

This document is a preview generated by EVS

**Natural gas - Determination of composition with defined uncertainty by gas chromatography - Part 5: Determination of nitrogen, carbon dioxide and C1 to C5 and C6+ hydrocarbons for a laboratory and on-line process application using three columns**

Natural gas - Determination of composition with defined uncertainty by gas chromatography - Part 5: Determination of nitrogen, carbon dioxide and C1 to C5 and C6+ hydrocarbons for a laboratory and on-line process application using three columns

## EESTI STANDARDI EESSÕNA

## NATIONAL FOREWORD

<p>Käesolev Eesti standard EVS-EN ISO 6974-5:2002 sisaldab Euroopa standardi EN ISO 6974-5:2001 ingliskeelset teksti.</p> <p>Käesolev dokument on jõustatud 14.02.2002 ja selle kohta on avaldatud teade Eesti standardiorganisatsiooni ametlikus väljaandes.</p> <p>Standard on kättesaadav Eesti standardiorganisatsioonist.</p>	<p>This Estonian standard EVS-EN ISO 6974-5:2002 consists of the English text of the European standard EN ISO 6974-5:2001.</p> <p>This document is endorsed on 14.02.2002 with the notification being published in the official publication of the Estonian national standardisation organisation.</p> <p>The standard is available from Estonian standardisation organisation.</p>
--	---

<p><b>Käsitlusala:</b> This part of EN ISO 6974 describes a gas chromatographic method for the quantitative determination of natural gas constituents using a three-column system. This method is applicable to natural gases of limited range, on-line and automatically calibrating on a regular basis for gas samples not containing any hydrocarbon condensate and/or water.</p>	<p><b>Scope:</b> This part of EN ISO 6974 describes a gas chromatographic method for the quantitative determination of natural gas constituents using a three-column system. This method is applicable to natural gases of limited range, on-line and automatically calibrating on a regular basis for gas samples not containing any hydrocarbon condensate and/or water.</p>
--	--

ICS 75.060

**Võtmesõnad:** chemical analysis and testin, column chromatogra, composition, determination, determination of content, gas analysis, gas chromatography, gas phase chromatography, hydrocarbons, information retrieval, natural gas, nitrogen content, on-line systems, uncertainties

English version

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

Part 5: Determination of nitrogen, carbon dioxide and C<sub>1</sub> to C<sub>5</sub> and C<sub>6+</sub> hydrocarbons for a laboratory and on-line process application using three columns (ISO 6974-5 : 2000)

Gaz naturel – Détermination de la composition avec une incertitude définie par chromatographie en phase gazeuse – Partie 5: Détermination de l'azote, du dioxyde de carbone et des hydrocarbures (C<sub>1</sub> à C<sub>5</sub> et C<sub>6+</sub>) pour l'application du processus en continu employant trois colonnes (ISO 6974-5 : 2000)

Erdgas – Bestimmung der Zusammensetzung mit definierter Unsicherheit durch Gaschromatographie – Teil 5: Bestimmung von Stickstoff, Kohlenstoffdioxid und C<sub>1</sub>- bis C<sub>5</sub>- und C<sub>6+</sub>-Kohlenwasserstoffen für eine Labor- und Online-Prozessanwendung mit drei Säulen (ISO 6974-5 : 2000)

This European Standard was approved by CEN on 2001-06-22.

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.

The European Standards exist 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, the Czech Republic, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, the Netherlands, Norway, Portugal, Spain, Sweden, Switzerland, and the United Kingdom.

**CEN**

European Committee for Standardization  
Comité Européen de Normalisation  
Europäisches Komitee für Normung

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

## Foreword

International Standard

ISO 6974-5 : 2000 Natural gas – Determination of composition with defined uncertainty by gas chromatography – Part 5: Determination of nitrogen, carbon dioxide and C<sub>1</sub> to C<sub>5</sub> and C<sub>6+</sub> hydrocarbons for a laboratory and on-line process application using three columns,

which was prepared by ISO/TC 193 'Natural gas' of the International Organization for Standardization, has been adopted by CEN/CMC as a European Standard.

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

In accordance with the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard:

Austria, Belgium, the Czech Republic, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, the Netherlands, Norway, Portugal, Spain, Sweden, Switzerland, and the United Kingdom.

## Endorsement notice

The text of the International Standard ISO 6974-5 : 2000 was approved by CEN as a European Standard without any modification.

