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Petroleum products — Determination of flash point — Pensky-Martens closed-cup method

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FOREWORD

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Draft International Standards adopted by the Technical Committees are circulated to the Member Bodies for approval before their acceptance as International Standards by the ISO Council.

International Standard ISO 2719 was drawn up by Technical Committee ISO/TC 28, *Petroleum products*, and circulated to the Member Bodies in July 1972.

It has been approved by the Member Bodies of the following countries :

Australia	Iran	Romania
Belgium	Ireland	South Africa, Rep. of
Canada	Israel	Spain
Chile	Mexico	Sweden
Czechoslovakia	Netherlands	Thailand
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Germany	Norway	United Kingdom
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India	Portugal	U.S.S.R.

The Member Body of the following country expressed disapproval of the document on technical grounds :

France

Printed in Switzerland

Petroleum products — Determination of flash point — Pensky-Martens closed cup method

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a method, using the Pensky-Martens closed cup apparatus, of determining the flash point of fuel oils, lubricating oils, liquids that tend to form a surface film under test conditions, and other liquids.

Open cup flash and fire points of petroleum products may be determined by the use of ISO 2592, *Petroleum products — Determination of flash and fire points — Cleveland open cup method*. Flash points of drying oils and suspensions of solids may be determined by the use of ISO 1523, *Paints and varnishes — Determination of flash point (closed cup method)*.

NOTE — The method described in this International Standard may be employed for the detection of contamination of lubricating oils by minor amounts of volatile material.

2 PRINCIPLE

The test portion is heated at a slow, constant rate with continual stirring. A small flame is directed into the cup at regular temperature intervals with simultaneous interruption of stirring. The flash point is the lowest temperature at which application of the test flame causes the vapour above the test portion to ignite.

3 APPARATUS

3.1 Pensky-Martens closed cup apparatus, as described in Annex A.

NOTE — Automatic flash point testers are available and in use which may be advantageous in the saving of testing time, permit the use of smaller samples, and have other factors which may merit their use. If automatic testers are used, the user must be sure that all 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 referee test.

3.2 Thermometers, partial immersion type, conforming to the appropriate specification in the table :

— low range for samples giving a flash point between 12 °C and 110 °C;

— high range for samples giving a flash point between 107 °C and 370 °C.

NOTE — For samples giving a flash point between 107 °C and 110 °C, either thermometer may be used.

TABLE — Thermometer specifications

	Low range	High range
Range	−7 to + 110 °C	+ 90 to + 370 °C
Immersion	57 mm	57 mm
Graduation	0,5 °C	2 °C
Longer lines at each	1 °C	10 °C
Figured at each	5 °C	20 °C
Scale error not to exceed	0,5 °C	1,5 °C
Expansion chamber permitting heating to	160 °C	370 °C
Overall length	287 ± 5 mm	287 ± 5 mm
Stem diameter	6 to 7 mm	6 to 7 mm
Bulb length	9 to 13 mm	8 to 10 mm
Bulb diameter	> stem	4,5 to 6 mm
Distance from bottom of bulb to line at	0 °C : 85 to 98 mm	110 °C : 86 to 99 mm
Distance from bottom of bulb to line at	100 °C : 221 to 237 mm	360 °C : 227 to 245 mm
Stem enlargement		
Diameter	7,5 to 8,5 mm	7,5 to 8,5 mm
Length	2,5 to 5,0 mm	2,5 to 5,0 mm
Distance to bottom	64 to 66 mm	64 to 66 mm

4 PREPARATION OF APPARATUS

Support the apparatus on a level, steady table. Unless tests are made in a draught-free room or compartment, it is good practice, but not obligatory, to surround the tester on three sides with a shield, each section of which is about 46 cm wide and 61 cm high.

5 PREPARATION OF SAMPLE

5.1 Samples of very viscous materials may be warmed until they are reasonably fluid before they are tested. However, no sample should be heated more than is absolutely necessary. A sample shall never be heated above a temperature 17 °C below its expected flash point.

5.2 Samples containing dissolved or free water may be dehydrated with calcium chloride or by filtering through a qualitative filter paper or a loose plug of dry absorbent cotton. Warming the sample is permitted, but it shall not be heated for prolonged periods or above a temperature 17 °C below its expected flash point.

NOTE — If it is suspected that the sample contains volatile contaminants, the treatment described in 5.1 and 5.2 shall be omitted.

