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ISO RECOMMENDATION

R 1523

PAINTS AND VARNISHES

DETERMINATION OF FLASHPOINT

(CLOSED CUP METHOD)

1st EDITION

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BRIEF HISTORY

The ISO Recommendation R 1523, *Paints and varnishes – Determination of flashpoint (Closed cup method)*, was drawn up by Technical Committee ISO/TC 35, *Paints and varnishes*, the Secretariat of which is held by the Nederlands Normalisatie-instituut (NNI).

Work on this question led to the adoption of Draft ISO Recommendation No. 1523, which was circulated to all the ISO Member Bodies for enquiry in November 1969. It was approved, subject to a few modifications of an editorial nature, by the following Member Bodies :

Austria	Italy	Sweden
Chile	Netherlands	Switzerland
Denmark	New Zealand	Turkey
France	Poland	U.A.R.
Germany	Portugal	United Kingdom
Greece	South Africa, Rep. of	U.S.S.R.
Israel	Spain	Yugoslavia

No Member Body opposed the approval of the Draft.

This Draft ISO Recommendation was then submitted by correspondence to the ISO Council, which decided to accept it as an ISO RECOMMENDATION.

PAINTS AND VARNISHES
DETERMINATION OF FLASHPOINT
(CLOSED CUP METHOD)

INTRODUCTION

This ISO Recommendation is one of a series dealing with the sampling and testing of paints, varnishes and related products. It should be read in conjunction with ISO Recommendation R 1516, *Paints and varnishes – Determination of the danger classification by flashpoint (closed cup method)*.

By the procedure described in this ISO Recommendation, differences between test apparatuses of various standard designs are minimised by ensuring that the test is carried out only when the product under test, and the air/vapour mixture above it in the test vessel, are exactly in temperature equilibrium.

1. SCOPE

This ISO Recommendation describes a method for determining the flashpoint of a paint, varnish or related product when tested in the manner described. The method is suitable for use over the temperature range 5 to 65 °C although some of the apparatus listed in the Appendix cannot cover all this range using the thermometers supplied with the apparatus.

NOTE. – In some countries existing regulations may require the use of other methods over at least a part of the temperature range 5 to 65 °C.

The procedure also makes allowance for variations from standard barometric pressure.

2. DEFINITION

Flashpoint (closed cup). The lowest temperature in degrees Celsius (at an air pressure of 101.3 kN/m²) 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.

3. PRINCIPLE

The test portion is heated in a closed cup of standard design by immersing it to the required level in a suitable water-bath. The temperature of the water-bath is slowly raised at such a rate that the difference in temperature between the water-bath and the test portion in the cup never exceeds 2 °C.

This slow rate of heating is necessary because of the low thermal conductivity of paints, varnishes and related products and also because heat transfer by convection is hindered by the high viscosity of many of these products. A stirring device may be used to aid thermal conduction but if used must not be in operation while an ignition trial is being carried out.

During the heating-up period ignition trials are carried out at intervals of not less than 1.5 minutes. This time interval is necessary to ensure that a saturation concentration of vapour in the air space above the test portion is re-established after each ignition trial.

The heating procedure laid down ensures that the temperature of the test portion does not rise more quickly than about 0.5 °C in 1.5 minutes.

4. APPARATUS

- 4.1 *Test cup.* The test cup used must be a closed cup with an internal level indicator; the closed cups specified in a number of national standards satisfy the necessary requirements (see Appendix).

NOTE. - If a stirrer is fitted to the test cup used, it should be operated during the heating-up period but must be stopped during each ignition trial. If a stirrer originally fitted to the test cup is removed, the aperture in the cover should be securely plugged before starting the test.

Essentially the test cup must be fitted with a tightly fitting cover which carries an opening slide and an ignition device capable, when the slide is open, of inserting an ignition flame (diameter 3.5 ± 0.5 mm); when inserted the nozzle of the ignition device must be 1 ± 1 mm above the underside of the cover. The equipment is such that an ignition trial can be performed by opening the slide, inserting and removing the nozzle of the ignition device, and closing the slide again, in a period of 2.5 ± 0.5 seconds. A mechanically driven device for doing this is permitted. The source of flame in the ignition device may be any suitable flammable gas.

