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Plastics — Resins in the liquid state or as emulsions or dispersions — Determination of apparent viscosity using a single cylinder type rotational viscometer method

*Plastiques — Résines à l'état liquide ou en émulsions ou dispersions
— Détermination de la viscosité apparente par la méthode du
viscosimètre rotatif de type à un cylindre*



Reference number
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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 on 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 the following URL: www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 5, *Physical-chemical properties*.

This third edition cancels and replaces the second edition (ISO 2555:1989), which has been technically revised. The main changes compared to the previous edition are as follows:

- a) the name of the equipment has been changed to "single cylinder type rotational viscometer" and the method of determination of viscosity is described with more general terms to avoid reference to specific brand names;
- b) the commercially available main models and spindles have been summarized;
- c) digital type viscometer has been added;
- d) the accuracy of test method has been added;
- e) the calibration method has been added.

Introduction

A rotational viscometer is composed of a spindle, a torque measuring device and a spring. There are three types of rotational viscometers with regard to difference of the spindle:

- a) single cylinder type (used in this document, i.e. ISO 2555);
- b) coaxial cylinder type (used in ISO 3219^[2]);
- c) cone-plate type (used in ISO 3219^[2]).

Single cylinder type rotational viscometers measure viscosity under non-constant shear rate. Coaxial cylinder type and cone-plate type instruments measure viscosity under constant shear rate.

When using a single cylinder type instrument, the measured viscosity is relative to measuring conditions. Conditions are therefore intended to be specified for viscosity measurements.

For Newtonian fluids, the viscosity value remains the same even if different viscosity measuring methods from this document and ISO 3219 are used.

With non-Newtonian fluids the measured viscosity changes depending on shear rate. The viscosity determined using different measuring methods such as methods from this document and ISO 3219 therefore may differ from each other, depending on shear rates used during measurements.

The Brookfield method has contributed a lot to determination of liquid viscosity. With its simple measuring, low-cost equipment, the principles underlying the Brookfield technique still remain an important element in determination of viscosity of liquids.

This document is largely based on the Brookfield method established in 1989. However, some of the instruments mentioned in the previous edition of this document have long been discontinued. Moreover, although analogue (or the so-called "dial") type was the predominant viscometer type at that time, the use has now shifted to digital viscometers in the recent years, increasing the need for this document to be revised.

The terms and standards introduced in this new edition are based on ISO 1652^[1].

Currently, digital viscometer has become the mainstream. However, analogue (or dial) viscometer is still used and cannot be removed from the method. This document allows the use of both analogue (or dial) viscometer and digital viscometer.

Plastics — Resins in the liquid state or as emulsions or dispersions — Determination of apparent viscosity using a single cylinder type rotational viscometer method

1 Scope

This document specifies a method of determining apparent viscosity of resins in a liquid state using a single cylinder type rotational viscometer.

The method can be used for viscosity measurements in the range from 0,02 Pa · s to 60 000 Pa · s.

This document is applicable to both Newtonian and non-Newtonian liquids and the measured apparent viscosity depends on the velocity gradient to which the liquids are subjected during the measurement.

2 Normative references

There are no normative references in this document.

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

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

- IEC Electropedia: available at <http://www.electropedia.org/>
- ISO Online browsing platform: available at <https://www.iso.org/obp>

3.1 single cylinder type rotational viscometer

apparatus that determines viscosity by measuring the torque acting on a cylinder or a disc surface rotating in laminar flow condition at constant angular velocity

3.2 spindle

rotational symmetric object in the form of a cylinder or disc connected to a vertically rotating shaft

3.3 apparent viscosity

viscosity of liquids/fluids determined from the measured torque using predetermined calibration table

Note 1 to entry: For non-Newtonian fluids the apparent viscosity depends on the shear rate. 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.

4 Principle

4.1 General

A rotational symmetric spindle is driven at constant rotational speed in the liquid being measured.

The resistance exerted by the fluid on the spindle, which depends on the viscosity of the liquid, creates a torque which is measured by a suitable device.

The apparent viscosity measured using a single cylinder type rotational viscometer is obtained by multiplying the torque readout with a coefficient which depends on the rotational speed and characteristics of the spindle. With digital viscometers, viscosity is displayed by setting a certain rotational speed and entering the correct spindle number.

This method applies to Newtonian and non-Newtonian matter.