6 PROCEDURE

6.1 Thoroughly clean and dry all parts of the cup and its accessories before starting the test, being sure to remove any solvent which has been used to clean the apparatus. Fill the cup with the sample to be tested to the level indicated by the filling mark. Place the lid on the cup and set the latter in the stove. Ensure that the locating or locking device is properly engaged. Insert the thermometer. Light the test flame and adjust it to a diameter of approximately 4 mm. Heat at such a rate that the temperature as indicated by the thermometer increases 5 to 6 °C/min. Turn the stirrer 90 to 120 rev/min, stirring in a downward direction.

6.2 If the sample is expected to have a flash point of 104 °C or below, apply the test flame when the temperature of the sample is not higher than 17 °C below the flash point, and thereafter at a temperature reading that is a multiple of 1 °C. Apply the test flame by operating the mechanism on the cover which controls the shutter and test flame burner so that the flame is lowered into the vapour space of the cup in 0,5 s, left in its lowered position for 1 s, and quickly raised to its high position. Do not stir the sample while applying the test flame.

6.3 If the sample is expected to have a flash point above 104 °C, apply the test flame in the manner just described at each temperature that is a multiple of 2 °C, beginning at a temperature not higher than 17 °C below the flash point.

6.4 Record as the flash point the temperature read on the thermometer at the time the test flame application causes a distinct flash in the interior of the cup. Do not confuse the true flash point with the bluish halo that sometimes surrounds the test flame at applications preceding the one that causes the actual flash.

7 CORRECTION FOR BAROMETRIC PRESSURE

Observe and record the barometric pressure. Calculate the flash point corrected to standard barometric pressure of 1,013 bar by adding algebraically to the observed temperature the correction given in degrees Celsius by the formula :

$$\frac{1{,}013 - p}{0{,}033} \times 0{,}9$$

where p is the barometric pressure expressed in bars.

Round off the values thus obtained to the nearest whole number.

8 PRECISION

The following criteria shall be used for judging the acceptability of results (95 % probability).

8.1 Repeatability

Duplicate results by the same operator using the same apparatus shall be considered suspect if they differ by more than the following amounts :

Flash point range	Repeatability
104 °C and below	2 °C
above 104 °C	5,5 °C

8.2 Reproducibility

The results submitted by each of two laboratories shall be considered suspect if they differ by more than the following amounts :

Flash point range	Reproducibility
104 °C and below	3,5 °C
above 104 °C	8 °C

NOTE — The precision limits shown are not derived from a correlation programme conducted on degrees Celsius but are calculated from the programme based on degrees Fahrenheit.

9 TEST REPORT

Report the corrected flash point as the Pensky-Martens closed cup flash point, reference being made to this International Standard.

ANNEX A

PENSKY-MARTENS CLOSED CUP APPARATUS

A typical assembly of the apparatus, gas heated, is shown in Figure 1. The apparatus shall consist of a test cup, cover assembly, and stove conforming to the following requirements :

A.1 Test cup, of brass or other non-rusting metal of equivalent heat conductivity, and conforming to the dimensional requirements shown in Figure 2. The flange shall be equipped with devices for locating the position of the cup in the stove. A handle attached to the flange of the cup is a desirable accessory, but shall not be so heavy as to tip over the empty cup.

A.2 Cover assembly, comprising :

A.2.1 Cover (see Figure 3), of brass or other non-rusting metal of equivalent heat conductivity, and with a rim projecting downward almost to the flange of the cup. The rim shall fit the outside of the 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 cup. The four openings in the cover, A, B, C, and D, are shown in Figure 3. The upper edge of the cup shall be in close contact with the inner face of the cover throughout its circumference.

A.2.2 Shutter, of brass (see Figure 4), approximately 2,4 mm thick, operating on the plane of the upper surface of the cover. The shutter shall be so shaped and mounted that it rotates on the axis of the horizontal centre of the cover between two stops, so placed that when in one extreme position, the openings A, B, and C in the cover 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 operated to the other extreme, the three cover openings shall be exactly open and the tip of the flame-exposure device shall be fully depressed.

A.2.3 Flame-exposure device (see Figure 4), having a tip with an opening 0,69 to 0,79 mm in diameter. This tip shall be made preferably of stainless steel, although other suitable metals may be used. 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 cover at a point on a radius passing through the centre of the larger opening A (see Figure 3).

A.2.4 Pilot flame for automatic relighting of the exposure flame. A bead approximately 4 mm in diameter may be mounted on the cover so that the size of the test flame can be regulated by comparison. The tip of the pilot flame shall have an opening the same size as the tip of the flame exposure device, 0,69 to 0,79 mm in diameter.

