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Plastics — Resins in the liquid state or as emulsions or dispersions — Determination of apparent viscosity by the Brookfield Test method

*Plastiques — Résines à l'état liquide ou en émulsions ou dispersions —
Détermination de la viscosité apparente selon le Procédé Brookfield*



Reference number
ISO 2555 : 1989 (E)

Foreword

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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. They are approved in accordance with ISO procedures requiring at least 75 % approval by the member bodies voting.

International Standard ISO 2555 was prepared by Technical Committee ISO/TC 61, *Plastics*.

This second edition cancels and replaces the first edition (ISO 2555 : 1974), of which it constitutes a technical revision.

Annexes A and B form an integral part of this International Standard.

Plastics — Resins in the liquid state or as emulsions or dispersions — Determination of apparent viscosity by the Brookfield Test method¹⁾

1 Scope

This International Standard specifies a method of determining an apparent viscosity, by the Brookfield Test method, of resins in a liquid or similar state, using one of the types of rotational viscometer described in this Standard.

The application of this method to specific products is detailed in annex A.

The viscometers, types A, B and C, permit viscosity measurements from 0,02 Pa·s (20 cP) to 60 000 Pa·s (60×10^6 cP).

2 Principle

A spindle, of cylindrical or related form (disc), is driven at a constant rotational frequency in the product being studied.

The resistance exerted by the fluid on the spindle, which depends on the viscosity of the product, causes a torque which is indicated by a suitable meter. This measurement may be based on tightening of a spiral spring depending on the torque, indicated by the movement of a needle on a dial.

The apparent viscosity by the Brookfield Test method is obtained by multiplying this dial reading by a coefficient which depends on the rotational frequency and characteristics of the spindle.

The products to which this International Standard is applicable are generally non-Newtonian and the measured viscosity depends on the velocity gradient to which the products are subjected during the measurement.

With these types of viscometer, the velocity gradient is not the same for every point of the spindle. Thus, for a non-Newtonian fluid, the result is not strictly the true "viscosity at a known velocity gradient" and therefore is conventionally called the apparent viscosity.

3 Apparatus

3.1 Brookfield-type viscometer, type A, B or C, chosen according to the product to be tested and the desired precision.

The detailed working principle of this apparatus, its description and the characteristics of the three types are given in annex B.

Each viscometer consists of:

- the viscometer body;
- seven interchangeable spindles numbered from 1 to 7 (number 1 being the largest); these spindles carry a mark that indicates the immersion level in the liquid; they are the same for the three types of viscometer; do not use spindles which show signs of corrosion or eccentricity;
- a detachable guard stirrup (type A only).

The rotational frequencies available on the different types of Brookfield viscometer are given in table 1.

NOTE — In the case of the Brookfield apparatus, the relationship between the models and available frequencies and viscometer types A, B and C are given in table 1. Other rotational frequencies within the same limits may be chosen.

Table 1 — Rotational frequencies available for the three types of viscometer

Viscometer type	Model	Rotational frequencies, min ⁻¹						
A	RVF	2	4	10	20			
	RVF 100			10	20	50	100	
	RVT	0,5	1	2,5	5	10	20	50 100
B	HAF	1	2	5	10			
	HAT	0,5	1	2,5	5	10	20	50 100
C	HBF	1	2	5	10			
	HBT	0,5	1	2,5	5	10	20	50 100

The shapes and sizes of the spindles are such that the viscosities corresponding to a maximum torque indication on the meter, for the various rotational frequencies, are those given in table 2.

The adjustment and calibration of these viscometers are usually carried out by the manufacturer of the apparatus.

1) The Brookfield Test method may be carried out using test equipment supplied by a number of manufacturers.

