

Paints, varnishes and similar coating materials

Determination of flow time using the DIN flow cup

DIN
53 211

Lacke, Anstrichstoffe und ähnliche Beschichtungstoffe;
Bestimmung der Auslaufzeit mit dem DIN-Becher

Supersedes April 1974 edition.

In keeping with current practice in standards published by the International Organization for Standardization (ISO), a comma has been used throughout as the decimal marker.

1 Scope and field of application

The determination of the flow time of paints¹⁾, varnishes¹⁾ and similar coating materials¹⁾ serves to obtain a characteristic value which may be used as a simple aid to assess the flow behaviour and the applicability of such products.

The DIN flow cup is not suitable for determining flow times of less than 25 seconds (corresponding to a kinematic viscosity of about 90 mm²/s), for which ISO 2431 flow cups are to be used, nor for flow times exceeding 150 seconds (corresponding to a kinematic viscosity of about 700 mm²/s).

2 Concept

The flow time is the time, in seconds, determined as specified in this standard, that the product under test requires to flow from the flow cup.

The flow time is reported as a characteristic value followed by 'DIN seconds', e.g. 53 DIN seconds.

3 Sampling and sample preparation

A representative sample, free of impurities, shall be taken from the product to be tested as specified in DIN 53 225. It shall be introduced into a sample container which shall then be tightly sealed. Determination of the flow time shall not begin until the sample is bubble-free.

See subclause 6.1 for the temperature of the sample and flow cup.

4 Number of determinations

The flow time shall be determined twice, each time using a new sample from the sample container.

5 Apparatus

5.1 DIN flow cup²⁾

5.1.1 The flow cup specified in this standard shall have a capacity of (100 ± 1) ml and a fixed jet (see figure 1; the dimensions given in the illustration are guideline values for the cup manufacturer). It shall be made of a material that is not affected by the products to be tested. Unless otherwise agreed, the material used shall be an aluminium alloy with an anodized surface.

5.1.2 The jet (see figure 1) shall be made of stainless steel, its interior surface shall be polished. It shall have an inside diameter of 4 mm (with a JS 9 tolerance)³⁾ and shall be (4 ± 0,02) mm in length, the radius of curvature of the rim being at least 0,2 mm and no greater than 0,4 mm.

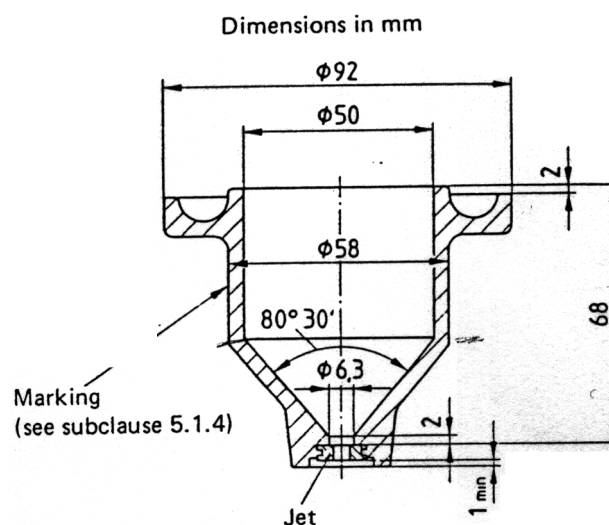


Figure 1. DIN 53 211 - 4 flow cup

Designation of the DIN flow cup for determining the flow time, with a jet inside diameter of 4 mm:

Flow cup DIN 53 211 - 4

5.1.3 The flow cup, when filled with oils of known kinematic viscosity (see note to clause 8), and with the temperature maintained to the nearest 0,1 °C, shall give flow times that do not deviate by more than 3% from the calibration graph shown in figure 2.

¹⁾ See DIN 55 945 for the definitions of 'paint', 'varnish' and 'coating material'.

²⁾ Information on sources of supply is obtainable from *Bezugsquellen für normgerechte Erzeugnisse im DIN*, Burggrafenstraße 6, D-1000 Berlin 30.

³⁾ Tolerance JS 9 means a permissible deviation of ± 0,015 mm; see DIN 7161.

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The calibration graph is defined by the following equations:

$$v = 4,57 t - 452/t; \text{ and } t = \frac{v}{9,14} + \sqrt{98,9 + \left(\frac{v}{9,14}\right)^2}$$

where

v is the kinematic viscosity, in mm^2/s ;

t is the flow time, in seconds.

