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Resins in the liquid state or as emulsions or dispersions — Determination of Brookfield RV viscosity

Résines à l'état liquide ou en émulsions ou dispersions — Détermination de la viscosité Brookfield RV

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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.

International Standard ISO 2555 (originally ISO/DIS 2555 and Addendum I) was drawn up by Technical Committee ISO/TC 61, *Plastics*, and circulated to the Member Bodies in November 1971 (Addendum I was circulated in June 1972).

ISO/DIS 2555 has been approved by the Member Bodies of the following countries:

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Germany
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Resins in the liquid state or as emulsions or dispersions — Determination of Brookfield RV viscosity

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a method of determining the viscosity, which is conventionally called "Brookfield viscosity", of resins in a liquid or similar state, using one of the rotation spindle types of Brookfield viscometers, RV Models.

The application of this International Standard to each of the products given in the title (liquids, emulsions, dispersions) requires another document which prescribes the measurement of this Brookfield RV viscosity and specifies particular conditions for the pertinent product, particularly the speed-spindle combination.

This document can be one of the following :

- a) An annex to the present International Standard, covering a product or group of products (for example annex A devoted to liquids).
- b) An International Standard for the designation or specification of a product;
- c) A national standard or commercial contract.

The Brookfield viscometers, RV models, permit viscosity measurements from 0,02 Pa·s (20 cP) to 8 000 Pa·s (8×10^6 cP).

2 PRINCIPLE

A spindle, of cylindrical or related form (disc), is driven by a synchronous motor at a constant speed in the product being studied.

The resistance exerted by the fluid on the spindle, which depends on the viscosity of the product, causes the tightening of a spiral spring which is indicated by the movement of a needle on a dial.

"Brookfield viscosity" is obtained by multiplying this dial reading by a coefficient which depends on the speed of rotation 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 product is subjected during the measurement.

With these types of viscometers 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 "Brookfield RV viscosity".

3 APPARATUS

3.1 Brookfield synchro-lectric viscometer, RV model (RVF, RVF-100 or RVT), chosen according to the product to be tested and the desired experimental accuracy.

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

Each viscometer consists of :

- viscometer body;
- seven interchangeable spindles numbered from 1 to 7 (number 1 being the largest); these spindles carry a mark which indicates the immersion level in the liquid; they are the same for the three types of viscometer;
- guard stirrup, detachable.

The rotation speeds available on the three types of the RV model of the Brookfield viscometer are :

Type	Speed in rev/min						
RVF		2	4	10	20		
RVF-100				10	20	50	100
RVT	0,5	1	2,5	5	10	20	50 100

The shapes and sizes of the spindles are such that the Brookfield RV viscosities corresponding to a maximum deflection of the needle on the dial, for the various speeds, are those given in table 1.

TABLE 1 — Brookfield RV viscosities

Viscometer type	Speed rev/min	Viscosity in Pa·s corresponding to full scale deflection for spindle number						
		1 (large)	2	3	4	5	6	7 (small)
RVT,RVF-100	100	0,1	0,4	1	2	4	10	40
	50	0,2	0,8	2	4	8	20	80
RVF,RVT,RVF-100	20	0,5	2	5	10	20	50	200
	10	1	4	10	20	40	100	400
RVT	5	2	8	20	40	80	200	800
RVF	4	2,5	10	25	50	100	250	1 000
RVT	2,5	4	16	40	80	160	400	1 600
RVF	2	5	20	50	100	200	500	2 000
RVT	1	10	40	100	200	400	1 000	4 000
	0,5	20	80	200	400	800	2 000	8 000

NOTE — 1 Pa·s = 10³ cP

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

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 Water bath, provided with a thermostat, to maintain the product being tested at the test temperature with an accuracy of $\pm 0,2^{\circ}\text{C}$ (usually, a reference temperature given in ISO/R 291).

3.3 Additional apparatus

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

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

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

4 CHOICE OF SPEED AND SPINDLE

Choose the speed-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.

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

The choice of speed automatically involves the choice of one or more types of viscometer. In this connection, it is recommended to use, if possible speeds of 10 and 20 rev/min, which are the only ones common to the three types.

