International Standard 3679

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION•МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ•ORGANISATION INTERNATIONALE DE NORMALISATION

Paints, varnishes, petroleum and related products — Determination of flashpoint — Rapid equilibrium method

Peintures, vernis, produits pétroliers et assimilés — Détermination du point d'éclair — Méthode rapide à l'équilibre

Second edition — 1983-12-01

UDC 667.612 : 536.468 **Ref. No. ISO 3679-1983 (E)**

Descriptors : paints, varnishes, petroleum products, tests, determination, flash point, test equipment, sampling.

Price based on 7 pages

Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of developing International Standards is carried out through ISO technical committees. Every member body interested in a subject for which a technical committee has been authorized 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.

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 3679 was developed jointly by Technical Committees ISO/TC 35, *Paints and varnishes*, and ISO/TC 28, *Petroleum products and lubricants*, and was circulated to the member bodies in June 1981.

It has been approved by the member bodies of the following countries :

Australia	Italy	Spain
Austria	Kenya	Sri Lanka
Belgium	Korea, Rep. of	Sweden
Brazil	Netherlands	Switzerland
Canada	New Zealand	Thailand
China	Norway	United Kingdom
Egypt, Arab Rep. of	Poland	USA
India	Portugal	USSR
Ireland	Romania	
Israel	South Africa, Rep. of	

The member bodies of the following countries expressed disapproval of the document on technical grounds :

France
Germany, F.R.

This second edition cancels and replaces the first edition (i.e. ISO 3679-1976).

Printed in Switzerland

Paints, varnishes, petroleum and related products — Determination of flashpoint — Rapid equilibrium method

0 Introduction

This International Standard describes one of two methods for the determination of the flashpoint of paints, varnishes, petroleum and related products and it should be read in conjunction with ISO 1523 when selecting a method. In ISO 1523, a similar determination is specified, using cups described in various national standards.

In both methods, the test is carried out only when the product under test and the air/vapour mixture above it in the test vessel are approximately in temperature equilibrium.

The apparatus specified in this International Standard enables a similar result to be determined using a more rapid procedure and with a smaller test portion (2 ml) of material than that given in ISO 1523. In addition, the apparatus can be made portable to the extent of being suitable for on-site testing as well as for normal use in laboratories. Collaborative work[1] has shown that results obtained by these procedures are comparable.

Nevertheless the interpretation of results obtained from solvent mixtures containing halogenated hydrocarbons should be considered with caution as these mixtures can give anomalous results.[2]

NOTE — The flash/no flash test using the same equipment under equilibrium conditions is given in ISO 3680.

1 Scope and field of application

This International Standard specifies a method for determining the flashpoint of a paint, varnish, paint binder, solvent, petroleum or a related product when the flashpoint is below 110 °C.

NOTE — Care should be taken in the interpretation of results obtained from solvent mixtures containing halogenated hydrocarbons (see clause 0).

2 References

ISO 1512, *Paints and varnishes — Sampling.*

ISO 1513, *Paints and varnishes — Examination and preparation of samples for testing.*

ISO 1523, *Paints, varnishes, petroleum and related products — Determination of flashpoint — Closed cup equilibrium method.*

ISO 3170, *Petroleum products — Liquid hydrocarbons — Manual sampling.*

ISO 3171, *Petroleum products — Liquid hydrocarbons — Automatic pipeline sampling.*

ISO 3680, *Paints, varnishes, petroleum and related products — Flash/no flash test — Rapid equilibrium method.*

3 Definition

flashpoint (closed cup) : Minimum temperature to which a product, confined in a closed cup, must be heated for the vapours emitted to ignite momentarily in the presence of a flame, when operating under standardized conditions.

NOTE — In this International Standard the flashpoint is corrected to an atmospheric pressure of 101,3 kPa (1 013 mbar).

