Ref. Ares(2025)10382012 - 27/11/2025

DELYCIOUS

Clean Hydrogen Partnership

Co-funded by the European Union

D3.2 Lab scale Raman test report

By HORIBA

The project is supported by the Clean Hydrogen Partnership and its members under the GA 101192075. Co-funded by the European Union. Views and opinions expressed are however those of the author(s) only and do not necessarily reflect those of the European Union or the Clean Hydrogen Partnership. Neither the European Union nor the granting authority can be held responsible for them.

Document control sheet

Project number	101192075		
Project name	**D**iagnostic tools for **EL**ectrol**Y**sers: **C**ost-efficient, **I**nnovative, **O**pen, **U**niversal and **S**afe		
Project acronym	DELYCIOUS		
Call	HORIZON-JTI-CLEANH2-2024		
Topic	HORIZON-JTI-CLEANH2-2024-01-04		
Type of action	HORIZON-JU-RIA (Research and Innovation Action)		
Granting authority	Clean Hydrogen Partnership and European Union		
Start date	01/01/2025	Duration	36 months
Project coordinator	Fraunhofer IWES		

Deliverable ID	**D3.2**
Deliverable Name	**Lab scale Raman test report**
Version	1
Type of Deliverable	R
Work Package No	WP3
Due date	30/11/2025
Actual submission date	27/11/2025

Lead Beneficiary	HORIBA	
Contributor(s)	Artur ALEKSANYAN	
Dissemination level	Public	☒
	Sensitive	☐
Reviewed by	Patrick Teppor (Cell and Stack Testing Manager, STARGATE)	
Document Approval	Internal Reviewer and WP leaders	

Clean Hydrogen Partnership
Co-funded by the European Union
The project is supported by the Clean Hydrogen Partnership and its members under the GA 101192075. Co-funded by the European Union. Views and opinions expressed are however those of the author(s) only and do not necessarily reflect those of the European Union or the Clean Hydrogen Partnership. Neither the European Union nor the granting authority can be held responsible for them.

About DELYCIOUS

As Europe accelerates its transition to clean energy, the Net Zero Industry Act identifies low-carbon hydrogen production as a strategic priority. DELYCIOUS tackles key challenges in water electrolysis technology, focusing on extending electrolyser lifespans and reducing operational costs under variable renewable energy sources. In DELYCIOUS, the development of cost-efficient, innovative, open, universal, and safe diagnostic tools to investigate the chemical and electrochemical properties of electrolysis systems is foreseen. This project combines Raman Spectroscopy (Raman) and Electrochemical Impedance Spectroscopy (EIS), to explore the chemical and electrochemical properties of electrolysis systems. By using both physical and data-driven modeling, it will be possible to identify important degradation parameters. To ensure that the diagnostic tools work effectively in various temperature ranges and across various electrolysis technologies, three technologies namely Alkaline Electrolysis (AEL), Proton Exchange Membrane Electrolysis (PEMEL), and Solid Oxide Electrolysis (SOEL) are addressed in this project, with a demonstration on alkaline electrolysers beyond 100 KW.

Consortium

Fraunhofer IWES
Coordinator

Air Liquide

University of Twente.

DUMAREY

HORIBA

etaflorence

stargate hydrogen

SIVONIC

Clean Hydrogen Partnership
Co-funded by the European Union

The project is supported by the Clean Hydrogen Partnership and its members under the GA 101192075. Co-funded by the European Union. Views and opinions expressed are however those of the author(s) only and do not necessarily reflect those of the European Union or the Clean Hydrogen Partnership. Neither the European Union nor the granting authority can be held responsible for them.

Summary

As part of the DELYCIOUS project, a Raman gas analyser prototype was developed and calibrated using certified gas mixtures to ensure accurate detection of hydrogen (H_2), oxygen (O_2), nitrogen (N_2), and water vapor (H_2O). Calibration was performed with Horiba's **Astra H2** software, which enables intuitive workflow setup and recalibration using minimal amount of reference gases.
Each gas was calibrated using at least two reference points for a full-scale measurement (0 to 100%). This method ensures reliable performance and easy recalibration at client sites.

