
INTERNATIONAL STANDARD



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Paints and varnishes — Rapid test for determination of flashpoint

Peintures et vernis — Essai rapide pour la détermination du point d'éclair

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FOREWORD

ISO (the International Organization for Standardization) is a worldwide federation of national standards institutes (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 set up 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 drawn up by Technical Committee ISO/TC 35, *Paints and varnishes*, and was circulated to the Member Bodies in January 1975.

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

Austria	Ireland	South Africa, Rep. of
Brazil	Israel	Spain
Bulgaria	Mexico	Switzerland
Canada	Netherlands	Turkey
Denmark	New Zealand	United Kingdom
Germany	Poland	U.S.A.
India	Portugal	Yugoslavia
Iran	Romania	

No Member Body expressed disapproval of the document.

Paints and varnishes — Rapid test for determination of flashpoint

0 INTRODUCTION

This International Standard is one of a series of standards dealing with the sampling and testing of paints, varnishes and related products. It should be read in conjunction with ISO 1512, ISO 1513 and ISO 1523. In ISO 1523, a similar determination is specified, using cups as described in various national standards. 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 (L.H. Bell, *Journal of Institute of Petroleum* Vol. 57, No. 556-July 1971) has shown that results obtained by these procedures are comparable.

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a method for determining the flashpoint of a paint, varnish, paint medium or related product when the flashpoint is between 0 and 105 °C.

2 REFERENCES

ISO 1512, *Paints and varnishes — Sampling.*

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

ISO 1523, *Paints and varnishes — Determination of flashpoint — Closed cup method.*

3 DEFINITION

flashpoint (closed cup) : The lowest temperature, in degrees

Celsius, (at an air pressure of 101,3 kPa*) at which solvent vapour from the product under test and confined in a closed cup gives rise, under the conditions of test, to an air/vapour mixture which can be ignited by an external source of ignition.

NOTE — This measure of the flammability of solvent vapour by external ignition gives an indication of the degree of fire hazard of the product.

4 APPARATUS

4.1 Flashpoint tester, consisting of an aluminium alloy block or other suitable corrosion-resistant metal block or a metal block plated with a 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 an ignition flame (diameter $3,5 \pm 0,5$ mm) into the well when the slide is open. When inserted, the nozzle of the ignition device shall be 1 ± 1 mm above the underside of the cover. 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.

* 1 kPa = 1 kN/m²

4.2 Thermometer, of the mercury-in-glass type, nitrogen filled for horizontal operation, conforming to the following specification :

Range	0 to 110 °C
Graduation at each	1 °C
Longer lines at each	10 °C
Figured at each	10 °C
Length of graduated portion	125 ± 10 mm
Scale error not to exceed	0,5 °C
Overall length	200 ± 5 mm
Stem diameter	6,5 ± 0,5 mm
Bulb length	12 ± 2 mm
Bulb diameter	5 ± 1 mm
Bulb shape	Elongated
Distance from bottom of bulb to 0 °C mark	50 ± 2 mm
Top finish	Plain
Immersion	44 mm
Average temperature of emergent column	30 °C over entire range

4.3 Hot-plate, fitted with a temperature-controlling device, or other means of heating the metal block. Where the apparatus is intended to be portable, the electrical heating device shall be a part of the complete apparatus. The heating device shall be 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.

4.4 Means of cooling the well.

Ice, solid carbon dioxide (CO₂), a cooling cylinder or a close-fitting cooling coil and ancillary cooling circuit may be used.

4.5 Syringe, capable of delivering 2 ml to an accuracy of ± 0,1 ml.

4.6 Fuel source for the ignition device, normally butane fuel, but coal gas or natural gas is a satisfactory alternative.

4.7 Suitable timing device.

5 SAMPLING

5.1 A representative sample of the product to be tested shall be taken as described in ISO 1512. The sample shall then be examined and prepared for testing as described in ISO 1513.

The sample shall be kept in an air-tight container, except during withdrawal of a test portion.

5.2 Because of the possibility of loss of volatile constituents, it is advisable, before opening the sample container and removing the test portion, to cool the sample to at least 10 °C below the expected flashpoint temperature, except when method 2 (see 7.2) is used. In this case, the sample shall be cooled to 3 to 5 °C below the expected flashpoint temperature.

The sample shall receive only the minimum mixing treatment to ensure uniformity. After removal of the test portion, the sample container shall be immediately closed tightly to ensure that loss of volatile flammable components from the container is minimized (failure to do this will make the sample unsuitable for repeat tests).

6 PREPARATION OF APPARATUS

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

7 PROCEDURE

7.1 Method 1 : For liquids the expected flashpoint of which is between ambient temperature and 105 °C

(When the expected flashpoint is close to ambient temperature, it may be more appropriate to use method 2 or a combination of methods 1 and 2.)

7.1.1 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.

