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Plastics — Poly(vinyl alcohol) (PVAL) materials —

Part 2:
Determination of properties

Plastiques — Matériaux en poly(alcool de vinyle) (PVAL) —

Partie 2: Détermination des propriétés

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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 61, *Plastics*, Subcommittee SC 9, *Thermoplastic materials*.

This second edition cancels and replaces the first edition (ISO 15023-2:2003), which has been technically revised. The main changes compared to the previous edition are as follows:

- the normative references have been updated;
- adjustments have been made due to a new designation system in ISO 15023-1.

A list of all parts in the ISO 15023 series can be found on the ISO website.

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.

Plastics — Poly(vinyl alcohol) (PVAL) materials —

Part 2:
Determination of properties

1 Scope

This document specifies the methods to be used in determining the properties of poly(vinyl alcohol), which is normally prepared by hydrolysis of poly(vinyl acetate) and whose composition comprises vinyl alcohol monomeric units and vinyl acetate monomeric units. This document is applicable to poly(vinyl alcohol) with a vinyl alcohol unit content (degree of hydrolysis) from 70 mol% to 100 mol%.

In addition to the designatory properties specified in ISO 15023-1 (degree of hydrolysis and viscosity of an aqueous solution), this document includes a number of other properties which are commonly used to specify PVAL materials (see Table 1).

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 976, *Rubber and plastics — Polymer dispersions and rubber latices — Determination of pH*

ISO 6587, *Paper, board and pulps — Determination of conductivity of aqueous extracts*

ISO 8130-1, *Coating powders — Part 1: Determination of particle size distribution by sieving*

ISO 12058-1, *Plastics — Determination of viscosity using a falling-ball viscometer — Part 1: Inclined-tube method*

3 Terms and definitions

No terms and definitions are listed in this document.

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/

4 Determination of properties

In the determination of properties and the presentation of results, the standards, methods and special conditions listed in Table 1 shall apply. The properties listed in Table 1 are those appropriate to poly(vinyl alcohol).

Table 1 — Properties and test conditions

Property	Method	Unit	Test conditions and supplementary instructions
Volatile-matter content	Annex A	% by mass	105 °C, 3 h
Sodium acetate content	Annex B	% by mass	Titration or conductivity method
Ash	Annex C	% by mass	void
Degree of hydrolysis	Annex D	mol%	Titration method
Viscosity of 4 % aqueous solution	Annex E	mPa·s	Rotational or inclined-tube falling-ball viscometer, 20 °C
Particle size distribution	ISO 8130-1	%	void
pH of aqueous solution	ISO 976	—	Concentration (4,0 ± 0,2) %

Annex A
(normative)

Determination of volatile-matter content

A.1 Overview

This annex specifies the method to be used for the determination of the volatile-matter content of PVAL.

A.2 Principle

The volatile-matter content is calculated from the loss in mass of a specimen heated at 105 °C for 3 h.

A.3 Test method

A.3.1 Apparatus

A.3.1.1 Constant-temperature oven, able to maintain a temperature of (105 ± 2) °C.

A.3.1.2 Weighing dish, shallow, about 60 mm in diameter and 30 mm in height, of glass, aluminium or preferably stainless steel, with a lid.

A.3.1.3 Balance, capable of weighing to 0,001 g.

A.3.1.4 Desiccator, containing silica gel as a drying agent.

A.3.2 Procedure

Carry out the determination in duplicate.

Weigh the dish (A.3.1.2) with its lid to the nearest 0,001 g (m_0), after heating it in the oven (A.3.1.1) maintained at (105 ± 2) °C for 1 h and cooling it to room temperature in the desiccator (A.3.1.4). Spread about 5 g of PVAL resin evenly over the bottom of the dish, replace the lid and weigh to the nearest 0,001 g (m_1). Place the assembly in the oven at (105 ± 2) °C, remove the lid (leaving it in the oven) and close the oven door. After 3 h ± 5 min, remove the assembly from the oven (lid on), allow to cool in the desiccator and weigh to the nearest 0,001 g (m_2).

