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Plastics — Ethylene/vinyl acetate copolymer (EVAC) thermoplastics — Determination of vinyl acetate content

Plastiques — Copolymères éthylène/acétate de vinyle (EVAC) thermoplastiques — Dosage de l'acétate de vinyle

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Foreword

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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).

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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*, in collaboration with the European Committee for Standardization (CEN) Technical Committee CEN/TC 249, *Plastics*, in accordance with the Agreement on technical cooperation between ISO and CEN (Vienna Agreement).

This third edition cancels and replaces the second edition (ISO 8985:1998), which has been technically revised.

The main changes compared to the previous edition are as follows:

- the normative references have been updated to the latest version;
- the mandatory terms and definitions clause has been added (see Clause 3);
- a “thermogravimetry test method” has been added;
- infrared spectrometer has been modified to be Fourier infrared spectrometer;
- the example of infrared spectrum has been modified from transmission to absorbance;
- the example of calibration curve has been modified.

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.

This corrected version of ISO 8985:2022 incorporates the following corrections:

- the missing content has been reinstated in subclauses 4.3.3.2.5, 4.3.5, 5.2.5.2 and 5.4.5.3;
- the values of Formula (6) has been corrected;
- the subtitles of Figure 2 have been corrected;
- the unit in Figure 5 has been corrected;

- the legend for Formula (11) has been corrected.

Plastics — Ethylene/vinyl acetate copolymer (EVAC) thermoplastics — Determination of vinyl acetate content

SAFETY PRECAUTIONS — Persons using this document should be familiar with normal laboratory practice, if applicable. This document does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user to establish appropriate safety and health practices.

1 Scope

This document specifies two categories of method for the determination of the vinyl acetate (VAC) content of ethylene/vinyl acetate (EVAC) copolymer, for use in the designation of such copolymers according to ISO 21301-1. One category is referred to as "reference methods", the other as "test methods".

The "reference methods" are used to calibrate the method used for the determination of the vinyl acetate content of ethylene/vinyl acetate copolymers.

The "test methods" are other methods which can be used for the determination if they are calibrated using one of the reference methods described in Clause 4, provided they show a certain permissible repeatability.

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 472, *Plastics — Vocabulary*

ISO 4799, *Laboratory glassware — Condensers*

ISO 11358-1:2014, *Plastics — Thermogravimetry (TG) of polymers — Part 1: General principles*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 472 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 https://www.electropedia.org/

4 Reference methods

4.1 Reference method 1: Hydrolysis and back titration

4.1.1 Principle

A test portion is dissolved in xylene and the acetate groups are hydrolysed with alcoholic potassium hydroxide solution. An excess of sulfuric or hydrochloric acid is added. The acid is back titrated with a standard sodium hydroxide solution in the presence of phenolphthalein as indicator.

4.1.2 Reagents

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

4.1.2.1 Xylene.

4.1.2.2 Sulfuric acid, approximately 5 g/l solution, or hydrochloric acid, approximately 3,7 g/l solution.

4.1.2.3 Potassium hydroxide, approximately 5,6 g/l ethanol solution.

Dissolve 5,6 g of solid potassium hydroxide (KOH) in 500 ml of ethanol, make up to 1 000 ml, leave to settle until the next day and decant the clear part of the solution.

4.1.2.4 Sodium hydroxide, standard solution, c(NaOH) = 0,1 mol/l.

4.1.2.5 Phenolphthalein, indicator solution.

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

4.1.3 Apparatus

Standard laboratory apparatus, plus the following:

4.1.3.1 Burette, 50 ml capacity, for the sodium hydroxide solution (4.1.2.4).

4.1.3.2 Pipette, 30 ml capacity, for the acid solution (4.1.2.2).

4.1.3.3 Pipette, 25 ml capacity, for the potassium hydroxide solution (4.1.2.3).

4.1.3.4 Test tube, 50 ml capacity, for the xylene (4.1.2.1).

4.1.3.5 Flask, up to 300 ml capacity, with stopper.

4.1.3.6 Dropping bottle, for the phenolphthalein indicator solution (4.1.2.5).

4.1.3.7 Reflux condenser, at least 500 mm long, in accordance with ISO 4799.

4.1.3.8 Heating equipment, sand bath, oil bath or heating jacket, adjustable to 200 °C.

4.1.3.9 Analytical balance, with an accuracy of 0,1 mg.

4.1.4 Procedure

4.1.4.1 Determination

4.1.4.1.1 Weigh a quantity of dry polymer as shown in Table 1 into the flask (4.1.3.5) to the nearest 0,1 mg. The mass of each sample particle shall be less than approximately 0,05 g.

