Publishable Summary for 16ENG01 MetroHyVe
Metrology for Hydrogen Vehicles

Overview

This project in great demand by the hydrogen industry such as hydrogen vehicle manufacturers including BMW, Daimler and Toyota, hydrogen refuelling station (HRS) operators, gas producers and standardisation bodies. A large hydrogen infrastructure is currently under development across Europe. However, the hydrogen industry is required to meet certain measurement requirements, set by European legislation that cannot currently be followed due to the lack of available methods and standards. This project will therefore address these issues and will develop metrology that will support the safe use of hydrogen in refuelling stations for the transport sector. The project will also be the first large scale project of its kind to tackle such measurement challenges in hydrogen flow metering, hydrogen quality assurance, hydrogen quality control and hydrogen sampling.

Need

In order to determine the main measurement needs for hydrogen industry, a survey was undertaken by NPL to understand the measurement challenges that this industry currently faces. The survey involved key stakeholders of the hydrogen industry including hydrogen producers, station operators, automotive manufacturers and standardisation bodies and the results clearly demonstrated that there are four key technical measurement challenges that prevent a hydrogen economy from growing in Europe:

- It is not currently possible to accurately calculate the amount of hydrogen dispensed when filling hydrogen into a fuel cell vehicle and therefore customers are not able to be charged correctly when buying hydrogen from HRS.
- Hydrogen provided by the refuelling stations will need to meet the hydrogen purity specifications of ISO 14687/EN 17124; however no laboratory in the world can currently perform all of these measurements under accreditation.
- HRSs will typically need to install instruments that can continuously monitor key impurities online to ensure harmful impurities do not reach the fuel cell of vehicles; these instruments are currently in development but have not yet been tested or validated.
- To date there are no verified techniques that can be followed, or validated sampling vessels available, for when HRSs sample hydrogen and send it to laboratories for purity analysis. Thus there is a high risk that the sample received by the laboratory is not representative of the hydrogen dispensed into vehicles.

Objectives

The overall aim of this project is to develop metrology that will support the safe use of hydrogen in refuelling stations for the transport sector. The specific objectives are:

1. Flow metering – To develop a metrological framework for testing hydrogen meters used to measure the mass of hydrogen dispensed into a fuel cell vehicle from a refuelling station and support laboratories by providing a good practice guide describing the calibration and validation of flow meters used at HRSs. The metrological and technical requirements stipulated in OIML R 139-1 and international standard SAE J2601 - Fuelling Protocols for Light Duty Gaseous Hydrogen Surface Vehicles should be followed, with a target accuracy of 1 %.
2. Hydrogen quality assurance – To support hydrogen purity testing as specified in ISO 14687 by developing traceable offline gas analysis methods, stable and accurate primary reference gas mixtures and the metrological tools to enable the introduction of low cost gas analysers suitable for use by

commercial gas analysis laboratories. An interlaboratory comparison will be held to allow commercial laboratories to prove their competency in hydrogen purity testing which will allow them to gain evidence for obtaining accreditation for this service. In addition, to develop a robust method for accurately performing online measurement of particulates (to determine whether levels are above or below 1 mg/kg) in hydrogen provided at the refuelling station, as specified in ISO 14687.

3. Hydrogen quality control – To perform purity measurements of hydrogen following the implementation of quality control techniques specified in ISO 19880-8 and validate continuous online hydrogen purity analysers for measuring canary species (the key impurities that guarantee global quality of the hydrogen) at the HRS. A good practice guide will be developed for the use and calibration of hygrometers for online water analysis. Low cost sensors will be investigated for use at HRS for performing online monitoring of impurities for quality control.
4. Sampling – To develop a robust protocol for taking a representative sample of hydrogen gas from a refuelling station and testing suitability of high pressure sampling vessels for delivering hydrogen to gas analysis laboratories for offline purity analysis; as required by ISO 14687.
5. Impact – To facilitate the take up of the technology and measurement infrastructure developed in the project by the measurement supply chain (accredited laboratories, instrument manufacturers), standards developing organisations (ISO, CEN/CENELEC) and end users (hydrogen industry, vehicle manufacturers and suppliers).