A.4 Expression of results

Calculate the volatile-matter content, w_{VM}, as a percentage by mass using Formula (A.1):

$$w_{VM} = \frac{m_1 - m_2}{m_1 - m_0} \times 100 \quad \text{(A.1)}$$

where

m_0 is the mass, in g, of the dish;

m_1 is the initial mass, in g, of the dish plus test portion;

m_2 is the mass, in g, of the dish plus test portion after heating.

Calculate the mean of the results of the two determinations and express the final result to two places of decimals.

A.5 Test report

The test report shall include the following particulars:

a) a reference to this document, i.e. ISO 15023-2:2019;

b) all details necessary for complete identification of the material tested;

c) the volatile-matter content, calculated as the arithmetic mean of the two determinations;

d) the individual results of the two determinations;

e) the date of the test.

Annex B
(normative)

Determination of sodium acetate content

B.1 Overview

This annex specifies the method to be used for the determination of the sodium acetate content of PVAL. The content can be determined either by a titration method or by a conductivity method. Additives can interfere with the determination, and these methods may not be suitable for PVAL containing them.

B.2 Principle

B.2.1 Titration method

The sample is dissolved in water and the solution titrated with hydrochloric acid using methylene blue and methyl yellow mixture as indicator. The sodium acetate content is calculated as a percentage by mass.

B.2.2 Conductivity method

The conductivity of an aqueous solution of the sample is measured in a conductometer. The sodium acetate content of the solution is determined by calibrating the conductometer with solutions containing known amounts of sodium acetate.

B.3 Titration method

B.3.1 Reagents

B.3.1.1 Hydrochloric acid, 0,1 mol/l.

B.3.1.2 Methylene blue/methyl yellow mixed indicator, mixed methylene blue/methyl yellow indicator, prepared by mixing equal parts of a 0,1 % solution of methylene blue in reagent-grade ethanol with a 0,1 % solution of methyl yellow in reagent-grade ethanol.

B.3.2 Apparatus

B.3.2.1 Conical flask, 300 ml, with ground-glass stopper.

B.3.2.2 Cylinder, 200 ml, graduated in 2 ml.

B.3.2.3 Burette, 50 ml, graduated in 0,1 ml.

B.3.3 Procedure

Carry out the determination in duplicate.

Weigh about 5 g of sample to the nearest 0,001 g into the conical flask (B.3.2.1), add about 150 ml of water and dissolve by heating.

A sample with a low degree of hydrolysis may sometimes cause the solution to become turbid. If this occurs, cool the solution slowly while stirring gently. Alternatively, a 3:1 water/methanol mixture may be used.

After dissolution, cool and titrate with 0,1 mol/l hydrochloric acid to an end point where the solution turns from green to light purple.

Carry out a blank test separately.

B.3.4 Expression of results

Calculate the sodium acetate content, w_{NaAc}, as a percentage by mass using Formula (B.1):

$$w_{\text{NaAc}} = \frac{(V_1 - V_0) \times c \times M_{\text{NaAc}}}{m \times 1000} \times 100 \qquad \text{(B.1)}$$

where

V_1 is the volume, in ml, of hydrochloric acid required for the test solution;

V_0 is the volume, in ml, of hydrochloric acid required for the blank;

c is the actual concentration, in mol/l, of the hydrochloric acid;

M_{NaAc} is the molecular mass of sodium acetate, in g/mol (82,03 g/mol);

m is the mass, in g, of the test portion.

Calculate the mean of the results of the two determinations and express the final result to two places of decimals.

In high alkali and low alkali PVAL manufacturing process sodium hydroxide may be present in the product sample, special attention should be drawn to the influence of sodium hydroxide on sodium acetate and hydrolysis result. However, if the determination result of sodium acetate is merely used for ash content calculation, the influence could be ignored.

B.4 Conductivity method

B.4.1 Reagent

B.4.1.1 Sodium acetate, reagent grade.

B.4.2 Apparatus

B.4.2.1 Conical flask, 100 ml, with ground-glass stopper.

B.4.2.2 Cylinder, 100 ml, graduated in 1 ml.

B.4.2.3 Conductivity meter, as specified in ISO 6587.

B.4.2.4 Thermometer, graduated in 0,1 °C.

B.4.2.5 Volumetric flask, 100 ml, with ground-glass stopper.