## Contents

Page

Foreword.....	2
Introduction .....	3
1 Scope .....	3
2 Normative references .....	4
3 Principle.....	4
4 Materials .....	5
5 Apparatus .....	5
6 Procedure .....	6
6.1 Gas chromatographic operating conditions.....	6
6.2 Calibration .....	7
6.3 Performance requirements .....	8
6.3.1 Peak resolution .....	8
6.3.2 Chromatogram .....	8
6.4 Determination.....	8
6.4.1 Sample valve purge .....	8
6.4.2 Analysis .....	9
7 Expression of results .....	9
7.1 Precision and accuracy.....	9
8 Test report .....	9
Annex A (informative) Procedure for setting valve timings and restrictor setting .....	13
Annex B (informative) Typical precision values .....	15
Bibliography .....	15

## Introduction

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

This method is based on a automatic column-switching technique in which multiple columns, chosen for their separating ability for particular groups of components, are switched automatically.

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.

## 1 Scope

This part of ISO 6974 describes a gas chromatographic method for the quantitative determination of natural gas constituents using a three-column system. This method is applicable to natural gases of limited range, on-line and automatically calibrating on a regular basis for gas samples not containing any hydrocarbon condensate and/or water. It is applicable to the analysis of gases containing constituents within the mole fraction ranges given in Table 1. 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 if used in conjunction with parts 1 and 2 of ISO 6974.

**Table 1 — Application ranges**

Component	Mole fraction range %
Nitrogen	0,001 to 15,0
Carbon dioxide	0,001 to 8,5
Methane	75 to 100
Ethane	0,001 to 10,0
Propane	0,001 to 3,0
<i>iso</i> -Butane (2-methylpropane)	0,001 to 1,0
<i>n</i> -Butane	0,001 to 1,0
<i>neo</i> -Pentane (2,2-dimethylpropane)	0,001 to 0,5
<i>iso</i> -Pentane (2-methylbutane)	0,001 to 0,5
<i>n</i> -Pentane	0,001 to 0,5
Hexanes + sum of all C <sub>6</sub> and higher hydrocarbons	0,001 to 1,0

NOTE 1 Hydrocarbons higher than *n*-pentane are expressed as the "pseudo-component" C<sub>6+</sub> which is measured as one composite peak and calibrated as such. The properties of C<sub>6+</sub> are calculated from detailed analyses of the individual C<sub>6</sub> and higher hydrocarbons by extended analysis or from historical data.

NOTE 2 Oxygen is not a normal constituent of natural gas and would not be expected to be present in gas sampled for an on-line instrument. If any oxygen is present as a result of air contamination, it will be measured with the nitrogen. The resulting (nitrogen + oxygen) value will be in error to a small extent because of the slight difference between detector responses for

oxygen and nitrogen. Nonetheless, the result for the natural gas/air mixture will be reasonably accurate since neither component contributes to the calorific value.

NOTE 3 The content of helium and argon are assumed to be negligible and unvarying such that helium and argon need not be determined.

## 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 6142, *Gas analysis — Preparation of calibration gas mixtures — Gravimetric method.*

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

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

ISO 7504:1984, *Gas analysis — Vocabulary.*

## 3 Principle

Determination of nitrogen, carbon dioxide and hydrocarbons from C<sub>1</sub> to C<sub>6+</sub> by gas chromatography using a three-column switching/backflush arrangement, configured as shown in Figure 1. The three chromatographic columns are connected by two six-port valves for handling sample injection and backflushing operations (or alternatively a single ten-port valve is used) to a thermal conductivity detector (TCD) which is used for quantification.

A single sample is injected first onto a boiling-point separation column, divided into short and long sections. The C<sub>6</sub> and heavier hydrocarbons are initially retained on the short section of this column. The long section of this column retains C<sub>3</sub> to C<sub>5</sub> hydrocarbons. The lighter components (nitrogen, methane, carbon dioxide and ethane) pass rapidly and unresolved through the boiling-point separation column onto a porous polymer-bead column, suitable for their retention and separation. Following an accelerated backflush of the short column situated closest to the detector, the heavier C<sub>6+</sub> hydrocarbons (determined as a recombined "pseudo component" rather than by the summation of individual component measurements) elute first and are quantified as a single peak. Next, from the longer section of this column situated farther from the detector, the C<sub>3</sub> to C<sub>5</sub> hydrocarbons are separated then quantified by TCD. Finally, by redirecting carrier gas onto the porous polymer-bead column, the lighter components, i.e. nitrogen, carbon dioxide, methane and ethane, are separated then quantified by the detector. A six-port valve either connects this column to the carrier-gas flow or by-passes it during measurement of C<sub>3</sub> to C<sub>5</sub> components.

The separations that occur in the columns are as follows:

Column 1 Retains C<sub>6+</sub> components ready for backflushing as one composite peak.

Column 2 Separates propane, *iso*-butane, *n*-butane, *neo*-pentane, *iso*-pentane and *n*-pentane, (which elute after C<sub>6+</sub> has left column 1).

---

1) To be published.