A.2.5 Stirring device (see Figure 4), mounted in the centre of the cover and carrying two 2-bladed metal propellers. A stirrer shaft may be coupled to the motor by a flexible shaft or a suitable arrangement of pulleys.

A.3 Stove. Heat shall be supplied to the cup by means of a properly designed stove which is equivalent to an air bath. The stove shall consist of an air bath and a top plate on which the flange of the cup rests.

A.3.1 Air bath, with a cylindrical interior and conforming to the dimensional requirements shown in Figure 1. The air bath may be either a flame- or electrically-heated metal casting (see Note 1), or an electric-resistance element (see Note 2). In either case, the air bath must be suitable for use without deformation at the temperatures to which it will be subjected.

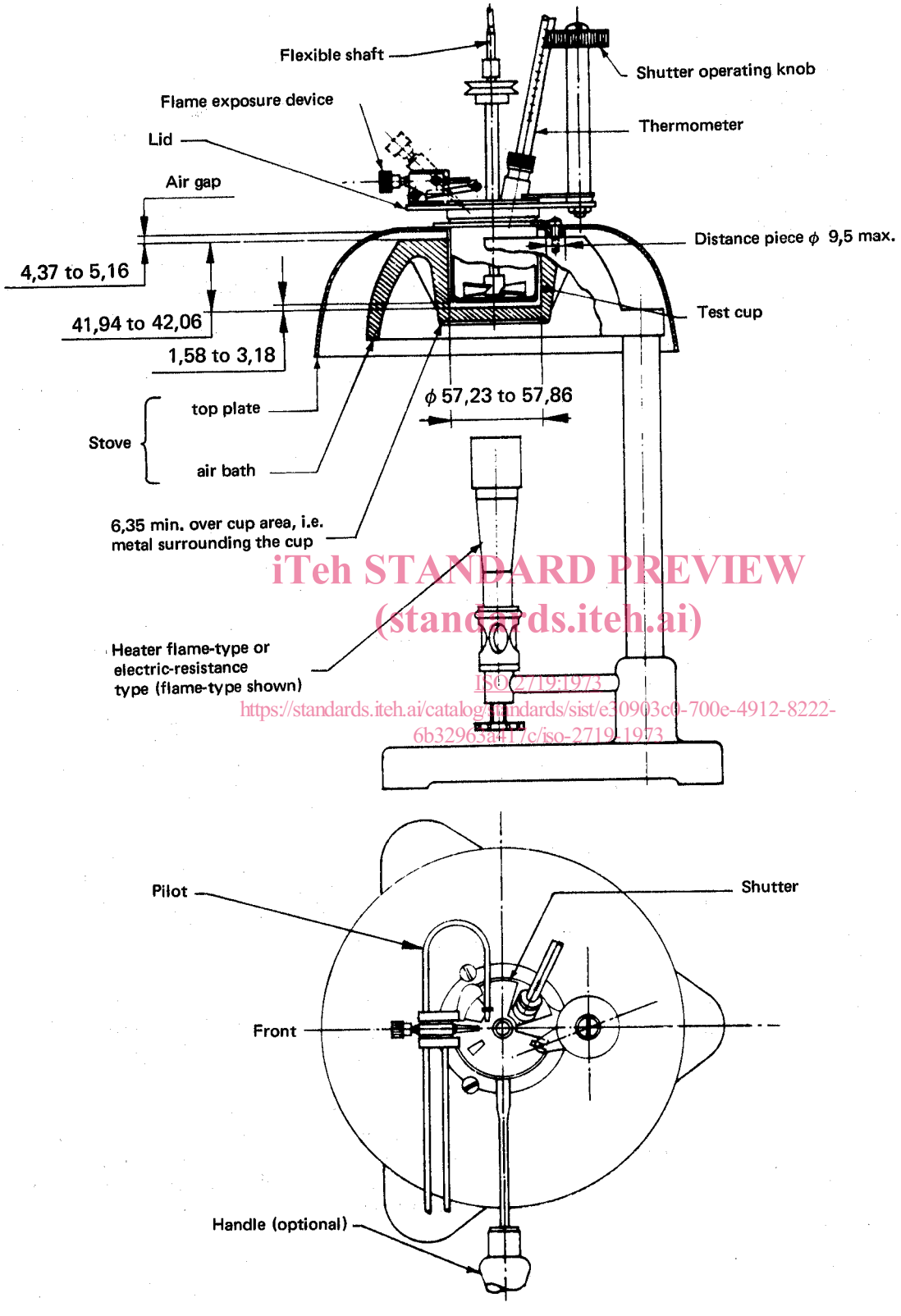
NOTES

1 If the heating element is a flame- or electrically-heated metal casting, it shall be so designed and used that the temperatures of the bottom and the walls are approximately the same. On this account it shall be not less than 6 mm thick. The casting shall be designed so that products of combustion of the flame cannot pass up and come into contact with the cup.