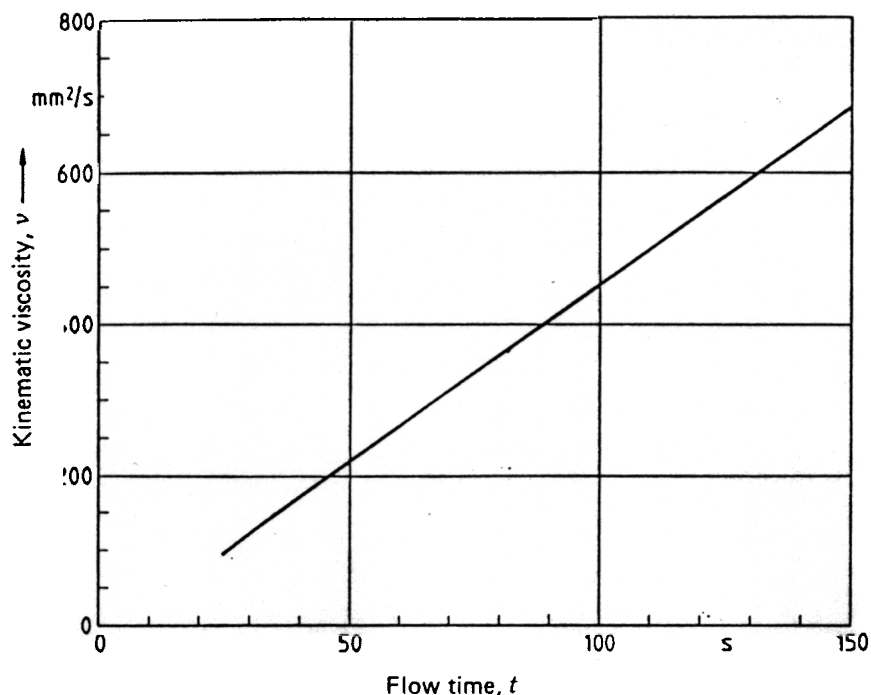


Figure 2. Calibration graph for DIN 53 211 – 4 flow cup

The equations are valid for temperatures between 15 °C and 25 °C.

5.1.4 Only flow cups that have been checked for compliance with this standard by the manufacturer shall be permanently marked (see figure 1) with the information given in figure 3, i.e. manufacturer's name, manufacturer's identification number and year of manufacture.

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Figure 3. Inscription

Proof of compliance with the specifications of this standard shall be provided by a test certificate as specified in DIN 50 049.

5.2 Thermometer

A thermometer with scale intervals of not less than 0,2 °C, e.g. precision thermometer as specified in DIN 12 775, shall be used.

5.3 Stand.

5.4 Glass plate with rounded (ground) edges.

5.5 Timing device

Stopwatch with a scale division of 0,2 seconds or less, with a limit of error of 0,1 % over a 60 minute period, or electronic timer with the same accuracy.

6 Procedure

6.1 The temperature of the sample prepared as described in clause 3 and of the flow cup shall, unless otherwise agreed (e.g. 20 °C) be $(23 \pm 0,5)$ °C before and during measurement of the flow time. Where other temperatures and limit deviations are agreed upon (e.g. in conditions of delivery or acceptance specifications), they shall be indicated together with the test result (see clause 7).

Note. A test temperature of $(20 \pm 0,5)$ °C has been specified for the flow times given in the *Verordnung über brennbare Flüssigkeiten* (German Regulation on combustible liquids). If the samples meet the minimum flow time requirement at 23 °C, it may be assumed that they will also meet this requirement at 20 °C, conversion of the flow times measured at 23 °C to their equivalent at 20 °C being only possible if the dependence of the flow time of the product to be tested on the temperature is known. As the flow time is largely a function of the temperature of the sample, great care shall be taken to ensure that the specified temperature is maintained throughout the

test. The use of a temperature-controlled enclosure facilitates maintenance of the flow cup at constant temperature.

6.2 Secure the flow cup to the stand so that its upper rim is in a horizontal plane. Close the orifice with a finger and pour the bubble-free sample into the cup so that it flows over the inner rim of the cup, preventing any air bubbles to form in the sample. Then, slide the glass plate over the rim so that no air bubbles form between the glass and the surface of the sample. Then draw the glass plate horizontally across the rim of the cup and remove it scraping off any liquid sticking to it by means of the rim.

6.3 Remove the finger from the orifice and simultaneously start to measure the flow time; stop as soon as the first break occurs in the stream of sample close to the orifice.

Note. Clean the flow cup (particularly the jet) carefully immediately after the test and before the sample begins to dry, using a soft cloth or a soft brush and a suitable solvent. Never use metal or hard cleaning tools. Testing in compliance with the requirements of this standard is only possible if the flow cup is clean and undamaged and the jet is in perfect order.

7 Test report

The test report shall include at least the following information:

- a) type and designation of product tested;
- b) a reference to this standard;
- c) test temperature, reported to the nearest 0,2 °C;
- d) flow time:
 - mean value (rounded to the nearest whole number), e.g. '53 DIN seconds'; in cases of dispute, individual

values (to the nearest 0,2 seconds) shall also be reported;

- e) deviations from mean value if greater than 2 %;
- f) any deviation, by agreement or otherwise, from the test procedure described;
- g) date of test.

