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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

Reference number
ISO 15023-2:2003(E)

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Foreword

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International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

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.

ISO 15023-2 was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 9, *Thermoplastic materials*.

ISO 15023 consists of the following parts, under the general title *Plastics — Poly(vinyl alcohol) (PVAL) materials*:

- *Part 1: Designation system and basis for specifications*
- *Part 2: Determination of properties*

INTERNATIONAL STANDARD **ISO 15023-2:2003(E)**

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

Part 2:
Determination of properties

1 Scope

This part of ISO 15023 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 part of ISO 15023 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 part of ISO 15023 includes a number of other properties which are commonly used to specify PVAL materials (see Table 1).

2 Normative references

The following referenced documents are indispensable for the application 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 976:1996, *Rubber and plastics — Polymer dispersions and rubber latices — Determination of pH*

ISO 6587:1992, *Paper, board and pulps — Determination of conductivity of aqueous extracts*

ISO 8130-1:1992, *Coating powders — Part 1: Determination of particle size distribution by sieving*

ISO 12058-1:1997, *Plastics — Determination of viscosity using a falling-ball viscometer — Part 1: Inclined-tube method*

ISO 15023-1:2001, *Plastics — Poly(vinyl alcohol) (PVAL) materials — Part 1: Designation system and basis for specifications*

3 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	
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	%	
pH of aqueous solution	ISO 976	—	Concentration (4,0 ± 0,2) %

Annex A
(normative)

Determination of volatile-matter content

A.1 Scope

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 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 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, 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, from the following equation:

$$w_{\text{VM}} = \frac{m_1 - m_2}{m_1 - m_0} \times 100$$

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 part of ISO 15023;

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 Scope

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 methyl orange 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 **Methyl orange indicator,** 1 g/l solution in ethanol, or **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 orange-yellow to red if methyl orange is used as indicator or from green to light purple if methylene blue/methyl yellow is used.

Carry out a blank test separately.

B.3.4 Expression of results

Calculate the sodium acetate content w_{NaAc}, as a percentage by mass, from the following equation:

$$w_{\text{NaAc}} = \frac{(V_1 - V_0) \times c \times 0{,}082\ 03}{m} \times 100$$

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;
0,082 03	is the molecular mass of sodium acetate divided by 1 000;
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.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, from the following equation:

$$w_{\text{NaAc}} = \frac{\rho_{\text{NaAc}}}{m} \times 100$$

where

ρ_{NaAc} is the concentration of sodium acetate in the test solution, in g/100 ml, 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 part of ISO 15023;

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 Scope

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, from the following equation:

$$w_{\text{Ash}} = w_{\text{NaAc}} \times 0{,}378$$

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 part of ISO 15023;

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 Scope

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 must 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 Terms and definitions

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

D.2.1
degree of hydrolysis
the molar percentage, in mol %, of the vinyl alcohol unit in the poly(vinyl alcohol), as given by the equation:

$$\text{Degree of hydrolysis} = \frac{\text{Vinyl alcohol unit content}}{\text{Vinyl alcohol unit content} + \text{Vinyl acetate unit content}} \times 100$$

D.3 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.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.

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.6.4 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.6.5 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.6.6 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.6.7 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** mol/l
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 %, from the following equations:

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

$$X_2 = \frac{44{,}05 \times X_1}{60{,}05 - 0{,}42 \times X_1}$$

$$H = 100 - X_2$$

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 equation 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 part of ISO 15023;

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 Scope

This annex specifies methods of determining the viscosity of a 4 % aqueous solution of PVAL by the Brookfield Test method 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 concentration-viscosity graph.

E.3 Brookfield Test method

E.3.1 Apparatus

E.3.1.1 Brookfield-type viscometer, 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 may 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

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

E.3.2.2.1 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).

E.3.2.2.2 Add water in amounts calculated using the following equation to prepare aqueous solutions with concentrations of 3,8 %, 4,0 % and 4,2 % by mass:

$$m_\text{w} = \frac{m_0 \times (100 - w_\text{VM})}{c_\text{s}} - m_0$$

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.

E.3.2.2.3 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 Note) 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.

NOTE 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.3.2.3.4 Start the motor and run at the chosen rotational frequency (see note to E.3.2.3.3), 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.3.2.3.4 Repeat with the remaining test solutions.

E.3.2.4 Measurement of exact concentrations of test solutions

E.3.2.4.1 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.

E.3.2.4.2 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.

E.3.2.4.3 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.

E.3.2.4.4 After drying, allow the dish to cool to room temperature in the desiccator and weigh to the nearest 0,001 g.

E.3.3.4.5 Repeat with the remaining test solutions.

E.3.2.5 Calculation

E.3.2.5.1 Calculate the exact concentration c_S of each test solution, as a percentage by mass, using the following equation:

$$c_\mathrm{S} = \frac{m_2 - m_3}{m_1 - m_3} \times 100$$

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.3.2.5.2 Calculate the viscosity η, in mPa·s, of each test solution using the following equation:

$$\eta = \kappa \times \theta$$

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.

E.4.1.4 **Oven,** able to maintain a temperature of (105 ± 2) °C.

E.4.1.5 **Desiccator,** containing silica gel as the drying agent.

E.4.1.6 **Stopwatch,** able to measure to 0,1 s accurately.

E.4.2 Procedure

E.4.2.1 Number of determinations

Carry out the determination in duplicate.

E.4.2.2 Dissolution of sample

Make up three test solutions following the same procedure as in E.3.2.2.

E.4.2.3 Measurement of viscosity

E.4.2.3.1 Place each of the test solutions prepared in the falling-ball viscometer and adjust the temperature of the solution to (20,0 ± 0,1) °C.

E.4.2.3.2 Measure to the nearest 0,1 s with the stopwatch the time required for the steel ball to travel the distance between the upper and lower graduations.

E.4.2.4 Determination of exact concentrations of test solutions

Determine the exact concentration of each test solution by the procedure given in E.3.2.4 and the calculation given in E.3.2.5.1.

E.4.2.5 Calculation

Calculate the viscosity η, in mPa·s, of each test solution using the following equation:

$$\eta = K \times (\rho_1 - \rho_2) \times t$$

where

ρ_1 is the density, in g/cm^3, of the ball at 20 °C;

ρ_2 is the density, in g/cm^3, of a 4 % aqueous solution of PVAL at 20 °C (normally taken as 1,008 g/cm^3);

t is the time, in seconds, taken for the ball to travel between the two graduations;

K is the viscometer calibration constant for the ball used, obtained as follows:

$$K = \eta_0 / (\rho_1 - \rho_3) \times t$$

where

η_0 is the absolute viscosity, in mPa·s, of a standard solution (see Note) used to calibrate the viscometer at 20 °C;

ρ_1 is the density, in g/cm^3, of the ball at 20 °C;

ρ_3 is the density, in g/cm^3, of a standard solution (see Note) used to calibrate the viscometer at 20 °C;

t is the time, in seconds, taken for the ball to travel between the two graduations.

NOTE The standard solution is a pure-Newtonian liquid of known viscosity, with which the time taken for the ball to travel between the two graduations is at least 10 s.

E.5 Expression of results

Plot a graph with the ordinate representing the viscosity and the abscissa the concentration determined for each test solution, and read off it the viscosity, in mPa·s, at 4 % concentration.

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

E.6 Test report

The test report shall include the following particulars:

a) a reference to this part of ISO 15023;

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

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

d) the individual results of the two determinations;

e) the date of the test.

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