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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):

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

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.