International Standard 2431

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Paints and varnishes — Determination of flow time by use of flow cups

Peintures et vernis — Détermination du temps d'écoulement au moyen de coupes d'écoulement

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

This third edition was submitted directly to the ISO Council, in accordance with clause 6.11.2 of part 1 of the Directives for the technical work of ISO. It cancels and replaces the second edition (i.e. ISO 2431-1980), which had been approved by the member bodies of the following countries :

Australia	Iran	Romania
Austria	Israel	South Africa, Rep. of
Brazil	Italy	Sweden
Bulgaria	Kenya	Switzerland
Czechoslovakia	Korea, Rep. of	Turkey
Egypt, Arab Rep. of	Mexico	United Kingdom
Germany, F.R.	Netherlands	USSR
India	Norway	

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

France
New Zealand
Poland

Paints and varnishes — Determination of flow time by use of flow cups

0 Introduction

The first edition of this International Standard, published in 1972, specified only one flow cup of orifice diameter 4 mm. The second edition specified three flow cups of orifice diameter 3 mm, 4 mm and 6 mm. This third edition corrects errors in figures 2 and 4 and the equations for those figures. Also, this edition incorporates the corrections given in Erratum 1, published by ISO in 1981. It is one of a series of standards dealing with the sampling and testing of paints, varnishes and related products.

As is well known, many countries over the years have developed their own standard flow cups and the difficulty in correlation between them has led to considerable confusion in comparing values. The standardization of an improved design of flow cup has been recommended after careful consideration, by an expert working group, of the role of flow cups for the measurement of flow time of paints, varnishes and related products.

It is recognized that the flow times are reproducible only for products of newtonian or near-newtonian flow properties. This effectively limits their practical use. Nevertheless, for checking purposes, these flow cups do serve a useful purpose. Furthermore, the measurement of flow time is often used to confirm the application consistency.

Paints often contain flow arresting agents to confer increased viscosity. Such paints exhibit anomalous flow properties that can only be properly assessed using viscometers operating at high velocity gradients, such as that described in ISO 2884.

Resins and varnishes, however, may exhibit newtonian or near-newtonian flow at much higher viscosities and, where this applies, flow cups can provide a useful means of controlling the consistency. To meet this requirement this International Standard provides flow cups suitable for viscosities up to about 700 mm^2/s.

1 Scope and field of application

1.1 This International Standard specifies a method for the determination of the flow time of paints, varnishes and related products that may be used to control consistency. A method for the adjustment of paints to the correct application consistency at the application temperature is described in the annex.

1.2 Three flow cups of similar dimensions, but having orifice diameters of 3, 4 and 6 mm, are specified. The method for their calibration is given.

1.3 The method is limited to testing materials for which the breakpoint of the flow from the orifice of the flow cup can be determined with certainty. This point is difficult to determine and reproduce for materials with flow times in excess of 100 s due to slowing-down effects.

2 References

ISO 468, *Surface roughness — Parameters, their values and general rules for specifying requirements.*

ISO 1512, *Paints and varnishes — Sampling.*

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

ISO 2884, *Paints and varnishes — Determination of viscosity at a high rate of shear.*

3 Definitions

3.1 flow time : The elapsed time from the moment when the material under test starts to flow from the orifice of the filled cup to the moment when the flow stream of material first breaks close to the orifice.

3.2 newtonian flow : The type of flow exhibited by a material in which the ratio of the shear stress to the velocity gradient does not vary either with time or with the velocity gradient. 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 : The type of flow exhibited by a material in which, at a constant temperature, the ratio of the shear stress to the velocity gradient varies either with time or with rate of shear. 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 dynamic viscosity : The ratio of the applied shear stress to the velocity gradient.

NOTE — The SI unit for dynamic viscosity is the pascal second (Pa·s). The traditional unit is the centipoise (cP); 1 cP = 1 mPa·s.

3.5 kinematic viscosity : The ratio of the dynamic viscosity to the density of the liquid.

NOTE — The SI unit for kinematic viscosity is the square metre per second (m^2/s). The traditional unit is the centistokes (cSt); 1 cSt = 1 mm^2/s.

4 Temperature considerations

The effect of temperature on flow time is highly significant in respect of application properties and varies with the type of product.

For international reference purposes, it is essential to standardize one test temperature, and 23 ± 0,5 °C is specified in this International Standard . However, it may be more convenient to carry out comparative testing at some other agreed temperature (for example, 25 °C) because of prevailing temperature conditions.