B.4.3 Procedure

Carry out the determination in duplicate.

Weigh 0,5 g of sample to the nearest 0,001 g into a conical flask, add about 50 ml of water and dissolve by heating.

A sample with a low degree of hydrolysis may sometimes cause the solution to become turbid. If this occurs, cool the solution slowly while stirring gently.

Transfer the solution to a 100 ml volumetric flask and make up to the mark with water.

Transfer about 50 ml of the aqueous solution in the volumetric flask to the measuring cell of the conductivity meter. Measure the conductivity at 30 °C ± 0,1 °C.

B.4.4 Calibration curve

Prepare the calibration curve as follows.

- Prepare four or five aqueous sodium acetate solutions covering a suitable range of concentrations and measure their conductivities.
- Prepare the calibration curve by plotting the conductivity values against the corresponding sodium acetate concentrations (g/100 ml).

B.4.5 Expression of results

Calculate the sodium acetate content, w_{NaAc}, as a percentage by mass using Formula (B.2):

$$w_{\mathrm{NaAc}} = \frac{m_{\mathrm{NaAc}}}{m} \times 100 \qquad \text{(B.2)}$$

where

m_{NaAc} is the mass of sodium acetate in the 100 ml test solution, expressed in g, obtained from the calibration curve;

m is the mass, in g, of the test portion.

Calculate the mean of the results of the two determinations and express the final result to two places of decimals.

B.5 Test report

The test report shall include the following particulars:

a) a reference to this document, i.e. ISO 15023-2:2019;

b) all details necessary for complete identification of the material tested;

c) the sodium acetate content, calculated as the arithmetic mean of the two determinations;

d) the individual results of the two determinations;

e) the date of the test.

Annex C
(normative)

Calculation of ash

C.1 Overview

This annex specifies the method to be used for the calculation of the ash of PVAL.

C.2 Principle

The ash expressed as sodium oxide is calculated from the sodium acetate content determined in Annex B.

C.3 Calculation

From each of the sodium acetate contents determined in Annex B, calculate the ash, w_{Ash}, as a percentage by mass, using Formula (C.1):

$$w_{\mathrm{Ash}} = w_{\mathrm{NaAc}} \times 0{,}378 \tag{C.1}$$

where

w_{NaAc} is the sodium acetate content, in %;

0,378 is a factor for converting the mass of sodium acetate into the corresponding mass of sodium oxide (molecular mass of sodium oxide × 0,5/molecular mass of sodium acetate).

Calculate the mean of the two results and express the final result to two places of decimals.

C.4 Test report

The test report shall include the following particulars:

a) a reference to this document, i.e. ISO 15023-2:2019;

b) all details necessary for complete identification of the material tested;

c) the ash, calculated as the arithmetic mean of the two results;

d) the individual results of the two calculations;

e) the date of the test.

Annex D
(normative)

Determination of degree of hydrolysis

D.1 Overview

This annex specifies the method to be used for the determination of the degree of hydrolysis of PVAL. The method is intended for PVAL without plasticizers and free of additives, fillers, dyes and any other materials which could interfere with the determination. When they are present, they shall be separated by a method agreed between the contracting parties. This method is applicable to PVAL with a nominal degree of hydrolysis from 70 mol% up to 100 mol%.

D.2 Principle

The degree of hydrolysis is obtained by determining the residual acetic acid group content, in mol%, by titration with sodium hydroxide and subtracting the result from 100 mol%.

D.3 Calculation

Calculate the degree of hydrolysis, *H*, of the vinyl alcohol unit in the poly(vinyl alcohol), in mol%, as given by Formula (D.1):

$$H = \frac{\text{Vinyl alcohol unit content}}{\text{Vinyl alcohol unit content} + \text{Vinyl acetate unit content}} \times 100 \qquad \text{(D.1)}$$

D.4 Reagents

D.4.1 Sodium hydroxide solution, 0,1 mol/l.

D.4.2 Sodium hydroxide solution, 0,5 mol/l.

D.4.3 Sulfuric acid or hydrochloric acid, 0,1 mol/l.

D.4.4 Sulfuric acid or hydrochloric acid, 0,5 mol/l.

