Publishable Summary for 21GRD05 Met4H2
Metrology for the hydrogen supply chain

Overview
The urgent need to mitigate climate change and to limit greenhouse gas emissions is driving actions to reduce the use of fossil fuels. However, meeting current and future energy needs necessitates the increased use of alternative energy sources such as hydrogen from renewable sources. To achieve this goal, the metrological infrastructure for hydrogen needs to address all parts of the supply chains. This project will provide novel and improved standards for the safe application of hydrogen flow measurement, hydrogen quality assessment and custody transfer. Together with outcomes from previous projects, an infrastructure will be established that provides measurement data that are fit for demonstrating compliance with regulations and contracts. This infrastructure will facilitate ramping up the use of hydrogen and society to adapt to using hydrogen instead of fossil fuels.

Need
The report from the Intergovernmental Panel for Climate Change of 2021 underlined once more the urgent need to reduce greenhouse gas emissions to mitigate climate change. The European Commission developed the European Green Deal to decarbonise energy use, shifting from fossil fuels to renewable fuels. One of the pillars is a phased approach to the introduction of hydrogen to replace fossil fuels in electrical power generation, transport, industry and the built environment. In addition, recent geopolitical developments on the edges of Europe have further highlighted the need for diverse, reliable and resilient, non-fossil fuel-based energy supplies in Europe. To apply hydrogen safely within the existing gas grid network and other infrastructure, traceable measurements for leak testing, material compatibility, sensors for monitoring processes and odorization are required to demonstrate compliance with legislation. Traceable flow measurement and hydrogen quality assessment are necessary for custody transfer and fair trade. Demonstrating compliance with hydrogen quality specifications, such as ISO 14687, and legal metrology requirements, such as International Organization of Legal Metrology (OIML) R140 and OIML R137, for metrological type approval and for assessing the performance of measuring systems, is therefore essential. Hydrogen sampling methods for applications below 20 MPa, such as gas grids, need to be developed and validated, to ensure metrological traceability and reliability of data obtained using those methods.

Metrological traceability and accuracy already developed need to be deployed to onsite measurement systems, so that robust and comparable results are obtained that support their use beyond monitoring processes. Finally, there is a need to improve and expand documentary standards for totalisation of quantity and energy used for custody transfer, such as OIML R140, ISO 15112 and EN 1776, to make them fit for purpose for use with hydrogen-enriched natural gas and hydrogen and to cover the totalisation of impurity content.

Objectives
The overall objective of the project is to further develop and integrate the metrology necessary to support the entire supply chain of hydrogen, from production to storage and end use. The project will disseminate metrological traceability to the field, so that measurement results become fit-for-purpose with respect to health, safety, environmental, and fiscal purposes.

The specific objectives of the project are:

1. To develop calibration and measurement methods to support reliable, traceable, and accurate measurements of hydrogen in production processes and end-user applications, in view of safety, process efficiency and environmental issues, such as for purity, leak detection, odorization, and

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materials performance, ensuring that online measurement instruments and sensors are operating within their specifications (e.g., to ISO 14687, OIML R139, and OIML R140).

2. To develop measurement standards to enable calibration and validation of flow metering equipment under actual conditions (pressure, temperature), used to accurately quantify flow rates of hydrogen (including blended hydrogen) through the hydrogen supply chain, and to facilitate compliance with respect to, e.g., OIML R137, OIML R140, and the Measurement Instruments Directive.
3. To develop and improve measurement standards and methods to enable traceable validation and performance evaluation of gas quality measurement methods for hydrogen, to thus improve on the current lack of equivalence for impurities, e.g., oxygen, hydrogen sulphide, moisture content, and for reactive components such as hydrogen chloride and chlorine. To develop and improve analysers for critical impurities for online monitoring of changes in gas quality, through the supply chain and processing equipment, to ensure the gas quality meets the required specifications (ISO 14687).
4. To develop novel methods for the evaluation of measurement uncertainty along the supply chain as a whole, namely with regard to the measurement of total quantity, and energy and impurity content of hydrogen and hydrogen blends.
5. To facilitate, in cooperation with the European Metrology Network Energy Gases, the take up of the technology and measurement infrastructure developed in the project by the measurement supply chain (accredited laboratories, instrumentation manufacturers for hydrogen), standards developing organisations (ISO, OIML) and end users (e.g., industry, consumers, power generation and transport).

