HYDRAITE – Hydrogen Delivery Risk Assessment and Impurity Tolerance Evaluation
Grant agreement no: 779475

D4.1 Report on European analytical capabilities for hydrogen purity according to ISO 14687 and quality assurance

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Confidentiality: Public

Submission date: 29.10.2019

Revision: 1.0

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FCH JU Project officer Alberto García Hombrados	**Grant agreement no:** 779475
Project name Hydrogen Delivery Risk Assessment and Impurity Tolerance Evaluation	**Project short name** HYDRAITE
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Summary

According to a recent report from the European Commission, hydrogen could represent 32% of the European fuel mix in 2050. The European Directive 2014/94/EU on the deployment of an alternative fuels infrastructure sets out that “The hydrogen purity dispensed by hydrogen refuelling points shall comply with the technical specifications included in the ISO 14687 standard”. Hydrogen fuel quality measurement are required to ensure compliance to international standards, however, no European laboratory had capability to perform all the measurements in 2017. The European hydrogen industry was relying on North American and Asian laboratories for performing all the analysis required by the international standard ISO 14687. The report presents the three European laboratories selected to developed ISO 14687 and EN 17124:2018 analytical capability. NPL and ZSW are able to perform all the analysis required by ISO 14687 and EN 17124:2018 on HYDRAITE milestone. Evidences were reported for NPL and ZSW as inter-laboratory comparison, bilateral comparison with Smart Chemistry or evaluation of analytical performance using NPL reference materials in hydrogen matrix. ZBT will achieve similar analytical capability by end of 2019.

HYDRAITE milestone is successfully passed with two laboratories out of three capable of performing all the required measurements in ISO 14687 and EN 17124:2018. Therefore European laboratories are fully capable of performing hydrogen fuel quality measurement required in work package 3 of HYDRAITE project. The next step is to continue working for agreement with future standard ISO/DIS 21087 and organise bilateral comparison between NPL, ZSW and ZBT to ensure equivalence and compliance of the European laboratories. It is the objective of the task 4.2 of HYDRAITE project.

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Contents

1. Hydrogen fuel quality and analytical laboratory worldwide

According to a recent report from the European Commission, hydrogen could represent 32% of the European fuel mix in 2050 [1]. The expansion of fuel cell electrical vehicles (FCEV) is becoming a crucial step to decarbonise the transport sector in Europe [2]. The European hydrogen fuel infrastructure (hydrogen refuelling stations: HRS) will increase significantly over the few next years with the following objectives in Europe: France: 100 HRS by 2023 and 400-1000 by 2028 [3]; Germany: 100 HRS in 2019 and 400 by 2023 [4]; the Netherlands: 50 HRS by 2025 [5] and United Kingdom: 20 HRS by 2020 [6]). FCEV are developing quickly across a wide range of vehicle types, from passenger vehicles (e.g. Toyota Mirai [7]) to truck (e.g. Hyundai lorries in Switzerland [8]) or trains [9]. The absence of contaminants in the hydrogen delivered at the HRS is critical to ensure the length of life of FCEV (durability > 5000 hours [10]). The international standard ISO 14687:2012 [11] defined the maximum amount fraction of each contaminant in hydrogen for FCEV applications. Therefore, hydrogen suppliers and producers need to ensure hydrogen quality in accordance to ISO 14687-2:2012 [11] or EN 17124:2018 [12] at HRS. The European Directive 2014/94/EU on the deployment of an alternative fuels infrastructure [13] sets out that “The hydrogen purity dispensed by hydrogen refuelling points shall comply with the technical specifications included in the ISO 14687-2 standard (which will be replaced by the European standard EN 17124:2018 [12]).

Based on the requirement of ISO/FDIS 19880-8 [14] for fuel quality control for HRS and EN 17124:2018 [12], a quality control plan has to be developed and implemented for each HRS. Hydrogen fuel quality measurements need to be performed on a regular basis. Scarce information is currently available in Europe on hydrogen fuel quality dispensed at the HRS nozzle. The publically available information is from European projects funded by the FCH JU as HyCoRA [15] or H2moveScandinavia [16]. These projects required a US based laboratory to perform hydrogen fuel quality measurement as the capability was not available commercially and independently in Europe.

