21NRM04 BiometCAP

D3 - New work item proposal (NWIP) (and draft text for a new ISO standard) describing the protocol for the performance evaluation of the gas analysers used for biomethane conformity assessment, submitted for consideration by ISO/TC193/SC1/WG25

Organisation name of the lead participant for the deliverable: NPL

Due date of the deliverable: March 2025

Actual submission date of the deliverable: 5th of August 2025

Confidentiality Status: PU - Public, fully open (remember to deposit public deliverables in a trusted repository)

Deliverable Cover Sheet

The project has received funding from the European Partnership on Metrology, co-financed from the European Union's Horizon Europe Research and Innovation Programme and by the Participating States.

The new work item proposal (NWIP) below (draft ISO standard text, describing the protocol for performance evaluation of the gas analysers that are used in biomethane conformity assessment) has been submitted to ISO/TC193/SC1 W25 Biomethane (secretariat and convenor) on the 24th of July 2025. WG25 confirmed the reception of two documents: the NWIP and the ISO Form 4. The NWIP contains annexes giving practical examples on how to use the performance assessment protocol for at least three different types of analytical methods and for a number of (at least 7) EN 16723 impurity groups also including terpenes and nitrogen.

These two documents have been combined here to form D3.

ISO #####-#:####(X)
ISO/TC 193/SC 1/WG 25
Date: 2025-MM-DD

Gas analysis - Biomethane - Protocol for the performance evaluation of gas analysers used for biomethane conformity assessment

WD stage

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Foreword

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This document was prepared by Technical Committee *[or Project Committee]* ISO/TC *[or ISO/PC]* ISO/TC 193, *[Natural gas]*, Subcommittee SC1, *[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

Biomethane is a sustainable alternative to fossil fuels used across Europe. Ensuring its quality is essential to protect natural gas infrastructure and end-user appliances. Harmful impurities must be kept below thresholds set by EN 16723. Various analytical techniques exist to analyse specific impurities in biomethane, and new methods are continually being developed. These techniques include gas chromatography, spectroscopy, and spectrometry-based principles. To ensure reliable and comparable quality measurements, it is essential to use equipment with verified performance, validated through traceable evaluation procedures. Performance evaluations shall be carried out after installation and thereafter periodically to ensure that the method is fit for purpose.

This document contains a validated protocol specifically designed as a tool for evaluating gas analysers in biomethane applications. The protocol applies to various techniques used for biomethane conformity assessment. Examples are given for analysers based on chromatography, spectroscopy, and spectrometry.

Gas analysis - Biomethane - Protocol for the performance evaluation of gas analysers used for biomethane conformity assessment

1 Scope

This document specifies a method for evaluating if an analytical system for biomethane composition analysis is fit for a defined purpose. It can be used:

a) to determine a range for each specified analyte over which the errors and uncertainties in measured composition do not exceed a predefined measurement requirement, using specified calibration gas(es);

b) to determine errors and uncertainties in a measured composition over a predefined range for each analyte, using specified calibration gas(es).

It is assumed that the analytical system is applied to compositions that vary over the typical ranges found within biomethane.

Performance evaluation of an analytical system is intended to be performed following initial installation to ensure that errors associated with assumed response functions are fit for purpose. Thereafter, periodic performance evaluation is recommended, or whenever any critical component of the analytical system is adjusted or replaced. The appropriate interval between periodic performance evaluations will depend upon both how instrument responses vary with time and how large an error may be tolerated. This first consideration is dependent upon instrument/operation; the second is dependent on the application. It is not appropriate, therefore, for this Standard to offer specific recommendations on intervals between performance evaluations.

Examples specific to gas chromatography, spectroscopy, and spectrometry are provided throughout the document.

2 Normative references

The following documents are referred to in the text in such a way that some or all 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 6145-7:2018 Gas analysis — Preparation of calibration gas mixtures using dynamic methods — Part 7: Thermal mass-flow controllers;

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 7504, Gas analysis — Vocabulary;

ISO 10715, Natural gas — Sampling guidelines;

ISO 10723, Natural gas - Performance evaluation for analytical systems;

ISO 14532, Natural gas — Vocabulary;

ISO/IEC guide 98-3:2008, 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 4224:2000, ISO 14532, IEC 61207-7:2013, ISO 19739:2004] and the following apply.

[redacted]

amount fraction

amount fraction, *x*, quotient of the amount of substance of a specified analyte and the sum of the amounts of substance of all components of a gas mixture

NOTE 1 to entry: The amount fraction is independent of the pressure and the temperature of the gas mixture.

[ISO 14912:2006, 2.1.1]

[redacted]

analysis function

relationship describing analyte content as a function of measurement system response

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/

[redacted]

amine

class of chemical compounds comprising nitrogen atom(s) bound to hydrogen and/or carbon atoms having the general formula R_3N

[SOURCE: ISO/TR 27912:2016, 3.5]

[redacted]

biogas

gas, comprising principally methane and carbon dioxide, obtained from the anaerobic digestion of biomass

[redacted]

biomass

biological material from living or recently living organisms, typically plants or plant-derived materials

[redacted]

biomethane

gas comprising principally methane, obtained either from upgrading biogas or methanation of bio-syngas

[redacted]

bio-syngas

gas comprising principally carbon monoxide and hydrogen, obtained from gasification of biomass

[redacted]

calibration function

relationship describing measurement system response as a function of analyte content

[redacted]

certified reference material (CRM)

reference material, characterized by a metrologically valid procedure for one or more specified properties, accompanied by a certificate that provides the value of the specified property, its associated uncertainty, and a statement of metrological traceability

[redacted]

calibration gas mixture (CGM)

gas mixture whose composition is sufficiently well established and stable to be used as a working measurement standard of composition

NOTE 1 to entry: a CGM is used for routine analyte calibration of the analyser. It is independent of the WMSs used to perform the evaluation.

[redacted]

gas chromatography

analytical method that is used to separate and determine the components of complex mixtures based on partitioning between a gas phase and a stationary phase.

[redacted]

mass concentration

concentration of a substance in a waste gas expressed as mass per volume

Note 1 to entry: Adapted from ISO 12039:2001, 3.10.

Note 2 to entry: Mass concentration is often expressed in milligrams per cubic metre (mg/m^3).

[redacted]

interference

negative or positive effect upon the response of the measuring system, due to a component of the sample that is not the measurand

[ISO 13199:2012, 3.4]

[redacted]

interferent

interfering substance

substance present in the air mass under investigation, other than the measurand, that affects the response

[ISO 9169:2006, 2.1.12]

[redacted]

measurand

particular quantity subject to measurement

[ISO/IEC Guide 98-3:2008, B.2.9]

[redacted]

performance characteristic

one of the quantities assigned to equipment in order to define its performance

[ISO 13199:2012, 3.9]

[redacted]

response

output signal of the measuring system for each specified analyte

NOTE 1 to entry: In the case of gas chromatography this will be either peak area or peak height, depending upon the measurement system configuration

[redacted]

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

[ISO 9169:2006, 2.2.4]

[redacted]

response function

functional relationship between measurement system response and analyte content

NOTE 1to entry: The response function can be expressed in two different ways as a calibration function or an analysis function, depending on the choice of the dependent and the independent variable.

NOTE 2 to entry: The response function is conceptual and cannot be determined exactly. It is determined approximately through calibration.

