# 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*

Reference number
ISO 8985:1998(E)

## Foreword

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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Note – The abbreviation previously used for ethylene/vinyl acetate copolymer (E/VAC) has been replaced by the abbreviation EVAC (see ISO 1043-1:1997, *Plastics – Symbols and abbreviated terms – Part 1: Basic polymers and their special characteristics*).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 phenophthalein 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 **hydrochoric 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(NaOH) = 0,1 mol/l.

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

**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 |
|---|---|
| $w(VAC) \leq 10$ | 1 |
| $10 < w(VAC) \leq 20$ | 0,5 |
| $20 < w(VAC) \leq 40$ | 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:

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

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.

**3.2.2.1 Xylene.**

**3.2.2.2 Hexan-1-ol.**

**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(HCl) = 0,3mol/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 |
|---|---|
| $w(VAC) \leq 2$ | 1 |
| $2 < w(VAC) \leq 5$ | 0,5 |
| $5 < w(VAC) \leq 30$ | 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(VAC), expressed as a percentage by mass, is given by the formula:

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

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

The test report shall contain the following information:

a) a reference to this International Standard and the method used;

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

c) 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 $O_2 < 1\%$

### 3.3.3.1 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:

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

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.

#### 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

$Q_o$ 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.

#### 3.3.3.2 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

- **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:

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

$$CO_2 + Na_2O \rightarrow Na_2CO_3$$

### 3.3.3.3.2 Apparatus

The apparatus is shown in figure 1.

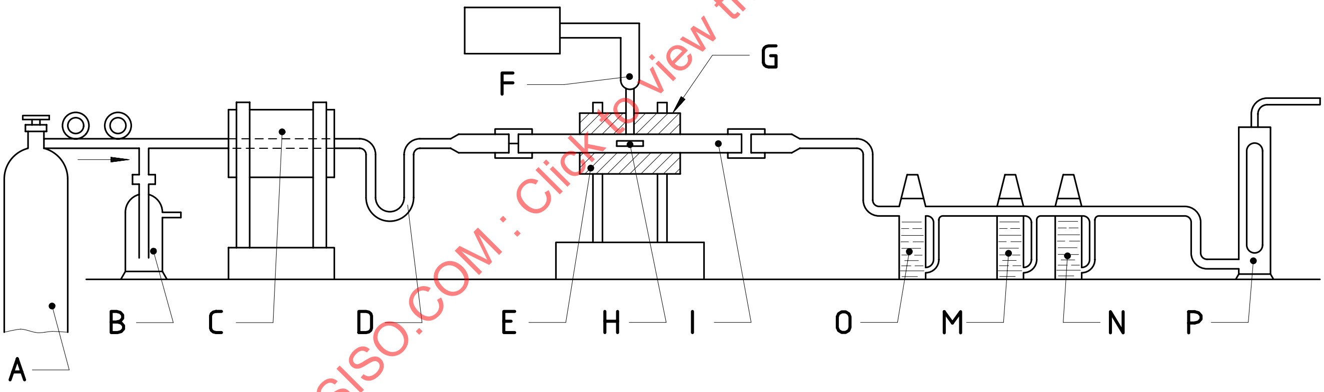


**Figure 1 — Apparatus for gravimetric analysis**

**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. |

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 hours 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.

### 3.3.3.3.3 Reagents

- **Anhydrous magnesium perchlorate.**

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

- **Iodine pentoxide.**

### 3.3.3.3.4 Procedure

First heat the pyrolysis unit to approx. 950°C for 2h under argon, then place in a desiccator. Adjust the oven temperature to 1100°C and the argon flow rate to 20 litres per hour. Purge the sodium oxide absorber for 5min, remove from the apparatus and weigh to an accuracy of one tenth of a milligram at ambient temperature after 5min. Place a test portion of approx.1g (weighed to the nearest tenth of a milligram) in the pyrolysis unit and put in the oven. Carry out pyrolysis for 30min. Then turn off the gas supply, switch off and disconnect the absorber and weigh at ambient temperature after 10min. Reconnect the absorber, turn on the argon supply once more and weigh again as before after 5min. The pyrolysis and gas absorption shall be considered complete if the mass of the absorber has not varied by more than 0,5mg between the two weighings.

