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IS 101-1-6 (1987): Methods of sampling and test for paints, varnishes and related products, Part 1: Test on liquid paints (general and physical), Section 6: Flash point [CHD 20: Paints, Varnishes and Related Products]



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“Knowledge is such a treasure which cannot be stolen”



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*Indian Standard*

# METHODS OF SAMPLING AND TEST FOR PAINTS, VARNISHES AND RELATED PRODUCTS

## PART 1 TESTS ON LIQUID PAINTS ( GENERAL AND PHYSICAL )

### Section 6 Flash Point

*( Third Revision )*

**1. Scope** — Prescribes a test and determination of flash point by closed cup equilibrium method. This standard prescribes flash/no flash test and actual determination, and is applicable between the temperature range of 5° and 110°C.

**2. Principle** — In case of flash/no flash test, the sample is heated in a suitably designated closed cup in a suitable water bath. The ignition trial is carried out after the test portion has been maintained under equilibrium conditions for at least 10 minutes at the selected equilibrium temperature. In the actual determination, the sample is heated in a suitably designated closed cup by immersing it to the required level in a suitable bath. The temperature of the bath is slowly raised at such a rate that the difference in temperature between the liquid in the bath and the test portion in the cup never exceeds 2°C, and the heating procedure ensures that the temperature of the test portion does not rise more quickly than about 7.5°C in 1-5 minutes.

### 3. Apparatus

**3.1 Test Cup** — A closed cup with an internal level indicator; if a stirrer is fitted to the test cup used, it may be operated during the heating-up period but shall be stopped during the ignition trial. If a stirrer originally fitted to the test cup is removed, the aperture in the cover shall be securely plugged before starting the test.

Essentially, the test cup shall be fitted with a cover which carries an opening slide and an ignition device which is inserted to a prescribed level into one of the openings in the cover when a test is made. The details of the cover, slide, ignition device and its movement shall be in accordance with the appropriate test given in this standard. The equipment is such that an ignition 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  $2.5 \pm 0.5$  s. A mechanically driven device for the opening of the slide and applying the ignition device to the air/vapour mixture in the cup is permissible and the source of flame for the ignition device may be any suitable flammable gas.

**3.2 Bath** — Any suitable bath capable of being adjusted to the required temperature and of adequate heating capacity to meet the requirements.

**3.3 Thermometers** — The test cup shall be fitted with a thermometer of appropriate range and dimensions that, when immersed in the test portion, measures its temperature with error not greater than 0.5°C.

The bath shall be fitted with a thermometer of equal precision when required, the accuracy of the thermometer's shall be checked against a reference standard by an authorized laboratory using the stipulated immersion.

**3.4 Support** — for holding the test cup in the bath so that the cover and upper edge are horizontal. The cup is immersed in direct contact with the water in the bath in such a position that the level of the test portion in the cup is the same as that of the water in the bath ( see Fig. 1 ).

### 4. Sampling and Sample Treatment

**4.1** Take a representative sample of the product to be tested using appropriate sampling procedure for the product concerned as given in IS:101 (Part 1/Sec 1)-1985 'Methods of sampling and test for paints, varnishes and related products: Part 1 Tests on liquid paints (general and physical), Section 1 Sampling (third revision)'. The sample shall be kept in air-tight container until it is to be tested. The ullage shall be not more than 10 percent of the total capacity of the container. Samples shall not be stored in plastic containers. Because of the possibility of loss of volatile constituents, the sample containers shall be cooled to at least 10°C below the expected flash point 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.