- 4.2 *Water-bath.* Any suitable water-bath capable of being adjusted to the required temperature (see clause 6.2.1.2) and of adequate heat capacity to meet the requirements of clause 6.2.3.5 may be used. A bath fitted with a stirrer and an adjustable thermostat is convenient.
- 4.3 *Thermometers.* The test cup should be fitted with a thermometer of appropriate dimensions and suitable range and accuracy, which is immersed in the test portion for measuring its temperature.
- The water-bath should be fitted with a thermometer of equal precision for measuring the water temperature. The thermometers for the cup and water-bath should be of similar range and accuracy.

NOTE. - The thermometers used would normally be those supplied with the particular apparatus, but in any case should be accurate to 0.5°C .

- 4.4 *Support.* A suitable support should be provided for holding the test cup in the water-bath so that the cover and upper edge are horizontal and the cup is immersed in direct contact with the water in such a position that the level of the test portion in the cup is the same as or below that of the water in the water-bath.

5. SAMPLING

- 5.1 A representative sample of the product under test should be obtained and prepared by the methods described in ISO Recommendation R 1512, *Paints and varnishes - Sampling* and R 1513, *Paints and varnishes - Examination and preparation of samples for testing*. The sample should be provided and kept in an air-tight container.
- 5.2 Because of the possibility of loss of volatile constituents, the sample should receive only the minimum treatment to ensure uniformity. After removing the test portion, the sample container should be immediately closed tightly to ensure that no volatile flammable components escape from the container (otherwise an entirely new sample will be required).

6. PROCEDURE

6.1 Preliminary test

Determine the approximate flashpoint of the sample by one or more preliminary tests. This determines the starting temperature for the definitive test, which should be about 5°C below the expected value.

6.2 Definitive test

6.2.1 Preparation of apparatus

- 6.2.1.1 Set up the apparatus in a draught-free position in a room having a temperature controlled at about 20°C .

- 6.2.1.2 Adjust the water-bath to a temperature 5 °C below the approximate flashpoint as determined in clause 6.1.
- 6.2.1.3 Carefully clean and dry the test cup, the cover and the cup thermometer and bring them to approximately the same temperature as the water-bath in clause 6.2.1.2.
- 6.2.2 *Test portion.* Obtain and prepare the test sample as described in section 5 above, and ensure that at all times during this preparation its temperature is at least 10 °C below the expected flashpoint temperature.
- Fill the test cup with the test sample until the internal level indicator just disappears under the surface of the liquid. Take care to avoid the formation of bubbles and contact between the sample and the cup wall above the level indicator. If either of these conditions occurs to a significant extent, empty the cup, prepare it again according to clause 6.2.1.3, and fill it with a fresh portion of the sample.

6.2.3 Determination

- 6.2.3.1 Immediately after filling the test cup, place the cover in position and support the cup in the bath so that the cover is horizontal and the cup is immersed in direct contact with the water and with the surface of the test portion at the same level as or below that of the water in the bath. Confirm that the bath is at the required temperature as defined in clause 6.2.1.2.
- 6.2.3.2 Light the flame of the ignition device and adjust it to the size of a bead of diameter 3.5 ± 0.5 mm.
- 6.2.3.3 As soon as the test portion has attained the same temperature as the water-bath (i.e. the starting temperature of the definitive test) perform an ignition trial by opening the slide, inserting and removing the nozzle of the ignition device, and closing the slide again, over a period of 2.5 ± 0.5 seconds.
- 6.2.3.4 If ignition occurs (see Note 1), the initial temperature selected was too high, and the complete procedure of clause 6.2.3 should be repeated with a fresh portion of the sample at a temperature about 5 °C lower.
- 6.2.3.5 If no ignition occurs (see Note 1), heat the bath at a rate such that the difference in temperature between the bath and the test portion never exceeds 2 °C. When the test portion has increased in temperature by 0.5 °C (i.e. after not less than 1.5 minutes), repeat the ignition test and if no ignition occurs repeat the procedure until a temperature is reached at which ignition occurs (see Note 2). Read to the nearest 0.5 °C the temperature indicated by the cup thermometer, correct this reading for any known thermometer correction, and record the result as the flashpoint temperature at the barometric pressure prevailing during the test. Record also the barometric pressure in kilonewtons per square metre, millibars, or millimetres of mercury.