8 Precision

(as specified in DIN 51 848 Part 1)

Repeatability: 4 %.

Reproducibility: 10 %.

Note. The repeatability has been limited to 4 % because of the limit deviation of $\pm 0,5$ °C specified for the temperature and the temperature dependence of the viscosity indicated in subclause 6.1. This limit can be met without difficulty if the flow behaviour of the product to be tested is normal. The reproducibility has been fixed at 10 % in consideration of the limit deviation for the DIN flow cup characteristic from the calibration graph of ± 3 %. Greater scatter of the test results may be due to evaporation of volatile components or anomalous flow of the product under test (non-newtonian behaviour, thixotropy) (see also Explanatory notes). If the flow time is not to be determined in conjunction with the *Verordnung über brennbare Flüssigkeiten*, it is recommended to determine the flow behaviour of thixotropic liquids using a rotating viscometer.

9 Testing for referee purposes

Only calibrated DIN 53 211 – 4 flow cups and verified thermometers shall be used for tests for referee purposes.

Standards and other documents referred to

DIN 1451 Part 4	Typefaces; grotesque; lettering for engraving stencils and templates
DIN 7161	ISO tolerances for internal dimensions (holes), for nominal sizes from 1 to 500 mm
DIN 12 775	Laboratory glassware; laboratory thermometers with 0,1 °C, 0,2 °C and 0,5 °C scale intervals
DIN 50 049	Materials testing certificates
DIN 51 848 Part 1	Testing of petroleum products; precision; general, concepts and their application to petroleum standards specifying requirements
DIN 53 225	Testing of paints and varnishes; sampling
DIN 55 945	Paints, varnishes and similar coating materials; terminology
ISO 2431	Paints, varnishes and similar coating materials; determination of flow time by use of flow cups
<i>Verordnung über Anlagen zur Lagerung, Abfüllung und Beförderung brennbarer Flüssigkeiten zu Lande</i> (German Regulation on installations for the storage, filling and transport by land of combustible liquids), as of 3 May 1982, <i>BGBI.</i> (German Federal Law Gazette) I, 569.	

Other relevant documents

Internationale Verordnung für die Beförderung gefährlicher Güter mit der Eisenbahn (International Regulation on the transport of dangerous goods by rail) – Annex I.

Europäisches Übereinkommen über die internationale Beförderung gefährlicher Güter auf der Straße (European Agreement on the international transport of dangerous goods by road) – Annexes A and B.

International Maritime Dangerous Goods Code (Recommendations issued by the Intergovernmental Maritime Consultative Organizations).

Transport of Dangerous Goods Recommendation prepared by the United Nations

Verordnung über die innerstaatliche und grenzüberschreitende Beförderung gefährlicher Güter auf Straßen (German Regulation on the transport of dangerous goods by road within and across national borders), as of 22 July 1985, BGBl. I, 1550.
 Verordnung über die innerstaatliche und grenzüberschreitende Beförderung gefährlicher Güter mit Eisenbahnen (German Regulation on the transport of dangerous goods by rail within and across national borders), as of 22 July 1985, BGBl. I, 1560.

Verordnung über die Beförderung gefährlicher Güter mit Seeschiffen (German Regulation on the transport of dangerous goods by sea), as of 27 June 1986, BGBl. I, 961.

Previous editions

DIN 53 211: 07.41, 06.62x, 12.70, 04.74.

Amendments

The following amendments have been made to the April 1974 edition:

- a) reference has been made to the flow cups specified in ISO 2431 for measuring flow times not lying within the range 25 to 150 seconds;
- b) a graph has been included comparing the measuring ranges of the flow cups specified in DIN 53 211 and ISO 2431;
- c) information on the temperature range within which the calibration graph is valid has been included;
- d) the standard has been editorially revised and updated.

Explanatory notes

This standard has been prepared by Technical Committee 7 Anstrichstoffe und ähnliche Beschichtungsstoffe of the Normenausschuß Anstrichstoffe und ähnliche Beschichtungsstoffe (Paints and Similar Coating Materials Standards Committee).

General

Besides flow cups with 4 mm orifice diameter as specified in this standard, ISO 2431 specifies flow cups with orifice diameters 3, 4 and 6 mm (see figure 4). It should be noted, however, that rotating viscometers are now generally preferred for paints and varnishes with higher viscosities.

In international recommendations, guidelines and specifications on the storage and transport of dangerous goods only ISO flow cups are specified for test purposes. Even the relevant German specifications now require the use of flow cups as specified in ISO 2431.

