BRITISH STANDARD

BS EN ISO 6974-3:2001

Incorporating Amendment No.1 to BS ISO 6974-3:2000 (renumbers the BS ISO as BS EN ISO 6974-3:2001)

Natural gas — Determination of composition with defined uncertainty by gas chromatography —

Part 3: Determination of hydrogen, helium, oxygen, nitrogen, carbon dioxide and hydrocarbons up to C8 using two packed columns

The European Standard EN ISO 6974-3:2001 has the status of a British Standard

ICS 75.060

British Standards

National foreword

This British Standard is the official English language version of EN ISO 6974-3:2001. It is identical with ISO 6974-3:2000.

The UK participation in its preparation was entrusted to Technical Committee PTI/15, Natural gas and gas analysis, which has the responsibility to:

- aid enquirers to understand the text;
- present to the responsible international/European committee any enquiries on the interpretation, or proposals for change, and keep the UK interests informed;
- monitor related international and European developments and promulgate them in the UK.

A list of organizations represented on this committee can be obtained on request to its secretary.

Cross-references

The British Standards which implement international or European publications referred to in this document may be found in the BSI Standards Catalogue under the section entitled "International Standards Correspondence Index", or by using the "Find" facility of the BSI Standards Electronic Catalogue.

A British Standard does not purport to include all the necessary provisions of a contract. Users of British Standards are responsible for their correct application.

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This British Standard, having been prepared under the direction of the Materials and Chemicals Sector Policy and Strategy Committee, was published under the authority of the Standards Policy and Strategy Committee on 15 August 2000

Summary of pages

This document comprises a front cover, an inside front cover, the EN ISO title page, the EN ISO foreword page, the ISO title page, pages ii to v, a blank page, pages 1 to 14, the Annex ZA page and a back cover.

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Amendments issued since publication

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EUROPEAN STANDARD
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EN ISO 6974-3

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ICS 75.060

English version

Natural gas - Determination of composition with defined uncertainty by gas chromatography - Part 3: Determination of hydrogen, helium, oxygen, nitrogen, carbon dioxide and hydrocarbons up to C8 using two packed columns (ISO 6974-3:2000)

Gaz naturel - Détermination de la composition avec une incertitude définie par chromatographie en phase gazeuse - Partie 3: Détermination de l'hydrogène, de l'hélium, de l'oxygène, de l'azote, du dioxide de carbone et des hydrocarbures jusqu'à C8 à l'aide de deux colonnes remplies (ISO 6974-3:2000)

Erdgas - Bestimmung der Zusammensetzung mit definierter Unsicherhei1 durch Gaschromatographie - Teil 3: Bestimmung von Wasserstoff, Helium, Sauerstoff, Stickstoff, Kohlenstoffdioxid und Kohlenwasserstoffen bis zu C8 mit zwei gepackten Säulen (ISO 6974-3:2000)

This European Standard was approved by CEN on 22 June 2001.

CEN members are bound to comply with the CEN/CENELEC Internal Regulations which stipulate the conditions for giving this European Standard the status of a national standard without any alteration. Up-to-date lists and bibliographical references concerning such national standards may be obtained on application to the Management Centre or to any CEN member.

This European Standard exists in three official versions (English, French, German). A version in any other language made by translation under the responsibility of a CEN member into its own language and notified to the Management Centre has the same status as the official versions.

CEN members are the national standards bodies of Austria, Belgium, Czech Republic, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and United Kingdom.

EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
EUROPÄISCHES KOMITEE FÜR NORMUNG

Management Centre: rue de Stassart, 36 B-1050 Brussels

Ref. No. EN ISO 6974-3:2001 E

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Foreword

The text of the International Standard from Technical Committee ISO/TC 193 "Natural gas" of the International Organization for Standardization (ISO) has been taken over as a European Standard by CMC.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by February 2002, and conflicting national standards shall be withdrawn at the latest by February 2002.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Czech Republic, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and the United Kingdom.

NOTE Normative references to International Standards are listed in annex ZA (normative).

