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Paints and varnishes — Determination of flow time by use of a flow cup

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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 2431 was drawn up by Technical Committee ISO/TC 35, *Paints and varnishes.*

It was approved in August 1971 by the Member Bodies of the following countries :

Austria	Ireland	Romania
Belgium	Israel	South Africa, Rep. of
Canada	Italy	Sweden
Egypt, Arab Rep. of	Netherlands	Switzerland
France	New Zealand	Turkey
Germany	Poland	United Kingdom
India	Portugal	U.S.S.R.

No Member Body expressed disapproval of the document.

Paints and varnishes — Determination of flow time by use of a flow cup

0 INTRODUCTION

This International Standard 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/R 1512 and ISO/R 1513.

The standardization for ISO purposes of a single improved design of flow cup is recommended after careful consideration by an expert task group of the role of flow cups for the measurement of flow time of paints, varnishes and related products. It is recognised that such flow times are of limited usefulness as the cups are only suitable for products of newtonian or near-newtonian flow properties. This effectively limits their practical use for paints to products of not more than 150 cSt (centistokes), since paint products of higher viscosity than this invariably have anomalous flow properties which can only be properly assessed using viscometers operating at high rates of shear. As is well known, many countries have over the years developed their own standard flow cups and the correlation of these has led to considerable confusion in comparing values, mainly because the products under test are seldom newtonian in behaviour.

The design adopted for the ISO flow cup has been carefully chosen to give the best compromise between accuracy in use in the viscosity range up to 150 cSt, ease of use including cleaning, and ease of manufacture. The ISO cup is intended in the first place for use as an international flow cup suitable for application in interchanging results between countries, and it is hoped that as its usefulness is appreciated it may increasingly replace existing national standard cups as a rapid means of assessing the flow characteristics of many paints, varnishes and related products.

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies the dimensions and the method of use of a flow cup, suitable for measuring the flow time of paints, varnishes and related products of newtonian and near-newtonian flow properties, of kinematic viscosity in the range of about 25 to $150 . 10^{-6} \, m^2/s$ (25 to 150 cSt).

NOTE — The method is only suitable when the breakpoint of the flow of product from the orifice of the cup can be defined with certainty. For paint products, this is normally found only in paints having a kinematic viscosity of not more than 150 cSt, such as the thinner brushing paints and those used for spraying, dipping, flow coating, etc.

2 REFERENCES

ISO/R 1512, *Paints and varnishes — Sampling.*

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

3 DEFINITIONS

3.1 flow time : The time in seconds which elapses from the moment when the material under test starts to flow from the orifice of the filled cup to the moment when the stream of material flowing out breaks.

3.2 newtonian flow : A material is considered to have newtonian flow when the ratio of the rate of shear to the shearing stress does not vary at different rates of shear. When variations in this ratio are small, the effect on viscosity of mechanical disturbance, such as stirring, is negligible and the material is said to have near-newtonian flow.

3.3 anomalous flow : A material is considered to have anomalous flow when at a constant temperature the ratio of the shear rate to the shearing stress varies either with time or with shear rate. For example, with so-called thixotropic materials stirring or other such mechanical disturbance immediately before test will reduce the flow time below that for an unstirred sample. With such materials uncertain and variable values for flow time are obtained in all flow cups.

3.4 viscosity units : Absolute or dynamic viscosity is defined in newton seconds per square metre ($N \cdot s/m^2$) but for laboratory purposes is frequently measured in poises and centipoises ($1 \, cP = 10^{-3} \, N \cdot s/m^2$). For the flow of liquids through tubes under gravity the density of the material must also be taken into account. The ratio absolute viscosity/density is known as the kinematic viscosity, which is defined in square metres per second

(m^2/s) but for laboratory purposes is frequently measured in stokes and centistokes (1 cSt = 10^{-6} m^2/s). The flow time of newtonian and near-newtonian materials in flow cups is related to the kinematic viscosity.