Limit deviations of 3% from the calibration graph are permitted both in ISO 2431 and DIN 53 211 for the flow times measured. However, it should be noted that a relative change of 3% in the flow time corresponds to a relative change in the viscosity which is greater than 3% for all cups, and which increases in inverse proportion to the flow time.⁴⁾

DIN cups with orifices of different diameters

There has been some doubt as to whether the jets of flow cups as specified in this standard may be interchanged. It is a requirement that DIN 53 211 — 4 flow cups be supplied with a test report in accordance with DIN 50 049. Such a test report would be meaningless if the jets were interchangeable. Thus, the conclusion is that flow cups with interchangeable jets do not comply with the requirements specified in the present standard.

⁴⁾ H. Bauer, U. Krämer. *Farbe und Lack*, 1983: 89, 518

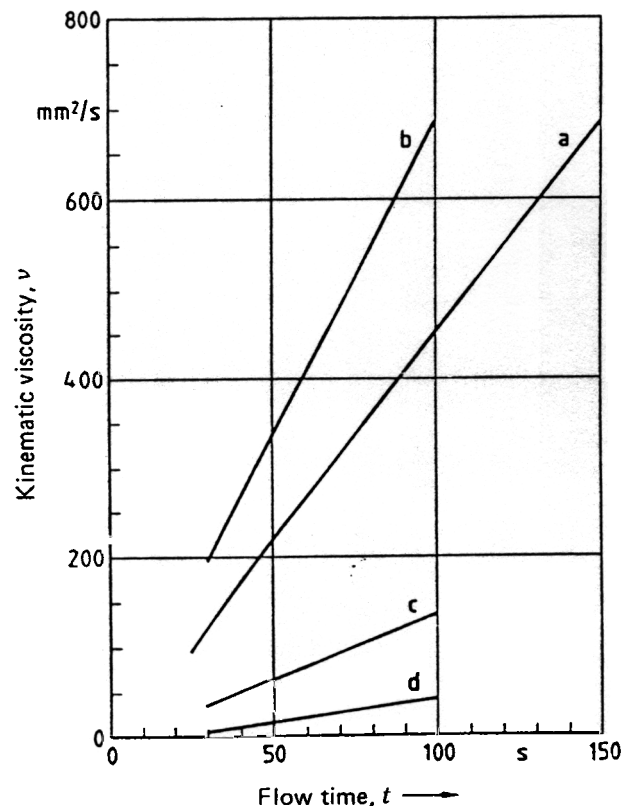


Figure 4. Comparison of flow times measured with different flow cups

- a = Flow cup as specified in DIN 53 211.
- b = Flow cup with orifice diameter 6 mm as specified in ISO 2431.
- c = Flow cup with orifice diameter 4 mm as specified in ISO 2431.
- d = Flow cup with orifice diameter 3 mm as specified in ISO 2431.

Flow behaviour anomalies

Greater dispersion of the measured values is always likely where anomalies in flow behaviour occur as a result of thixotropy or structural viscosity, such anomalies being indicated in the test report by stating the actual repeatability values attained.

If possible, flow cups should not be used for thixotropic liquids (see note to clause 8), which are characterized by a reversible gel/sol transition. Thus, slightly different ways in handling them when carrying out the determination may result in fluctuations of the measured values exceeding the limit deviations specified in this standard. As highly thixotropic materials may easily be identified as such beforehand, no determination in accordance with this standard should be carried out. However, there are numerous materials whose thixotropy is less pronounced and which cannot be identified without equipment. If it is suspected that the materials concerned are thixotropic, this can be checked by determinations in which the waiting time prior to the substance being released is varied, using the following basic procedure.

International Patent Classification

C 09 D 5/00
C 09 D 7/00
G 01 N 33/32
G 01 N 11/02

Stir the product to be tested using a high-speed stirrer or some other mechanical stirring device for several minutes, taking care to avoid the introduction of air bubbles and any increase in the sample temperature. The duration of the filling procedure should be about the same for all the determinations (approximately one minute). Then slide a glass plate over the upper rim of the cup as described in subclause 6.2.

In the first determinations, then open the orifice immediately, slide the glass plate off the upper rim of the cup, and determine the mean value for at least three flow times. For subsequent determinations, after the filling and prior to release of the flow, allow the material to remain undisturbed in the cup for three minutes before the finger is removed from the orifice. Here, too, determine the mean value for at least three flow times.

If the mean values of the flow times measured by the two methods above differ by more than 4 %, the material shall be deemed thixotropic so that the flow time cannot be determined by the method given in this standard. ⁵⁾

⁵⁾ Zorll, U. *Messung thixotroper Substanzen mit dem Auslaufbecher* (Measurement of thixotropic substances using a flow cup), *Industrie-Lackier-Betrieb*, 1965: 33, 231–235.