Publishable Summary for 21NRM04 BiometCAP
Protocol for SI-traceable validation of methods for biomethane conformity assessment

Overview

The conformity assessment of biomethane requires further standardisation in order to support Europe's green energy future. The overall EU target for Renewable Energy Sources consumption by 2030 has been raised to 32 % in the RED II directive [Directive (EU) 2018/2001, 2018]. This project will deliver accessible traceability to the stakeholder community by developing efficient and cost-effective methods for the preparation of traceable gas transfer standards for the performance evaluation of biomethane monitoring systems. Using these, a robust performance assessment protocol will be developed and validated in order to benchmark and characterise analytical systems (e.g. gas analysers). The outputs, including trial applications, will be directly fed into standardisation development. This project will bridge the gap between previously developed primary standards and the industry's need for accessible, traceable performance evaluation against a validated protocol.

Need

Biomethane is already used widely within Europe as a means to sustainably displace fossil fuels. Its usage is projected to increase significantly (doubling by 2030 from the current 26 Terawatt-hour (TWh) produced in Europe) [EBA "European Biogas Association Statistical Report: 2019 European Overview", 2020] as a result of European green energy targets [European Commission, A European Green Deal, 2019]. Biomethane quality monitoring is essential to prevent damage to the existing natural gas infrastructure and to end user appliances that can be caused by harmful impurities in biomethane. In addition, these impurities need to be kept below limit thresholds (as specified in EN 16723 for gas grids [EN 16723 1 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 vehicles [EN 16723 2 Natural gas and biomethane for use in transport and biomethane for injection in the natural gas network Part 2: Automotive fuel specifications, 2017]).

Reliable and traceable purity measurements can only be obtained with equipment of known performance, from which the sensitivity, selectivity, precision and bias have been traceably evaluated (as required under e.g. ISO/IEC 17025 §7.2 [ISO/IEC 17025, Testing and Calibration Laboratories, 2017]) (Objective 3). Instrument manufacturers and end users require a standardised protocol (Objective 2) in order to meaningfully demonstrate instrument performance in both laboratory and field settings. Despite similar approaches existing for other green fuels, e.g. hydrogen in the form of ISO 21087 [ISO 21087 Gas analysis — Analytical methods for hydrogen fuel — Proton exchange membrane (PEM) fuel cell applications for road vehicles, 2019], a suitable biomethane evaluation protocol does not yet exist.

An additional challenge is bias prevention, as existing measurement methods (e.g. as developed in EMPIR JRP 16ENG05 Biomethane) have not been traceably tested for cross-interference bias caused by gas matrix and impurity variation. This type of selectivity evaluation is essential to prevent bias in reported results, to prevent interruptions in the supply of biomethane into the European gas infrastructure (caused by over-reporting), and to prevent damage to infrastructure (caused by under-reporting).

To deliver this on a practical level, cost effective transfer standards need to be developed to disseminate traceability from the primary standards in an accessible format for the biomethane industry, which includes many small-scale producers (Objective 1). These transfer standards need to be fit-for-purpose in terms of measurement uncertainty (≤10 %) and shelf life (≥12 months). Improving accessibility to gas standards and a

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validated protocol for their application will allow for a safe and effective expansion of the biomethane industry within Europe.

Objectives

The overall objective of this project is to support the development of standardisation and to provide the traceability, reliability and characterisation necessary for the conformity assessment of biomethane.

The specific objectives of the project are:

1. To develop and validate methods for the static and dynamic preparation of gas transfer standards containing different groups of impurities. These should be suitable for use in the SI-traceable validation and performance evaluation of current and future analytical instruments and methods that are used in the conformity assessment of biomethane in accordance with EN 16723. In addition, this should include determining the key metrological parameters of each method. Uncertainties of 1 % - 10 % are targeted for EN 16723 limit values.
2. To develop a comprehensive protocol for the validation and performance evaluation of the analytical instruments and methods that are used in the conformity assessment of biomethane (applicable to both current and future methods). The validated and implemented methods should be able to generate reproducible and SI-traceable measurement results and a NWIP and draft ISO text should be submitted to ISO.
3. To use the protocol, developed in objective 2, to evaluate the performance of commercially available industrial gas analysers, based on e.g. spectroscopy or gas chromatography, which are used for laboratory and field-based biomethane (test) measurements. The protocol should also be used to evaluate all relevant measurement methods. A review will be undertaken to evaluate its effectiveness and reproducibility across a wide variety of methods and compounds.
4. To maintain constant contact with the EMN for Energy Gases and collaborate with the technical committee ISO/TC193/SC1/WG25 “Biomethane” and the users of the standards they develop to ensure that the outputs of the project are aligned with their needs and are incorporated into future standards at the earliest opportunity and that user training and knowledge transfer is maximised throughout the project. This will include, in particular, the protocol developed in objective 2.

