometric pressure reading

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

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

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

$$\text{Reading in mmHg} \times 0,1333 = \text{kPa}$$

11.2 Correction of observed flash point to standard atmospheric pressure

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

$$T_c = T_o + 0,25(101,3 - p)$$

where

T_o is the flash 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.

12 Expression of results

Report the flash point, corrected to standard atmospheric pressure, rounded to the nearest 0,5 °C.

13 Precision

13.1 General

The precision, as determined by statistical examination of interlaboratory test results using ISO 4259^[7], is given in 13.2 and 13.3.

13.2 Repeatability, r

The difference between two test results obtained by the same operator with the same apparatus under constant operating conditions on identical test material would, in the long run, in the normal and correct operation of the test method, exceed the values given in Tables 1 and 2 in only one case in twenty.

Table 1 — Repeatability values for Procedure A

Material	Flash point range °C	Repeatability, r °C
Paints and varnishes	—	1,5
Distillates and unused lubricating oils	40 to 250	0,029 X^a

^a X is the average of the test results being compared.

Table 2 — Repeatability values for Procedure B

Material	Flash point range °C	Repeatability, r °C
Residual fuels and cutback bitumen	40 to 110	2,0
Used lubricating oils	170 to 210	5 ^a
Liquids that tend to form a surface film, liquids with suspensions of solids, highly viscous materials	—	5,0 ^b

^a Data generated from one sample of used diesel fuel engine oil by 20 laboratories.

^b The precision data was determined by ASTM Committee D-1.

13.3 Reproducibility, R

The difference between two single and independent test results obtained by different operators working in different laboratories on identical test material would, in the long run, in the normal and correct operation of the test method, exceed the values given in Tables 3 and 4 in only one case in twenty.

Table 3 — Reproducibility values for Procedure A

Material	Flash point range °C	Reproducibility, R °C
Paints and varnishes	—	—
Distillates and unused lubricating oils	40 to 250	0,071 X^a

^a X is the average of the test results being compared.

Table 4 — Reproducibility values for Procedure B

Material	Flash point range °C	Reproducibility, R °C
Residual fuels and cutback bitumen	40 to 110	6,0
Used lubricating oils	170 to 210	16 ^a
Liquids that tend to form a surface film, liquids with suspensions of solids, highly viscous materials	—	10,0 ^b

^a Data generated from one sample of used diesel fuel engine oil by 20 laboratories.

^b The precision data was determined by ASTM Committee D-1.

14 Test report

The test report shall contain at least the following information:

- a reference to this International Standard and the procedure used;
- the type and complete identification of the sample tested;
- the preheating temperature and the preheating time, if applicable (see 9.1.4);
- the ambient barometric pressure in the vicinity of the apparatus (see 10.2.1 and 10.3.1);
- the result of the test (see clause 12);
- any deviation, by agreement or otherwise, from the procedure specified;
- the date of the test.

Annex A (informative)

Apparatus verification

A.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^[2] and ISO Guide 35^[3], or an in-house reference material/SWS prepared in accordance with one of the procedures given in A.2.2. The performance of the apparatus should be assessed in accordance with the guidance given in ISO Guide 33^[1] and ISO 4259^[7].

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

A.2 Verification check standards

A.2.1 Certified reference material (CRM), comprising of 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.

A.2.2 Secondary working standard (SWS), comprising of a stable petroleum product or a single hydrocarbon or other stable substance with a flash point determined either by

- 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
- conducting an interlaboratory method-specific test program 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.

A.3 Procedure

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

Table A.1 — Approximate values of the closed cup flash points of hydrocarbons

Hydrocarbon	Nominal flash point °C
Decane	53
Undecane	68
Dodecane	84
Tetradecane	109
Hexadecane	134

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.

A.3.2 For new apparatus, and at least once a year for working apparatus, conduct a verification check using a CRM (A.2.1) tested in accordance with 10.2.

A.3.3 For intermediate verification, conduct a verification check using a SWS (A.2.2) tested in accordance with 10.2.

A.3.4 Correct the result for barometric pressure in accordance with 11.2. Note the corrected result, to the nearest 0,1 °C, in a permanent record.

A.4 Evaluation of test result

A.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 equations given in A.4.1.1 and A.4.1.2, it is assumed that reproducibility has been estimated in accordance with ISO 4259^[7] and that certified value of the CRM, or the assigned value of the SWS, has been obtained by the procedures set out in ISO Guide 35^[3] 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 .

