
Polimerni materiali - Termoplastični kopolimer etilena in vinil acetata (EVAC-kopolimer) - Ugotavljanje deleža vinil acetata (ISO/DIS 8985:2020)

Plastics - Ethylene/vinyl acetate copolymer (EVAC) thermoplastics - Determination of vinyl acetate content (ISO/DIS 8985:2020)

Kunststoffe - Ethylen-Vinylacetat-Copolymer (EVAC)-Thermoplasten - Bestimmung des Vinylacetatgehalts (ISO/DIS 8985:2020)

Plastiques - Copolymères éthylène/acétate de vinyle (EVAC) thermoplastiques - Dosage de l'acétate de vinyle (ISO/DIS 8985:2020)

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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).

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For an explanation on 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 the following URL: www.iso.org/iso/foreword.html.

The committee responsible for this document is Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 9, *Thermoplastic materials*.

This third edition cancels and it replaces the second edition (ISO 8985:1998(E)), which has been technically revised by adding a "thermogravimetry test method" and modifying the method of infrared spectrum.

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 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 international standard 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 in accordance with ISO 21301-1:2019. 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 21301-1, *Plastics — Ethylene-vinyl acetate (EVAC) moulding and extrusion materials — Part 1: Designation system and basis for specifications*

ISO 472, *Plastics — Vocabulary*

ISO 4799, *Laboratory glassware — Condensers*

ISO 11358-1, *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:

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

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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**, approx. 5g/l solution, or hydrochloric acid, approx. 3,7g/l solution.

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

Dissolve 5,6g of solid potassium hydroxide (KOH) in 500ml of ethanol, make up to 1000ml, leave to settle until the next day and decant the clear part of the solution.

4.1.2.4 **Sodium hydroxide**, standard solution, $c(\text{NaOH}) = 0,1 \text{ mol/l}$.

4.1.2.5 **Phenolphthalein**, indicator solution.

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

4.1.3 Apparatus

Standard laboratory apparatus, plus the following:

4.1.3.1 **Burette**, 50ml capacity, for the sodium hydroxide solution ([4.1.2.4](#)).

4.1.3.2 **Pipette**, 30ml capacity, for the acid solution ([4.1.2.2](#)).

4.1.3.3 **Pipette**, 25ml capacity, for the potassium hydroxide solution ([4.1.2.3](#)).

4.1.3.4 **Test tube**, 50ml capacity, for the xylene ([4.1.2.1](#)).

4.1.3.5 **Flask**, up to 300ml 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 500ml 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 0,1mg.

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 0,1mg. The mass of each sample particle shall be less than approx. 0,05g.

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

Assumed vinyl acetate content, w(VAC) %	Approximate mass of test portion g
w(VAC) ≤ 10	1
10 < w(VAC) ≤ 20	0,5
20 < w(VAC) ≤ 40	0,3
40 < w(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 50ml of xylene ([4.1.2.1](#)) to the contents of the flask and 25ml 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 hours using the heating apparatus. After hydrolysis, remove the flask from the heating apparatus and allow to cool to ambient temperature. Add 30mL 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.

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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(VAC), expressed as a percentage by mass, is given by the formula:

$$w(\text{VAC}) = \frac{8,609 \times (v_1 - v_2) \times c_1}{m} \quad (1)$$

where

v_1 is the volume, in millilitres, of sodium hydroxide solution used for the determination;

v_2 is the volume, in millilitres, of sodium hydroxide solution used for the blank test;

c_1 is the actual concentration, expressed in moles per litre, of the sodium hydroxide solution used for the titration;

m is the mass, in grams, of the test portion (see [4.1.4.1.1](#)).

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.

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4.1.6 Test report

The test report shall contain the following information:

- a) a reference to this International Standard 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, approx. 28g/l ethanolic solution.

4.2.2.4 Acetone.

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4.2.2.5 Hydrochloric acid, standard solution, $c(\text{HCl}) = 0,3\text{mol/l}$.

4.2.2.6 Lithium chloride, 40g/l ethanolic solution.

4.2.3 Apparatus

Standard laboratory equipment, plus the following:

4.2.3.1 Potentiometric titrator, with a 10ml capacity burette graduated every 0,02ml, 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 50ml, for the xylene ([4.2.2.1](#)) and the acetone ([4.2.2.4](#)).

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

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

4.2.3.5 Flask, capacity 100ml.

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

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

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

4.2.3.9 Magnetic stirrer.

4.2.4 Procedure

4.2.4.1 Determination

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

Table 2 — Guide to the mass of 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 of all carry out a preliminary test under conditions which are valid for a copolymer containing 5% to 30% vinyl acetate.

4.2.4.1.1 Add 25ml 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 ([4.2.2.7](#)) set at boiling temperature. After 30 min., remove the flask from the heating apparatus and allow to cool for 5-6 min., then introduce 35ml 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 ([4.2.2.9](#)), otherwise transfer the solution to a beaker first. 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](#)).

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 0,04ml to 0,06ml increments.

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.