4.2 Operating principle

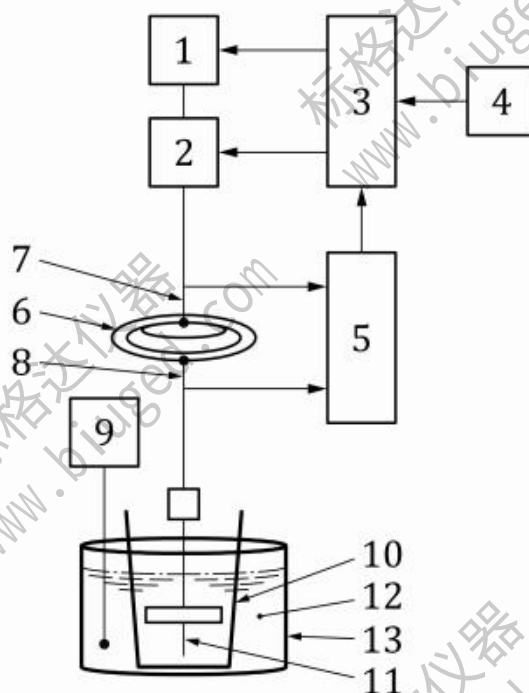
With analogue viscometer, a synchronous motor rotates a vertical shaft through a gear-box. This vertical shaft drives, through a spiral spring, a second lower shaft, forming an extension of the first. A removable spindle is attached to this second shaft and is immersed in the liquid under test. These 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 resistance of the liquid to the rotation of the spindle, i.e. the liquid viscosity. 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.

With digital viscometers, an angle deviation between a vertical shaft and a second lower shaft is automatically read using the device for angle deviation measurement. [Figure 1](#) shows a schematic drawing of the operating principle of a digital viscometer.

4.3 Brief description

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

Digital viscometer body is equipped with an electric switch, synchronous motor, the input device of the rotational speed, the input of the spindle, spiral spring, spindle, CPU, display and measurement of the angular deviation system.

**Key**

- 1 viscosity (display)
- 2 motor
- 3 CPU
- 4 input device of the rotational speed and spindle number
- 5 device of angle deviation measurement, between the spindle and the motor
- 6 spring
- 7 first vertical shaft
- 8 second lower shaft
- 9 temperature measuring device
- 10 guard stirrup
- 11 spindle
- 12 liquid
- 13 beaker
- ← electrical signals or input

Figure 1 — Schematic drawing of the operating principle of a digital viscometer

5 Apparatus

5.1 Single cylinder type rotational viscometer

Depending on the viscosity of liquid and the desired precision, viscometer models L, R, H(A) or H(B) are chosen for measurement of sample viscosity. These viscometer models are categorized, based on the torque range. R is used as a standard format. L is used when the viscosity is low. H(A), H(B) is used when the viscosity is high.

The interchangeable spindles are made of metal and are in the form of cylinders or discs, which can be fixed on to main shaft of the viscometer. L1 to L4 are to be used with model L and R1 to R7 can be used for models R, H(A) and H(B) type viscometers.

A guard stirrup as specified in Annex B can be used to protect the device and the spindle. Using a guard stirrup shall not change measurement conditions.

The guard stirrups consist of a metal plate with a "U" form to protect the spindles.

The shape and dimensions of guard stirrups and spindles are given in Annex B. The size of the beaker shall be adapted depending on use of a guard stirrup.

With an analogue viscometer, the 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 rotating in air, the needle shall be calibrated to the "0" graduation of the dial.

A digital viscometer measures automatically the deviation between the spindle and the motor.

Figure 2 shows an example of a single cylinder type rotational viscometer for model L. Figure 3 shows an example of a single cylinder type rotational viscometer for model R, H(A) and H(B).



Figure 2 — Example of single cylinder type rotational viscometer for Model L



Figure 3 — Example of single cylinder type rotational viscometer for model R, H(A) and H(B)

Each viscometer consists of the following:

- the viscometer body; model L, R, H(A), H(B) depending on the liquid to be tested; with plastics, model R is most commonly used, with highly viscous samples model H(A) or H(B) is recommended, while with less viscous samples model L is recommended. The maximum value of the measurement, $L < R < H(A) < H(B)$;
- for model L: Four interchangeable spindles numbered from L1 to L4 (L1 being the largest);
- for model R, H(A), H(B): Seven interchangeable spindles numbered from R1 to R7 (R1 being the largest).

