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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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Contents

Page

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).

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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.

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(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 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(VAC), expressed as a percentage by mass, is given by the Formula (1):

$$w(\mathrm{VAC}) = \frac{0{,}08\ 609 \times (V_1 - V_2) \times c_1}{m} \times 100 \tag{1}$$

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);

0,08 609 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 %

Table 3 — Traditional methods of elementary organic analysis

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

4.3.3 procedure

4.3.3.1 Determination by acidimetric coulometry

4.3.3.1.1 Principle

The oxygen is transformed into carbon monoxide by pyrolysis at 1 070 °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 1 120 °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.

4.3.3.1.2 Coulometric measurement of the carbon dioxide

The carbon dioxide is absorbed by the cathodic part of a coulometric cell, containing an approximately 5 g/l solution of barium perchlorate, in accordance with the following formula:

$$CO_2 + H_2O + Ba^{2+} \rightarrow BaCO_3 + 2H^+$$

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

$$2H_2O + 2e^- \rightarrow H_2 + 2OH^-$$

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.

4.3.3.1.3 Analysis

Although coulometry is an absolute method, the specific conditions of analysis require calibration and the measurement of scatter in accordance with 4.3.5.

The "response factor" of the apparatus, F_0, is given by Formula (3):

$$F_0 = \frac{m_0}{Q_R - Q_0} = 0{,}082\ 9 \qquad (3)$$

The percentage of oxygen, *y*, is given by Formula (4):

$$y = \frac{0{,}082\ 9 \times (Q_x - Q_0)}{m} \times 100 \quad (4)$$

where

m_0 is the mass of oxygen, in mg, in reference portion;

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 mg, of a reference substance containing mass m_0, in mg, of oxygen;

Q_x is the quantity of electricity measured during the analysis of mass m, in mg, of the unknown substance;

0,0829 is the quantity of oxygen, in mg, corresponding to 1 °C in the conditions above;

m is the mass, in mg, of the test portion..

4.3.3.2 Determination by infrared absorption

4.3.3.2.1 Principle

The test portion is degraded by pyrolysis under an inert gas (either nitrogen or argon). The gases produced are reduced over carbon at 1 120 °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.

4.3.3.2.2 Apparatus

— **Elemental analyser**, with infrared detector.

— **Analytical balance**, accurate to 1 mg.

— **Crucibles**, suitable for pyrolysis.

4.3.3.2.3 Reagents

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

4.3.3.2.4 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 4.3.5).

4.3.3.2.5 Calculations

Potassium iodide/potassium (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).

4.3.3.3 Determination by gravimetric analysis

4.3.3.3.1 Principle

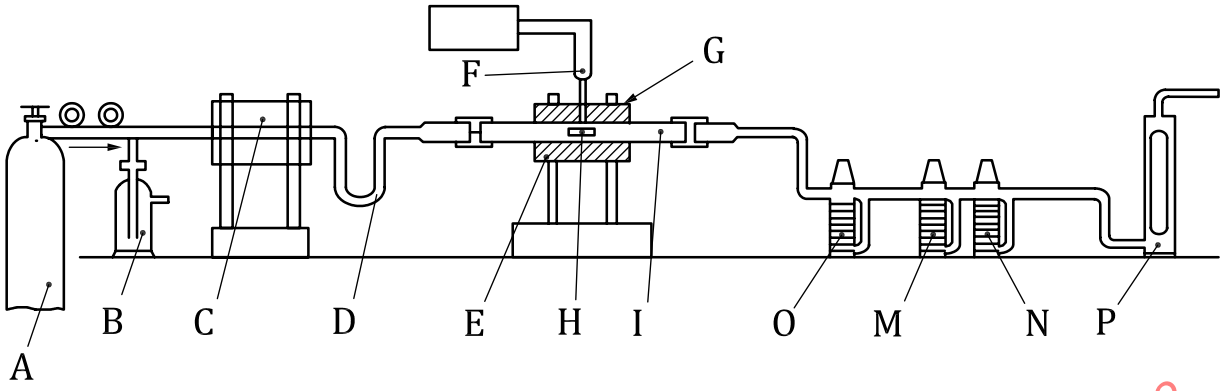
A test portion is pyrolysed under argon gas at 1 100 °C, followed by oxidation of the carbon monoxide formed by iodine pentoxide. The carbon dioxide is passed through a pre-weighed absorber containing sodium oxide on a suitable support. The increase in mass of the absorber is proportional to the oxygen content of the test portion. The reactions are shown below:

$$I_2O_5 + 5CO \rightarrow 5CO_2 + I_2$$

$$CO_2 + Na_2O \rightarrow Na_2CO_3$$

4.3.3.3.2 Apparatus

The apparatus is shown in Figure 1.