D.4.5 Phenolphthalein solution, 10 g/l, in 90 % (by volume) ethyl alcohol.

D.5 Apparatus

D.5.1 Conical flask, 300 ml, with ground-glass stopper.

D.5.2 Burettes, 25 ml, graduated in 0,1 ml.

D.5.3 Measuring cylinder, 100 ml, graduated in 1 ml.

D.5.4 Dissolving apparatus, capable of heating with stirring.

D.5.5 **Balance**, accurate to 0,001 g.

D.6 Procedure

D.6.1 Carry out the determination in duplicate.

D.6.2 Weigh, to the nearest 0,001 g, a test portion of about the mass specified in Table D.1 into the conical flask (D.5.1).

D.6.3 Add 100 ml of water and 3 drops of phenolphthalein solution (D.4.5) and heat to a temperature of at least 90 °C for 30 min in the dissolving apparatus (D.5.4) to dissolve the test portion completely, leaving the conical flask open to allow volatile organic matter in the test portion to escape.

D.6.4 A sample with a low degree of hydrolysis may sometimes cause the solution to become turbid. If this occurs, cool the solution slowly while stirring gently. Alternatively, a 3:1 water/methanol mixture may be used.

D.6.5 After cooling to room temperature, add 25 ml of 0,1 mol/l or 0,5 mol/l sodium hydroxide solution (D.4.1 or D.4.2) as specified in Table D.1 from a burette (D.5.2). Stopper the flask, stir, and allow to stand at room temperature for 2 h or more (or boil under reflux).

D.6.6 Add to the flask from a burette (D.5.2) 25 ml of sulfuric or hydrochloric acid (D.4.3 or D.4.4) of the same concentration as the sodium hydroxide solution used in D.6.4, stopper the flask and stir well.

D.6.7 Titrate excess sulfuric or hydrochloric acid with sodium hydroxide solution, of the same concentration as used in D.6.4, to a faint pink end point.

D.6.8 Separately, carry out a blank test following the procedure described in D.6.3 to D.6.6, i.e. without the test portion.

Table D.1 — Test portion size and concentration of reagent solutions used

Estimated degree of hydrolysis mol%	Test portion g	Concentration of reagent solutions used g/mol
At least 97	3	0,1
At least 90 but less than 97	3	0,5
At least 80 but less than 90	2	0,5
At least 70 but less than 80	1	0,5

D.7 Expression of results

Calculate the degree of hydrolysis, *H*, in mol%, using Formulae (D.2) to (D.4):

$$X_1 = \frac{(V_1 - V_0)\times c\times 0{,}060\,05}{m\times\left(1-\frac{w_{\text{VM}}+w_{\text{NaAc}}}{100}\right)}\times 100 \qquad \text{(D.2)}$$

$$X_2 = \frac{44{,}05\times X_1}{60{,}05-0{,}42\times X_1} \qquad \text{(D.3)}$$

$$H = 100 - X_2 \tag{D.4}$$

where

X_1	is the amount of acetic acid, in percent by mass, corresponding to the residual acetate content;
X_2	is the residual acetate content, in mol%;
V_1	is the volume, in ml, of 0,1 mol/l or 0,5 mol/l sodium hydroxide solution consumed by the test solution;
V_0	is the volume, in ml, of 0,1 mol/l or 0,5 mol/l sodium hydroxide solution consumed by the blank;
c	is the concentration of the reagent solutions used (0,1 mol/l or 0,5 mol/l);
0,060 05	is the molecular mass of acetic acid divided by 1 000;
m	is the mass, in g, of the test portion;
w_{VM}	is the volatile-matter content, in percent by mass, of the PVAL (see Annex A);
w_{NaAc}	is the sodium acetate content, in percent by mass, of the PVAL (see Annex B);
44,05	is the molecular mass of a vinyl alcohol unit;
60,05	is the molecular mass of acetic acid;
0,42	is a factor obtained from the following formula relating X_1 and X_2:

$$X_1 = \frac{X_2 \times 60{,}05}{X_2 \times 86{,}09 + (100 - X_2) \times 44{,}05} \times 100$$

where 86,09 is the molecular mass of vinyl acetate.

