
**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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[ISO 8985:1998](https://standards.iteh.ai/catalog/standards/sist/00f2e0bf-2d9a-4cbf-9421-1fb6f8bdda72/iso-8985-1998)

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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.

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Note – The abbreviation previously used for ethylene/vinyl acetate copolymer (E/VAC) has been replaced by the abbreviation EVAC (see ISO 1043-1:1997, *Plastics – Symbols and abbreviated terms – Part 1: Basic polymers and their special characteristics*).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

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/00f2e0bf-2d9a-4cbf-9421-1fb6f8bdda72/iso-8985-1998>

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(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.

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.

3.2.4.2 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.

3.2.5 Expression of results

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

$$w(\text{VAC}) = \frac{8,609(v_3 - v_4)c_2}{m}$$

where

v_3 is the volume, in millilitres, of standard hydrochloric acid used for the blank test;

v_4 is the volume; in millilitres, of standard hydrochloric acid solution used for the determination;

c_2 is the actual concentration, in moles per litre, of the standard hydrochloric acid solution used for the titration;

m is the mass, in grams, of the test portion (see 3.2.4.1.1).

3.2.5.2 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.

3.2.6 Test report

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The test report shall contain the following information:

- a reference to this International Standard and the method used;
- all details necessary for complete identification of the sample;
- the result, expressed in accordance with 3.2.5.2.

3.3 Reference method 3: Measurement of oxygen

3.3.1 Principle

The determination of the oxygen content is carried out using one of the traditional methods of elementary organic analysis. Therefore the following methods apply:

Method	Reaction	Detection method	Test sample	Measurement	Detection range %	Absolute dispersion %
3.3.3.1	Pyrolysis and reoxidation	Coulometry	Micro	Absolute	0,2	(0,02)
3.3.3.2	Pyrolysis	Infrared absorption	Micro	Comparative	0,03	10 relative
3.3.3.3	Pyrolysis and reoxidation	Gravimetric analysis	Macro	Absolute	0,05	0,05

3.3.2 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 $O_2 < 1\%$

3.3.1 Determination by acidimetric coulometry

3.3.3.1.1 Principle

The oxygen is transformed into carbon monoxide by pyrolysis at 1070°C, in a helium or nitrogen atmosphere, of a micro-analytical sample and passing the pyrolysis gases through an oven lined with amorphous carbon heated to 1120°C.

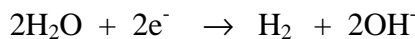
Any acid components are absorbed by a mixture of soda lime and magnesium perchlorate. The carbon monoxide is then passed over CuO at 250°C where it is oxidized to carbon dioxide which is determined by coulometry.

3.3.3.1.2 Coulometric measurement of the carbon dioxide

The carbon dioxide is absorbed by the cathodic part of a coulometric cell, containing an approx. 5% (m/V) solution of barium perchlorate, in accordance with the following formula:



The H^+ ions are neutralized by adding OH^- ions, which are produced electrolytically by the coulometer, in accordance with the following formula:



The electricity supply from the coulometer is controlled by a silver/silver chloride and a glass electrode which detect the variations in the pH of the solution, and the quantity of carbon dioxide is calculated from the quantity of electricity supplied.

3.3.3.1.3 Analysis

Although coulometry is an absolute method, the specific conditions of analysis require calibration and the measurement of scatter in accordance with 3.3.5.

The "response factor" of the apparatus is:

$$F_o = \frac{m_o}{Q_R - Q_o} = 0,0829$$

The percentage of oxygen is then:

$$y = \frac{0,0829(Q_x - Q_o)100}{m}$$

where

- Q_0 is the quantity of electricity measured during a blank test, i.e. an analysis carried out with an empty dish;
- Q_R is the quantity of electricity measured during the analysis of mass m , in milligrams, of a reference substance containing mass m_0 , in milligrams, of oxygen;
- Q_x is the quantity of electricity measured during the analysis of mass m , in milligrams, of the unknown substance.

0,0829 is the quantity of oxygen, in milligrams, corresponding to 1 C in the conditions above.

3.3.2 Determination by infrared absorption

3.3.3.2.1 Principle

The test portion is degraded by pyrolysis under an inert gas (either nitrogen or argon). The gases produced are reduced over carbon at 1120°C. All the oxygen is converted into carbon dioxide which is passed into the measurement cell through which a monochromatic infrared beam passes (using suitable light filters).