For control by flow time, it is normal practice to condition the test sample to an agreed temperature and to ensure that the temperature variation does not exceed 0,5 °C during testing.

5 Apparatus

5.1 Flow cups

5.1.1 Dimensions

The dimensions of the ISO flow cups 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, because the flow time is inversely proportional to the fourth power of this dimension. The jet of the cup shall be made of stainless steel or sintered carbide unless otherwise specified, and the body of the cup shall be made of a material which is corrosion resistant and is 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 shall not be immediately adjacent to the jet, so as to prevent a capillary action when the material under test flows out.

5.1.3 Finish

The interior surfaces of the cups, including the orifice, shall be smooth and free from turning marks, crevices, ledges and burrs which may cause random flow or trap 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

Dimensionally similar cups will give, with newtonian liquids, similar flow times, provided that the temperature of testing is precisely the same. The use of such liquids to calibrate cups provides a useful means of initially checking that dimensionally similar cups are within the accepted tolerances of performance and also for checking from time to time whether any wear or damage has taken place sufficient to bring a cup outside the accepted tolerances.

For calibration of any particular cup, use a standard oil [2] of known kinematic viscosity and draw a graph of kinematic viscosity versus temperature from the data given by the supplier for the oil.

Using the relevant procedure described in clause 7, determine the flow time of the oil at a known temperature within the range 20 to 30 °C, measured to the nearest 0,1 °C.

Record this flow time, which should be in the range 30 to 100 s and preferably near the mid-point of this range, to an accuracy of 0,2 s.

From the prepared graph, read the kinematic viscosity at the test temperature.

Using the appropriate calibration graph of figure 2, 3 or 4, read the flow time corresponding to this kinematic viscosity.

If the two values of flow time obtained do not differ by more than 3 %, the cup may be deemed satisfactory for use.

For reference purposes, a correction factor corresponding to the flow time deviation from that obtained using the oil may be applied.

5.1.5 Marking

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

a) designation of cup : ISO 2431 No. 3, 4 or 6;

b) manufacturer's identification number;

c) manufacturer's name or trade-mark.

1) In the sense defined in ISO 468, i.e. the arithmetical mean deviation R_a from the mean line of the profile.

2) Information on suppliers of suitable oils can be obtained from national standards organizations.

5.1.6 Care and checking of flow cups

Clean the cup immediately after use and before the sample starts to dry, using a suitable solvent. Never use metal cleaning tools or wire. If the orifice becomes contaminated with dried deposits, soften these with a suitable solvent and clean carefully, for example with a soft cloth pulled through the orifice.

Check the cups periodically for wear or damage by the calibration procedure specified in 5.1.4.

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.

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 0,5 s or finer and accurate to within 0,2 % when tested over a 60 min period.

5.2.6 Temperature-controlled room or enclosure, capable of maintaining the cup and sample at a recommended, constant temperature. (See clause 4.)

6 Sampling

Take a representative sample of the material to be tested as described in ISO 1512. Examine and prepare the sample for testing as described in ISO 1513. Before testing, it is advisable to strain the sample through an appropriate sieve into a clean dry container. This is mandatory for referee purposes. 150 ml of strained material is sufficient for carrying out one test. Take care to mix the material thoroughly, while at the same time avoiding, as far as possible, loss of solvent by evaporation.

7 Procedure

7.1 Preliminary check

NOTE — This check is carried out to show that the material is suitable for the test (i.e. is newtonian or near-newtonian).

7.1.1 Choose a flow cup that will give a flow time of between 30 and 100 s for the material.

7.1.2 Determine the flow time by the procedure specified in 7.2, making sure that the material is well agitated before pouring into the cup. Remove the finger within 5 s of filling the flow cup.

7.1.3 Repeat the determination but this time allow the material to remain in the flow cup for 60 s before removing the finger.

7.1.4 If the second result differs from the first result by more than 10 %, the material shall be deemed to be non-newtonian and therefore unsuitable for consistency control by flow-time measurement.

7.2 Determination of flow time

7.2.1 Choice of flow cup

Choose a flow cup that will give a flow time between 20 and 100 s, but preferably between 30 and 100 s, for the test material.