A.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.

A.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 or replicate tests carried out on the CRM or SWS.

A.4.2 If the test result conforms to the tolerance requirements, record this fact.

A.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.

A.4.4 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.

Annex B (normative)

Pensky-Martens closed cup test apparatus

B.1 General

This annex describes the manual, gas/electrically heated, flame-ignition-device apparatus. This shall consist of a test cup, lid assembly and heating chamber as described in B.2 to B.4. A typical assembly of the apparatus, in this case gas heated, is shown in Figure B.1.

B.2 Test cup

The test cup shall be made of brass or another non-rusting metal of equivalent heat conductivity, and shall conform to the dimensional requirements shown in Figure B.2. The flange shall be equipped with devices for positioning the test cup in the heating chamber. A handle attached to the flange of the test cup is a desirable accessory, but shall not be so heavy as to tip over the empty test cup.

B.3 Cover assembly

B.3.1 The cover assembly shall comprise the following items.

B.3.2 Lid, made of brass or another non-rusting metal of equivalent heat conductivity, and with a rim projecting downward almost to the flange of the test cup, as shown in Figure B.3. The rim shall fit the outside of the test cup with a clearance not exceeding 0,36 mm on the diameter. There shall be a locating or locking device, or both, engaging with a corresponding device on the test cup. Three openings in the lid, A, B and C, are shown in Figure B.3. The upper edge of the test cup shall be in close contact with the inner face of the lid round the whole of its circumference.

B.3.3 Shutter, made of brass, approximately 2,4 mm thick, operating in the plane of the upper surface of the lid, as shown in Figure B.4. The shutter shall be shaped and mounted so that it rotates about the axis of the horizontal centre of the cover between two stops, placed so that, when in one extreme position, the openings A, B and C in the lid are completely closed, and when in the other extreme position, these openings are completely opened. The mechanism operating the shutter shall be of the spring type and constructed so that, when at rest, the shutter exactly closes the three openings. When the mechanism is operated to the other extreme, the three cover openings shall be exactly open and the tip of the flame-exposure device (see B.3.4) shall be fully depressed.

B.3.4 Flame-exposure device, having a tip with an opening 0,7 mm to 0,8 mm in diameter, as shown in Figure B.4. This tip shall be made of stainless steel or another suitable material. The flame-exposure device shall be equipped with an operating mechanism which, when the shutter is in the "open" position, depresses the tip so that the centre of the orifice is between the planes of the lower and upper surfaces of the lid at a point on a radius passing through the centre of the largest opening A (see Figure B.3).

NOTE A bead made of suitable material, of dimensions which represent the size of the test flame (3 mm to 4 mm), may be mounted in a visible position on the lid.

B.3.5 Pilot flame for automatic relighting of the exposure-flame. The tip of the pilot flame jet shall have an opening 0,7 mm to 0,8 mm in diameter.

B.3.6 Stirring device, mounted in the centre of the lid (see Figure B.4) and carrying two two-bladed metal propellers. The lower propeller shall measure approximately 38 mm from tip to tip, each of its two blades being 8 mm in width and having a pitch of 45°. The upper propeller shall measure approximately 19 mm from tip to tip, each of its two blades also being 8 mm in width and having a pitch of 45°. Both propellers shall be located on the stirrer shaft in such a manner that, when viewed from underneath the stirrer, the blades of one propeller are at 0° and 180° while the blades of the other propeller are at 90° and 270°.

NOTE The stirrer shaft may be coupled to a motor, stirring in a downward direction, by means a flexible shaft or a suitable arrangement of pulleys.

B.4 Heating chamber and top plate

Heat shall be supplied to the test cup by means of a properly designed heating chamber which is equivalent to an air bath. The heating chamber shall consist of an air bath and a top plate on which the flange of the test cup rests.