Table 2 — Maximum value of viscosity (Pa·s)^{*)} as a function of viscometer type, rotational frequency and spindle

Viscometer type	Rotational frequency min ⁻¹	Spindle number						
		1	2	3	4	5	6	7
A	100	0,1	0,4	1	2	4	10	40
	50	0,2	0,8	2	4	8	20	80
	20	0,5	2	5	10	20	50	200
	10	1	4	10	20	40	100	400
	5	2	8	20	40	80	200	800
	4	2,5	10	25	50	100	250	1 000
	2,5	4	16	40	80	160	400	1 600
	2	5	20	50	100	200	500	2 000
	1	10	40	100	200	400	1 000	4 000
	0,5	20	80	200	400	800	2 000	8 000
B	100	0,2	0,8	2	4	8	20	80
	50	0,4	1,6	4	8	16	40	160
	20	1	4	10	20	40	100	400
	10	2	8	20	40	80	200	800
	5	4	16	40	80	160	400	1 600
	2,5	8	32	80	160	320	800	3 200
	2	10	40	100	200	400	1 000	4 000
	1	20	80	200	400	800	2 000	8 000
	0,5	40	160	400	800	1 600	4 000	16 000
C	100	0,8	3,2	8	16	32	80	320
	50	1,6	6,4	16	32	64	160	640
	20	4	16	40	80	160	400	1 600
	10	8	32	80	160	320	800	3 200
	5	16	64	160	320	640	1 600	6 400
	2,5	32	128	320	640	1 280	3 200	12 800
	2	40	160	400	800	1 600	4 000	16 000
	1	80	320	800	1 600	3 200	8 000	32 000
	0,5	160	640	1 600	3 200	6 400	16 000	64 000
*) 1 Pa·s = 10 ³ cP								

It is recommended that the adjustment and calibration be checked from time to time by means of Newtonian liquids of known viscosity, either by the user's laboratories or by official standardizing laboratories.

3.2 Thermostatic liquid bath, to maintain the product being tested at the test temperature with an accuracy of $\pm 0,2\text{ }^{\circ}\text{C}$.

Recommended test temperatures, in degrees Celsius, are: 23, 25, 40, 55, 70, 85 and 100.

NOTE — If the test has to be carried at higher temperatures, it is advisable to use an extension shaft between the spindle and the apparatus.

3.3 Additional apparatus

3.3.1 Support, which holds the viscometer and moves it in a vertical plane.

3.3.2 Beaker, 90 mm to 92 mm in diameter and 115 mm to 160 mm in height.

3.3.3 Thermometer, graduated in $0,1\text{ }^{\circ}\text{C}$, to measure the temperature of the product being tested.

4 Choice of rotational frequency and spindle

Choose the rotational frequency-spindle combination taking into account the value of the viscosity to be measured, the desired precision and the velocity gradient. It is necessary to make this choice in such a way that no measurement corresponds to less than 20 % or more than 95 % of full-scale deflection. However, for the best accuracy it is advisable to keep to the range 45 % to 95 % of full scale.

If a comparison of viscosities between non-Newtonian products is desired, it is necessary to use the same rotational frequency-spindle combination for all the measurements, even if the accuracy of one of the measurements is markedly decreased.

The choice of rotational frequency automatically involves the choice of one or more types of viscometer. In this connection, it is recommended that, if possible, a rotational frequency of 10 min^{-1} be used.

The rotational frequency-spindle combination to be used shall be specified in the document that prescribes the measurement (see annex A, clause A.1). The choice may be made in terms of the stated or expected viscosity, using table 2 and noting that the most suitable range is between 45 % and 95 % of full scale.

5 Procedure

Mount the viscometer, with its guard stirrup, when applicable (see sub-clause B.3.3 in annex B) on its support. Fill the beaker (3.3.2) with the product to be tested, taking care not to introduce air bubbles, then place it in the bath (3.2) for a sufficient time to reach the desired temperature. If the product contains volatile matter or is hygroscopic, take care to close the beaker tightly during this operation.

With the beaker still in the bath, hold the spindle at an angle of approximately 45° to the surface of the product and immerse it in the product. Orientate the spindle vertically, and connect it to the shaft of the apparatus.

Check that the spindle is vertical, using the bubble level, that the lower end of the spindle is more than 10 mm from the bottom of the beaker and that the spindle is immersed to the underside of the mark on its shaft. Immerse the thermometer (3.3.3) in the product.