Progress beyond the state of the art and results

To develop and validate methods for the static and dynamic preparation of gas transfer standards containing different groups of impurities. These should be suitable for use in the SI-traceable validation and performance evaluation of current and future analytical instruments and methods that are used in the conformity assessment of biomethane in accordance with EN 16723. In addition, this should include determining the key metrological parameters of each method. Uncertainties of 1 % - 10 % are targeted for EN 16723 limit values. (Objective 1)

This project will develop novel, cost effective solutions in the form of multi-component gas standards and improved dynamic methods for the preparation of gas transfer standards, with a focus on the impurities and limit levels specified in EN 16723. This cost-effective approach will allow laboratories to perform verification, validation and quality control as required by, e.g. ISO/IEC 17025, with reduced and affordable costs, thus increasing access to traceability for the industry. These standards will also resolve the issue of some standards not being available for field use on biomethane sites due to their complexity, non-portability and cost.

Novel cost-effective standards will also be developed for the evaluation of the effects of the variable biomethane gas matrix and cross-interferences caused by impurities simultaneously present in biomethane. This project is the first research project addressing this issue in full and the project’s output will help to secure the reliability of the analytical measurements of the impurities in biomethane, avoiding biases that are commonplace (e.g. terpenes/siloxane interferences via certain GC methods and methane interference with certain spectroscopic methods).

A stakeholder survey and consultation was completed, the outcome of which was shared with the participants and the stakeholder committee. The results were used to define a specification for the compositions of static and dynamic standards which will be prepared within the project. The development of static gas standards has progressed according to plan and are scheduled for dispatch to relevant participants.

To develop a comprehensive protocol for the validation and performance evaluation of the analytical instruments and methods that are used in the conformity assessment of biomethane (applicable to both current and future methods). The validated and implemented methods should be able to generate reproducible and SI-traceable measurement results and a NWIP and draft ISO text should be submitted to ISO. (Objective 2)

Currently, gas analysers for biogas and biomethane applications cannot be reliably evaluated for performance. ISO 10723 [ISO 10723, Natural gas — Performance evaluation for analytical systems, 2012] describes the performance evaluation for analytical systems for natural gas composition, and ISO 21087 is in place for hydrogen applications, however methods and protocols for biogas and biomethane applications still need to be developed. This project will develop a robust protocol for the sampling, analysis and performance evaluation of analytical instruments (gas analysers) and methods that are used for biomethane conformity assessment. The protocol will be designed to be suitable for current and future measurement techniques.

The consortium is progressing with the development of the protocol with completion expected in autumn 2023. The consortium published 2 open access reports summarising the requirements for the protocol (validation parameters, existing protocols, and standardised methods), and the requirements for sampling including recommendations for maintaining the integrity of the sample (available on the BiometCAP website).

To use the protocol, developed in objective 2, to evaluate the performance of commercially available industrial gas analysers, based on e.g. spectroscopy or gas chromatography, which are used for laboratory and field-based biomethane (test) measurements. The protocol should also be used to evaluate all relevant measurement methods. A review will be undertaken to evaluate its effectiveness and reproducibility across a wide variety of methods and compounds. (Objective 3)

The performance assessment protocol will be validated using lab-based analysers, and traceably validated methods that were previously developed as part of targeted research (e.g. EMRP JRP ENG54 Biogas, EMPIR JRP 16ENG05 Biomethane). The application of the performance assessment protocol will then be expanded to industrial analysers and a review will be undertaken of the results of its application, comparing the variables of location, analyte and technique in order to produce a good practice guide for the repeatable and accurate implementation of the performance assessment protocol. Such a comparison has not been undertaken for the biomethane industry and the results will provide a valuable route for more solutions to enter the market and for stakeholder knowledge to be enhanced.

Several measurement techniques have been reviewed and three techniques have been selected (FTIR/NDIR, micro-GC/GC and TDLS/CRDS) in close discussion with the participants and stakeholders. Four biogas production sites, two in Denmark and two in Finland have been identified.

Outcomes and impact

To promote the project, and to share insights generated throughout the project, the project was introduced to scientific and industrial end-users. One paper has been submitted to Accreditation and Quality Assurance. One presentation was made at the 2023 Emerging Fuel Symposium conference in USA, and a poster was presented at a workshop organised by the EMN for Energy Gases in Portugal. The project was introduced to the ISO/TC 193 WG25 and two reports were sent to the WG afterwards. The project was also introduced to the WG Biomethane of the GERG network.