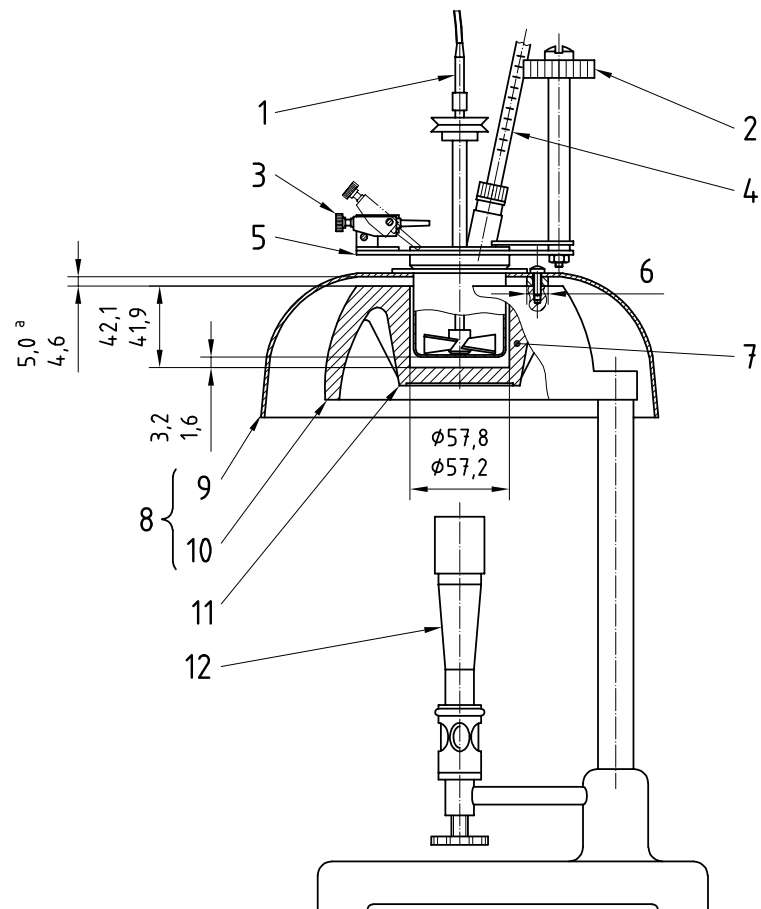
The air bath shall have a cylindrical interior and conform to the dimensional requirements shown in Figure B.1. It shall be either a flame-heated or an electrically heated metal casting, or have an internal electrical-resistance element. In either case, the air bath shall be suitable for use without deformation at the temperatures to which it will be subjected.

If the air bath is a flame-heated or electrically heated metal casting, it shall be designed so that the temperatures of the bottom and the walls are approximately the same. For this reason, it shall be not less than 6 mm thick. If flame-heated, the casting shall be designed so that the products of combustion of the flame cannot pass up and come into contact with the test cup.

If the air bath has an electrical-resistance element, it shall be constructed so that all parts of the interior surface are heated uniformly. The walls and bottom of the air bath shall be not less than 6 mm thick.

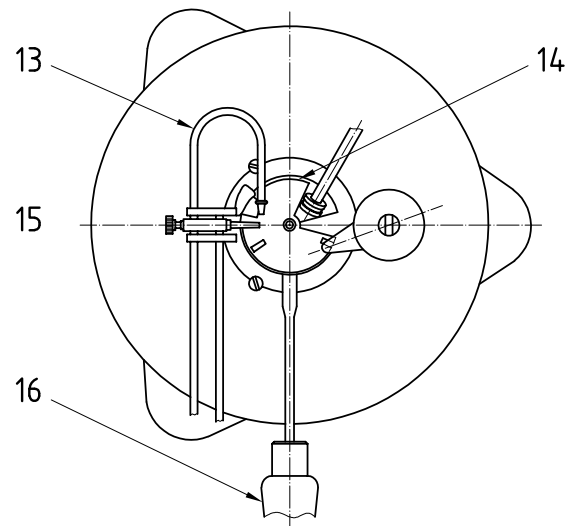
The top plate shall be made of metal and mounted with an air gap between it and the air bath. It shall be attached to the air bath by means of three screws and spacing bushings. The bushings shall be of sufficient thickness to define an air gap of $4,8 \text{ mm} \pm 0,2 \text{ mm}$, and they shall be not more than 9,5 mm in diameter.