The absorption of the infrared radiation by the carbon dioxide causes both a weakening in the luminous intensity and also an increase in the temperature and pressure in the cell. An electrical signal is generated either by a photometer or a pressure sensor. It is often compared with an argon or nitrogen cell sensor for cross-reference purposes. The size of the electrical signal is proportional to the amount of carbon dioxide in the gas mixture and therefore to the amount of oxygen in the sample.

3.3.3.2.2 Apparatus

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- **Elemental analyser**, with infrared detector.
- **Analytical balance**, accurate to 1 mg.
- **Crucibles**, suitable for pyrolysis.

3.3.3.2.3 Reagents

Reagents are specified for each method and type of apparatus. Reagents of a quality referred to as "for organic micro-analysis" are required.

3.3.3.2.4 Procedure

The procedures are specific to each type of apparatus. Certain handling precautions are essential, as follows:

- handle crucibles using tongs only;
- use a spatula when weighing out test portions;
- carry out the correct calibration procedures (see 3.3.5).

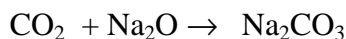
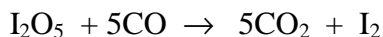
3.3.3.2.5 Calculations

The oxygen concentrations are determined by comparing (automatically or otherwise) the incoming signals received by the detector during analysis of the unknown samples with the coefficients of proportionality resulting from the analysis of the reference samples (apart from the blank test). They are expressed as a percentage by mass (x).

3.3.3.3 Determination by gravimetric analysis

3.3.3.3.1 Principle

A test portion is pyrolysed under argon gas at 1100°C, followed by oxidation of the carbon monoxide formed by iodine pentoxide. The carbon dioxide is passed through a pre-weighed absorber containing sodium oxide on a suitable support. The increase in mass of the absorber is proportional to the oxygen content of the test portion. The reactions are shown below:



3.3.3.3.2 Apparatus

The apparatus is shown in figure 1.

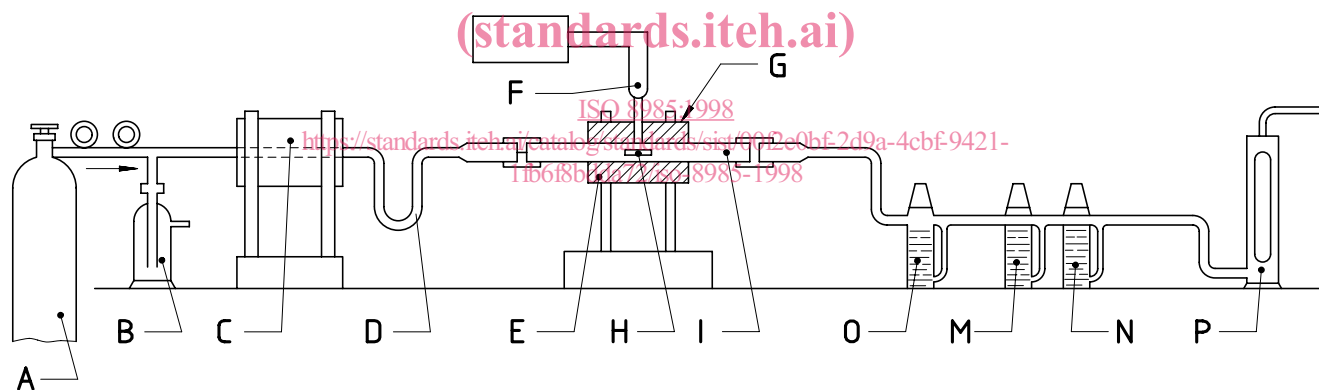


Figure 1 — Apparatus for gravimetric analysis

Key:

- A Argon supply with pressure regulator.
- B Mercury valve.
- C Contact oven with a non-porous porcelain combustion tube containing platinum on a suitable support.
- D Drying and purification unit using argon, containing anhydrous magnesium perchlorate MgClO_4 and sodium oxide on a suitable support, separated by glass wool (in tubes of diameter 25mm and a height of approx. 100mm), the unit being connected by a clip to the oven.
- E Electric or resistance oven, made of carborundum or metal, capable of heating the combustion tube to 1350°C.