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 25 mm and a height of approximately 100 mm), 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 1 350 °C

F thermocouple, to measure the temperature of the oven. The pointer on the thermocouple, protected by a sleeve, shall be close to the external surface of the combustion tube. The relationship between the internal temperature of the tube and the figures on the pyrometer scale shall be set beforehand

G refractory tube (non-porous at the test temperature), with an internal diameter of 20 mm to 30 mm and a minimum length of 650 mm so that its ends do not become warm during combustion

H fireclay dish, calcined beforehand in argon for 2 h at 950 °C

I quartz wool plug, to filter the gases

M absorption unit to absorb the water vapour produced, containing anhydrous magnesium perchlorate.

N absorption unit to absorb the carbon dioxide produced, containing sodium oxide on a suitable support

O column containing iodine pentoxide

P flowmeter

Figure 1 — Apparatus for gravimetric analysis

4.3.3.3.3 Reagents

— **Anhydrous magnesium perchlorate**.

— **Sodium oxide**, on a suitable support.

— **Iodine pentoxide**.

4.3.3.3.4 Procedure

First heat the pyrolysis unit to approximately 950 °C for 2 h under argon, then place in a desiccator. Adjust the oven temperature to 1 100 °C and the argon flow rate to 20 l/h. Purge the sodium oxide absorber for 5 min, remove from the apparatus and weigh to an accuracy of one tenth of a milligram at ambient temperature after 5 min. Place a test portion of approximately1 g (weighed to the nearest 0,1 mg) in the pyrolysis unit and put in the oven. Carry out pyrolysis for 30 min. Then turn off the gas supply, switch off and disconnect the absorber and weigh at ambient temperature after 10 min. Reconnect the absorber, turn on the argon supply once more and weigh again as before after 5 min. The pyrolysis and gas absorption shall be considered complete if the mass of the absorber has not varied by more than 0,5 mg between the two weighings.

4.3.3.3.5 Calculations

The oxygen content, *y*, expressed as a percentage by mass, is given by the Formula (5):

$$y = \frac{\left(m_a^{'} - m_a\right) \times 0{,}363}{m} \times 100 \tag{5}$$

where

$m_a^{'}$ is the mass, in g, of the absorber after pyrolysis;

m_a is the mass, in g, of the absorber before pyrolysis;

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

0,363 is the conversion factor for carbon dioxide into oxygen (the fact that the carbon monoxide is oxidized to carbon dioxide by adding an oxygen atom from the reagent and not from the test portion has been taken into account).

4.3.4 Sampling

The mass of test portion suitable for the "micro" methods is approximately 30 mg.

The mass of test portion suitable for the "macro" method is about 3 g.

4.3.5 Calibration

Certain precautions are common to micro-analytical methods, i.e.:

Calibration is carried out using the same procedure as for the unknown samples, using analytical-quality reagents containing known amounts of oxygen.

Recommended reagents are showed Table 4.

Table 4 — Recommended reagents

Reagents	Amounts of oxygen %
Cholesterol	4,14
Pyramidon	6,92
Acetanilide	11,84
Tryptophane	15,68

— A calibration stage during which the analytical parameters of the analyser are determined by processing the signals from the reference samples in order to establish the coefficient of proportionality between the signal received by the detector and the quantity of oxygen in the test portion.

— An analysis stage during which the analysis of the unknown samples is carried out by comparing the signals obtained with those from the calibration stage. It is necessary to verify whether the coefficients of proportionality remain constant. This verification is carried out every fifth analysis. The values of the coefficients shall not show a scatter greater than 0,3 % relative.

4.3.6 Calculation of vinyl acetate content

If y is the percentage of oxygen in the copolymer, determined by one of the three methods described in 4.3, the vinyl acetate content w(VAC), expressed as a percentage by mass, is given by the Formula (6):

$$w(\mathrm{VAC}) = 2{,}668\, y \tag{6}$$

where 2,668 is the conversion factor for oxygen to the vinyl acetate.

4.3.7 Test report

The test report shall include the following information:

a) all details necessary for complete identification of the sample analysed;

b) the method used;

c) the individual results and their mean;

d) details of any operations not specified in this document, as well as any incidents which might have affected the results.