7.1.2 Turn on the heating device. When the thermometer reads approximately 3 °C below the expected flashpoint temperature of the product to be tested, slowly adjust the control of the heating device to the point at which the signal light is just extinguished.

7.1.3 Allow the well to become stable at this temperature, as indicated by the signal light cycling ON/OFF.

7.1.4 Ensure that the syringe is clean and dry. Charge the syringe with 2 ml of the cooled sample and transfer to the filling orifice taking care not to lose any of the contents.

NOTE — When 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 discharged with a micro-pipette or a spatula into the well while the cover is open.

Immediately after filling the well, the cover shall be tightly closed. Using this method, the sample and its container shall be cooled to at least 10 °C below the expected flashpoint temperature.

7.1.5 Discharge the 2 ml of sample into the well (see note in 7.1.4). Remove the syringe and immediately start the timing device.

7.1.6 Open the gas control valve and light the pilot and test flames. Adjust the test flame size to $3,5 \pm 0,5$ mm diameter.

7.1.7 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 jet, and closing the slide again over a period of $2,5 \pm 0,5$ s. Watch for a flash.

NOTE – When the vapour mixture under test is near the flashpoint temperature, 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.

If a large blue flame does not appear as a flash, but instead a continuous luminous flame burns in the orifice caused by opening the slide when the ignition flame is introduced, then the flashpoint of the product lies considerably below the test temperature and the determination shall be repeated.

7.1.8 Close the gas control valve.

7.1.9 If no flash was observed, clean out the well and repeat the test at 5°C higher intervals, using a fresh test portion in each test, until a flash is observed.

If a flash was 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 portions of the test sample shall be used for each successive test.

7.1.10 Having established a flash between two temperatures 5°C apart, repeat the procedures in 7.1.1 to 7.1.8, using fresh test portions at 1°C intervals above the lower of the two temperatures until a flash is observed. Read, to the nearest $0,5^\circ\text{C}$, the temperature indicated by the thermometer when this flash occurs, and record the result as the flashpoint temperature at the barometric pressure prevailing during the test (see clause 8). Record the barometric pressure.

7.2 Method 2 : For liquids the expected flashpoint of which is between 0°C and ambient temperature

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

7.2.2 Cool the well (4.4) until its temperature is 3 to 5°C below the expected flashpoint temperature. Wipe out the well to ensure that it is dry and free from any residue of carbon dioxide, close the cover and ensure that the slide is in the closed position.

7.2.3 Ensure that the syringe is clean and dry. Charge the syringe with 2 ml of the cooled sample and transfer to the filling orifice, taking care not to lose any of the contents (see note in 7.1.4).

7.2.4 Discharge the 2 ml of sample into the well (see note in 7.1.4). Remove the syringe.

7.2.5 Open the gas control valve and light the pilot and test flames. Adjust the test flame size to $3,5 \pm 0,5$ mm diameter.

7.2.6 When the temperature of the test portion well reaches the expected flashpoint temperature, perform the ignition trial by opening the slide, inserting and removing the jet and closing the slide again over a period of $2,5 \pm 0,5$ s. Watch for a flash (see note in 7.1.7).

7.2.7 Close the gas control valve.

7.2.8 If no flash was observed, clean out the well and repeat the test at 5°C higher intervals, using a fresh test portion in each test, until a flash is observed.

If a flash was 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 portions of the test sample shall be used for each successive test.

7.2.9 Having established a flash between two temperatures 5°C apart, repeat the procedures in 7.2.1 to 7.2.7 using fresh test portions at 1°C intervals above the lower of the two temperatures until a flash is observed. Read to the nearest $0,5^\circ\text{C}$ the temperature indicated by the thermometer when this flash occurs, and record the result as the flashpoint temperature at the barometric pressure prevailing during the test (see note in 7.1.7). Record also the barometric pressure.

8 CALCULATION OF CORRECTED FLASHPOINT

Calculate the flashpoint, corrected to standard barometric pressure of 101,3 kPa (1 013 mbar; 760 mmHg), by adding algebraically to the observed temperature the correction in degrees Celsius, given by one of the following formulae :

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

where

p_0 is the barometric pressure, expressed in kilopascals;

p_1 is the barometric pressure, expressed in millibars;

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

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

9 PRECISION

9.1 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.

9.2 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.

NOTE — These precision limits are the same as in ISO 1523; however, from the limited work carried out on paints, the indications are that the methods are capable of a much higher precision.

10 TEST REPORT

The test report shall include at least the following information :

- a) a reference to this International Standard and the method used : method 1 or 2 (as appropriate), or to a corresponding national standard;
- b) the type and identification of the product tested;
- c) the corrected flashpoint calculated as in clause 8;
- d) any deviation, by agreement or otherwise, from the test procedure specified;
- e) the date of the test.