2 If the air bath is of the electric-resistance heated type, 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.

A.3.2 Top plate, of metal, mounted with an air gap between it and the air bath. It may 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 mm, and they shall be not more than 9,5 mm in diameter.

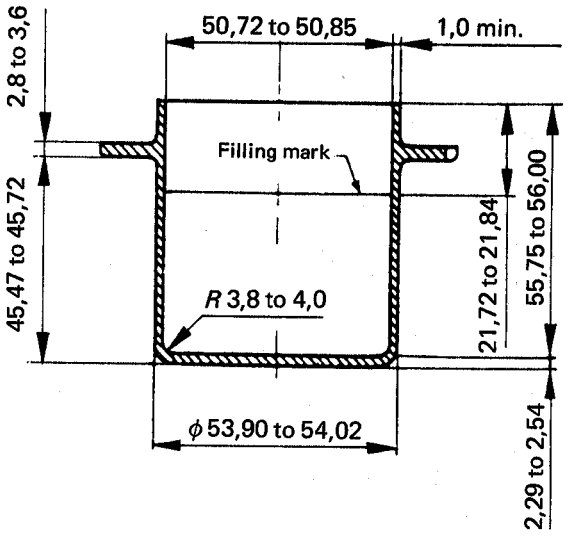
Dimensions in millimetres

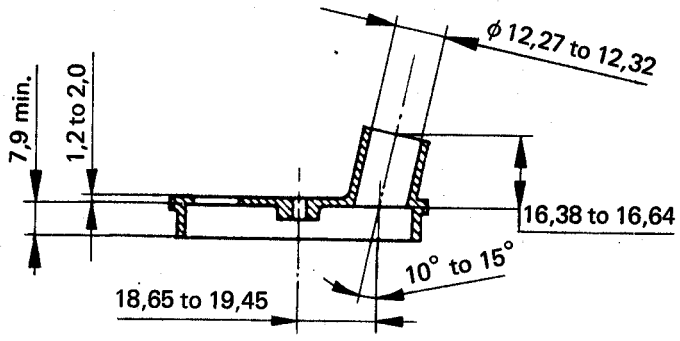


NOTE — Lid assembly may be positioned either right- or left-handed.

