International Standard 6379

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Ethylene for industrial use — Determination of hydrocarbon impurities — Gas chromatographic method

Éthylène à usage industriel — Dosage des impuretés hydrocarbonées — Méthode par chromatographie en phase gazeuse

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Foreword

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Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council.

International Standard ISO 6379 was developed by Technical Committee ISO/TC 47, *Chemistry*, and was circulated to the member bodies in October 1979.

It has been approved by the member bodies of the following countries :

Australia	Germany, F.R.	Poland
Austria	Hungary	Portugal
Belgium	India	Romania
China	Italy	South Africa, Rep. of
Czechoslovakia	Korea, Rep. of	Switzerland
France	Netherlands	USSR

No member body expressed disapproval of the document.

This International Standard has also been approved by the International Union of Pure and Applied Chemistry (IUPAC).

Printed in Switzerland

Ethylene for industrial use — Determination of hydrocarbon impurities — Gas chromatographic method

1 Scope and field of application

This International Standard specifies a gas chromatographic method for the determination of hydrocarbon impurities in ethylene for industrial use.

The method is applicable to the determination of the impurities listed in annex B and principally to the determination of

— methane (CH_4) at concentrations greater than 5 ml/m³;

— ethane (CH_3-CH_3) at concentrations greater than 5 ml/m³;

— acetylene (ethyne) ($CH\equiv CH$) at concentrations greater than 1 ml/m³;

— propane ($CH_3-CH_2-CH_3$) at concentrations greater than 2 ml/m³;

— propylene (propene) ($CH_2=CH-CH_3$) at concentrations greater than 2 ml/m³.

2 Reference

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

3 Principle

Selection of a gas chromatography column allowing the separation of the impurities to be determined.

Passage of a gaseous test portion through the column, detection by flame ionization and comparison of the peaks obtained with those derived from an external standard.

4 Materials

4.1 Carrier gas

Nitrogen or helium of the best available commercial quality, having water and oxygen contents each less than 5 ml/m³.

4.2 Standards

Prepare (or obtain) standard mixtures in which the concentration of each impurity to be determined is within the concentration limits which are encountered in the product to be analysed.

5 Apparatus

Ordinary laboratory apparatus and

5.1 Chromatograph

Use a gas chromatograph complying with the requirements specified below and 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.

5.1.1 Injection device (see ISO 6377), permitting the introduction into the column of a test portion of about 1 to 5 ml, constant to within ± 1 %.

5.1.2 Columns.

A number of columns which have been found suitable are described in annex A. Use one of these columns, or several of them in succession, or any other columns giving satisfactory separation for the determination required.

5.1.3 Detector, flame ionization type.

5.1.4 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 Preparation of sample

See ISO 6377, clause 4.

7 Procedure

7.1 Preparation of the apparatus

Select a column suitable for the analysis to be performed and condition it by keeping it for at least 12 h at a temperature at least 20 °C higher than the operating temperature, using a carrier gas flow rate equal to that to be used in the analysis.

Set up the column and carry out the adjustments necessary to produce the optimum operating conditions (see annex A). Wait a sufficient time for these conditions to become stable (the production of a stable base line).

7.2 Injection of the test portion

See ISO 6377, clause 5.

7.3 Preliminary test

Inject a preliminary test portion in order to establish that the separation of the peaks corresponding to the impurities to be determined is sufficient. If the contents of the impurities are to be calculated from the peak heights, determine, taking into account the capability of the recorder, the attenuation at which these peaks will be as high as possible.

7.4 Calibration

Inject, in succession, the standard mixtures (4.2) so as to display three peaks, at three different concentrations, for each impurity to be determined.

7.5 Determination

Pass two test portions, in succession, through the chromatograph.

7.6 Examination of the chromatograms

7.6.1 Typical chromatogram

See annex C.

7.6.2 Retention times

See annex B.

7.6.3 Calculation

See ISO 6377, clause 7.

8 Expression of results

For each impurity determined, calculate the mean of the two determinations and express the results in millilitres per cubic metre of product, or in milligrams per kilogram of product.

9 Test report

See ISO 6377, clause 9.

Annex A

Columns and operating conditions which have been found suitable for the determination of hydrocarbon impurities in ethylene

Column	Squalane[1)]	Alumina
Length, m	11	3
Internal diameter, mm **Material**	4 to 5 Stainless steel	2 Stainless steel
Stationary phase	30 % (*m*/*m*) squalane	None
Support	Chromosorb P 60/80	Alumina 100/200
Temperature, °C	50	60 for 5 min, 10 °C/min up to 300 °C
Carrier gas	Helium	Nitrogen
Flow rate, ml/min	42	15

1) Squalane $C_{30}H_{62}$ = 2,6,10,15,19,23-hexamethyltetracosane.

NOTE — Information on proprietary products may be obtained from the ISO Central Secretariat or from the Secretariat of ISO/TC 47/SC 14 (AFNOR).

Annex B

Absolute retention times (in minutes)

Column	Squalane	Alumina
Methane	6,75	3
Ethane	11,5	5
Acetylene (ethyne)	7,6	15,7
Propane	19,6	9,5
Propylene (propene)	17,6	13
***Iso*-butane (2-methylpropane)**	38,9	13,6
***n*-butane**	53,1	14,2
2-butene	56,8	17
1,3-butadiene	56,8	19,7

Annex C

Typical chromatogram from an alumina column