INTERNATIONAL STANDARD

ISO 6974-3

First edition
2000-04-01

Natural gas — Determination of composition with defined uncertainty by gas chromatography —

Part 3:
Determination of hydrogen, helium, oxygen, nitrogen, carbon dioxide and hydrocarbons up to C8 using two packed columns

Gaz naturel — Détermination de la composition avec une incertitude définie par chromatographie en phase gazeuse —

Partie 3: Détermination de l'hydrogène, de l'hélium, de l'oxygène, de l'azote, du dioxyde de carbone et des hydrocarbures jusqu'à C8 à l'aide de deux colonnes remplies

Reference number
ISO 6974-3:2000(E)

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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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 3.

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.

Attention is drawn to the possibility that some of the elements of this part of ISO 6974 may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

International Standard ISO 6974-3 was prepared by Technical Committee ISO/TC 193, *Natural gas*, Subcommittee SC 1, *Analysis of natural gas*.

This part as well as the other five parts of ISO 6974 cancel and replace ISO 6974:1984 which specified only one method.

ISO 6974 consists of the following parts, under the general title *Natural gas — Determination of composition with defined uncertainty by gas chromatography*:

— *Part 1: Guidelines for tailored analysis*

— *Part 2: Measuring-system characteristics and statistics for data treatment*

— *Part 3: Determination of hydrogen, helium, oxygen, nitrogen, carbon dioxide and hydrocarbons up to* C_8 *using two packed columns*

— *Part 4: Determination of nitrogen, carbon dioxide and* C_1 *to* C_5 *and* C_{6+} *hydrocarbons for a laboratory and on-line measuring system using two columns*

— *Part 5: Determination of nitrogen, carbon dioxide and* C_1 *to* C_5 *and* C_{6+} *hydrocarbons for a laboratory and on-line process application using three columns*

— *Part 6: Determination of hydrogen, helium, oxygen, nitrogen, carbon dioxide and hydrocarbons up to* C_8 *using three capillary columns*

Annexes A and B of this part of ISO 6974 are for information only.

Introduction

This part of ISO 6974 describes a precise and accurate method for the analysis of natural gas, which permits the determination of the composition of natural gas. The compositional data obtained are used for the calculation of calorific value, relative density and the Wobbe index.

This method requires the use of two columns which are put into one or two gas chromatographs. The constituents of the eluent of the first column are detected by a thermal conductivity detector (TCD). The constituents of the eluent of the second column are detected by a TCD and flame ionization (FID) in series.

If the two columns are put into one chromatograph the gas chromatographic conditions are described in informative annex A.

This part of ISO 6974 provides one of the methods that may be used for determining the composition of natural gas in accordance with parts 1 and 2 of ISO 6974.

Natural gas — Determination of composition with defined uncertainty by gas chromatography —

Part 3:

Determination of hydrogen, helium, oxygen, nitrogen, carbon dioxide and hydrocarbons up to C8 using two packed columns

1 Scope

This part of ISO 6974 describes a gas chromatographic method for the quantitative determination of the content of helium, hydrogen, oxygen, nitrogen, carbon dioxide and C_1 to C_8 hydrocarbons in natural gas samples using two packed columns. This method is applicable to determinations made in on-line processes or in the laboratory. It is applicable to the analysis of gases containing constituents within the mole fraction ranges given in Table 1 and which do not contain any hydrocarbon condensate. These ranges do not represent the limits of detection, but the limits within which the stated precision of the method applies. Although one or more components in a sample may not be detected present, the method can still be applicable.

This part of ISO 6974 is only applicable in conjunction with parts 1 and 2 of ISO 6974.

Table 1 — Application ranges

Component	Mole fraction range %
Helium	0,01 to 0,5
Hydrogen	0,01 to 0,5
Oxygen	0,1 to 0,5
Nitrogen	0,1 to 40
Carbon dioxide	0,1 to 30
Methane	50 to 100
Ethane	0,1 to 15
Propane	0,001 to 5
Butanes	0,000 1 to 2
Pentanes	0,000 1 to 1
Hexanes to octanes	0,000 1 to 0,5

2 Normative references

The following normative documents contain provisions which, through reference in this text, constitute provisions of this part of ISO 6974. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this part of ISO 6974 are encouraged to investigate the possibility of applying the most recent editions of the normative documents indicated below. For undated references, the latest edition of the normative document referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