Progress beyond the state of the art

The current state-of-the-art for the objectives above and the outcomes from this project to go beyond this are:

Flow metering – Currently there is no capability for calibrating hydrogen flow meters to be used at HRSs for monitoring the amount of hydrogen dispensed into the vehicle up to pressures of 875 bar (required for stations providing hydrogen at nominal working pressures of 700 bar). The project has already developed a gravimetric methods (Primary Standards) to calibrate and verify flow meters with hydrogen at a nominal working pressure of 700 bar, as well as new methods which allow non-flammable substances to be used to calibrate these flow meters such as nitrogen, air or water.

Hydrogen quality assurance – Existing hydrogen purity laboratories are unable to provide traceable ISO 14687 hydrogen purity measurements because the methods and standards have not been developed for all of the impurities specified. The project has begun to develop the missing methods and standards, and if it is not possible to develop a particular measurement a statement will be provided to ISO TC 197 recommending a revision of the ISO 14687 standard and the proposed alternative measurement that should be included instead. A new requirement in ISO 14687 (as of 2019) is that all methods must meet the criteria set in ISO 21087 and therefore the project has included an additional activity to assess all new methods against this standard.

Hydrogen quality control – Although an offering of online hydrogen purity analysers should be possible through the latest developments in spectroscopic technologies, these instruments have not been tested and validated using traceable standards to ensure that the performance meets the criteria for implementing hydrogen quality control strategies as suggested in ISO 19880-8. Project partners have now developed and validate three online hydrogen purity analysers utilising different measurement technologies which can be installed at HRSs as part of quality control strategies. A comparison will take place at NPL to test performance of five such online purity analysers.

Sampling – The issue of correct sampling is a common topic among the hydrogen industry as it is possible that samples of hydrogen contain impurities (such as water and air) caused by bad sampling techniques. The project performed robust tests on sampling (both particles and gas) in order to develop a good practice guide which will provide a protocol for correct sampling at HRS and selection criteria for sampling vessels.

Results

Flow metering

Partners have surveyed seven different hydrogen refueling station operators to understand their different operating conditions and where in the system their flow meter is situated. This has helped the project's understanding of the suitable maximum permissible error for hydrogen refueling stations. A report of these findings has been written and following a final review will be sent to the convener of OIML TC8 SC7 to support a revision of OIML R139-1 (which is the current international recommendation for flow metering at the hydrogen refueling station). Primary gravimetric flow metering standards have also been produced and validated as part of this project (Figure 1); these mobile facilities are capable of calibrating flow meters in-situ at hydrogen refueling stations at the extreme operating conditions expected during refueling (700 bar).

Figure 1: The new Primary Standard built by METAS

Hydrogen quality assurance

A literature review is underway to identify new methods for measuring impurities in hydrogen as required by ISO 14687-2. The last literature review of this kind was performed in 2015 meaning that new methods should be available over the past three years. Method development is ongoing at project partners CEM, AP2E, IFE, NPL, RISE, SINTEF and VSL focusing on analytical techniques that can measure low level reactive impurities. In an effort to ensure the project remains up to date with outside activities, all activities involving development of ISO 14687 methods was updated following the development of ISO 21087 which provides criteria for accepting ISO 14687 methods. All partners have agreed to perform additional work to compare their methods against ISO 21087. Further to this, a hydrogen impurity enrichment device has been tested for the first time with low level sulphur, and the results have indicated that a palladium-gold membrane (which was expected to be suitable for this technique) was in fact not suitable and led to severe reduction in hydrogen flux. Ten new membranes have now been prepared (more than the two originally specified) and the best performing membrane will be used to enrich a hydrogen sample taken from a UK HRS. A method for filter weighing (during analysis of particles in hydrogen) has also been developed and validated. Following support from stakeholders a proposal was made for a low cost system of analysers to cover key impurities from ISO 14687. This is now in development between RISE and AP2E and combines Gas Chromatography methods with optical feedback cavity enhanced absorption spectroscopy. Hydrogen mixtures are currently being prepared for the MetroHyVe Offline Comparison which will assess 13 laboratories from around the world (Europe, Asia and USA) in performing hydrogen purity analysis against ISO 14687.

