This document

Natural gas - Determination of composition with defined uncertainty by gas chromatography - Part 4: Determination of nitrogen, carbon dioxyde and C1 to C5 and C6+ hydrocarbons for a laboratory and online measuring system using two columns

Natural gas - Determination of composition with defined uncertainty by gas chromatography - Part 4: Determination of nitrogen, carbon dioxyde and C1 to C5 and C6+ hydrocarbons for a laboratory and on-line measuring system using two columns



EESTI STANDARDI EESSÕNA

NATIONAL FOREWORD

Käesolev Eesti standard EVS-EN ISO 6974-4:2002 sisaldab Euroopa standardi EN ISO 6974-4:2001 ingliskeelset teksti.

This Estonian standard EVS-EN ISO 6974-4:2002 consists of the English text of the European standard EN ISO 6974-4:2001.

Käesolev dokument on jõustatud 14.02.2002 ja selle kohta on avaldatud teade Eesti standardiorganisatsiooni ametlikus väljaandes.

This document is endorsed on 14.02.2002 with the notification being published in the official publication of the Estonian national standardisation organisation.

Standard on kättesaadav Eesti standardiorganisatsioonist.

The standard is available from Estonian standardisation organisation.

Käsitlusala:

This part of EN ISO 6974 describes a gas chromatographic method for the quantitative determination of natural gas constituents using a two-column system. This method is applicable to determinations made in on-line processes or in the laboratory.

Scope:

This part of EN ISO 6974 describes a gas chromatographic method for the quantitative determination of natural gas constituents using a two-column system. This method is applicable to determinations made in on-line processes for in the laboratory.

ICS 75.060

Võtmesõnad: chemical, chemical analysis and testin, column chromatography, composition, determination, determination of content, gas analysis, gas chromatography, gas phase chromatography, hydrocarbons, measuring systems, natural gas, nitrogen content, online, uncertainties

EUROPEAN STANDARD NORME EUROPÉENNE EUROPÄISCHE NORM

EN ISO 6974-4

August 2001



English version

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

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 (ISO 6974-4 : 2000)

Gaz naturel – Détermination de la composition avec une incertitude définie par chromatographie en phase gazeuse – Partie 4: Détermination de l'azote, du dioxyde de carbone et des hydrocarbures (C₁ à C₅ et C₆₊) pour un laboratoire et un système de mesure en continu employant deux colonnes (ISO 6974-4: 2000)

Erdgas – Bestimmung der Zusammensetzung mit definierter Unsicherheit durch Gaschromatographie – Teil 4: Bestimmung von Stickstoff, Kohlenstoffdioxid und C_1 - bis C_5 - und C_{6+} -Kohlenwasserstoffen für ein Labor- und Online-Messsystem mit zwei Säulen (ISO 6974-4: 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

6 2 2

Foreword

International Standard

ISO 6974-4: 2000 Natural gas - Determination of composition with defined uncertainty by gas chromatography – Part 4: Determination of nitrogen, carbon dioxide and C₁ to C₅ and C₆₊ hydrocarbons for a laboratory and on-line measuring system using two 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 6974-4: 2000 was approved by CEN as a European Standard without any modification.

daro Contents Page

Forewo	ord	2
Introdu	uction	3
1	Scope	3
2	Normative references	4
3	Normative references Principle	4
4	Materials	
5	Apparatus	4
6	Apparatus	6
6.1	Gas chromatographic operating conditions	6
6.2	Performance requirements — Peak resolution	9
6.3	Determination — Outline of the analysis	. 10
7	Expression of results	. 10
7.1	Expression of results Calculation Precision and accuracy	. 10
7.2	Precision and accuracy	. 10
8	Test report	. 10
Annex	A (informative) Procedure for setting valve timings and restriction setting	
Annex	B (informative) Final time settings	. 11
Annex	C (informative) Typical precision values	. 12
	graphy	

Introduction

This part of ISO 6974 specifies 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 requires the use of two columns placed in series in a single-oven gas chromatograph.

The combination of two columns makes backflushing possible. All the constituents including the backflush peak are detected by a thermal conductivity detector (TCD).

This method is derived from ISO 6568, Natural gas — Simple analysis by gas chromatography.

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 two-column system. 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. 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 10
Methane	75 to 100
Ethane	0,001 to 10,0
Propane	0,001 to 3,0
iso-Butane (2-methylpropane)	0,001 to 1,0
<i>n</i> -Butane	0,001 to 1,0
neo-Pentane (2,2-dimethylpropane)	0,001 to 0,5
iso-Pentane (2-methylbutane)	0,001 to 0,5
n-Pentane	0,001 to 0,5
Hexanes + sum of all C ₆ and higher hydrocarbons	0,001 to 0,2

NOTE 1 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 2 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 6143, Gas analysis — Determination of the composition of calibration gas mixtures — Comparison methods.

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 7504:1984, Gas analysis — Vocabulary

3 Principle

Determination of nitrogen, carbon dioxide, methane, ethane, propane, butanes and pentanes by gas chromatography using two columns (a short one and a long one) of DC-200 on Chromosorb PAW in a backflush arrangement. The short column retains hydrocarbons heavier than normal pentane which are eluted after backflushing as a C_{6+} composite peak. The long column is used for the determination of nitrogen, carbon dioxide, methane up to normal pentane. Detection is carried out by a thermal conductivity detector (TCD). Oxygen, argon, hydrogen and helium are not measured by this method.

4 Materials

- **4.1 Helium carrier gas**, > 99,99 % pure.
- **4.2 Working-reference gas mixtures** (WRM), the composition of which shall be chosen to be similar to that of the anticipated sample.

Prepare a cylinder of a working-reference gas mixture, by a gravimetric method, in accordance with ISO 6142, and/or certified and validated in accordance with ISO 6143. The working-reference gas mixture shall contain at least nitrogen, carbon dioxide, methane, ethane, propane, *n*-butane, *iso*-butane and *possibly neo*-pentane, *iso*-pentane and *n*-pentane.

5 Apparatus

- **5.1 Laboratory gas chromatographic** (GC) **system**, consisting of the following components.
- **5.1.1** Gas chromatograph (GC), capable of isothermal operation and equipped as follows:

1) To be published.