ISO 6974-1:2000, *Natural gas — Determination of composition with defined uncertainty by gas chromatography — Part 1: Guidelines for tailored analysis.*

ISO 6974-2:—[1], *Natural gas — Determination of composition with defined uncertainty by gas chromatography — Part 2: Measuring-system characteristics and statistics for data treatment.*

ISO 7504, *Gas analysis — Vocabulary.*

3 Principle

Determination of nitrogen, carbon dioxide and hydrocarbons from C_1 to C_8 by gas chromatography using two chromatographic columns. A molecular sieve 13X column coupled with a thermal conductivity detector (TCD) is used for the separation and detection of hydrogen, helium, oxygen and nitrogen, and a Porapak R column coupled with a TCD and a flame ionization detector (FID) in series is used for the separation and detection of nitrogen, carbon dioxide and hydrocarbons from C_1 to C_8. The two analyses are carried out independently and the results are combined.

If oxygen is seen to be present at a mole fraction greater than 0,02 % when measured using the molecular sieve column, then the nitrogen value shall be taken from the molecular sieve analysis. If the mole fraction of oxygen is less than 0,02 % and assuming that hydrogen is absent from the gas sample, the nitrogen value can be taken from the Porapak R analysis.

Quantitative results are achieved by determining the response of the TCD detector with reference-gas mixtures and using relative response factors of the FID detector.

The resulting composition of the natural gas is normalized to 100 %.

4 Materials

4.1 For the determination of helium, hydrogen, oxygen and nitrogen, (separation on molecular sieve 13X column) consisting of the following.

4.1.1 Argon carrier gas, > 99,99 % pure, free from oxygen and water.

If the purity of the gas is less than that specified, it is essential to check that the type of impurity present does not interfere with the analysis. Also, even if the carrier gases argon and/or helium fall within the specification, some of the impurities present in the gases can nevertheless interfere with the analysis. Under these circumstances, appropriate purification is essential.

4.1.2 Working-reference gas mixtures (WRM), consisting of:

4.1.2.1 Gas mixtures containing helium and hydrogen with nitrogen or argon as the matrix gas.

4.1.2.2 Gas mixtures containing oxygen and nitrogen with argon as the matrix gas.

NOTE 1 Take care to prevent explosion of gas mixtures.

NOTE 2 In the case of analysis using only one instrument, the WRM with oxygen and nitrogen as components and argon as the matrix gas can be replaced by oxygen with nitrogen as matrix gas. By addition of helium to the WRM this gas could also be used for the daily calibration.

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[1] To be published.

4.2 For the determination of nitrogen, carbon dioxide and hydrocarbons from C_1 to C_8 (separation on Porapak column), consisting of the following.

4.2.1 Helium carrier gas, > 99,99 % pure, free from oxygen and water.

4.2.2 Working-reference gas mixtures (WRM), consisting of multi-component gas mixtures containing: nitrogen, carbon dioxide and hydrocarbons from C_1 to C_3 (optional to C_4)

An example of the composition of the working-reference gas mixture is given in Table 2.

Table 2 — Example of the composition of the working-reference gas mixture

Component	Mole fraction %
Nitrogen	6
Methane	80,5
Carbon dioxide	9
Ethane	4
Propane	0,5
n-Butane	0,5 (optional)

4.2.3 FID gases, consisting of

a) **hydrogen**, > 99,99 % pure, free from corrosive gases and organic compounds;

b) **air**, free from hydrocarbon impurities.

5 Apparatus

5.1 Laboratory gas chromatographic (GC) system, consisting of two columns, a molecular sieve 13X column and a Porapak column, which are contained in two column-ovens or can be placed in the same column-oven.

The gas sample is injected on each column by means of a 6-way sample valve. Signal responses of components in the gas sample are detected using TCD and/or FID detectors.

NOTE The gas sample can be injected into the Porapak and molecular sieve column in series using a column isolation technique.

5.1.1 For the determination of helium, hydrogen, oxygen and nitrogen, equipped with the following specific components and characteristics.

