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Analysis of natural gas - Biomethane - Determination of ammonia content by Tuneable Diode Laser Absorption Spectroscopy (ISO/DIS 2612:2023)

Analyse von Erdgas - Biomethan - Bestimmung von Ammoniakanteil durch Absorptionsspektroskopie mittels durchstimmbarer Laserdioden (ISO/DIS 2612:2023)

Analyse du gaz naturel - Biométhane - Détermination de la teneur en ammoniac par spectroscopie d'absorption laser à diode accordable (ISO/DIS 2612:2023)

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Analysis of natural gas — Biomethane — Determination of ammonia content by Tuneable Diode Laser Absorption Spectroscopy

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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.

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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

Ammonia is a common trace impurity found in biomethane. It is a product from the anaerobic digestion of biomass, formed from the breakdown of amino acids during the production of biogas. As an impurity in biogas and biomethane, ammonia is corrosive when it dissolves in the presence of water, damaging process equipment and leads to an increase in anti-knock processes in combustion engines when used as a fuel for vehicles. Ammonia is detrimental to the environment and as an air pollutant forms particulates which are damaging to public health. Additionally, when present in the combustion of biomethane, ammonia can lead to the formation of nitrogen oxides (NO_x), which are regulated pollutants as they are toxic and affect air quality. Therefore, the presence of ammonia in biogas and biomethane is undesirable to gas distributers and their customers.

Measuring ammonia content in mixtures of methane at the trace level (i.e. mg m^{-3}) is technically difficult due to the adsorptive nature (i.e. "stickiness") of ammonia. Particularly spectral NH_3 measurements can be severely hampered by spectral interferences from the matrix gas components, which further increases the complexity of these measurements. Measurements in biogas or biomethane are also dangerous due to the potentially explosive nature of methane, when mixed with an oxidizer like ambient air.

This method supports the implementation of specifications for biomethane and biogas such as EN 16723-1[1] and EN 16723-2[2] when used in the natural gas grids and when using it as a transportation fuel. Implementation of these specifications require fit-for-purpose measurement methods with known performance and acceptable metrological traceability to support the trade of renewable gases as well as conformity assessment. Currently, methods are referenced in standards such as EN 16723-1 which have not been validated for use with biomethane and biogas. This standard describes measurement methods that meet these requirements and can be implemented by laboratories and industry, also those seeking accreditation on the basis of, e.g., ISO/IEC 17025.

The methods described are based on commercially available spectroscopic analysers, specific to the measurement of ammonia. They have been shown to perform at an acceptable level when quantifying the ammonia content of biomethane at the 10 mg m^{-3} level, as specified in e.g. EN 16723-1.

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WARNING — The use of this document can involve hazardous materials, operations and equipment. This document does not aim to address all of the safety problems associated with the materials specified. It is the responsibility of the user of this document to establish appropriate safety and health practices and to determine the applicability of any other restrictions prior to use.

1 Scope

This document describes several test methods for measuring the ammonia amount fraction in natural gas and biomethane at the trace level (µmol mol^{-1}). The suitable handling and sampling of pressurised mixtures of ammonia in methane that are applied to several different ammonia measurement systems are described. The measurement systems comprise of readily available commercial spectroscopic analysers that are specific to ammonia. These NH_3 analysers are considered as a *black box* in terms of their operation, which is dependent on the instructions of the manufacturer. The document describes suitable calibration and measurement strategies to quantify ammonia in (bio)methane around and above the 10 mg m^{-3} (14 µmol mol^{-1}) level and applies to analysis within absolute pressure ranges of 1 bar – 2 bar, temperatures of 0 °C – 40 °C and relative humidity < 90 %.

References are also made to additional standards that are applied either to Natural Gas analysis or Air Quality measurements. In this document the matrix gas is always methane or biomethane and the measurand is the amount fraction NH_3.

NOTE 1 bar = 0,1 MPa = 10^5 Pa; 1 MPa = 1 N/mm^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 6143, *Gas analysis — Comparison methods for determining and checking the composition of calibration gas mixtures*

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

ISO 7504, *Gas analysis — Vocabulary*

ISO 9169, *Air quality — Definition and determination of performance characteristics of an automatic measuring system*

ISO 10715, *Natural gas — Gas sampling*

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

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

ISO 14532, *Natural gas — Vocabulary*

ISO 16664, *Gas analysis — Handling of calibration gases and gas mixtures — Guidelines*

IEC 61207-7, *Expression of performance of gas analyzers - Part 7: Tuneable semiconductor laser gas analyzers*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 7504, ISO 6143, ISO 10715, ISO 10723, ISO 14532, IEC 61207-7, ISO 9169 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
diode laser
semiconductor laser which is formed from a p-n junction and powered by injected electric current

[SOURCE: IEC 61207-7:2013, 3.2]

3.2
Ultraviolet-visible spectroscopy
UV-Vis
spectroscopy of radiation that consists of electromagnetic radiation with wavelengths in the ultraviolet and/or visible regions

[SOURCE: ISO/TS 80004-6:2021(en),[3] 5.6]

3.3
span gas
gas or gas mixture used to adjust and check the span point on the response line of the measuring system

Note 1 to entry: This amount fraction is often chosen around 70 % to 90 % of full scale.

[SOURCE: ISO 12039:2019,[4] 3.15]

3.4
span point
value of the output quantity (measured signal) of the automatic measuring system for the purpose of calibration, adjustment, etc. that represents a correct measured value generated by reference material

[SOURCE: ISO 13199:2012,[5] 3.14]

3.5
performance characteristic
one of the quantities assigned to the analytical instrument in order to define its performance

[SOURCE: ISO 13199:2012, 3.9]

3.6
response time
time interval between the instant when a stimulus is subjected to a specified abrupt change and the instant when the response reaches and remains within specified limits around its final stable value, determined as the sum of the lag time and the rise time in the rising mode, and the sum of the lag time and the fall time in the falling mode

[SOURCE: ISO 9169:2006, 2.2.4]