Wait until the temperature of the product is between the prescribed limits. Start the motor and run at the desired rotational frequency, taking into account the manufacturer's recommendations.

Allow the instrument to run and read the torque meter to the nearest 0,25 % of maximum value indication, in accordance with the manufacturer's recommendations, when a stable value has been reached on the meter. Lock the needle and stop the motor to take the reading.

NOTES

1 If the reading of the torque meter changes slowly, this may indicate that the product is thixotropic or rheopectic. The document that prescribes the measurement (see annex A, clause A.1) should state whether the reading of the torque meter should be taken after a defined time, or as soon as it has become constant. It is also possible to plot the curve of viscosity as a function of period of rotation.

2 Different liquids may have different rheology. For liquids with a thixotropic or rheopectic behaviour, the period of rotation should be fixed, e.g. 1 min (use only 1 period since the reading of the torque meter may depend on time).

Restart the motor and make another measurement.

Continue taking measurements until two consecutive values are obtained that do not differ by more than 3 % from each other (unless otherwise specified in the documents mentioned in annex A, clause A.1).

Take the average of these two values.

After each determination, detach the spindle from the apparatus and wash it thoroughly in a suitable solvent.

6 Expression of results

Calculate the apparent viscosity, in pascal seconds, of the product tested, using the formula

$$\frac{A \times k \times l}{1\,000}$$

where

A is a coefficient depending on the type of apparatus whose value is 1 for type A, 2 for type B and 8 for type C;

k is a coefficient depending on the rotational frequency-spindle combination used; in the case of apparatus as specified in annex B, clause B.3, the values of k are as shown in table 3;

l is the mean of the two values read on the scale.

Table 3 — Coefficient k (scale 0 to 100) for each rotational frequency-spindle combination¹⁾

Rotational frequency min ⁻¹	Coefficient k for spindle number						
	1 (largest)	2	3	4	5	6	7 (smallest)
100	1	4	10	20	40	100	400
50	2	8	20	40	80	200	800
20	5	20	50	100	200	500	2 000
10	10	40	100	200	400	1 000	4 000
5	20	80	200	400	800	2 000	8 000
4	25	100	250	500	1 000	2 500	10 000
2,5	40	160	400	800	1 600	4 000	16 000
2	50	200	500	1 000	2 000	5 000	20 000
1	100	400	1 000	2 000	4 000	10 000	40 000
0,5	200	800	2 000	4 000	8 000	20 000	80 000

1) It may be necessary to calibrate each spindle with liquids of known viscosity and to adopt slightly different values of k (see clause B.4 in annex B).

Express the results to three significant figures, indicating the type of viscometer (A, B or C), the number of the spindle and the rotational frequency, as in the following example:

Brookfield viscosity (A/3/20) = 4,25 Pa·s
(1 Pa·s = 10³ cP).

NOTE — If the measurement has been made under the conditions described in the notes to clause 5 (thixotropic or rheopectic products), follow the special instructions of the document mentioned in annex A, clause A.1.

7 Test report

The test report shall include the following particulars:

- a reference to this International Standard;
- the designation of the product being tested;
- the test temperature;
- the model of viscometer used;
- the value of the apparent viscosity calculated in accordance with the directions of clause 6.

Annex A (normative)

General application to resins in the liquid state, emulsions and dispersions

A.1 Field of application

This annex shall be used for determining the apparent viscosity of resins in the liquid state, unless any indication to the contrary is given.

It shall be used whenever a standard or a commercial contract concerning the product recommends the use of ISO 2555 and its annex A.

A.2 Choice of test conditions

A.2.1 Rotational frequency

Owing to the generally non-Newtonian behaviour of the products covered by this annex, the results obtained with different rotational frequencies cannot theoretically be compared.

The choice of the frequency depends on the viscosity range to be measured. It shall be stated for each type of product.

It is necessary to establish a diagram showing the viscosity range covered for the viscometer used at the specified frequency (see figure A.1 for an example of the diagram for viscometers A, B and C at a frequency of 10 min⁻¹).

