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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

ISO 2431:2019(E)

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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 preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established 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. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 35, *Paints and varnishes*, Subcommittee SC 9, *General test methods for paints and varnishes*.

This sixth edition cancels and replaces the fifth edition (ISO 2431:2011), which has been technically revised. The main changes compared to the previous edition are as follows:

— a general reference to ISO 4618 has been added in Clause 3;

— the information in Clause 4 on measuring at other temperatures and humidities specified in this document has been amended;

— Figure 1 has been corrected;

— information on conduction of measurements in a fume cupboard has been added to Clause 4.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Introduction

The first edition of this document, ISO 2431, published in 1972, specified only one flow cup with an orifice diameter of 4 mm. The second edition specified three flow cups with an orifice diameter of 3 mm, 4 mm and 6 mm. The third edition corrected errors in Figures 2 and 4 and the formulae for those figures. The fourth edition specified four flow cups with an orifice diameter of 3 mm, 4 mm, 5 mm and 6 mm. In the fifth edition the curves in Figures 2 to 5 have been placed in a single figure (Figure 2) and the formulae for the conversion of flow time to kinematic viscosity and vice versa represented by the curves in these figures have been moved from the figures to a table (Table 1). The procedure for checking the flow cups for wear and tear has been revised to include two alternative methods (one using a certified reference material or secondary working standard, the other using a certified flow cup) and has been moved to an informative annex. The main changes made in this sixth edition are given in the foreword.

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 of the role of flow cups for the measurement of the flow time of paints, varnishes and related products.

Flow times are a measure for sample viscosity only for products with Newtonian or near-Newtonian flow properties. This effectively limits the practical use of flow cups. 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 thickening agents to ensure increased viscosity. Such paints exhibit non-Newtonian flow properties. Their viscosity during application can only be properly assessed using viscometers such as that described in ISO 3219.

Resins and varnishes can exhibit Newtonian or near-Newtonian flow at much higher viscosities than most paints and, where this applies, flow cups can provide a useful means of controlling the consistency. As a result, this document provides flow cups suitable for viscosities up to about 700 mm^2/s.

With thixotropic materials, stirring or other such mechanical stress immediately before testing will reduce the flow time compared with that for an unstirred sample. With such materials, uncertain and variable flow time values are obtained with all the flow cups. The repeatability and reproducibility limits given in Clause 9 cannot be achieved in the determination of the flow time of such materials.

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

1 Scope

This document specifies a method for determining the flow time of paints, varnishes and related products that can be used to control consistency.

Four flow cups of similar dimensions, but having orifice diameters of 3 mm, 4 mm, 5 mm and 6 mm, are specified. Two methods for checking the flow cups for wear and tear are given (see Annex A).

Flow cups with a replaceable jet are not covered by this document as the close tolerances on the supply of the material under test to the jet are not met.

Commonly used dipping flow cups are also not covered by this document.

NOTE   Since the fabrication tolerances for such flow cups are greater than those of the flow cups specified in this document, flow time determinations with dipping flow cups give a precision which is lower than that obtained with the flow cups specified in this document (see Clause 9).

The method described in this document 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 near the upper limit of the measurement range (100 s) due to slowing-down effects.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 1513, *Paints and varnishes — Examination and preparation of test samples*

ISO 4618, *Paints and varnishes — Terms and definitions*

ISO 15528, *Paints, varnishes and raw materials for paints and varnishes — Sampling*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 4618 and the following apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at http://www.electropedia.org/

3.1
flow time
t

time that elapses from the moment when the material under test starts to flow from the orifice of the filled flow cup to the moment when the flow stream of material first breaks off close to the orifice

3.2
Newtonian flow
behaviour where the shear viscosity is independent of shear rate, shear stress and time

3.3
non-Newtonian flow
behaviour where the shear viscosity is dependent either on shear rate and shear stress or on shear rate, shear stress and time

3.4
kinematic viscosity
ν
ratio of shear viscosity and density

Note 1 to entry: The kinematic viscosity ν has the unit square metres per second $(m^2 \cdot s^{-1})$.