7.2.2 Temperature adjustment

Adjust the temperature of the strained sample, and the flow cup, to 23 ± 0,5 °C, or to an alternative agreed temperature (see clause 4). If the temperature-controlled enclosure (5.2.6) is used, as recommended, it is advisable to condition the cup and the sample before straining, by placing them in the enclosure before use. The sample shall be considered ready for test immediately after any air bubbles entrained during the preparation and sieving procedures have dispersed. Carry out a final check that the temperature of the sample is within 0,5 °C of the agreed test temperature immediately prior to filling the cup.

7.2.3 Preparation of the flow cup

Place the flow cup on the stand (5.2.2), in a position free from draughts and, by using the spirit level (5.2.3) and adjusting the levelling screws of the stand, ensure that the upper rim of the flow cup is in a horizontal plane.

7.2.4 Filling the flow cup

With the orifice closed by a finger, fill the cup with the freshly strained, 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. Remove any meniscus formed either by drawing the straight-edge scraper (5.2.4) 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. Then draw this plate horizontally across the rim of the cup so that, when the plate is removed, the level of the sample coincides with the top rim of the cup.

7.2.5 Measurement of flow time

Place a suitable receiver under the flow cup so 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 (5.2.5), stopping it as soon as the first break occurs in the stream of sample close to the orifice. Record the flow time to the nearest 0,5 s.

If the test is not carried out in the temperature-controlled enclosure, place the thermometer (5.2.1) in the stream of sample so as not to interfere with observation of the break in the flow.

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 and not less than 100 mm from the orifice. It is convenient to use the same thermometer as is used to adjust the temperature of the sample initially. Any difference in temperature from the initially adjusted temperature shall not be greater than 0,5 °C.

7.2.6 Repeat determinations

Carry out a second determination on another portion of the originally prepared sample and check carefully that the temperature of testing is within the prescribed limits. Record the flow time to the nearest 0,5 s. Calculate the mean of the two determinations.

If the two determinations differ by more than 5 %, carry out a third determination. If the third determination and either of the previous determinations do not differ by more than 5 %, discard the other determination. Calculate the result as the mean of the two accepted determinations.

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.

8 Precision

The precision of the method, as obtained by statistical examination of interlaboratory test results, is a follows :

8.1 Repeatability (r)

The difference between two results (each the mean of two accepted determinations) obtained by the same operator with the same apparatus under constant operating conditions on identical test material shall, at the 95 % confidence level, not exceed 5 %.

8.2 Reproducibility (R)

The difference between two results (each the mean of two accepted determinations) obtained by different operators in different laboratories on identical test material shall, at the 95 % confidence level, not exceed 10 %.

9 Test report

The test report shall include at least the following information :

a) the type and identification of the product tested;

b) a reference to this International Standard (ISO 2431) and to the designation (No. 3, 4 or 6) of the cup used;

c) the manufacturer's identification number of the flow cup used;

d) the temperature of testing;

e) the flow time (for referee purposes, individual values should also be reported);

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

g) the date of the test.

Annex

Use of flow cups for the adjustment of paint consistency

A.1 Introduction

In certain circumstances it may be necessary to adjust the consistency of paint to the required flow time at the temperature of application.

Since paint consistency varies with temperature, it would be helpful if manufacturers of a paint supplied a chart showing the relationship of temperature to the quantity of thinner to be added to obtain a desired consistency.

A.2 Use of flow cups to determine the quantity of thinner required to adjust a paint to a specified flow time

A.2.1 The application consistency of paints is readily adjusted by addition of thinner to give the required flow time at the temperature of application. When the application conditions are known to vary over a range of temperatures, such as seasonal variations or in different work places, the volume ratios of thinner to paint can be predetermined and expressed graphically or in tabular form for the convenience of the applicator. Even so, the actual flow time should be checked and adjustment made if necessary, just prior to application.

A.2.2 When thinning paint to a specified consistency, the user should have either

a) a graph or table indicating the volume ratio of thinner to paint required to give a predetermined flow time depending on temperature variations at the work place, or

b) information from the supplier indicating the application flow time and the approximate thinning ratio.

A.2.3 The flow cup specified should give, for the material under test, a flow time in excess of 20 s to allow a sufficient degree of accuracy in the test.

A.2.4 Thin a representative sample of the bulk paint with the appropriate volume of thinner and stir until it is well mixed.

A.2.5 Place the clean flow cup in the stand provided and ensure that the top of the cup is level when it is seated in the stand.

A.2.6 With the orifice closed by the finger, fill the cup with the thinned paint until it flows evenly over the rim of the gallery. (If the cup is level, the overflow will be even over the rim.)