5 Examples of test methods

5.1 Method using infrared spectrometry

5.1.1 Principle

The vinyl acetate content is determined using infrared spectrometry. This method involves determining the ratio of the absorbance in the n(CH_2) band at 2 678 cm^{-1} and the 2n(CO) band at 3 460 $cm^{-1}$of an EVAC film with a thickness of between 50 µm and 300 µm. The absorbance ratio is converted to vinyl acetate content using a calibration curve established using standard EVAC reference specimens with known vinyl acetate contents measured as in Clause 4. It is not necessary to know the exact thickness of the film as an infrared band is used as an internal reference.

NOTE 1 This method is suitable for samples containing 3 % or more of VAC.

NOTE 2 The internal reference peak at 3 605 cm^{-1} can be used as the internal reference instead of the peak at 2 678 cm^{-1} for samples with a thickness greater than 200 µm. If manual measurement of thickness is possible, it can be used instead of the absorbance at the internal reference peak.

NOTE 3 A drawback to this method is the possible interference by stabilizers or additives. Other CO peaks exist at 610 cm^{-1}, 1 020 cm^{-1}, 1 250 cm^{-1} and 1 743 cm^{-1}, however, they can be used instead of the peak at 3 460 cm^{-1}, with a sample thickness appropriate to the VAC content of the sample.

5.1.2 Apparatus and materials

Standard laboratory equipment, plus the following:

5.1.2.1 Fourier infrared spectrometer, wave number range 4 000 cm^{-1} to 400 cm^{-1}, capable of resolution to 1 cm^{-1}.

5.1.2.2 Specimen holder.

5.1.2.3 Hot-pressing machine, supplying at least 10 MPa pressure, with hotplates which can achieve a temperature of 150 °C.

5.1.2.4 EVAC reference specimens, the vinyl acetate contents of which have been determined using one of the reference methods described in Clause 4.

5.1.3 Procedure

5.1.3.1 Preparation of films

Prepare films of constant thickness using the hot-pressing machine (5.1.2.3) at approximately 150 °C. For VAC contents of less than or equal to 10 %, use 200 µm to 300 µm. For VAC contents greater than 10 % use 50 µm to 150 µm.

NOTE To assist with shaping and to prevent EVAC sticking to the plate surface, a thin film of polytetrafluoroethylene (PTFE) can be used. Where the VAC content is less than 20 %, the PTFE film can be replaced by aluminium foil.

5.1.3.2 Measurement

5.1.3.2.1 Set the wave number range of the Fourier infrared spectrometer to 4 000 cm^{-1} to 400 cm^{-1}, the wave number of resolution to 4 cm^{-1}.

5.1.3.2.2 Scan the background without placing an EVAC specimen in the specimen holder (5.1.2.2).

5.1.3.2.3 Insert an EVAC reference specimen (5.1.2.4) prepared in accordance with 5.1.3.1 into the specimen holder (5.1.2.2). Scan the sample under the same conditions as the background scan to obtain the infrared spectrum of the sample with the background subtracted.

5.1.3.2.4 Draw baselines tangential to the trace at 3 520 cm^{-1} and 3 400 cm^{-1} (for the 3 460 cm^{-1} band) and at 3 280 cm^{-1} and 2 430 cm^{-1} (for the 2 678 cm^{-1} band) as shown in Figure 2.

