International Standard 8174

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Ethylene and propylene for industrial use — Determination of acetone, acetonitrile, propan-2-ol and methanol — Gas chromatographic method

Éthylène et propylène à usage industriel — Dosage de l'acétone, de l'acétonitrile, du propanol-2 et du méthanol — Méthode par chromatographie en phase gazeuse

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Foreword

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Ethylene and propylene for industrial use — Determination of acetone, acetonitrile, propan-2-ol and methanol — Gas chromatographic method

1 Scope and field of application

This International Standard specifies a gas chromatographic method for the determination of acetone, acetonitrile, propan-2-ol and methanol in ethylene and propylene (propene) for industrial use.

The method is applicable to products having acetone, propan-2-ol and methanol concentrations greater than 1 mg/kg, and acetonitrile concentrations greater than 10 mg/kg.

2 References

ISO 6377, *Light olefins for industrial use — Determination of impurities by gas chromatography — General considerations.*

ISO 7382, *Ethylene for industrial use — Sampling in the liquid and the gaseous phase.* [1]

ISO 8563, *Propylene and butadiene for industrial use — Sampling in the liquid phase.* [1]

3 Principle

Passage of a gaseous test portion through water to absorb acetone, acetonitrile, propan-2-ol and methanol, and subsequent gas chromatographic analysis of the aqueous solution, using a flame ionization detector, and comparison of the peaks obtained, with those derived from an external standard.

4 Reagents and materials

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

4.1 Nitrogen, having a water content less than 5 ml/m^3.

4.2 Air, compressed, dry.

4.3 Acetone.

4.4 Propan-2-ol.

4.5 Methanol.

4.6 Acetonitrile.

4.7 Standard mixture, aqueous solution containing 20 mg of each impurity to be determined per litre.

5 Apparatus

5.1 Absorption train (see figure 1), comprising

— a flow meter capable of measuring flow rates between 5 and 100 l/h;

— three absorption flasks (A, B and C) with sintered glass discs (see figure 2);

— a gas meter, graduated every 10 ml.

5.2 Water bath, capable of being controlled between 0 and 5 °C.

5.3 Vaporization device (see ISO 6377, clause 4).

5.4 Chromatograph, fitted with a flame ionization detector, which will yield a peak height of at least five times the noise level at concentrations for each of the impurities as given in clause 1.

5.4.1 Injection device (see ISO 6377, sub-clause 3.2), permitting the introduction of a test portion of 2 µl constant to within ± 1 %.

1) At present at the stage of draft.

5.4.2 Column, such as that described below which has been found suitable, or any other columns giving satisfactory separation for the determination required :

5.4.2.1 Tube

material : stainless steel
length : 1 m
internal diameter : 4 mm
external diameter : 6 mm

5.4.2.2 Packing

Support

material : polytetrafluoroethylene resin, such as Chromosorb T
particle size : 250 to 420 µm

Stationary phase

material : polyethylene glycol 400 and (*N*, *N*-dimethylstearamide), such as Hallcomid M-18

Packing composition

Coat 100 g of Chromosorb T with 10 g of polyethylene glycol 400 and 10 g of Hallcomid M-18.

5.4.3 Recorder, having a response time, on the normal scale, of 2 s or less and a noise level less than 0,1 % on this scale.

6 Sampling and preparation of the sample

Take the laboratory sample in a stainless steel cylinder as specified in ISO 7382 or ISO 8563, and carry out preparation of the sample as indicated in ISO 6377, clause 4.

7 Procedure

WARNING — The operations specified must be carried out under a well-ventilated hood and in a completely flame-free environment and the test samples must be vented to an extraction system. During the manipulations, it is essential to connect the sample cylinder to earth by means of an equipotential clamp.

7.1 Absorption

Assemble the absorption train (5.1), immerse the three absorption flasks (A, B and C) in the water bath (5.2) and introduce 10,0 ml of water into each flask.

Connect the absorption train (5.1) to the vaporization device (5.3) and pass the sample for 2 h, at a rate of 50 l/h. With the gas meter, measure the exact volume taken.

Then pass 2 l of nitrogen (4.2) through the absorption train at a rate of 5 to 10 l/h.

Transfer the contents of the first two absorption flasks (A and B) into a 25 ml one-mark volumetric flask, dilute to the mark and mix.