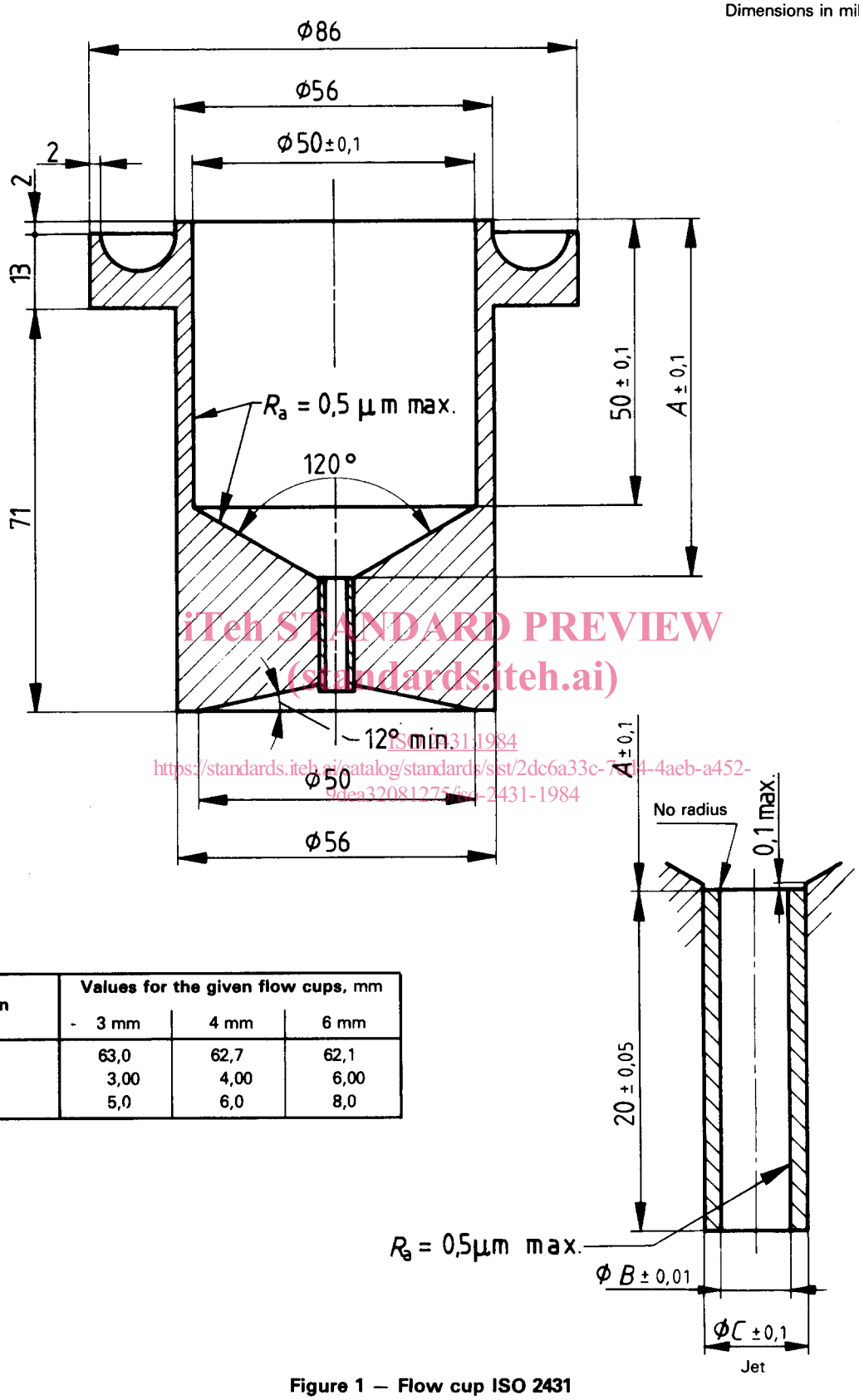
A.2.7 Remove the finger from the orifice and simultaneously start the timing-device, stopping it as soon as the first break occurs in the stream of paint close to the orifice. Record the flow time to the nearest second.

Add more thinner if the flow time is too long or more paint if the flow time is too short. After each addition, mix the thinned paint well and redetermine the flow time until the required limits are met.