FIGURE 1 — **Pensky-Martens closed cup apparatus**

Dimensions in millimetres





Section X–X

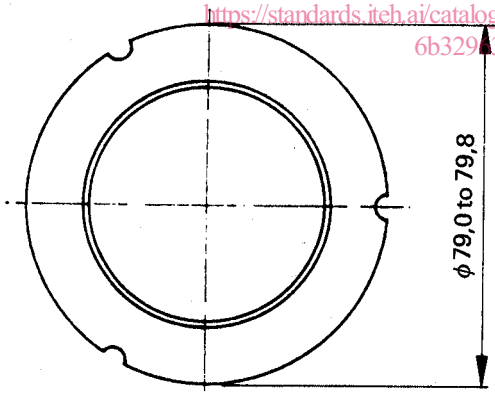


FIGURE 2 — **Test cup**

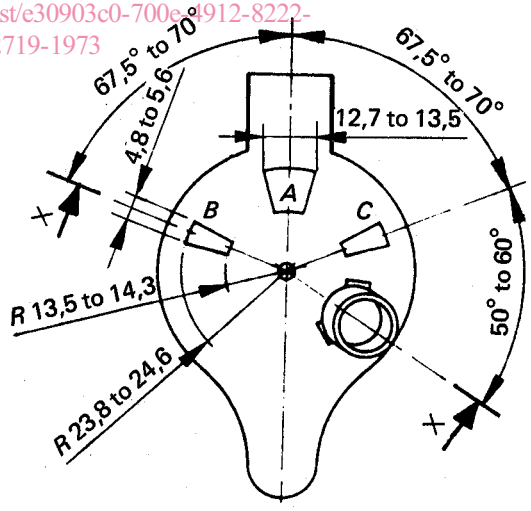


FIGURE 3 — **Cover**

Dimensions in millimetres

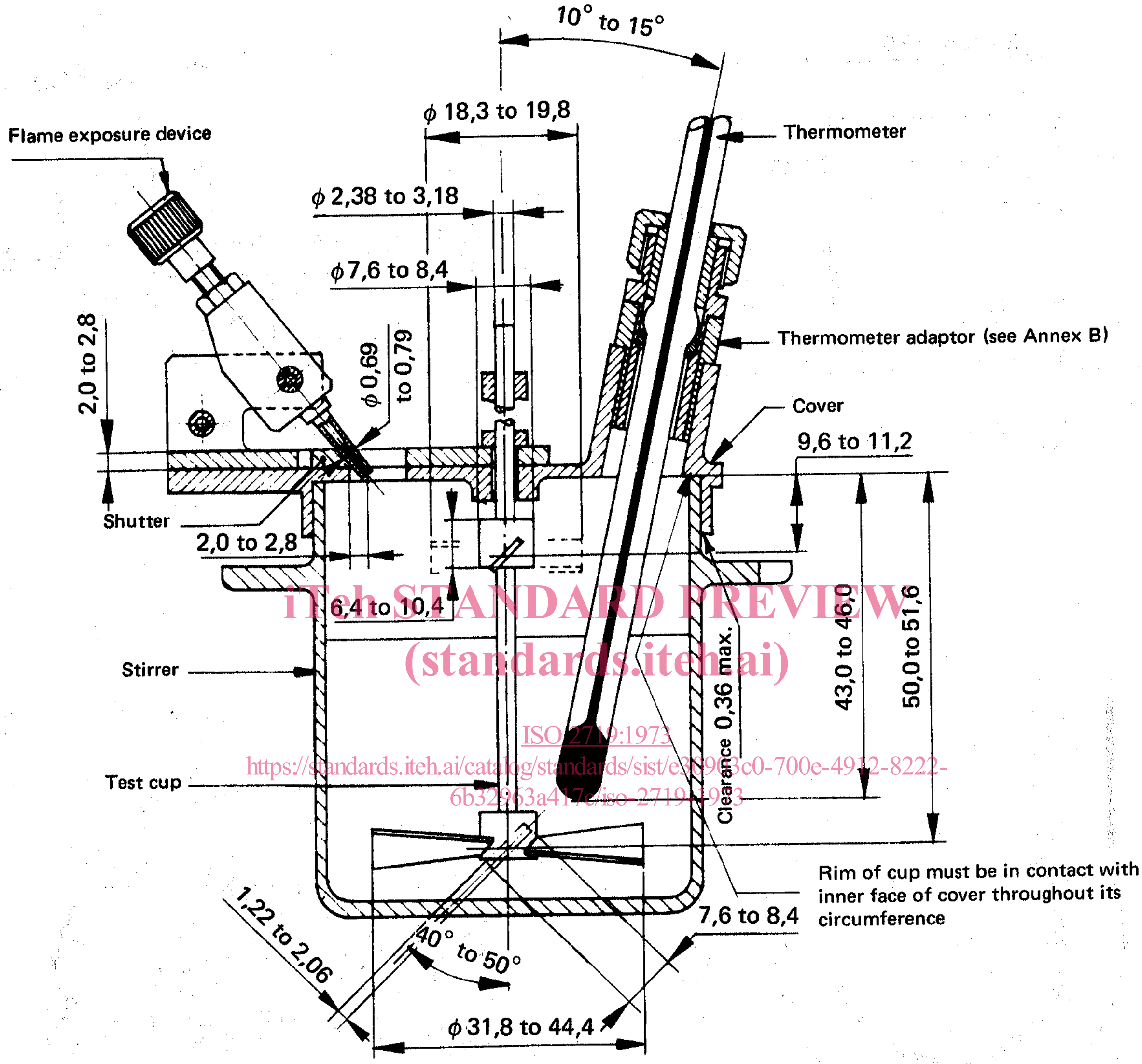


FIGURE 4 — **Test cup and cover assembly**

ANNEX B

MANUFACTURING STANDARDIZATION OF THERMOMETER AND FERRULE

B.1 The low-range thermometer, which conforms also to the specifications for the cup thermometer in the Tag closed tester and which frequently is fitted with a metal ferrule intended to fit the collar on the cover of the Tag closed tester, can be supplemented by an adapter (Figure 5) to be used in the larger diameter collar of the Pensky-Martens apparatus. Differences in dimensions of these collars, which do not affect test results, are a source of unnecessary trouble to manufacturers and suppliers of instruments, as well as to users.