[redacted]

siloxane

class of chemical compounds comprising at least two silicon atom connected via an oxygen atom having the general formula $(R_2Si)_nO$, where $n > 1$.

[redacted]

terpene

products consisting mainly of terpenic hydrocarbons obtained as by-products of an essential oil by distillation, concentration, or other separation techniques.

[redacted]

upgrading of biogas

removal of carbon dioxide and contaminants from biogas

[redacted]

uncertainty of measurement

parameter, associated with the result of a measurement, that characterizes the dispersion of values that can reasonably be attributed to the measurand.

NOTE to entry: in keeping with ISO/IEC Guide 98-3, in this International Standard the uncertainty of the composition is expressed as a standard uncertainty or as an expanded uncertainty calculated through the use of an appropriate coverage factor.

[redacted]

working measurement standard

WMS

standard that is used routinely to calibrate or verify measuring instruments or measuring systems

[ISO/IEC Guide 99:2007, 5.7]

NOTE 1 to entry: working measurement standard is usually calibrated against a CRM.

4 Principles of analysis

Performance characteristics of the measurement system are determined when used in combination with a specified calibration gas mixture. Therefore, the evaluation procedure can be used to:

-determine the errors and uncertainties in the measured composition and properties over a predefined range for each specified analyte; and

-determine a range for each specified analyte over which the errors and uncertainties in measured composition and properties do not exceed a predefined measurement requirement.

In each case, the performance characteristics are calculated for the measurement systems when used in combination with a specified calibration gas of known composition and uncertainty.

NOTE 1: The method can also be used to establish the most appropriate composition of the calibration gas mixture to be used routinely with the measurement system such that the errors and uncertainties are minimised over a predefined range of use.

A complete assessment of the errors and uncertainties arising from the use of a measurement system could be performed by measuring an infinite series of well-defined reference gas mixtures whose compositions lie within the specified range of operation. However, this is practically impossible. Instead, the principle used in this International Standard is to measure a smaller number of well-defined reference gases and to determine a mathematical description of the response function for each specified analyte over a predefined range. The performance of the measurement system can then be modelled using these “true” response functions, the response functions assumed by the measurement system’s data system and the reference data for the calibration gas mixture specified for the measurement system. The measurement of many gas mixtures can then be simulated using numerical methods to determine performance benchmarks inherent in the measurement system.

The general procedure for determining the performance characteristics of the measurement system is summarised in Table 1.

Table 1 — Overview of performance evaluation process

Step	Phase	Description
1	Planning	Specify the analytes required to be measured by the measurement system and the measurement range for each over which the measurement system shall be evaluated. For each analyte, determine the extent of validation required.
2	Planning	Establish the functional descriptions of the response functions assumed by the measurement system for each specified analyte.
3	Planning	Specify a set of reference gas mixtures with compositions covering all ranges for all analytes specified in Step 1.
4	Planning	Establish the composition and uncertainty of the calibration gas mixture(s) to be used for routine calibration of the measurement system.
5	Experimental	Perform a multi-point calibration experiment by collecting measurement system response data for measurements of the reference gas mixtures specified in Step 4. The entire experiment should be conducted within a period equivalent to that between routine calibrations.
6	Calculation	Calculate the calibration functions and analysis functions for each specified analyte using regression analysis and validate the compatibility of the functions with the calibration data set.
7	Calculation	Calculate measurement system errors and uncertainties for each analyte over a specified range of compositions using the functions and reference data collated in Step 5 and 6.
8	Calculation	From the distribution of errors and the unbiased uncertainty estimates calculated in Step 7, determine the mean error and its uncertainty for each measurand.

NOTE 2: The response functions in Step 2 are referred to as the assumed response functions of the system at the time of calibration/evaluation. These are generally analysis functions used by the measurement system to determine the amount from the measured response, $x = G_{asm}(y)$.

NOTE 3: The response functions in Step 6 are referred to as the true response functions of the system at the time of calibration/evaluation, $y = F_{true}(x)$ and $x = G_{true}(y)$.

The mean errors and their uncertainties on analyte content and properties resulting from Step 8 can be compared to performance requirements for the analytical system. If performance benchmarks are poorer than the analytical requirements of the measurement, then the method fails to provide the desired performance over the fully specified range. The method shall be modified accordingly, and the entire evaluation procedure repeated. Alternatively, the offline calculations shall be repeated over a restricted range of operation to improve system performance. In this case, the measurement system may be shown to perform adequately over a limited range.

It may be possible to modify the data system on the measurement system to allow for the difference between the true response functions and the analysis function assumed by the measurement system. In this case, the measurement system should be adjusted following the evaluation to account for this difference. If the function form of G_{true} and G_{asm} are the same, then the parameters of G_{asm} in the measurement system data system can be updated with those determined for G_{true} in Step 6 above, thereby eliminating systematic errors due to the measurement system. However, it is important to remember that the parameters of G_{true} are only valid for each analyte over the content range used to establish the analysis function. That is, the measurement system should not be used outside the ranges defined and evaluated in Step 1, 2, and 3.

Performance evaluation procedure

Step 1: Specification of analytes, ranges and extent of validation

Users of this International Standard should first decide which analyte measured by the instrument are to be used in the evaluation of the performance. These are termed specified analytes. For each specified analyte, the range of amount fractions over which the response function is to be evaluated shall then be decided.

Typical impurity components that are required to be measured within biomethane include total silicon (as Si), siloxanes (L2, L3, L4, L5, D3, D4, D5, and D6), nitrogen, carbon monoxide, oxygen, hydrogen, ammonia, amines, total sulfur, water, compressor oil, dust, and chlorinated and fluorinated compounds.

The measurement ranges for these analytes shall be decided based on the application. Such ranges shall generally be greater than that which is expected to be measured by the instrument in regular duty. If the data from the performance evaluation is used subsequently to update the response functions assumed by the instrument, then it is vital that the analyte content ranges used in the evaluation extend beyond the specified operating range. Should this not be the case, considerable measurement errors might result from extrapolation outside the determined response function.

Example values for gas grid injection and vehicle fuel use are available in EN 16723-1 (EN16723-1:2016 Natural gas and biomethane for use in transport and biomethane for injection in the natural gas network, part 1: Specifications for biomethane for injection in the natural gas network, 2016) and EN 16723-2 (EN16723-2:2017 Natural gas and biomethane for use in transport and biomethane for injection in the natural gas network, part 2: Automotive fuels specification, 2017), respectively.

