INTERNATIONAL STANDARD

ISO 14663-2

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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

Reference number
ISO 14663-2:1999(E)

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.

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.

International Standard ISO 14663-2 was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 9, *Thermoplastic materials*.

ISO 14663 consists of the following parts, under the general title *Plastics — Ethylene/vinyl alcohol (EVOH) copolymer moulding and extrusion materials*:

— *Part 1: Designation system and basis for specifications*

— *Part 2: Preparation of test specimens and determination of properties*

Annexes A, B and C form an integral part of this part of ISO 14663.

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

Part 2:
Preparation of test specimens and determination of properties

1 Scope

This part of ISO 14663 specifies the methods of preparation of test specimens and the test methods to be used in determining the properties of EVOH moulding and extrusion materials. Requirements for handling test material and for conditioning both the test material before moulding and the specimens before testing are given here.

Procedures and conditions are described for the preparation of test specimens, and procedures for measuring properties of the materials from which these specimens are made are given. Properties and test methods which are suitable and necessary to characterize EVOH moulding and extrusion materials are listed.

The properties have been selected from the general test methods in ISO 10350. Other test methods in wide use for or of particular significance to these moulding and extrusion materials are also included in this part of ISO 14663, as is the designatory property specified in part 1: melt mass-flow rate.

In order to obtain reproducible and comparable test results, it is necessary 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 standards contain provisions which, through reference in this text, constitute provisions of this part of ISO 14663. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this part of ISO 14663 are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 75-1:1993, *Plastics — Determination of temperature of deflection under load — Part 1: General test method.*

ISO 75-2:1993, *Plastics — Determination of temperature of deflection under load — Part 2: Plastics and ebonite.*

ISO 178:1993, *Plastics — Determination of flexural properties.*

ISO 179-1:—[1], *Plastics — Determination of Charpy impact properties — Part 1: Non-instrumented impact test.*

ISO 180:—[2], *Plastics — Determination of Izod impact strength.*

ISO 291:1997, *Plastics — Standard atmospheres for conditioning and testing.*

ISO 294-2:1996, *Plastics — Injection moulding of test specimens of thermoplastic materials — Part 2: Small tensile bars.*

ISO 306:1994, *Plastics — Thermoplastic materials — Determination of Vicat softening temperature (VST).*

ISO 527-1:1993, *Plastics — Determination of tensile properties — Part 1: General principles.*

ISO 527-2:1993, *Plastics — Determination of tensile properties — Part 2: Test conditions for moulding and extrusion plastics.*

ISO 899-1:1993, *Plastics — Determination of creep behaviour — Part 1: Tensile creep.*

ISO 1133:1997, *Plastics — Determination of the melt mass-flow rate (MFR) and the melt volume-flow rate (MVR) of thermoplastics.*

ISO 1183:1987, *Plastics — Methods for determining the density and relative density of non-cellular plastics.*

ISO 2039-2:1987, *Plastics — Determination of hardness — Part 2: Rockwell hardness.*

ISO 2818:1994, *Plastics — Preparation of test specimens by machining.*

ISO 3146:1985, *Plastics — Determination of melting behaviour (melting temperature or melting range) of semi-crystalline polymers.*

ISO 3167:1993, *Plastics — Multipurpose test specimens.*

ISO 3451-1:1997, *Plastics — Determination of ash — Part 1: General methods.*

ISO 10350:1993, *Plastics — Acquisition and presentation of comparable single-point data.*

ISO 14663-1:1999, *Plastics — Ethylene/vinyl alcohol (EVOH) copolymer moulding and extrusion materials — Part 1: Designation system and basis for specifications.*

IEC 60093:1980, *Methods of test for volume resistivity and surface resistivity of solid electrical insulating materials.*

IEC 60112:1979, *Method for determining the comparative and the proof tracking indices of solid insulating materials under moist conditions.*

IEC 60243-1:1998, *Electrical strength of solid insulating materials — Test methods — Part 1: Tests at power frequencies.*