Outcomes for industrial and other user communities

By gaining an insight into the user communities measurement needs, the project's outcomes will enable fit-for-purpose biomethane conformity assessment measurement services to be provided to industries, testing laboratories, research organisations and other end-users. Such services will include: a) calibration gas mixtures b) calibration and measurement facilities and services c) performance evaluation of gas analysers; d) direct characterisation of biomethane quality in the field; e) proficiency testing; f) consultancy and g) training. Use of the good practice guide produced by the project will allow end users to have an accessible format to refer to for use with biomethane conformity testing, thus allowing the project's outcomes to be shared. Accreditation can be sought against e.g. ISO 17025.

Instrument (e.g. gas analysers) and sensor manufacturers will receive access to a performance assessment protocol which they can use to benchmark their products and use during the product development process as

a means of quality control. Their customers will be able to utilise the performance assessment protocol for their own quality control checks when e.g. developing and characterising methods.

Biomethane producers will be able to use the performance assessment protocol in combination with transfer standards and training material outputs to repeatably and accurately quantify analyser performance, which will reduce the opportunity for measurement bias and improve the efficiency of biomethane production. This will ensure that they meet the quality assurance requirements of e.g. national regulations.

As outlined above, it is anticipated that a major outcome of this project will be the widespread uptake and use of the biomethane performance assessment protocol for biomethane conformity assessment throughout Europe and globally. Another outcome will be the increase in new industrial products and services for biomethane quality monitoring using the new solutions developed in this project (e.g. traceably validated industrial biomethane measurement systems as validated within this project).

Outcomes for the metrology and scientific communities

Laboratories, research organisations and academia will be able to use the state-of-the-art gas transfer standards and the performance assessment protocol developed within this project to perform further research within the biomethane purity area to support the development of new biomethane measurement technologies. Using the outcomes from this project, their performance will be able to be traceably quantified to the SI.

Metrology institutes will have new capability in the form of gas transfer standards and a performance assessment protocol by which to evaluate further techniques and methods they wish to develop within the biomethane area for research and delivery of measurement services to industry. This knowledge will be transferred from the experienced to the less experienced metrology institutes.

Outcomes for relevant standards

The project's outputs will provide direct input to ISO/TC193/SC1/WG25 "Biomethane" by providing them with a validated protocol for the sampling, analysis and performance evaluation of gas analysers. The results of the performance evaluation of the industrial gas analysers will give a realistic overview of (industrial) measurement capabilities, which will allow standards to be tailored to the real needs of industry and it will promote their widespread uptake. The protocol and method developed for the performance evaluation of gas analysers will be submitted to ISO/TC193/SC1/WG25 for consideration as a new ISO standard (in the form of a New Work Item Proposal (NWIP) and draft ISO standard text). The results obtained from the project will also be disseminated to CEN to enable it to update EN 16723 under its mandate M/475.

The project will also provide input to the activities of other committees, such as ISO/TC158, CEN/TC408, BIPM CCQM Gas Analysis Working Group (GAWG), EURAMET/Metchem SC-GAS, and national working groups and mirror committees.

Longer-term economic, social and environmental impacts

In the longer term, the project's outputs will enable fit-for-purpose services to be provided to industries, testing laboratories, research organisations and other end-users. End users will be able to verify the performance of their measurement equipment with a validated protocol using the developed gas transfer standards. The need for fewer gas transfer standards will significantly lower the cost of biomethane conformity assessment, making biomethane production cost-effective for small producers. These service receivers will be able to undertake e.g. SI-traceable calibrations of equipment and the calibration of gas mixtures, to improve the quality of their measurement results, and they will be able to use traceable and cost-effective gas transfer standards for measuring impurities in biogas and biomethane.

The accurate measurement of trace-level toxic impurities in biomethane will, in the longer term, ensure that regulations limiting the contents of these compounds to safe (non-toxic) levels can be enforced robustly.

This project will accelerate the increased use of biomethane and upgraded biogas and it will enrich the European natural gas supply chains. Therefore, it will also help to reduce Europe's dependence on natural gas imports and it will promote the realisation of the EU target on Renewable Energy.

Increased use of biomethane will help reduce emissions of greenhouse gases and reliance on fossil fuels, as biomethane is produced from renewable sources, such as organic waste, landfills, pulp sludge or manure. Its use in road vehicles will significantly limit harmful emissions. This project will assist the EU in moving towards solving the major global problem of decarbonisation.

List of publications

n/a

This list is also available here: https://www.euramet.org/repository/research-publications-repository-link/

Project start date and duration:		1 October 2022, 36 months
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Internal Beneficiaries: 1. RISE, Sweden 2. BFKH, Hungary 3. CMI, Czechia 4. IMBiH, Bosnia and Herzegovina 5. PTB, Germany 6. TUBITAK, Türkiye 7. VSL, Netherlands 8. VTT, Finland	External Beneficiaries: 9. DTU, Denmark 10. GERG, Belgium 11. TFS, Germany	Unfunded Beneficiaries:
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