4 Temperature considerations

Temperature and humidity are important parameters affecting test results. Deviations from the requirements specified can lead to results that are not comparable. However, the interested parties may agree upon alternative parameters and these parameters shall be reported.

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

For reference purposes, (23,0 ± 0,5) °C is specified as the test temperature in this document. However, it might be more convenient to carry out comparative testing at some other agreed temperature (for example, 25 °C) because of prevailing temperature conditions (see also Annex B).

For control by flow time, the test sample and flow cup shall be conditioned to an agreed or specified temperature and it shall be ensured that the temperature variation does not exceed 0,5 K during testing. The flow cup shall be in a place which is free from draughts.

If testing is conducted in a fume cupboard and the air suction is left on, this shall be noted in the test report.

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 shown in Figure 1.

NOTE   The most critical tolerance is the internal diameter of the jet of the flow cup, because the flow time is inversely proportional to the fourth power of this dimension.

5.1.2 Material

The jet of the flow cup shall be made of stainless steel or sintered carbide, and the body of the flow cup shall be made of a material which is corrosion-resistant and is not affected by the products to be tested.

5.1.3 Construction

The dimensions not specified, such as wall thickness, shall be such that no distortion of the flow cup can occur in use. The external shape should preferably be as shown in Figure 1, but may be modified for convenience of use, or manufacture, provided that the protruding jet of the flow 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 any capillary action when the material under test flows out.

Flow cups having an additional jacket for temperature control are preferred.

5.1.4 Finish

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

The standard of finish required is equivalent to a maximum roughness Ra (as defined in ISO 4287) of not more than 0,5 μm.

Dimensions in millimetres
Roughness in micrometres

Key

1   jet

a   Sharp edge (not rounded).
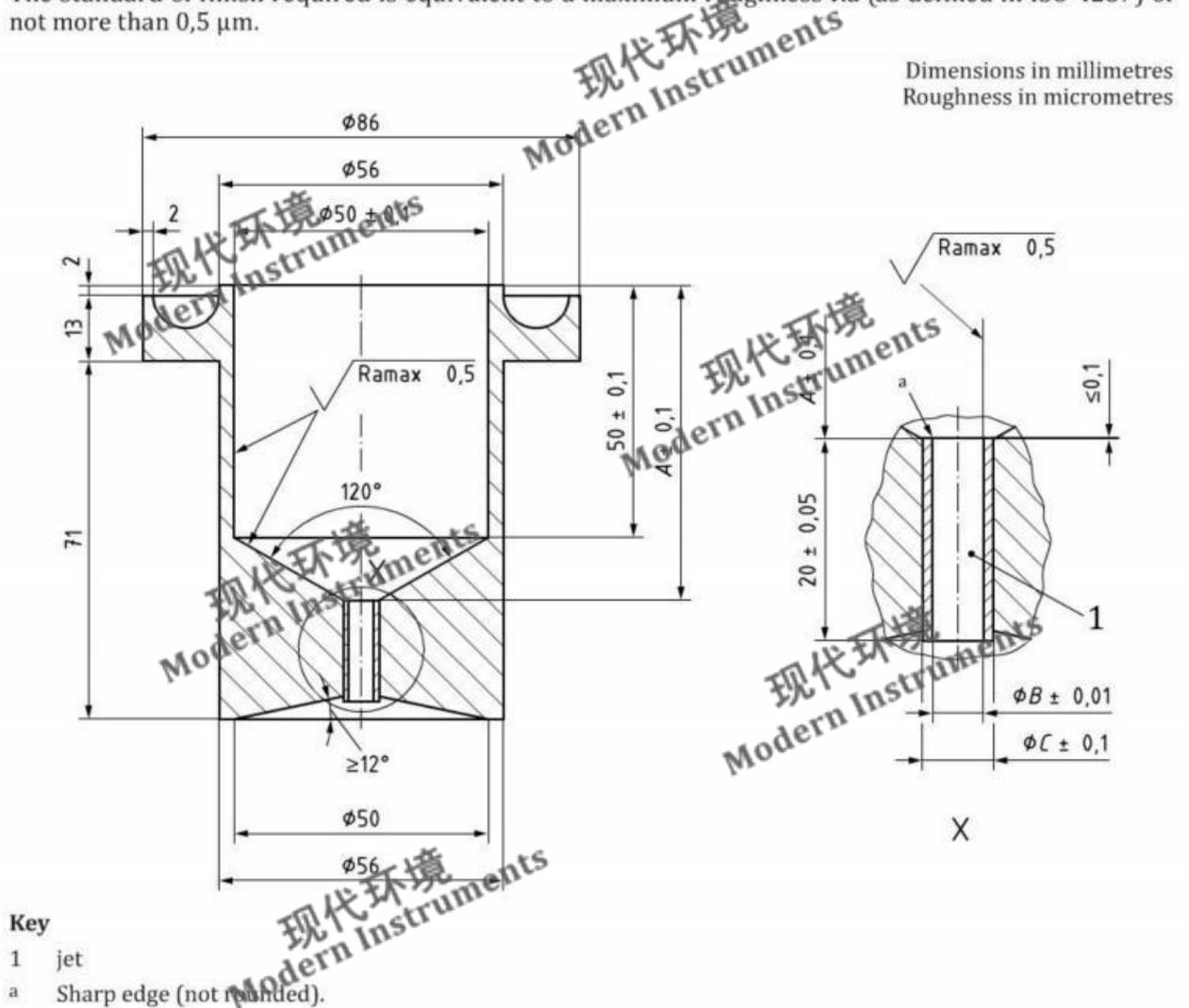
Dimension	Values[b] for the flow cups given			
	3-mm (No 3) flow cup	4-mm (No 4) flow cup	5-mm (No 5) flow cup	6-mm (No 6) flow cup
A	63	62,7	62,4	62,1
B	3	4	5	6
C	5	6	7	8
[b] For tolerances, see the enlarged section of the jet.				