The lack of European analytical laboratory capable of analysing hydrogen contaminant amount fraction as required in ISO 14687:2012 and prEN 17124 was evident in 2017. At this date, only NPL was close to having the complete analytical capability to perform all the analysis required in ISO 14687:2012. The European hydrogen industry was relying on North American and Asian laboratories for performing all the analysis required by the international standard ISO 14687:2012. The dependency of the European market on laboratories outside Europe lead to logistic issues (transport, stability of impurities during transport), delay in analysis and a lack of control on the quality (i.e., difficulty to perform audit, no requirement to follow European standards).

The HYDRAITE project supports the implementation of three European laboratories with full capability of performing ISO 14687 purity measurements in order to support the growing European hydrogen industry. HYDRAITE is collaborating with other national (e.g. German funded project Hy-Lab) and European funded projects (EMPIR MetroHyve [17]) while developing analytical capabilities for hydrogen purity across Europe.

This deliverable presents the results of the implementation of the analytical methods for the complete scope of ISO/FDIS 14687 [18] and EN 17124:2018 [12] in three European laboratories and the development of quality assurance and the validation approaches as specified by ISO TC 158 JWG7.

2. Implementation of three European independent analytical laboratories with ISO 14687 scope

2.1 Scope and independence

The first objective of the project was to define the scope of the laboratories. Two parameters are critical for the laboratory implementation in the HYDRAITE project:

- The independence of the laboratory: it is critical to implement laboratories which are independent from any hydrogen producer. It allows access to anyone in the hydrogen industry (i.e., SME or large company, university or consortium) to gain access to reliable and accurate hydrogen fuel quality measurement. The laboratory should be able to offer services to the whole H_2 industry. Therefore, NPL, ZSW and ZBT are considered independent institutes.
- The list of parameters in the international standard ISO 14687: the hydrogen fuel quality standard has evolved quickly in the past years. The ISO 14687-2:2012 [11] standard was revised and ISO/FDIS 14687 [18] will be released in the incoming month with new threshold amount fractions and compound enlisted (i.e., methane and sum of CO, formic acid and formaldehyde). At the European level, CEN-CENELEC published a new standard EN 17124:2018 [12] with a list of compounds and threshold for hydrogen fuel quality. The future released ISO/DIS 14687 [18] and EN 17124:2018 [12] will be aligned in term of compounds' list and threshold amount fraction (Table 1). For this reason, the consortium's objective is related to EN 17124:2018 [12] and the new standard ISO/FDIS 14687 [18].

The three laboratories, NPL, ZSW and ZBT, are independent institutes and aimed to be capable of measuring all the gas contaminants according to the threshold of ISO/FDIS 14687 [18] and EN 17124:2018 [12]. Particulates were excluded from the objective as particulate measurement needs to be done at HRS with dedicated sampling equipment. It was not considered to be analysis required for central analytical laboratory.

The report will present the analytical laboratory capability available in July 2019 and the evidences confirming the achievements.

Table 1. Internation standard for hydrogen fuel quality for fuel cell electrical vehicles. The threshold for the contaminants are reported with notes.

Contaminant	ISO 14687-2: 2012 [11] SAE J2719:2011 [19]		ISOF/DIS 14687 [18] EN 17124:2018 [12]	
	Max. admissible value [µmol/mol]	notes	Max. admissible value [µmol/mol]	notes
Water	5	-	5	-
Total hydrocarbons	2	Due to CH4, TC > 2 µmol/mol	2 except CH_4	including oxygenated organic species
Methane	-	-	100	-
Oxygen	5	-	5	-
Helium	300	-	300	-
Nitrogen	100	N2+Ar<100	300	-
Argon	100	N2+Ar<100	300	-
carbon dioxide	2	-	2	-
Carbon monoxide	0.2	-	0.2	CO+HCHO+HCOOH < 0.2µmol/mol
Total sulphur compounds	0.004	H2S, COS, CS2, mercaptans (NG)	0.004	H2S, COS, CS2, mercaptans (NG)
Formaldehyde	0.01	-	0.2	CO+HCHO+HCOOH < 0.2µmol/mol
Formic acid	0.2	-	0.2	CO+HCHO+HCOOH < 0.2µmol/mol
Ammonia	0.1	-	0.1	-
Halogenated compounds	0.05 (total)	i.e. HBr, HCl Cl2, organic R-X	0.05	HCl, organic R-Cl
Max. particulate conc.	1 mg/kg	-	1 mg/kg	-

2.2 Independent analytical laboratory capability

2.2.1 National Physical Laboratory

The National Physical Laboratory (Teddington, UK) developed hydrogen quality measurement over the recent years. The NPL Hydrogen quality laboratory is capable of providing analysis according to ISO/FDIS 14687 and EN17124:2018 using the analytical methods described in Table 2. As NPL is a metrological institute, and due to stringent, quality control, it is expected to deliver accurate and traceable measurements for analytical services.

