

Communication

First Hydrogen Fuel Sampling from a Fuel Cell Hydrogen Electrical Vehicle—Validation of Hydrogen Fuel Sampling System to Investigate FCEV Performance

Thomas Bacquart ^{1,*} , Niamh Moore ¹, Vincent Mattelaer ², James Olden ², Abigail Siân Olivia Morris ¹, Ward Storms ² and Arul Murugan ¹ 

¹ National Physical Laboratory, Atmospheric Environmental Science Department, Hampton Road, Teddington, Middlesex TW11 0LW, UK

² Toyota Motor Europe, R&D, B-1930 Zaventem, Belgium

* Correspondence: thomas.bacquart@npl.co.uk; Tel.: +44-(0)20-8943-6652

Abstract: Fuel cell electric vehicles (FCEV) are developing quickly from passenger vehicles to trucks or fork-lifts. Policymakers are supporting an ambitious strategy to deploy fuel cell electrical vehicles with infrastructure as hydrogen refueling stations (HRS) as the European Green deal for Europe. The hydrogen fuel quality according to international standard as ISO 14687 is critical to ensure the FCEV performance and that poor hydrogen quality may not cause FCEV loss of performance. However, the sampling system is only available for nozzle sampling at HRS. If a FCEV may show a lack of performance, there is currently no methodology to sample hydrogen fuel from a FCEV itself. It would support the investigation to determine if hydrogen fuel may have caused any performance loss. This article presents the first FCEV sampling system and its comparison with the hydrogen fuel sampling from the HRS nozzle (as requested by international standard ISO 14687). The results showed good agreement with the hydrogen fuel sample. The results demonstrate that the prototype developed provides representative samples from the FCEV and can be an alternative to determine hydrogen fuel quality. The prototype will require improvements and a larger sampling campaign.

Keywords: fuel cell electrical vehicles; ISO14687; gas analysis; gas sampling; hydrogen refueling station; hydrogen quality



Citation: Bacquart, T.; Moore, N.; Mattelaer, V.; Olden, J.; Morris, A.S.O.; Storms, W.; Murugan, A. First Hydrogen Fuel Sampling from a Fuel Cell Hydrogen Electrical Vehicle—Validation of Hydrogen Fuel Sampling System to Investigate FCEV Performance. *Processes* **2022**, *10*, 1709. <https://doi.org/10.3390/pr10091709>

Academic Editor: Alfredo Iranzo

Received: 5 June 2022

Accepted: 17 August 2022

Published: 27 August 2022

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1. Introduction

Vehicle electrification is now seen as the main decarbonization pathway for nearly all road-based transportation [1]. Moreover, it is not certain that electrical vehicles powered by Li-ion batteries will be suitable for every vehicle market, owing to inherent limits in their energy storage capacity, charging infrastructure availability, and achievable cost [2]. Alternative technologies as hydrogen fuel cell will complement battery electrical vehicles for the long-range, higher mass and high-utilization transportation markets (e.g., trucks or forklifts). Across Europe, many countries have initiated national policies to introduce over the next few decades hydrogen mobility technologies, especially for heavy-duty vehicles (e.g., trucks or forklifts), to the market. The European hydrogen market for fuel cell electrical vehicles (FCEV) is anticipated to increase significantly over the next years [3] especially following the European Commission target of 4500 hydrogen refueling stations (HRS) in Europe by 2030 [4]. Aligned with the increase of HRS infrastructure worldwide, FCEVs mass production is engaged with the ambition of automotive manufacturers such as Toyota [5] or Hyundai [6] and fuel cell stack producers (i.e., Symbio [7]). The industrial engagement is in coherence with policy makers ambition (i.e., Europe target 13 million FCEVs by 2030 [8]).