Figure 1 — Flow cup

5.1.5 Measurement range

Flow cups shall be used within the measurement range given in Table 1. Meaningful data can only be obtained in this range. In addition, the conversion of flow time to kinematic viscosity and *vice versa*, shall be carried out using the formulae given in Table 1.

Table 1 — Measurement range of flow cups and conversion of flow time to kinematic viscosity and *vice versa*

Flow cup	Flow time, t s	Kinematic viscosity, v mm²/s	Measurement range s
No 3	$t = \dfrac{v}{0,89} + \sqrt{451,5 + \left(\dfrac{v}{0,89}\right)^2}$	$v = 0,443 \times t - \dfrac{200}{t}$	$30 \le t \le 100$
No 4	$t = \dfrac{v}{2,74} + \sqrt{146,0 + \left(\dfrac{v}{2,74}\right)^2}$	$v = 1,37 \times t - \dfrac{200}{t}$	$30 \le t \le 100$
No 5	$t = \dfrac{v}{6,56} + \sqrt{67,1 + \left(\dfrac{v}{6,56}\right)^2}$	$v = 3,28 \times t - \dfrac{200}{t}$	$30 \le t \le 100$
No 6	$t = \dfrac{v}{13,8} + \sqrt{82,6 + \left(\dfrac{v}{13,8}\right)^2}$	$v = 6,90 \times t - \dfrac{570}{t}$	$30 \le t \le 100$

The curves corresponding to the formulae given in Table 1 are plotted in Figure 2.

NOTE   These curves are given for information only.