Keep the contents of the third absorption flask (C) to check for complete absorption in the first two absorption flasks. Inject into the chromatograph (5.4) an aliquot from flask (C) to verify the absence of the components to be determined.

7.2 Setting the chromatograph

a) Injection device

temperature : 150 °C

b) Column

temperature : 65 °C

c) Carrier gas

nitrogen flow rate : 50 ml/min

d) Detector

temperature : 150 °C

7.3 Calibration

Inject 2 µl of the standard mixture (4.8) and record the chromatogram, then identify the peaks and calculate the areas.

7.4 Determination

Inject 2 µl of the solution obtained as in 7,1, record the chromatogram (see figure 3), identify the peaks and calculate the areas.

8 Expression of results

8.1 Calculation in case of ethylene

The acetone, acetonitrile, propan-2-ol, or methanol contents, expressed in milligrams per kilogram, are given by the formula :

$$20 \times \frac{25}{1\,000} \times \frac{A_1}{A_0} \times \frac{1}{V \times 0{,}001\,26} \times \frac{(273 + t)}{273} \times \frac{101\,325}{p}$$

$$= 147\,283 \times \frac{A_1 (273 + t)}{A_0 \times V \times p}$$

where

A_0 is the area, in square millimetres, of the peak of the impurity to be determined obtained with the standard mixture;

A_1 is the area, in square millimetres, of the corresponding peak, obtained with the sample;

V is the volume, in litres, of ethylene, passed through the absorption train;

p is the pressure, in pascals, at which the volume V is measured;

t is the temperature, in degrees Celsius, at which the volume V is measured;

0,001 26 is the density, in kilograms per litre, of the ethylene at 0 °C and at 101 325 Pa.

8.2 Calculation in case of propylene

The acetone, acetonitrile, propan-2-ol or methanol contents, expressed in milligrams per kilogram, are given by the formula specified in 8.1 but taking the value of 0,001 916 instead of 0,001 26 corresponding to the density, in kilograms per litre, of propylene at 0 °C and at 101 325 Pa.

The formula becomes :

$$96\,856 \times \frac{A_1\,(273 + t)}{A_0 \times V \times p}$$

8.3 Precision

In the case of ethylene, a round-robin analysis carried out by 2 laboratories yielded the results from which it was calculated that, at 10 mg of methanol per kilogram, the expected repeatability is ± 1,6 mg/kg and the expected reproducibility is ± 2,5 mg/kg.

9 Test report

The test report shall include the following information :

a) all information necessary for the complete identification of the sample (lot, date, time and duration of each sampling, etc.);

b) a reference to this International Standard;

c) the concentration of each impurity as required;

d) the nature of the hydrocarbon and its concentration in the gaseous standard mixture;

e) a statement of any experimental conditions which are regarded as optional;

— a description of the column or combination of columns used, or reference to the column or combination of columns specified in the International Standard,

— the nature of the carrier gas,

— the pressure, in bars*, of the carrier gas at the entrance to the column or in the first part of the column,

— the flow rate of the carrier gas, in litres per hour, measured at standard atmospheric pressure,

— the volume, in microlitres, measured at standard atmospheric pressure, of solution injected for each test,

— the duration of recording;

f) details of any unusual features noted during the determination;

g) details of any operations not included in this International Standard, or in the International Standards to which reference is made, or regarded as optional.