FCEVs have strict specification for hydrogen fuel quality. The presence of contaminants can impact the lifetime of fuel cells [9] and of the FCEV. Thirteen gaseous contaminants

and particulates should be monitored according to international standards ISO 14687 [10] or EN 17124 [11]. According to the European Directive 2014/94/EU [12], hydrogen fuel supplied to FCEV should be of a suitable quality (compliance with ISO14687:2019). Users and producers have to ensure that contaminants in hydrogen are measured accurately. As part of the measurement process, performing reliable and representative sampling of the hydrogen fuel at the HRS nozzle is mandatory. Sampling at the HRS has been studied [13,14], standardised (ASTM D7606 [15]) and referenced in ISO 19880-1 [16]. The recent review by Arrhenius et al. [17] presented sampling systems currently available for hydrogen fuel sampling at HRS nozzle like H2 Qualitizer and Hy-SAM [17].

The current challenge is that HRS sampling and analysis are not sufficiently frequent enough to identify unexpected issues at HRS (i.e., insufficient purge after maintenance, compressor oil leakage, unsuitable sealing material) [18]. Several studies pointed that contamination may be found at hydrogen refuelling station, for example newly commissioned [19] or from new contaminants from maintenance and operations [18]. A recent study in Europe showed that a fraction of the sample taken (29%) were in violation with the fuel tolerance limit [13]. Moreover, receiving the results of analysis may take at least a few days due to logistics and analysis time. Therefore, a hydrogen fuel issue may be observed in FCEV performance even before any hydrogen fuel sampling is performed. In case there is suspicion of unexplained poor FCEV performance, it is critical to determine if the hydrogen fuel may be responsible. Considering the complexity of hydrogen fuel online monitoring and the potential delay between any hydrogen fuel analysis from the HRS nozzle and FCEV performance observation, there is a need for an alternative option to investigate the hydrogen fuel quality. In case of FCEV events like power loss, or unplanned stopping, the hydrogen fuel has to be sampled from the FCEV itself and analysed to investigate the root cause of the event.

The hydrogen fuel quality from the FCEV itself has never been reported due to the lack of sampling system and procedure. The objective of this short communication is to present the first hydrogen fuel sampling from a FCEV. To demonstrate the representativity of National Physical Laboratory (NPL) FCEV sampling rig, this study will present a direct comparison between sampling at HRS nozzle and FCEV sampling during the same refilling event. As it is not expected to see differences between the hydrogen fuel from the HRS nozzle and the FCEV tank, the comparison should demonstrate the equivalence of the two approaches. The hydrogen fuel analysis according to ISO 14687 was performed on both samples. Finally, the results will highlight the validity of the sampling procedure and present the base of the future studies to be performed to improve the sampling strategy for HRS and FCEV.

2. Materials and Methods

2.1. Comparative Sampling between FCEV and HRS Nozzle

The comparison of hydrogen fuel quality sampling at the HRS nozzle and from the FCEV requires that the FCEV is refilled while the nozzle sample is taken as described in Figure 1. This allows in theory the analysis of the same hydrogen fuel taken at the nozzle and then sampled from the FCEV. It is possible using the H2 Qualitizer which allows hydrogen sampling from the HRS without the requirement to override safety elements of the control system [14]. The sampler is principally a tee where a sample is collected while an FCEV is refueled [14]. A Generation 1 Toyota Mirai was selected, and the hydrogen fuel tank was almost emptied before the sampling to avoid bias due to hydrogen fuel present in the FCEV before the refuel. The HRS nozzle sampling was performed at a public accessible HRS in Europe, the Toyota Mirai used during the refueling event was sampled less than 30 km after.

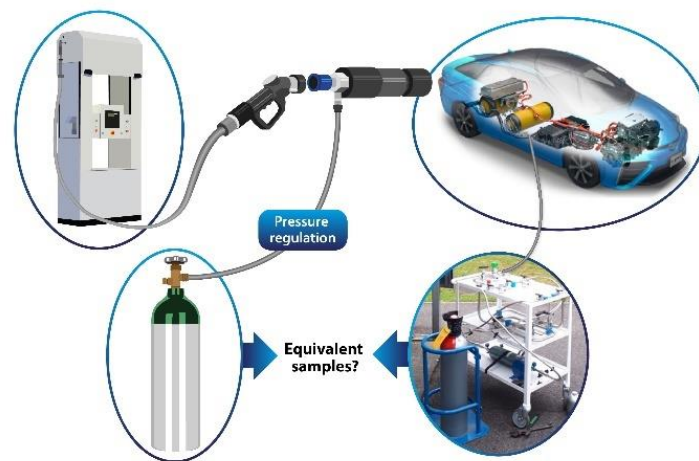


Figure 1. Schematic of the comparison principles between HRS nozzle sampling and FCEV sampling to evaluate the representativity of the sample taken by the FCEV sampling system.