A.2.2 Spindle

If the value of apparent viscosity to be measured is known approximately, choose the spindle with the help of a diagram (see A.2.1), showing, for each type of viscometer, the range of viscosity covered by each spindle.

For the comparison of results, it is better to keep the same spindle even if one must use it within a range where its use is

only "permitted", than to work with different spindles. Practice shows that the results are not always the same between successive spindles.

If the value of viscosity to be measured is not known, make measurements with successive spindles, beginning with number 7 until the requirements of figure A.1 are met.

A.2.3 Particular conditions recommended for certain products

Table A.1 summarizes the particular test conditions recommended for a number of products in the light of international practice. The spindle shall be chosen in accordance with the recommendations of clause 4 of this standard, taking into account the viscosity value.

Table A.1 — Recommended test conditions

Product	Visco- meter type	Rota- tional fre- quency min ⁻¹	Temp- erature °C	Special remarks
Phenolic resins	A ¹⁾	50	23	Read after 1 min.
Polyester resins	A ¹⁾	10	23	
Epoxy resins	A ¹⁾	10	23	
Adhesives	A ¹⁾	10 or 20	23	
Aqueous dispersions	A ¹⁾	50	23	
PVC pastes	A ¹⁾	20	23	

1) The use of types B or C is allowed when the viscosity of the product is greater than the viscosity limit measurable with type A at the rotational frequency recommended.

A.3 Procedure

Follow carefully the instructions given in clause 5.

Take particular care only to measure the viscosity of products completely free of bubbles.

If necessary, bubbles can be eliminated by applying a vacuum or by any other appropriate method.

In the case of products which are volatile, or have volatile constituents, it is, of course, necessary to use closed vessels.

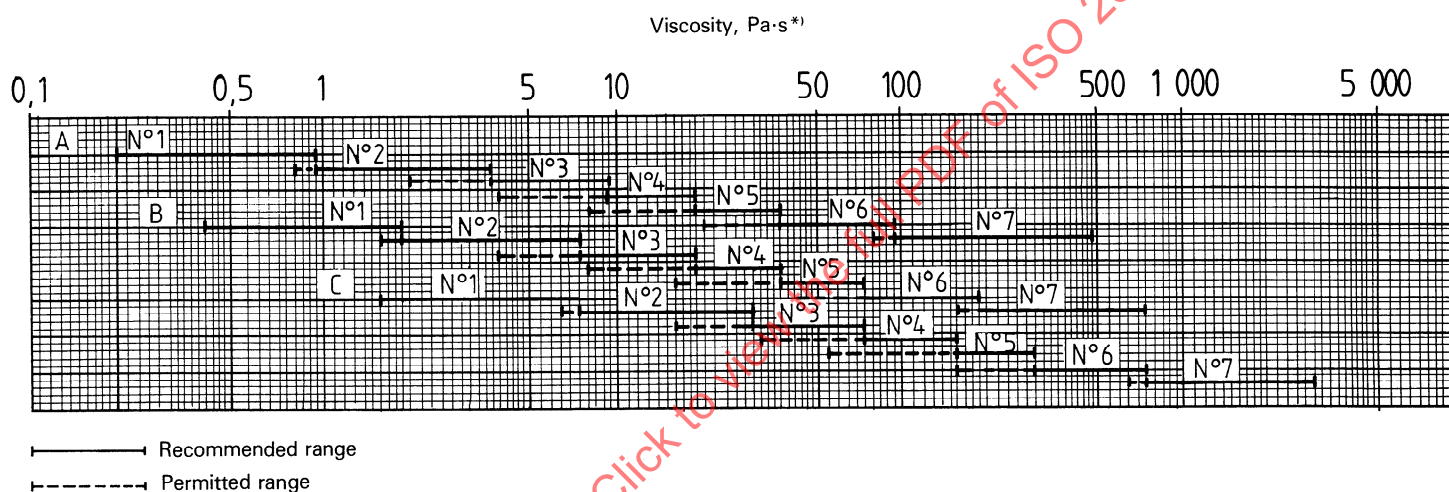


Figure A.1 — Viscosity range (logarithmic scale) for every spindle of the A, B and C viscometers at a rotational frequency of 10 min⁻¹

^{*)} 1 Pa·s = 10³ cP

Annex B (normative)

Principle, description and characteristics of viscometers A, B and C

B.1 Operating principle

The viscometer comprises a synchronous motor rotating a vertical shaft through a gear-box.

