



SLOVENSKI STANDARD
SIST EN ISO 8985:1999

01-maj-1999

Polimerni materiali – Kopolimer etilen vinil acetat (EVAC) plastomeri – Določevanje deleža vinilacetata (ISO 8985:1998)

Plastics - Ethylene/vinyl acetate copolymer (EVAC) thermoplastics - Determination of vinyl acetate content (ISO 8985:1998)

Kunststoffe - Ethylen-Vinylacetat-Copolymer (EVAC)-Formmassen - Bestimmung des Vinylacetatgehalts (ISO 8985:1998)

Plastiques - Copolymères éthylène/acétate de vinyle (EVAC) thermoplastiques - Dosage de l'acétate de vinyle (ISO 8985:1998)

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EUROPEAN STANDARD
NORME EUROPÉENNE
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EN ISO 8985

March 1998

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English version

Plastics - Ethylene/vinyl acetate copolymer (EVAC)
thermoplastics - Determination of vinyl acetate content (ISO
8985:1998)

Plastiques - Copolymères éthylène/acétate de vinyle
(EVAC) thermoplastiques - Dosage de l'acétate de vinyle
(ISO 8985:1998)

This European Standard was approved by CEN on 15 February 1998.

CEN members are bound to comply with the CEN/CENELEC Internal Regulations which stipulate the conditions for giving this European Standard the status of a national standard without any alteration. Up-to-date lists and bibliographical references concerning such national standards may be obtained on application to the Central Secretariat or to any CEN member.

This European Standard exists in three official versions (English, French, German). A version in any other language made by translation under the responsibility of a CEN member into its own language and notified to the Central Secretariat has the same status as the official versions.

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EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
EUROPÄISCHES KOMITEE FÜR NORMUNG

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Foreword

The text of the International Standard ISO 8985:1998 has been prepared by Technical Committee ISO/TC 61 "Plastics" in collaboration with Technical Committee CEN/TC 249 "Plastics", the secretariat of which is held by IBN.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by September 1998, and conflicting national standards shall be withdrawn at the latest by September 1998.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Czech Republic, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and the United Kingdom.

Endorsement notice

The text of the International Standard ISO 8985:1998 was approved by CEN as a European Standard without any modification.

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INTERNATIONAL STANDARD

**ISO
8985**

Second edition
1998-03-01

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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Reference number
ISO 8985:1998(E)

ISO 8985:1998(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 8985 was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 9, *Thermoplastic materials*.

This second edition cancels and replaces the first edition (ISO 8985:1989), which has been technically revised.

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

1 Scope

This International Standard specifies two categories of method for the determination of the vinyl acetate (VAC) content of ethylene/vinyl acetate (EVAC) copolymers, for use in the designation of such copolymers in accordance with ISO 4613-1:1997. One category is referred to as "reference methods", the other as "test methods".

Note – The abbreviation previously used for ethylene/vinyl acetate copolymer (E/VAC) has been replaced by the abbreviation EVAC (see ISO 1043-1:1997, *Plastics – Symbols and abbreviated terms – Part 1: Basic polymers and their special characteristics*).

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 3 provided they show a certain permissible repeatability.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard 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 4613-1:1993, *Plastics – Ethylene/vinyl acetate (E/VAC) moulding and extrusion materials – Part 1: Designation and specification*.

ISO 4799:1978, *Laboratory glassware – Condensers*.

3 Reference methods

3.1 Reference method 1: Hydrolysis and back titration

3.1.1 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.

3.1.2 Reagents

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

3.1.2.1 Xylene.

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

3.1.2.3 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.

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

Note – The nomenclature $c(\text{xxxx})$ is preferred to $[\text{xxxx}]$ and used throughout, e.g. $c(\text{NaOH}) = [\text{NaOH}]$, when referring to concentrations.

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3.1.2.5 Phenolphthalein, indicator solution.