4 Principle

4.1 Method 1 (For liquids whose expected flashpoint is between ambient temperature and 110 °C)

The test portion is heated in the specified apparatus. The ignition trial is carried out after the test portion has been maintained under equilibrium conditions for 60 s at a temperature approximately 3 °C below the expected flashpoint.

The trial is repeated at other temperatures until a flash is observed at a temperature which is not more than 1 °C above a

[1] BELL, L.H. *J. Inst. Petrol.* **57** (556) July 1971.

[2] RYBICKY, J. and STEVENS, J.R. *J. Coatings Technol.* **53** (676) May 1981 : 40-42.

temperature at which no flash was observed. The temperature at which the flash occurs is recorded as the flashpoint at the atmospheric pressure prevailing during the test and this temperature is then corrected to the standard atmospheric pressure of 101,3 kPa (1 013 mbar).

4.2 Method 2 (For liquids whose expected flashpoint is below ambient temperature)

The test portion is cooled to at least 3 °C below the expected flashpoint and then, in the specified apparatus, an ignition trial is carried out as in 4.1 after the test portion has been maintained under equilibrium conditions for 60 s.

The trial is repeated at other temperatures until a flash is observed at a temperature which is not more than 1 °C above a temperature at which no flash was observed. The temperature at which the flash occurs is recorded as the flashpoint at the atmospheric pressure prevailing during the test and this temperature is then corrected to the standard atmospheric pressure of 101,3 kPa (1 013 mbar).

5 Apparatus

5.1 Flashpoint tester, consisting of a block of aluminium alloy or other suitable corrosion-resistant metal of high thermal conductivity. The block has a cylindrical depression or test portion well, of depth approximately 10 mm and diameter approximately 50 mm, over which is fitted a cover. A thermometer is embedded in the block. A plan diagram is given in figure 1 and the essential dimensions are given in figures 2 to 5.

The cover is fitted with an opening slide and a device capable of inserting a test flame (diameter 3,5 ± 0,5 mm) into the well when the slide is open.

When inserted, the extremity of the nozzle of the ignition device shall just intersect the plane of the underside of the cover with a tolerance of ± 0,1 mm. The cover is also provided with an orifice extending into the well for insertion of the test portion and with a suitable clamping device for securing the cover tightly to the metal block so that the three openings in the cover are within the diameter of the well.

It is important that, when the slide is in the open position, the two openings in the slide coincide exactly with the two corresponding openings in the cover. It is also important that, when the slide is in the shut position, all the three openings in the cover are closed by the slide.

5.2 Thermometer, of appropriate range and dimensions which measures the temperature of the block within an error no greater than 0,5 °C. A thermometer having a graduation at each 0,5 °C is recommended. When required, the accuracy of the thermometer shall be checked against a reference standard by an authorized laboratory, using the stipulated immersion.

5.3 Heating device, fitted with a temperature controller such that the temperature of the metal block can be maintained within ± 0,2 °C of the required temperature. A signal light is necessary to indicate when heating is on. If the apparatus is intended to be portable, the heating device shall be electrical and shall be part of the complete apparatus.

The heating device shall be capable of controlling the rate of increase in the temperature of the flashpoint tester to within 0,5 °C in 30 s.

5.4 Means of cooling the well : Ice, solid carbon dioxide (CO_2), or a Peltier or other suitable cooling device.

If a continuously operating cooling device is used in method 2 (8.2) to control the well temperature, it should be possible to stabilize the temperature to within ± 0,2 °C of the expected flashpoint for a period of 60 s after the test portion has been discharged into the well. This ensures that equilibrium conditions are attained.

5.5 Syringe, capable of delivering 2 ml to an accuracy of ± 0,1 ml or, for use with highly viscous products, a **micropipette** or **spatula** (see the note in 8.1.3).

5.6 Fuel source for the ignition device : flammable gas, for example butane.

5.7 Suitable timing device.

6 Sampling and sample treatment

6.1 Take a representative sample of the product to be tested using the appropriate sampling procedure for the product concerned. References to sampling procedures for various products are given in the annex.