Dimensions in millimetres



Key

- 1 Flexible shaft
- 2 Shutter-operating knob
- 3 Flame-exposure device
- 4 Thermometer
- 5 Lid
- 6 Distance piece $\varnothing 9,5$ max.
- 7 Test cup
- 8 Heating chamber
- 9 Top plate
- 10 Air bath
- 11 6,5 min. thickness over cup area, i.e. metal surrounding the cup
- 12 Heater, flame-type or electric-resistance type (flame-type shown)
- 13 Pilot
- 14 Shutter
- 15 Front
- 16 Handle (optional)

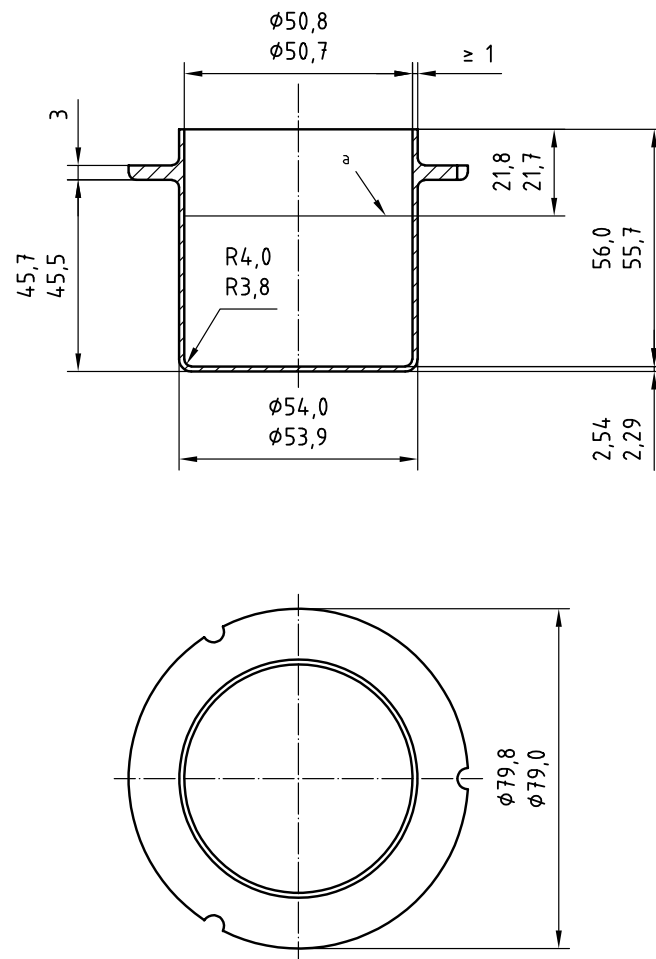


NOTE The lid assembly may be positioned either right- or left-handed.

^a Air gap.

Figure B.1 — Pensky-Martens closed cup apparatus

Dimensions in millimetres



^a Filling mark.

