FINAL PUBLISHABLE REPORT

Grant Agreement number	16ENG01
Project short name	MetroHyVe
Project full title	Metrology for hydrogen vehicles

Project start date and duration:		1st June 2017 (36 months)
Coordinator: Arul Murugan (NPL) Tel: +44 20 8943 6382 E-mail: arul.murugan@npl.co.uk Project website address: www.metrohyve.eu		
Internal Funded Partners: 1. NPL, United Kingdom 2. CEM, Spain 3. Cesame, France 4. FORCE, Denmark 5. JV, Norway 6. NEL, United Kingdom 7. RISE, Sweden 8. VSL, Netherlands	External Funded Partners: 9. Air Liquide, France 10. AP2E, France 11. CT, United Kingdom 12. FHA, Spain 13. IFE, Norway 14. ITM, United Kingdom 15. Linde, Germany 16. NEN, Netherlands 17. SINTEF, Norway	Unfunded Partners: 18. Empa, Switzerland 19. METAS, Switzerland 20. Shell, Netherlands
RMG: -		

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1 Overview

This project was in great demand by the hydrogen industry, such as by 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 previously could not be followed due to the lack of available methods and standards. This project was funded to address these issues and to develop new metrology infrastructure that aims to support the safe use of hydrogen in refuelling stations for the transport sector. The project was 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. The project provides the first traceable Primary Standard for flow metering to allow accurate calculating of hydrogen dispensed into vehicles from refuelling stations and development of new sampling and hydrogen purity methods as required by ISO 14687 to allow station operators to check that quality of hydrogen does not damage end-user fuel cell vehicles.

2 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 faced. 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 were four key technical measurement challenges that prevented a hydrogen economy from growing in Europe:

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

3 Objectives

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

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 hydrogen refuelling stations 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. Creating 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).

4 Results

The MetroHyVe project supported the introduction of hydrogen vehicles to European market by solving four key measurement challenges that was faced by the industry (flow metering, gas quality assurance, gas quality control and sampling). This section summarises the results of:

Objective 1 – Flow metering

Objective 2 – Gas quality assurance

Objective 3 – Gas quality control

Objective 4 – Sampling

Objective 1: Flow metering

Identifying and assessing the uncertainty sources for hydrogen metering

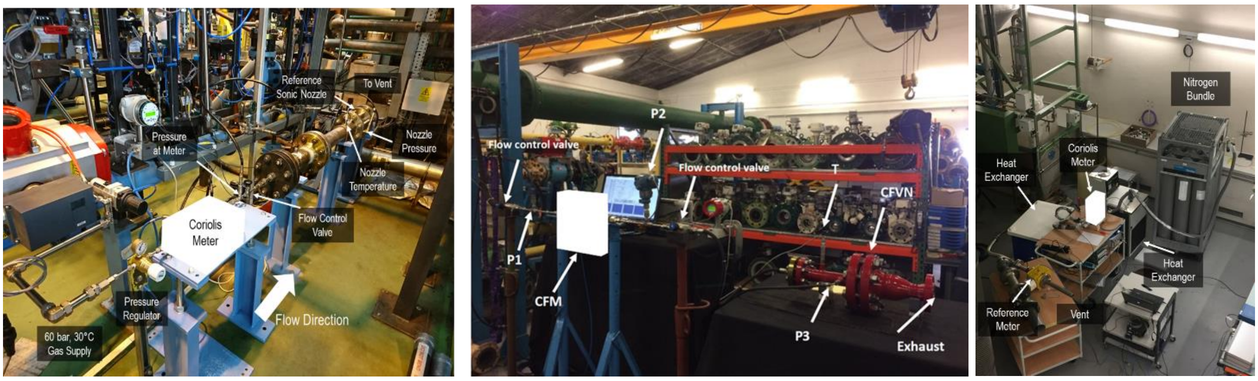
A survey designed and circulated by Cesame with the support of Empa, FORCE, JV, METAS, NEL, RISE and VSL was used to collect data from hydrogen industry regarding operational conditions of a hydrogen refuelling station. The survey included questions such as location, type and model of flow meter used, types of heat exchange and compression. The results of the survey are published in "Public report on operating conditions and uncertainty sources of a hydrogen refuelling station." The report also includes a loss of uncertainty sources and assessment on contributions to the measurement error. The report also includes an update on OIML R 139 recommendations which provides the maximum error of uncertainty that should be allowed in flow metering measurements at gas refuelling stations.

