INTERNATIONAL STANDARD

ISO 15023-2

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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
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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.

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*

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:

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

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:

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

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:

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

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.