5.1.1.1 Gas chromatograph, capable of temperature-programmed operation and equipped with a TCD and the following specific equipment:

a) **column oven and temperature controller,** consisting of:

— **column oven**, capable of maintaining the temperature of the column to within ± 0,5 °C over a temperature range from 35 °C to 350 °C;

NOTE 1 To obtain a temperature of 35 °C when testing at high ambient temperatures, a provision for cooling may be required, for example using an accessory for cooling with liquid carbon dioxide or liquid nitrogen.

NOTE 2 Alternative procedures for analysis on the molecular sieve 13X column are given in annex A.

- **temperature controller,** consisting of a linear programmer suitable for providing a rate of temperature increase of 30 °C/min over the specified range.

b) **flow regulator**, capable of maintaining suitable carrier-gas flow rates.

5.1.1.2 **Injection device,** consisting of a by-pass-type injector (gas-sampling valve) having an injection capacity of 1 ml and capable of being heated to a temperature setting of 110 °C.

The sample volume shall be reproducible such that successive runs agree within 1 % for each component.

5.1.1.3 **Columns,** two with the same type of packing and with the same dimensions.

The second column is normally used for drift compensation during the temperature programme. If drift is compensated by means of an electronic integrator, the second column is not necessary.

Columns shall satisfy the following requirements:

a) **metal tubing,** having the following characteristics:

- nature: stainless steel, number 20 (AISI type 316), cleaned and degreased
- length: 1 m
- diameter: 2 mm internal diameter (i.d.)
- form: appropriate for the chromatograph
- radius: appropriate for the chromatograph

NOTE If a column of 3 m is used, increase the column oven temperature to 40 °C (see annex A).

b) **packing, Molecular sieve 13X,** particle size from 150 µm to 180 µm (80 ASTM mesh to 100 ASTM mesh);

- method of packing: any suitable packing method providing uniform column packing;
- conditioning: overnight at approximately 350 °C under a flow of carefully dried carrier gas.

NOTE Some injection devices are unable to deal with temperatures above 250 °C and may cause conditioning problems.

5.1.1.4 **Thermal conductivity detector** (TCD).

5.1.2 **For the determination of nitrogen, carbon dioxide and hydrocarbons from C_1 to C_8,** equipped with the following specific components and characteristics.

5.1.2.1 **Gas chromatograph**, suitable for dual-column application and equipped in series with a TCD and an FID.

a) column oven and temperature controller, consisting of:

- **column oven**, capable of maintaining the temperature of the column to within ± 0,5 °C over a temperature range from 35 °C to 230 °C.

  NOTE To obtain a temperature of 35 °C an accessory for cooling with liquid carbon dioxide or liquid nitrogen may be necessary.

- **temperature controller**, consisting of a linear programmer suitable for providing a rate of temperature increase of 15 °C/min over the specified range.

b) **flow regulator,** capable of maintaining suitable carrier-gas flow rates.

5.1.2.2 **Injection device,** consisting of a by-pass-type injector (gas-sampling valve) having an injection capacity of 1 ml and capable of being heated to a temperature setting of 110 °C.

5.1.2.3 **Columns,** two of the same type of packing and with the same dimensions.

The second column is normally used for drift compensation during the temperature programme. If drift is compensated by means of an electronic integrator, the second column is not necessary.

a) **metal tubing,** having the following characteristics:

— nature: stainless steel, number 20 (AISI type 316), cleaned and degreased

— length: 3 m

— diameter: 2 mm i.d.

— form: appropriate for the chromatograph

— radius: appropriate for the chromatograph

b) **Porapak R packing,** particle size from 150 µm to 180 µm (80 ASTM mesh to 100 ASTM mesh);

— method of packing: any suitable packing method providing uniform column packing

— conditioning: overnight at approximately 230 °C under a flow of carefully dried carrier gas

5.1.2.4 **Detectors,** having the following characteristics:

— for components including hydrocarbons up to C_3: thermal conductivity detector (TCD)

— for hydrocarbons from C_4 to C_8: flame ionization detector (FID)

Ethane and propane can be detected by an FID if the mole fraction is less than 1 %. In either case, the time constant shall not be greater than 0,1 s. If C_3 is used as reference component, it shall be detected by an FID.