Adopted 30 April 1987

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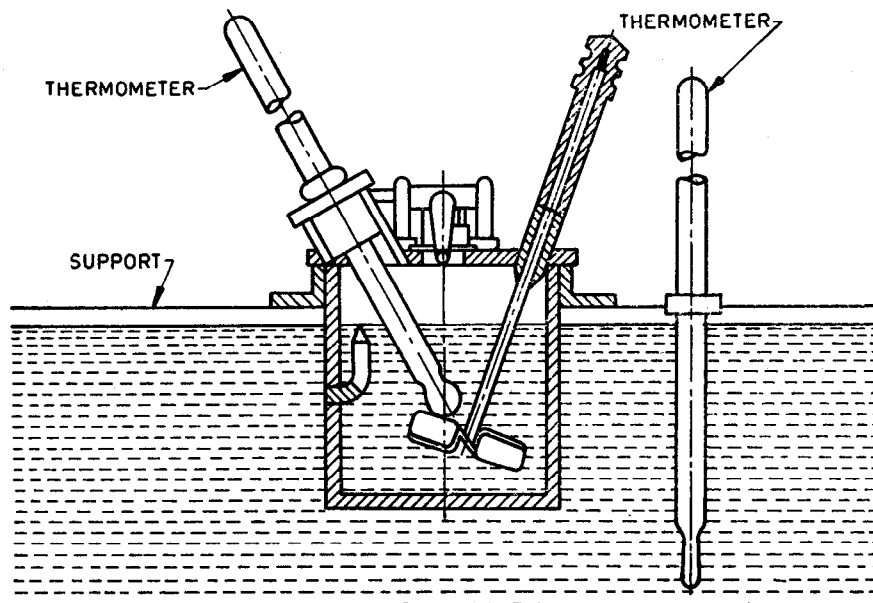


FIG. 1 CLOSED CUP, WITH FITTED STIRRER, IMMERSSED IN THE BATH  
( THE STIRRER FOR THE BATH IS NOT SHOWN )

## 5. Procedure

**5.1 Flash/No Flash Test**— Adjust the temperature of the bath and maintain it at the selected equilibrium temperature. The selected equilibrium temperature is corrected for variation from an atmospheric pressure of 101.3 kPa by raising the value for a higher pressure or lowering the value for a lower pressure at the rate of 1°C for each 4 kPa difference. Carefully clean and dry the test cup, its cover and thermometer. Bring them to a temperature at least 2°C below the selected equilibrium temperature. Fill the test cup with test portion until the internal level indicator just disappears under the surface of the liquid. Take care to avoid both 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 as above and fill it with a fresh test portion. Immediately after filling the test cup, place the cover and thermometer 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 bath liquid and with the surface of the test portion at the same level as the water in the bath. Light the flame of the ignition device and adjust it to the size of a bead of diameter  $3.5 \pm 0.5$  mm. Allow the temperature of the test portion to rise within 0.5°C of the selected equilibrium temperature. Maintain these conditions for 10 min, or such longer time interval as is necessary, to permit the temperature of the test portion to reach the selected equilibrium temperature. Then perform the ignition trial by opening the slide, inserting and removing the nozzle of the ignition device and closing the slide again, over a period of  $2.5 \pm 0.5$  seconds. Watch for a flash and close the slide. Record whether a flash has occurred. If no flash was observed, maintain the test portion at the test temperature for ten minutes and repeat the test. If the second test results in a flash, the product shall be considered to have flashed at the selected equilibrium temperature.

**5.2** Determine the approximate flash point of the sample by 5.1. This determines the starting temperature for the determination which shall be about 5°C below the expected value. Set up the apparatus in a draught free position and preferably in subdued light. Adjust the temperature of the liquid in the bath to 5°C below the approximate flash point as found out by 5.1. Carefully clean and dry the test cup, its cover and thermometer. Bring them to approximately the same temperature as the bath. Prepare the sample as given in 4 and ensure that, at all times during this preparation, its temperature is at least 10°C below the expected flash point temperature. Fill the cup with the test portion until the internal indicator first disappears under the surface of the liquid. Take care to avoid both the formation of bubbles and contact between the sample and the cup wall above the level indicator. If the either of these conditions occurs to a significant extent, empty the cup, prepare it again as above and fill it with a fresh test portion. Immediately after filling the cup, place the cover and thermometer in position. Support the cup in the bath so that the cover is horizontal and the cup is immersed in direct contact with the liquid in the bath and with the surface of the test portion at the same level as that of the liquid in the bath. Confirm that the bath is at the required temperature. Light the flame of the ignition device and adjust it to an approximately spherical shape of diameter  $3.5 \pm 0.5$  mm. As soon as the test portion has attained the same temperature as the liquid in the bath, perform an ignition trial by opening the slide, inserting and removing the nozzle of the ignition