Key

1   3-mm flow cup
2   4-mm flow cup
3   5-mm flow cup
4   6-mm flow cup
t flow time, in seconds
v kinematic viscosity, in square millimetres per second
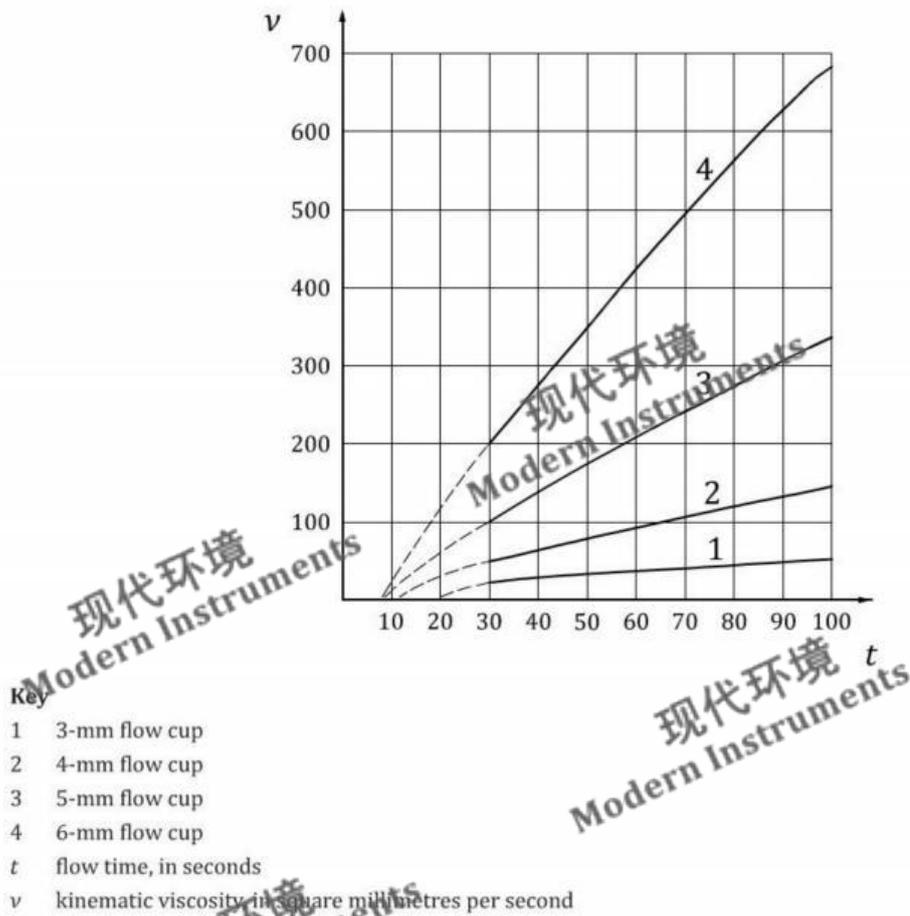
Figure 2 — Conversion curves for 3-mm, 4-mm, 5-mm and 6-mm flow cups

5.1.6   Marking

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

a)   designation of flow cup:

Flow cup ISO 2431, No 3, No 4, No 5 or No 6;

b)   manufacturer's identification number;

c)   manufacturer's name or trade mark.

5.1.7   Care and checking of flow cups

Clean the flow cup immediately after use and before the sample starts to dry, using a suitable solvent. Never use metal tools or a wire scourer for cleaning purposes. 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 flow cups periodically for wear and tear using one of the procedures specified in Annex A.

5.2 Supplementary apparatus

5.2.1 Thermometer, graduated at intervals of 0,2 °C or finer.

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 with rounded edges or **straight-edge scraper.**

5.2.5 Stopwatch, or other suitable timing-device, with scale divisions of 0,2 s or finer.

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

NOTE   This will not be needed if the flow cup has a jacket for temperature control.

6 Sampling

Take a representative sample of the material to be tested, as described in ISO 15528. Examine and prepare the sample for testing, as described in ISO 1513.

A sample of 150 ml 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 for Newtonian flow

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

7.1.2   Determine the flow time as specified in 7.2.

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 used to close off the orifice (see 7.2.4).

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 of between 30 s and 100 s for the test material.

7.2.2 Temperature adjustment

Adjust the temperature of the sample, and the flow cup, to (23,0 ± 0,5) °C, or to an alternative agreed temperature (see Clause 4).

If the temperature-controlled enclosure (5.2.6) is used, it is advisable to condition the flow cup and the sample by placing them in the enclosure before use.