All spindles carry a mark that indicates the immersion level in the liquid (see F in [Table B.1](#)).

Characteristics of single cylinder type rotational viscometer models are given in [Table 1](#).

Table 1 — Characteristics of single cylinder type rotational viscometer models

Viscometer model	Maximum torque $\mu\text{N}\cdot\text{m}$	Spindles numbered
L	67,37	L1 L2 L3 L4
R	718,7	R1 R2 R3 R4 R5 R6 R7
H(A)	1 437,4	R1 R2 R3 R4 R5 R6 R7
H(B)	5 749,6	R1 R2 R3 R4 R5 R6 R7

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](#) and [Table 3](#).

Table 2 — Maximum value of viscosity as a function of rotational speed and type of spindle for viscometer model L

Viscometer model	Rotational speed r/min	Viscosity Pa · s ^a			
		Spindle			
		L1	L2	L3	L4
L	100	0,06	0,3	1,2	6
	60	0,1	0,5	2	10
	30	0,2	1	4	20
	12	0,5	2,5	10	50
	6	1	5	20	100
	3	2	10	40	200
	1,5	4	20	80	400
	0,6	10	50	200	1 000
	0,3	20	100	400	2 000

^a 1 Pa · s = 10³ cP.

Table 3 — Maximum value of viscosity as a function of rotational speed and type of spindle for viscometer models R, H(A) and H(B)

Viscometer model	Rotational speed r/min	Viscosity Pa · s ^a						
		Spindle						
		R1	R2	R3	R4	R5	R6	R7
R	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	18	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
H(A)	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
	4	5	10	20	50	100	500	2 000
	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	100	400	800	1 600	4 000	16 000

^a 1 Pa · s = 10³ cP.

Table 3 (continued)

Viscometer model	Rotational speed r/min	Viscosity Pa · s ^a						
		Spindle						
		R1	R2	R3	R4	R5	R6	R7
H(B)	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
	4	20	80	200	400	800	2 000	8 000
	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

^a 1 Pa · s = 10³ cP.

Calibration shall be carried out once per year using traceable standard liquids.

5.2 Thermostatic liquid bath

It shall be capable to maintain the liquid being tested at the test temperature with an accuracy of $\pm 0,2$ °C.

Recommended test temperatures are: 20 °C, 23 °C, 25 °C, 40 °C, 55 °C, 70 °C, 85 °C and 100 °C.

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

5.3 Additional apparatus

5.3.1 Support, used to hold the viscometer and move it vertically.

5.3.2 Beaker. Glass beaker suitable for the intended spindle and guard stirrup.

The size of the beaker may be arbitrary. However, it is necessary to use the beaker of the same size when comparing the results of a measurement.

5.3.3 Temperature measuring device.

— Resolution: $\pm 0,1$ °C.

— Accuracy: $\pm 0,2$ °C.

6 Choice of spindle and rotational speed

Choose the spindle and rotational speed taking into account the value of the viscosity to be measured, the desired precision and the velocity gradient. For best accuracy a measurement range of 45 % to 95 % of full scale deflection is preferred. By no means shall measurements be done at less than 20 % or more than 95 % of full-scale deflection.

The choice of rotational speed implies use of one or more suitable models of viscometer. If more than one viscometer models are used the same rotational speed should be used for each model. The choice may be made based on stated or expected viscosity, using [Tables 2 and 3](#).

To allow comparison of results, it is better to keep the same spindle even if this requires use outside of the preferred range of 45 % to 95 % of full scale deflection. Practice shows that the results may deviate between successive spindles.

If the value of viscosity to be measured is not known, measurements shall be performed with successive spindles, beginning with number (R7 or L4).

Choice of test conditions for general application to resins in the liquid state, emulsions and dispersions is shown in [Annex A](#).

7 Procedure

Mount the viscometer, with its guard stirrup, when applicable, on its support. Fill the beaker (5.3.2) with the liquid to be tested, taking care not to introduce air bubbles, then place it in the bath (5.2) for a sufficient time to reach the desired temperature. If the liquid contains volatile matter or is hygroscopic, take care to close the beaker tightly during heating to the desired temperature and measurement. Take particular care to measure the viscosity of products completely free of bubbles only. If necessary, bubbles can be eliminated by applying a vacuum or by any other appropriate method.