2.2. Hydrogen Sampling from Fuel Cell Electrical Vehicles

2.2.1. NPL FCEV Sampling Rig

The NPL FCEV sampling rig is designed to realise reliable sampling of hydrogen fuel from FCEV (see Figure 2). The system was designed for low-pressure sampling (e.g., pressures below 8 MPa). The system is designed with three paths: one purging/venting path (V1 valve) and two sampling paths with a flow-through sampling path (VS valves and double-ended cylinder) and parallel sampling (VB valves and 10 L cylinder with one valve outlet). All outlets were jointly vented in a safe manner through a chimney with a flame arrestor, non-return valves, and a silencer. The complete system is made from Swagelok parts made of stainless steel 316 (Swagelok, UK). The sampling rig is compatible with various types of cylinders from stainless steel 1–4 L vessels to larger cylinders (i.e., aluminum cylinder 10 L) and with single valve outlet or double valve outlets cylinder. The sampling rig protocol was developed to ensure proper purging of the system before sampling in the gas cylinder.

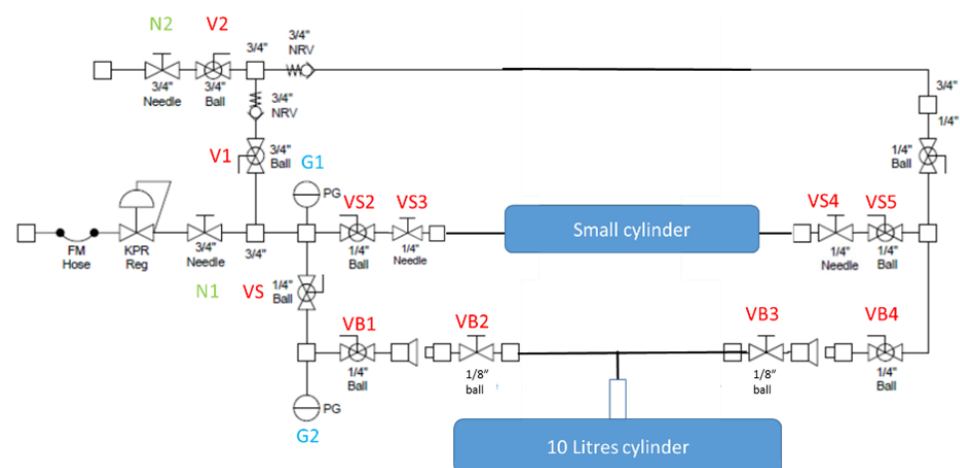


Figure 2. Schematic of the sampling system used to obtain representative hydrogen fuel sample from the FCEV at low-pressure point close to the vehicle fuel tank. V1, V2, V3, VS2, VS5, VB1, VB2, VB3 and VB4: two-way valve; N1, N2: metering valve (needle valve); VS3, VS4: small cylinder valve (metering valve); small cylinder was double-ended 1–4 L cylinder (i.e., 4 L sulfinert coated stainless steel vessel (Restek, UK)); 10 litres cylinder is 10 L aluminum treated Spectraseal® cylinder (BOC, Woking, UK).

2.2.2. Gas Cylinder Preparation

Cylinder preparation requires that the cylinders were evacuated for at least 2 h for the 4 L sulfinert coated stainless steel vessel (Restek, Ripley, UK) and more than 8 h for the 10 L aluminum treated Spectraseal[®] cylinder (BOC, Woking, UK). The cylinders were evacuated to a high vacuum (below 5×10^{-7} mbar) at ambient temperature. The high vacuum system was composed of a scroll pump (Scrollvac 15 plus, Leybold, Chessington, UK) and a turbomolecular pump with magnetic rotor suspension (Turbovac Mag, Leybold, Chessington, UK).