Figure B.2 — Test cup

Dimensions in millimetres

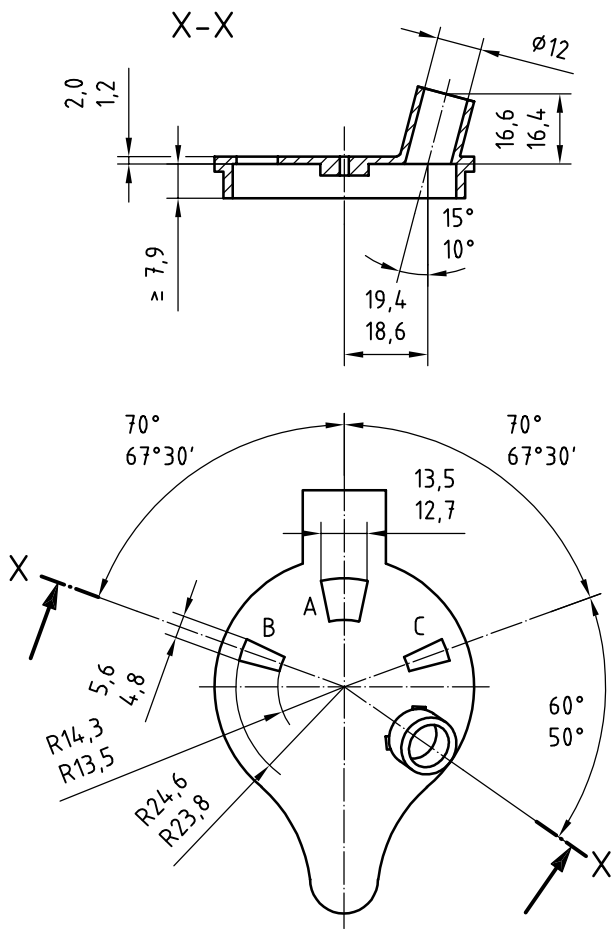
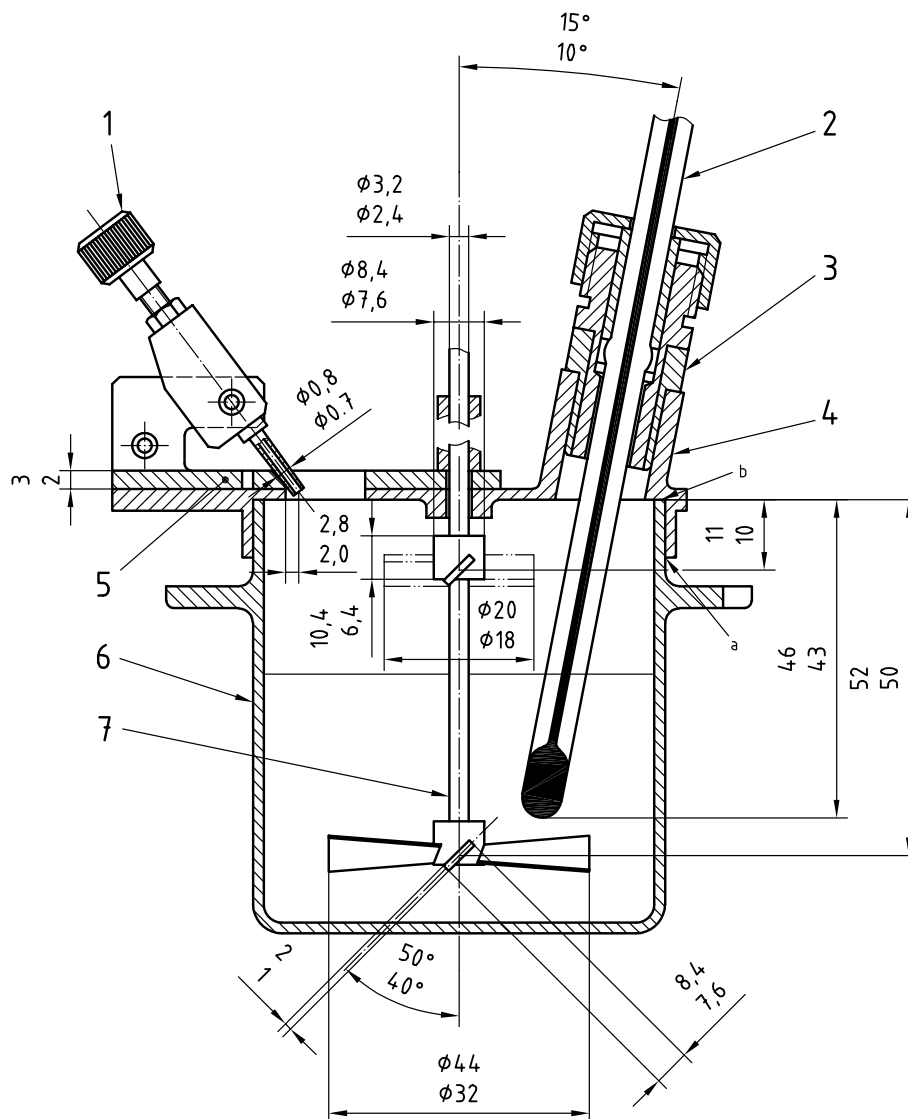


Figure B.3 — Lid

Dimensions in millimetres



Key

- 1 Flame-exposure device
- 2 Thermometer
- 3 Thermometer adaptor (see annex D)
- 4 Lid
- 5 Shutter
- 6 Test cup
- 7 Stirrer

^a Clearance 0,36 max.

^b Rim of cup shall be in contact with inner face of lid throughout its circumference.

Figure B.4 — Test cup and lid assembly

Annex C (normative)