Investigate alternative methods for type approval testing using substitute substances to hydrogen

The aim was for NEL, Cesame, METAS and VSL to use gases other than hydrogen to perform approval testing of flow meters. This would allow laboratories to use non-flammable fluids (such as nitrogen or water) to perform calibrations. The work included testing the effects of temperature changes as would be expected during a refuelling. Four flow meters were tested which were all Coriolis type.

Testing showed that the largest errors occurred at the low flow rates, and at medium to high flow rates errors for most meters were within ±1 %. The work showed the potential of using alternative fluids to hydrogen to perform calibration in a safer manner.

Regarding influence of temperature, at stable conditions greater errors and wider spread of errors occurred at -40 °C compared to 20 °C. Errors of up to 10% were observed at the lowest flow rates, but within ±2 % at moderate to high flow rates. When the incoming gas was much colder than the meter, performance of the meter shifted significantly as temperatures stabilised. Errors ranged -15 to 15 % and depended more on the difference in temperature at upstream compared to flow meter, rather than the gas flow rate.



The flow laboratories from NEL, CESAME and METAS, respectively, that were used to perform the testing.

Investigation on the high-pressure dependence (up to 875 bar) of Coriolis mass flow meters

A novel high-pressure flow test facility was built at RISE and used to perform high-pressure measurements with high-pressure flow meters of three different brands: RHEONIK, HEINRICHS, KEM at ambient temperature in a pressure range between 5 bar and 850 bar using water as test liquid. Additional (low-pressure) calibrations need to be performed in order to correct for the temperature effect and hence to separate temperature and pressure effects. A complete data set regarding the influence of pressure on mass flow measurement accuracy for all three flow meters has been published.

Development of a gravimetric method to calibrate and verify HRS flow meters at 875 bar

METAS and CESAME performed field testing using gravimetric primary standards at 700 bar. It was shown that the gravimetric method was suitable for achieving the required accuracy of 0.3 % and could be used for type-approval testing of hydrogen refuelling stations. It was noted during testing that venting times (required to remove hydrogen gas from the standard following measurement) was long and limited the number of measurements that could be performed in a day. The evaluation of a station takes several days with the gravimetric standard, and therefore it was recommended that an alternative method to using gravimetric standards should be developed to reduce measurement time and costs (including labour).

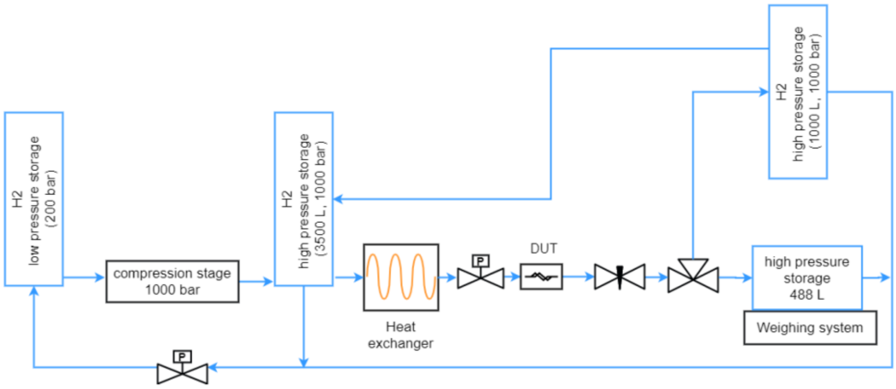
The master meter method was tested in comparison to the gravimetric method, and showed good results when tested in the warm zone including meeting requirements for a Class 4 station as defined by OIML R 139. In the cold zone, the master meter showed a large positive error (> 5 %) with low repeatability.

Uncertainty budget for the type approval testing, the periodic verification and gravimetric facility

Four reports were written providing assessments of uncertainties for using alternative gases or water to perform flow meter calibrations and using the gravimetric approach to calibrate flow meters at the refuelling station. A final report was provided on the requirements for developing a primary test bench to perform traceable calibration of hydrogen flow meters following technical requirements stipulated in OIML R139-1 and SAE J2601. In this document, the authors presented specifications and a tentative design for a primary calibration test rig for hydrogen flow metering up to 900 bar and its associated uncertainty budget. Based on the design, a bill of material was created which led to a cost assessment.