The temperature of the liquid shall be kept constant during measurement. The time to reach the desired temperature of measurement may be different depending on type and viscosity of the liquid and shall be checked by preliminary testing. If the viscosity of the liquid is high it may take up to 24 h to reach temperature equilibrium.

In order to remove the air bubbles attached to the spindle, with the beaker still in the bath, hold the spindle at an angle of approximately 45° relative to the surface of the liquid upon immersion. R1 spindle is easy to fall out because the R1 spindle has a hole on the top surface (see [Figure 4](#)). R1 spindle is used with low viscosity liquids. It is necessary to help the air bubbles escape. Orientate the spindle vertically, and connect it to the shaft of the apparatus.



Figure 4 — R1 spindle

Check that the spindle is vertical, using the bubble level of the viscometer, 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 (5.3.3) in the liquid. Figure 5 shows the bubble level indicator.



Figure 5 — Bubble level indicator

Wait until the temperature of the liquid has reached the desired test temperature.

With digital viscometers, input the rotational speed and the spindle number to be used.

Start the motor and run at the desired rotational speed.

With analogue viscometers, allow the instrument to run and read the dial to the nearest 0,25 % of maximum value. When a stable value has been reached on the dial, lock the needle and stop the motor to take the reading.

With digital viscometers, viscosity is automatically calculated and displayed.

Slowly changing dial readings may indicate thixotropic or rheopectic behaviour of the liquid. Refer to the manufacturer instructions to take the reading of the dial 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.

Different liquids may have different rheology. For liquids with thixotropic or rheopectic behaviour, the period of rotation should be fixed, e.g. 1 min.

Continue taking measurements until two consecutive values are obtained that do not differ by more than 3 % from each other.

Take the average of these two values.

After each determination, detach the spindle from the apparatus and wash it thoroughly in a suitable solvent. For example, light oil, neutral detergent, alkaline cleaning solution or water.

8 Expression of results

Calculate the apparent viscosity, in pascal seconds (Pa·s), of the product tested, using [Formula \(1\)](#):

$$\eta = \frac{A \cdot k \cdot l}{1\,000}$$
 (1)

where

- η is the apparent viscosity;
- A is a coefficient depending on the viscometer model having a value of 1 for model L, 1 for model R, 2 for model H(A) and 8 for model H(B);
- k is a coefficient depending on the rotational speed-spindle combination used; in the case of apparatus as specified in [Annex B](#), the values of k are as shown in [Tables 4](#) and [5](#);
- l is the mean of the two values read on the dial.

With digital viscometers, there is no need for calculation using the above formula; by selecting the rotational speed and entering the correct spindle number, viscosity is automatically calculated and displayed.

Table 4 — Coefficient k for different rotational speed-spindle combination

Viscometer model	Rotational speed r/min	Coefficient k^a			
		Spindle			
		L1	L2	L3	L4
L	100	0,6	3	12	60
	60	1	5	20	100
	30	2	10	40	200
	12	5	25	100	500
	6	10	50	200	1 000
	3	20	100	400	2 000
	1,5	40	200	800	4 000
	0,6	100	500	2 000	10 000
	0,3	200	1 000	4 000	20 000
^a It may be necessary to calibrate each spindle with liquids of known viscosity and to adopt slightly different values of k .					

Table 5 — Coefficient k for different rotational speed-spindle combination

Viscometer model	Rotational speed r/min	Coefficient k^a						
		Spindle						
		R1	R2	R3	R4	R5	R6	R7
R, H(A), H(B)	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

^a It may be necessary to calibrate each spindle with liquids of known viscosity and to adopt slightly different values of k .

Express the results to three significant figures, indicating the model of viscometer (L, R, H(A), or H(B)), the number of the spindle and the rotational speed, as in the following example:

Viscosity (R/3/20) = 4,25 Pa · s

[1 Pa · s = 10³ cP]

9 Test report

The test report shall include the following particulars:

- a reference to this document (i.e. ISO 2555);
- the designation of the liquid being tested;
- the test temperature;
- the viscometer model, rotational speed, spindle;
- the value of the apparent viscosity;
- the measurement time;
- the size of the beaker;
- the presence or absence of the guard stirrup.