The sample shall be considered ready for test immediately after all the air bubbles entrained during the preparation procedure have dispersed. Carry out a final check that the temperature of the sample is within 0,5 K of the agreed test temperature immediately prior to filling the flow 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 flow cup with the well stirred, bubble-free sample, pouring slowly to avoid the formation of air bubbles. If bubbles are formed, allow them to rise to the surface and remove them.

NOTE   If the flow 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 (see 5.2.4) over the entire rim of the flow 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, subsequently drawing this plate horizontally across the rim of the flow cup so that, when the plate is removed, the level of the sample coincides with the top rim of the flow 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 rounded flow time to the nearest 0,5 s.

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 outlier. 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 non-Newtonian flow behaviour, and consideration shall be given to other methods of test, e.g. to measuring the viscosity using a rotational viscometer.

8   Marking of products tested

The product tested may be marked with a marking indicating the results of the test.

If such a marking is made, it shall comprise a reference to this document, the designation number of the flow cup used and the flow time, in seconds. For example,

$$\text{ISO } 2431 - 5 - 65$$

where

ISO 2431   is the number of this document;

5   is the designation number of the flow cup used;

65   is the flow time, in seconds.

9   Precision

9.1   General

An interlaboratory test was planned and executed and the results evaluated, in accordance with ISO 5725-2. 11 laboratories participated in the test. Liquids were tested using 4 mm and 6 mm flow cups. Except for the varnish when tested using the 6 mm flow cup, the liquids showed Newtonian flow behaviour. The flow behaviour of the varnish when tested using the 6 mm flow cup was nearly Newtonian, but, when the preliminary check specified in 7.1 was carried out, the second result did not differ from the first result by more than 10 % (see 7.1.4).

9.2   Repeatability limit, *r*

The value below which the absolute difference between two single test results, each the mean of duplicates, obtained on identical material by one operator in one laboratory within a short interval of time using the standardized test method, might be expected to lie with a 95 % probability is approximately 2 s.

Details of the results are given in Table 2.

Table 2 — Repeatability limit, *r*

Test liquid	Orifice diameter	Mean value of flow time	Repeatability limit *r*
	mm	s	
Engine oil	4	5?	1,7
Varnish, based on organic solvents	4	56	1,7
Engine oil	6	4?	2,6
Varnish, based on organic solvents	6	43	1,7

9.3   Reproducibility limit, *R*

The value below which the absolute difference between two single test results, each the mean of duplicates, obtained by operators on identical material in different laboratories using the standardized test method, might be expected to lie with a 95 % probability is approximately 3 s for the 4 mm flow cup and approximately 6 s for the 6 mm flow cup.

Details of the results are given in Table 3.

Table 3 — Reproducibility limit, R

Test liquid	Orifice diameter	Mean value of flow time	Reproducibility limit R
	mm	s	s
Engine oil	4	55	2,2
Varnish, based on organic solvents	4	56	3,2
Engine oil	6	60	3,9
Varnish, based on organic solvents	6	43	5,5

10 Test report

The test report shall include at least the following information:

a) all details necessary to identify the product tested;

b) a reference to this document (ISO 2431:2019) and to the designation (No 3, No 4, No 5 or No 6) of the flow 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 shall also be reported);

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

g) any unusual features (anomalies) observed during the test;

h) the date of the test.

Annex A
(normative)

Checking flow cups for wear and tear

A.1 General

To monitor wear and tear, flow cups should be checked regularly with one of the two methods in defined intervals, e.g. every six months. In method A, the flow cup is checked by using a certified reference material (CRM) or reference material (RM) (see ISO Guide 30). In method B, the flow cup is checked against a certified flow cup.

A.2 Standards required

A.2.1 **Certified Reference Material (CRM)**, comprising a standard Newtonian oil of known kinematic viscosity and known shelf life.

A.2.2 **Reference Material (RM)**, comprising e.g. a commercially available engine oil or other substance with a kinematic viscosity determined by testing representative subsamples at least three times, utilizing an instrument previously verified using a CRM, statistically analysing the results and, after the removal of any outliers, calculating the arithmetic mean of the results.

Store RMs in containers which will retain the integrity of the RM, out of direct sunlight, at a temperature not exceeding 10 °C.