The estimated cost for developing a primary test rig for hydrogen flow metering up to 900 bar amounts to around 1.4 M€. This amount only covers the material needed for the test rig and does not take into account the needed infrastructure (e.g. building, parcel and technical requirements of the building). Neither it takes into account the cost in man month for building the test rig. It can be safely said that such an investment is most probably beyond the capabilities of a single national metrology institute and that a consortium would need to be created to develop, build and run such a test rig.

The key outputs of Objective 1 are: Developing new capabilities and facilities that could be used to accurately and traceably calibrate flow meters used to determine amount of hydrogen dispensed into vehicles during refuelling. This includes offline calibration in laboratories and online calibration directly at refuelling stations using mobile primary standards. This objective was successfully achieved.



Piping and instrumentation diagram for the primary test rig for high-pressure calibration

Objective 2: Hydrogen quality assurance

Validated analytical methods for measuring reactive compounds

A literature review was performed including all known methods that have been developed worldwide to perform quality assurance measurements for hydrogen in accordance with ISO 14687 Grade D. One of the more challenging methods was for measurement of low level halogenates; methods were developed by RISE (thermal desorption plus gas chromatography mass spectrometry, TD-GC-MS), VSL (cavity ringdown spectroscopy, CRDS), CEM (micro gas chromatography with thermal conductivity detector), IFE (gas chromatography with pulsed discharge helium ionisation detector, GC-PDHID) and NPL (impinger trapping followed by ion chromatography and TD-GC-MS). New methods were developed for measurement of formic acid, formaldehyde and ammonia using optical feedback cavity enhanced adsorption spectroscopy by AP2E, Fourier Transform Infrared Spectroscopy (FTIR) by Linde and CRDS by VSL. NPL used a range of methods to perform measurement of ammonia, formaldehyde and formic acid include GC-MS, FTIR, GC-PDHID, GC methaniser FID and selected-ion flow-tube MS (SIFT-MS). New methods for measuring sulphur compounds were tested using GC-PDHID (IFE) and OFCEAS (RISE).

Validated methods for performing traceable measurement of particulates

Testing was performed to understand sources of uncertainty for filter weighing used to calculate particle mass concentration in accordance with ISO 14687 Grade D. Initially a test was performed to identify potential mass contamination issues from filter preparation at the refuelling stations, and the results concluded that there would be none. A good practice guide was written for the handling, transporting and weighing of filters for this measurement which included a method for accurate measurement (building on expertise gained from the area of ambient air monitoring) and storage of filters. It was shown that by carefully controlling the temperature and humidity during filter weighing it was possible to decrease the measurement uncertainty by a factor of 10. The importance of selecting the right filter type and material was highlighted.

Primary reference gas mixtures and dynamic reference standards for low level impurities in hydrogen

An assessment of suitable passivation treatments for cylinders used to collect hydrogen with various impurities was performed. It was found that there was limited data in the literature to these types of results, and in some cases the passivation treatment appeared to not be reported. A report was written reviewing all known passivation treatments used for storing impurities in hydrogen. New gas standards and dynamic standards were prepared and developed, respectively, by CEM, NPL and VSL which will be used to provide high accuracy gas standards to support hydrogen purity measurements.

Optimisation of impurity enrichment devices

The aim was to support the development of NPL's Hydrogen Impurity Enrichment Device to allow it to be used to perform impurity enrichment using a real sample of hydrogen obtained for an operational hydrogen refuelling station. A new procedure for spiking krypton into a hydrogen sample was developed involving calculating of required krypton mass in correlation to expected hydrogen pressure. New compositions of membranes were tested with the aim of improving speed of the enrichment process whilst reducing adsorption of reactive species to the membrane.