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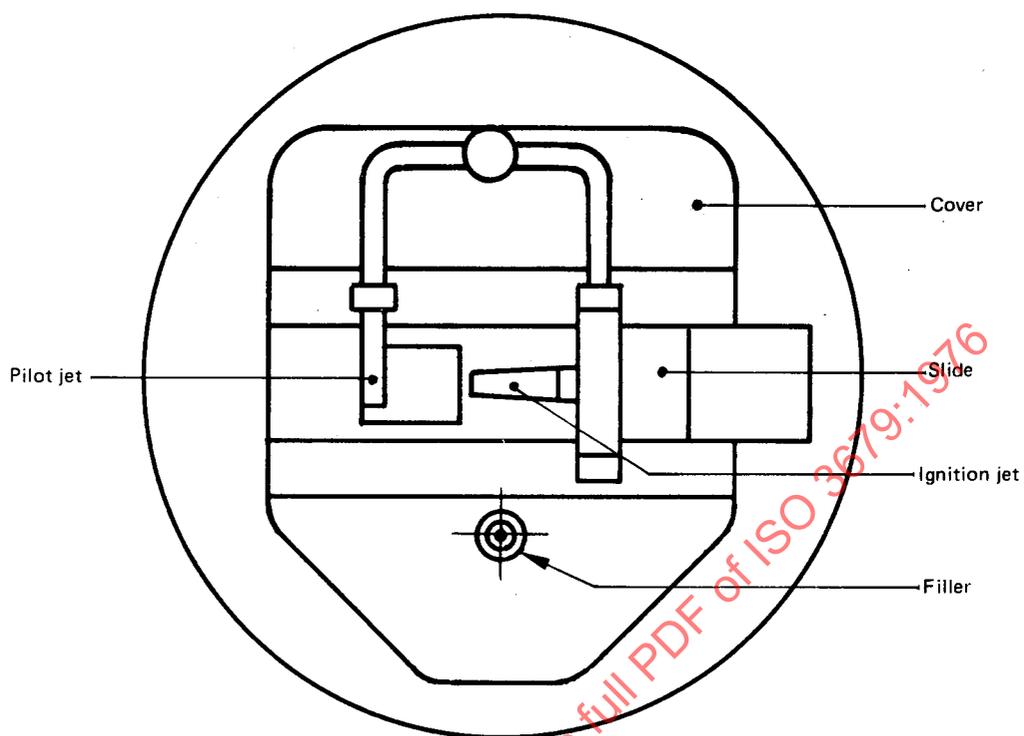


FIGURE 1 – Plan diagram of flashpoint tester

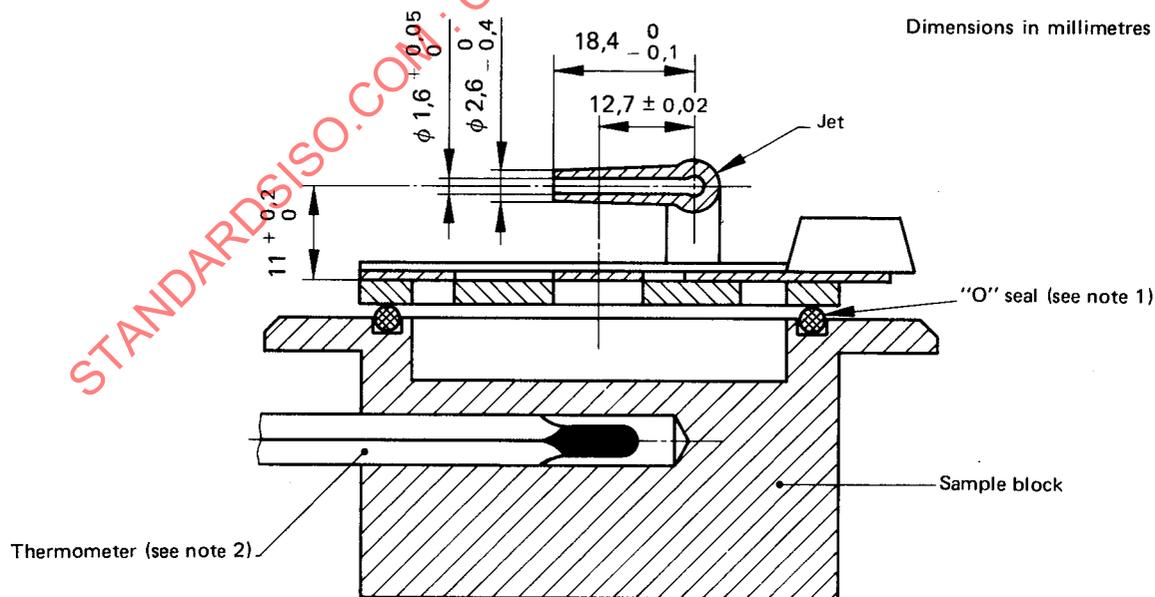


FIGURE 2 – Section of block through jet

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.