4 TEMPERATURE CONSIDERATIONS

4.1 The effect of temperature on flow time is very significant and it is for this reason that the use of a controlled temperature enclosure (for example, a cabinet or room) is recommended and is considered essential for international referee purposes. If this is not available then it should be ensured that the temperature change in the material during the testing, as determined by comparing the material temperature just before testing with that recorded by a thermometer in the efflux stream, shall be kept as low as possible and in any case shall not exceed 0.5 °C. It is not possible to apply a general temperature correction factor to flow times because the types of materials likely to be tested are known to have very different temperature coefficients.

4.2 For international referee purposes it is essential to standardize one temperature of test and this is recommended to be 23 °C. However it may be more convenient to carry out comparative testing at some other temperature (for example 25 °C) because of prevailing temperature conditions. For such testing the test temperature shall be specified in the national standard or if none exists the temperature shall be agreed between the interested parties. It is still essential to use a controlled temperature enclosure or to ensure that temperature variation during testing does not exceed 0.5 °C.

5 APPARATUS

5.1 Flow cup

5.1.1 *Dimensions*

The dimensions of the ISO flow cup and the tolerances allowed in manufacture shall be as given in Figure 1. The most critical tolerance is the internal diameter of the jet of the cup since the flow time is inversely proportional to the fourth power of this dimension. The jet of the cup shall be made of stainless steel and the body of the cup shall be made of a material which is corrosion resistant and not affected by the products to be tested.

5.1.2 *Construction*

The dimensions not specified, such as wall thickness, shall be such that no distortion of the cup can occur in use. The external shape shown in Figure 1 is recommended, but may be modified for convenience of use or manufacture, provided that the protruding jet of the cup is protected from accidental damage as far as possible by an external protective sleeve. Such a protective sleeve must not be immediately adjacent to the jet so as to cause a capillary action when the material under test flows out.

5.1.3 *Finish*

The interior surfaces of the cup, including the orifice, shall be smooth and free from turning marks, crevices, ledges and burrs which may cause random flow or trap some sample or cleaning material. The standard of finish required is equivalent to a maximum roughness[1] of not more than 0.5 μm.

5.1.4 *Calibration*

The cup shall be calibrated using a standard mineral oil of known kinematic viscosity. The procedure used is described in the Appendix.

5.1.5 *Marking*

Each flow cup shall have the following inscriptions permanently and legibly marked on it :

1) designation of cup : "ISO 2431";

2) identification number (manufacturer's);

3) maker's name or trade mark.

5.2 Supplementary apparatus

5.2.1 *Thermometer,* accurate to 0.2 °C and graduated at 0.1 °C intervals.

5.2.2 *Stand,* suitable for holding the flow cup and provided with levelling screws (see also 7.4).

5.2.3 *Spirit level,* preferably of the circular type.

5.2.4 *Flat glass plate* or *straight edge scraper.*

5.2.5 *Stop watch,* or other suitable timing device with scale divisions of 0.2 s or finer and accurate to within 0.1 % when tested over a 60 min period.

5.2.6 *Temperature controlled room* or *enclosure* for maintaining the cup and sample at a constant temperature (recommended).

6 SAMPLING

A representative sample of the material to be tested shall be taken as described in ISO/R 1512. The sample shall then be prepared for testing as described in ISO/R 1513.

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1) In the sense defined as the arithmetic mean deviation R_a from the mean line of the profile, in ISO/R 468, *Surface roughness.*

Before testing, the sample shall be strained through a sieve of aperture size 125 μm into a clean container. 150 ml of strained material is sufficient for carrying out one test. Care shall be taken to mix the paint thoroughly, while at the same time avoiding, as far as possible, loss of solvent by evaporation.