Table 2 — Example of biomethane components and ranges

Component	Example upper limit value	Reference	Example calibration range
Total volatile silicon (as Si). Including L2, L3, L4, L5, D3, D4, D5, and D6 Siloxanes	1 mgSi m^{-3}	EN 16723-1:2016	0.3 – 1.5 mgSi m^{-3}
Carbon monoxide	1000 µmol mol^{-1}	EN 16723-1:2016	500 - 5000 µmol mol^{-1}
Ammonia	10 mg m^{-3}	EN 16723-1:2016	5 - 20 mg m^{-3}
Amines	10 mg m^{-3}	EN 16723-1:2016	5 - 20 mg m^{-3}
Hydrogen sulfide + carbonyl sulfide (as sulfur)	5 mg m^{-3}	EN 16723-2:2017	1 - 10 mg m^{-3}
Total sulfur	30 mgS m^{-3}	EN 16723-2:2017	10 - 50 mgS m^{-3}
Methane	-	-	40 – 100 cmol mol^{-1}
Carbon dioxide	5 cmol mol^{-1}	EN 16723-2:2017	40 – 100 cmol mol^{-1}
Nitrogen	5 cmol mol^{-1}	EN 16723-2:2017	1 – 10 cmol mol^{-1}
Hydrogen	2 cmol mol^{-1}	EN 16723-2:2017	1 - 5 cmol mol^{-1}
Oxygen	1 cmol mol^{-1}	EN 16723-2:2017	0.5 - 5 cmol mol^{-1}
Water			
Compressor oil	de minimis	EN 16723-1:2016	
Dust impurities	de minimis	EN 16723-1:2016	
Chlorinated compounds	de minimis	EN 16723-1:2016	
Fluorinated compounds	de minimis	EN 16723-1:2016	

The extent of the performance characteristic validation is summarised in Table 3.

Table 3 — Extent of validation

Performance characteristic	Analytical application		
	Analyte identification	Analyte detection	Analyte quantification
Selectivity	x	x	x
Limit of detection		x	
Limit of quantification			x
Working range (linearity)			x
Trueness			x
Precision			x

Step 2: Response function specification

The measurement system data system will assume a relationship between the response and content of a analyte in the gas. This is the assumed analysis function of the measurement system, $x = G_{\mathrm{asm}}(y)$. Many measurement systems assume a simple first-order polynomial function in the form $x = b_1y$, where b_1 is often referred to as the response factor (RF) for that analyte. In this case, a single calibration gas mixture is used, and a first-order response function is assumed, passing through the origin. Alternatively, the measurement system may assume a higher-order polynomial functional description or even an exponential or power function.

In some cases, the response may be calculated relative to that of another (reference) analyte. Such a relative response factor shall have a response function similar to that of the reference analyte.

NOTE 1: Occasionally, functional types other than polynomials, such as exponential relationships, are implemented by a measurement system's data system. If the measurement system uses functional types other than polynomials, it is appropriate to use these in the determination of the analysis functions.

Step 3: Reference gas suite specification

Traceable reference gas standards (also known as calibration gases) are required to be used for the performance evaluation process. The amount fraction and standard uncertainty of the analyte shall be obtained or derived from the calibration certificate. If the uncertainty is not a standard uncertainty, it shall be divided by the manufacturer's stated coverage factor (*k*).

NOTE 1: The design of the calibration gas suite can have significant influence on the distribution of bias errors for the instrument. Similarly, the uncertainties on the amount fraction of each analyte in the calibration gas can make a significant contribution to the uncertainty on the measurement results. Hence, the design and uncertainty of composition of the calibration mixture shall be chosen carefully based on the application.

The mixtures may be multi-component mixtures or binary mixtures. The matrix gas shall be chosen to ensure it is suitable for the measurement purpose. For example, measurements of impurities within biomethane shall use calibration standards in a methane matrix gas, whereas measurements of biogases, shall have a mixed matrix gas containing all relevant primary components (methane, carbon dioxide, and nitrogen) to ensure experiments are representative.

Ensure the calibration gases have sufficient volume and pressure of gas and are within their defined stability period for the duration of the performance evaluation experiments.

The minimum number of calibration points recommended to give sufficient degrees of freedom for the unbiased estimate of the response function is as follows:

- 3 for a first-order polynomial;
- for a second-order polynomial; and
- for a third-order polynomial.

The calibration gas mixtures shall be selected such that their amount fractions are approximately equally spaced across the defined evaluation range (see 5.1), with one at or below the lower limit and one at or above the higher limit.

NOTE 2: Depending on the intended application, the lower end of the range might be close to the limit of detection, in which case it might not be possible to include a component amount fraction below the lower end of the application range.

Step 4: Routine calibration gas specification

Specify the composition and uncertainty of the calibration gas mixture(s) required to be used for routine calibrations. The composition and uncertainty of the mixture shall be designed to ensure that measurements are fit for their intended purpose.

Steps 5 – 8: Experimental performance evaluation and calculations

General note on experimental design

With appropriate planning, several performance characteristics can be evaluated from the same set of experiments. Selectivity is important to evaluate early in the process to provide information on the validity of subsequent parameter evaluations.

Experimental design shall consider the following:

- Reference gases to be measured;
- Experimental conditions (e.g. ambient temperature and pressure);
- Stabilisation time of the analytical equipment (including sampling equipment);
- The number of replicate measurements per test; and
- The number of repeat tests.

Data evaluation shall consider the following:

- Statistical parameters to be calculated (e.g., mean, standard deviation);
- How performance characteristics will be calculated; and
- How ‘fitness of purpose’ of the performance characteristic will be assessed.

Selectivity

Analytical selectivity is defined as “the ability of the method to determine particular analytes in mixtures or matrices without interferences from other components of similar behaviour”.

As the composition of biomethane can be highly variable, interferences may be present within certain measurement techniques and shall be evaluated as part of the validation process.

To perform the selectivity evaluation, use a reference material containing both the analyte of interest and other components that are expected to be present within the biomethane samples.

NOTE 1: the analyte and components are required to be present in the reference mixture at amounts above the expected limit of detection of the method and spanning the expected analytical working range.

Examples of common interferences observed in the analysis of biomethane are described in Table 4.

Table 4 — Examples of interferences within biomethane analysis

Technique	Examples of possible interferences
GC-FID	dimethyl-octadiene, alpha-pinene
GC-FID	beta-phellandrene, 3-carene, p-cymene
GC-FID	eucalyptol, d-limonene
GC-TCD	O_2, argon, Ar, H_2
FTIR	CH_4, CO_2, H_2O, HCl, NH_3, CO
NDIR	CH_4, CO_2, H_2O, HCl, NH_3
mGC-TCD	O_2, Ar, N_2, H_2

Example A: Gas chromatography (GC)

A chromatographic peak may be wrongfully identified as the analyte of interest if several analytes, including the analyte of interest, elute from the column at the same retention time (referred to as co-elution). Identification based on retention time alone is not sufficient without an assessment of the retention times of expected interferences. For example, if measuring siloxanes via GC, it should be noted that siloxanes, terpenes, BTEX (benzene, toluene, ethylbenzene and xylenes), and other VOCs may have similar retention times. Therefore, the method shall first be evaluated for selectivity by measuring reference standards containing those components using the same method.

An example for evaluating selectivity for gas chromatography techniques is as follows: after measuring a sample containing both the analyte of interest and an interferent, the selectivity can be calculated following Equation (1).

$$R = \frac{t_{R2} - t_{R1}}{0.5(W_2 + W_1)} \qquad (1)$$

Where:

R is the *resolution of the analyte;*

t_{R2}, t_{R1} *are the retentions times for the two analyte peaks; and*

W_2, W_1 *are the respective peak widths at baseline.*

A resolution of 1.5 or more occurs when the signal returns to its baseline between two peaks, indicating good separation.

Corrective measures for poor selectivity:

Repeating the work using an analytical column of increased length or different polarity;

Decreasing the over temperature, or adjusting the temperature program; or

Employ a detection method with high selectivity (e.g. mass spectrometry).