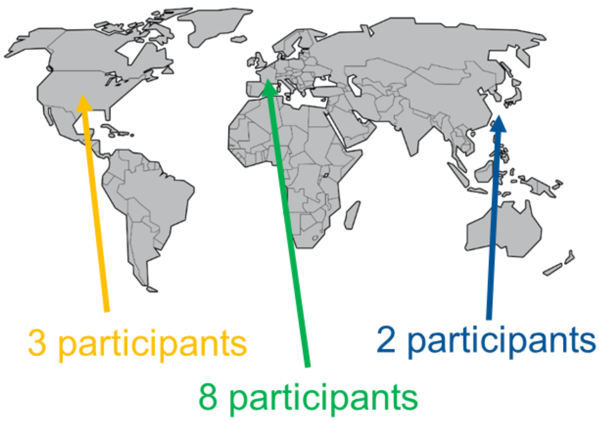

Figure 2: Location of confirmed participants of the MetroHyVe Offline Comparison

Hydrogen quality control

Three partners (Shell, Cascade Technologies and AP2E) have developed online instruments for measuring key impurities and are completing testing against gas mixtures containing low level hydrogen sulphide, carbon monoxide, oxygen and water in hydrogen which were produced by NPL. The project has received several hygrometers from external collaborators for testing at NPL's humidity facilities which will be complete in early 2019. A high pressure particle atomiser is also being built at NPL that will be used to produce particles in hydrogen mixtures for testing online particulate analysers. Further to this, a scoping exercise is underway to determine the important factors that define a good low cost sensor for monitoring impurities at a HRS. The scheme for an intercomparison for online purity analysers has been formulated and is being planned to take place at NPL; already five participants (Shell, Cascade Technologies, AP2E, Tiger Optics and Protea) have been selected who will be providing their own commercial online analysers.

Figure 3: The high pressure particle atomiser will generate the first particles in hydrogen standard

Sampling

Hydrogen sampling was performed at the HRS at NPL. During sampling, a few samples were taken following different numbers of purge cycles to test how many purges are required to remove water and air contaminants. The sampling has now incorporated testing of commercial sampling devices for gas sampling (Linde Qualitiser) and particle sampling (HyDAC). A teleconference was hosted by IFE to discuss how to contaminate IFE's HRS with carbon monoxide and hydrogen sulphide and a plan has been made going forward to purchase all parts required for this. Developing such a facility will for the first time allow assessment of a laboratory to provide a full sampling and analysis service (where traceability comes from the station's ability to accurately blend impurities in the hydrogen). A selection of sorbent tubes to test for sampling directly at a HRS has also been made.

Figure 4: Testing a commercial hydrogen sampling device

Impact

To ensure the project activities remains aligned with stakeholder needs, the consortium has built up a stakeholder advisory board, with around 50 members on it, including vehicle manufacturers, gas producers and laboratories. Representatives of key standardisation committees are also included in the stakeholder advisory board, including experts and chair persons.

Several dissemination activities have occurred which include a keynote speech on the project at the World Hydrogen Energy Conference in Brazil (2018), 9 other conference presentations, presentations at standardisation committees including a talk on this project at the plenary meeting for ISO TC 197 Hydrogen Technologies. Now that the project has produced some key results that need to be disseminated to the hydrogen community, the project is organising training sessions and workshops to be held in 2019 and 2020.