The sample shall be kept in an airtight container until it is to be tested. The ullage, i.e. the air space above the contents of the container, shall not be more than 10 % of the total capacity of the container.

Samples shall not be stored in plastics (polyethylene, polypropylene, etc.) bottles.

6.2 Because of the possibility of loss of volatile constituents, the sample container shall be cooled to at least 10 °C below the expected flashpoint before opening it to remove the test portion, except when method 2 (see 8.2) is used. In this case, the sample shall be cooled to 3 to 5 °C below the expected flashpoint before opening it to remove the test portion. The sample shall receive only the minimum mixing treatment to ensure uniformity. After removal of the test portion, the sample container shall immediately be tightly closed to ensure that loss of volatile components from the container is minimized. If this is not carried out, the product sample shall be deemed unsuitable for further testing.

7 Preparation of apparatus

Place the test apparatus in a position where it is not exposed to draughts, and preferably in subdued light.

8 Procedure

8.1 Method 1 (Determination of flashpoint when the expected flashpoint is between ambient temperature and 110 °C)

NOTE — When the expected flashpoint is close to ambient temperature, it may be more appropriate to use method 2.

8.1.1 Ensure that the well and cover/slide are clean and free from contamination, using a paper tissue if necessary. Close the cover and ensure that the slide is in the closed position.

8.1.2 Turn on the heating device (5.3). When the thermometer (5.2) reads approximately 3 °C below the expected flashpoint of the product to be tested, slowly adjust the controller of the heating device to the point at which the signal light is just extinguished. Allow the temperature of the well to stabilize, as indicated by the signal light cycling ON/OFF.

8.1.3 Ensure that the syringe (5.5) is clean and dry. Charge the syringe with 2 ml of the cooled sample (6.2) and transfer the syringe to the filling orifice, taking care not to lose any of the contents. Quickly discharge the test portion into the well, remove the syringe, and immediately start the timing device (5.7). Check that the slide of the cover is still in the closed position.

NOTE — If the viscosity of the product under test is so high as to prevent discharge through the orifice, a test portion of 2 to 3 ml may be transferred with a micropipette or a spatula into the well while the cover is open.

Immediately after filling the well, close the cover tightly.

8.1.4 Open the gas control valve and light the pilot and test flames. Adjust the test flame size to an approximately spherical shape of diameter 3,5 ± 0,5 mm.

8.1.5 When 60 s have elapsed, by which time the test portion is deemed to have reached the test temperature, perform the ignition trial by opening the slide, inserting and removing the nozzle, and closing the slide again over a period of 2,5 ± 0,5 s. Watch for a flash between opening and closing the slide.

8.1.6 Record whether a flash has occurred.

NOTES

1 When the vapour mixture under test is near the flashpoint, application of the ignition flame may give rise to a halo; however, the product is only deemed to have flashed if a comparatively large blue flame appears and propagates itself over the surface of the liquid.

2 If a continuous luminous flame burns in the orifice when the slide is opened and the ignition flame is introduced, then the flashpoint lies considerably below the test temperature.

8.1.7 Close the gas control valve and clean the apparatus.

8.1.8 If no flash is observed, repeat the test at 5 °C higher intervals, using a fresh test portion in each test, until a flash is observed.

If a flash is observed, repeat the test at 5 °C lower intervals, using a fresh test portion in each test, until no flash is observed.

CAUTION — Once a test flame has been applied to the test portion, the test is terminated and fresh test portions shall be used for each successive test.

8.1.9 Having established a flash between two temperatures 5 °C apart, repeat, using fresh test portions the procedures in 8.1.1 to 8.1.7 at 1 °C intervals above the lower of the two temperatures until a flash is observed. Read to the nearest 0,5 °C the temperature indicated by the thermometer when this flash occurs, correct this reading for any known thermometer correction, and record the result as the flashpoint at the atmospheric pressure prevailing during the test (see clause 9). Record also the atmospheric pressure in kilopascals, millibars, or millimetres of mercury.