5.1.3.2.5 Determine the absorbance at 3 460 cm^{-1} and 2 678 cm^{-1} (see the example given in Figure 2).

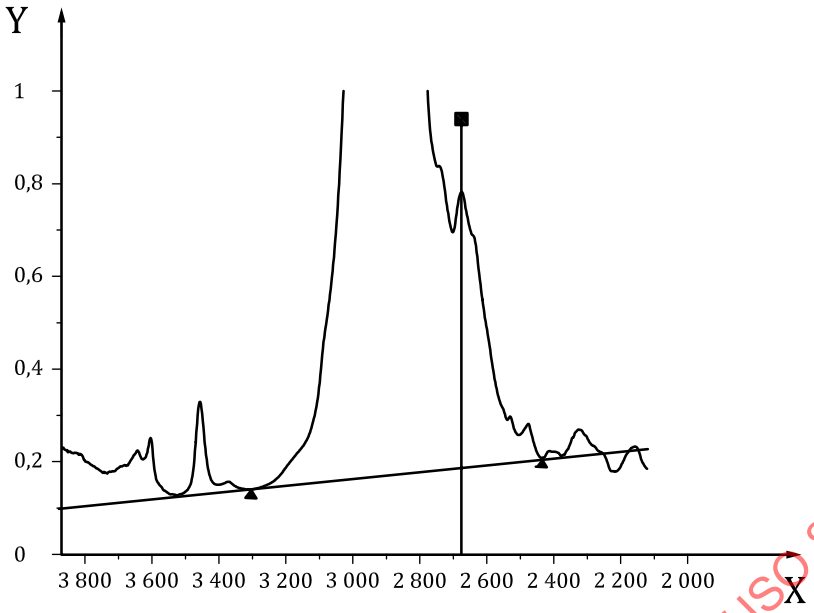
5.1.3.2.6 Repeat the measurements as detailed in 5.1.3.2.2 to 5.1.3.2.5 for each of the other reference specimens and for the test (unknown) specimen. The spectrometer operating conditions, such as sweep rate, measurement range, balance, slit width and sensitivity, shall remain unaltered during this process.

5.1.3.3 Calibration curve

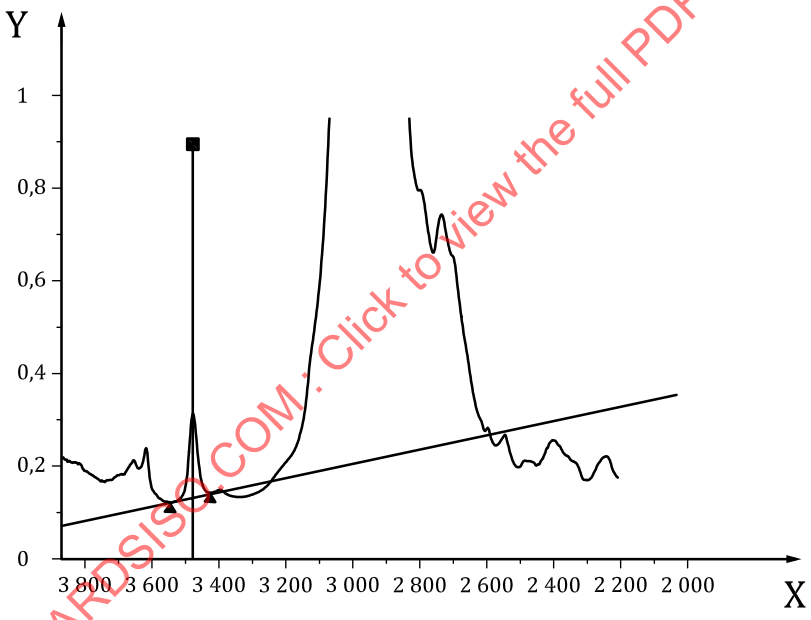
Plot a calibration curve using the values of the VAC content determined by one of the reference methods in Clause 4 and the values of the absorbance ratio calculated for the EVAC reference specimens. An example of such a curve is given in Figure 3.

To guarantee the regression coefficient of calibration curve, stepwise regression was used, for example, the 0,5 % to 10,0 % vinyl acetate range and the 10,0 % to 28,0 % vinyl acetate range.

A minimum of 5 reference values shall be used for the linear regression, of the 0,5 % to10,0 % vinyl acetate range, 5,0 % to 28,0 % vinyl acetate range. The number of standards used for the nonlinear regression, of more vinyl acetate ranges shall not be less than six.