2.2.3. Hydrogen Sampling Rig Operation

The sampling protocol involves four steps including system connection, sampling system purge, sampling, and disconnection of the system. It is mandatory to perform and agree on the safety risk assessment before any actual sampling.

The FCEV connection point should be compatible with the NPL car sampling rig. The exact detail of these items cannot be disclosed as it is FCEV dependent. A flexible hose was used to connect the FCEV to the NPL FCEV sampling rig. The NPL FCEV sampling rig and the sampling cylinders were connected (using NPL minimum dead volume connection or Swagelok quick connect fitting) and secured to a solid point. The sampling system was leak checked using pressure drop, liquid leak detector Snoop[®] (Swagelok, Kings Langley, UK), and electronic detector (HydrogenPower, Umweltsensortechnik GmbH, Geschwenda, DE). Prior to any sampling, hydrogen from the FCEV was vented for a significant amount of time (>5 min) at high flow rate (>5 L/min) to ensure that hydrogen flow path is free from contamination until the sampling system (path N1-V1-V2-N2). The venting time depends on the FCEV type, the connection point in the FCEV, and the length of the connection between the FCEV and the sampling system. A sequence of three purges was performed: 1—flow through the sampling system purge; 2—cycling purges; 3—flow through the sampling system purge. The volume of hydrogen vented was superior to 50 L during these experiments. The cycling purges were performed to ensure that the sampling line was free of contaminants especially water that may be present in the dead volume of the sampling system and may not be easily purged during flow-through purges. The cycling purges were repeated more than 5 times. The hydrogen fuel from the FCEV was then allowed to flow through the sampling line for more than 10 min at flow above 5 L/min then the valve VB3 or VS4 was closed to allow the sampling of hydrogen into the cylinders. The sampling cylinder gas was left to equilibrate with the FCEV for at least 1 min. Then the cylinder was closed, and the sampling tool was depressurized and disconnected. The same procedure was repeated for the two sampling cylinders taken.

2.3. Hydrogen Sampling at Hydrogen Refueling Station (High Pressure 700 Bar)

The hydrogen H2 Qualitizer (Linde, AT) was used for sampling at a hydrogen refueling station. The sampling adaptor itself is a “T-piece” inserted between a receptacle, that receives the HRS nozzle, and a nozzle, that attaches to the FCEV. The Linde H2 Qualitizer is based on a Tescom pressure regulator, positionned between the t-piece and the sampling vessel. The regulator has a maximum inlet pressure of 875 bar and a temperature range of −40 to 85 °C. The adaptor is not equipped with an infrared communication interface. The sampling was performed according to the procedure described by Bacquart et al. [14].

2.4. Analytical Methods

The National Physical Laboratory (UK) is the UK’s national metrology laboratory and it developed analytical methods to measure all of the hydrogen fuel contaminants listed in ISO 14687. The analyses were performed using NPL internal methods. NPL methods were accredited to ISO 17025 for N₂, O₂, Ar, CO, CO₂, CH₄, non-methane hydrocarbons (NMHC), total sulphur, H₂O and He. Analyses were calibrated using NPL gravimetric gas standards in hydrogen matrix gas or NPL liquid standards for organo-halogenated. Nitrogen, oxygen and argon were analysed by gas chromatography (Agilent, UK) with pulsed discharge

helium ionization detector (PDHID, VICI, CH) using helium as a high purity carrier gas (Helium BIP[®], Airproducts, UK). The GC/PDHID sampling loop was 1 mL. The sample was transferred onto capillary column molsieve 5A plot (30 m × 0.53 mm × 50 µm) and a second capillary column molsieve 5A plot (50 m × 0.53 mm × 50 µm). The GC oven was set at 30 degrees Celsius. Water was measured using quartz crystal microbalance, QMA401 (Michell, USA). Gases are sampled directly from the gas cylinder to the analyser, a valve was used to restrict the flow to 0.333 L/min for the QMA. Detailed information about the analytical methods is provided in Supplementary Materials.

