
**Analysis of natural gas — Silicon
content of biomethane —**

Part 2:

**Determination of siloxane content by
gas chromatography with ion mobility
spectrometry**

Analyse du gaz naturel — Teneur en silicium du biométhane —

*Partie 2: Détermination de la teneur en siloxanes par
chromatographie en phase gazeuse avec spectrométrie de mobilité
ionique*

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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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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*, in collaboration with the European Committee for Standardization (CEN) Technical Committee CEN/TC 408, *Natural gas and biomethane for use in transport and biomethane for injection in the natural gas grid*, in accordance with the Agreement on technical cooperation between ISO and CEN (Vienna Agreement).

A list of all parts in the ISO 2613 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.

Introduction

This document describes a method for the determination of the siloxane content of biomethane that is applicable to both laboratory and online analyses.

Siloxanes are a common contaminant in biogas, caused by their extensive use in common household products such as cosmetics and detergents. These products eventually become entrained in waste streams that are used to generate biogas. Additionally, siloxanes are sometimes added to digesters as anti-foaming agents to improve generation efficiency.

If not effectively removed, siloxanes can cause damage to gas processing and vehicle injection equipment through formation of silica deposits at high temperatures. Many gas distributors require measurement reports to demonstrate that the resultant biomethane conforms with their requirements, which typically includes siloxane content due to its potential to cause damage. Additionally, producers may wish to monitor siloxane content for process optimisation purposes.

The technique described in this document is commercially available and tailored specifically to measuring siloxanes within biomethane at low concentrations (for instance, as specified in EN 16723-1^[10] and EN 16723-2^[11]). It is suitable for use both in the laboratory and field.

Analysis of natural gas — Silicon content of biomethane —

Part 2: Determination of siloxane content by gas chromatography with ion mobility spectrometry

1 Scope

This document describes a gas chromatography – ion mobility spectroscopy (GC-IMS) method for the determination of the concentration of siloxanes in biomethane. The method is applicable to the following siloxanes:

- hexamethyldisiloxane (L2);
- octamethyltrisiloxane (L3);
- decamethyltetrasiloxane (L4);
- dodecamethylpentasiloxane (L5);
- hexamethylcyclotrisiloxane (D3);
- octamethylcyclotetrasiloxane (D4);
- decamethylcyclopentasiloxane (D5);
- dodecamethylcyclohexasiloxane (D6).

This document describes suitable calibration and measurement strategies to quantify siloxanes in (bio) methane around and above the $0,3 \text{ mg m}^{-3}$ ($14 \text{ } \mu\text{mol mol}^{-1}$) level and applies to analyses within absolute pressure ranges of 1 bar – 2 bar¹⁾, temperatures of 0 °C – 40 °C and relative humidity < 90 %.

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 6142-1, *Gas analysis — Preparation of calibration gas mixtures — Part 1: Gravimetric method for Class I mixtures*

ISO 6143, *Gas analysis — Comparison methods for determining and checking the composition of calibration gas mixtures*

ISO 6144, *Gas analysis — Preparation of calibration gas mixtures — Static volumetric method*

ISO 6145-1, *Gas analysis — Preparation of calibration gas mixtures using dynamic methods — Part 1: General aspects*

ISO 7504, *Gas analysis — Vocabulary*

ISO 10715, *Natural gas — Gas sampling*

1) 1 bar = 0,1 MPa = 10^5 Pa; 1 MPa = 1 N/mm².

ISO 10723, *Natural gas — Performance evaluation for analytical systems*

ISO 14532, *Natural gas — Vocabulary*

ISO 14912, *Gas analysis — Conversion of gas mixture composition data*

ISO/IEC Guide 98-3, *Uncertainty of measurement — Part 3: Guide to the expression of uncertainty in measurement (GUM:1995)*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO/IEC Guide 98-3, ISO 7504, ISO 14532 and the following apply.

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

- ISO Online browsing platform: available at <https://www.iso.org/obp>
- IEC Electropedia: available at <https://www.electropedia.org/>

3.1 ion mobility spectrometry IMS

analytical method used to separate and identify ionised molecules in the gas phase based on their mobility in a carrier gas under an electric field

4 Principle

A gaseous biomethane sample is drawn into a sample loop via a vacuum pump. The sample is injected onto a GC column, which separates the individual components in the gas mixture. As they exit the column, the components are ionised and passed into a drift tube. The drift tube applies a defined electric field, and a flow of inert nitrogen drift gas causes the ions to drift at different rates based on their mass and geometric structure onto a detector.

The instrument is calibrated by sampling an appropriate range of siloxane concentrations and using the detector output to generate a calibration function. The function can then be applied to unknown samples to calculate the concentration of siloxanes. This can then be summed to calculate total siloxanes and total silicon.

The method is applicable to siloxanes within the concentration ranges provided in [Table 1](#).

Table 1 — Application ranges

Component	Abbreviation	Formula	Lower limit (mg m ⁻³)	Upper limit (mg m ⁻³)
hexamethyldisiloxane	L2	C ₆ H ₁₈ OSi ₂	0,1	10,0
octamethyltrisiloxane	L3	C ₈ H ₂₄ O ₂ Si ₃	0,1	10,0
decamethyltetrasiloxane	L4	C ₁₀ H ₃₀ O ₃ Si ₄	0,1	10,0
dodecamethylpentasiloxane	L5	C ₁₂ H ₃₆ O ₄ Si ₅	0,1	10,0
hexamethylcyclotrisiloxane	D3	C ₆ H ₁₈ O ₃ Si ₃	0,1	10,0
octamethylcyclotetrasiloxane	D4	C ₈ H ₂₄ O ₄ Si ₄	0,1	10,0
decamethylcyclopentasiloxane	D5	C ₁₀ H ₃₀ O ₅ Si ₅	0,1	10,0
dodecamethylcyclohexasiloxane	D6	C ₁₂ H ₃₆ O ₆ Si ₆	0,3	10,0

NOTE Trimethylsilanol and trimethylsilane are outside of the scope of this document, but these silicon-containing compounds can also be found as impurities within biomethane.