This vertical shaft drives, through a spiral spring, a second lower shaft, forming an extension of the first. A removable spindle is attached to this second shaft and is immersed in the liquid under test.

These two shafts rotate at the same frequency but, when the spindle is immersed there is an angular deviation between them which is a function of the liquid resistance to the rotation of the spindle, i.e. the liquid viscosity.

This deviation is measured by a horizontal needle, fixed to the spindle shaft, moving on a horizontal dial fixed to the first (motor) shaft and consequently rotating with this shaft. When the spindle is turning in air, the needle corresponds to the "0" graduation on the dial.

Because of the difficulty of taking readings while the needle and dial are both rotating, a dial-needle locking system allows the reading to be taken after the motor has stopped.

B.2 Brief description

The viscometer body is equipped with an electric switch, synchronous motor, gear-box with its rotational frequency change button, spiral spring, dial and needle, and dial-needle locking system.

The interchangeable spindles have the form of cylinders or discs of polished metal fixed on a shaft. They can be used on the three types of viscometer.

The guard stirrup consists of a metal plate with a "U" form to protect the spindles.

B.3 Characteristics

B.3.1 Viscometer body

B.3.1.1 Rotational frequencies of the spindles

4 or 8 rotational frequencies according to the type of apparatus; the rotational frequencies are given in table 1.

B.3.1.2 Dial graduation

From 0 to 100 in half-divisions.

B.3.1.3 Torque of the spiral spring

For full-scale deflection:

718,7 $\mu\text{N}\cdot\text{m}$, for A type

1 437,4 $\mu\text{N}\cdot\text{m}$, for B type

5 749,6 $\mu\text{N}\cdot\text{m}$, for C type

B.3.2 Interchangeable spindles

Shapes and dimensions are given in figures B.1, B.2 and B.3.

The values of these dimensions result from the conversion of values expressed in inches, and the number of significant figures is not necessarily an indication of the precision of the original values.

B.3.3 Guard stirrup

The shape and dimensions are given in figure B.1.

It ensures the protection of the spindle and a minimum distance of 10 mm between the end of the spindle and the bottom of the beaker. Its use is optional.

B.4 Calibration of apparatus

It is advisable to calibrate the apparatus periodically by measuring the viscosity of pure Newtonian liquids and checking the rotational frequencies of the spindles.