A.3 Test report

The test report shall include the required ratio of thinner to paint and, if required, items a), b), c), f), and g) of clause 9, together with a record of the temperature of test, to the nearest 0,5 °C, and the flow time, reported to the nearest second.

Dimensions in millimetres



Dimension	Values for the given flow cups, mm		
	3 mm	4 mm	6 mm
A	63,0	62,7	62,1
B	3,00	4,00	6,00
C	5,0	6,0	8,0

Figure 1 — Flow cup ISO 2431

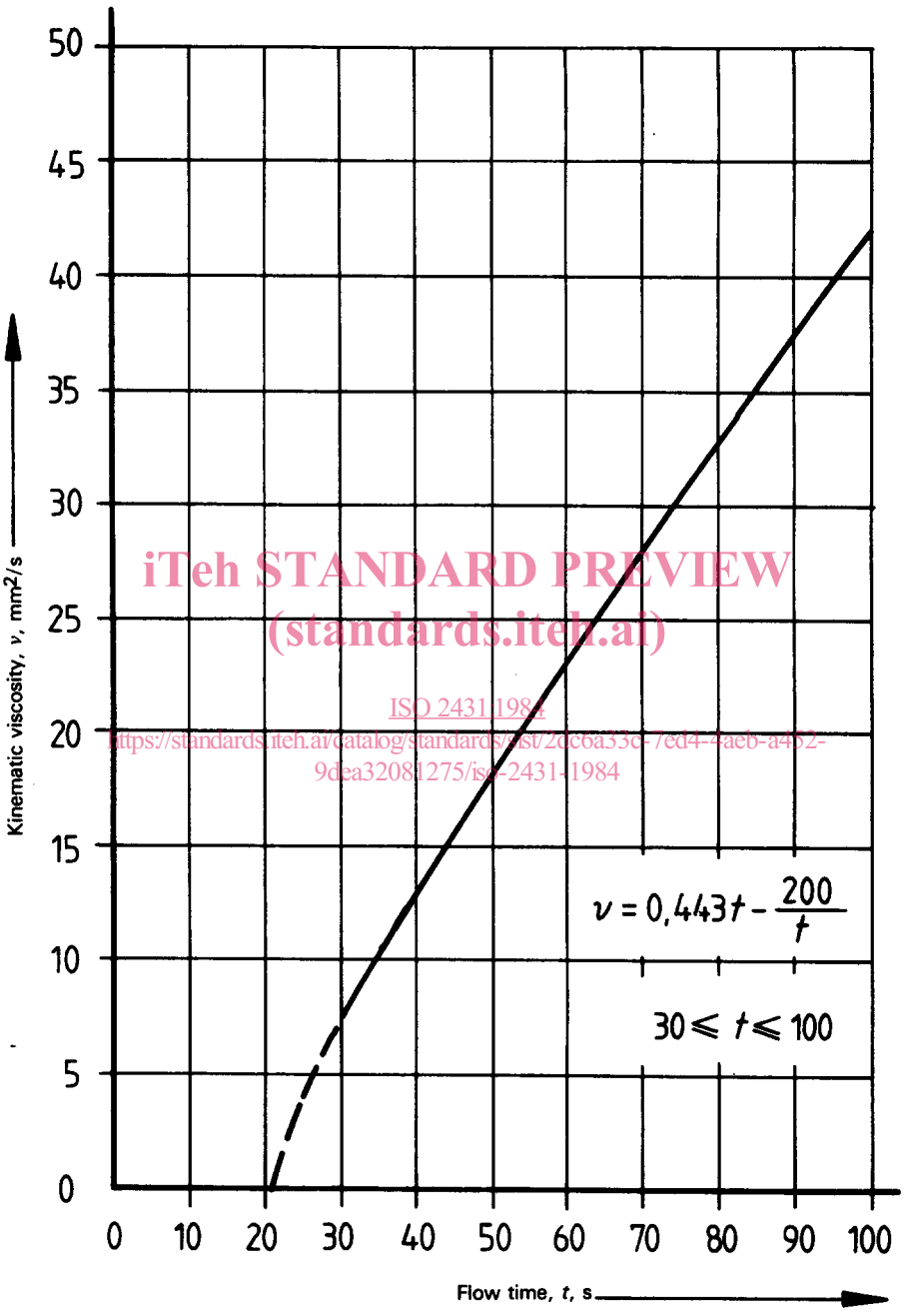


Figure 2 — Calibration curve for 3 mm cup