Example B: Spectrometry

The selectivity of atomic emission spectroscopy analysers for silicon refers to their ability to specifically detect and quantify silicon atoms among other elements present in a sample. These analysers achieve selectivity through the precise measurement of emitted light wavelengths characteristic of silicon atoms, allowing for accurate determination even in complex matrices.

Given that samples containing silicon derived from siloxane compounds typically exhibit low total dissolved solids (TDS) and a clean matrix devoid of spectral interfering elements, sensitivity served as the primary guiding principle for emission line selection. The chosen emission lines are as follows: 250.590 nm, 251.432 nm, 251.611 nm (the most sensitive line), 288.158 nm, and 390.552 nm (the least sensitive line). Prior to analysis, the viewing position in the plasma for each line and nebulizer pressure were optimized using sample solutions, accounting for any matrix effects.

AES analytical method for quantifying total silicon in biomethane involves two crucial steps: firstly, the concurrent design and optimization of measurement system detection and quantification methods suitable for analysing silicon in an acidic liquid medium; secondly, the development of a derivatization procedure to convert siloxane compounds into the analytical form of hexafluorosilicate ion.

Example C: Spectroscopy

FTIR/NDIR analysers are typically measurements in a broad spectral range where the absorption spectra a dominated by CH_4. Impurities absorption is in opposite quite weak compared to the CH_4 one. Measurement system producers typically include reference spectra databases for single gas components and some spectra modelling tools to perform data (spectra) analysis in case if several gas components are in a spectral overlap, Therefore, it is recommended to perform a periodic analyser calibration check with:

1) single reference calibration gas such as CH_4 (99.998%);

2) with at least one critical impurity, e.g. NH_3 (50ppm) in N_2; and

3) a mixture with known concentrations, e.g. CH_4 (99%) + NH_3 (50 ppm) + N_2 (balance).

The same is applicable for any GC-based systems.

Limit of Detection and Limit of Quantification

The Limit of Detection is defined as “the lowest content of analyte that can be detected by the method at a specified level of confidence”. Similarly, the Limit of Quantification is the lowest content of analyte that can be quantified by the method at a specified level of confidence.

The LOD and LOQ are calculated in one of two ways: via replicate measurements of blank samples, or via replicate measurements of test samples with a suitably low amount fraction of the analyte. LOD and LOQ are calculated by multiplying the standard deviation (s_0') by suitable factors (typically 3 for LOD and 10 for LOQ). To obtain an adequate estimation of s_0', 10 replicate measurements are recommended.

If results do not require blank correction during routine method usage, then Equation (2) should be used.

$$s_0' = \frac{s_0}{\sqrt{n}} \tag{2}$$

If results do require blank correction during routine method usage, then Equation (3) should be used.

$$s_0' = s_0\sqrt{\frac{1}{n} + \frac{1}{n_b}} \tag{3}$$

Where:

s_0 = *the estimated standard deviation of a number of single results at or near zero amount fraction;*

s_0' = *the standard deviation used for calculating LOD and LOQ;*

n = *the number of replicate observations averaged when reporting results where each replicate is obtained following the measurement procedure; and*

n_b = *the number of blank observations used to calculate the blank correction.*

The LOD and LOQ can then be calculated according to Equations (4) and (5) respectively.

$$\text{LOD} = 3 \times s_0' \tag{4}$$

$$\text{LOQ} = 10 \times s_0' \tag{5}$$

For measurements of biomethane, high purity methane can be used for the blanks. It is important to ensure that the blank does not contain any analytes or interferences at or above the expected LOD.

Example A: Gas chromatography (GC)

Chromatography relies on a peak being detected above the noise level, therefore a reference standard containing the analyte at an amount fraction close to the expected LOD is required. It is important that the sampling of the reference standard follows the same procedure as that of the analysis method to be assessed.

Example B: Spectrometry

<>

Example C: Spectroscopy

In laser spectroscopy typically the Allan-Werle deviation of a measurement at a low, stable concentration is determined to derive the optimal integration time (i.e., number of averages) and the corresponding limit of detection [3]. LoDs at other integration times can also be derived from the Allan-Werle plot. Note that the composition of the matrix may affect the LoD.

Working range

Working range is defined as “The interval over which the method provides results with an acceptable uncertainty”.

IUPAC distinguishes the linear range (concentration range over which the intensity of the signal obtained has a linear relationship with concentration of the analyte) and the dynamic range (the ratio between the maximum usable indication and the minimum usable indication). In the dynamic range, the response may be non-linear, especially at higher concentrations.

To assess the measurement system working range:

- identify the range of interest (recommended to span ± 10% of the expected calibration range) and measure blanks and calibration standards at 6 - 10 amount fractions spread evenly across this range; and
- plot response against amount fraction and visually examine plot to identify the approximate linear range.

To quantify linearity over the identified linear range, measure blanks and calibration standards at 6 - 10 amount fractions spread evenly across the linear range.

Plot response against amount fraction and calculate appropriate regression statistics. Plot the residuals and inspect their distribution; random distribution of the residuals around zero confirms linearity.

NOTE 1: If the standard deviation is proportional to the concentration, then a weighted regression calculation may be more appropriate than a non-weighted linear regression. Outliers shall not be removed without justification using further measurements at nearby amount fractions. In certain circumstances for measurement system calibration, it may be better to try to fit a non-linear curve to the data. The number of samples then needs to be increased. Functions higher than quadratic are generally not advised.

Finally, to assess if the chosen measurement system ranges and calibration procedures are fit-for-purpose, calibrate the measurement system according to the routine calibration procedure. Measure the blank and reference materials 2-3 times each at 6 - 10 amount fractions, spread across the range of interest.

NOTE 2: Assessment of the working range shall be supported by precision and bias studies from amount fractions covering the whole working range.

NOTE 3: Each matrix requires a separate working range assessment due to potential biases due to interferences that can cause non-linear responses for analytes, and effects that may be caused on analyte recovery.

Example A: Gas chromatography

The upper boundary of the working range in GC may be caused by saturation of certain detectors, which may be observable for example, as a poor-quality peak shape

Example B: Spectrometry

Atomic emission spectrometry: The upper boundary limit for the working range of atomic emission spectroscopy (AES) depends on several factors, including the instrument's design, the specific analytical conditions, and the properties of the sample being analyzed. However, there are some general considerations to define the upper limit of the working range:

Dynamic Range of the Detector: The upper limit of the working range is often determined by the dynamic range of the detector used in the AES instrument. Detectors have a maximum signal intensity they can accurately measure without saturation. Beyond this point, the detector response becomes non-linear.

Spectral Interference: At high analyte concentrations, spectral interferences may occur due to overlapping emission lines from other elements or molecular bands. This interference can affect the accuracy and precision of the analysis.

Matrix Effects: High analyte concentrations or complex sample matrices can lead to matrix effects, where the sample matrix interferes with the atomization and excitation processes, affecting the intensity of the emission signal.

Sensitivity and Signal-to-Noise Ratio (SNR): As analyte concentrations increase, the signal-to-noise ratio may decrease, making it challenging to accurately measure low-intensity emission lines.

Instrument Calibration: The upper limit of the working range may also be influenced by the calibration range of the instrument. Calibrating the instrument over a wide concentration range ensures accurate measurements within that range.

Sample Dilution: For samples with analyte concentrations above the upper limit of the working range, dilution may be necessary to bring the concentrations within the instrument's linear range.