An RM may be used up to three times. It shall then be tempered and allow to stand for devolatilization for at least 3 h.

A.2.3 **Flow cup**, certified.

A.3 Method A — Checking using a CRM or RM

To check a particular flow cup, use a CRM or an RM of known kinematic viscosity at (23 ± 0,2) °C. Choose a CRM or RM with a flow time which is in the range 30 s to 100 s, and preferably near the midpoint of this range, for the flow cup concerned.

Condition the CRM or RM, and the flow cup being checked, at (23 ± 0,2) °C for at least 2 h. Determine the flow time of the CRM or RM, following the procedure specified in Clause 7, to the nearest 0,2 s.

Carry out the determination three times.

Calculate the mean value of the three determinations and, using the appropriate formula in Table 1, the kinematic viscosity of the liquid as given by the flow cup.

Calculate the deviation between the certified and the measured viscosity using Formula (A.1):

$$\Delta v = \frac{(v_m - v_{ce}) \times 100}{v_{ce}} \tag{A.1}$$

where

Δv is the deviation between the certified and the measured viscosity, expressed as a percentage;

v_m is the viscosity calculated from the flow time measured, in square millimetres per second;

v_{ce} is the viscosity of the CRM or RM, in square millimetres per second.

If the two values of the calculated kinematic viscosity obtained do not differ by more than 3 %, the flow cup is deemed to be satisfactory for use.

NOTE   When using an RM, instead of calculating the kinematic viscosity the flow times measured can be used directly to calculate the deviation, expressed as Δt [see Formula (A.2)].

A.4   Method B — Checking using a certified flow cup

To check a particular flow cup, use a certified reference flow cup of the same type. Choose an RM with a flow time which is in the range 30 s to 100 s, and preferably near the midpoint of this range, for the flow cup concerned.

Condition the certified reference flow cup, the flow cup being checked and the RM in a temperature-controlled enclosure at a temperature between 20 °C and 25 °C for at least 2 h. During this conditioning and the subsequent flow time measurement, the temperature shall remain constant to within ±0,2 °C. Determine the flow time of the RM, following the procedure specified in Clause 7, to the nearest 0,2 s.

Carry out the determination three times. Calculate the result as the mean of the three determinations.

Calculate the deviation between the flow time given by the certified reference flow cup and that given by the flow cup being checked, using Formula (A.2):

$$\Delta t = \frac{(t_{ch} - t_{ce}) \times 100}{t_{ce}} \tag{A.2}$$

where

Δt is the deviation between the flow time given by the certified flow cup and that given by the flow cup being checked, expressed as a percentage;

t_{ch} is the flow time determined with the flow cup being checked, in seconds;

t_{ce} is the flow time determined with the certified reference flow cup, in seconds.

If the two values of the determined flow times obtained do not differ by more than 3 %, the flow cup is deemed to be satisfactory for use.

Annex B
(informative)

Conversion of flow times from one temperature to another

If the flow time cannot be measured at the specified temperature, it is necessary to convert it from the measurement temperature. This annex describes an interpolation method for the conversion. The method is not suitable for the extrapolation of data outside the defined temperature range.

Vogel's equation is a well-proven and frequently used approximation of the viscosity/temperature behaviour of paints. It is given, for flow times, by Formula (B.1):

$$\ln t = A + \frac{B}{T+C} \tag{B.1}$$

in which the constants A, B and C are given by Formula (B.2) to (B.4):

$$C = \frac{(\ln t_1 - \ln t_3)(T_1 - T_2)T_3 - (\ln t_1 - \ln t_2)(T_1 - T_3)T_2}{(\ln t_1 - \ln t_3)(T_1 - T_3) - (\ln t_1 - \ln t_3)(T_1 - T_2)} \tag{B.2}$$

$$A = \frac{(T_1 + C)\ln t_1 - (T_2 + C)\ln t_2}{(T_1 - T_2)} \tag{B.3}$$

$$B = (T_2 + C)(\ln t_2 - A) \tag{B.4}$$

where

T is the temperature, in degrees Celsius;

T_1 is the lower temperature limit, in degrees Celsius;

T_2 is the temperature in the middle of the range, in degrees Celsius;

T_3 is the upper temperature limit, in degrees Celsius;

t_1 is the flow time, in seconds, at temperature T_1;

t_2 is the flow time, in seconds, at temperature T_2;

t_3 is the flow time, in seconds, at temperature T_3.