8.1.10 Repeat the determination (8.1.1 to 8.1.9) and calculate the mean corrected flashpoint (see clause 9) to the nearest 0,5 °C.

8.2 Method 2 (Determination of flashpoint when the expected flashpoint is below ambient temperature)

8.2.1 Ensure that the sample and its container are at 3 to 5 °C below the expected flashpoint.

8.2.2 Cool the well (see 5.4) until its temperature is 3 to 5 °C below the expected flashpoint. Wipe out the well to ensure that it is clean and dry, and that it is free from any residue of carbon dioxide, if used. Close the cover and ensure that the slide is in the closed position.

8.2.3 Ensure that the syringe (5.5) is clean and dry. Charge the syringe with 2 ml of the cooled sample (6.2) and transfer the syringe to the filling orifice, taking care not to lose any of the contents. Quickly discharge the test portion into the well (see the note to 8.1.3), remove the syringe, and immediately start the timing device (5.7). Check that the slide of the cover is still in the closed position.

8.2.4 Open the gas control valve and light the pilot and test flames. Adjust the test flame size to an approximately spherical shape of diameter 3,5 ± 0,5 mm.

8.2.5 Ensure that the temperature of the well does not rise more quickly than about 0,5 °C in 30 s. When the temperature of the test portion well reaches the expected flashpoint, perform the ignition trial by opening the slide, inserting and removing the nozzle, and closing the slide again over a period of 2,5 ± 0,5 s. Watch for a flash between opening and closing the slide (see notes to 8.1.6).

8.2.6 Record whether a flash has occurred.

8.2.7 Close the gas control valve and clean the apparatus.

8.2.8 If no flash is observed, repeat the test at 5 °C higher intervals, using a fresh test portion in each test, until a flash is observed.

If a flash is observed, repeat the test at 5 °C lower intervals, using a fresh test portion in each test, until no flash is observed (see notes to 8.1.6).

CAUTION — Once a test flame has been applied to the test portion, the test is terminated and fresh test portions shall be used for each successive test.

8.2.9 Having established a flash between two temperatures 5 °C apart, repeat, using fresh test portions the procedures in 8.2.1 to 8.2.7 at 1 °C intervals above the lower of the two temperatures until a flash is observed. Read to the nearest 0,5 °C the temperature indicated by the thermometer when this flash occurs, correct this reading for any known thermometer correction, and record the result as the flashpoint at the atmospheric pressure prevailing during the test (see clause 9). Record also the atmospheric pressure in kilopascals, millibars, or millimetres of mercury.

8.2.10 Repeat the determination (8.2.1 to 8.2.9) and calculate the mean corrected flashpoint (see clause 9), to the nearest 0,5 °C.

9 Calculation

Calculate the flashpoint, in degrees Celsius, corrected to standard atmospheric pressure of 101,3 kPa (1 013 mbar or 760 mmHg), by adding algebraically to the observed temperature the correction from one of the following equations

$$C = \frac{101{,}3 - p_0}{4} \quad \text{or} \quad \frac{1\,013 - p_1}{40} \quad \text{or} \quad \frac{760 - p_2}{30}$$

where

C is the correction, in degrees Celsius;

p_0 is the atmospheric pressure, expressed in kilopascals;

p_1 is the atmospheric pressure, expressed in millibars;

p_2 is the atmospheric pressure, expressed in millimetres of mercury.

Record the mean corrected flashpoint to the nearest 0,5 °C.

NOTE — Whilst these formulae are strictly correct only within the atmospheric pressure range from 98,0 to 104,7 kPa, for pressures outside this range the error is sufficiently small to be ignored.

10 Precision

10.1 Repeatability (r)

The value below which the absolute difference between two single test results obtained on identical material by one operator in one laboratory using the same equipment within a short interval of time using the standardized test method, may be expected to lie with a 95 % probability, is 2 °C.