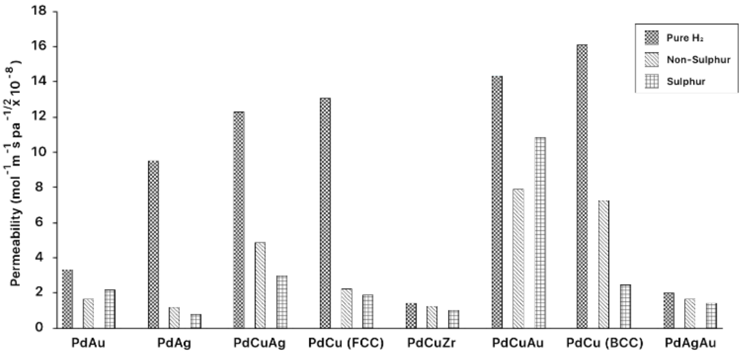


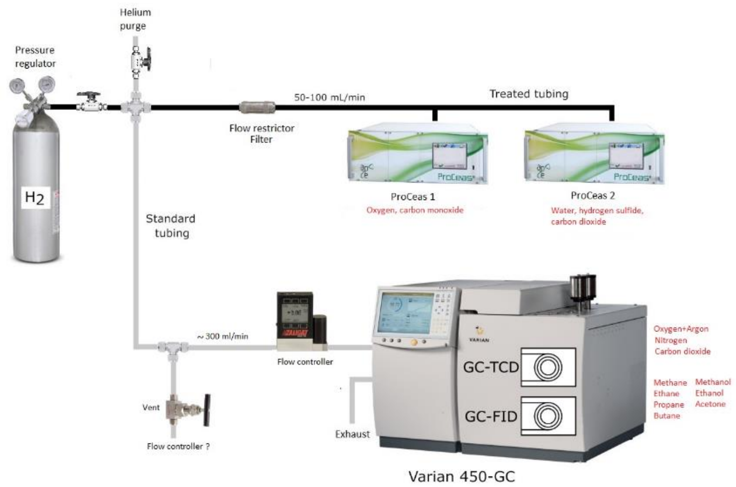
Figure 5.2: Permeability data for pure hydrogen, non-sulphur, and sulphur permeation tests

Composition of membranes tested in the Hydrogen Impurity Enrichment Device

Finally, impurity enrichment of a real sample of hydrogen taken from an operation hydrogen refuelling station was performed; the results showing that it was possible to further reduce limit of detection for analytical instruments through the device. In this case, an enrichment of 42 was successfully employed, but increase enrichment was possible by changing starting and finishing pressures as well as increasing volume of the sampling cylinder.

<u>A cost-efficient offline system for ISO 14687 purity analysis</u>

An analytical system combining gas chromatography with OFCEAS was developed to design a system that could simultaneously perform measurement of several impurities in hydrogen. As advised by the project's stakeholder advisory board, the scope of impurities for the analyser was reduced from full ISO 14687 Grade D specifications to only impurities relevant for the PEM water electrolysis.



Cost efficient offline analysis system for ISO 14687 Grade D developed by RISE

A peer-reviewed paper was published discussing development of the device.

<u>Interlaboratory comparison for ISO 14687 hydrogen measurements</u>

An offline interlaboratory comparison was hosted by NPL and VSL to test laboratories from Europe, Asia and USA in performing measurements of selected impurities in hydrogen according to ISO 14687 Grade D. The comparison involved 13 participants who were asked to measure water, nitrogen, carbon monoxide and hydrogen sulphide. Not all participants measured all impurities. A study on the reference materials prepared for the comparison showed that carbon monoxide and nitrogen were stable, whereas the water and hydrogen sulphides drifted downwards (i.e. decreasing amount fraction over time). However, a correction was applied to ensure the reference value for each participant was relevant.

The results of the comparison showed that not all participants could meet the reference value, possibly due to underestimating uncertainty or improper validation of the technique. It was noted that stability of the mixtures needed to be improved and delivery of mixtures to non-European countries took longer than expected.

The key outputs of Objective 2 are: To develop Primary Reference Materials (gas standards) and analytical methods that industry requires to perform accurate and traceable hydrogen purity measurements in accordance with ISO 14687. Methods were also developed to improve and reduce costs for hydrogen purity by investigating all-in-one analysers and hydrogen impurity enrichment. Additionally, the first worldwide comparison for offline hydrogen purity analysis was intended to demonstrate the importance of providing these kinds of testing schemes and provide the first example of how these schemes should be run in future. This objective was successfully achieved.

Objective 3: Hydrogen quality control

Online measurement of gaseous impurities

Three online analysers were developed by CT, Shell and AP2E to perform measurement of one or more impurities from water, carbon monoxide, oxygen and hydrogen sulphide in hydrogen. The instruments were successfully developed and joined in an online comparison.