NOTES

1. 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 should be repeated from clause 6.1.

2. In view of the volatility of the solvent in the materials likely to be tested, the total duration of the test should not exceed about 2 hours.

7. CALCULATION OF CORRECTED FLASHPOINT

Calculate the flashpoint corrected to standard barometric pressure of 101.3 kN/m² (1013 mbar; 760 mmHg), by adding algebraically to the observed temperature the correction given in degrees Celsius by one of the following formulae :

$$\frac{101.3 - p_0}{4} \quad \text{or} \quad \frac{1013 - p_1}{40} \quad \text{or} \quad \frac{760 - p_2}{30}$$

where

- p_0 is the barometric pressure expressed in kilonewtons per square metre;
- p_1 is the barometric pressure expressed in millibars;
- p_2 is the barometric pressure expressed in millimetres of mercury.

8. PRECISION

- 8.1 Repeatability (one observer, one instrument) : ± 2 °C.
- 8.2 Reproducibility (different observers, different instruments) : ± 3 °C.

NOTE. - For reference tests a better level of precision is required, and the procedure described in section 10 should be followed.

9. TEST REPORT

The test report should contain the following information :

- (a) a reference to this ISO Recommendation or to a corresponding national standard;
- (b) type and identification of the product under test;
- (c) reference to the standard describing the test cup, and details of any modifications made;
- (d) corrected flashpoint calculated as in section 7;
- (e) any deviation, by agreement or otherwise, from the test procedure described;
- (f) date of the test.

10. REFERENCE TESTS

- 10.1 For reference tests two or more individual measurements should be made.
- 10.2 If the difference between the results of two individual tests, calculated in accordance with section 7, does not exceed 1.0 °C, report the mean value as the flashpoint.
- 10.3 If the difference between the first two results exceeds 1.0 °C, a third test must be made. If the greatest difference between the three results does not exceed 1.5 °C, report the mean value as the flashpoint.
- 10.4 If the greatest difference between the first three results exceeds 1.5 °C, two further tests must be made. If only one of the five results differs by more than 1.5 °C from the mean value, reject this result and report the mean value of the other four results as the flashpoint.
- 10.5 If more than one of the five results differs by more than 1.5 °C from the mean value, report this mean value as the flashpoint but state also the individual values and add a note to the test report on the irregular flashing of the sample.

APPENDIX

The cups listed below of closed cup apparatus described in national standards are known to satisfy the necessary requirements of this method of test for the determination of flashpoint. The method requires immersion of the cup in a water-bath and if a suitable bath is not included in the standard apparatus one must be provided.

Abel cup	French Standard	NF T 66-009
Abel cup	French Standard	NF M 07-011
Abel cup	British Standard	BS 3442 (and IP 33 and IP 170)
Abel-Pensky cup	German Standard	DIN 51 755
Abel-Pensky cup (+ stirrer)	Swedish Standard	SIS 02 18 11
Abel-Pensky cup (modified)	German Standard	DIN 53 213
Pensky-Martens cup	British Standard	BS 2839 (and IP 34)
Pensky-Martens cup	<u>French Standard</u>	NF M 07-019
Pensky-Martens cup	German Standard	DIN 51 758
Pensky-Martens cup	Netherlands Standard	NEN 3205
Pensky-Martens cup	Swedish Standard	SIS 02 18 12
Pensky-Martens cup	USA Standard	Z 11.7 (and ASTM D 93)
Tag cup	USA Standard	Z 11.24 (and ASTM D 56)