Thermometer specifications

Table C.1 — Thermometer specifications

	Low range	Medium range	High range
Range	−5 °C to +110 °C	20 °C to 150 °C	90 °C to 370 °C
Immersion, mm	57	57	57
Graduations:			
Subdivisions	0,5 °C	1 °C	2 °C
Long lines at each	1 °C and 5 °C	5 °C	10 °C
Numbers at each	5 °C	5 °C	20 °C
Scale error, maximum	0,5 °C	1 °C	1 °C to 260 °C 2 °C over 260 °C
Expansion chamber: permits heating to	160 °C	200 °C	370 °C
Total length, mm	282 to 295	282 to 295	282 to 295
Stem diameter, mm	6,0 to 7,0	6,0 to 7,0	6,0 to 7,0
Bulb length, mm	9 to 13	9 to 13	7 to 10
Bulb diameter, mm	Not less than 5,5 and not greater than stem	Not less than 5,5 and not greater than stem	Not less than 5,5 and not greater than stem
Distance from bottom of bulb to line at	0 °C: 85 mm to 95 mm	20 °C: 85 mm to 95 mm	90 °C: 80 mm to 90 mm
Length of graduated portion, mm	140 to 175	140 to 180	145 to 180
Stem enlargement:			
Diameter, mm	7,5 to 8,5	7,5 to 8,5	7,5 to 8,5
Length, mm	2,5 to 5,0	2,5 to 5,0	2,5 to 5,0
Distance to bottom, mm	64 to 66	64 to 66	64 to 66
NOTE 1 IP 15C/ASTM 9C, IP 16C/ASTM 10C, IP 101C and ASTM 88C meet the above requirements.			
NOTE 2 See annex D for the description of an adaptor for low-range thermometers.			

Annex D (informative)