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

3.1.3 Apparatus

Standard laboratory apparatus, plus the following:

3.1.3.1 Burette, 50ml capacity, for the sodium hydroxide solution (3.1.2.4).

3.1.3.2 Pipette, 30ml capacity, for the acid solution (3.1.2.2).

3.1.3.3 Pipette, 25ml capacity, for the potassium hydroxide solution (3.1.2.3).

3.1.3.4 Test tube, 50ml capacity, for the xylene (3.1.2.1).

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

3.1.3.6 Dropping bottle, for the phenolphthalein indicator solution (3.1.2.5).

3.1.3.7 Reflux condenser, at least 500ml long, in accordance with ISO 4799.

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

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

3.1.4 Procedure

3.1.4.1 Determination

3.1.4.1.1 Weigh a quantity of dry polymer as shown in table 1 into the flask (3.1.3.5) 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
$w(\text{VAC}) \leq 10$	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.

3.1.4.1.2 Add 50ml of xylene (3.1.2.1) to the contents of the flask and 25ml of potassium hydroxide (3.1.2.3), using the pipette (3.1.3.3). Heat the flask, topped with the reflux condenser (3.1.3.7), 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 (3.1.2.2), using the pipette (3.1.3.2), stopper the flask and shake vigorously. Add several drops of phenolphthalein solution (3.1.2.5) and titrate the excess acid with standard sodium hydroxide solution (3.1.2.4), shaking the flask during the titration.

3.1.4.2 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.

3.1.5 Expression of results

3.1.5.1 The vinyl acetate content w(VAC), expressed as a percentage by mass, is given by the formula:

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

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 3.1.4.1.1).

3.1.5.2 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.

3.1.6 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 3.1.5.2.

3.2 Reference method 2: Saponification and potentiometric titration

3.2.1 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.

3.2.2 Reagents

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

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3.2.2.1 Xylene.

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3.2.2.2 Hexan-1-ol. <https://standards.iteh.ai/catalog/standards/sist/3a1736df-55aa-49f5-8529-d74766c03603/sist-en-iso-8985-1999>

3.2.2.3 Potassium hydroxide, approx. 28g/l ethanolic solution.

3.2.2.4 Acetone.

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

3.2.2.6 Lithium chloride, 40g/l ethanolic solution.

3.2.3 Apparatus

Standard laboratory equipment, plus the following:

3.2.3.1 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 (3.2.2.6). Other types of potentiometric titrator may be used.

3.2.3.2 Test tube, capacity 50ml, for the xylene (3.2.2.1) and the acetone (3.2.2.4).

3.2.3.3 Burette, capacity 5ml, for the potassium hydroxide solution (3.2.2.3).

3.2.3.4 Pipette, capacity 10ml, for the hexan-1-ol (3.2.2.2).

3.2.3.5 Flask, capacity 100ml.

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

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

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

3.2.3.9 Magnetic stirrer.

3.2.4 Procedure

3.2.4.1 Determination

3.2.4.1.1 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 specimen g
$w(\text{VAC}) \leq 2$	1
$2 < w(\text{VAC}) \leq 5$	0,5
$5 < w(\text{VAC}) \leq 30$	0,2
$30 < w(\text{VAC})$	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.

3.2.4.1.2 Add 25ml of xylene (3.2.2.1) to the contents of the flask and 10ml of hexan-1-ol (3.2.2.2) and 5ml of potassium hydroxide solution (3.2.2.3). Heat the flask, topped with the reflux condenser (3.2.3.6), for 30 min., using the heating apparatus (3.2.2.7) 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 (3.2.2.4) through the top of the condenser. Remove the condenser and place the flask (if conical) on the magnetic stirrer (3.2.2.9), otherwise transfer the solution to a beaker first. Immerse the glass electrode (see 3.2.3.1) 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 3.2.3.1) in the beaker filled with the ethanolic solution of lithium chloride (3.2.2.6).

Carry out the potentiometric titration immediately, adding standard hydrochloric acid (3.2.2.5) 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.