device and closing the slide again, in a period of  $2.5 \pm 0.5$  s. Watch for a flash between opening and closing the slide. Record whether a flash has occurred. If a flash occurs carry out the procedure again with a fresh test portion but starting the test at a temperature about  $5^{\circ}\text{C}$  lower than selected previously. If no flash occurs, heat the bath at a rate such that the difference in temperature between the bath and test portion never exceeds  $2^{\circ}\text{C}$ . When the test portion has increased in temperature by  $0.5^{\circ}\text{C}$ , repeat the ignition test and if no flash is observed repeat the procedure until a temperature is reached at which a flash occurs. Read to the nearest  $0.5^{\circ}\text{C}$  the temperature indicated by the cup thermometer, correct this reading for any known thermometer correction, and record the result as the flash point at that atmospheric pressure prevailing during the test. Record the atmospheric pressure in kPa. Clean the cup and carry out a second determination using a fresh test portion and repeating the procedures described above. Calculate the mean corrected flash point to the nearest  $0.5^{\circ}\text{C}$ . When the test is required for reference purposes, where a higher level of precision is required, the procedure prescribed in 8 shall be followed.

## 6. Calculation

**6.1** Calculate the flash point, in degree Celsius, corrected to standard atmospheric pressure of 101.3 kPa by adding algebraically to the observed temperature the correction from one of the following equations:

$$C = \frac{101.3 - P_0}{4} \text{ or } \frac{1013 - P_1}{40} \text{ or } \frac{760 - P_2}{30}$$

where

$C$  = correction, in  $^{\circ}\text{C}$ ;

$P_0$  = atmospheric pressure, kPa;

$P_1$  = atmospheric pressure, mbar; and

$P_2$  = atmospheric pressure, mm Hg.

Record the mean corrected flash point to the nearest  $0.5^{\circ}\text{C}$ .

## 7. Precision

**7.0** Precision data is not quoted for flash/no flash test.

**7.1 Repeatability**—The repeatability for actual determination is expected to lie within  $2^{\circ}\text{C}$  with 95 percent probability.

**7.2 Reproducibility**—The reproducibility for actual determination is expected to lie within  $3^{\circ}\text{C}$  with 95 percent probability.

## 8. Test for Referee Purposes

**8.1** More than 2 individual measurements may be required to achieve the necessary agreement. If the difference between the results of two individual tests, calculated according to 6, does not exceed  $1.0^{\circ}\text{C}$ , report the mean value to the nearest  $0.5^{\circ}\text{C}$  as the flashpoint. If the difference exceeds  $1.0^{\circ}\text{C}$ , a third test shall be made. If the greatest difference between the 3 results does not exceed  $1.5^{\circ}\text{C}$ , report the mean value to the nearest  $0.5^{\circ}\text{C}$  as the flash point. If the greatest difference between the first 3 results exceeds  $1.5^{\circ}\text{C}$ , two further tests shall be made. If only one of the five results differs by more than  $1.5^{\circ}\text{C}$  from the mean value, reject this result and report the mean value of the other four results to the nearest  $0.5^{\circ}\text{C}$  as the flash point. If more than one of the five results differ by more than  $1.5^{\circ}\text{C}$  from the mean value, report this mean value to the nearest  $0.5^{\circ}\text{C}$  as the flash point but state also the individual values and add a note to the test report on the irregular flashing of the product tested.

## EXPLANATORY NOTE

This Indian Standard is one of the series dealing with the testing of paints, varnishes and related products. In the preparation of this standard, considerable assistance has been derived from ISO 1516-1981 Paints, varnishes, petroleum and related products—Flash/no flash test—Closed cup equilibrium method and ISO:1523-1983 Paints, varnishes, petroleum and related products—Determination of flash point—Closed cup equilibrium method issued by the International Organization for Standardization (ISO). This standard supersedes 24 of IS:101-1964 'Methods of test for ready mixed paints and enamels (second revision)'.