The online analysers developed by AP2E, Cascade and Shell

Online measurement of humidity

Hygrometers are used to monitor levels of water in hydrogen refuelling stations for quality control; water must be kept below 5 µmol mol^{-1} to meet the specifications of ISO 14687 Grade D. NPL tested six commercially available metal-oxide hygrometers owned by collaborators including one in service in the ITM Power refuelling station at NPL used in H2 at 2 MPa provided by ITM. Tests were performed in nitrogen and hydrogen against the NPL multi-gas, multi-pressure Standard Humidity Generator including dew-point temperatures from -65 °C to -20 °C at pressures of 0.17 MPa and 2 MPa (20 bar) corresponding to amount fractions of water vapour from 0.5 to 50 µmol mol^{-1} at 2 MPa. Evaluation of drift was performed by repeated full measurement set after ~6 and ~12 months. The results which were reported in a good practice guide available to industry showed that there were significant drifts observed which would severely affect accuracy of the devices and could lead to water levels reaching above ISO 14687 Grade D threshold levels.

Online measurement of particulate mass concentration and sizing

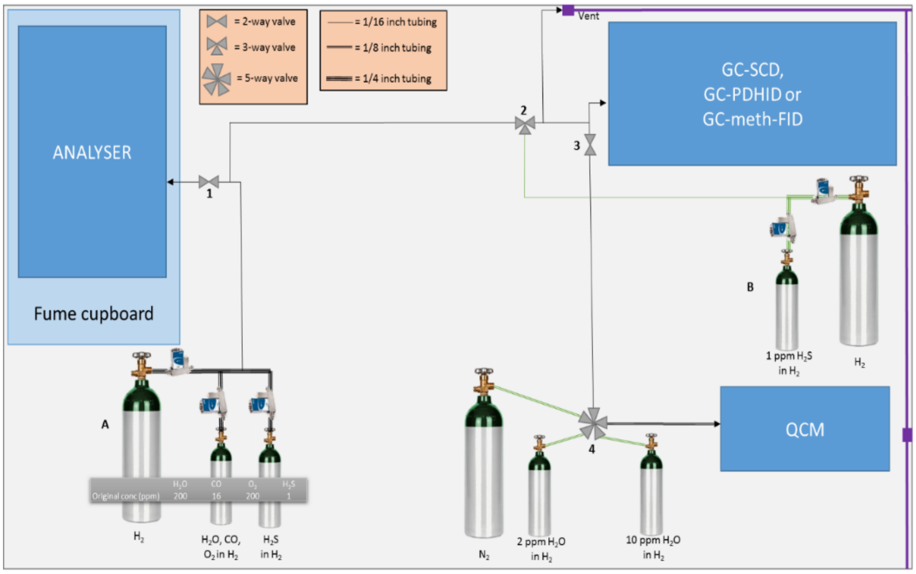
A Tapered Element Oscillating Microbalance (TEOM) was purchased to perform online measurement of particulate mass concentration. The instrument was used to directly measure particulates in a gas stream produced by an aerosol generator. A report indicating the results from the feasibility study was written up, where conclusions indicated that the TEOM could be used safely with hydrogen gas and the method to set this up was described. However, there were noted issues with the particulate generation method that needed to be addressed.

Low cost sensors for hydrogen purity

A report on low cost sensors was written for the target impurities of carbon monoxide, carbon dioxide, water and hydrogen sulphide in hydrogen. The report included results from a survey of suitable sensors, recommendations and testing of a selected sensor. The stakeholder advisory board gave initial feedback regarding the definition of "low cost" and the other parameters that would be key to develop a successful device. Three low cost sensors were purchased and tested at Air Liquide including the CO-B4 Alphasense, TFS3879 Figaro and Alphasense H2S-B4.

Intercomparison of online purity analysers

A comparison exercise took place at NPL to test the performance of five online analysers for measuring impurities (water, oxygen, carbon monoxide and hydrogen sulphide) in hydrogen.