Progress beyond the state of the art and results

To develop calibration and measurement methods to support reliable, traceable, and accurate measurements of hydrogen in production processes and end-user applications, in view of safety, process efficiency and environmental issues, such as for purity, leak detection, odorization, and materials performance, ensuring that online measurement instruments and sensors are operating within their specifications (e.g., to ISO 14687, OIML R139, and OIML R140).

Calibration standards for measuring hydrogen leaks have been designed and are being constructed. Different measurement principles are used, such as the pressure-temperature-volume behaviour of the fluid, refractometry and gravimetric flow measurement. The first version of the transfer standard was designed and used in the preliminary metrological validation of one of the designed calibration standards.

A high-pressure permeation setup was designed considering the recommendations and specifications of ISO 15105-1. This standard describes a general method for measuring the gas-transmission rate in plastics. A permeation cell was designed for the setup, as well as the two sample holders for O-rings and sheets/foils. Permeation measurements at pressure differences up to 300 bar on high performance polymer foils and hydrogen certified O-rings were successfully performed.

A comprehensive protocol for the evaluation of the performance of sensors used in hydrogen production, upgrading, transportation and use is being developed. The following metrological metrics (e.g., accuracy, baseline, cross-sensitivity (selectivity), drift, environmental effects, final indication, hysteresis, limit of quantification, linear range/measuring range, noise, operational range, uncertainty, response/recovery time, reversibility, resolution, saturation, sensitivity) are being taken into account.

Based on the literature review of odorization of natural gas and hydrogen, reference gas mixtures were specified and will be prepared in duplicate. Analytical methods for various sulphur compounds at ppm-level have been optimised. Experiments were performed to assess the matrix effect of hydrogen, hydrogen-enriched natural gas and methane as matrix. A sulphur-free odorant was selected to prepare gravimetric reference gas mixtures at the ppm level in hydrogen in cylinders with potentially suitable passivation treatment. One gas mixture for each odorant is being prepared. Based on stakeholder input, two mixtures with 2-hexyn in hydrogen are being prepared and will follow the stability by using an internal standard (benzene) in the mixture.

To develop measurement standards to enable calibration and validation of flow metering equipment under actual conditions (pressure, temperature), used to accurately quantify flow rates of hydrogen (including blended hydrogen) through the hydrogen supply chain, and to facilitate compliance with respect to, e.g., OIML R137, OIML R140, and the Measurement Instruments Directive.

A literature study related to the use of hydrogen in pipelines in Europe has been prepared. It details the gas distribution grids and specific plans to use the grid to transport hydrogen.

An evaluation of existing gas models for gas flow suitable for hydrogen was produced and accepted by the consortium. It assesses whether models for air or natural gas can correctly model hydrogen flow for different applications and flow conditions.

A document investigating the development of supply chain containing liquid hydrogen is in preparation.

The preparation of a travelling standard for the comparison of primary flow measurement standards for hydrogen-enriched natural gas, and the construction and characterisation of the transfer package have started. A protocol is being developed detailing the comparison process and calendar.

For the development of a primary flow standard for domestic gas flow meters, a reference flow meter was chosen. The development of the facilities was started by designing these and procuring the parts necessary to the construction. The facilities are at different stages of advancement depending on the partners, from under procurement to being operational.

Finally, with input from the Stakeholder Committee a survey was created and distributed among the networks of the participants to enquire the need for calibration facilities for large-capacity flow meters for hydrogen flow in Europe. The purpose is to create a roadmap for such a pan-European infrastructure, so that the gas industry has access to metrological traceability for hydrogen flow meters for gas grids.

To develop and improve measurement standards and methods to enable traceable validation and performance evaluation of gas quality measurement methods for hydrogen, to thus improve on the current lack of equivalence for impurities, e.g., oxygen, hydrogen sulphide, moisture content, and for reactive components such as hydrogen chloride and chlorine. To develop and improve analysers for critical impurities for online monitoring of changes in gas quality, through the supply chain and processing equipment, to ensure the gas quality meets the required specifications (ISO 14687).