Gravimetric standards and/or dynamic standards were used to generate calibration curves covering the EN 17124 and ISO 14687 threshold and the measured values. The dynamic standards were prepared by dilution of NPL gravimetric gas standards (NPL, UK) with high purity hydrogen (BIP+ quality, Air Products, UK) using mass flow controller systems (Bronkhorst, NL).

The data was investigated however no result was discarded without a technical reason. The calibration curve, results of analysis and uncertainties associated were determined using NPL software XLGENline [20]. An expanded uncertainty using a *k* value of 2 was used. In some cases, a more conservative uncertainty was derived from scientific experience.

3. Results and Discussion

3.1. Comparison of Hydrogen Quality between HRS Nozzle and FCEV Tank Sampling

The first sampling of hydrogen fuel from an FCEV was performed in conjunction with the sampling of a hydrogen refueling station. It was critical to establish a comparison between the established sampling procedure at the nozzle of the HRS using H2 Qualitizer and the first FCEV hydrogen fuel sampling system. The FCEV was almost entirely refueled (tank mostly emptied). The refuelling achieved approximately 80% of the tank capacity due to the lack of communication between the H2 Qualitizer and the FCEV. The samples taken from the FCEV and the HRS nozzle were analysed in repeatability conditions at NPL laboratory (Teddington, UK). The analyses were performed within 8 weeks after the sampling at NPL laboratory (Teddington, UK).

3.1.1. Comparison of Hydrogen Quality for all ISO 14687 Gas Contaminants between HRS Nozzle and FCEV Tank Sampling

The results of analysis (Table 1) showed that the hydrogen fuel was compliant with the ISO 14687:2019 requirements at HRS. No gas contaminants were found above the ISO 14687 threshold from HRS nozzle sampling. Most of the contaminants were found below the detection limit of NPL analytical methods except nitrogen, argon, and water. The amount fractions of nitrogen and argon in the gas samples were not significantly different at 95% confidence level from the HRS nozzle sampling (H2 Qualitizer) and from the FCEV (NPL FCEV sampling rig). The difference in limits of detection on sulphur amount fraction between “FCEV sampling 1”, “HRS sample”, and “FCEV sampling 2” is related to analysis on different days and a change in the sensitivity of the GC-SCD analyser. A small amount of carbon dioxide was observed in the FCEV sampling 2 however the measured amount fraction was close to the NPL limit of detection and consequently has a large uncertainty. It is not considered as disagreeing with the other results of analysis reporting less than the limit of detection.

Table 1. Results of analysis for all the contaminants mentioned in ISO 14687 standard from a sample obtained at the HRS and from samples obtained from the FCEV. (1) total halogenated compounds reported as the sum of the amount fraction of organo-halogenated compounds listed in ASTM D7892-15. (2) individual organo-halogenated compounds refer to the highest limit of detection for an individual halogenated compound listed in ASTM D7892-15. (3) reported as an amount fraction of total sulphur in the sample. n.a. means not applicable; n.m. means not measured as the analysis was not realised due to insufficient gas.