### 3.3.3.3.5 Calculations

The oxygen content y, expressed as a percentage by mass, is given by:

$$y = \frac{\left(m_a{}' - m_a\right) \times 36{,}3}{m}$$

where

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

$m_a$ is the mass, in grams, of the absorber before pyrolysis;

$m_a'$ is the mass, in grams, of the absorber after pyrolysis;

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).

### 3.3.4 Sampling

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

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

### 3.3.5 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:

| | % $O_2$ |
|---|---|
| Cholesterol | 4,14 |
| Pyramidon | 6,92 |
| Acetanilide | 11,84 |
| Tryptophane | 15,68 |

The exact procedure may differ slightly from one type of analysis to another, but shall include:

– 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.

### 3.3.6 Calculation of vinyl acetate content

If y is the percentage of oxygen in the copolymer, determined by one of the three methods described in 3.3, the vinyl acetate content w(VAC), expressed as a percentage by mass, is given by:

$$w(VAC) = 2{,}688y$$

### 3.3.7 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 International Standard, as well as any incidents which may have affected the results;

e) the date of the test.

# 4 Examples of test methods

## 4.1 Method using infrared spectrometry

### 4.1.1 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 2678cm$^{-1}$ and the 2n(CO) band at 3460cm$^{-1}$of an EVAC film with a thickness of between 50µm and 300µm. 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 3. 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 10% (m/m) or more of VAC.

Note 2 – The internal reference peak at 3605cm$^{-1}$ may be used as the internal reference instead of the peak at 2678cm$^{-1}$ for samples with a thickness greater than 200µm. If manual measurement of thickness is possible, it may be used instead of the absorbance at the internal reference peak.

Note 3 – A drawback to this method is the possible interference by stabilisers or additives. Other CO peaks exist at 610cm$^{-1}$, 1020cm$^{-1}$, 1250cm$^{-1}$ and 1743cm$^{-1}$, however, and they can be used instead of the peak at 3460cm$^{-1}$, with a sample thickness appropriate to the VAC content of the sample.

### 4.1.2 Apparatus and materials

Standard laboratory equipment, plus the following:

**4.1.2.1 Spectrometer**, wave number range 4000cm$^{-1}$ to 600cm$^{-1}$, capable of resolution to 1 cm$^{-1}$.

**4.1.2.2 Specimen holder.**

**4.1.2.3 Beam shutter.**

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

**4.1.2.5 EVAC reference specimens**, the vinyl acetate contents of which have been determined using one of the reference methods described in clause 3.

### 4.1.3 Procedure

#### 4.1.3.1 Preparation of films

Prepare films of constant thickness using the hot-pressing machine (4.1.2.4) at approx. 150°C. For VAC contents of less than or equal to 10%, use 200µm to 300µm. For VAC contents greater than 10% use 50µm to 150µm.

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

#### 4.1.3.2 Measurement

**4.1.3.2.1** Calibrate the abscissa (wave number) and adjust the absorbance zero for 100% transmission through the spectrometer (4.1.2.1).

**4.1.3.2.2** Insert an EVAC reference specimen (4.1.2.5) prepared in accordance with 4.1 3.1 into the specimen holder (4.1.2.2) and place it in the spectrometer measurement beam.