Laboratory set up used by NPL to perform testing of online analysers

Z-scores were provided to all participants to show the accuracy of their instruments. Time response and range were also tested. Based on this comparison it was recommended that analysers are first tested by an independent party (e.g. in the form of a round-robin comparison). Some instruments seem satisfactory while others require improvements. Scope of the comparison was necessarily limited, but further extensive field tests were recommended before installing these instruments at refuelling stations (operation in the long-term, different weather conditions and determination of analyser drift, calibration and maintenance intervals etc).

The results of the comparison showed that there was a lack of traceable and stable reference materials in hydrogen for validation of hydrogen purity analysers (e.g. for sulphur compounds but also mixtures containing oxygen due to the observed reaction with hydrogen). There was a considered need for wide availability of such reference materials to support the world-wide uptake of hydrogen for fuel cell applications.

The results of the comparison (with participants anonymised) were published in a public report that is available from the project website.

The key outputs of Objective 3 are: To develop new online analysis instruments for the four key impurities from ISO 14687 that require quality control at hydrogen refuelling stations. Furthermore, the first worldwide comparison of online instruments was performed including testing of important parameters such as trueness, repeatability, drift and response time. Additionally, the activities looked at developing online particulate measurement solutions and the feasibility of low cost sensors. This objective was successfully achieved.

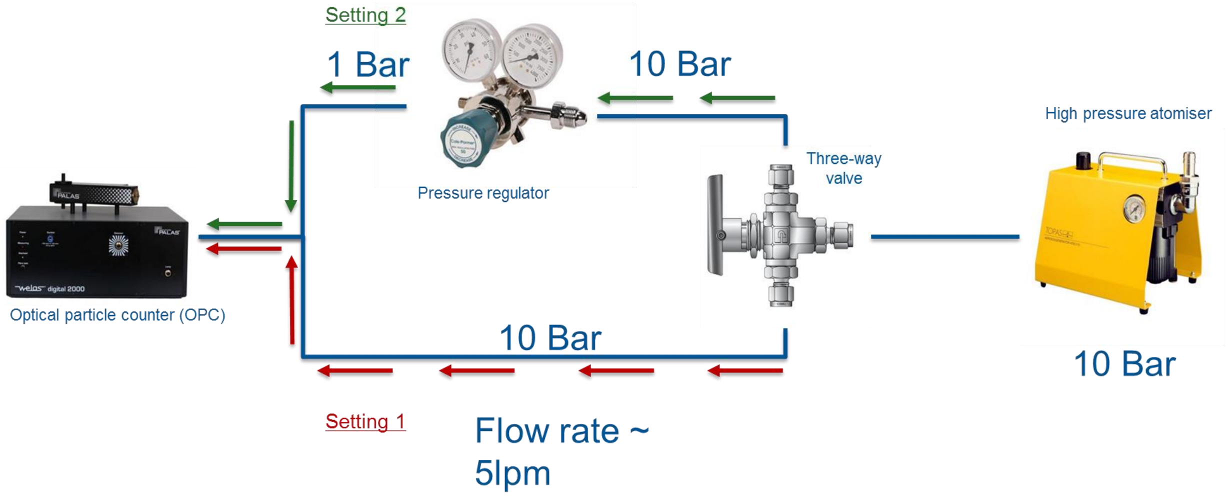
Objective 4: Sampling

Suitable sampling techniques at the hydrogen refueller

FHA with support from Air Liquide, ITM, RISE and NPL performed a survey to understand the sample requirements from the gas analysis laboratories and the working conditions at the refuelling station. Procedures for evacuating gas cylinder used for gas sampling was provided by NPL and SINTEF. The approach used by NPL included evacuation down to 1 x 10^{-7} mbar whilst monitoring for impurities in outgas. NPL also developed a method for purging the Linde Qualitiser before sampling by pre-filling the sampling cylinder with a small amount of ultra-high purity hydrogen at a slight overpressure. A correction factor was applied to account for any small dilution of hydrogen sample with the pre-filled hydrogen.

Validation of particulate sampling using filters

NPL performed a review of particulate sampling techniques and highlighted that the best option was the HYDAC PSA-H70. A study was performed to determine particulate mass loss through a pressure regulator. This was carried out by producing particulates in hydrogen using a high pressure atomiser and comparing the amount of particulates measured at an optical particle counter with and without a pressure regulator present.