Sampling Location	n.a.	Hydrogen Refueling Station—700 Bar	FCEV Sampling—1	FCEV Sampling—2
Sampling system	n.a.	H2 Qualitizer 10 L cylinder	NPL prototype 10 L cylinder	NPL prototype Sulfinert 4L
Sampling vessel	n.a.	aluminum Spectraseal	aluminum Spectraseal	cylinder double valve outlet
Compounds	ISO 14687:2019 threshold ($\mu\text{mol/mol}$)	Measured amount fraction and expanded uncertainty ($k = 2$)/($\mu\text{mol/mol}$)		
Nitrogen	300	24.7 ± 0.7	25.0 ± 0.6	n.m.
Helium	300	<14	<14	<14
Argon	300	0.870 ± 0.035	0.835 ± 0.031	n.m.
Water	5	1.27 ± 0.26	4.4 ± 0.9	n.m.
Oxygen	5	<0.5	<0.5	n.m.
Carbon dioxide	2	<0.020	<0.020	0.017 ± 0.007
Methane	100	<0.020	<0.020	<0.020
Non-methane hydrocarbons	2	<0.040	<0.040	<0.040
Carbon monoxide	0.2	<0.030	<0.030	<0.030
Formic acid	0.2	<0.040	<0.08	<0.08
Ammonia	0.1	<0.07	<0.07	<0.07
Formaldehyde	0.2	<0.05	<0.05	<0.05
Total halogenated compounds ⁽¹⁾	0.05	<0.032	<0.030	<0.031
Individual organo halogenated compounds ⁽²⁾	n.a.	<0.0030	<0.0030	<0.0030
Total sulphur compounds ⁽³⁾	0.004	<0.0010	<0.0030	<0.0010

3.1.2. Water Amount Fraction Discrepancies between HRS Nozzle and FCEV Tank Sampling

The amount fraction of water was significantly higher in the “FCEV sample 1” compared to the “HRS sample”. The difference may have different explanations: humidity in the FCEV tank before the sampling, insufficient purging of the NPL prototype sampling rig, or the reliability of parallel sampling at HRS for water amount fraction. A study from Bacquart et al. highlighted the difficulty to purge water from H2 Qualitizer system as water vapour tends to be purged by displacement rather than dilution [21] and it tends to adsorb on the steel surface [22]. During the NPL FCEV sampling rig testing in the laboratory, no hydrogen contamination by water was observed following the procedure. However, the NPL laboratory experiment using hydrogen cylinders as the source gas is slightly different from real FCEV tank sampling due to the complexity of the FCEV system, the different gas flow systems involved in the FCEV and the type IV tank.

The water amount fraction disagreement requires additional studies to find the source of the discrepancies. An important aspect to consider is the assessment of the amount fraction of water in the FCEV tank type IV. It would require a dedicated experimental setup to understand the behaviour of water amount fraction in this type of cylinder. Another perspective is the deployment of an analyser that can measure water amount fraction while the sampling is realised, it would provide a better understanding of the water amount fraction behavior during the sampling (i.e., insufficient purging, stable reading). Regarding the other contaminants, the study demonstrates the equivalence between the HRS sampling and the FCEV sampling.

3.1.3. Sampling Procedure HRS Nozzle and FCEV Tank

The sampling comparison study confirmed that the hydrogen fuel was similar in both sampling regarding air contamination (oxygen, argon, nitrogen). Therefore, it demonstrated that no air contamination was introduced during the FCEV sampling. It demonstrates that the sampling system and procedure proposed by NPL is suitable for hydrogen quality sampling from the FCEV.

3.2. Perspective of the FCEV Sampling System

The NPL FCEV sampling rig showed good performance and agreement with established sampling system. However, the NPL FCEV sampling rig may require further improvements to be implemented more widely and to extend the number of FCEV to gather larger information on potential FCEV issues.

3.2.1. Improvement of the Sampling System

There are three areas of improvement for the system: 1—simplification for wider-use, 2—representativity for reactive compounds and 3—understanding FCEV tank homogeneity.

The design proposed by NPL may require improvement to be more widely used by the industry. The concept was successfully demonstrated and will require improvement in the design to simplify its use (less valves, single flow path, smaller footprint). A more user-friendly version would allow deployment of the capability for hydrogen car specialists and support better investigation of FCEV performance considering hydrogen fuel quality.

The representativity of reactive compounds is difficult to evaluate from hydrogen fuel sampling as it is often free of any reactive contaminants (i.e., sulphur, ammonia, formaldehyde). It is known that such compounds at low amount fraction (i.e., nmol/mol) tend to adsorb on surface. Therefore, the current system may require laboratory evaluation for such reactive gas or to be entirely sulfonert treated to reduce the potential adsorption on the FCEV sampling rig. NPL is currently developing a new version of the sampling system considering this aspect. The water amount fraction will need additional investigation to determine the reason behind the difference in the results obtained from HRS nozzle sampling and within the FCEV. Water amount fraction measurement at 5 $\mu\text{mol/mol}$ level is still a measurement requiring more focus from analytical laboratory due to the nature of water and its adsorption to surface.

