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Plastics — Ethylene/vinyl alcohol (EVOH) copolymer moulding and extrusion materials —

Part 2:

Preparation of test specimens and determination of properties

Plastiques — Matériaux à base de copolymères éthylène/alcool vinylique (EVOH) pour moulage et extrusion —

Partie 2: Préparation des éprouvettes et détermination des propriétés

ISO 21309-2:2019

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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 first edition of ISO 21309-2 cancels and replaces ISO 14663-2:1999, which has been technically revised. The main changes compared to the previous edition are as follows:

- the number of the standard has been changed;
- the normative references have been updated.

A list of all parts in the ISO 21309 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 — Ethylene/vinyl alcohol (EVOH) copolymer moulding and extrusion materials —

Part 2:

Preparation of test specimens and determination of properties

1 Scope

This document specifies the methods of preparation of test specimens and the test methods to be used in determining the properties of ethylene/vinyl alcohol (EVOH) copolymer moulding and extrusion materials. It gives requirements for handling test material and for conditioning both the test material before moulding and the specimens before testing.

This document describes procedures and conditions for the preparation of test specimens, and procedures for measuring properties of the materials from which these specimens are made. Properties and test methods which are suitable and necessary to characterize EVOH moulding and extrusion materials are listed in this document.

The properties have been selected from the general test methods in ISO 10350-1. Other test methods in wide use for or of particular significance to these moulding and extrusion materials are also included in this document, as is the melt mass-flow rate designatory property specified in ISO 21309-1.

In order to obtain reproducible and comparable test results, it is intended to use the methods of specimen preparation and conditioning, the specimen dimensions and the test procedures specified herein. Values determined will not necessarily be identical to those obtained using specimens of different dimensions or prepared using different procedures.

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 75-1:2013, Plastics — Determination of temperature of deflection under load — Part 1: General test method

ISO 75-2:2013, Plastics — Determination of temperature of deflection under load — Part 2: Plastics and ebonite

ISO 178, Plastics — Determination of flexural properties

ISO 179-1:2010, Plastics — Determination of Charpy impact properties — Part 1: Non-instrumented impact test

ISO 180:2000, Plastics — Determination of Izod impact strength

ISO 291:2008, Plastics — Standard atmospheres for conditioning and testing

ISO 294-2, Plastics — Injection moulding of test specimens of thermoplastic materials — Part 2: Small tensile bars

ISO 306, Plastics — Thermoplastic materials — Determination of Vicat softening temperature (VST)

ISO 21309-2:2019(E)

ISO 527-1, Plastics — Determination of tensile properties — Part 1: General principles

ISO 527-2, Plastics — Determination of tensile properties — Part 2: Test conditions for moulding and extrusion plastics

ISO 899-1, Plastics — Determination of creep behaviour — Part 1: Tensile creep

ISO 1133-1, Plastics — Determination of the melt mass-flow rate (MFR) and melt volume-flow rate (MVR) of thermoplastics — Part 1: Standard method

ISO 1183-1:2012, Plastics — Methods for determining the density of non-cellular plastics — Part 1: Immersion method, liquid pyknometer method and titration method

ISO 2039-2, Plastics — Determination of hardness — Part 2: Rockwell hardness

ISO 3146:2000, Plastics — Determination of melting behaviour (melting temperature or melting range) of semi-crystalline polymers by capillary tube and polarizing-microscope methods

ISO 3451-1:2008, Plastics — Determination of ash — Part 1: General methods

 ${\tt ISO~10350-1}, \textit{Plastics} - \textit{Acquisition and presentation of comparable single-point data} - \textit{Part 1: Moulding materials}$

ISO 20753, Plastics — Test specimens

ISO 21309-1, Plastics — Ethylene/vinyl alcohol (EVOH) copolymer moulding and extrusion materials — Part 1: Designation system and basis for specifications

IEC 60112, Method for determining the comparative and the proof tracking indices of solid insulating materials under moist conditions

IEC 60243-1, Electrical strength of solid insulating materials — Test methods — Part 1: Tests at power frequencies

IEC 60250, Recommended methods for the determination of the permittivity and dielectric dissipation factor of electrical insulating materials at power, audio and radio frequencies including metre wavelengths

IEC 60296, Specification for unused mineral insulating oils for transformers and switchgear

IEC 60695-11-10, Fire hazard testing — Part 11-10: Test flames — 50 W horizontal and vertical flame test methods

IEC 62631-3-1, Dielectric and resistive properties of solid insulating materials — Part 3-1: Determination of resistive properties (DC methods) — Volume resistance and volume resistivity — General method

IEC 62631-3-2, Dielectric and resistive properties of solid insulating materials — Part 3-2: Determination of resistive properties (DC methods) — Surface resistance and surface resistivity

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:

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

3.1

oxygen gas transmission rate O₂GTR

volume of oxygen gas passing through unit area of the parallel surfaces of a film per unit time under specific conditions

Note 1 to entry: The rate is usually expressed in cubic centimetres (at 0 °C under standard atmospheric pressure) per square metre per 24 h under a pressure difference of 1 atm [cm 3 /(m 2 ·24 h·atm)].