Calculate the mean of the results of the two determinations and express the final result to two places of decimals.

D.8 Test report

The test report shall include the following particulars:

a) a reference to this document, i.e. ISO 15023-2:2019;

b) all details necessary for complete identification of the material tested;

c) the degree of hydrolysis, calculated as the arithmetic mean of the two determinations;

d) the individual results of the two determinations;

e) the date of the test.

Annex E

(normative)

Determination of viscosity of 4 % aqueous solution by the Brookfield test method or the inclined-tube falling-ball method

E.1 Overview

This annex specifies methods of determining the viscosity of a 4 % aqueous solution of PVAL by the Brookfield test method (see ISO 1652) or the inclined-tube falling-ball method.

E.2 Principle

Three aqueous solutions of PVAL spanning a range of concentrations each side of 4 % are prepared and their viscosities measured with a Brookfield-type viscometer or an inclined-tube falling-ball viscometer. The viscosity at a concentration of 4 % is obtained from a natural logarithm of viscosity-concentration graph.

E.3 Brookfield test method

E.3.1 Apparatus

E.3.1.1 Brookfield-type viscometer[1], Model LVF or Model LVT (see Note), chosen according to the viscosity range in which the measurements are to be made (see Table E.2). The Brookfield UL adapter accessory can be used with both models. Do not use spindles which show signs of corrosion or eccentricity.

NOTE The Model LVF has four rotational frequencies, and the Model LVT has eight (see Table E.1). Each has a set of four spindles. The Brookfield test method can be carried out using equipment supplied by a number of manufacturers.

Table E.1 — Rotational frequencies available for the two models of viscometer

Model	Rotational frequencies min^{-1}							
LVF	60	30	12	6	—	—	—	—
LVT	60	30	12	6	3	1,5	0,6	0,3

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[1] Suitable instruments are commercially obtainable from several sources, such as the Brookfield-type viscometer. This information is given for the convenience of the users of this International Standard and does not constitute an endorsement by ISO of these products.

Table E.2 — Maximum value of viscosity as a function of viscometer type, rotational frequency and spindle

Model	Rotational frequency min^{-1}	Spindle				
		UL adapter	No. 1	No. 2	No. 3	No. 4
		Maximum viscosity mPa·s				
LVF and LVT	60	10	100	500	2 000	10 000
	30	20	200	1 000	4 000	20 000
	12	50	500	2 500	10 000	50 000
	6	100	1 000	5 000	20 000	100 000
LVT only	3	200	2 000	10 000	40 000	200 000
	1,5	400	4 000	20 000	80 000	400 000
	0,6	1 000	10 000	50 000	200 000	1 000 000
	0,3	2 000	20 000	100 000	400 000	2 000 000

Adjustment and calibration of these viscometers are usually carried out by the manufacturer.

It is recommended that the adjustment and calibration be checked from time to time by means of pure-Newtonian liquids of known viscosity, either in the user's laboratory or by an accredited calibration laboratory.

E.3.1.2 Conical flasks, 500 ml.

E.3.1.3 Beaker, of diameter at least 65 mm and capacity at least 300 ml.

E.3.1.4 Thermostatic water bath, able to maintain a temperature of (20,0 ± 0,1) °C.

E.3.1.5 Weighing dish, shallow, 60 mm in diameter and 30 mm in height, of glass, aluminium or (preferably) stainless steel, with a lid.

E.3.1.6 Drying oven, able to maintain a temperature of (105 ± 20) °C.

E.3.1.7 Desiccator, with silica gel as drying agent.

E.3.1.8 Water bath, usable with boiling water, in which the weighing dish (E.3.1.5) can be placed.

E.3.2 Procedure

E.3.2.1 Number of determinations

Carry out the determination in duplicate.

E.3.2.2 Dissolution of sample

Weigh, to the nearest 0,001 g, three test portions, each of at least 15 g, into three separate conical flasks (E.3.1.2).

Add water in amounts calculated using Formula (E.1) to prepare aqueous solutions with concentrations of 3,8 %, 4,0 % and 4,2 % by mass:

$$m_W = \frac{m_0 \times (100 - w_{VM})}{c_s} - m_0 \tag{E.1}$$

where

m_W is the mass, in g, of water to be added;

m_0 is the mass, in g, of the test portion;

w_{VM} is the volatile-matter content, as a percentage by mass;

c_s is the desired concentration of the solution.