— the TCD and FID detectors shall be connected in series

NOTE If the mole fraction of oxygen is less than 0,02 %, the nitrogen value can be taken from the Porapak R analysis, assuming that hydrogen is not present in the gas sample.

6 Procedure

6.1 Gas chromatographic operating conditions

6.1.1 For the determination of helium, hydrogen, oxygen and nitrogen

Set the operating conditions for the apparatus (5.1.1) as follows.

a) Oven and column:

  — initial temperature: 35 °C for 7 min

  — temperature rate: 30 °C/min to 250 °C

- final temperature: maintain at 250 °C for 10 min

NOTE Alternative procedures for analysis on the molecular sieve 13X column are described in annex A. Variations in the programming may give a better separation.

b) Carrier gas flow rate: 10 ml/min of argon

c) Detector: TCD

- set according to manufacturer's instructions
- temperature: between 140 °C and 160 °C
- carrier gas: argon

6.1.2 For the determination of nitrogen, carbon dioxide and hydrocarbons from C_1 to C_8

6.1.2.1 GC conditions

Set the operating conditions for the apparatus (5.1.2) as follows:

a) Oven and column:

- initial temperature: 35°C for 3 min
- temperature rate: 15 °C/min to 200 °C
- final temperature: maintain at 200 °C for 30 min

b) Carrier gas flow rate: 35 ml/min of helium

c) Detector:

- set according to manufacturer's instructions
- FID
    i) temperature: between 290 °C and 310 °C
    ii) carrier gas: helium
- TCD
    i) temperature: between 240 °C and 260 °C
    ii) carrier gas: helium

6.1.2.2 Column stability check

Check the baseline stability of the column by means of blank runs.

No individual peak shall originate from a constituent having a mole fraction exceeding 0,04 %. If larger peaks are seen, repeat blank runs until satisfactory. If necessary, prepare new columns, preferably from a different batch of Porapak R.

NOTE 1 Different batches of Porapak R often show variation in performance. For example the retention sequence of benzene and cyclohexane may be reversed. It is therefore recommended that the retention time of benzene and cyclohexane be determined from time to time, and certainly after new columns have been installed.

NOTE 2 Baseline stability may be checked as follows:

a) raise the oven to the final temperature to clear any accumulated contamination;

b) cool to the initial temperature;

c) inject a calibration gas mixture containing a low mole fraction of butane and start the temperature programme;

d) at the end of the calibration gas run, cool to the initial temperature. Perform a blank run by injecting carrier gas in place of a sample and start the temperature programme;

e) calculate the mole fractions of the constituents of which the peaks are recognized by the integrator in the C_5 to C_8 region by comparison with butane in the calibration gas.

6.2 Performance requirements

6.2.1 Resolution efficiency

6.2.1.1 Molecular sieve 13X column

The height of the valley between the peaks above the baseline shall be no greater than 10 % of the height of the larger peak under the operating conditions following injection of a sample containing equivalent amounts (a mole fraction of about 0,4 %) of hydrogen and helium (see Table 3). If this criterion is not met, condition the packing for a longer period or prepare a new column.

Assess the peak resolution in accordance with ISO 7504.

Table 3 — Required peak resolution

Component 1	Component 2	Resolution
Hydrogen	Helium	0,1 (at least)

6.2.1.2 Porapak R column

The height of the valley between the 2-methylbutane and pentane peaks above the baseline shall be no greater than 10 % of the height of the larger peak under the operating conditions following injection of a sample. If this criterion is not met, condition the packing for a longer period or prepare a new column.

6.2.2 Response

Determine the response characteristics for each of the gases determined in accordance with ISO 6974-2 at least once a year.

6.2.3 Relative response factor

Determine the relative response factors in accordance with ISO 6974-2.

6.3 Determination

6.3.1 Outline of the analysis

The analysis is outlined as follows.

a) Analyse the working-reference gas mixture and the sample in accordance with clause 11 of ISO 6974-1:2000.

b) Measure the content of methane, ethane, propane, optional butane, nitrogen and carbon dioxide directly by means of determined response curves.

c) Measure the content of oxygen, hydrogen and helium directly using two certified-reference gas mixtures.

d) No groups shall be determined.

e) No backflush shall be performed.

f) Use the relative response to determine the content of higher hydrocarbons, i.e. from C_3 and upwards. Use propane as the reference compound (optional: butane).