**4.1.3.2.3** Adjust the baseline using the shutter (4.1.2.3) in the reference beam.

**4.1.3.2.4** Record the spectrum from 4000cm$^{-1}$ to 2000cm$^{-1}$.

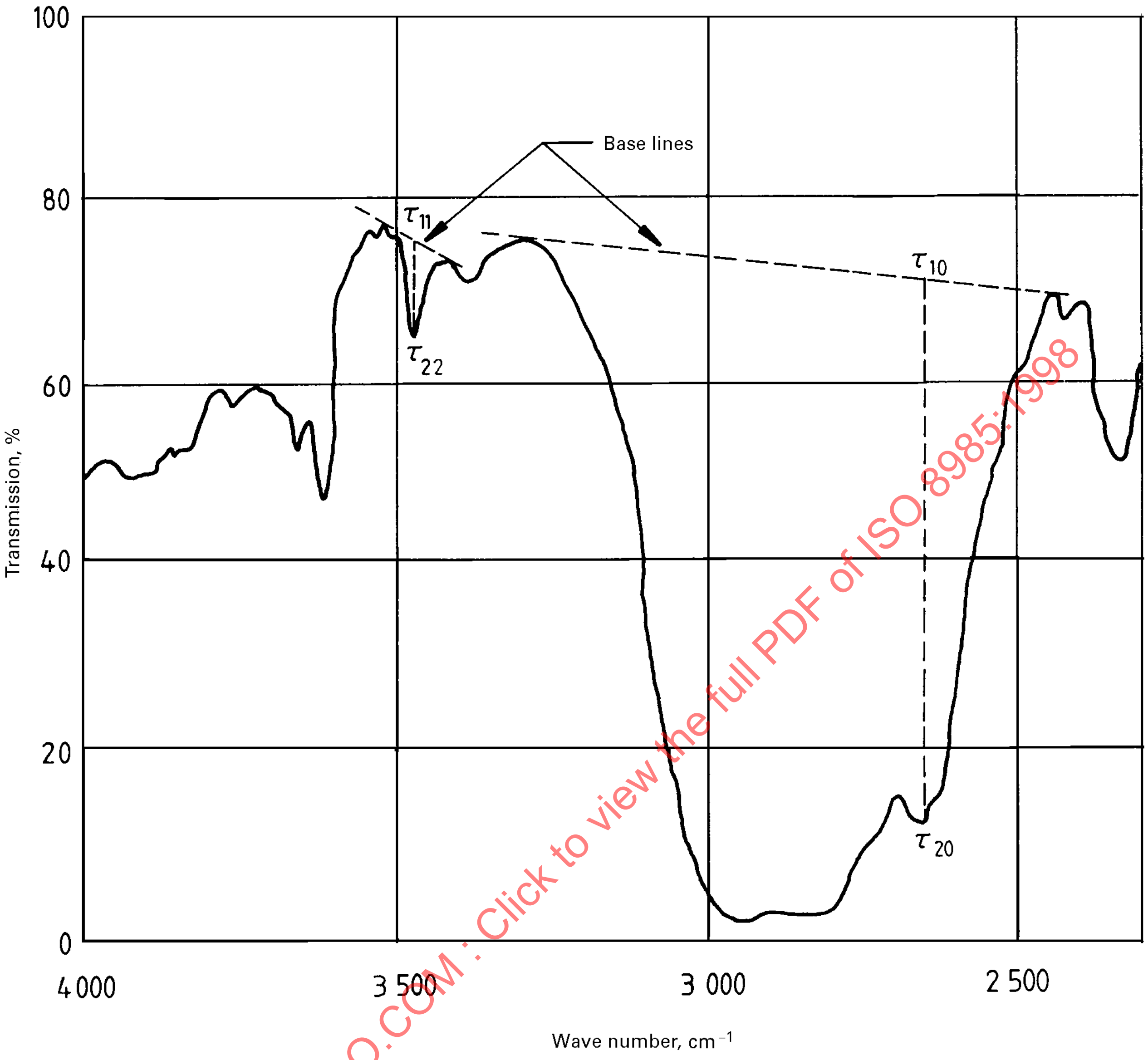
**4.1.3.2.5** Draw baselines tangential to the trace at 3520cm$^{-1}$ and 3400cm$^{-1}$ (for the 3460cm$^{-1}$ band) and at 3280cm$^{-1}$ and 2430cm$^{-1}$ (for the 2678cm$^{-1}$ band) as shown in figure 2.

**4.1.3.2.6** Determine the absorbance at 3460cm$^{-1}$ and 2678cm$^{-1}$ (see the example given in figure 2).

**4.1.3.2.7** Repeat the measurements as detailed in 4.1.3.2.2 to 4.1.3.2.6 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.