B.2 Conformity to the requirements of the dimensions as shown in Figure 5 is not mandatory, but is desirable to users as well as suppliers of the Pensky-Martens closed cup apparatus.

B.3 The length of the enlargement and the distance from the bottom of the enlargement to the bottom of the bulb shall be measured with the test gauge as shown in Figure 6.

Dimensions in millimetres

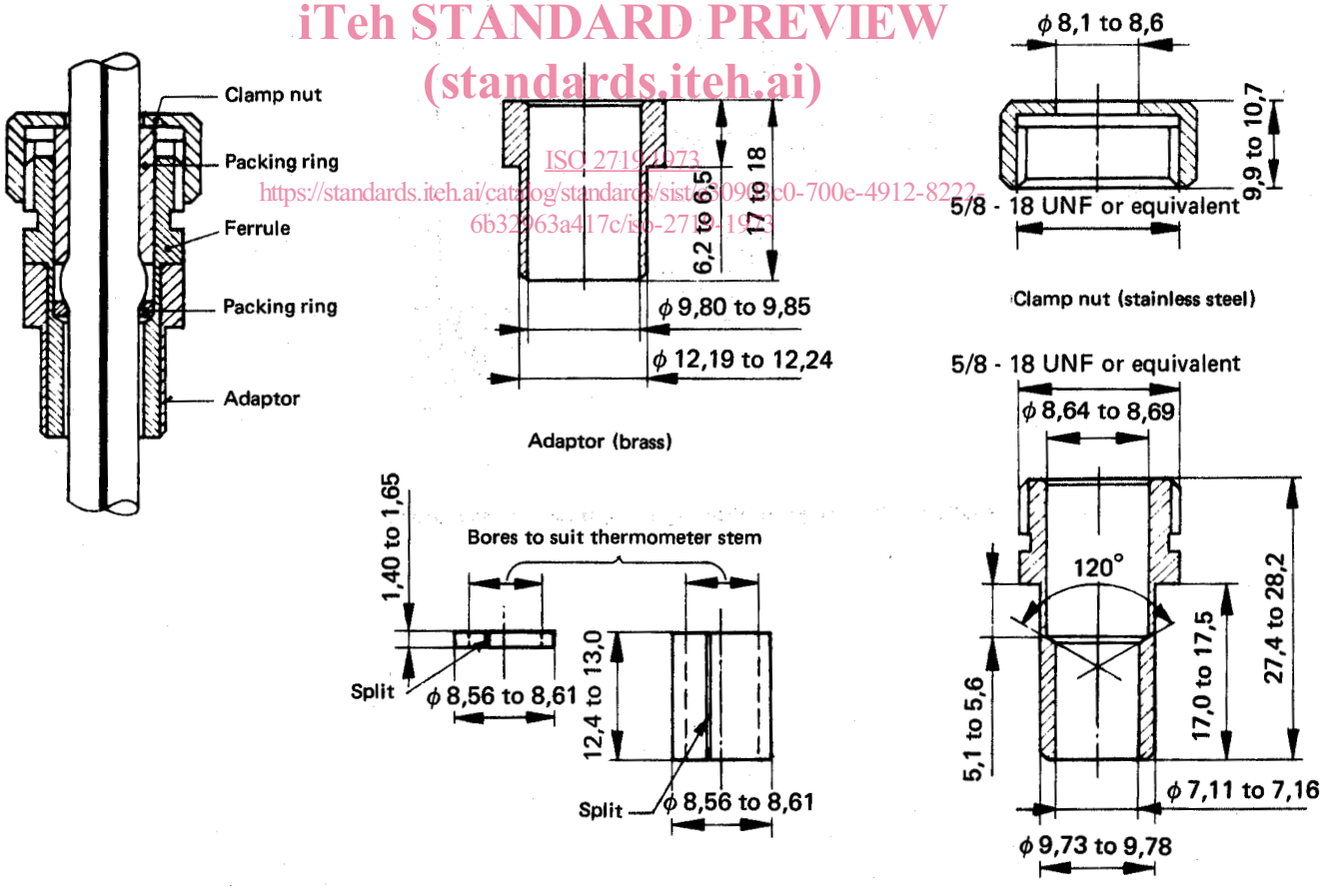


FIGURE 5 — **Dimensions for thermometer adaptor, ferrule, and packing ring.**