Impact on industrial and other user communities:

- The activities and reports produced by the project are regularly being disseminated to OIML TC8 SC7, and this will impact OIML R139-1 which provides maximum permissible errors for flow metering at HRS.
- The sampling campaign performed in this project proves that there is a certain minimum number of purges that is required to remove air and water contaminants from sampling devices. The results are being written into a report which will guide HRS operators through this process. The impact is significant as HRS need to be able to sample without contaminating it otherwise laboratories will provide test reports showing that their hydrogen fails to meet ISO 14687.
- Online instruments have been developed to measure key impurities in hydrogen and validation is underway against primary reference materials. Once this is done, the instrument manufacturers will be able to show HRS clear evidence that their instruments work.
- The offline comparison will robustly assess commercial hydrogen purity laboratories in their ability to accurately provide hydrogen purity measurements as specified by ISO 14687. The good practice guide will be produced by partners in assessing their purity methods against ISO 21087 and will be disseminated in an effort to support other laboratories.
- An online virtual measurement service hub (*via* a website) has been created at NPL and has started to list laboratories and their hydrogen capabilities. The hub will allow end-users to easily find and access these laboratories.

Impact on the metrology and scientific communities:

- Four primary gravimetric standards are currently being built to verify hydrogen flow meters under real conditions using 875 bar hydrogen.
- New calibration gas standards are being made that will have improved stability for low level reactive impurities in hydrogen. Gas producers will be able to use these to certify their calibration standards for all impurities in ISO 14687 to ensure they are providing traceable values.
- A selection process is underway to pick suitable sorbent tubes and gas vessels for different types of impurities in hydrogen. Gas analysis laboratories will be able to use the project’s future report on this subject to select suitable sampling vessels for customers requiring their services.

Impact on relevant standards

- ISO 14687 has now been revised based on influence from MetroHyVe partners to remove ‘key halogenated compounds’ from the standard. This was due to the fact that no methods exist to perform this measurement, which was an activity of the project.
- ISO 21087 includes an updated review which was performed by NPL and took information directly from the MetroHyVe project regarding new analytical methods.
- ISO 19880-8 has yet to be revised, but MetroHyVe partners will ask to include an update on available online purity analysers.

- ISO 19880-1 has yet to be revised, but MetroHyVe partners will ask to include an updated appendix on sampling techniques including best practice gained from this project.

Longer-term economic, social and environmental impacts

The long term goal of this project is to support the introduction of hydrogen vehicles which if replacing conventional petrol cars can help Europe to reach its challenging emission targets.

Two instruments have been selected by this project that when combined will be able to provide full analysis of all key impurities of ISO 14687. In the longer term this will mean that gas analysis laboratories will be able to spend less on investment for analytical instruments in order to provide a hydrogen purity service. Development of hydrogen purity methods will also help to prevent serious damage to fuel cell vehicles. Such damage would be costly for the automotive manufacturers to replace and therefore its prevention will promote the introduction and manufacture of hydrogen fuel cell vehicles.

The implementation of online hydrogen purity analysers developed in this project will in the long-term reduce the need for contracting regular hydrogen purity analysis by a commercial laboratory with an important cost reduction in quality assurance of hydrogen. New players entering the hydrogen industry will also learn the importance of laboratory accreditation and how to prove competency through interlaboratory comparisons. Both developments will support the use of hydrogen as a fuel for fuel cell vehicles and will also provide suppliers and end-users with confidence in the quality of their hydrogen fuel.

<table>
<tr><td colspan="2">Project start date and duration:</td><td>June 1st 2017, 36 months</td></tr>
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1 NPL, UK
2 CEM, Spain
3 Cesame, France
4 Force, Denmark
5 JV, Norway
6 NEL, UK
7 RISE, Sweden
8 VSL, Netherlands</td><td>External Funded Partners:
9 Air Liquide, France
10 AP2E, France
11 CT, UK
12 FHA, Spain
13 IFE, Norway
14 ITM, UK
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