Note 2 to entry: The SI unit for O_2GTR is the femtometre per pascal second [fm/(Pa·s)]:

1 fm (femtometre) = 10^{-15} m 1 atm = 101,3 kPa

 $1 \text{ cm}^3/(\text{m}^2 \cdot 24 \text{ h·atm}) = 0.114 \text{ 3 fm}/(\text{Pa·s})$ $1 \text{ fm}/(\text{Pa·s}) = 8.752 \text{ cm}^3/(\text{m}^2 \cdot 24 \text{ h·atm})$

4 Preparation of test specimens

4.1 General

Specimens shall be prepared by injection moulding or by cutting from film. The method to be used is indicated in the list of properties (see <u>Table 2</u>) for each test specimen.

It is essential that specimens are always prepared using the same processing conditions. The material shall be kept in moisture-proof containers until it is required for use.

The moisture content of filled or reinforced materials shall be expressed as a percentage of the total mass of the compound.

4.2 Treatment of the material before moulding

Before processing, the moisture content of the sample shall not exceed 0,3 % (mass fraction). If the moisture level exceeds this limit, the material shall be dried for 24 h \pm 2 h at a temperature of 105 °C \pm 5 °C *in vacuo* or in a stream of dried nitrogen.

To ensure that the moisture content remains low, it is recommended that the material in the feed hopper of the injection-moulding machine be blanketed with any suitable gas (dried air, nitrogen or argon, for example). Better results may be obtained using a dehumidifier hopper dryer.

4.3 Injection moulding

Injection-moulded specimens shall be prepared in accordance with ISO 294-2, using the conditions specified in <u>Table 1</u>.

1	Table 1 — Conditions for injection moulding of test specimens										

Ma	iterial	Melt	Mould	Avionaga	Average Hold Maximum Cooling To		Total		
Filler con- tent	Ethylene content	tempera- ture	tempera- ture	injection velocity	Hold pressure	pressure time	injection pressure	Cooling time	cycle time
mol%	mol%	°C	°C	mm/s	MPa	S	MPa	S	S
0	>15 but ≤30	220	50	150	80	15	80	45	50
0	>30 but ≤45	200	50	150	80	15	80	45	50
0	>45 but ≤60	180	50	150	80	15	80	45	50
≤30	>15 but ≤60	230	60	150	80	12	80	35	40
>30	>15 but ≤60	250	80	150	80	12	100	35	40

4.4 Preparation of film specimens

Film specimens shall be cut from cast, blown or any other type of film. The recommended thickness is $20~\mu m \pm 10~\mu m$. The thickness of the specimens shall be determined from the average thickness of the sample, measured mechanically. The variation in thickness shall not exceed $2~\mu m$. The specimens shall have smooth surfaces and shall be free from marks and other visible defects (streaks, pinholes, fisheyes, etc.).

5 Conditioning of test specimens Preview

5.1 General

Properties shall be determined on specimens in the dry-as-moulded (DAM) state or on specimens in the moist state or on specimens in either state. The state of the specimens shall be reported.

5.2 Dry-as-moulded (DAM) state

Specimens shall be moulded from dry granules (see 4.2 and 4.3.). Specimens are considered to be in the dry-as-moulded state when they have been placed immediately after moulding in a moisture-proof container and stored at 23 °C ± 2 °C for at least 24 h. The moisture content of DAM specimens may not exceed 0,3 % (mass fraction). Drying of specimens with moisture contents above this limit in order to reach this moisture content is not allowed.

To keep the absorbed moisture at a low level, DAM specimens shall be tested in as short a time as possible (maximum 30 min) after removal from the moisture-proof container.

5.3 Moist state

Test specimens are considered to be in the moist state when they have been conditioned at 23 °C \pm 2 °C and 50 % \pm 5 % relative humidity until equilibrium has been reached (see ISO 291:2008, Annex A).

5.4 Film conditioning

Non-oriented film shall be heat-treated under the following conditions:

- temperature: 20 °C ± 2 °C below the melting temperature;
- time: 10 min.

Film shall be held in a frame by clamps during the heat treatment so that the dimensions do not change.

6 Determination of properties

In the determination of properties and the presentation of data, the standards, supplementary instructions and notes given in ISO 10350-1 shall be applied. All tests shall be carried out in the standard laboratory atmosphere of 23 °C \pm 2 °C and 50 % \pm 5 % relative humidity unless specifically stated otherwise in Tables 2 or 3.

<u>Table 2</u> is taken from ISO 10350-1 and the properties listed are those appropriate to ethylene/vinyl alcohol copolymer moulding and extrusion materials. These properties are those considered useful for comparisons of data generated for different thermoplastics.

<u>Table 3</u> contains those properties, not found specifically in <u>Table 2</u>, which are in wide use or of particular significance in the practical characterization of ethylene/vinyl alcohol copolymer moulding and extrusion materials. Comparisons of different materials using these properties may well be restricted to those thermoplastics in the same generic families.