Examples of typical chromatograms of this analysis are given for information in Figures A.1 and A.2 of annex A.

6.3.2 Estimation of other components

Estimate the fraction of other components in accordance with ISO 6974-1.

Backflushing shall not be carried out.

7 Expression of results

7.1 Calculation

7.1.1 Mole fractions

Refer to ISO 6974-1.

7.1.2 Correction for the presence of oxygen

Normally, natural gas does not contain oxygen. If, however, natural gas samples are found to contain oxygen and if this is due to contamination by improper sampling of the gas then the mole fraction of nitrogen and all other components shall be corrected according to the following procedure:

a) the sample contains more than 0,02 % of a mole fraction of oxygen. If the mole fraction of oxygen > 0,02 %, correct the mole fraction of nitrogen according to the equation:

$$x_{N_{2,c}} = x_{N_2} - \frac{78}{21} x_{O_2} \qquad (1)$$

where

$x_{N_{2,c}}$	is the mole fraction, expressed as a percentage, of nitrogen after correcting the mole fraction for air contamination;
x_{N_2}	is the mole fraction, expressed as a percentage, of nitrogen in the sample after normalization;
x_{O_2}	is the mole fraction, expressed as a percentage, of oxygen in the sample after normalization.

In equation (1), it is assumed that the TCD responses for N_2 and O_2 are equal.

b) the sample contains less than 0,02 % of a mole fraction of oxygen. If the mole fraction of oxygen < 0,02 %, correct the mole fraction of nitrogen according to equations (1) or (2).

   1) If nitrogen analysis has been carried out on a molecular sieve 13X column, equation (1) shall be applied.

   2) If nitrogen analysis has been carried out on a Porapak R column, apply the following equation:

$$x_{N_{2,c}} = x_{N_2} - \frac{100}{21} x_{O_2} \tag{2}$$

   In equation (2) it is assumed that the TCD responses for N_2 and O_2 are equal.

The mole fraction, $x_{j,c}$, expressed as a percentage, of component j in the sample corrected for the presence of oxygen is normalized to 100 % according to the equation:

$$x_{j,c} = \frac{x^*_{j,s}}{\sum_{j=1}^{n-2} x_{j,s} + x_{N_{2,c}}} \times 100 \tag{3}$$

where

$x_{j,s}$ is the normalized mole fraction, expressed as a percentage, of component j in the sample;

$x^*_{j,s}$ is the non-normalized mole fraction, of component j in the sample;

n is the total number of components;

n–2 is the total number of components except oxygen and nitrogen, determined separately on both the molecular sieve 13X and Porapak R column.

7.2 Precision and accuracy

Refer to ISO 6974-2.

See annex B for typical precision values.

8 Test report

Report the results in accordance with clause 14 of ISO 6974-1:2000.

Annex A
(informative)

Single-oven gas-chromatographic system consisting of two columns

Both analytical columns are placed in a single column oven and provided with a linear temperature programmer capable of obtaining a rate of temperature increase of 30 °C/min over the specified range.

A molecular sieve 13X column is used for the determination of helium, hydrogen, oxygen. The detection of these components is carried out by TCD. The gas sample is injected using a by-pass-type injector with an injection capacity of 1 ml. Flow regulators are used to give suitable argon gas-flow rates.

A Porapak R column is used for the determination of nitrogen, carbon dioxide, methane to normal octane. The detection is carried out by TCD in series with an FID. The gas sample is injected using a by-pass-type injector (gas sampling valve) with an injection capacity of 1 ml. Flow regulators are used to give suitable helium gas flow rates.

The configuration of such a chromatographic system is given in Table A.1.