Saturation Effects: At very high analyte concentrations, the emission signal may saturate the detector, leading to inaccurate measurements. Dilution or sample preparation techniques may be required to avoid saturation effects.

Manufacturer Specifications: The instrument's specifications provided by the manufacturer often indicate the upper limit of the working range based on the instrument's design and performance characteristics.

Example C: Spectroscopy

An example of what would cause the upper boundary of the working range in UV/VIS spectroscopy are effects encountered due to plateauing at high absorbance values.

Trueness (bias)

Trueness is defined as “the closeness of agreement between the average of an infinite number of replicates measured quantity values and a reference quantity value”.

As it is not possible to take an infinite number of measurements, trueness cannot be measured. A practical assessment of the trueness can however be made. This assessment is normally expressed quantitatively in terms of ‘bias’. The bias can be estimated using several approaches.

Approach 1: evaluation against a reference material.

Compare mean measured value, $\bar{x}$, with the reference value, x_{ref},for the reference material. Calculate bias, b, percent relative bias, $b(\%)$ or the relative percent recovery (apparent recovery) $R(\%)$, according to Equations (6)-(8).

$$b = \bar{x} - x_{ref} \tag{6}$$

$$b(\%) = \frac{\bar{x} - x_{ref}}{x_{ref}} \times 100 \tag{7}$$

$$R(\%) = \frac{\bar{x}}{x_{ref}} \times 100 \tag{8}$$

Approach 2: Evaluation using sample spiking.

Compare the difference between mean spiked value $\bar{x}'$ and mean value $\bar{x}$ with the added concentration x_{spike}. Calculate the relative spike recovery $R'(\%)$ at each concentration according to Equation (9).

$$R'(\%) = \frac{\bar{x}' - \bar{x}}{x_{spike}} \times 100 \tag{9}$$

Approach 3: Evaluation against an alternative validated method.

Compare mean measured value, $\bar{x}$ with the reference value $x_{refmethod}$ of measurements made using an alternative validated reference method. Calculate bias, b, per cent relative bias, $b(\%)$ or the relative per cent recovery (apparent recovery) $R(\%)$ according to Equations (10)-(12).

$$b = \bar{x} - x_{refmethod} \tag{10}$$

$$b(\%) = \frac{\bar{x} - x_{refmethod}}{x_{refmethod}} \times 100 \tag{11}$$

$$R(\%) = \frac{\bar{x}}{x_{refmethod}} \times 100 \tag{12}$$

Precision (repeatability, intermediate precision, and reproducibility)

Precision is defined as “the closeness of agreement between measured quantity values obtained by replicate measurements under specified conditions” and can be evaluated in terms of the repeatability, intermediate precision, and reproducibility:

- repeatability (S_r): the variability in results when measurements are performed in a single laboratory over a short timescale;
- intermediate precision/within-lab reproducibility (S_{RW}): the variability in results when measurements are made in a single laboratory under conditions that are more variable than repeatability conditions (on different days); and
- reproducibility/between-lab reproducibility (S_R): the variability in results when measurements are made in different laboratories.

To evaluate repeatability and intermediate precision, measure the analyte(s) of interest under repeatability conditions, collecting at least 6 repeat results on at least 3 different days. After grouping the data by day, perform a one-way Analysis of Variance (ANOVA) calculation on the groups of data. The calculation can be performed by hand, by constructing an ANOVA table as shown in Table 5, or using a software package such as Microsoft Excel.

Table 5 — ANOVA Table

Source of Variation	Sum of Squares	Degrees of Freedom	Mean Squares	F
Between group	$SS_b = \sum_{i=1}^{k} n\left(\bar{X}_i - \bar{X}\right)^2$	$DF_b = k - 1$	$MS_b = \frac{SS_b}{DF_b}$	$F = \frac{MS_b}{MS_w}$

Within group	$SS_w = \sum_{i=1}^{k}\sum_{j=1}^{n}\left(X_{ij} - \overline{X}_i\right)^2$	$DF_w = nk - k$	$MS_w = \frac{SS_w}{DF_w}$	
Total	$SS_t = \sum_{i=1}^{k}\sum_{j=1}^{n}\left(\overline{X}_{ij} - \overline{X}\right)^2$	$DF_t = nk - 1$		

Where:

$\overline{X}_{ij}$	*is the mean measurement result for measurement “j” of group “i”;*
$\overline{X}_i$	*is the mean measurement result for group “i";*
$\overline{X}$	*is the mean measurement result for all data;*
n	*is the number of measurements in a group;*
k	*is the number of groups;*
SS	*is the sum of squares;*
MS	*is the mean of squares;*
DF	*is the degrees of freedom; and*
F	*is the F distribution.*

Once the ANOVA table is populated by results from a single laboratory on different days under repeatability conditions, then the repeatability and intermediate precision can be calculated using Equations (13) – (15).

$$S_r = \sqrt{MS_w} \tag{13}$$

Where S_r is the absolute repeatability. Relative repeatability can be calculated by dividing S_r by the mean measurement value, $\overline{X}$, and multiplying by 100%.

$$S_{between} = \sqrt{\frac{MS_b - MS_w}{n}} \tag{14}$$

Where $S_{between}$ is the absolute contribution to the total variation from the grouping factor. The intermediate precision can then be calculated as the square root of the sum of squares of the repeatability and the between group variation.

$$S_I = \sqrt{s_r^2 + s_{between}^2} \tag{15}$$

Where S_I is the absolute intermediate precision. Relative intermediate precision can be calculated by diving S_I by the mean measurement value, $\overline{X}$, and multiplying by 100%.

Similarly, the reproducibility can be evaluated by carrying out the intermediate precision analysis using data from separate laboratories.

Example A: Gas chromatography

Repeatability can be evaluated by performing repeat injections of a reference gas mixture using a defined method. The area of the chromatograms are integrated (either automatically as specified within the measurement system software, or it may be possible to manually integrate the analyte peaks). The standard deviation of the peak area of the analyte is used to determine repeatability.

Intermediate precision is evaluated by repeating the repeatability test over a period in which laboratory or field conditions are expected to vary by routine amounts (e.g. different temperature, pressure and humidity conditions).

Example B: Spectrometry

Example C: Spectroscopy

Measurement uncertainty

Expanded measurement uncertainty quantifies an interval around a measurement result within which the true value of the measurand is likely to be found within a stated degree of confidence. It offers a quantitative measure of the reliability of a measurement result. Several methods exist to estimate the uncertainty from the results, importantly the approach chosen shall consider [ref]:

- the long-term precision of the method (*i.e.,* reproducibility); and
- the bias and its uncertainty, including the statistical uncertainty involved in the bias measurements, and the uncertainty in the reference value.