Requirements were drafted for sampling points which are considered representative of pressure, temperature, humidity, sampling frequency, safety, and volume. Existing hydrogen sampling standards, guidelines and current practices were reviewed, including ISO/AWI 19880-9. Based on these outcomes, a sampling rack was designed and built. The sampling system was also specified for pressures up to 70 MPa, which is equipped with a safe venting of hydrogen. A second rig is being constructed, suitable for operation at 1-to-10 bar.

A transportable precision humidity generator (TPHG) was designed, and developed and commissioned to provide state-of-the-art on-site traceability to trace water measurements in hydrogen. The TPHG. first of its kind, is cooled with ethanol and operates between 0.2 MPa and 5.5 MPa, with water amount fractions from 0.5 ppm to 50 ppm or, equivalently, between -55 °C and -10 °C pressure dew point. Various instruments, based on different technologies, are available for validating the new TPHG in an ATEX-grade laboratory. Trials were started to prepare a primary gas reference material of water vapour in 3 types of hydrogen gas cylinders. Currently the fillings are up to 3 MPa at 5 ppm water in nitrogen. The short-term stability is good, and the agreement is within ± 0.1 ppm when compared to dynamically prepared gas mixtures. Additionally, an existing high-pressure dewpoint generator (HPDG) is being adapted for trace water measurements in hydrogen. The HPDG is designed to operate between 0.1 MPa and 6 MPa, with water amount fractions from 0.5 ppm to 100 ppm. The adaptation of the HPDG is nearing completion and the performance of the HPDG with H_2 carrier gas will be characterised with a cavity ring-down spectrometer.

The feasibility of preparing mixtures of chlorine in nitrogen was assessed, starting from a commercial mixture of chlorine in nitrogen. Analyses were performed with cavity-enhanced absorption spectroscopy. Using mass-flow controllers to create calibration gas mixtures at different amount fractions, a calibration curve was recorded.

Not much literature is available on the stability of hydrogen chloride in hydrogen. The preparations have started on a stability study of these calibration gas mixtures and a new HCl analyser has been purchased. The upgrading of the portable reference gas generator (based on the evaporation method) for hydrogen chloride in a hydrogen matrix at amount fraction 10 nmol/mol to 10 µmol/mol with relative uncertainties between 3 % to 5 % has started. The generator will prepare calibration gas mixture with about 0.1 % (volume fraction) water.

The development of a multicomponent gas analyser based on the far-UV spectroscopy, suitable for on-line/off-line simultaneous measurements of multiple compounds i.e. impurities (e.g., hydrogen sulphide, water and carbon dioxide) in hydrogen has started. The analyser is equipped with temperature sensors and a pressure sensor for pressures up to 5 MPa and suitable for measurements up to 4 MPa in H_2. In the design of the analyser experience and results gained in high-pressure permeation work have been accounted. First measurements with ultra-pure gases (6.0 grade) such as Ar, N_2 and H_2 up to 50 bar have been done. The measurements show that simultaneous measurements of e.g. H_2S, CO_2, H_2O, O_2, CO, HCl and Cl_2 under given amount fractions should be possible to perform in on-line continuous analyser operation. The analyser will next be validated using a transportable dynamic vapor generator developed in the project.

The first mixtures necessary for improving the total sulphur analysis in hydrogen were prepared, starting at the low ppm-level hydrogen sulphide. A two-step dynamic dilution setup was designed. Parts, including the mass flow controllers were ordered.

Work has started on the development of a traceable laser spectroscopic method for ammonia in the range 50 nmol/mol to 500 nmol/mol. The setup is ready for the first tests.

A compact versatile photoacoustic on-line analyser was developed. The analyser uses NIR and/or MIR spectroscopy capable of monitoring the most important impurities. Currently the design is being further developed and detailed. The analyser is under construction.

To develop novel methods for the evaluation of measurement uncertainty along the supply chain as a whole, namely with regard to the measurement of total quantity, and energy and impurity content of hydrogen and hydrogen blends.

A general framework for the calculation of uncertainties in the total volume, energy and purity exposure based on models described in, e.g., OIML R140, EN 1776 and ISO 15112 was set up. The framework includes the AGA8 and GERG-2008 equations of state. Uncertainty evaluation uses both the law of propagation of uncertainty and the Monte Carlo method. The input of data to the computational framework is standardised, using a modular approach that is adaptable to metering station configuration and other calculation methods.

Using the computational framework, an outline of software for calculating the uncertainties in the measurands of interest (total quantity, energy and purity exposure) was set up and implemented. The framework provides the quantities relevant to volumetric quantity and energy measurement. In a later stage, simpler cases such as measuring quantity on a mass basis will be implemented.

Existing uncertainty models were assessed and analysed to address the correlation among measurements taken with the same instrument. Data from natural gas metering stations was used to make the assessments. Models were developed for assessing correlations between flow rate, temperature, pressure and composition measurements. Work has started on the analysis of the time variation of parameters of interest such as quantity, energy content, and impurity, due to the presence of, e.g., daily or seasonal cycles. The present serial correlation under more or less steady-state conditions inflates uncertainty by some 30 %, assessed on two datasets. Different computational techniques for the totalization of relevant parameters were assessed. A distinction between deterministic and random signals was made by applying time-domain filtering method. The discretization error of the totalization was estimated and found to be proportional to N^{-2}.

Outcomes and impact

The project was presented at several conferences and international meetings. A website was created, and a LinkedIn page created. Two meetings with the Stakeholder Committee (SAB) took place, providing guidance on the implementation of the project. The first meeting with the SAB took place on M3, the second on M12, following a Stakeholder Workshop open to the public. The work on odorants attracted the interest of ISO/TC193/WG5 and MARCOGAZ. Several other standardisation committees were contacted and the information about the project activities was shared. The project team organised a bilateral meeting with the EMN Energy Gases at the beginning of the project to agree processes for information sharing; since then, communication actions were coordinated.

Outcomes for industrial and other user communities

This project will support the industry involved in the hydrogen supply chain from hydrogen production to transport and end use.

Novel measurement standards for hydrogen leak rate measurement will enable industry to have reliable devices to monitor the integrity of gas grids, thereby ensuring safety when feeding hydrogen into these grids. The methods and standards for material compatibility assessment will enable universities, research institutes and industry to assess materials for their suitability to be used with hydrogen, ensuring that potential hazardous situations are recognised at an early stage. The odorization standards will enable gas grid operators, for example, to confirm that the odorant level in hydrogen enriched natural gas (HENG) and hydrogen meets the specifications, thereby ensuring the safe distribution of these gases to the built environment. They also enable research institutes and other bodies to assess the olfactometric properties of these odorants with hydrogen-containing energy gases.

The rigs developed for the calibration and evaluation of sensors for hydrogen quality will enable users and producers of these sensors to have them assessed, so that these sensors have a known performance, and the results obtained are metrologically traceable. This traceability in turn enables users of the sensors to apply them beyond the monitoring of processes, thereby avoiding the need to measure again for, e.g., assessment of compliance with specifications.

The validated primary standards for flow metering of hydrogen-containing energy gases will enable custody transfer for these gases, in combination with the methods for sampling and hydrogen quality assessment, and the improved methods for totalisation and the associated measurement uncertainty evaluation.

The hydrogen quality measurements performed at two industrial sites, electrolyser plant and gas pipeline, will demonstrate to the industry how metrological traceability and accuracy can be delivered in real-life situations. These demonstrations and the good practice guidelines derived from them, will create a close link with the hydrogen production and transport sector as well as with the measurement system manufacturers and therefore, ensure a swift take up of project outcome.

Outcomes for the metrology and scientific communities
To facilitate the take up of hydrogen in Europe and worldwide, a well-established measurement infrastructure is a must. This project focuses on developing, optimising and comparing traceable measurement standards and methods, so that this infrastructure is created.

The novel flow measurement standards will enable NMIs, DIs and calibration laboratories to provide measurement services for the hydrogen supply chain and industry to have their instruments calibrated. These in turn enable research into the development of gas meters for HENG and hydrogen. The sampling methods, standards and methods for hydrogen quality assessment will enable services to be provided by the gas industry in the form of secondary and working gas standards and measurements and will provide research groups with the necessary tools to ensure their measurement results are metrologically traceable, so that conclusions from their work can directly be taken up by others.

The improved methods for hydrogen quality assessment will enable metrological traceability to be disseminated to laboratories, which in turn can seek accreditation based on ISO/IEC 17025 for their services related to ISO 14687, including sampling. Research groups will benefit from these capabilities in that they can assess improvements in processes along the supply chain with the necessary standards for calibration of their equipment.

Legal metrology organisations and their national bodies benefit from the work related to especially OIML R137 and OIML R140, as well as the calibration and measurement services enabling assessment of the performance of measurement equipment supporting conformity assessment and type approval.

Outcomes for relevant standards
The project will provide enhanced guidance for calculating the total quantity, energy and impurity exposure of supplied or received gas and good practices in taking into account the dynamics of the gas grid and gas properties in the uncertainty evaluation to OIML R140 and ISO 15112. The work on flow measurements will provide evidence as to whether OIML R140 can also be applied to metering and custody transfer of hydrogen and HENG. Material compatibility testing is covered in standards such as ISO 15105 and ISO 2782; this project contributes approaches for increasing the sensitivity of the measurement and set-up adjustments allowing for extension of the parameter range in terms of the boundary conditions. The results in WP3 will demonstrate that the scope of ISO 21087 can be extended to supply chains other than just PEM fuel cells. The materials compatibility overview for calibration gas mixtures in ISO 16664 can be updated based on the stability study data for static gas standards with, e.g., hydrogen chloride and moisture.

Longer-term economic, social and environmental impacts
As natural gas is the primary fuel source for heating in Europe, the market is approximately 550 billion cubic metres per year. The introduction of hydrogen in this part of the gas supply relieves the pressure on the electrical grids. This project will provide the tools to adapt the measurement infrastructure to distributing HENG in the first instance, and hydrogen at a later stage. Using HENG comes with relatively small changes for end users, and thus is a very economical measure to decarbonise the gas supply in the short term. It is also far less disruptive than requesting end-users to switch from gas to electricity for these purposes.

The outcomes support the safe application of hydrogen in gas transmission and distribution systems, as well as charging end-users and industry for their gas use in accordance with current requirements. Thereby it facilitates the transition from fossil fuels to net-zero carbon dioxide emission renewable fuels. Feeding in hydrogen enables end-users and industry to gradually adapt to this future, with as little disruption as necessary. In the coming years, feeding in green hydrogen in the natural gas grids leads to a reduction of carbon dioxide emissions, thereby contributing to meeting intermittent goals of the European energy system.

List of publications

n/a

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

<table>
<tr><td colspan="2">Project start date and duration:</td><td>01 October 2022, 36 months</td></tr>
<tr><td colspan="3">Coordinator: Adriaan van der Veen, VSL Tel: +31 6 12021712 E-mail: avdveen@vsl.nl
Project website address: https://met4h2.eu/</td></tr>
<tr><td>Internal Beneficiaries:
1. VSL, Netherlands
2. BAM, Germany
3. BEV-PTP, Austria
4. CEM, Spain
5. Cesame, France
6. CMI, Czechia
7. CNAM, France
8. DFM, Denmark
9. FORCE, Denmark
10. INRIM, Italy
11. JV, Norway
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16. DTU, Denmark
17. ENVIPARK, Italy
18. GERG, Belgium
19. Nippon Gases, Italy
20. NORCE, Norway
21. POLITO, Italy
22. UL, Slovenia
23. ZAE Bayern, Germany - ceased participation on 31 December 2022
24. CAE, Germany - joined the consortium on 1 January 2023</td><td>Unfunded Beneficiaries:
25. SICK, Germany</td></tr>
<tr><td colspan="3">Associated Partners:
26. METAS, Switzerland,
27. NEL, United Kingdom,
28. NPL, United Kingdom</td></tr>
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