7 PROCEDURE

7.1 Temperature adjustment

Adjust the strained sample, and the flow cup, to a temperature of 23 ± 0.5 °C or to an alternative agreed test temperature (see 4.2). If a controlled temperature enclosure is used, as recommended, it is advisable to condition the sample before sieving, and also the cup, by placing them in the enclosure before use. The sample is considered ready for test immediately after any air bubbles entrained during the preparation and sieving procedures have dispersed. A final check that the temperature of the sample is within 0.5 °C of the agreed test temperature shall be made immediately prior to filling the cup.

7.2 Preparation of the flow cup

Place the flow cup on the stand provided, in a position free from draughts, and by using a spirit level and adjusting the levelling screws of the stand ensure that the upper rim of the flow cup is in a horizontal plane.

7.3 Filling the flow cup

With the orifice closed by the finger, fill the cup with the freshly sieved, bubble-free sample, pouring slowly to avoid the formation of air bubbles. If any bubbles are formed allow them to rise to the surface and remove them. If the cup has been properly levelled the sample will overflow evenly over the rim, into the gallery. Any meniscus formed is removed by drawing a straight edge over the entire rim of the cup or by sliding over the rim a flat glass plate with rounded edges so that no air bubbles form between the glass and the surface of the sample, and then drawing this plate horizontally across the rim of the cup so that, when it is removed, the level of the sample concides with the top rim of the cup.

7.4 Measurement of flow time

Place a suitable receiver under the flow cup such that the distance between the orifice of the flow cup and the surface of the received sample is never less than 100 mm. Remove the finger from the orifice and simultaneously start the timing device, stopping it as soon as the first break occurs, close to the orifice, in the stream of sample. Record the flow time to the nearest 0.2 s.

If the test is not carried out in a controlled temperature enclosure, place the thermometer in the stream of sample.

(It is convenient to use the same thermometer as used to adjust the temperature of the sample initially). This is conveniently done by holding the thermometer in a suitable clamping device with the bulb so placed that it is at an angle to the direction of flow and completely immersed in the emergent stream. Any difference in temperature from the initially adjusted temperature shall not be greater than 0.5 °C.

7.5 Precision

7.5.1 *Repeat determination*

A second determination shall be carried out using another portion of the originally prepared sample and checking carefully that the temperature of testing is within the prescribed limits. The flow time in each case shall be recorded to the nearest 0.2 s. The results of the two determinations shall not differ from their mean value by more than 2 %; if they do, a third determination shall be made. If the results of this determination and either one of the previous determinations do not differ from their mean value by more than 2 %, the other of the previous results shall be discarded. If the third determination does not provide this measure of agreement, the method of test is unlikely to be suitable because of anomalous flow behaviour, and consideration shall be given to other methods of test.

7.5.2 *Reproducibility*

Results from different laboratories shall not be considered suspect unless they differ by more than 5 % absolute.

8 TEST REPORT

The test report shall include the following information :

a) a reference to this International Standard or to a corresponding national standard;

b) type and identification of the product under test;

c) description of the flow cup used, including identification number;

d) temperature of testing to the nearest 0.2 °C; and an explanation if this temperature, by agreement, differs from 23 ± 0.5 °C;

e) the flow time reported as a mean value (of two results) rounded off to the nearest second, provided that the individual results do not differ by more than 2 % from the mean value before rounding; for referee purposes the individual values recorded to the nearest 0.2 s shall also be reported;

f) any deviation, by agreement or otherwise, from the test procedure described;

g) date of the test.

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9 CARE AND CHECKING OF FLOW CUPS

Clean the cup immediately after use and before the sample starts to dry, using a suitable solvent. Metal cleaning tools or wire must never be used. If the orifice becomes contaminated with dried deposits, these must be softened with a suitable solvent and cleaned carefully, for example with a soft cloth pulled through the orifice.