The uncertainty in the bias, u_{bias}, can be estimated according to Equations (16)-(18).

$$bias = \bar{x} - x_{refmethod} \tag{16}$$

$$RMS_{bias}^2 = \sqrt{\frac{\sum(bias_i)^2}{n_{CRM}}} \tag{17}$$

$$u_{bias} = \sqrt{RMS_{bias}^2 + u(c_{ref})^2} \tag{18}$$

Where:

$u(c_{ref})$ *= the standard uncertainty of certified concentration; and*

n_{CRM} *= the number of CRMs used.*

If only one CRM is used, then the standard deviation of measured concentration, S_{bias}, shall be considered according to Equation (19).

$$u_{bias} = \sqrt{bias^2 + (\frac{S_{bias}}{\sqrt{n}})^2 + u(c_{ref})^2} \tag{19}$$

Where:

n *= the number of measurements; and*

$bias$ *= is calculated according to Equation 20.*

$$bias = \frac{c_{-}c_{ref}}{c_{ref}} x\ 100\% \tag{20}$$

Where:

c_{ref} *= the certified concentration; and*

c_i *= the measured concentration.*

The combined standard uncertainty, u_c, can be calculated according to Equation (21).

$$u_c = \sqrt{u_r^2 + u_{day}^2 + \Delta_m^2} \tag{21}$$

Where:

u_r = *is the relative uncertainty due to repeatability (see 5.11);*

u_{day} = *is the relative uncertainty due to reproducibility (see 5.11); and*

Δ_m = *is the relative bias of the analytical method (see 5.10).*

The expanded measurement uncertainty is then given by Equation (22).

$$U = k \cdot u_c \tag{22}$$

where k is the coverage factor.

A coverage factor of two corresponds to a confidence level of approximately 95%.

Recommended target measurement uncertainties are 1 to 10 % as proposed by the BiometCAP project. This requires the use of reference materials with high accuracy.

Interpretation of results

Evaluation report and documentation

Proper documentation is required for auditing and evaluation purposes. National accreditation bodies may give specific requirements for this documentation. The validation report shall contain the following sections:

Table 6 — Evaluation Report

Section	Description
Method title	“The determination of A in the presence of B in C using D” Where: A is the analyte or measurand, B are the interference(s) tested, C is the sample matrix, D is the measurement technique.
Scope	Description of the measurement method principle, method purpose and the parameters evaluated for the performance evaluation
Normative references	As appropriate to the evaluation
Definitions	Use ISO definitions where possible
Performance requirements	Statement of maximum possible error and maximum possible bias
Methodology	Description of the performance evaluation, including reference materials, equipment and sampling method.
Results	Results of the performance evaluation, including a statement on uncertainty.

Annex A

Example validation plans and reports

Table A.1 — Validation Plan: example for bulk composition

<table>
<tr><th>Method title</th><td colspan="4">The determination of hydrogen, carbon monoxide, oxygen and nitrogen in a matrix of methane using gas chromatography with thermal conductivity detection</td></tr>
<tr><th>Normative references</th><td colspan="4">ISO 6974-1:2017 Determination of composition with defined uncertainty by gas chromatography.</td></tr>
<tr><th>Scope</th><td colspan="4">Bulk composition (H_2, CO, O_2, N_2) are separated from CH_4 on two capillary columns, with helium and argon carrier gasses. The individual components are detected and quantified using TCD detectors.</td></tr>
<tr><th>Analytes</th><td colspan="4">N_2, CO, O_2, H_2.</td></tr>
<tr><th>Range (µmol mol^{-1})</th><td colspan="4">H_2: 100 - 100 000; CO: 1000;
O_2: 10 - 10 000; N_2: 100 - 100 000.</td></tr>
<tr><th>Matrix</th><td colspan="4">Gas (biomethane).</td></tr>
<tr><th>Reference materials</th><td colspan="4">A reference mixture produced by a national measurement institute in a methane matrix gas, (cylinder ID: D133074) containing 94213.25 ± 24.63 µmol/mol of N2, 962.81 ± 0.32 µmol/mol of CO; 1922.80 ± 1.32 µmol/mol of O2; and 104060.89 ± 192.36 H2 was dynamically diluted with N5.0 grade methane to generate the calibration curve.
The curve was verified by calibrating a second reference mixture (cylinder ID: D113080) containing 19973.57 ± 15.97 µmol/mol of N2, 1009.28 ± 0.26 µmol/mol of CO; 406.68 ± 0.43 µmol/mol of O2; and 20160.2 ± 25.24 H2.</td></tr>
<tr><th>Sampling</th><td colspan="4">Regulator, passivated stainless steel tubing.</td></tr>
<tr><th>Metrological traceability</th><td colspan="4">Calibration curves were generated by diluting traceable reference standards prepared and validated by a national measurement institute.</td></tr>
<tr><th>Performance requirements</th><td colspan="4">The method shall be optimised to prevent bias from ambient nitrogen and oxygen. Bias target value was set to 5 %, precision of 2.5 % and an expanded measurement uncertainty of 10%.</td></tr>
<tr><th rowspan="4">Extent of validation</th><td>Precision: Repetability</td><td>☒</td><td>Selectivity</td><td>☒</td></tr>
<tr><td>Precision: Intermediate precision</td><td>☒</td><td>Working range and linearity</td><td>☒</td></tr>
<tr><td>Precision: Reproduceability</td><td>☒</td><td>LOD & LOQ</td><td>☒</td></tr>
<tr><td>Trueness</td><td>☒</td><td>Expanded measurement uncertainty</td><td>☒</td></tr>
</table>

Table A.2 — Validation results: example for bulk composition

Results
Selectivity: Good selectivity is achieved by using helium and argon carrier gas channels. ***LOD:*** N_2 – 83 µmol/mol; CO 133 µmol/mol; O_2 – 65 µmol/mol;. H_2 – 6 µmol/mol. ***Working range and linearity:*** The correlation coefficient for all components measured with GC-TCD were close to 1, suggesting that the equation for the linear regression fits the data. This observation implies that the method encompasses a linear working range of 488-94 213 µmol/mol for N_2; 175-960 µmol/mol for CO; 48-1922 for O_2; and 540 – 194 969 µmol/mol for H_2. The distribution of residuals is random, confirming the linearity and working range ***Bias:*** The bias for N2 was 2.17 %; for CO 0.17 %; for O_2 6.44 %; and for H_2 is 5.00 %. All components other than O_2 met the 5% bias target value. ***Precision:*** Precision was assessed by measuring duplicates of the calibration curve over several days. The relative repeatability and reproducibility were calculated using the one-way ANOVA function, giving 0.41 and 2.04 % for N_2; 0.04 % and 0.15 % for CO; 0.48 % and 1.82 % for O_2; and 0.58 % and 1.36 % for H_2. ***Measurement uncertainty:*** The expanded uncertainty (k=2) for each component was calculated as a root of the sum of the squares of the trueness, repeatability, and reproducibility, giving 6.01 % for N_2; 0.46 % for CO; 13.42 % for O_2; and 10.43 % for H_2.
Conclusions
ISO ISO 6974-1:2017 therefore found to be fit-for-purpose, reliable and highly sensitive for the analysis of N_2, CO, and H_2. The expanded uncertainty for O_2 was above the targeted level of 10 %, due to significant bias.