Table 2 — General properties and test conditions (selected from ISO 10350-1)

Property	Unit	Standard	Specimen type (dimensions in mm)	Specimen preparation ^a	Test conditions and supplementary instructions	
Rheological properties					, , ,	
Melt mass-flow rate	g/10 min	ISO 1133-1	Moulding compound	_	Temperature 210 °C, load 2,16 kg (see also conditions given in ISO 21309-1)	
Mechanical properties	(http	c.//ctan	lards ita	h ai)		
Tensile modulus	MPa	S.//Stallt	adi usitt	11.41)	Test speed 1 mm/min	
Yield stress	МРа	ocumon	t Preview		Test speed 50 mm/min	
Yield strain	%	ocumen	r I levie		Test speed 50 mm/min	
Strain at break	%	ISO 527-1		.,	Test speed 50 mm/min	
Stress at 50 % strain	МРа	ISO 527-2 ISO 2130	ISO 20753, type A1	М	Test speed 50 mm/min	
Stress at break	MPa	lards/iso/3ad88hac	-df3b-4783-9e9b		Test speed 50 mm/min	
Nominal strain at break	%	aras, 150, 5 aro 0 o ar	4150 1705 7070		Test speed 5 mm/min. Only to be quoted if stress at 50 % strain cannot be measured.	
Tensile creep modulus	МРа	ISO 899-1	See ISO 20753	M	At 1 h At 1 000 h } Strain ≤ 0,5 9	
Flexural modulus	MD-	MPa	a ISO 178	80 × 10 × 4	М	Test speed 2 mm/min
Flexural strength	MI a	130 170	00 ^ 10 ^ 4	IVI	Test speed 2 mm/ mm	
Charpy impact strength	kJ/m ²		80 × 10 × 4	M	Method 1e (edgewise impact)	
Charpy notched impact strength	kJ/m²	ISO 179-1	80 × 10 × 4 V-notch, r = 0,25	М	Method 1eA (edgewise impact)	
Thermal properties						
Melting temperature	°C	ISO 3146:2000	Moulding compound	_	Method C (DSC or DTA). Use 10 °C/min	
Temperature of deflection under load	°C	ISO 75-1:2013 ISO 75-2:2013	110 × 10 × 4 edgewise or 80 × 10 × 4 flatwise	М	Method A (1,8 MPa)	
Vicat softening temperature	°C	ISO 306	10 × 10 × 4	M	Heating rate 50 °C/h, load 50 N	
Electrical properties						
Relative permittivity	_	IEC (0250	>00 v > 00 ·· 1	М	Frequency 100 Hz and 1 MHz (com	
Dissipation factor	_	IEC 60250	≥80 × ≥80 × 1	IVI	pensate for electrode edge effect)	
Volume resistivity	Ω·m	IEC 62631-3-1	. (0 . (0 . 2	М	W. I. FOOM	
Surface resistivity	Ω	IEC 62631-3-2	≥60 × ≥60 × 2		Voltage 500 V	

 Table 2 (continued)

Property	Unit	Standard	Specimen type (dimensions in mm)	Specimen preparation ^a	Test conditions and supplementary instructions		
Electric strength	kV/mm	IEC 60243-1	≥80 × ≥80 × 1 ≥80 × ≥80 × 3	М	Use 25 mm/75 mm coaxial-cyl- inder electrode configuration. Immerse in IEC 60296 transformer oil. Use short time (rapid rise) test.		
Comparative tracking index	_	IEC 60112	≥15 × ≥15 × 4	М	Use solution A		
Other properties							
Density	kg/m ³	ISO 1183-1:2012	_	М	Use method B (pyknometer method). Use toluene/carbon tetrachloride as immersion liquids.		
Flammability	S	IEC 60695-11-10	125 × 10 × 4	M	Method B — after flame time of horizontal specimens		
a M = Injection moulding.							

 $\begin{tabular}{l} \textbf{Table 3-Additional properties and test conditions of particular utility to EVOH moulding and extrusion materials} \\ \end{tabular}$

Property	Unit	Standard	Specimen type (dimensions in mm)	Specimen preparation ^a	Test conditions and supplemen- tary instructions
Mechanical properti	es	ileh Si	tandard	S	
Yield stress	MPa	ISO 527-1, ISO 527-2	ISO 20753,	iteh _M ai)	Test speed 5 mm/ min (for materials with fillers or rein-
Yield strain	%		type A1		forcements)
Izod impact strength	kJ/m ²	ISO 180:2000	80 × 10 × 4	M	Method 1A
Rockwell hardness	_	ISO 2039-2	≥20 × ≥20 × ≥6	M	
Other properties		<u>ISO 21</u>	309-2:2019		
https://standards.ite Ash	eh.ai/catalog/stand %	ISO 3451-488 1:2008	Moulding com- pound	e9b-86ea3b03 M	Method A: 600 °C ± 25 °C
Volatile matter	%	Annex A		M	
Ethylene content	%	Annex B		M	
Oxygen-gas transmis- sion rate	cm ³ / (m ² ·24·h × atm)	Annex C		F	
a M = Injection mould	ing; F = Film.				