10.2 Reproducibility (R)

The value below which the absolute difference between two single test results obtained on identical material by operators in different laboratories, using the standardized test method, may be expected to lie with a 95 % probability, is 3 °C.

11 Test report

The test report shall contain at least the following information :

a) the type and identification of the product tested;

b) a reference to this International Standard (ISO 3679) and the method used : method 1 or 2 (as appropriate);

c) the mean corrected flashpoint, in degrees Celsius, calculated as in clause 9;

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

e) the date of the test.

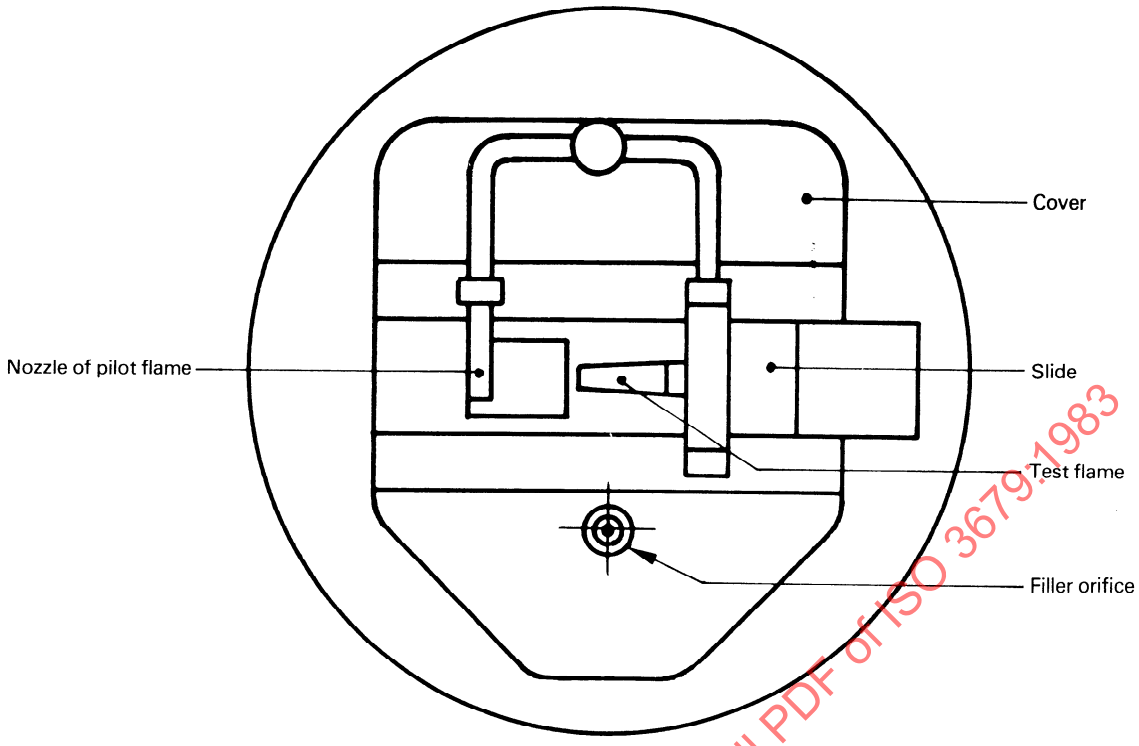

Figure 1 — Plan diagram of flashpoint tester