Table A.1 — Configuration of the chromatographic system

Determination	Helium, hydrogen, oxygen	Nitrogen, carbon dioxide, methane to normal octane
Column		
Packing	Molecular sieve 13X	Porapak R
Length	3 m	3 m
Internal diameter	2 mm	2 mm
ASTM mesh size	80 to 100	80 to 100
Metal tubing	Stainless steel	Stainless steel
Carrier gas	Argon; 30 ml/min	Helium; 30 ml/min
Detector	TCD	TCD and FID
Injection device		
Capacity	1 ml	1 ml
Valve temperature	110 °C	110 °C
Temperature setting		
Initial temperature	40 °C	40 °C
Initial time	12 min	12 min
Temperature rate	15 °C/min	15 °C/min
Final temperature	200 °C	200 °C
Final time	30 min	30 min

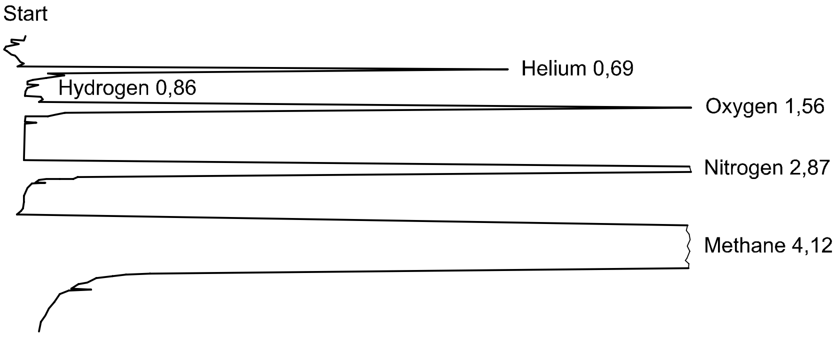

Figure A.1 — Typical chromatogram of helium, hydrogen, oxygen and nitrogen using a Molecular sieve 13X column (with indication of the absolute retention time in minutes)