a) Vinyl acetate absorbance peak



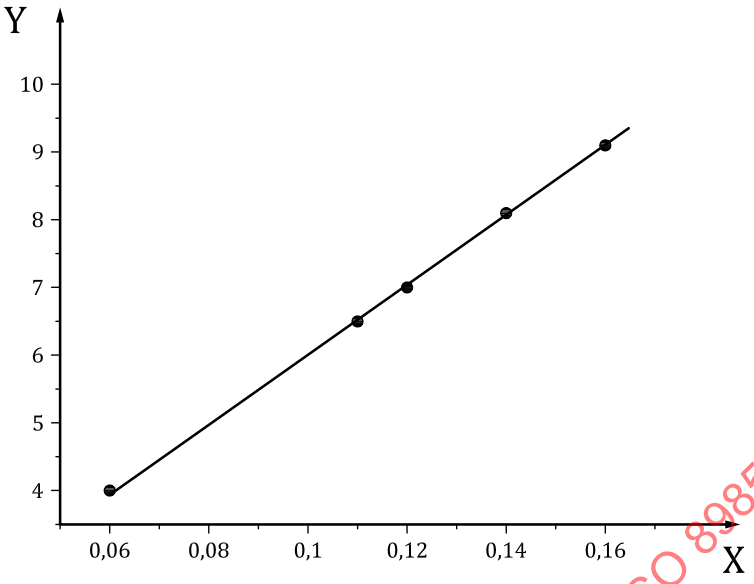
b) Acetic acid absorbance peak

Key

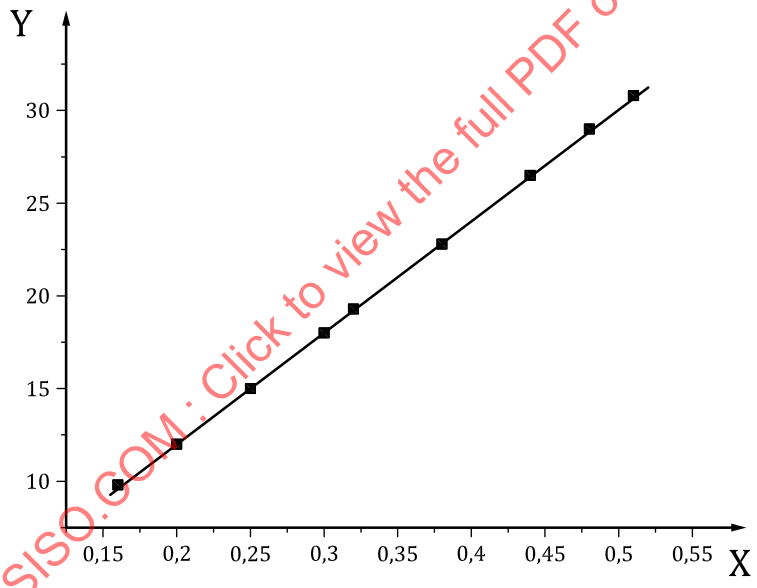
Y absorbance

X wave number,cm^{-1}

Figure 2 — Example of IR spectrum and determination of absorbance



a) Calibration curve(3 % ≤ *w*(VAC) < 10 %)



b) Calibration curve(*w*(VAC) ≥ 10 %)

Key

Y VAC content (%)

X absorbance ratio ($A_{(3\,460)}/A_{(2\,678)}$)

Figure 3 — Example of a calibration curve

5.1.4 Expression of results

The vinyl acetate content of the sample is determined by reading, from the calibration curve, the VAC content, expressed as a percentage by mass, corresponding to the absorbance ratio obtained for the test specimen. If the absorbance ratio for the test specimen lies on the linear section of the calibration curve, the vinyl acetate content can also be calculated using the Formula (7):

$$w(\mathrm{VAC}) = K \times \frac{A_{(3\ 460)}}{A_{(2\ 678)}} \tag{7}$$

where

K is the ratio of the VAC content to the absorbance ratio for the linear section of the curve, determined using the least-squares method;

$A_{(3\ 460)}$ is the absorbance of 3 460 cm^{-1} peak;

$A_{(2\ 678)}$ is the absorbance of 2 678 cm^{-1} peak.

5.1.5 Test report

The test report shall include the following information:

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

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

c) the result, expressed in accordance with 5.1.4;

d) details of any operations not specified in-this document, as well as any incidents which might have affected the results.

5.2 Acidimetric method

5.2.1 Principle

A test portion is placed in an oven set at 350 °C. The inside of the oven is connected, via a glass tube, to a wash bottle containing a solution of potassium hydroxide. The pyrolysis products are transferred, by means of a hot flow of nitrogen, to the wash bottle, in which the acid decomposition gases are absorbed by the potassium hydroxide solution. The volume of potassium hydroxide solution necessary for the absorption phase is then determined by titration with a standard solution of hydrochloric acid, using phenolphthalein as indicator.