Conclusions on method applicability	☒ The method meets the requirements and can be implemented	☐ The method does not meet the requirements

Table A.3 — Validation Plan: example for siloxanes

Method title	The determination of siloxanes L2, L3, D3, D4 and D5 in a matrix of methane using thermal desorption gas chromatography with flame ionization detection and mass spectrometry detection			
Normative references	ISO 2620 - Analysis of natural gas — Biomethane — Determination of VOCs by thermal desorption gas chromatography with flame ionization and/or mass spectrometry detectors			
Scope	Siloxanes in gas are enriched on the adsorbent Tenax TA, thermally desorbed for gas chromatographic separation on a non-polar capillary column. The majority of the sample goes to a flame ionization detector (FID) and a smaller portion goes to a mass selective detector (MS) for identification of individual components and quantification.			
Analytes	hexamethyldisiloxane (L2), octamethyltrisiloxane (L3), hexamethylcyclotrisiloxane (D3), octamethylcyclotetrasiloxane (D4) and decamethylcyclopentasiloxane (D5)			
Range (mg/m^3)	0.005 - 20			
Matrix	Gas (biomethane).			
Reference materials	A reference mixture produced by a national measurement institute in methane containing 0.0874 ± 0.0044 µmol/mol of L2, 0.0557 ± 0.0034 µmol/mol of L3, 0.0542 ± 0.0038 µmol/mol of D3, 0.0429 ± 0.0026 µmol/mol of D4 and 0.0326 ± 0.002 µmol/mol of D5 was used for the validation.			
Sampling	Flow through Tenax TA sorbent tubes			
Metrological traceability	Analytical instruments have been calibrated with standards traceable to the National Metrology Institute (NMI), which are linked to the International System of Units (SI).			
Performance requirements	The method should be optimized to avoid co-elution of the analytes of interest and the other components present in the sample. Trueness target value was set to 5 %, precision 2.5 % and an expanded measurement uncertainty of 10% .			
Extent of validation	Precision: Repetability	☒	Selectivity	☒
	Precision: Intermediate precision	☐	Working range and linearity	☒
	Precision: Reproduceability	☐	LOD & LOQ	☒
	Trueness	☒	Expanded measurement uncertainty	☒

Table A.4 — Validation results: example for siloxanes

Results		
Selectivity: D4 eluate close to a dimethyl-octene using FID but shows good separation when using MS. ***LOD:*** Varies between 0.18 and 0.9 ng which is equal to 1.8 µg/m^3 and 9 µg/m^3 calculated with a volume of 100ml. ***Working range and linearity:*** the correlation coefficient for all siloxanes measured with GC-MS/FID were close to 1, suggesting that the equation for the linear regression fits the data. This observation implies that the method encompasses a linear working range within 3 to around 120 ng. The distribution of residuals is random, confirming the linearity and working range ***Bias:*** The mean bias for L2 was calculated to -21 and the relative bias was calculated to -3.6%. Using this result, the standard deviation of the measured concentration, SRW, was calculated to be 1.8 %. Taking into account the uncertainty of the reference standard, the total bias, u(bias), was calculated to be 4.56% which is in good agreement with the targeted value of 5%. ***Precision:*** Precision was assessed by measuring 10-12 duplicates of L2, L3, D3, D4 and D5 at varying quantities using TD-GC-MS/FID on several days. Standard deviation, as well as relative standard deviation, were determined and the pooled standard deviation was calculated. The measured Sr varies between 0.8 to 3.9 %. Taking into account, the contribution of control samples (here toluene), the within-laboratory reproducibility, u(Rw), was evaluated to be 3.04% for L2 in MS, a bit higher than the targeted value of 2.5% ***Measurement uncertainty:*** The expanded uncertainty (k=2) for L2 was calculated using the software MUKit. The calculations show that the measurement uncertainty is 11%.		
Conclusions		
ISO 2620:2024 is therefore found to be fit-for-purpose, reliable and highly sensitive. As the working range is at least 2 to 100 ng, it can be used to analyse samples with amount fractions of siloxanes from 2 nmol/mol to 1 µmol/mol (using volumes of 5 to 200 ml per tube).		
Conclusions on method applicability	☒ The method meets the requirements and can be implemented	☐ The method does not meet the requirements

Bibliography

[1] *ISO 6142-1:2015/Amd 1:2020 Gas analysis — Preparation of calibration gas mixtures — Part 1: Gravimetric method for Class I mixtures — Amendment 1: Corrections to formulae in Annex E and G;*

[2] *ISO 6143:2025 Gas analysis — Comparison methods for determining and checking the composition of calibration gas mixtures;*

[3] *ISO 6144:2003 Gas analysis — Preparation of calibration gas mixtures — Static volumetric method;*

[4] *ISO 6145-7:2018 Gas analysis — Preparation of calibration gas mixtures using dynamic methods — Part 7: Thermal mass-flow controller;*

[5] ISO 6974-1:2012, *Natural gas — Determination of composition and associated uncertainty by gas chromatography Part 1: General guidelines and calculation of composition;*

[6] *ISO 6974-2:12012, Natural gas — Determination of composition and associated uncertainty by gas chromatography — Part 2: Uncertainty calculations;*

[7] *ISO 5725-1:2023, Accuracy (trueness and precision) of measurement methods and results;*

[8] *ISO 15796:2005, Gas analysis – investigation and treatment of analytical bias;*

[9] *ISO 7504:2015, Gas analysis — Vocabulary;*

[10] *ISO 10715:2022, Natural gas — Sampling guidelines;*

[11] *ISO 10723:2012, Natural gas - Performance evaluation for analytical systems;*

[12] *ISO 14532:2014, Natural gas — Vocabulary;*

[13] *ISO/IEC guide 98-3:2008, Uncertainty of measurement — Part 3: Guide to the expression of uncertainty in measurement (GUM:1995);*

ISO FORM 4

NEW WORK ITEM PROPOSAL (NP)

DATE OF CIRCULATION:
Click here to enter a date.

PROPOSER:

☐ ISO member body:
Click or tap here to enter text.

☒ Committee, liaison or other:
ISO/TC 193/SC 1 'Analysis of natural gas'

CLOSING DATE FOR VOTING:
Click here to enter a date.

REFERENCE NUMBER:
Click or tap here to enter text.

☒ **WITHIN EXISTING COMMITTEE**
Document Number: Click or tap here to enter text.
Committee Secretariat: NEN

☐ **PROPOSAL FOR A NEW PC**

A proposal for a new work item within the scope of an existing committee shall be submitted to the secretariat of that committee.

A proposal for a new project committee shall be submitted to the Central Secretariat, which will process the proposal in accordance with ISO/IEC Directives, Part 1, Clause 2.3.

Guidelines for proposing and justifying new work items or new fields of technical activity (Project Committee) are given in ISO/IEC Directives, Part 1, Annex C.

IMPORTANT NOTE: Proposals without adequate justification and supporting information risk rejection or referral to the originator.

PROPOSAL

(to be completed by the proposer, following discussion with committee leadership if appropriate)

English title

Biomethane — performance evaluation for analytical systems

French title

Biométhane — Évaluation des performances des systèmes d'analyse

(Please see ISO/IEC Directives, Part 1, Annex C, Clause C.4.2).
In case of amendment, revision or a new part of an existing document, please include the reference number and current title

SCOPE

(Please see ISO/IEC Directives, Part 1, Annex C, Clause C.4.3)

This specifies a method for determining whether an analytical system for biomethane composition analysis is fit for a defined purpose. It can be used:

a) to determine a range for each specified component over which the errors and uncertainties in measured composition do not exceed a predefined measurement requirement, using specified calibration gas(es);

b) to determine errors and uncertainties in a measured composition over a pre-defined range for each analyte, using specified calibration gas(es).

It is assumed that the analytical system is applied to compositions that vary over the typical ranges found within biomethane.

Examples specific to gas chromatography, spectroscopy and spectrometry are provided throughout the document.