NPL is using gas calibrant in hydrogen gas matrix for analysis. Calibration of the instruments is performed on each measurement occasion. Methods are following a validation process to be in compliance with ISO 21087:2019. NPL has accreditation ISO 17025 testing scope for CO, CO_2, Ar, N_2, total sulphur compounds, methane, total hydrocarbons and water in hydrogen gas matrix (UKAS testing N.0002). All analytical methods have a limit of detection compliant with ISO/FDIS 14687 and EN 17124:2018. NPL also provides an uncertainty budget for their analytical results based on a 95 % probability (coverage factor k=2). The "total halogenated" analysis is performed with compliance for ASTM D7892-15 [20]. This implies that inorganic halogens like HCl, HBr, Cl_2 and Br_2 have not been analysed.

Table 2. Analytical methods available at NPL for hydrogen fuel quality analysis. The limit of detection are reported as indicative with the required amount of gas to perform the analysis.

Contaminant	ISO/FDIS 14687 EN 17124 :2018 [µmol/mol]	Analytical Method	Detection Limit [µmol/mol]	Required amount of gas [L]
Water	5	Quartz crystal microbalance CRDS	0.2 0.030	30 L 30 - 60 L
Total Hydrocarbons	2	GC-Methaniser-FID	0.05	2 L
Methane	100	GC-Methaniser-FID	0.05	2 L
Oxygen	5	GC-PDHID	0.3	2 L
Helium	300	GC-TCD	10	2 L
Argon **Nitrogen**	300	GC-PDHID	0.3 1	2 L
Carbon Dioxide	2	GC-Methaniser-FID	0.02	2 L
Carbon Monoxide	0.2	GC-Methaniser-FID	0.02	2 L
Total sulphur compounds	0.004	GC-SCD	0.001	1 L
Formaldehyde	0.2	GC-Methaniser-FID SIFT-MS	0.05 0.02	2 L 2 L
Formic Acid	0.2	FTIR SIFT-MS	0.05 0.02	30 L 2 L
Ammonia	0.1	GC-MS FTIR SIFT-MS	1 0.05 0.02	2 L 30 L 2 L
Key halogenated compounds according to ASTM D7892-15	0.05	TD-GC-MS	0.016	1.5 L

2.2.2 Zentrum für Sonnenenergie- und Wasserstoff-Forschung Baden-Württemberg (ZSW)

The Zentrum für Sonnenenergie- und Wasserstoff-Forschung Baden-Württemberg (ZSW, Center for Solar Energy and Hydrogen Research) is a non-profit Research and Development Organisation working in the field of renewable energies. The ZSW analytical laboratory is located in Ulm in southern Germany. ZSW developed an entire analytical laboratory dedicated to hydrogen fuel quality in 2018 within the national project Hy-Lab. A large range of analytical techniques are available and part of the capability of ZSW to determine the 13 compounds in hydrogen fuel (Table 3).

Table 3. Analytical methods available at ZSW for hydrogen fuel quality analysis. The limit of detection are reported as indicative with the required amount of gas to perform the analysis.