Annex A
(informative)

Choice of test conditions for general application to resins in the liquid state, emulsions and dispersions

Figure A.1 is a diagram showing the viscosity range covered for the viscometer used at the specified rotational speed.

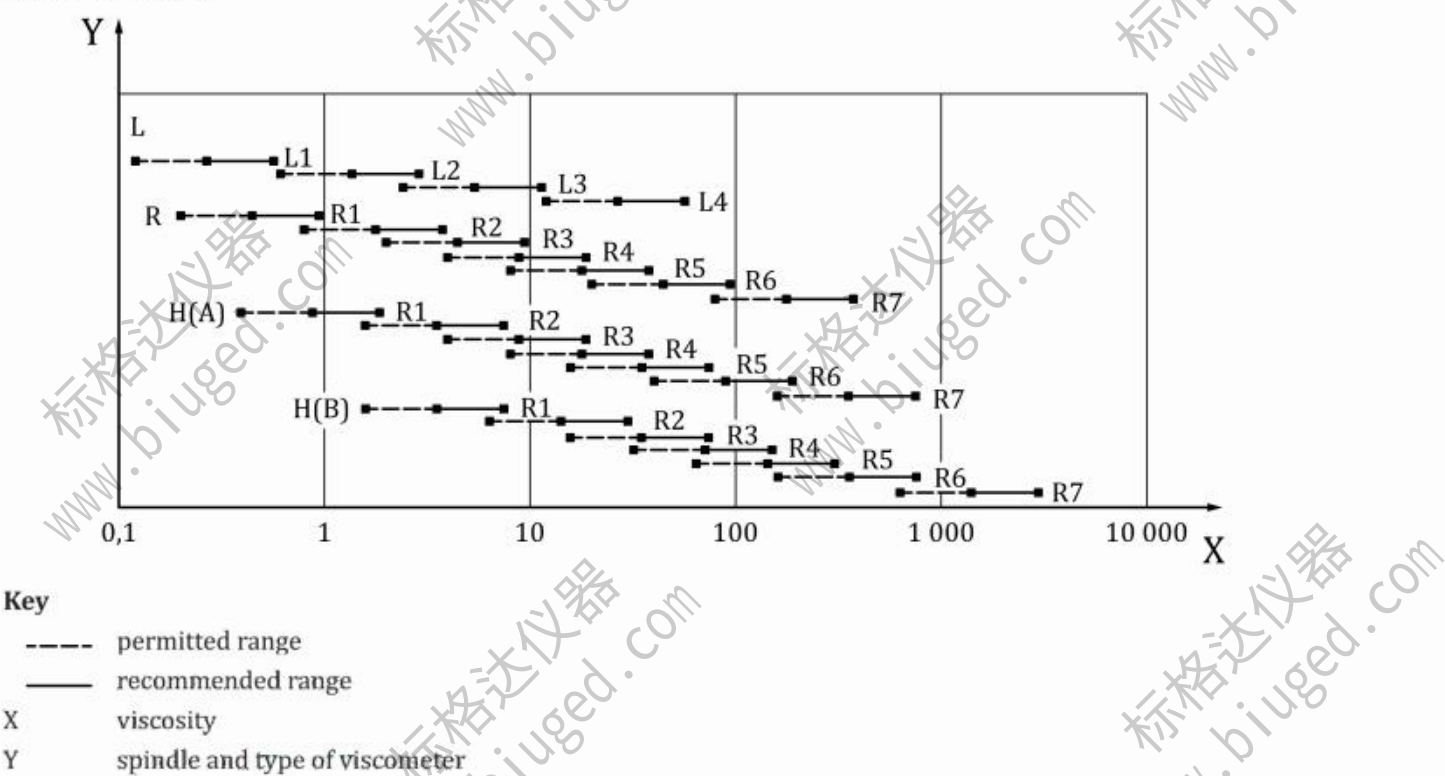


Figure A.1 — Viscosity range (logarithmic scale) for every spindle of the L, R, H(A), and H(B) viscometers at a rotational speed of 10 r/min

Particular conditions may be recommended for certain products.

Table A.1 summarizes the particular test conditions for a number of products in the light of international practice. Choose spindle in response to the viscosity value.