PURPOSE AND JUSTIFICATION

(Please see ISO/IEC Directives, Part 1, Annex C and additional guidance on justification statements in the brochure Guidance on New Work)

Click or tap here to enter text. (Please use this field or attach an annex)

PROPOSED PROJECT LEADER (name and email address)

Lucy Culleton, lucy.culleton@npl.co.uk

PROPOSER (including contact information of the proposer's representative)

Lucy Culleton, lucy.culleton@npl.co.uk

☒ **The proposer confirms that this proposal has been drafted in compliance with ISO/IEC Directives, Part 1, Annex C**

PROJECT MANAGEMENT

Preferred document
☒ International Standard
☐ Technical Specification
☐ Publicly Available Specification*

* While a formal NP ballot is not required (no eForm04), the NP form may provide useful information for the committee P-members to consider when deciding to initiate a Publicly Available Specification.

Proposed Standard Development Track (SDT – to be discussed by the proposer with the committee manager or ISO/CS)

☒ 18 months ☐ 24 months ☐ 36 months

Proposed date for first meeting: 2025-06-19

Proposed TARGET dates for key milestones

- Circulation of 1st Working Draft (if any) to experts: 2025-05-30

- Committee Draft consultation (if any): 2025-09-30
- DIS submission*: 2025-11-28
- Publication*: 2026-11-30

* Target Dates for DIS submission and Publication should be set a few weeks ahead of the limit dates automatically determined when selecting the SDT.

It is proposed that this DOCUMENT will be developed by:

☒ An existing Working Group, add title ISO/TC 193/SC 1/WG 25

☐ A new Working Group Click or tap here to enter text.
(Note that the establishment of a new Working Group requires approval by the parent committee by a resolution)

☐ The TC/SC directly

☐ To be determined

☐ This proposal relates to a new ISO document

☐ This proposal relates to the adoption, as an active project, of an item currently registered as a Preliminary Work Item

☐ This proposal relates to the re-establishment of a cancelled project as an active project

☐ Other: Click or tap here to enter text.

Additional guidance on project management is available here.

PREPARATORY WORK

☒ A draft is attached

☐ An existing document serving as the initial basis is attached

☐ An outline is attached
Note: at minimum an outline of the proposed document is required

The proposer is prepared to undertake the preparatory work required:

☒ Yes ☐ No

If a draft is attached to this proposal:

Please select from one of the following options:

☒ The draft document can be registered at Preparatory stage (WD – stage 20.00)

☐ The draft document can be registered at Committee stage (CD – stage 30.00)

☐ The draft document can be registered at enquiry stage (DIS – stage 40.00)

☐ If the attached document is copyrighted or includes copyrighted content, the proposer confirms that copyright permission has been granted for ISO to use this content in compliance with clause 2.13 of ISO/IEC Directives, Part 1 (see also the Declaration on copyright).

RELATION OF THE PROPOSAL TO EXISTING INTERNATIONAL STANDARDS AND ON-GOING STANDARDIZATION WORK

To the best of your knowledge, has this or a similar proposal been submitted to another standards development organization or to another ISO committee?

☐ Yes ☒ No

If Yes, please specify which one(s) Click or tap here to enter text.

☒ The proposer has checked whether the proposed scope of this new project overlaps with the scope of any existing ISO project

☐ If an overlap or the potential for overlap is identified, the proposer and the leaders of the existing project have discussed on:
  i. modification/restriction of the scope of the proposal to avoid overlapping,
  ii. potential modification/restriction of the scope of the existing project to avoid overlapping.

☐ If agreement with parties responsible for existing project(s) has not been reached, please explain why the proposal should be approved
Click or tap here to enter text.

☐ Has a proposal on this subject already been submitted within an existing committee and rejected? If so, what were the reasons for rejection?
Click or tap here to enter text.

This project may require possible joint/parallel work with
☐ IEC (please specify the committee) Click or tap here to enter text.
☐ CEN (please specify the committee) Click or tap here to enter text.
☐ Other (please specify) Click or tap here to enter text.

Please select any UN Sustainable Development Goals (SDGs) that this proposed project would support
(information about SDGs, is available at www.iso.org/SDGs)

☐ GOAL 1: No Poverty
☐ GOAL 2: Zero Hunger
☐ GOAL 3: Good Health and Well-being
☐ GOAL 4: Quality Education
☐ GOAL 5: Gender Equality
☐ GOAL 6: Clean Water and Sanitation
☒ GOAL 7: Affordable and Clean Energy
☒ GOAL 8: Decent Work and Economic Growth
☒ GOAL 9: Industry, Innovation and Infrastructure
☐ GOAL 10: Reduced Inequality
☐ GOAL 11: Sustainable Cities and Communities
☐ GOAL 12: Responsible Consumption and Production
☒ GOAL 13: Climate Action
☐ GOAL 14: Life Below Water
☐ GOAL 15: Life on Land
☐ GOAL 16: Peace, Justice and strong institutions
N/A GOAL 17: Partnerships for the goals

Identification and description of relevant affected stakeholder categories
(Please see ISO CONNECT)

Benefits/Impacts/Examples

Industry and commerce – large industry	Ensures quality of measurements performed on biomethane for use in gas transmission and distribution infrastructure.
Industry and commerce – SMEs	Facilitates the development of new products and services via a standardised performance assesment methodology. Facilitates validation of analysers used by SMEs for biomethane quality monitoring.
Government	A new standardised performance assesment methodology that can be used to inform policy
Consumers	Ensures quality of end products aquired by consumers via a standardised performance assesment methodology
Labour	Facilitates the development of new products and services via a standardised performance assesment methodology
Academic and research bodies	Facilitates the evaluation of analysers used for academic research, ensuring quality of results
Standards application businesses	A new standard is available for applcation in the field of biomethane conformity assesment.
Non-governmental organizations	Click or tap here to enter text.
Other (please specify)	Click or tap here to enter text.

Listing of countries where the subject of the proposal is important for their national commercial interests (Please see ISO/IEC Directives, Part 1, Annex C, Clause C.4.8)

Click or tap here to enter text.

Listing of external international organizations or internal parties (other ISO and/or IEC committees) to be engaged in this work (Please see ISO/IEC Directives, part 1, Annex C, Clause C.4.9)

Click or tap here to enter text.

Listing of relevant documents (such as standards and regulations) at international, regional and national level (Please see ISO/IEC Directives, Part 1, Annex C, Clause C.4.6)

Click or tap here to enter text.

ADDITIONAL INFORMATION

Maintenance Agencies (MAs) and Registration Authorities (RAs)

☐ This proposal requires the designation of a maintenance agency.
If so, please identify the potential candidate:
Click or tap here to enter text.

☐ This proposal requires the designation of a registration authority.
If so, please identify the potential candidate
Click or tap here to enter text.

NOTE: Selection and appointment of the MA or RA are subject to the procedure outlined in ISO/IEC Directives, Part 1, Annex G and Annex H.

Known patented Items (Please see ISO/IEC Directives, Part 1, Clause 2.14)

☐ Yes ☒ No

If Yes, provide full information as an annex

Is this proposal for an ISO management System Standard (MSS)?

☐ Yes ☒ No

Note: If yes, this proposal must have an accompanying justification study. Please see the Consolidated Supplement to the ISO/IEC Directives, Part 1, Annex SL or Annex JG