Contaminant	ISO/FDIS 14687 EN17124:2018	Analytical Method	Lower Detection Limit	Lower Quantification Limit	estimated amount of gas needed
	µmol/mol		[µmol/mol]	[µmol/mol]	[L]
Water	5	Dew Point mirror	0.1*	< 1*	45
Total Hydrocarbons	2	GC-FID	TBD	< 0.05*	16
Methane	100	GC-PDHID	TBD	< 0.1*	16
Oxygen	5	GC-PDHID	TBD	< 0.1*	16
Oxygen	5	GC-TCD	< 1*	< 5*	6
Helium	300	GC-TCD	1	10*	6
Argon	300	GC-PDHID	TBD	0.1*	16
Argon	300	GC-TCD	TBD	< 2.5*	6
Nitrogen	300	GC-PDHID	TBD	< 0.1*	16
Nitrogen	300	GC-TCD	TBD	10	6
Carbon Dioxide	2	GC-PDHID	TBD	< 0.9*	13.6
Carbon Monoxide	0.2	GC-PDHID	< 0.1	< 0.1*	16
Carbon Monoxide	0.2	OFCEAS	0.001	0.003**	10
Total sulphur compounds	0.004	TD-(GC-)FPD	TBD***	0.001 (< 0.001)***	1.2
Formaldehyde	0.2	OFCEAS	0.001*	0.003**	10
Formic Acid	0.2	OFCEAS	0.001*	0.003**	10
Ammonia	0.1	OFCEAS	0.003*	0.01**	10
Halogenated compounds	0.05	TD-(GC-)ECD	TBD***	< 0.005***	16

* signals from contaminations in the reported range were above 10σ of noise amplitude

** manufacturer specification

*** manufacturer specification – tweakable by loading time of thermodesorber unit

The reported lowest detection limit (LoD) and lowest quantification limit (LoQ) values are still mostly derived from manufacturer specifications or observed amplitudes of real samples. Formal proof is still pending, due to current unavailability of a dilution unit which can satisfy the dilution ratios needed. One such dilution unit is currently under in-house development within the German national project Hy-Lab. Completion and verification of the new diluter had to be shifted within the project towards Q3/Q4 2019. The experimentally proven levels will be gained during work on validation of methods to conform to the coming ISO 20187.

2.2.3 Zentrum für BrennstoffzellenTechnik GmbH (ZBT)

The Zentrum für BrennstoffzellenTechnik GmbH (ZBT) was founded as fuel cell research centre in 2001 by the State of North-Rhine Westphalia and the European Fund for Regional Development and is located in Duisburg, Germany. Focusing on application-oriented R&D, ZBT is a non-profit, limited company bridging the gap between basic research at the University and requirements of industry.

ZBT developed a complete hydrogen quality laboratory between 2018 – 2019 purchasing a set of state of the art analyser with large spectrum (IMR/EI-MS) and dedicated analyser (see Table 4) in order to cover the ISO/DIS 14687 requirements.

Table 4. Analytical methods available at ZBT for hydrogen fuel quality analysis. The values for the limit of detection, which are not marked are reported as specifications given by the manufacturer.

Impurity	ISO/FDIS 14687 EN 17124:2018 [µmol/mol]	Analytical Method	Detection Limit [µmol/mol]
Water	5	Quartz crystal microbalance IMR-MS	0.1 3.044**
Total Hydrocarbons	2	GC-PED IMR-MS	0.01 0.0105*
Methane	100	GC-PED IMR-MS	0.01 0.0117*
Oxygen	5	GC-PED IMR-MS	0.01 0.209*
Helium	300	EI-MS	0.0041*
Argon	300	GC-PED EI-MS	0.05 0.00039*
Nitrogen	300	GC-PED EI-MS	0.1 0.01*
Carbon Dioxide	2	IMR-MS	0.987**
Carbon Monoxide	0.2	GC-PED IMR-MS	0.001 0.06**
Total sulphur compounds	0.004	TD-GC-SCD IMR-MS	< 0.001 0.0009 (H_2S)*
Formaldehyde	0.2	IMR-MS	0.0015*
Formic Acid	0.2	IMR-MS	0.0039*
Ammonia	0.1	IMR-MS	0.0018*
Key halogenated compounds	0.05	IMR-MS	< 0.067**

* LoD determined as 3-fold standard deviation of background signal in H2 9.0 after calibration
** further optimization is in progress

The commissioning phase, the investigations on the limit of detection (LoD) and limit of quantification (LoQ) and their validation of the GC-PED, the GC-SCD with TD and the QCM are still ongoing. The LoD (values marked with *) of the mass spectrometer could already be determined as 3-fold standard deviation of background signal in H_2 9.0 after calibration, see *Table 4*. It can be seen that the device meets almost all necessary boundary conditions of LoD below respective threshold levels. Regarding moisture, carbon monoxide, carbon sulfide, and hydrogen chloride (values marked with **) further optimization is in progress. These results need to be verified and validated and cross sensitivities to be identified.