IEC 60250:1969, *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 60296:1982, *Specification for unused mineral insulating oils for transformers and switchgear.*

IEC 60695-11-10:1999, *Fire hazard testing — Part 11-10: Test flames — 50 W horizontal and vertical flame test methods.*

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[1] To be published. (Revision of ISO 179:1993)

[2] To be published. (Revision of ISO 180:1993)

3 Preparation of test specimens

Specimens shall be prepared by injection moulding or by cutting from film. The method to be used is indicated in the list of properties (Table 2) for each test specimen. The code letter M is used to show that injection moulding is to be used; F is the code letter for film.

It is essential that specimens are always prepared using the same processing conditions. The standard conditions are given below.

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.

3.1 Treatment of the material before moulding

Before processing, the moisture content of the sample shall not exceed 0,3 % (m/m). If the moisture level exceeds this limit, the material shall be dried for 24 h ± 2 h at a temperature of 105 °C ± 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.

3.2 Injection moulding

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

Table 1 — Conditions for injection moulding of test specimens

Material		**Melt temperature**	**Mould temperature**	**Average injection velocity**	**Hold pressure**	**Hold-pressure time**	**Maximum injection pressure**	**Cooling time**	**Total cycle time**
Filler content % (m/m)	Ethylene content % (m/m)	°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

3.3 Preparation of film specimens

Film specimens shall be cut from cast, blown or any other type of film. The recommended thickness is 20 µm ± 10 µm. 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 µm. The specimens shall have smooth surfaces and shall be free from marks and other visible defects (streaks, pinholes, fish-eyes, etc.).

4 Conditioning of test specimens

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

4.1 Dry-as-moulded (DAM) state

Specimens shall be moulded from dry granules (see 3.1 and 3.2). Specimens are considered to be in the dry-as-moulded (DAM) 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 % (m/m). 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.

4.2 Moist state

Test specimens are considered to be in the moist state when they have been conditioned at 23 °C ± 2 °C and (50 ± 5) % relative humidity until equilibrium has been reached (see annex A to ISO 291:1997).

4.3 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.

5 Determination of properties

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

Table 2 is taken from ISO 10350:1993, 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.

Table 3 contains those properties, not found specifically in Table 2, 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)

Property	**Unit**	**Standard**	**Specimen type** (dimensions in mm)	**Specimen preparation**1)	**Test conditions and supplementary instructions**
Rheological properties					
Melt mass-flow rate	g/10 min	ISO 1133	Moulding compound	—	Temperature 210 °C, load 2,16 kg (see also conditions given in ISO 14663-1)
Mechanical properties					
Tensile modulus	MPa	ISO 527-1 ISO 527-2	ISO 3167, type A	M	Test speed 1 mm/min
Yield stress	MPa				Test speed 50 mm/min
Yield strain	%				Test speed 50 mm/min
Strain at break	%				Test speed 50 mm/min
Stress at 50 % strain	MPa				Test speed 50 mm/min
Stress at break	MPa				Test speed 50 mm/min
Nominal strain at break	%				Test speed 5 mm/min. Only to be quoted if stress at 50 % strain cannot be measured.
Tensile creep modulus	MPa	ISO 899-1	See ISO 3167	M	At 1 h At 1 000 h Strain ⩽ 0,5 %
Flexural modulus	MPa	ISO 178	80 × 10 × 4	M	Test speed 2 mm/min
Flexural strength					
Charpy impact strength	kJ/m^2	ISO 179-1	80 × 10 × 4	M	Method 1e (edgewise impact)
Charpy notched impact strength	kJ/m^2		80 × 10 × 4 V-notch, *r* = 0,25	M	Method 1eA (edgewise impact)
Thermal properties					
Melting temperature	°C	ISO 3146	Moulding compound	—	Method C (DSC or DTA). Use 10 °C/min.
Temperature of deflection under load	°C	ISO 75-1 ISO 75-2	110 × 10 × 4 edgewise or 80 × 10 × 4 flatwise	M	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 60250	⩾ 80 × ⩾ 80 × 1	M	Frequency 100 Hz and 1 MHz (compensate for electrode edge effect)
Dissipation factor	—				
Volume resistivity	Ω·m	IEC 60093	⩾ 80 × ⩾ 80 × 1	M	Voltage 500 V
Surface resistivity	Ω				
Electric strength	kV/mm	IEC 60243-1	⩾ 80 × ⩾ 80 × 1 ⩾ 80 × ⩾ 80 × 3	M	Use 25 mm/75 mm coaxial-cylinder electrode configuration. Immerse in IEC 60296 transformer oil. Use short time (rapid rise) test.
Comparative tracking index	—	IEC 60112	⩾ 15 × ⩾ 15 × 4	M	Use solution A
Other properties					
Density	kg/m^3	ISO 1183	—	M	Use method B (pyknometer method). Use toluene/carbon tetrachloride as immersion liquids.
Flammability	s	IEC 60695-11-10	125 × 10 × 4	M	Method B — afterflame time of horizontal specimens