Adaptor for low-range thermometer

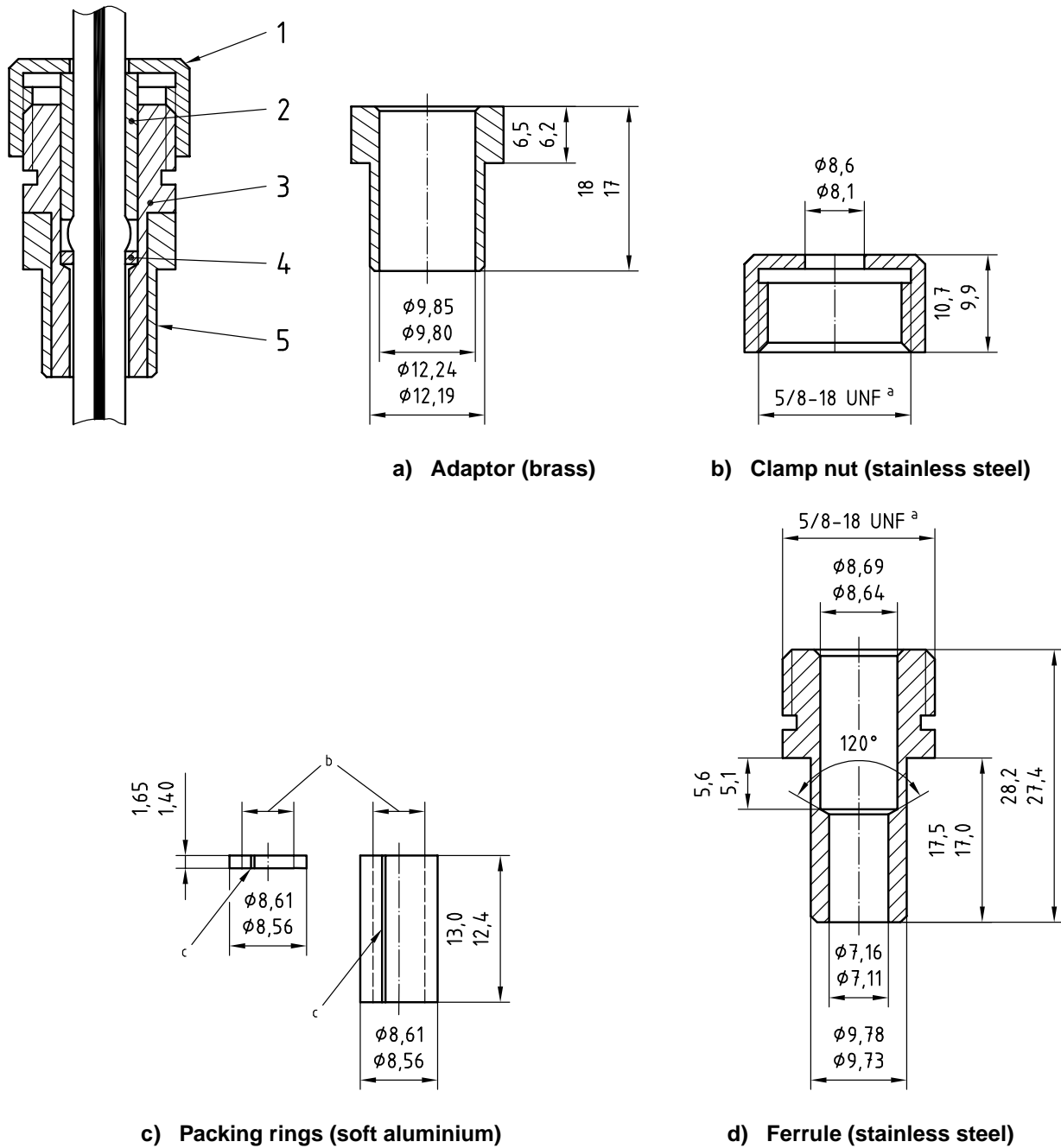
D.1 General

Low-range thermometers are sometimes fitted with a metal ferrule intended to fit the collar of the Tag flash tester (ASTM D56 ^[9]). This may be supplemented by an adaptor (see Figure D.1) for use in the larger-diameter collar of the Pensky-Martens apparatus.

D.2 Test gauge

The length of the enlargement and the distance from the bottom of the enlargement to the bottom of the bulb may be measured with the test gauge as shown in Figure D.2.

Dimensions in millimetres



Key

- 1 Clamp nut
- 2 Packing ring
- 3 Ferrule
- 4 Packing ring
- 5 Adaptor

^a Or equivalent.

^b Bores to suit thermometer stem.

^c Split.

Figure D.1 — Dimensions of thermometer adaptor, ferrule and packing ring

Dimensions in millimetres

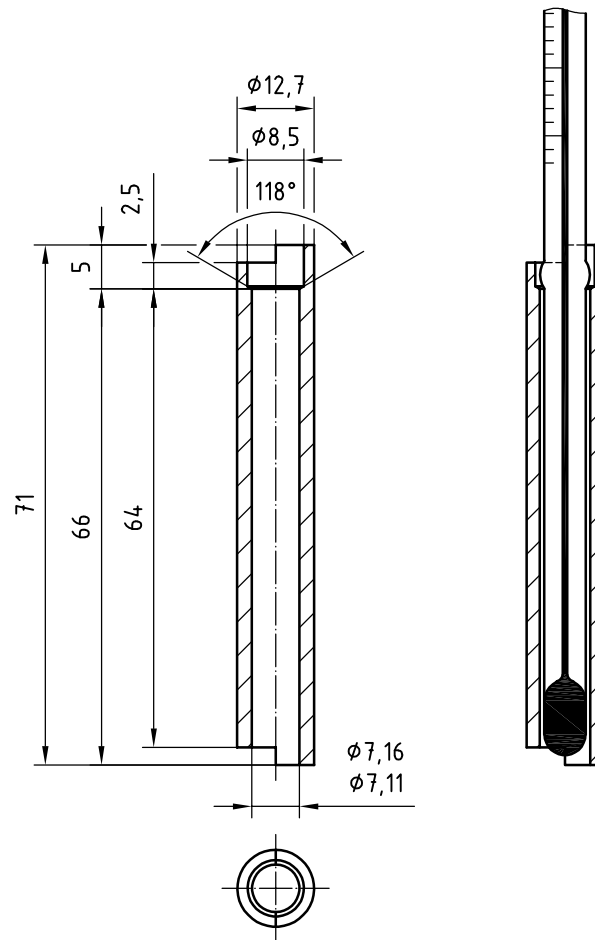


Figure D.2 — Test gauge for checking enlargements on thermometers

Bibliography

- [1] ISO Guide 33:1989, *Uses of certified reference materials*
- [2] ISO Guide 34:2000, *General requirements for the competence of reference material producers*
- [3] ISO Guide 35:1989, *Certification of reference materials — General and statistical principles*
- [4] ISO 1523:2002, *Determination of flash point — Closed cup equilibrium method*
- [5] ISO 2592:2000, *Determination of flash and fire points — Cleveland open cup method*
- [6] ISO 3679:—¹⁾, *Determination of flash point — Rapid equilibrium closed cup method*
- [7] ISO 4259:1992, *Petroleum products — Determination and application of precision data in relation to methods of test*
- [8] ISO 13736:1997, *Petroleum products and other liquids — Determination of flash point — Abel closed cup method*
- [9] ASTM D56-01, *Standard Test Method for Flash Point by Tag Closed Tester*

1) To be published. (Revision of ISO 3679:1983)