2.3 Achievements by July 2019

2.3.1 International benchmarking and evidences

Due to the lack of analytical laboratories able to perform hydrogen fuel quality measurement, it is extremely difficult to evaluate laboratory performance in the area of hydrogen fuel quality. Only few inter-comparison or bi-lateral comparison has been performed worldwide (i.e., EURAMET 1220 [21]). NPL participated successfully in the first hydrogen quality round robin test (EURAMET 1220) organised by EURAMET. For carbon monoxide and dihydrogen sulphide, NPL results were agreeing with all the other participants.

NPL performed a bi-lateral comparison on hydrogen fuel quality measurement with Smart Chemistry during the European project HyCoRA [15]. It demonstrated the capability of NPL to measure all the compounds listed in ISO/FDIS 14687 and EN 17124:2018 as Smart Chemistry. Several differences were observed between the two analytical laboratories. It is important to consider the differences with deeper investigations. It is the new step regarding quality of the analytical results reported.

The HYDRAITE project reported the results of a new inter-comparison between NPL and Smart Chemistry in HYDRAITE work package 3. For this milestone, NPL and Smart Chemistry were the two laboratories compared. NPL and Smart Chemistry are using different analytical techniques and strategies that may result in difference in the final results (i.e., NPL performs total sulphur analysis while Smart Chemistry is analysing a list of independent molecules containing sulphur atoms). One difference may be in the definition of total compounds (total sulphur, total halogenated and total hydrocarbons). Investigating these differences will be progressed in task 4.2 of HYDRAITE project. Comparison of the sampling campaign results of analysis will provide important data to understand differences especially on the measurement of total compounds compare to selected list of halogenated or sulphur compounds.

In the meantime, ZSW and ZBT are performing similar measurements on the hydrogen fuel samples of the first sampling campaign. Interpretation of the differences is complex to interpretas multiple parameters can influence the results (i.e., contaminant stability, analytical methods, analytes, time of analysis, calibrants). However, reaching the number of four laboratories performing similar measurement on the same samples (*n*: 4 – 10) would provide better statistic and understanding in the laboratory differences observed.

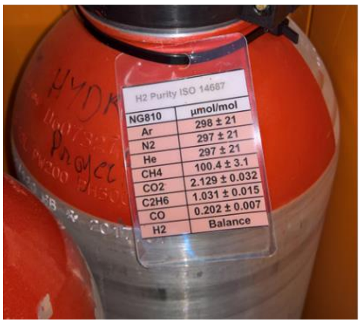

Figure 1. NPL reference materials supplied to ZSW and ZBT for evaluationg analytical performance according to ISO/FDIS 14687 and EN 17124:2018.

NPL prepared and certified gas standards containing the 7 out of 13 contaminants in hydrogen matrix according to ISO/FDIS 14687 and EN 17124:2018. The reference material were prepared in 10L aluminium cylinder with SpectraSeal® treatment (BOC, UK).

Table 5. Composition of NPL reference materials supplied to ZSW and ZBT. The amount fractions correspond to EN 17124:2018 thresholds. The uncertainty was below 10% relative for all contaminants in hydrogen gas.

Component	Amount fraction / [µmol/mol]
Helium	300
Argon	300
Nitrogen	300
Methane	100
Carbon dioxide	2
Ethane	1
Carbon monoxide	0.2
Hydrogen	Balance gas

ZSW performed analytical performance on hydrogen fuel quality using the two NPL gas standards. Even if all the 13 compounds were not present in the NPL reference materials, ZSW performed the measurement for all of them (Table 6). In the absence of the compounds, the value measured from ZSW should be below detection limit.

Table 6. Results of analytical performance from ZSW on NPL reference material using all their available analytical instruments.