A new research aspect is the homogeneity of the contaminants in the FCEV tank. There is no information available about contaminants behaviour in type IV tank and on potential segregation in the tank (i.e., pressure, density). It may be interesting to perform various sampling at different level of emptiness of the FCEV tank. Even if these preliminary results showed excellent agreement between the nozzle and the FCEV tank, additional bilateral comparisons should be performed to increase the confidence in the equivalence between the two sampling points.

3.2.2. Application on Real Case to Identify Hydrogen Fuel Issues and New Contaminants

The application of the FCEV sampling system on real events would allow correlating FCEV performance with fuel quality. It would be important for FCEV owners to understand the origin of low performance. This new type of sampling can be used after identifying performance issues on a FCEV. The application of the sampling on a real case will require being informed and gaining access FCEV which encounter performance issues which is currently difficult to find. This type of sampling may be useful to identify new types of contaminants that are not already regulated especially considering an increase in new hydrogen technologies and fast-paced implementation of the solutions to support an exponentially growing market. This type of sampling will help analytical laboratory and research project to focus on hydrogen fuel that had a performance impact on FCEV. It would simplify the research activity around new impurities profiling by using FCEV experience to quickly identify and regulate new potential contaminants. It may be quicker and more

efficient rather than trying to measure unknown compounds in hydrogen fuel from HRS which often has no clear fuel cell impact evaluation.

4. Conclusions

This study demonstrates the capability and the representativity of hydrogen sampling from a FCEV using NPL FCEV sampling rig. The study presented the first design for FCEV sampling rig and the first results from a FCEV sampling. The sampling rig and procedure allowed the authors to get a representative sample in agreement with hydrogen fuel quality from HRS nozzle sampling except for water. These preliminary results present the potential of the system and highlight the next steps (simplification of the system, representativity for reactive compounds, and understanding FCEV tank homogeneity). Due to the small dataset, it is recommended to realise additional comparisons between HRS and FCEV sampling to gather more reliable datasets.

The FCEV sampling system would extend the investigation capability on FCEV performance related to hydrogen fuel quality. It is a critical tool to better understand the durability of FCEV during its lifetime in real conditions, identify hydrogen fuel quality issues related to the FCEV performance, early identification of new contaminant issues in hydrogen fuel, or investigate issues between FCEV end-users and HRS operators on hydrogen fuel quality.

Supplementary Materials: The following supporting information can be downloaded at: <https://www.mdpi.com/article/10.3390/pr10091709/s1>, Section on analytical methods.

Author Contributions: Conceptualization, T.B., J.O. and V.M.; methodology, T.B., N.M., J.O. and V.M.; Sampling: T.B., N.M., J.O. and V.M.; formal analysis, T.B., N.M., A.S.O.M. and V.M.; investigation, T.B., N.M., A.S.O.M., V.M., W.S. and A.M.; resources, T.B., J.O., V.M. and W.S.; data curation, T.B., N.M., A.S.O.M. and A.M.; writing—original draft preparation, T.B.; writing—review and editing, T.B., N.M., A.S.O.M., V.M., W.S. and A.M.; supervision, T.B., V.M. and W.S.; project administration, T.B., V.M. and W.S.; funding acquisition, T.B., V.M. and W.S. All authors have read and agreed to the published version of the manuscript.

Funding: This research received no external funding.

Institutional Review Board Statement: Not applicable.

Informed Consent Statement: Not applicable.

Data Availability Statement: The data presented in this study are available in this article: First hydrogen fuel sampling from a fuel cell hydrogen electrical vehicle—Validation of hydrogen fuel sampling system to investigate FCEV performance.

Conflicts of Interest: The authors declare no conflict of interest.

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