The conversion of flow times from one temperature to another only gives reliable results if

— the chosen temperature range $T_3 - T_1$ is ≤20 °C;

— the conversion does not involve extrapolation outside the defined temperature range.

If the constants A, B and C are known (for an example, see Table B.1), the interpolation data within the interval from T_1 to T_3 can be calculated using Formula (B.1) and tabulated (for an example, see Table B.2). Additionally, the data can be plotted as an interpolation curve (for an example, see Figure B.1).

Table B.1 — Calculation of the constants A, B and C

Temperature °C		Flow time s		Calculated values of A, B and C	
T_1	16	t_1	62	A	3,641
T_2	23	t_2	54	B	8,552
T_3	30	t_3	50	C	1,609

Table B.2 — Interpolated values of flow time between the three base values

Temperature °C	Flow time s
16	62
17	60,4
18	59,0
19	57,8
20	56,7
21	55,7
22	54,8
23	54,0
24	53,3
25	52,6
26	52,0
27	51,4
28	50,9
29	50,4
30	50,0
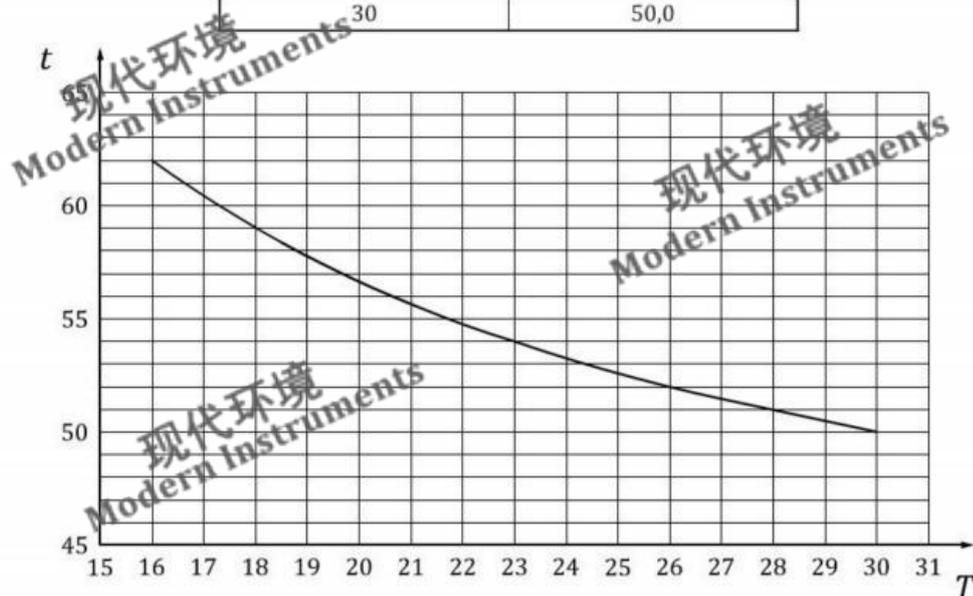
Key

t flow time, in seconds

T temperature, in degrees Celsius

Figure B.1 — Example of relationship between flow-time t and temperature T

Bibliography

[1]   ISO 3219, *Plastics — Polymers/resins in the liquid state or as emulsions or dispersions — Determination of viscosity using a rotational viscometer with defined shear rate*

[2]   ISO 4287, *Geometrical Product Specifications (GPS) — Surface texture: Profile method — Terms, definitions and surface texture parameters*

[3]   ISO 5725-2, *Accuracy (trueness and precision) of measurement methods and results — Part 2: Basic method for the determination of repeatability and reproducibility of a standard measurement method*

[4]   ISO Guide 30, *Reference materials — Selected terms and definitions*