Heat the flasks to dissolve the test portions completely, and allow them to cool to about 20 °C, de-aerating the solutions completely.

A sample with a low degree of hydrolysis may sometimes cause the solution to become turbid. If this occurs, cool the solution slowly while stirring gently.

E.3.2.3 Measurement of viscosity

E.3.2.3.1 Mount the viscometer (E.3.1.1), with its guard stirrup fitted if applicable, on its stand.

E.3.2.3.2 Fill the beaker (E.3.1.3) with one of the test solutions, taking care not to introduce air bubbles, then place it in the thermostatic water bath (E.3.1.4) for a sufficient time to reach (20,0 ± 0,1) °C.

E.3.2.3.3 With the beaker still in the bath, hold the chosen spindle (see E.3.2.3.4) at an angle of approximately 45° to the surface of the solution and lower it into the solution. Orientate the spindle vertically, and connect it to the shaft of the viscometer. Check that the spindle is vertical and that it is immersed to the underside of the mark on its shaft.

E.3.2.3.4 It is necessary to choose a spindle and rotational frequency such that no measurements correspond 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.

E.3.2.3.5 Start the motor and run at the chosen rotational frequency (see E.3.2.3.4), monitoring the torque meter. When a stable value has been reached on the meter, lock the needle and the motor to take the reading.

E.3.2.3.6 Repeat with the remaining test solutions.

E.3.2.4 Measurement of exact concentrations of test solutions

Place a previously washed weighing dish (E.3.1.5) in the drying oven (E.3.1.6) at (105 ± 2) °C and dry for at least 1 h. Allow the dish to cool to room temperature in the desiccator (E.3.1.7) and weigh to the nearest 0,001 g.

Place about 5 g of one of the test solutions prepared in E.3.2.2 in the weighing dish and weigh to the nearest 0,001 g.

Evaporate the solution to dryness on the boiling-water bath (E.3.1.8) and dry at (105 ± 2) °C in the oven for at least 4 h.

After drying, allow the dish to cool to room temperature in the desiccator and weigh to the nearest 0,001 g.

Repeat with the remaining test solutions.

E.3.2.5 Calculation

Calculate the exact concentration, c_s, of each test solution, as a percentage by mass, using Formula (E.2):

$$c_s = \frac{m_2 - m_3}{m_1 - m_3} \times 100 \qquad \text{(E.2)}$$

where

m_1 is the total mass, in grams, of the test solution and weighing dish before drying;

m_2 is the total mass, in grams, of the test solution and weighing dish after drying;

m_3 is the mass, in grams, of the weighing dish.

E.3.2.5.1 Calculate the viscosity, η, in mPa·s, of each test solution using Formula (E.3):

$$\eta = \kappa \times \theta \qquad \text{(E.3)}$$

where

κ is a coefficient depending on the rotational-frequency/spindle combination used (see Table E.3);

θ is the value read from the scale (0 to 100).

Table E.3 — Coefficient, κ, for each rotational-frequency/spindle combination

Spindle[a]	**Rotational frequency** min^{-1}							
	60	**30**	**12**	**6**	**3**	**1,5**	**0,6**	**0,3**
	Coefficient, κ							
UL adapter	0,1	0,2	0,5	1	2	4	10	20
No. 1	1	2	5	10	20	40	100	200
No. 2	5	10	25	50	100	200	500	1 000
No. 3	20	40	100	200	400	800	2 000	4 000
No. 4	100	200	500	1 000	2 400	4 000	10 000	20 000

[a] It may be necessary to calibrate each spindle with liquids of known viscosity and to use slightly different values of κ.

E.4 Inclined-tube falling-ball method

E.4.1 Apparatus

E.4.1.1 Conical flasks, 300 ml.

E.4.1.2 Thermostatic water bath, able to maintain a temperature of (20,0 ± 0,1) °C.

E.4.1.3 Inclined-tube falling-ball viscometer with steel balls, with a falling time of at least 10 s, as specified in ISO 12058-1.