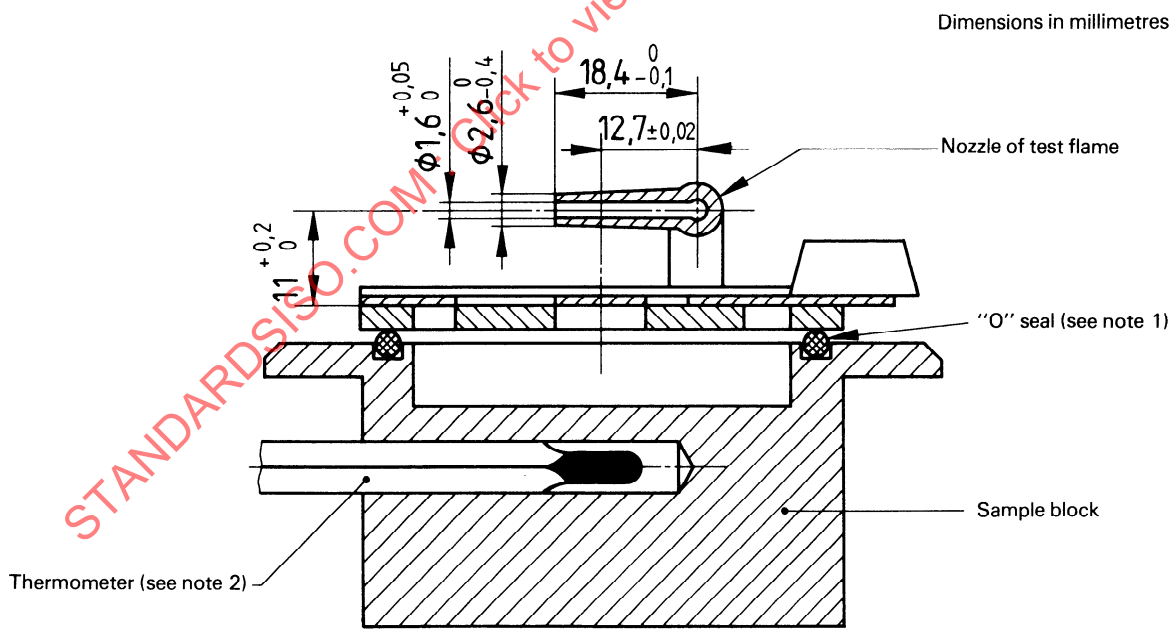

Figure 2 — Section of block through nozzle of test flame

NOTES

1 The "O" seal or gasket which provides a tight seal when the cover is shut shall be made of a heat-resistant material to withstand temperatures up to 150 °C.

2 When in position, the thermometer bulb shall be surrounded with a suitable thermal conducting thermoplastic compound. Silicone heat-sink compounds have been found suitable for the purpose.

3 The slide shall be fitted with a spring or other device to ensure that it stays in the fully closed position when shut.

Dimensions in millimetres

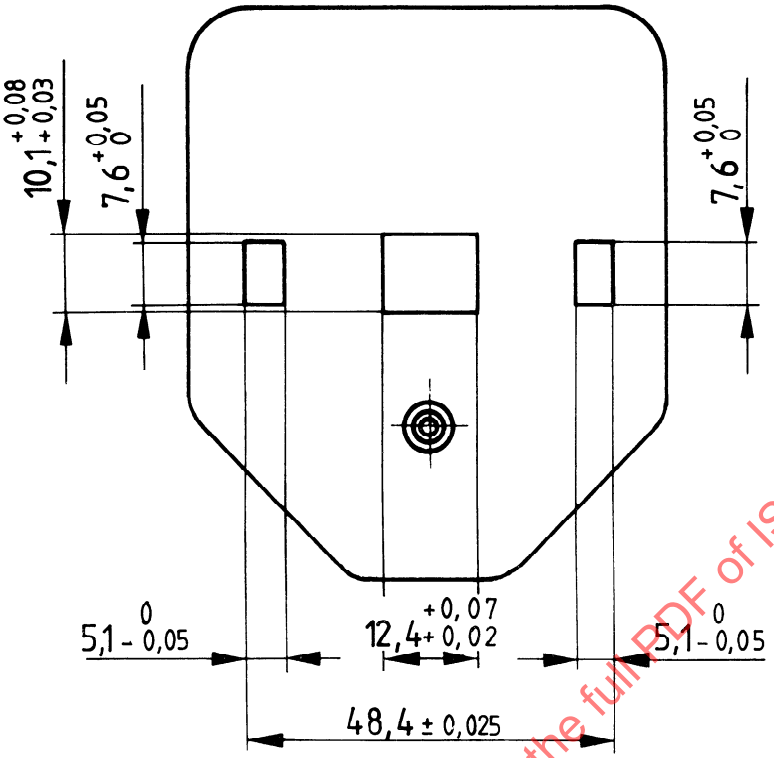

Figure 3 — Cover
(brass or other suitable metal, approximately 2 mm thick)