* 1 bar = 10^5 Pa

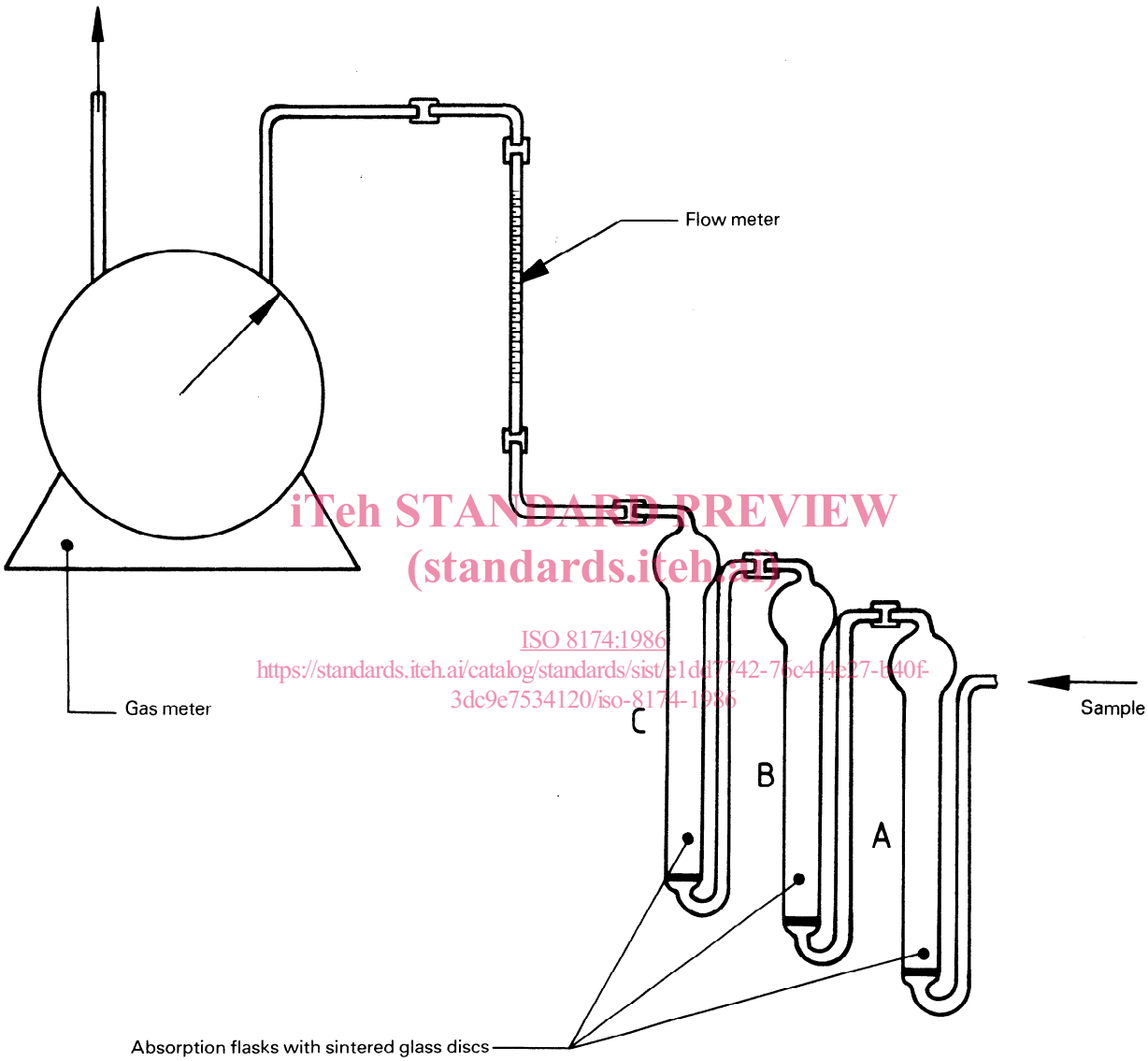

Figure 1 — Absorption train

Dimensions in millimetres

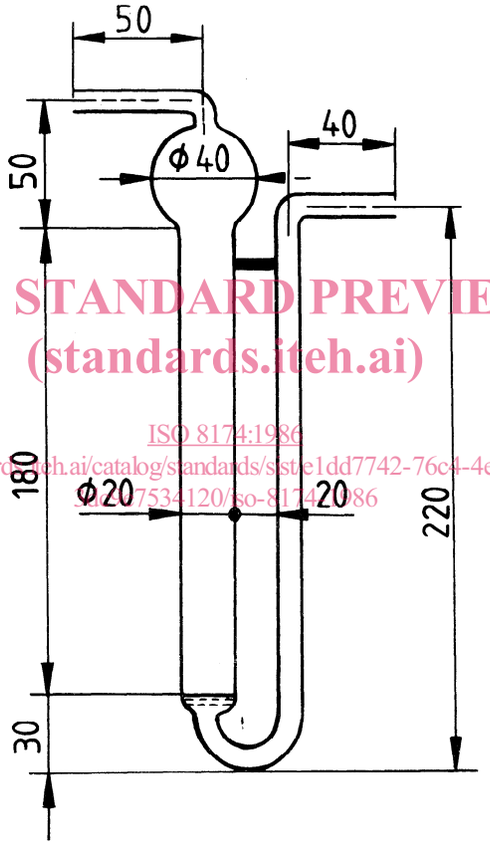

Figure 2 — Absorption flask details

Impurity	Relative retention time
Acetone	1,00
Methanol	1,88
Acetonitrile	2,42
Propan-2-ol	3,26