Note that the RVF type has a lower limit around 0,1 Pa·s (100 cP) while the RVF-100 and RVT types have a lower limit around 0,02 Pa·s (20 cP).

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

5 PROCEDURE

Mount the viscometer, with its guard stirrup, on its support.

Fill the beaker (3.3.2) with the product to be tested, taking care not to introduce air bubbles, then put it in the water bath thermostatically controlled within $\pm 0,2^{\circ}\text{C}$, 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.

Mount the selected spindle on the instrument shaft, keeping the shaft steady while screwing up the coupling.

While the beaker is in the water bath, lower the apparatus on its support so that the spindle is immersed in the liquid to the underside of the mark on its shaft. Check that the spindle is vertical, using a bubble level, and insert the thermometer (3.3.3) into the product.

Wait until the temperature of the product is between the prescribed limits.

Start the motor and run at the desired speed taking into account the manufacturer's recommendations.

Unlock the needle and allow the instrument to run until the needle reaches a stable position on the dial (this usually takes 5 to 10 s).

NOTE — If the needle is not completely stable, but moves slowly on the dial, this often indicates that the product is thixotropic or rheopectic. The document which prescribes the measurement (see clause 1) should state whether the position of the needle must be noted after a defined time or after stability of the needle. It is also possible to plot the curve of viscosity as a function of time of rotation.

Lock the needle and stop the motor to take the reading.

Read the value obtained on the scale to the nearest quarter division (0,25). (The dial is graduated in half-units.)

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 clause 1).

Take the average of these two values.

6 EXPRESSION OF RESULTS

Calculate the Brookfield RV viscosity, in pascal seconds, of the product tested by means of the formula

$$\frac{k \times \ell}{1\,000}$$

where

- k is a coefficient depending on the speed-spindle combination used and is shown in table 2;
- ℓ is the mean of the two values read on the scale.

TABLE 2 — Coefficient k (scale 0 to 100) for each speed-spindle combination

Speed rev/min	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

Express the results to three significant figures, indicating not only the Brookfield RV viscosity, but also the speed-spindle combination, as in the following example :

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

NOTE — If the measurement has been made in the conditions described in the note to clause 5 (thixotropic or rheopectic products) follow the special instructions of the documents mentioned in clause 1.

7 TEST REPORT

The test report shall include the following particulars :

- a) the designation of the product being tested;
- b) the test temperature;
- c) the model of the viscometer used;
- d) the value of Brookfield RV viscosity calculated according to the directions of clause 6.

ANNEX A

GENERAL APPLICATION TO RESINS IN THE LIQUID STATE

A.1 FIELD OF APPLICATION

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

It shall be used whenever an international or national standard or a commercial contract concerning the product recommends the use of International Standard ISO 2555, and its annex A.

A.2 CHOICE OF SPEED AND SPINDLE

A.2.1 If the value of "Brookfield RV viscosity" to be measured is known approximately, choose the speed-spindle combination from the table below, drawn up using only the 10 and 20 rev/min speeds that are common to the three types of RV viscometers.

Brookfield RV viscosity in Pa·s	from to (excl.)	0,200	0,475	0,950	1,90	3,80	4,75	9,50	19,0	38,0	47,5	95,0	190
		0,475	0,950	1,90	3,80	4,75	9,50	19,0	38,0	47,5	95,0	190	400
Speed rev/min		20	10	20	10	20	10	20	10	20	10	20	10
Spindle No.		1	1	2	2	3	3	5	5	6	6	7	7

NOTE – 1 Pa·s = 10³ cP

A.2.2 If the value of viscosity to be measured is unknown, make measurements trying successively the speed-spindle combinations indicated in the table, starting with one corresponding to a viscosity obviously greater than the actual value in order to avoid damage to the apparatus. Choose the combination giving the largest deflection not exceeding 95 % of the full scale.

A.3 PROCEDURE

Follow carefully the provisions of clause 5.

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

If necessary, bubbles can be eliminated by leaving the product standing for a sufficient time, at an elevated temperature if necessary.

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