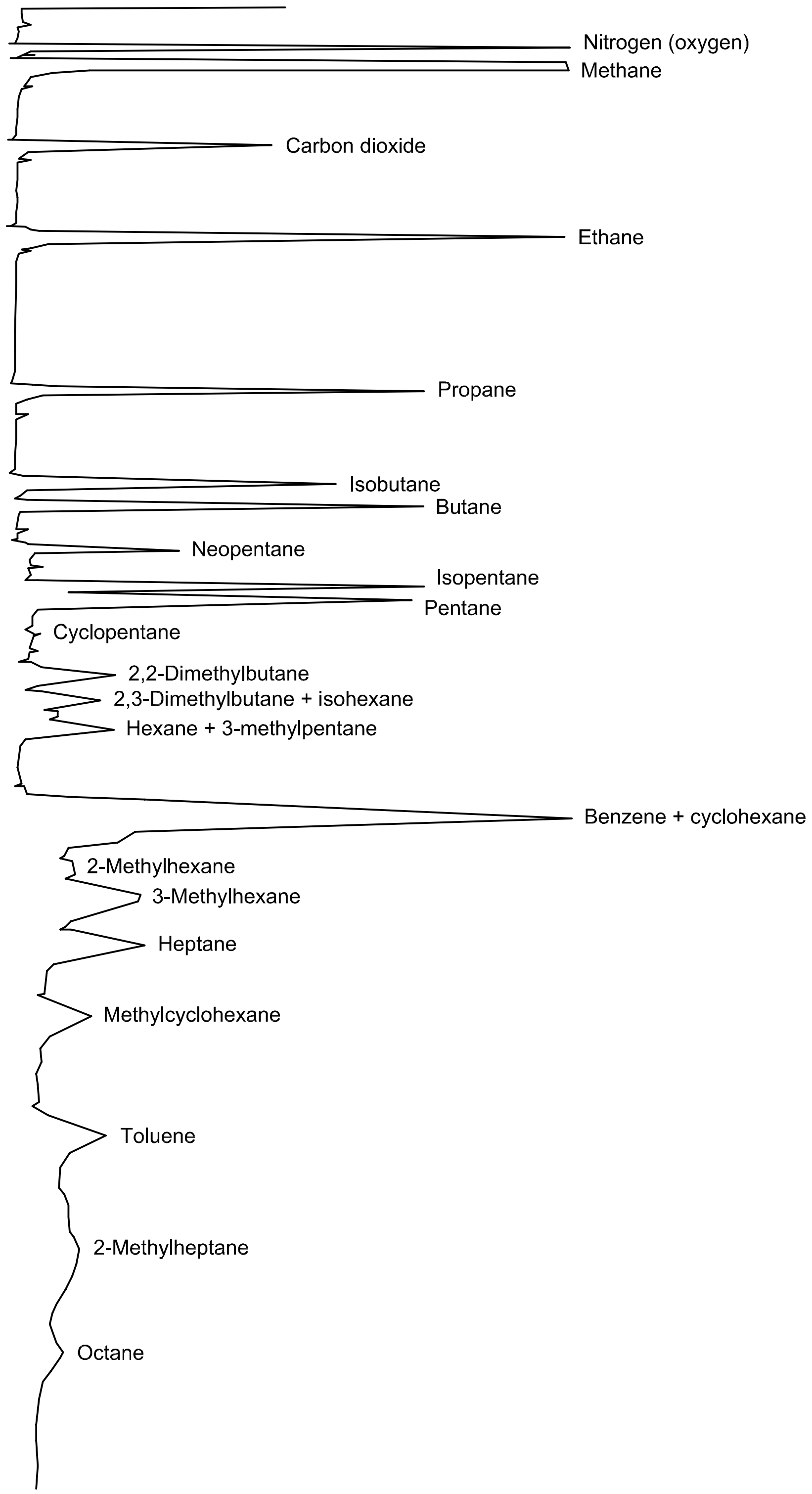

Figure A.2 — Typical chromatogram of nitrogen (oxygen), carbon dioxide and hydrocarbons from C_1 to C_8 using a Porapak R column

Annex B
(informative)

Typical precision values

Typical values for repeatability and reproducibility, which have been assessed in an interlaboratory test project of ISO/TC 158/SC 2 in October 1986 with participation of gas-producing, gas-supplying, gas-shipping companies and commercial laboratories from Belgium, Germany, Ireland, Norway, the Netherlands and the UK are given in Table B.1.

Table B.1 — Repeatability and reproducibility of measurement results

Mole fraction x %	**Repeatability**		**Reproducibility**	
	Absolute mole fraction (%)	Relative %	Absolute mole fraction (%)	Relative %
$x < 0,1$	0,003	—	0,006	—
$0,1 < x < 1$	—	3	—	6
$1 < x < 50$	—	1	—	3
$50 < x < 100$	—	0,1	—	0,2

NOTE These values have been obtained from practical experience and indicate the performance of the method. They cannot be compared as such with precision values mentioned in informative annexes of other parts of ISO 6974 because they result from the quality of the calibration gases and laboratory skills employed.

Bibliography

[1] ISO 6142, *Gas analysis — Preparation of calibration gas mixtures — Gravimetric method.*

[2] ISO 6143, *Gas analysis — Determination of the composition of calibration gas mixtures — Comparison methods.*

[3] ISO 6976, *Natural gas — Calculation of calorific values, density, relative density and Wobbe index from composition.*

[4] ISO 10723, *Natural gas — Performance evaluation for on-line analytical systems.*

[5] ISO 13275:—2), *Natural gas — Preparation of calibration gas mixtures — Gravimetric methods.*

[6] ISO 14111, *Natural gas — Guidelines to traceability in analysis.*

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2) To be published.

Annex ZA
(normative)

Normative references to international publications with their relevant European publications

This European Standard incorporates by dated or undated reference, provisions from other publications. These normative references are cited at the appropriate places in the text and the publications are listed hereafter. For dated references, subsequent amendments to or revisions of any of these publications apply to this European Standard only when incorporated in it by amendment or revision. For undated references the latest edition of the publication referred to applies (including amendments).

NOTE Where an International Publication has been modified by common modifications, indicated by (mod.), the relevant EN/HD applies.

Publication	Year	Title	EN	Year
ISO 6974-1	2000	Natural gas - Determination of composition with defined uncertainty by gas chromatography - Part 1: Guidelines for tailored analysis	EN ISO 6974-1	2001

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