#### 4.1.3.3 Calibration curve

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



Absorbance (2678) = $\log_{10} \tau_{10} - \log_{10} \tau_{20} = A_{10} - A_{20}$ = 0,857
Absorbance (3460) = $\log_{10} \tau_{11} - \log_{10} \tau_{22} = A_{11} - A_{22}$ = 0,062
Ratio: Absorbance (3460) / Absorbance (2678) = 0,072

**Figure 2 – Example of IR spectrum and determination of absorbance**

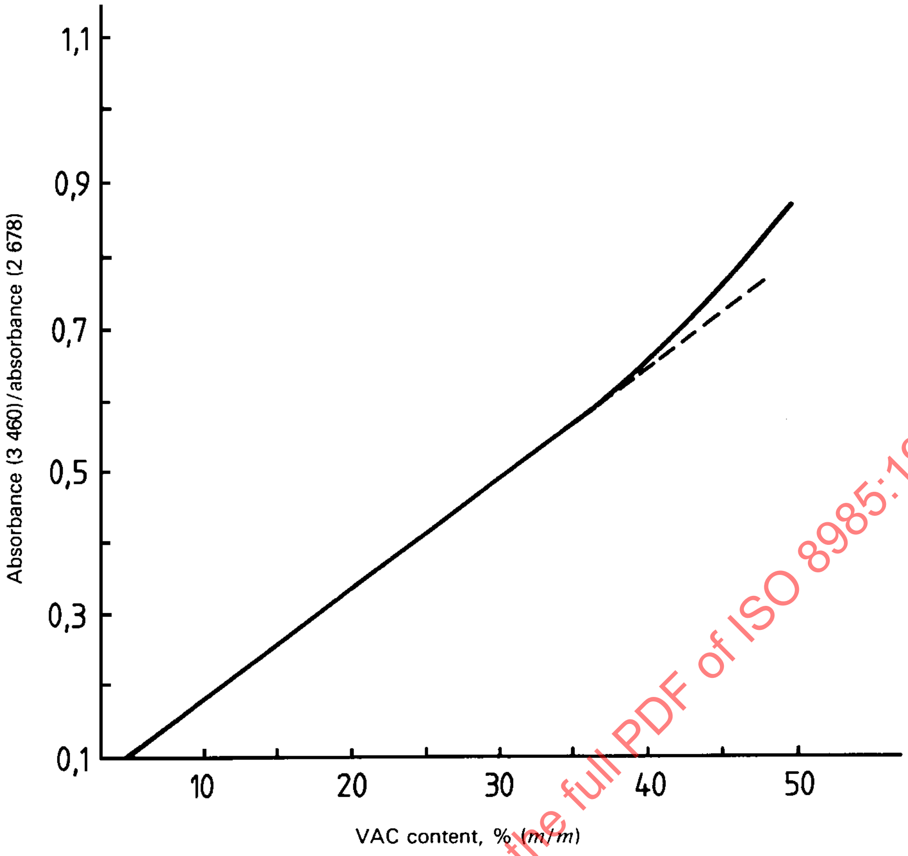


Figure 3 – Example of a calibration curve

### 4.1.4 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:

$$K \times \frac{\text{Absorbance (3460)}}{\text{Absorbance (2678)}}$$

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.

### 4.1.5 Test report

The test report shall include the following information:

a) a reference to this International Standard and the method used;

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

c) the result, expressed in accordance with 4.1.4;

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

## 4.2 Acidimetric method

### 4.2.1 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.

### 4.2.2 Reagents and materials

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

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

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

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

**4.2.2.4 Nitrogen.**

### 4.2.3. Apparatus

Standard laboratory equipment, plus the following:

**4.2.3.1 Pyrolysis oven**, adjustable to 350°C.

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

**4.2.3.3 Drechsel wash bottle**, 250ml capacity, with a sintered-glass plate (see the note). An example is shown in figure 4.