5.2.2 Reagents and materials

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

5.2.2.1 Potassium hydroxide, solution, ***c***(KOH) = 0,1 mol/l.

5.2.2.2 Hydrochloric acid, **standard solution**, ***c***(HCl) = 0,1 mol/l.

5.2.2.3 Phenolphthalein, indicator solution, made by dissolving 0,1 g of phenolphthalein in 100 ml of ethanol.

5.2.2.4 Nitrogen.

5.2.3 Apparatus

Standard laboratory equipment, plus the following:

5.2.3.1 Pyrolysis oven, adjustable to 350 °C.

5.2.3.2 Combustion boat, made of vitrified porcelain, 110 mm long, 12 mm wide and 8 mm high.

5.2.3.3 Drechsel wash bottle, 250 ml capacity, with a sintered-glass plate[1]. An example is shown in Figure 4.

5.2.3.4 Schellbach automatic-zero burette, 50 ml capacity.

5.2.3.5 Conical flask, 250 ml capacity.

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

5.2.4 Procedure

5.2.4.1 Determination

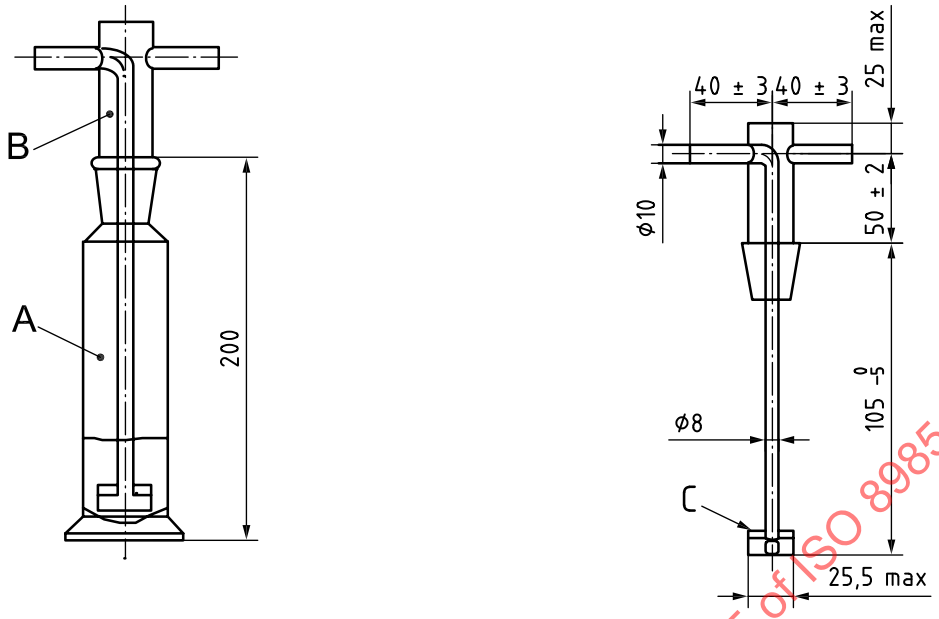
Weigh approximately 2,5 g of sample, to the nearest 0,1 mg, in a combustion boat (5.2.3.2). Place in the pyrolysis oven (5.2.3.1) which has been preheated to 350 °C. Connect the wash bottle (5.2.3.3), containing 100 ml of potassium hydroxide solution (5.2.2.1), to the interior of the oven using a glass tube. Verify that the seal between the oven and the wash bottle, which shall be replaced before each determination, is correctly positioned. Pass nitrogen (5.2.2.4) at a flow rate of 15 l/h into the oven and into the potassium hydroxide solution in the wash bottle. After approximately 2 hours, transfer 50 ml of the potassium hydroxide solution from the wash bottle to the conical flask (5.2.3.5), using a pipette. Determine by titration the amount of potassium hydroxide which has not been neutralised by the acid gases, using hydrochloric acid (5.2.2.2) and adding a few drops of phenolphthalein solution as indicator.

5.2.4.2 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.

1) A type D1 sintered-glass plate is available commercially. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of this particular product. Equivalent products can be used if they can be shown to lead to the same results.

Dimensions in millimetres



a) Bottle and insert **b) Detail of the insert**

Key

A bottle
B insert
C fritted glass plate

Figure 4 — Drechsel wash bottle

5.2.5 Expression of results

5.2.5.1 Method of calculation

The vinyl acetate content, expressed as a percentage by mass, is given by the Formula (8):

$$w(\mathrm{VAC}) = \frac{2\times(V_5 - V_6)\times 0{,}08\ 609\times c_2}{m}\times 100 \qquad (8)$$

where

V_5 is the volume, in ml, of the standard hydrochloric acid solution used for the blank test;

V_6 is the volume, in ml, of the standard hydrochloric acid solution used for the determination;

c_2 is the actual concentration of the hydrochloric acid solution;

m is the mass, in g, of the test portion.

5.2.6 Test report

The test report shall include the following information:

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

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