Laboratory set up used by NPL to test whether presence of a pressure regulator removed particulates from the hydrogen stream

The results showed that the pressure regulator removed a large amount of the particulates in the stream, and therefore should not be used as a method for depressurising hydrogen before sampling for particulate mass concentration. A good practice guide was produced to provide guidance on sampling particulates at a hydrogen refuelling station.

Efficiency of sorbent tubes

Several sorbents were tested for trapping targeted compounds among sulfur compounds, hydrocarbons and halogenated compounds by monitoring recovery yields (at the time of the sampling) and the storage stability (2 weeks). The sorbents that were selected were:

- Chromosorb 106: good for very low BP compounds, Tenax TA: good for higher BP compounds
- Tenax TA/Carboxen1003/CarbographTD: most ”universal” sorbent for the 3 species
- Chromosorb106 /Tenax TA in one tube.

It was found that one sorbent would not work for all compounds, although could work for a large number of compounds from the species specified in ISO 14687 Grade D as “total” compounds.

The possibility of directly sampling hydrogen at the refuelling station using sorbent tubes was also investigated. It was concluded that the pressure needed to be reduced to a maximum of 10 bar before reaching the tubes. Currently it is not possible to directly sample on tubes at the nozzle of the HRS; implementing sampling on tubes at the nozzle would be very challenging in many aspects including safety. However, two possibilities had been discussed which included making use of the hydrogen that is vented during current sampling procedures or using the “pressure pulse” at the start of the refuelling. If the station could be modified to accommodate the use of online analysers and/or sensors, there would also be the possibility to implement a design for the sampling on tubes. The transfer of hydrogen sample from the original vessel to sorbent tubes is not prohibited although this could risk impurity losses as well as further contamination of the sample.

Assessing suitability of commercially available sampling vessels

A robust study was performed to assess suitable sampling vessels for transferring samples of gaseous hydrogen from the refuelling station to the laboratory for quality assurance testing. The following were considered important factors for selecting a suitable sampling vessel:

- Compatibility with available H2-sampling devices (limiting factor for now)
- Size (ex 10 L or smaller: the volume must be enough to perform all required analyses)
- Configuration: two ended cylinders (easy to purge), one ended cylinders
- Materials (Aluminium, steel, alloys and composite materials)
- Different inner treatments as passivation
- Pressure requirements (to be certified for at least 100 bar or the sampling pressure)
- Price range
- Commercial availability.

VSL, NPL and RISE tested different sampling vessels and cylinders to monitor stability of different hydrogen mixtures containing low level impurities.

- VSL: 100 ppb HCl + 100 ppb CO in H2 gas mixture – prepared by VSL, tested in SilcoNert 2000-treated stainless-steel and Aculife IV-treated aluminium sampling vessel.
- NPL: 40 ppb H2S + 100 ppb CO in H2 gas mixture – prepared by NPL, tested in Spectraseal-treated aluminium and untreated aluminium sampling vessel.
- RISE: 40 ppb H2S + 100 ppb CO in H2 gas mixture – prepared by NPL, tested in Dursan-treated stainless steel and Sulfinert-treated stainless-steel sampling vessel.

Results were reported in a good practice guide available on the project website.

The key outputs of Objective 4 are: To provide good practice for sampling either gas impurities or particulates from a high pressure hydrogen refuelling station to ensure a representative sample is obtained after delivery to the laboratory for offline purity analysis. This includes best practice for use of gas sampling devices, particulate sampling devices (using filter), suitable gas cylinders with appropriate passivation treatments and feasibility of using sorbent tubes. This objective was successfully achieved.

5 Impact

To ensure the project activities remained aligned with stakeholder needs the consortium had built up a stakeholder advisory board of around 50 members; including vehicle manufacturers, gas producers and laboratories. Representatives of key standardisation committees were also included in the stakeholder advisory board, including experts and chair persons. To ensure the project remained relevant to hydrogen industry, the advisory board influenced changes to some activities.

Main dissemination activities included a keynote speech on the project at the World Hydrogen Energy Conference in Brazil (2018), 22 other conference presentations, presentations at standardisation committees including a talk on this project at the plenary meeting for ISO TC 197 Hydrogen Technologies. MetroHyVe held the first workshop on measurement challenges for hydrogen vehicles (in partnership with the Horizon 2020 Cross-Cutting project HYDRAITE), hosting around 80 participants from around the globe, and another two workshops to support industry to perform accurate sampling.

