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Analysis of natural gas — Biomethane — Determination of halogenated compounds —

Part 1: HCl and HF content by ion chromatography

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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.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT), see www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 193, *Natural gas*, Subcommittee SC 1, *Analysis of natural gas*.

A list of all parts in the ISO 2611 series can be found on the ISO website.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

ISO/DIS 2611-1:2023(E)**Introduction**

This document is part of a modular horizontal approach which includes a test method for total chlorine, fluorine and halogenated VOCs in biomethane.

For measuring hydrogen chloride (HCl) and hydrogen fluoride (HF) in biomethane, a method is described based on the absorption of these components on an alkali-impregnated quartz fiber filter. The anions chloride and fluoride are then analyzed by ion chromatography with conductimetric detection. The concentrations are expressed in equivalent hydrochloric acid and hydrofluoric acid at appropriate reference conditions.

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Analysis of natural gas — Biomethane — Determination of halogenated compounds —

Part 1: HCl and HF content by ion chromatography

1 Scope

This document specifies a method for the determination of the concentration hydrochloric acid (HCl) and hydrofluoric acid (HF) in biomethane, after absorption on an alkali-impregnated quartz fiber filter or in a sorbent trap, by ion chromatography (IC) with conductimetric detection.

The method is applicable to biomethane for concentration levels for HCl from 0,07 mg/m³ to 35 mg/m³; and for HF from 0,07 mg/m³ to 20 mg/m³.

Unless stated otherwise, all concentrations in this standard are given under standard reference conditions (see ISO 13443^[3]). Other conditions may be applied.

This method is also applicable to biogas. This method is intended to support conformity assessment of biomethane and biogas according to specifications, such as EN 16723^{[1][2]}.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 6974-1, *Natural gas — Determination of composition and associated uncertainty by gas chromatography — Part 1: General guidelines and calculation of composition*

ISO 6974-2, *Natural gas — Determination of composition and associated uncertainty by gas chromatography — Part 2: Uncertainty calculations*

ISO 6974-3, *Natural gas — Determination of composition and associated uncertainty by gas chromatography — Part 3: Precision and bias*

ISO 6976, *Natural gas — Calculation of calorific values, density, relative density and Wobbe indices from composition*

ISO 14532, *Natural gas — Vocabulary*

ISO 3696, *Water for analytical laboratory use — Specification and test methods*

ISO 10304-1, *Water quality — Determination of dissolved anions by liquid chromatography of ions — Part 1: Determination of bromide, chloride, fluoride, nitrate, nitrite, phosphate and sulfate*

3 Terms and definitions

For the purposes of this document, the terms and definitions in ISO 14532 and the following apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at <https://www.iso.org/obp>

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— IEC Electropedia: available at <https://www.electropedia.org/>

3.1

analyte

element, ion or substance to be determined by an analytical method

[SOURCE: EN 16687:2015, 4.1.11]

3.2

limit of quantification

LOQ

lowest analyte concentration that can be quantified with an acceptable level of precision and under the conditions of the test

[SOURCE: ISO 16140-1:2016(en), 2.36]

3.3

laboratory sample

sample intended for laboratory inspection or testing

[SOURCE: ISO 11074:2015, 4.3.7]

4 Symbols and abbreviations

4.1 Symbols

Symbol	Description	Unit
$\rho_{(x)}$	Concentration of gaseous hydrogen chloride or hydrogen fluoride in biethane	$\mu\text{g}/\text{m}^3$
$\rho_{(x-)}$	Concentration in ions chlorides or fluorides	$\mu\text{g}/\text{L}$
$M(x-)$	Molar mass of ions chlorides or fluorides	g/mol
$M(x)$	Molar mass of hydrogen chloride or hydrogen fluoride	g/mol
$m_{(x)}$	Mass of gaseous chlorides or fluorides collected	μg
V_s	Volume of extract solution	L
V_{gas}	Volume of the gas sampled	m^3
q_V	Volume flow rate of the gas during sampling	mL/min
p_0	Pressure at reference conditions	kPa
p_{gas}	Pressure at sampling conditions	kPa
t	Sampling time	min
T_0	Temperature at reference conditions	K
T_{gas}	Temperature at sampling conditions	K
Z	Compressibility factor	1

4.2 Abbreviations

CD	Coulometric detector
SI	International System of Units

5 Principle

Hydrochloric acid (HCl) and hydrofluoric acid (HF) contained in biomethane are trapped on an alkali-impregnated quartz fiber filter. The adsorbed inorganic halides are eluted by aqueous extraction with a sonification step.

NOTE Where “biomethane” is written, it is implied that it also covers biogas.

The instrumental analysis of chlorides and fluorides in the extracts is performed by ion chromatography with a conductimetric detector (CD).

When using CDs, it is essential that the eluents show a sufficiently low conductivity. For this reason, CDs are usually combined with a suppressor device (cation exchanger), which will reduce the conductivity of the eluent and transform the sample species into their respective acids.

6 Reagents and consumables

Use only reagents of recognized analytical grade. Weigh the reagents with a relative expanded uncertainty of $\pm 1\%$ ($k = 2$) of the nominal mass, unless stated otherwise.

6.1 Water

The water used in this method shall comply with grade 1 in accordance with ISO 3696.

6.2 Aqueous solutions

Sodium carbonate solution, Na_2CO_3 with a mass concentration of 50 g L^{-1} .

Sodium bicarbonate solution, NaHCO_3 with an amount-of-substance concentration of $0.0024 \text{ mol L}^{-1}$.

6.3 Chloride and fluoride stock standard solutions

The solutions shall have a mass concentration of $\rho_X = 1\,000 \text{ mg L}^{-1} \pm 10 \text{ mg L}^{-1}$ ($k = 2$) each.

Single anion and mixed anion stock solutions with adequate and required specification are commercially available. These solutions are stable for several months. Solutions used shall have certified concentrations with acceptable metrological traceability and a stated uncertainty.

6.4 Chloride and fluoride standard solutions

Depending on the concentrations expected, prepare single or mixed standard solutions of chloride and fluoride concentrations from the stock standard solution (6.4). Store the standard solutions in polyethylene bottles.

The equipment used (e.g., balances, volumetric glass ware) shall be calibrated or checked for performance. The calculation of the concentration(s) of the standard solution(s) shall include the evaluation of the measurement uncertainty associated with the concentration.

NOTE Guidance on the evaluation of measurement uncertainty is given in reference [4].