Cups shall be checked periodically for wear or damage by the calibration procedure described in the Appendix.
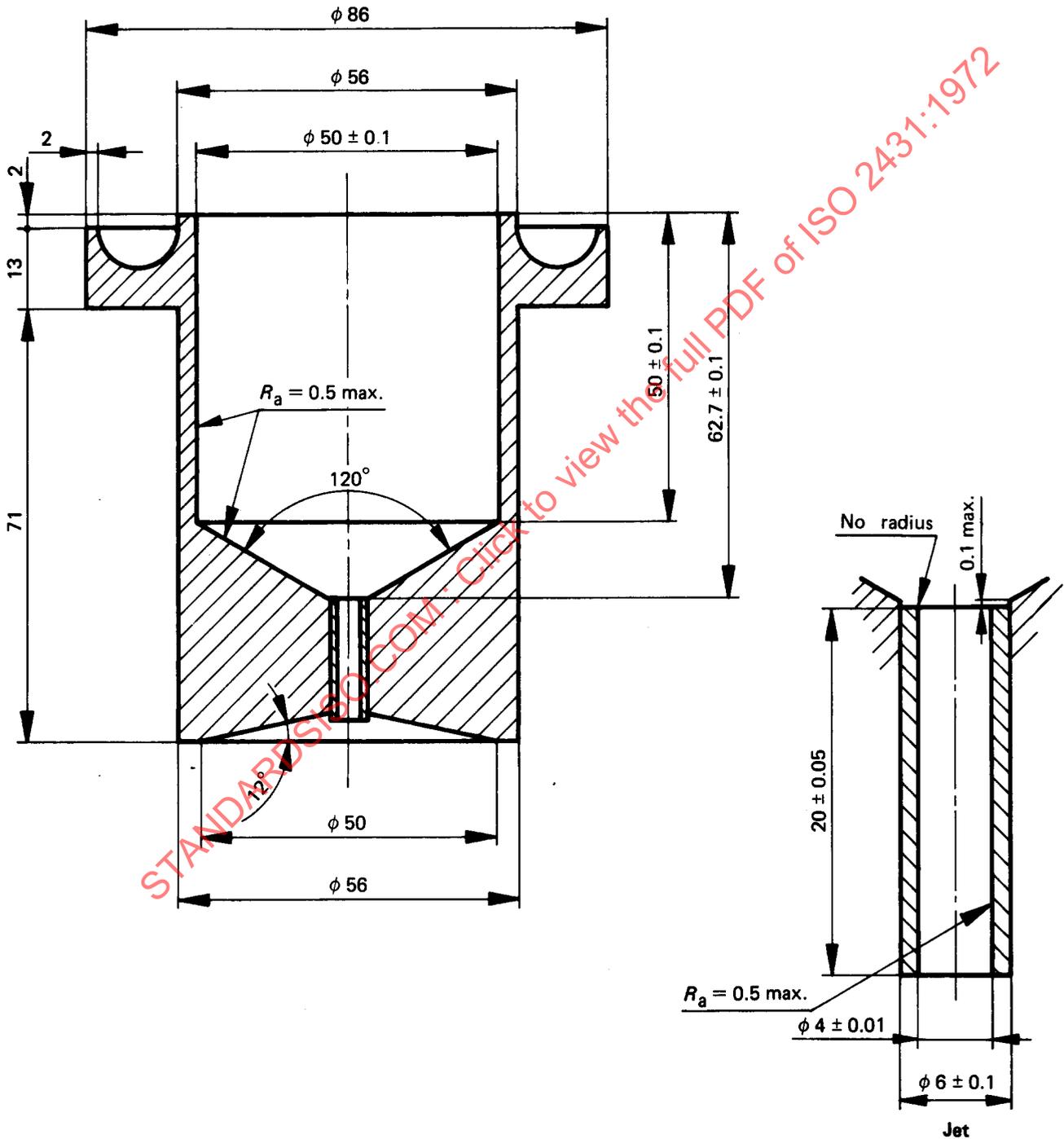
Dimensions in millimetres

FIGURE 1 — Flow cup ISO 2431