Measured species	Analytical Method	ISO/FDIS 14687 threshold	Certified amount fraction in NPL cylinder 1	Result NPL1_1	Result NPL1_2	Certified amount fraction in NPL cylinder 2	Result NPL2_1	Result NPL2_2
		µmol/mol	µmol/mol	µmol/mol	[µmol/mol]	[µmol/mol]	[µmol/mol]	[µmol/mol]
Water	Dew Point mirror	5	n.c.	4 ± 0.X	73.9 ± 3.1	n.c.	1 ± 0.X	221 ± 9.5
Total Hydrocarbons	GC-FID	n.d.	107.4	78.1 ± 1.4	88.3 ± 1.1	102.5	59.2 ± 3.9	82.8 ± 0.8
Total non methane hydrocarbons	Calculated	2	1.974	-5.8	6.7	2.062	-15.4	5.0
Methane	GC-PDHID	100	105.4	83.9 ± 2.4	81.7 ± 2.1	100.4	74.6 ± 2.9	77.8 ± 2
Oxygen	GC-PDHID	5	n.c.	< 0.1	< 0.1	n.c.	< 0.1	< 0.1
Oxygen	GC-TCD	5	n.c.	< 3	< 3	n.c.	< 3	< 3
Helium	GC-TCD	300	299	300.9 ± 7.1	291 ± 6.7	297	294.2 ± 6.1	295.2 ± 6.1
Argon	GC-TCD	300	306	326.5 ± 12.9	307 ± 13.2	298	318.3 ± 12.7	297.9 ± 12.2
Nitrogen	GC-PDHID	300	308	311.1 ± 16.6	233.5 ± 11.9	297	292.6 ± 15	234.9 ± 12.6
Nitrogen	GC-TCD	300	308	308.2 ± 19.5	303.8 ± 26.4	297	297.6 ± 20.3	292.6 ± 25.7
Carbon Dioxide	GC-PDHID	2	1.99	2.3 ± 0.2	2.1 ± 0.1	2.129	2.0 ± 0.2	2.1 ± 0.1
Carbon Monoxide	GC-PDHID	0.2	0.204	0.25 ± 0.03	0.19 ± 0.05	0.202	0.21 ± 0.03	0.17 ± 0.03
Carbon Monoxide	OFCEAS_1	0.2	0.204	0.111 ± 0.006	0.113 ± 0.006	0.202	0.113 ± 0.006	0.113 ± 0.006
Total sulphur compounds	TD-(GC-) FPD	0.004	n.c.	0.009 ± 0.001	0.013 ± 0.002	n.c.	< 0.001	< 0.001
Formaldehyde	OFCEAS_2	0.2	n.c.	0.008 ± 0.001	0.07 ± 0.031	n.c.	0.007 ± 0.001	0.005 ± 0.001
Formic Acid	OFCEAS_2	0.2	n.c.	0.008 ± 0.001	0.018 ± 0.015	n.c.	0.012 ± 0.001	< 0.003
Ammonia	OFCEAS_1	0.1	n.c.	< 0.01	< 0.01	n.c.	< 0.01	< 0.01
H2S	OFCEAS_1	Subset of total sulphur	n.c.	< 0.007	0.009 ± 0.001	n.c.	< 0.007	< 0.007
COS	OFCEAS_1	Subset of total sulphur	n.c.	0.104 ± 0.005	0.103 ± 0.005	n.c.	0.098 ± 0.005	0.098 ± 0.005
Halogenated compounds	TD-(GC-) ECD	0.05	n.c.	< 0.001	< 0.001	n.c.	< 0.001	< 0.001
Sum of CO/HCHO/HCOOH	GC-PDHID + OFCEAS_2	0.2	n.c.	0.26 ± 0.04	0.28 ± 0.1	n.c.	0.23 ± 0.03	0.18 ± 0.03

The results reported in Table 6 are compliant with the expected values from the NPL reference materials (except for water and for the calculated amount of total non-methane hydrocarbons). All the others compounds are agreeing with the expected values. Therefore, ZSW is considered capable of performing the measurement of all contaminants requested in ISO/FDIS 14687 and EN 17124:2018. The differences on the GC-PDHID and FID measurements are further being investigated. The high variance on the PDHID is suspected to come from potentially permanently contaminated chromatographic columns which will be replaced in Q3 2019. The differing measurements of total hydrocarbons via FID may come from manual integration methodology and unstable zero-level signals. The fault in water measurement is expected to be result of lacking experience with the newly commissioned device. The repeated measurements of the NPL gas standards will be done after all mediation approaches are implemented and validated against other available and ordered gas standards.

ZBT analyzed the NPL gas cylinder with the mass spectrometer with regard to the gas components mentioned. The results are shown in Table 7. The analyzer was calibrated with a certified test gas cylinder from Air Liquide.

Table 7. Results of analytical performance from ZBT on NPL reference material using mass spectrometer (IMR-/EI-MS) as analytical instrument.