Impact on industrial and other user communities:

The activities and reports produced by the project were regularly disseminated to OIML TC8 SC7, and along with this MetroHyVe partners attended committee meetings to support revision of OIML R139-1 which provides maximum permissible errors for flow metering at HRS. The follow-up EMPIR project 19ENG04 MetroHyVe 2 may consider proposing a revision to OIML R139-1.

The sampling campaign performed in this project proves that there is a certain minimum number of purges that are required to remove air and water contaminants from sampling devices. Tested methods for sampling particulates and gas have been written into reports and good practice guides which will support HRS operators through this process. The impact is significant as HRSs need to be able to sample hydrogen without contaminating it otherwise laboratories will provide test reports showing that their hydrogen fails to meet ISO 14687. Following from MetroHyVe activities, ISO TC 197 have now set up a new working group to focus on sampling methods at hydrogen refuelling stations which is being co-chaired by SINTEF and NPL (partners of this project).

Online instruments have been developed to measure key impurities in hydrogen, and since testing had been carried out at NPL, the project could determine their suitability as hydrogen quality control measures. These devices provided by AP2E, Shell and CT can either be further developed or sold commercially (where data from this project can be used to show instrument performance). Furthermore, in the online comparison two additional instruments were rigorously tested; and results from the comparison can be used to either further develop the instrument or be used to show customers that it performs well. MetroHyVe partners provided additional support to these participants by explaining possible issues with their measurement which was mainly regarding calculation of measurement uncertainty.

The offline comparison robustly assessed commercial hydrogen purity laboratories in their ability to accurately provide hydrogen purity measurements as specified by ISO 14687. In total 13 laboratories from Europe, Asia and USA participated, and the results first of all highlighted that not all laboratories could correctly measure the reference value; in these cases the results were fed back and where required NPL and VSL supported the laboratories by providing additional advice to correct measurements. The activity also impacted the hydrogen quality community by highlighting the importance of running annual schemes such as this one.

An online virtual measurement service hub (via a website) has been created at NPL and is listing laboratories and their hydrogen capabilities. The hub allows end-users to easily find and access these laboratories. The hub will remain open and managed by EMPIR project 19ENG04 MetroHyVe 2.

Impact on the metrology and scientific communities:

Four primary gravimetric standards were built to verify hydrogen flow meters under real conditions using 875 bar hydrogen. These standards can be used across Europe to ensure hydrogen fuel dispensed from stations is calculated accurately in accordance with the requirements of OIML R139-1.

New calibration gas standards were made with improved stability for low level reactive impurities in hydrogen. Gas producers are able to use these to certify their calibration standards for all impurities in ISO 14687 to ensure they are providing traceable values. These new standards are now being advertised and sold by the NMIs as new products. These can already be purchased by research organisations, manufacturers of fuel cells, laboratories, calibration gas providers and universities.

Testing of sorbent tubes and gas vessels for different types of impurities in hydrogen has been completed including an assessment of stability periods. Gas analysis laboratories are able to use the project's report on this subject to select suitable sampling vessels for customers requiring their services. This work will improve the stability of impurities in hydrogen samples.

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 existed to perform this measurement, which was an activity of the project. Another revision cycle may start in 2021 (to revise Grade D of ISO 14687 which is related to fuel cell hydrogen purity).

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 included an annex on sampling, but this part will now be written into an independent standard. SINTEF will head this activity.

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.

A low cost all-in-one analyser was developed by RISE in collaboration with AP2E which fulfilled the goal of lowering cost and labour to perform all impurity measurements required for electrolysis. The development of new hydrogen purity methods by the project will help to prevent serious damage to fuel cell vehicles by allowing the hydrogen industry to perform all hydrogen purity measurements as required by ISO 14687 Grade D. 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 be able to use the new reports and good practice guides to 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.

6 List of publications

Measurement challenges for hydrogen vehicles, A. Murugan, M. de Huu, T. Bacquart, J. van Wijk, K. Arrhenius, I. te Ronde and D. Hemfrey, International Journal of Hydrogen Energy, Volume 44, Issue 35, July 2019, Pages 19326-19333. https://doi.org/10.1016/j.ijhydene.2019.03.190

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7 Contact details

Dr Arul Murugan

arul.murugan@npl.co.uk

(+44)20 8943 6382