Table A.1 — Examples of test conditions

Product	Viscometer model	Rotational speed r/min	Temperature °C	Special remarks
Phenolic resins	R ^a	50	23	
Polyester resins	R ^a	10	23	
Epoxy resins	R ^a	10	23	
Adhesives	R ^a	10 or 20	23	
Aqueous dispersions	R ^a	50	23	Read after 1 min
PVC pastes	R ^a	20	23	Read after 1 min
^a The models H(A) and H(B) can be used when the viscosity of the product is greater than the viscosity limit measurable with model R at the rotational speed recommended. For less viscous product, model L may be used.				

Annex B
(normative)

Spindle and guard stirrup

The shape and dimensions of guard stirrups are given in [Figure B.1](#).

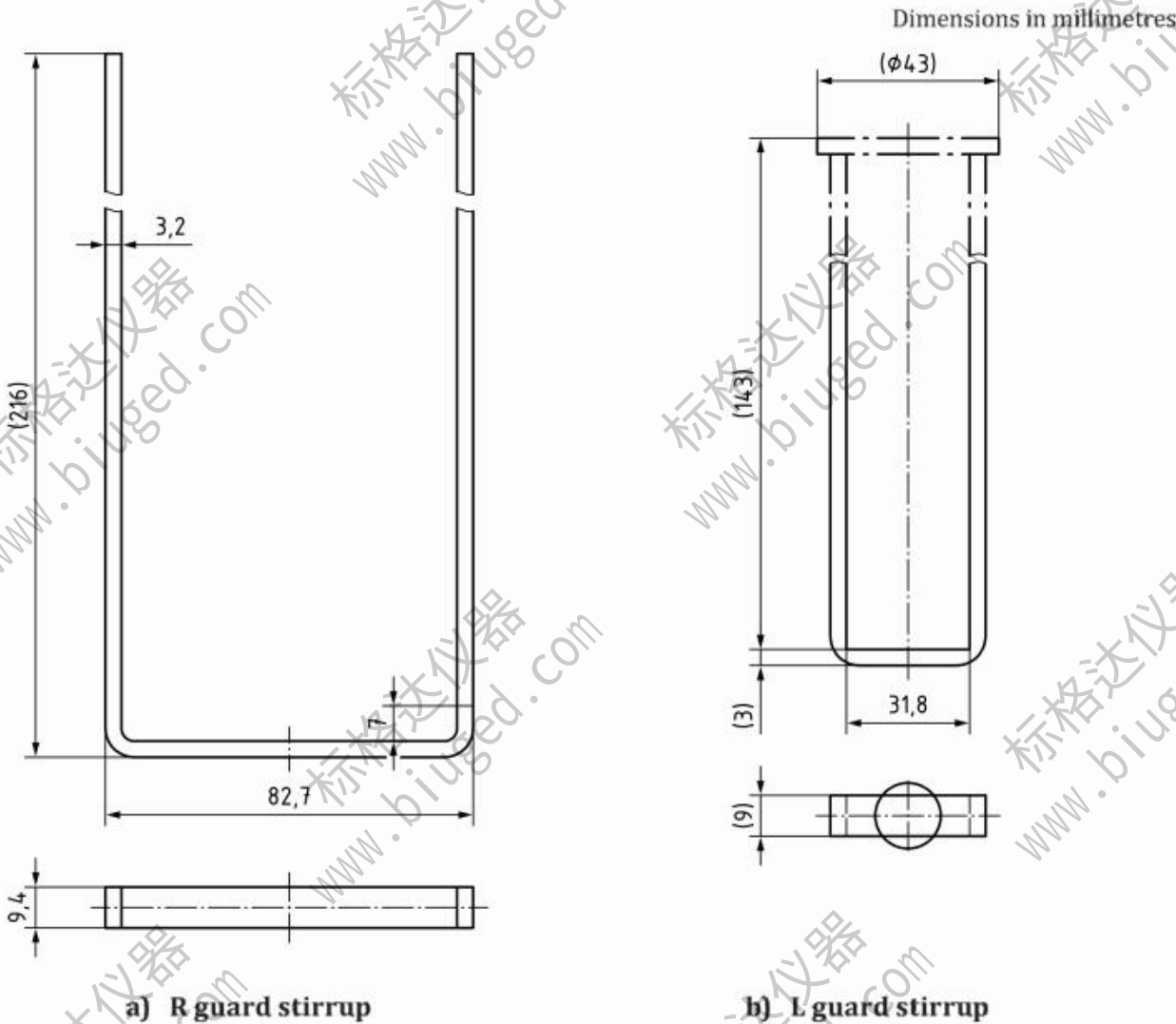


Figure B.1 — Shapes and dimensions of guard stirrups

The shapes and dimensions of spindles are given in [Figure B.2](#) and [Table B.1](#).