Dimensions in millimetres

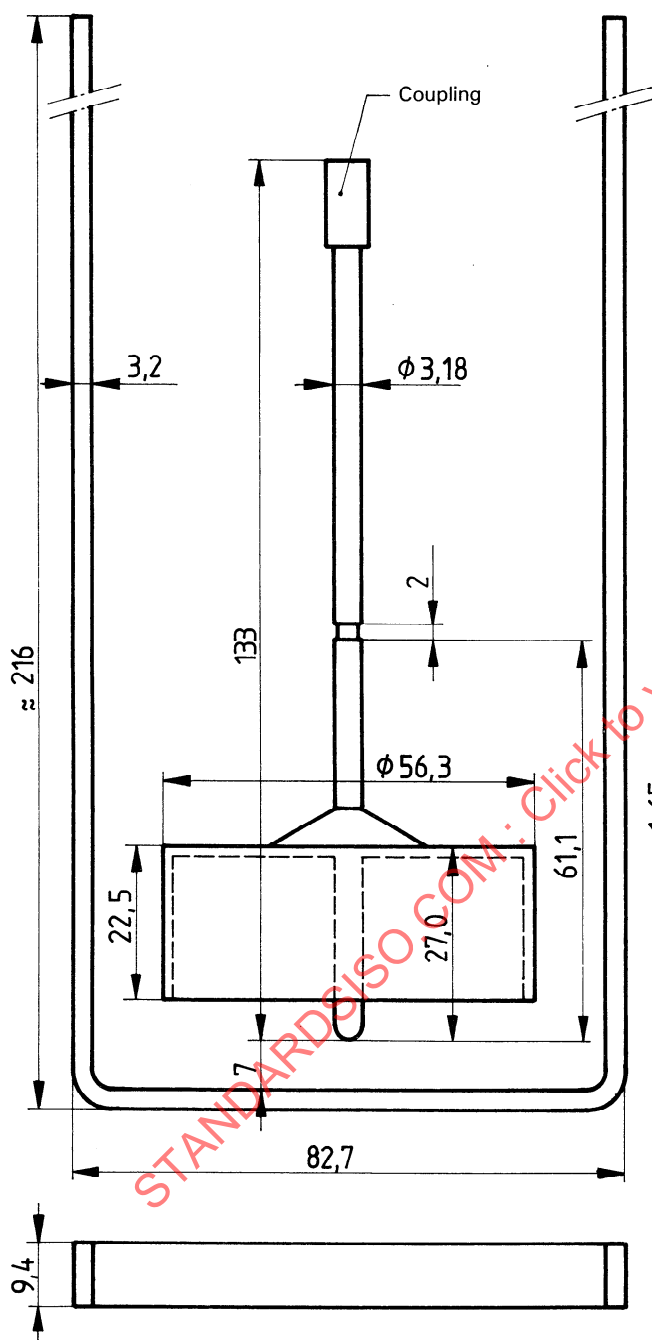


Figure B.1 — No. 1 spindle and guard stirrup

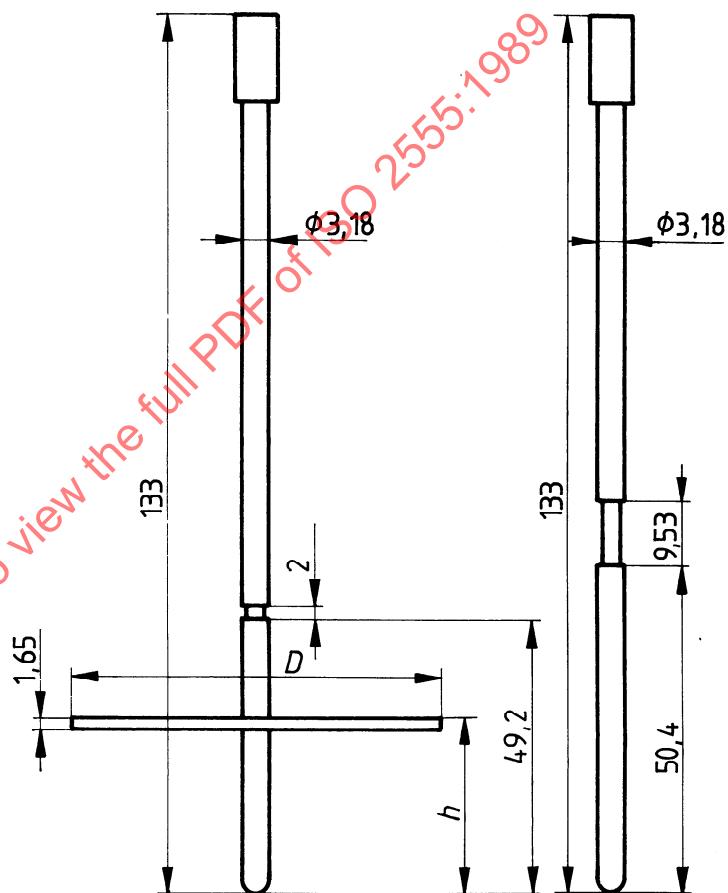


Figure B.2 — Spindles Nos. 2 to 6 Figure B.3 — Spindle No. 7

No.	D	h
2	47,0	27,0
3	34,7	27,0
4	27,3	27,0
5	21,1	27,0
6	14,6	30,2