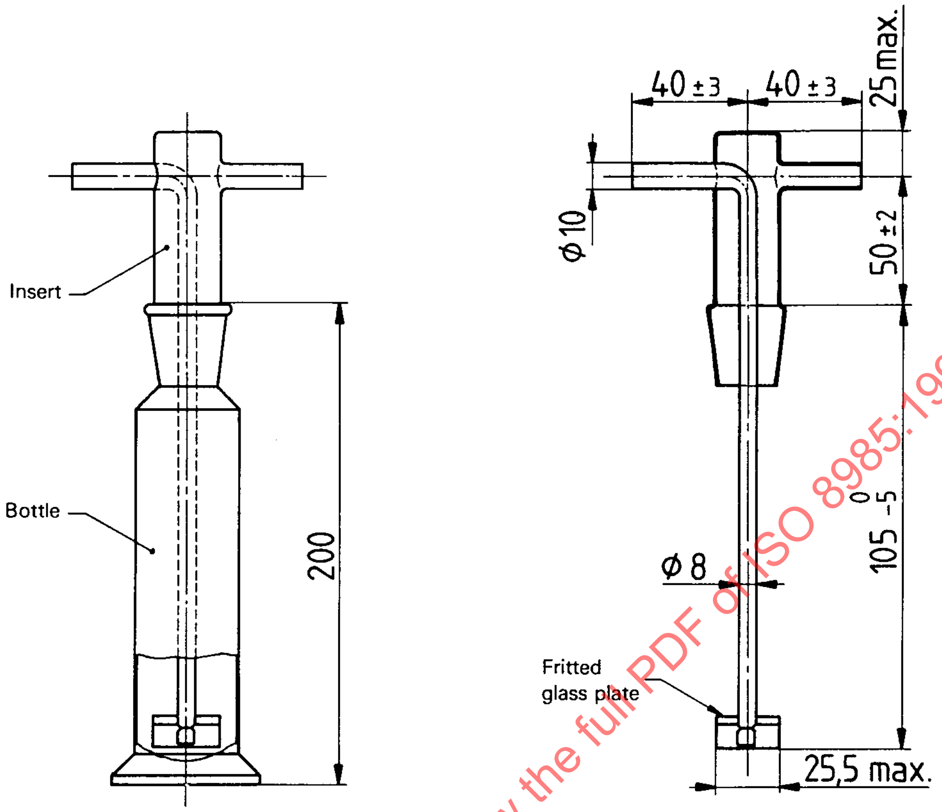
Note – A type D1 sintered-glass plate is available commercially. This information is given for the convenience of users of this International Standard and does not constitute an endorsement by ISO of this particular product. Equivalent products may be used if they can be shown to lead to the same results.

**4.2.3.4 Schellbach automatic-zero burette**, 50ml capacity.

**4.2 3.5 Conical flask**, 250ml capacity.

**4.2.3.6 Analytical balance**, with an accuracy of 0,1mg.

Dimensions in millimetres



a) Bottle and insert b) Detail of the insert

**Figure 4 – Drechsel wash bottle**

### 4.2.4 Procedure

#### 4.2.4.1 Determination

Weigh approx. 2,5g of sample, to the nearest 0,1mg, in a combustion boat (4.2.3.2). Place in the pyrolysis oven (4.2.3.1) which has been preheated to 350°C. Connect the wash bottle (4.2.3.3), containing 100ml of potassium hydroxide solution (4.2.2.1), 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 (4.2.2.4) at a flow rate of 15 litres/h into the oven and into the potassium hydroxide solution in the wash bottle. After approximately 2 hours, transfer 50ml of the potassium hydroxide solution from the wash bottle to the conical flask (4.2.3.6), using a pipette. Determine by titration the amount of potassium hydroxide which has not been neutralised by the acid gases, using hydrochloric acid (4.2.2.2) and adding a few drops of phenolphthalein solution as indicator.

#### 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.

### 4.2.5 Expression of results

#### 4.2.5.1 Method of calculation

The vinyl acetate content, expressed as a percentage by mass, is given by the formula: