Publishable Summary for 19ENG04 MetroHyVe 2
Metrology for hydrogen vehicles 2

Overview
To meet the 2050 Europe carbon neutral targets (net-zero emission from transport) many countries are now legislating to ban the sale of internal combustion engines. Electric vehicles are a viable alternative and can run on either hydrogen fuel cells (FC) or rechargeable batteries. According to 78 % of automotive executives polled, Fuel Cell Electric Vehicles (FCEV)s are "the real breakthrough for e-mobility" and could represent 32 % of fuel demand by 2050. Current barriers to mass implementation of hydrogen in transport arise from European Directive 2014/94/EU and International organisation of legal metrology (OIML) recommendations that must be met by all European hydrogen refuelling stations (HRS). This project will address these issues and will develop metrology that will enable hydrogen to become a conventional fuel and support the European energy transition. The project will tackle measurement challenges in hydrogen flow metering, hydrogen quality control and hydrogen sampling and fuel cell stack testing.

Need
Climate change, air quality and reliance on imported fuels from non-renewable sources require quick deployment of alternative fuels such as hydrogen to meet the 2050 Europe carbon neutral targets (net-zero emission from transport). The previous EMPIR project 16ENG01 MetroHyVe addressed hydrogen for transport measurement challenges and has established a solid basis of research for this project to build upon. Through engagement with key stakeholders such as consumers, hydrogen producers, HRS operators, automotive manufacturers and standardisation bodies, the previous project identified the following measurement challenges that need to be addressed in order to support the deployment of hydrogen as a transport fuel in Europe:

- Consumers need confidence that they are being charged correctly when buying hydrogen at a HRS. It is currently not possible to accurately measure the amount of hydrogen dispensed when filling hydrogen into a heavy-duty FCEV. (objective 1)
- HRS compliance to regulation is currently expensive to achieve and to maintain. To reduce the subsequent costs to the consumer, HRS operators need the ability to test their stations in a quicker, cheaper and more frequent manner. Methods using secondary standards for hydrogen flow dispensed at HRS calibrated by the European primary standards are urgently needed for both light and heavy-duty vehicles. (objective 1)
- To improve reliability of their results and hence facilitate improved confidence of end users, commercial gas analysis laboratories in Europe need hydrogen fuel reference materials for the measurement of hydrogen fuel quality according to ISO 14687:2019. (objective 2)
- There are no harmonised guidelines or ISO standards for hydrogen fuel sampling at HRS. Sampling bias due to improper strategy, materials, purging or delay for analysis may lead to biased information on hydrogen fuel quality. There is a high risk that the results received by consumers are not representative of the actual hydrogen dispensed into the FCEVs due to improper sampling. Standardisation bodies (ISO TC 197) need technical evidence to revise the standards (ISO 19880-1). (objective 3)
- HRSs operators need to install sensors that can continuously monitor key contaminants to ensure that such contaminants never reach the FC vehicles; some instruments have been tested in laboratories during the previous EMPIR project 16ENG01 but the sensors and analyser were never validated in real life conditions at HRS. (objective 4)
- A better understanding of the impact of contaminants on stacks is needed by automotive manufacturers, tier 1 suppliers, research institutes, hydrogen producers and normalisation committees to ensure the lifetime of FCEV. Studies based on using hydrogen recirculation loop produce more reliable data on effect of contaminants on FC stack. However, there is no harmonised methodology which may lead to unreproducible results or biased interpretation. As the current ISO 14687:2019 (on

Hydrogen fuel quality — Product specification) threshold values are based on such previous studies, it is vital to harmonise this methodology. (objective 4)

Additionally, there are no recommendations or good practice guides for European regulation bodies for the verification of HRS with respect to handling, duration and long-term behaviour.

Objectives

The overall aim of the project is to develop a broad underpinning metrological infrastructure to support the European hydrogen industry.

The specific objectives of the project are:

1. To develop a metrological framework for testing hydrogen meters used to measure the mass of hydrogen dispensed into light-duty to heavy-duty fuel cell electrical vehicle (FCEV) from HRS, traceability will be assured by developing new primary standards (< 0.5% accuracy) or secondary standards (< 2% accuracy) traceable to the primary standards of previous EMPIR project 16ENG01 MetroHyVe or newly developed primary standards. Guidelines and a good practice guide to ensure accurate measurements and minimised uncertainty due to hydrogen refuelling stations (HRS) design will be produced.
2. To develop reference materials (RM) suitable to monitor hydrogen quality laboratory performance and to undertake an inter-laboratory comparison on hydrogen fuel including all contaminants regulated in EN 17124:2018 and ISO 14687:2019. Guidelines for online analysers at HRS over long periods (including validation, calibration and quality control) and recommendations to ISO 21087:2019 for online analysers and sensors will be produced.
3. To develop and validate sampling equipment and procedures based on European and national projects feedback as well to harmonize proposals for hydrogen sampling methods with USA and Japan. Good practice guides on hydrogen sampling at HRSs (nozzle) and other locations (e.g. FCEV) to provide input for the revision of ISO 19880-1 will also be produced.
4. To develop standard test protocols, including reproducibility study and intercomparison evaluation for automotive fuel cell (FC) stack testing (with online analysis) to determine threshold limits of critical contaminants in hydrogen. Critical parameters for FC stack testing with anode recirculation loop will be defined. A good practice guide on the measurement of impact of contaminant on FC stack (with online analysis) with detailed description of parameters to monitor will be developed.
5. To facilitate the take up of the technology and measurement infrastructure developed in the project to the hydrogen industry including the measurement supply chain (such as hydrogen quality laboratories, instrument manufacturers), standards developing organisations (ISO/TC 197, CEN/CENELEC and IEC TC105) and end users (hydrogen fuelling stations, vehicle manufacturers).

Progress beyond the state of the art

Flow metering: Currently there are more than four primary standards installations in Europe (including one at METAS and one at JV) for calibrating hydrogen flow meters to be used at HRSs for light-duty vehicles. METAS and Cesame have demonstrated calibration procedures for monitoring the amount of hydrogen dispensed into the vehicle up to pressures of 875 bar (required for stations providing hydrogen at nominal working pressure (NWP) of 700 bar. The current primary standards are compliant for fuel delivery to light-duty vehicles according to the Society of Automotive Engineers (SAE) J2601 protocol but an international field comparison for clear acceptance by the HRS operators and notified bodies is missing. The implementation of heavy-duty vehicles such as buses and trucks into the hydrogen infrastructure requires primary standards that are capable of going up to higher flow rates and therefore require a dedicated standard, which is currently not available. The project will go beyond the state of the art by developing a gravimetric flow method to calibrate and verify flow meters with hydrogen that are compatible for heavy-duty vehicles. The project will also go beyond the state of the art by developing and demonstrating the traceability chain from primary standards to secondary standards for regular verification of small and light-duty vehicles as well as for heavy-duty vehicles. Additionally, regular monitoring of HRS with different designs using the already available gravimetric standards from the previous EMPIR project 16ENG01 MetroHyVe will allow a firm set of data to be established which can provide recommendations for the periodicity of verification.

Hydrogen quality control: European laboratories are developing the capability to measure the contaminants specified in ISO 14687:2019 and EN 17124:2018. However, the current lack of gas calibrants with sufficient uncertainty, reference materials (RM) for validation and inter-laboratory comparison scheme is a barrier for

European commercial laboratories to be able to prove their agreement to ISO 21087:2019. This project will go beyond the state of the art by developing the gas calibrants and the RMs that European laboratories need to validate their analytical methods according to ISO 21087:2019. In the previous project 16ENG01 MetroHyVe a hydrogen quality inter-comparison was performed with 4 of the contaminants specified in ISO 14687:2019. This project will go beyond state of the art by developing a new inter-comparison involving all 13 contaminants in ISO 14687:2019. The previous EMPIR project 16ENG01 MetroHyVe validated 5 online gas analysers in laboratory-based conditions. However, such online hydrogen analysers and sensors have yet to be validated and tested at HRSs to ensure that their performance meets the criteria for implementing hydrogen quality control strategies as suggested in ISO 19880-8. This project will go beyond state of the art by testing and validating online hydrogen purity analysers and sensors at HRSs as part of quality control strategies and will provide metrological guidance to support accuracy and traceability of such measurements.

Sampling: Representative sampling of hydrogen is critical and currently some contaminants such as H_2S or water are hidden or enhanced by inappropriate sampling strategies. The previous EMPIR project 16ENG01 MetroHyVe started investigating the stability of some contaminants, such as CO or H_2S in commercially available sampling vessels. However, the number of sampling cylinders and additional contaminants such as CH_2O, CH_2O_2 or NH_3 requires additional studies. This project will perform robust tests to evaluate reactive compounds stability in commercially available sampling vessels and in state-of-the-art sampling vessels. The sampling vessel is only a part of the sampling strategy and three sampling methods at an HRS nozzle have been recently reported in ISO 19880-1 annex K. However, the sampling methods were not validated or compared, which has raised concerns on sampling equivalence worldwide. The project will go beyond the state of the art by performing the first sampling inter-comparison of all the European hydrogen sampling strategies at the HRS nozzle including US and Japanese approaches.

FC stack testing: Harmonisation of automotive FC stack testing is needed for the assessment of their performance and durability under operating conditions (including reproducibility issues) and hence of their technological progress. The project will go beyond that state of the art by providing the first guideline for FC stack testing with a recirculation loop. The project will test the reproducibility of FC stack testing and highlight the critical parameters for their performance, as well as harmonising the test protocol.

Results

Objective 1 – Development of a metrological framework for testing hydrogen meters used to measure the mass of hydrogen dispensed into light-duty to heavy-duty fuel cell electrical vehicle (FCEV) from HRS

The consortium is progressing with the development of two first primary standards for heavy-duty vehicle (35 and 70 MPa) with completion expected in summer 2022. This activity included to evaluate uncertainty budget for heavy-duty primary standard. The results indicate that temperature stability for buoyancy correction is key to accurate measurements. The key comparison of flow metering primary standards started in Autumn 2021 and will continue during the first half of 2022. The field measurements campaign started at commercial HRSs and will continue for the next year to gather data on stability of the meter and establish verification periods. A method for field verification of HRS using calibrated master meter progressed significantly and will be shared in 2022. An uncertainty model for HRS dispensers to be used by HRS designers and notified bodies to better understand uncertainty sources will be available in 2022.

Objective 2 – Develop reference materials (RM) suitable to monitor hydrogen quality laboratory performance

The consortium published an open access report summarising the results of a survey on the requirements of gas calibrants for hydrogen fuel quality (available on MetroHyVe 2 website). The partners have planned and started the testing of three new multi-contaminant gas mixtures in hydrogen in a large variety of gas cylinders. The mixtures cover contaminants found in real hydrogen fuel samples and detailed in ISO 14687. The stability study will last for at least 12 months. The partners started the stability study for formaldehyde and ammonia in hydrogen matrix in different gas cylinder and preparation to develop new calibrant gas suitable for hydrogen purity laboratory. The partners are currently preparing the interlaboratory scheme covering eight contaminants (specified in ISO 14687) in hydrogen fuel. It is the most ambitious interlaboratory comparison worldwide as it involves production of complex gas mixture in batch (14 equivalent gas mixtures), largest number of contaminants in gas cylinder (8 compounds) including reactive compounds at ISO 14687 threshold (i.e. 4 nmol/mol of sulphur compounds). Currently 12 laboratories (including commercial laboratories) from Europe, North America and Asia registered for the intercomparison that will take place in 2022.

Guidelines for online analysers at HRS over long periods

A first peer review article on sensor for hydrogen fuel quality was published (https://doi.org/10.3390/pr10010020) highlighting the challenges of using low-cost sensors. Partners studied the performance of two sensors for water in hydrogen in laboratory condition. Partners, instrument providers and partners owning HRS started organising the installation of the online analysers at HRS. Modification of HRS to accommodate sensors and online analysers is under preparation with a plan for installation in 2022.

Objective 3 - Development and validation of sampling equipment and procedures

Partners published an extensive peer-review article on sampling containers, sampling equipment and methodologies used for hydrogen fuel sampling (https://doi.org/10.1016/j.ijhydene.2021.08.043). The partners have selected more than six types of cylinders for the testing covering most types of cylinders used in Europe, Japan and US. The study of reactive compounds behaviour in those cylinders over time started and will carry on in 2022. The partners achieved bilateral sampling comparison between Europe and US approach (in collaboration with ASTM). Two joint samplings at HRS in USA were performed by US representative and project partner in 2021. The analyses are in progress. The sampling inter-comparison in Europe and Asia are expecting to happen 2022 following delay due to travel restriction. The partners have updated the good practice guide for representative sampling and analysis of particulate matter in hydrogen fuel dispensed from HRS (available on MetroHyVe 2 website). First repeatability study of particulate sampling at HRS has been performed in 2021. First FCEV contamination was realised by partner to evaluate the representativity of sampling from FCEV. The first results will be used to improve the methodology of sampling. A new infrastructure to test the contaminant behaviour in FCEV tank has been implemented by project partner and the first experiment were performed on real tank to evaluate the methodology. As a new methodology for sampling contaminants, sorbent tubes were successfully tested at high pressure (few bar) for capturing and transporting very low-level reactive impurities.

Objective 4 - develop standard test protocols, including reproducibility study and intercomparison evaluation for automotive fuel cell (FC) stack testing

The consortium is finalising an extensive literature review and first comparison to evaluate their state-of-the-art systems. Partners have chosen 4 different stacks, which will support the further applicability of the recommendations to be formed. The measurements, comparison and evaluation started in the autumn of 2021 and will continue to the end of 2022. The study on clean-up protocol of fuel cell pipes after exposure to contaminant (inert, hydrocarbons, reactive species) in hydrogen has started. Finally, a new study on adsorption/desorption of reactive compounds (i.e. ammonia) started in a hydrogen fuel cell system in correlation with online measurement (i.e. FTIR) at partner laboratories.

Impact

The key dissemination activities of the project during the first 18 months were its contribution to SDO with participation in meetings for ISO TC 197 WG 24, 27 and 28, ASTM D03 and CEN TC268 and CEN/CLC JTC6. In addition, the partners supported the creation of a new ISO TC197 WG 33 for hydrogen sampling standardisation (convened by partners in this project) which was kicked off in 2021. The consortium was involved in international committees ISO TC197 WG 27 and WG 28 providing technical inputs. Two peer review articles were accepted in the International Journal of Hydrogen energy and Processes.

The project partners have also presented the importance and relevance of hydrogen fuel quality measurements during European and international workshops (Mission Hydrogen workshop 2021, ASTM and Hydrogen Europe events) and international conferences (World hydrogen technical conference, Conference international Metrology, Hypothesis). The project partners setup the MetroHyVe 2 project website. Technical reports and good practice guides (5 reports available for download) have been freely available on the website for the Hydrogen community.

Moreover, the project emulated research activities in related field. For example, a partner studied heat transfer during the refuelling of a hydrogen tank numerically and experimentally (submitted in peer review journal).

Impact on industrial and other user communities

The project will produce validated primary standards for type-approval and initial verification of HRS, which will be made available to HRS operators. A new traceability chain for secondary standards allowing quick and affordable verification of HRS for both light- and heavy-duty FCEVs will also be developed by the project. Additionally, new measurement capabilities to allow HRSs to charge customers correctly when refuelling heavy-duty FCEV will be made available as a result of the project. (Objective 1)

Commercial gas analysis laboratories and instrument providers will be able to demonstrate ISO 14687:2019 capabilities in compliance with ISO 21087:2019 using the RMs and gas calibrants developed in the project.

This will allow such commercial gas analysis laboratories and instrument providers to demonstrate they are competent and able to provide traceable measurements for hydrogen fuel quality. (Objective 2)

HRS operators can have the confidence to install and maintain accurate online hydrogen analysers and sensors. Therefore, HRSs will be able to obtain reliable hydrogen fuel quality analyses using external laboratories or online measurements to show compliance with ISO 14687:2019 and ISO 19880-8 as requested by EU Directive 2014/94/EU. (Objective 2)

Correct sampling including suitable sampling vessels, suitable equipment, procedures and delay for analysis can be followed by HRS operators and applied by analytical laboratories. It will support confidence of end-users and ensure lifetime of FCEVs. Bilateral comparison between EU and ASTM approaches was performed in 2021. (Objective 3)

Harmonised stack testing will support the FC industry, automotive manufacturers and suppliers in developing improved stacks in terms of their lifetime or the impact of contamination on them. The project's results will also help to potentially identify new types of stacks for the next FCEV generation. (Objective 4).

Gas analyser and sensors manufacturers will have access to the relevant standards/guideline needed to validate and operate their instruments at HRS.

The results will provide industry with increased confidence in the measurements from European laboratories and support the enforcement of hydrogen quality regulations as stated in EU directive 2014/94/EU on the on the deployment of alternative fuels infrastructure

Impact on the metrology and scientific communities

Primary standards to verify hydrogen flow meters under real conditions using 875 bar hydrogen will be made available by the project for a large spectrum of vehicles (light and heavy-duty) and high flow stations. Data sets for the establishment of recommendations for the periodicity of verification of HRS based on their design will also be developed by the project. NMIs and DIs, using the primary standards developed in this project will be able to provide traceability to industry secondary standards for flow verification and claim CMCs.

New reference materials, primary gas mixtures and certified gas mixtures will be made available to analytical laboratories, sensor, instrument manufacturers to develop the quality control chain for the hydrogen fuel industry (HRS, automotive manufacturers).

Electrochemistry laboratories and automotive industry will be able to perform reproducible stack test with recirculation loop using harmonised protocol. It will allow comparison of results obtained in Europe and between institutes and industry and potentially support quicker identification of the new type of stack for the next FCEV generation.

The project will start and promote international collaboration and comparison on hydrogen quality (sampling and analysis between Europe, Asia and US) to support the global hydrogen economy.

Considering the target of 4000 HRS in EU by 2030, the use of primary standard to perform HRS verification is impossible to achieve. The secondary standards developed in this project will enable existing flow calibration laboratories to start providing a calibration service for the secondary standards needed by hydrogen industry enabling the requested number of verifications for European and local requirements.

Impact on relevant standards

The project will support the revision of OIML R139 with technical evidence and guidelines on calibration, achievable uncertainty, flow metering for light and heavy-duty vehicles at hydrogen refuelling stations and verification periodicity.

The good practice guides on handling of sampling containers and hydrogen quality sampling procedures/methods will support the drafting of new sampling standard for hydrogen at HRS nozzle ISO 19880-9 under ISO/TC 197/WG 33. This will include equivalent sampling approaches at the hydrogen stations, sampling vessels and delay for analysis.

The harmonised test protocol on the reproducibility of FC stack as well as the recommendations and good practice guide will be provided to ISO/TC 197/WG27 to revise the ISO 14687 on hydrogen fuel standard contaminant limits. Moreover, as the ISO 14687:2019 threshold values are based on such studies, reproducibility and harmonised test protocol will support the WG in revising the standard with reliable technical results. This work will be valuable to support the revision of IEC 62282-7-1 for FC stack testing with anode gas recirculation within IEC TC105.

Proposal for guidelines on online analyser and sensor validation and quality control at HRS will be provided to the ISO TC197 WG28 and ISO TC 158 JWG7 (for revision of ISO 21087:2019 and ISO 19880-8)

The project will present the good practice guide for sampling and suitability of sampling vessels with CEN/CLC/TC 6. It will support CEN/CLC/TC 6's decision whether ISO 19880-9 can be adopted at CEN level.

The project will provide reports on hydrogen contaminants measurements, quality control of offline laboratory and online sensor and analyser, sampling strategies and recommendations to CEN/TC 268/WG 5 to support the revision EN 17124:2018. Finally, this project will also support the revision of IEC 62282-7-1 for FC stack testing with anode gas recirculation.

Longer-term economic, social and environmental impacts

HRS operators and notified bodies will have access to several internationally recognised primary standards for initial verification and type-approval as well as traceable secondary standard for flow verification according to OIML recommendation. Considering the deployment of 4500 HRS in Europe by 2030 compliant to local and European regulations, the project outcomes will support significant reduction in the cost and time of flow verification while ensuring accuracy and traceability.

The deployment of the accurate and traceable European hydrogen quality laboratories will help to prevent serious damage to FCEVs which would be costly for the automotive manufacturers and fleet operators. Considering the European target of 1 million FCEVs in 2030, the reduction in risk of FCEV damage due to low hydrogen fuel quality is a critical cost saving for fleet operators (estimated to be millions of euros).

The implementation of quality control using online hydrogen purity analysers and sensors will reduce the need for subcontracting regular hydrogen purity analysis by a commercial laboratory which will have an important cost reduction in the quality assurance of hydrogen. This will result in lowering the price of hydrogen compared to conventional fuel and will be beneficial to the end users, FCEVs owner and the automotive manufacturers.

Through the support into FCEV reliability, HRS fuel quality and cost reduction, the number of FCEVs will increase and become more visible to society. This will enhance acceptance of FCEVs as "normal" road vehicles rather than a prototype or small fleet. The societal acceptance of hydrogen fuel is essential to achieve the energy transition toward a greener society. In addition to supporting climate change targets, introducing taxis fleet, hydrogen bus and heavy-duty vehicles to market will improve cities air quality and provide health benefits as only water is emitted at the tailpipes; this will prevent people from breathing in toxic carbon dioxide and carbon monoxide emissions and reduce the frequency of pollution peak.

This project will continue to support the expansion of hydrogen vehicles. Replacing conventional petrol vehicles will help Europe reach the challenging emission targets. Deployment of hydrogen for transport will support the growth in hydrogen production using renewable energy such as solar or wind to decrease the entire emission footprint of the sector. The emissions from hydrogen vehicles (i.e. buses, vans, trucks, taxis) is safe for health and will help reduce urban pollution and pollution peak responsible of hundreds of thousands death per year.

The standards and methods developed in the project will ensure that FCEV and heavy-duty vehicles can go to market without hindrance from the technical specifications in European Directive 2014/94/EU. A quicker deployment of FCEVs fleet will help to reduce carbon dioxide emitted from cars especially freight which is crucial for reaching the very challenging climate change targets set at the Paris Climate Conference (COP21) and the new European commission target for 2050.

List of publications

[1] *Detection of Contaminants in Hydrogen Fuel for Fuel Cell Electrical Vehicles with Sensors—Available Technology, Testing Protocols and Implementation Challenges*, Arrhenius, K., Bacquart, T., Schröter, K., Carré, M., Gozlan, B., Beurey, C. and Blondeel, C, Processes, 10-1, page 20, DOI: 10.3390/pr10010020

[2] *Strategies for the sampling of hydrogen at refuelling stations for purity assessment*, Arrhenius, K., Aarhaug, T.A., Bacquart, T., Morris, A., Bartlett, S., Wagner, L., Blondeel, C., Gozlan, B., Lescornes, Y., Chramosta, N., Spitta, C., Basset, E., Nouvelot, Q. and Rizand, M, International Journal of Hydrogen Energy, 46-70, page 34839, DOI: 10.1016/j.ijhydene.2021.08.043

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

Project start date and duration:	01 August 2020, 36 months	
Coordinator: Dr Thomas Bacquart, NPL Tel: (+44) 20 8943 6652 E-mail: thomas.bacquart@npl.co.uk Project website address: https://www.sintef.no/projectweb/metrohyve-2/		
Internal Funded Partners: 1. NPL, United Kingdom 2. BEV-PTP, Austria 3. Cesame, France 4. JV, Norway 5. METAS, Switzerland 6. NEL, United Kingdom 7. RISE, Sweden 8. VTT, Finland	External Funded Partners: 9. Air Liquide, France 10. CEA, France 11. EMCEL, Germany 12. Empa, Switzerland 13. ENGIE, France 14. ITM, United Kingdom 15. Linde, Germany 16. SINTEF, Norway 17. ZBT, Germany	Unfunded Partners: 18. TME, Belgium
RMG: -		