1) M = Injection moulding

Table 3 — Additional properties and test conditions of particular utility to EVOH moulding and extrusion materials

Property	Unit	Standard	Specimen type (dimensions in mm)	Specimen preparation1)	Test conditions and supplementary instructions
Mechanical properties					
Yield stress	MPa	ISO 527-1, ISO 527-2	ISO 3167, type A	M	Test speed 5 mm/min (for materials with fillers or reinforcements)
Yield strain	%				
Izod impact strength	kJ/m^2	ISO 180	80 × 10 × 4	M	Method 1A
Rockwell hardness	—	ISO 2039-2	⩾ 20 × ⩾ 20 × ⩾ 6	M	
Other properties					
Ash	%	ISO 3451-1	Moulding compound	M	Method A: 600 °C ± 25 °C
Volatile matter	%	ISO 14663-2, annex A		M	
Ethylene content	%	ISO 14663-2, annex B		M	
Oxygen-gas transmission rate	cm^3/(m^2·24 h·atm)	ISO 14663-2, annex C		F	

1) M = Injection moulding
F = Film

Annex A
(normative)

Determination of volatile matter (including water)

A.1 Scope

This annex specifies a method of determining the volatile matter (including water) in ethylene/vinyl alcohol copolymers.

A.2 Principle

A test portion is heated at 120 °C ± 2 °C to constant mass in a weighing bottle.

A.3 Apparatus

A.3.1 Air-circulation oven, capable of maintaining the temperature at 120 °C ± 2 °C.

A.3.2 Weighing bottle, about 40 mm in diameter and 70 mm in height, made of glass, aluminum or, preferably, stainless steel, with lid.

A.3.3 Balance, accurate to 0,1 mg.

A.3.4 Desiccator, containing a suitable desiccant.

A.4 Procedure

Weigh the bottle (A.3.2) with its lid to the nearest 0,1 mg, after heating in the oven (A.3.1) at 120 °C ± 2 °C for 1 h and cooling to ambient temperature in the desiccator (A.3.4).

Place a mass m_0 (about 20 g) of resin in the bottle, replace the lid and weigh to the nearest 0,1 mg.

Place the assembly in the oven at 120 °C ± 2 °C, remove the lid (but leave it in the oven) and close the oven door.

After 24 h ± 0,5 h, remove the assembly from the oven, allow to cool in the desiccator and weigh to the nearest 0,1 mg. Keep the lid on during transfer and weighing. From this, calculate the mass m_1 of the residue.

Carry out two determinations.

For each determination, calculate the percentage of volatile matter from the formula given in clause A.5.

If the two percentages differ by less than 0,05 % in absolute value, use them to calculate the mean.

If not, carry out further determinations until two values satisfying this requirement are obtained.

However, if the two values obtained are each less than 0,05 % — no matter what the difference between them — new determinations are not necessary.

In certain special cases, it may be necessary to conduct the determination at a higher temperature of 150 °C ± 3 °C for 5 h ± 0,1 h. In reporting the results, give the reasons for choosing 150 °C.

A.5 Expression of results

For each of the determinations, calculate the percentage of volatile matter (including water) to two decimal places from the formula

$$\frac{m_0 - m_1}{m_0} \times 100$$

where

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

m_1 is the mass, in g, of the test portion after heating.

Calculate the mean, to two decimal places, of the two values obtained.

In the test report, give this mean as the percentage of volatile matter (including water).

NOTE For ordinary use, the expression of the result to one decimal place is generally sufficient.

A.6 Precision

Cooperative tests between different laboratories have shown a reproducibility, for the values determined, of 0,1 % (absolute).

A.7 Test report

The test report shall include the following particulars:

a) a reference to annex A of this part of ISO 14663;

b) all details necessary for complete identification of the sample;

c) if appropriate, the reason for carrying out the test at 150 °C;

d) the result, expressed in accordance with clause A.5;

e) any circumstances which may have affected the result;

f) the date of the test.

Annex B
(normative)

Determination of ethylene content

B.1 Scope

This annex specifies a method of determining the ethylene content of ethylene/vinyl alcohol copolymers.

NOTE This method cannot be applied to mixtures of EVOH with other polymers or fillers.

B.2 Principle

The hydroxyl groups of the resin are acetylated by reacting a solution of the resin in pyridine with acetic anhydride. The excess acetic anhydride and pyridine are washed out with water. The acetylated resin is dissolved in a methanol/water mixture and the acetate groups hydrolysed with sodium hydroxide. Excess sulfuric acid is added and the acid back-titrated with a standard volumetric solution of sodium hydroxide, using phenolphthalein as indicator.

B.3 Reagents

During the analysis, use only reagents of recognized analytical grade and only distilled water or water of equivalent purity.

B.3.1 Acetic anhydride.

B.3.2 Pyridine.

B.3.3 Acetone.

B.3.4 Methanol.

B.3.5 Sulfuric acid, standard volumetric solution, $c(H_2SO_4) = 0,25$ mol/l, or **hydrochloric acid,** standard volumetric solution, $c(HCl) = 0,5$ mol/l.

B.3.6 Sodium hydroxide, standard volumetric solution, $c(NaOH) = 0,2$ mol/l.

B.3.7 Sodium hydroxide, standard volumetric solution, $c(NaOH) = 0,5$ mol/l.

B.3.8 Phenolphthalein, indicator solution.

Dissolve 0,7 g of phenolphthalein in 100 ml of ethanol.

B.4 Apparatus

B.4.1 Conical flask, capacity 50 ml, with a ground-glass neck and ground-glass stopper.

B.4.2 Conical flask, capacity 100 ml, with a ground-glass neck and ground-glass stopper.

B.4.3 Conical flask, capacity 300 ml, with a ground-glass neck.

B.4.4 Beaker, capacity 2 litres.

B.4.5 Reflux condenser, length about 400 mm, with a ground-glass cone capable of being fitted on the 300 ml conical flask (B.4.3).

B.4.6 Measuring cylinder, capacity 10 ml, for the acetic anhydride (B.3.1).

B.4.7 Pipettes, capacity 15 ml, one for the 0,5 mol/l sodium hydroxide solution (B.3.7), the other for the sulfuric or hydrochloric acid (B.3.5).

B.4.8 Measuring cylinders, capacity 20 ml, one for the pyridine (B.3.2), the other for the acetone (B.3.3).

B.4.9 Measuring cylinder, capacity 50 ml, for the methanol (B.3.4).

B.4.10 Burette, capacity 25 ml, graduated in 0,1 ml steps, for the 0,2 mol/l sodium hydroxide solution (B.3.6).

B.4.11 Dropping bottle, for the phenolphthalein solution (B.3.8).

B.4.12 Magnetic stirrer, with a magnetic bar covered with a corrosion-resistant material (for example PTFE).

B.4.13 Glass stirring rod.

B.4.14 Oil bath, capable of being maintained at 105 °C ± 5 °C, or **other suitable means** of keeping the contents of the flask at this temperature.

B.4.15 Water bath, capable of being maintained at 60 °C ± 2 °C, or **other suitable means** of keeping the contents of the flask at this temperature.

B.4.16 Vacuum desiccator, capable of being maintained at 70 °C ± 10 °C and below 5,3 kPa.

B.4.17 Balance, accurate to 0,1 mg.

B.5 Procedure

B.5.1 Acetylation

B.5.1.1 Weigh, to the nearest 0,01 g, 2 g ± 0,1 g of the resin into the 100 ml conical flask (B.4.2). Add 6 ml of acetic anhydride (B.3.1), 12 ml of pyridine (B.3.2) and the stirrer bar (see B.4.12). Stopper the flask.

B.5.1.2 Heat the flask on the oil bath (B.4.14) at 105 °C ± 5 °C, using the magnetic stirrer (B.4.12) and removing the stopper at regular intervals. Keep heating for 2 h after the test portion has dissolved.

B.5.1.3 Remove the flask from the oil bath and allow to cool to ambient temperature.

B.5.1.4 Pour the contents (acetylated resin) slowly into 1 litre of distilled water in the 2 litre beaker (B.4.4), stirring with the glass rod (B.4.13). Decant off the acetylated resin.

B.5.1.5 Add 1 litre of water, leave for 30 min and decant off the acetylated resin. Repeat this washing process three times.

B.5.1.6 Put the acetylated resin in the 50 ml conical flask (B.4.1). Add 20 ml of acetone (B.3.3) and dissolve the acetylated resin by shaking the flask and, if necessary, by heating gently.

B.5.1.7 Pour the solution slowly into 1 litre of distilled water in the 2 litre beaker, stirring with the glass rod. Decant off the acetylated resin.

B.5.1.8 Repeat the operations in B.5.1.6 and B.5.1.7 twice more.

B.5.1.9 Process the acetylated resin (B.5.1.8) into a thin film and dry it at 70 °C ± 10 °C for 16 h in the vacuum desiccator (B.4.16).

B.5.2 Determination

B.5.2.1 Weigh, to the nearest 0,1 mg, 0,2 g of the dried acetylated resin into the 300 ml conical flask (B.4.3). Add 15 ml of 0,5 mol/l sodium hydroxide solution (B.3.7) by means of a pipette (B.4.7), and 30 ml of methanol (B.3.4).

B.5.2.2 Heat the flask fitted with the condenser (B.4.5) for 4 h on the water bath (B.4.15) at 60 °C ± 2 °C.

B.5.2.3 When hydrolysis is complete, remove the flask with the condenser from the water bath and allow to cool to ambient temperature. Rinse the inside wall of the condenser and the flask with 30 ml of methanol poured into the top of the condenser. Remove the condenser from the flask.

B.5.2.4 Add 15 ml of sulfuric or hydrochloric acid (B.3.5) by means of a pipette (B.4.7).

B.5.2.5 Add a few drops of phenolphthalein solution (B.3.8) and shake gently.

B.5.2.6 Titrate the excess acid with 0,2 mol/l sodium hydroxide solution (B.3.6).

B.5.3 Blank test

Carry out a blank test under the same conditions as for the determination, but without the dried acetylated resin prepared in B.5.1.9.

B.5.4 Number of determinations

Carry out two determinations. If the results, calculated from the formula given in clause B.6, differ by more than 1 % (*m*/*m*), repeat the determinations. Report the result as the arithmetic mean of two valid determinations.

B.6 Expression of results

For each of the determinations, calculate the ethylene content, expressed as a percentage by mass, as follows:

$$\text{Vinyl alcohol content} = (V_1 - V_2) \times c \times \frac{1{,}722}{m}$$

$$\text{Ethylene content} = 100 - (\text{vinyl alcohol content})$$

where

V_1 is the volume, in millilitres, of standard volumetric sodium hydroxide solution (B.3.6) used in the determination;

V_2 is the volume, in millilitres, of standard volumetric sodium hydroxide solution (B.3.6) used in the blank test;

c is the actual concentration, in moles per litre, of the sodium hydroxide solution (B.3.6);

m is the mass, in grams, of the test portion;

1,722 is the mass, in grams, of vinyl acetate corresponding to 1,000 ml of sodium hydroxide solution, $c(\text{NaOH}) = 0{,}200$ mol/l.

B.7 Test report

The test report shall include the following particulars:

a) a reference to annex B of this part of ISO 14663;

b) all details necessary for complete identification of the sample;

c) the result, expressed in accordance with clause B.6;

d) any circumstances which may have affected the result;

e) the date of the test.

Annex C
(normative)

Determination of steady-state rate of transmission of oxygen gas through ethylene/vinyl alcohol copolymer in the form of film using a coulometric sensor

C.1 Scope

This annex specifies a method of determining the steady-state rate of transmission of oxygen gas through ethylene/vinyl alcohol copolymer in the form of film using a coulometric sensor. It gives the oxygen gas transmission rate (O_2GTR).

C.2 Definition

For the purposes of this annex, the following definition applies:

C.2.1
oxygen gas transmission rate
O_2GTR
the volume of oxygen gas passing through unit area of the parallel surfaces of a film per unit time under the conditions of test

NOTE 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 [$cm^3/(m^2 \cdot 24\,h \cdot atm)$].

The SI unit for O_2GTR is the femtometre per pascal second [$fm/(Pa \cdot s)$]:

1 fm (femtometre) = 10^{-15} m

1 atm = 101,3 kPa

1 $cm^3/(m^2 \cdot 24\,h \cdot atm)$ = 0,114 3 $fm/(Pa \cdot s)$

1 $fm/(Pa \cdot s)$ = 8,752 $cm^3/(m^2 \cdot 24\,h \cdot atm)$

C.3 Principle

A film specimen is mounted in a diffusion cell composed of two chambers. One chamber is slowly purged at a defined nitrogen (carrier gas) flow rate, the other chamber being supplied with oxygen. Oxygen gas which permeates through the film into the carrier gas is determined by a coulometric sensor which generates an electrical current proportional to the amount of oxygen flowing into the sensor per unit time.

C.4 Materials

C.4.1 Carrier gas, consisting of a nitrogen/hydrogen mixture in which the percentage of hydrogen is between 0,5 % (*V*/*V*) and 3,0 % (*V*/*V*). The carrier gas shall be dry and shall not contain more than 100 ppm of oxygen. A commercially available mixture known as "forming gas" is suitable.

NOTE The presence of certain contaminants in the carrier gas stream may give rise to an erroneous sensor signal. Such interfering substances include free chlorine and other strong oxidizing agents. In addition, exposure to carbon dioxide should be minimized to avoid deteriorIation of the sensor through reaction with the potassium hydroxide electrolyte.

C.4.2 Oxygen test gas, dry, purity not less than 99,9 %.

C.4.3 Sealing grease: a high-viscosity silicone stopcock grease or high-vacuum grease, for sealing the specimen film in the diffusion cell.

C.5 Apparatus

An oxygen gas transmission apparatus is shown in Figure C.1, including the following elements:

C.5.1 Diffusion cell, designed to allow oxygen gas to permeate through the specimen.

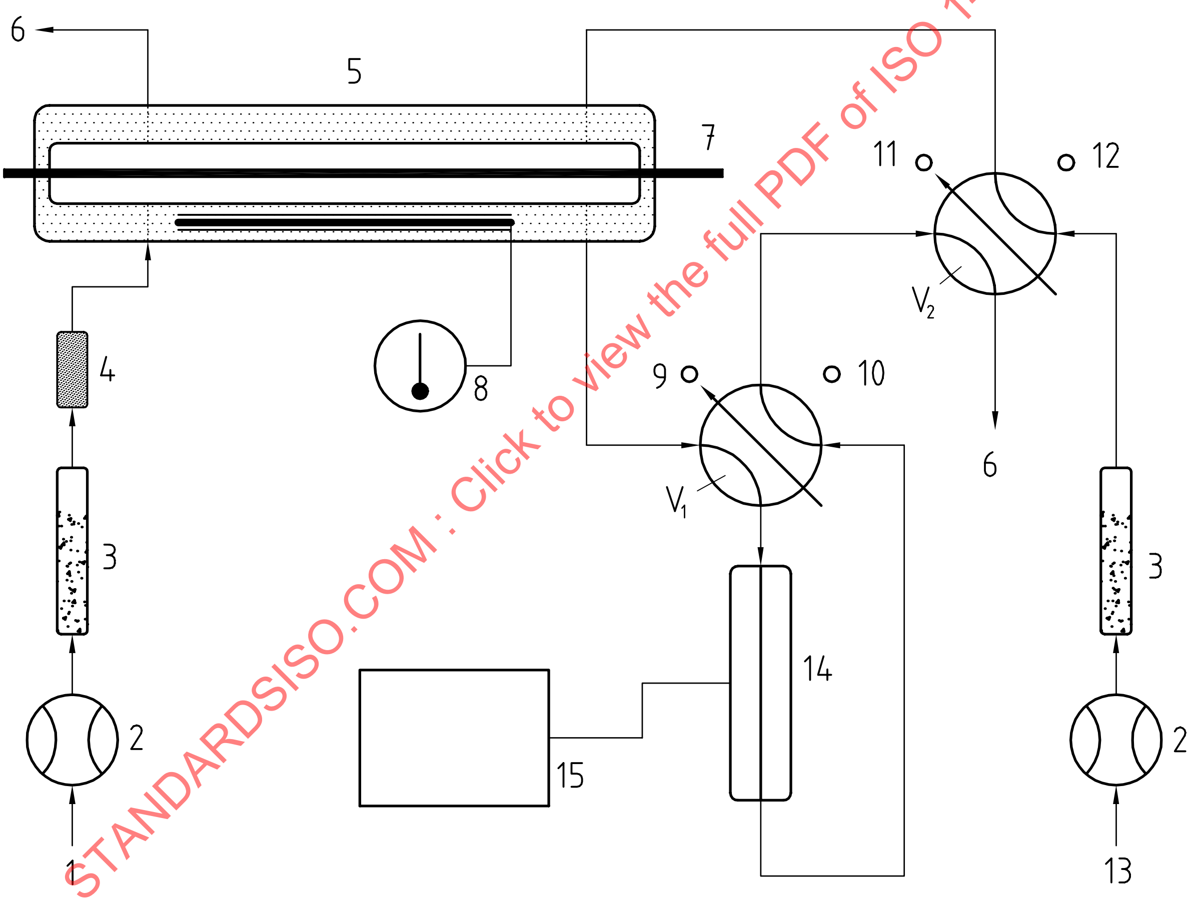
C.5.2 Coulometric sensor, to monitor the oxygen permeation rate, producing an electric current which is converted into a voltage.

C.5.3 Catalyst bed, containing a platinum or palladium catalyst on alumina, to remove essentially all the oxygen from the carrier gas.

C.5.4 Flowmeters.

C.5.5 Humidistats.

NOTE A suitable apparatus is available commercially.



Key

1 Nitrogen
2 Flowmeter
3 Humidistat
4 Catalyst bed
5 Diffusion cell
6 Exhaust
7 Specimen
8 Temperature control
9 "Insert sensor" position
10 "Sensor bypass" position
11 "Oxygen purge" position
12 "Carrier purge" position
13 Oxygen
14 Coulometric sensor
15 Recorder

Figure C.1 — Example of an oxygen transmission rate apparatus