Impurity	Analytical Method	ISO/FDIS 14687 [µmol/mol]	Calibration gas from Air Liquide [µmol/mol]	Certified amount in NPL cylinder [µmol/mol]	Result from ZBT [µmol/mol]
Methane	IMR-MS	100	2.509 ± 0.05	99.2 ± 3.0	> UDL
Total non methane Hydrocarbons	Calculated	2	n.c.	1.018 ± 0.015	1.024 ± 0.02
Helium	EI-MS	300	312.2 ± 6.244	293 ± 21	460.98 ± 20.8
Argon	EI-MS	300	96.6 ± 1.932	293 ± 21	292.18 ± 2.0
Nitrogen	EI-MS	300	101.7 ± 5.09	294 ± 21	348.70 ± 7.6
Carbon Dioxide	IMR-MS	2	2.155 ± 0.043	2.103 ± 0.032	2.398 ± 0.1
Carbon Monoxide	IMR-MS	0.2	2.053 ± 0.041	0.199 ± 0.007	<LOD*

* due to cross sensitivities a.o. to ethane

When determining gas components with the mass spectrometer, cross sensitivities must be considered. For example ethane (C_2H_6) has a strong cross sensitivity to CO and CO_2. Investigations on cross sensitivities are ongoing. As today, these investigations are not finalized and because there is ethane in the NPL bottle, CO_2 and especially CO can not be analyzed properly currently. In addition, the manufacturer of the mass spectrometer is working on optimization of the parameters. After these optimizations, the deviations of the analysis results should be significantly lower. Furthermore, the other detectors for CO, sulfur, water etc. will be in use soon.

2.3.2 European Laboratory compliance and milestone

Based on the achievements of NPL and ZSW, the consortium concluded that these two European laboratories out of the three laboratories selected in HYDRAITE project are currently capable of providing the measurement of hydrogen contaminants as requested in ISO 14687 and EN17124:2018.

ZBT is currently finalising the implementation of their analytical laboratory. ZBT should be operational and capable of providing the measurement of hydrogen contaminants as requested in ISO 14687 and EN17124:2018 by December 2019.

The milestone is considered acceptable as 2 out of 3 independent European laboratories are capable of performing hydrogen fuel quality measurement according to the international and European standards.

2.3.3 Perspective and ongoing progress

As a part of this project, NPL and ZSW have already demonstrated capability to perform hydrogen fuel quality measurements according to ISO/FDIS 14687 and EN 17124:2018. ZBT will achieve similar capability before the next sampling campaign in 2020. It is critical to understand that performing the measurement and ensuring accurate results is different. Currently, no laboratory worldwide can demonstrate full accuracy of measurement regarding ISO 14687 as no inter-laboratory comparison is covering all the compounds or reference materials available for all contaminants at the respective threshold amount fraction.

The objective of HYDRAITE is not only to implement three European laboratories with the analytical capabilities, but also to provide a strong evidence on the quality and the reliability of their results to provide the consumers and the end-users with trust in the reported value. The laboratories are currently working towards ensuring adherence to a future standard ISO 21087:2019 [22]. The standard will require method validation for all analytical methods used to perform hydrogen fuel quality. As the standard was prepared during the timeline of the project, the three laboratories are still working to provide all documentations and compliance to the standard when it will be published (expected in 2019).

In the next part of the project, the three laboratories will perform the measurement of hydrogen fuel quality from hydrogen refuelling stations in Europe. The focus will be on comparing the results of analysis, evaluating and understanding the differences. This quality control study will be performed by bilateral comparison using same samples between the three laboratories and participating in inter-laboratory comparisons (i.e., EMPIR MetroHyVe inter-comparison). During the course of HYDRAITE project, other commercial laboratories have developed capability to perform ISO/DIS 14687 hydrogen fuel quality analysis such as Linde's cental laboratory in Germany or Air Liquide's CEMIAG laboratory in France. HYDRAITE partners will propose bilateral comparison with these laboratories in order to continue the effort in quality and accuracy improvement of the European laboratories.

Acknowledgements

This project has received funding from the Fuel Cells and Hydrogen 2 Joint Undertaking under grant agreement No 779475. This Joint Undertaking receives support from the European Union's Horizon 2020 research and innovation programme, Hydrogen Europe and Hydrogen Europe research.

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