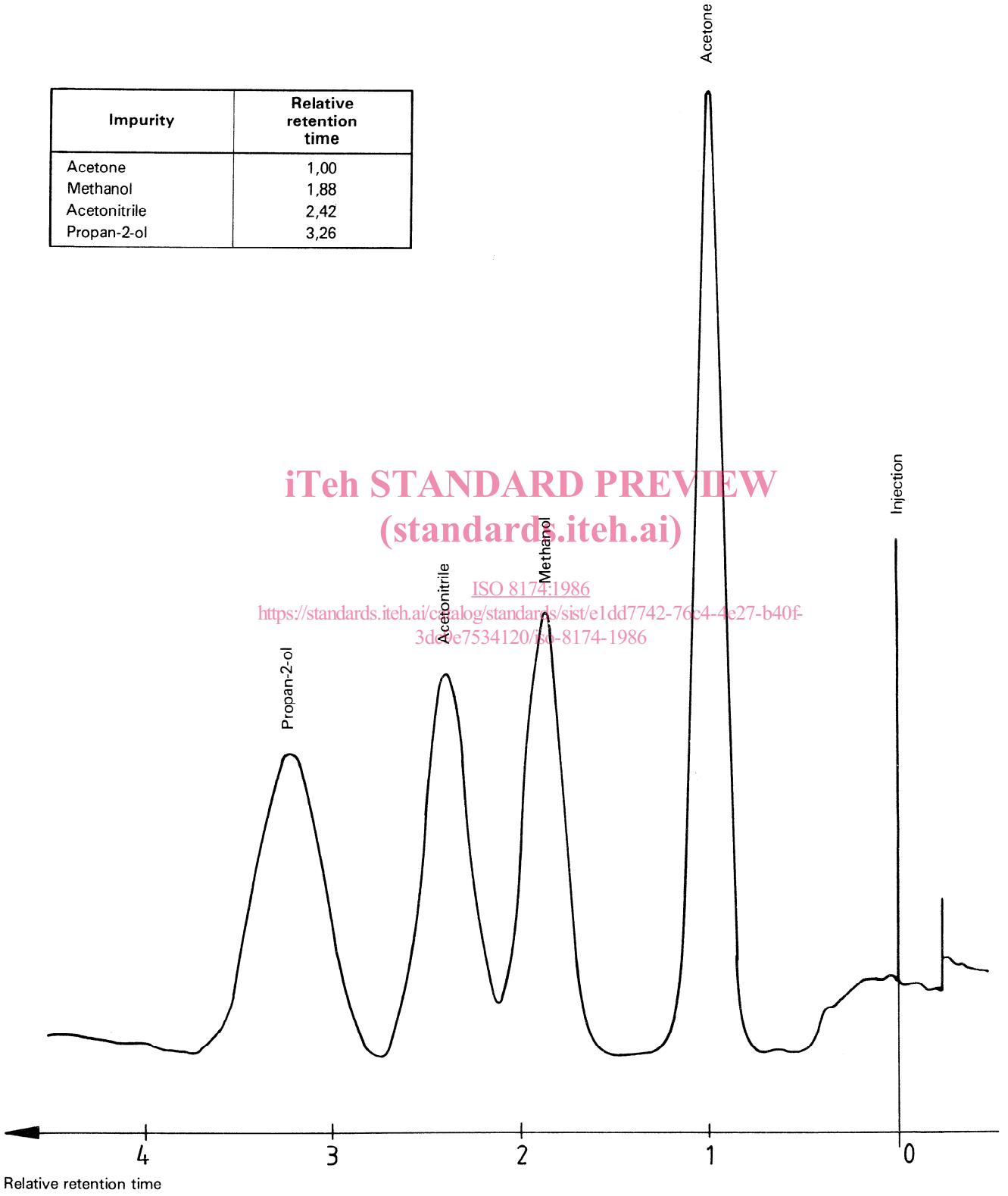

Figure 3 — Typical chromatogram — Determination of acetone, methanol, propan-2-ol and acetonitrile