Table 1 — Guide to the mass of the sample to be used

Assumed vinyl acetate content $w(\text{VAC})$ %	Approximate mass of test portion g
$w(\text{VAC}) \leq 10$	1
$10 < w(\text{VAC}) \leq 20$	0,5
$20 < w(\text{VAC}) \leq 40$	0,3
$40 < w(\text{VAC})$	0,2

When analysing an unknown sample, first carry out a preliminary test under conditions which are valid for a copolymer containing 20 % to 40 % VAC.

4.1.4.1.2 Add 50 ml of xylene (4.1.2.1) to the contents of the flask and 25 ml of potassium hydroxide (4.1.2.3), using the pipette (4.1.3.3). Heat the flask, topped with the reflux condenser (4.1.3.7), for 2 h using the heating apparatus. After hydrolysis, remove the flask from the heating apparatus and allow to cool to ambient temperature. Add 30 ml of sulfuric or hydrochloric acid (4.1.2.2), using the pipette (4.1.3.2), stopper the flask and shake vigorously. Add several drops of phenolphthalein solution (4.1.2.5) and titrate the excess acid with standard sodium hydroxide solution (4.1.2.4), shaking the flask during the titration.

4.1.4.2 Blank test

Carry out a blank test in parallel with the determination, following the same procedure and using the same reagents but omitting the test portion.

4.1.5 Expression of results

4.1.5.1 The vinyl acetate content $w(\text{VAC})$, expressed as a percentage by mass, is given by the Formula (1):

$$w(\text{VAC}) = \frac{0{,}08609 \times (V_1 - V_2) \times c_1}{m} \times 100 \qquad (1)$$

where

V_1	is the volume, in ml, of sodium hydroxide solution used for the determination;
V_2	is the volume, in ml, of sodium hydroxide solution used for the blank test;
c_1	is the actual concentration, expressed in mol/l, of the sodium hydroxide solution used for the titration;
m	is the mass, in g, of the test portion (see 4.1.4.1.1);
0,08609	is the molar mass of vinyl acetate, in kg/mol.

4.1.5.2 Carry out two determinations. If the results differ by more than 1 %, discard them and run the determinations again. Report the arithmetic mean of the two determinations.

4.1.6 Test report

The test report shall contain the following information:

a) a reference to this document (i.e. ISO 8985:2022) and the method used;

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

c) the result, expressed in accordance with 4.1.5.2.

4.2 Reference method 2: Saponification and potentiometric titration

4.2.1 Principle

A test portion is dissolved in a mixture of xylene and hexan-1-ol and the acetate groups are hydrolysed with alcoholic potassium hydroxide solution. Acetone is added to prevent copolymer precipitation. The excess alkali is titrated with standard hydrochloric acid using a potentiometric titrimeter.

4.2.2 Reagents

During analysis use only reagents of recognized analytical quality and distilled water or water of equivalent purity.

4.2.2.1 Xylene.

4.2.2.2 Hexan-1-ol.

4.2.2.3 Potassium hydroxide, approximately 28 g/l ethanolic solution.

4.2.2.4 Acetone.

4.2.2.5 Hydrochloric acid, standard solution, c(HCl) = 0,3 mol/l.

4.2.2.6 Lithium chloride, 40 g/l ethanolic solution.

4.2.3 Apparatus

Standard laboratory equipment, plus the following:

4.2.3.1 Potentiometric titrator, with a 10 ml capacity burette graduated every 0,02 ml, a millivoltmeter or other suitable type of voltmeter, a glass measurement electrode, a silver/silver chloride reference electrode and a connecting bridge and beaker filled with an ethanolic solution of lithium chloride (4.2.2.6). Other types of potentiometric titrator may be used.

4.2.3.2 Test tube, capacity 50 ml, for the xylene (4.2.2.1) and the acetone (4.2.2.4).

4.2.3.3 Burette, capacity 5 ml, for the potassium hydroxide solution (4.2.2.3).

4.2.3.4 Pipette, capacity 10 ml, for the hexan-1-ol (4.2.2.2).

4.2.3.5 Flask, capacity 100 ml.

4.2.3.6 Reflux condenser, at least 300 mm long, in accordance with ISO 4799.

4.2.3.7 Heating apparatus, sand bath, oil bath or heating jacket, adjustable to approximately 200 °C.

4.2.3.8 Analytical balance, with an accuracy of 0,1 mg.

4.2.3.9 Magnetic stirrer.

4.2.4 Procedure

4.2.4.1 Determination

4.2.4.1.1 Weigh a quantity of dry polymer as shown in Table 2 into the flask to the nearest 0,1 mg. The mass of each sample particle shall be less than approximately 0,05 g.

Table 2 — Guide to the mass of the sample to be used

Assumed vinyl acetate content w(VAC) %	Approximate mass of test portion g
w(VAC) ≤ 2	1
2 < w(VAC) ≤ 5	0,5
5 < w(VAC) ≤ 30	0,2
30 < w(VAC)	0,1

When analysing an unknown sample, first carry out a preliminary test under conditions which are valid for a copolymer containing 5 % to 30 % vinyl acetate.

4.2.4.1.2 Add 25 ml of xylene (4.2.2.1) to the contents of the flask and 10ml of hexan-1-ol (4.2.2.2) and 5ml of potassium hydroxide solution (4.2.2.3). Heat the flask, topped with the reflux condenser (4.2.3.6), for 30 min, using the heating apparatus (4.2.3.7) set at boiling temperature.

4.2.4.1.3 After 30 min, remove the flask from the heating apparatus and allow to cool for 5 min to 6 min., then introduce 35 ml of acetone (4.2.2.4) through the top of the condenser. Remove the condenser and place the flask (if conical) on the magnetic stirrer (4.2.3.9), otherwise transfer the solution to a beaker first.

4.2.4.1.4 Immerse the glass electrode (see 4.2.3.1) and one of the ends of the connecting bridge into the flask or beaker. Immerse the other end of the connecting bridge and the silver/silver chloride reference electrode (see 4.2.3.1) in the beaker filled with the ethanolic solution of lithium chloride (4.2.2.6).

4.2.4.1.5 Carry out the potentiometric titration immediately, adding standard hydrochloric acid (4.2.2.5) until the first drop in potential and stirring all the time. When close to the end point, add acid in 0,04 ml to 0,06 ml increments.

4.2.4.1.6 When the end point is reached, read off the voltage, in millivolts, on the titrator as well as the volume of hydrochloric acid added.

The end point of the titration is that point at which the greatest variation in potential occurs for a given increment of acid added. In the event of two such points occurring, take the first value as the end point. The end point may also be determined graphically.

4.2.4.2 Blank test

Carry out a blank test in parallel with the determination, following the same procedure and using the same reagents but omitting the test portion. Plot the titration curve. The mean value of the peak on the titration curve is taken as the end point.

4.2.5 Expression of results

4.2.5.1 The vinyl acetate content w(VAC), expressed as a percentage by mass, is given by the Formula (2):

$$w(\mathrm{VAC}) = \frac{0{,}08\ 609 \times (V_3 - V_4) \times c_2}{m} \times 100 \qquad (2)$$

where

V_3 is the volume, in ml, of standard hydrochloric acid used for the blank test;

V_4 is the volume, in ml, of standard hydrochloric acid solution used for the determination;

c_2 is the actual concentration, expressed in mol/l, of the standard hydrochloric acid solution used for the titration;

m is the mass, in g, of the test portion (see 4.2.4.1.2).

4.2.5.2 Carry out two determinations. If the results differ by more than 1 %, discard them and run the determination again. Report the arithmetic mean of the two determinations.

4.2.6 Test report

The test report shall contain the following information:

a) a reference to this document (i.e. ISO 8985:2022) and the method used;

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

c) the result, expressed in accordance with 4.2.5.2.

4.3 Reference method 3: Measurement of oxygen

4.3.1 Principle

The determination of the oxygen content is carried out using one of the traditional methods of elementary organic analysis. Therefore, the following three methods as shown Table 3 are applied.

4.3.2 Apparatus

Any apparatus (commercial or otherwise) may be used, provided it meets the following requirements.

Detection range: 0,2 %

Dispersion: 0,2 % absolute or 10 % relative if $O_2 < 1$ %