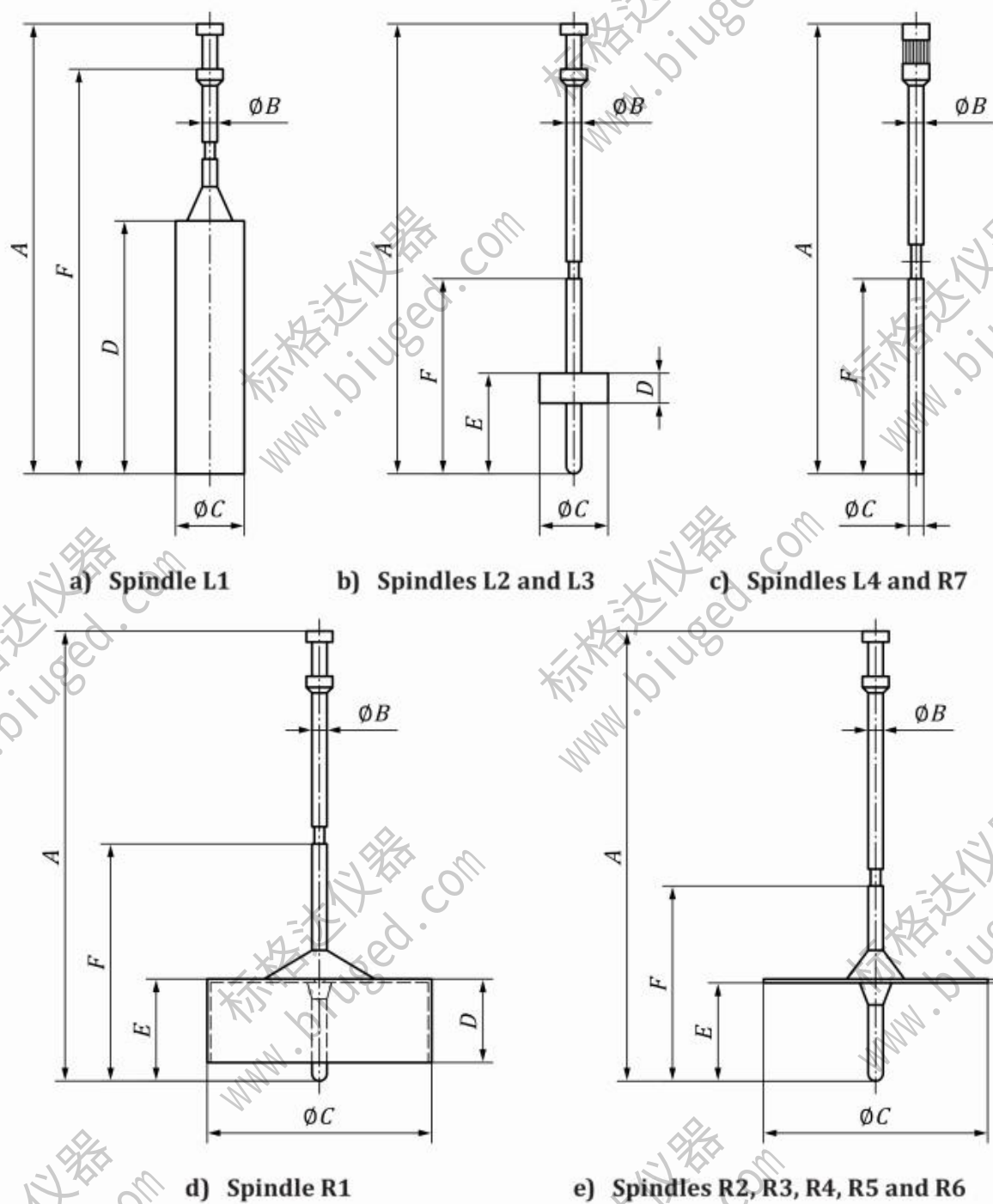


Figure B.2 — Shapes and dimensions of spindles