SLOVENSKI STANDARD
oSIST prEN ISO 2620:2023

01-september-2023

Analiza zemeljskega plina - Biometan - Določevanje hlapnih ogljikovodikov (VOC) s plinsko kromatografijo s termično desorpcijo in s plamensko ionizacijskim in/ali masno selektivnim detektorjem (TD-GC-FID/MS) (ISO/DIS 2620:2023)

Analysis of natural gas - Biomethane - Determination of VOCs by thermal desorption gas chromatography with flame ionization and/or mass spectrometry detectors (ISO/DIS 2620:2023)

Analyse von Erdgas - Biomethan - Bestimmung von flüchtigen organischen Verbindungen durch thermische Desorptionsgaschromatographie mit Flammenionisations- und/oder Massenspektrometriedetektoren (ISO/DIS 2620:2023)

Analyse du gaz naturel - Biométhane - Détermination des COV par chromatographie en phase gazeuse à désorption thermique avec détecteurs à ionisation de flamme et/ou spectrométrie de masse (ISO/DIS 2620:2023)

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Analysis of natural gas — Biomethane — Determination of VOCs by thermal desorption gas chromatography with flame ionization and/or mass spectrometry detectors

Analyse du gaz naturel — Biométhane — Détermination des COVs par chromatographie en phase gazeuse à désorption thermique avec détecteurs à ionisation de flamme et/ou spectrométrie de masse

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ISO copyright office
CP 401 • Ch. de Blandonnet 8
CH-1214 Vernier, Geneva
Phone: +41 22 749 01 11
Email: copyright@iso.org
Website: www.iso.org

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Foreword

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

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 supports the implementation of specifications for biomethane and biogas when used in the natural gas grids and when used as a transport fuel. Implementation of these specifications require fit-for-purpose test methods with known performance and acceptable metrological traceability to support the trade in renewable gases and conformity assessment.

Depending on the production method, biogas usually contains volatile organic compounds (VOCs) such as terpenes, siloxanes, hydrocarbons, sulfur containing compounds, oxygenated hydrocarbons, halogenated hydrocarbons, ketones, alcohols, and esters. VOCs can also be found in the biomethane even after upgrading.

Analysis of natural gas — Biomethane — Determination of VOCs by thermal desorption gas chromatography with flame ionization and/or mass spectrometry detectors

1 Scope

This document describes a method for sampling and analysis of volatile organic compounds (VOCs), including siloxanes, terpenes, organic sulfur compounds, in natural gas and biomethane matrices, using thermal desorption gas chromatography with flame ionization and/or mass spectrometry detectors (TD-GC-FID/MS).

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 14532, *Natural gas — Vocabulary*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 14532 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/

4 Principle

A measured volume of sample gas is drawn through one sorbent tube where VOCs are retained while highly volatile organic compounds as methane matrix gas pass through. Desorption of the tubes is then carried out by thermal desorption (TD) in which the adsorbed substances are released with heat and then transferred into a gas chromatograph (GC) equipped with a capillary column and a mass spectrometer (MS). MS data of the separated VOC components are compared with a Mass Spectral Library for compound identification. The use of the specific ions in addition to the retention time ensure positive identification of a given VOC. Retention indices can also be used to identify peaks by comparing measured retention indices with tabulated values. Quantification is performed using either the FID and/or the MS detector. The expected quantification limit is 2-5 ng which is equivalent to 20-50 µg/m^3 in the sampled gas assuming a 100 ml gas sample volume.

5 Reagents and materials

5.1 Individual VOCs, for calibration purposes

For calibration purposes. Purity > 99 %.

5.2 Dilution solvent

Dilution solvent of chromatographic quality, for preparing blend solutions for liquid spiking shall be free from compounds co-eluting with the compound(s) of interest (e.g. in methanol or diethylether).

5.3 Sorbents

Sorbent preconditioned and kept in a clean atmosphere.

NOTE Example of sorbent: 2,6-diphenylphenylene oxide polymer.

Tubes compatible with the thermal desorption instrument to be used (6.8), typically containing 200 mg sorbent; shall be sealed with for example screw-cap fittings with polytetrafluoroethylene.

The conditioning temperature should not exceed the maximum rated temperature for the adsorbent resin. The desorption tube should preferably be conditioned 25 °C higher than the temperature at which it will be desorbed.

5.4 Micro-syringe, 5 or 10 µl liquid syringe readable to 0,1 µl.

5.5 Gas-tight syringe, 100 ml readable to 5 ml, for indirect sampling.

5.6 Tubings, silicon-free and sulfur-free tubings.

5.7 Volumetric flask, in inert material e.g. glass, quartz, polytetrafluoroethylene, 50 ml, class A.

5.8 Flow controller, volumetric, 50 to 500 ml/min readable to 2 ml/min.

6 Apparatus

6.1 Sampling system

Sampling system capable of accurately and precisely drawing natural gas or biomethane flow through the sorbent tubes.

6.2 Gas chromatography/mass spectrometer /flame ionization detector

Mass spectrometer in Electron Ionization mode (EI).

6.2.1 Capillary column for gas chromatography

NOTE Example of column: 95 % dimethylpolysiloxane, 5 % diphenyl, 60 m long, 0,32 mm inner diameter and 1 µm film thickness.

6.3 Thermal desorption instrument

Preferably two-stage thermal desorption in which the adsorbed substances are first released with heat and then transferred to a cooling trap for focusing. The cooling trap is reheated quickly and the substances are released and transported onto the gas chromatography column for separation.

7 Preparation

7.1 Field tube blanks

Determine levels of VOCs on unspiked, conditioned tubes.