Dimensions in millimetres

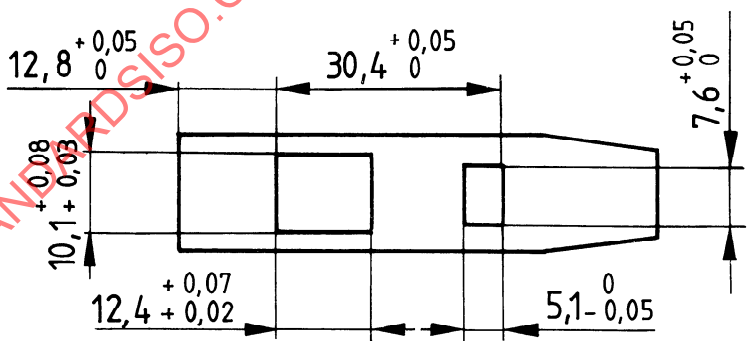

Figure 4 — Slide
(stainless steel or other suitable metal, approximately 1,2 mm thick)

Dimensions in millimetres

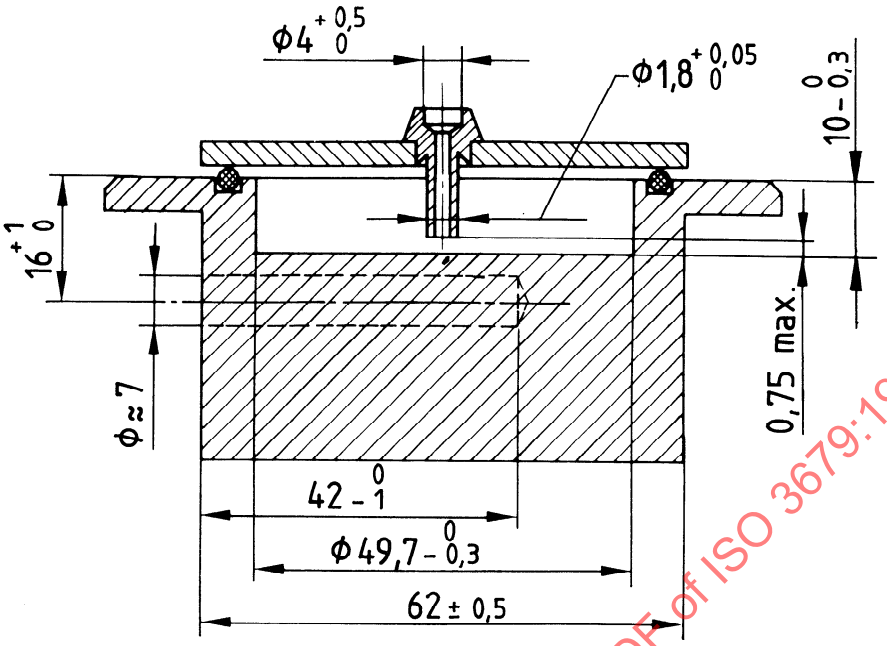

Figure 5 — Section of block through filler hole

Annex

Sampling procedures

A.1 Paints, varnishes and related products

Take a representative sample of the product to be tested as described in ISO 1512 and examine and prepare it for testing as described in ISO 1513. In addition observe the precautions of 6.2, as appropriate.

A.2 Petroleum and related products

Take a representative sample of the product to be tested as described in ISO 3170 or ISO 3171, as appropriate. In addition observe the precautions of 6.2, as appropriate.