Key results:

- The analyser accurately measured dry and humid gas mixtures with a limit of detection (LOD) of 1% and with acquisition times optimized at 3–4 seconds.
- In humid mixtures, the analyser successfully identified water vapor peaks and quantified concentrations of gas matrix. Raman gas analyser prototype is not compatible with liquid water; hence, it needs to be removed from the gas stream before the measurements.
- The system supports both **quantitative** and **qualitative** gas detection, including low concentration components below 1%. Quantitative measurement allows for monitoring the concentration dynamics in the framework of manipulations with electrolysers. Qualitative measurement gives additional information about the composition of the gas matrix which could be crucial to detect; Indeed, contaminants and undesirable chemical components can potentially deteriorate the stack.

Clean Hydrogen Partnership
Co-funded by the European Union

The project is supported by the Clean Hydrogen Partnership and its members under the GA 101192075. Co-funded by the European Union. Views and opinions expressed are however those of the author(s) only and do not necessarily reflect those of the European Union or the Clean Hydrogen Partnership. Neither the European Union nor the granting authority can be held responsible for them.

Contents

List of Figures

List of Tables

Clean Hydrogen Partnership
Co-funded by the European Union
The project is supported by the Clean Hydrogen Partnership and its members under the GA 101192075. Co-funded by the European Union. Views and opinions expressed are however those of the author(s) only and do not necessarily reflect those of the European Union or the Clean Hydrogen Partnership. Neither the European Union nor the granting authority can be held responsible for them.

1. Device calibration

The Raman gas analyser prototype was factory-calibrated before delivery, using certified gas mixtures to ensure accurate detection of O_2, H_2, N_2, and H_2O concentrations. Calibration was performed with the **Astra H2** software developed by Horiba, which provides both gas composition analysis and an intuitive step-by-step calibration workflow.

Each gas component requires at least two reference concentrations for proper calibration since it is considered that the concentration scales linearly with the Raman signal strength. For instance, hydrogen calibration involves using 100% N_2 as the zero point and a 10% H_2/N_2 mixture as the span point. This approach allows for efficient recalibration at the client site using a minimal set of certified gas bottles.

Figure 1 illustrates a typical calibration curve for the analyser, showing how unknown gas concentrations are derived from the reference data. The curve enables precise interpolation of Raman signal intensities into concentration values for each gas.

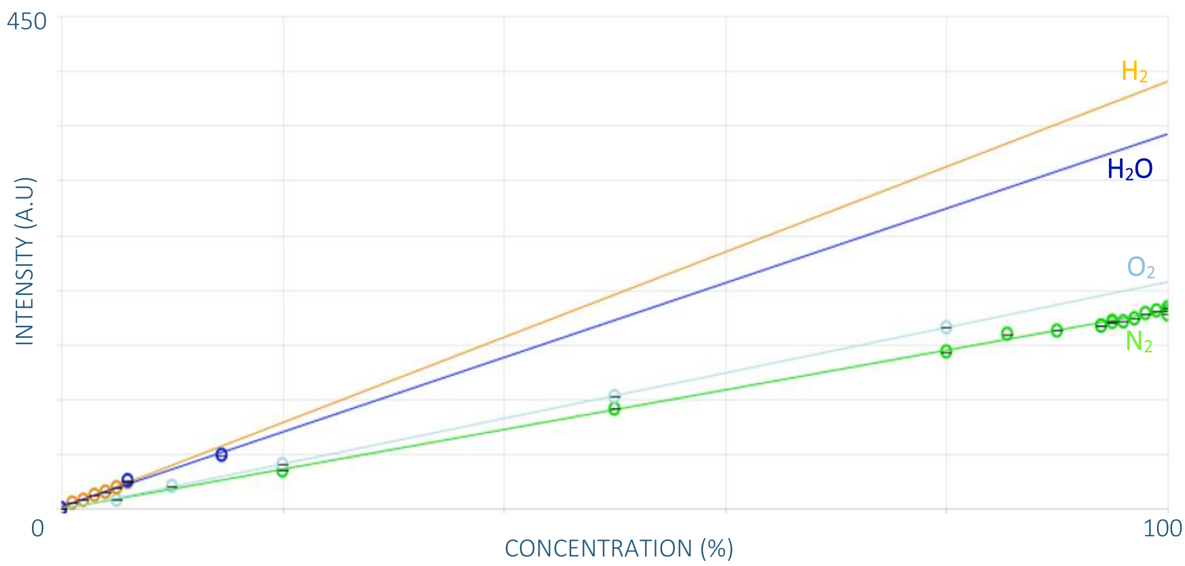

Figure 1 : Calibration curve for each gas component. Orange: H_2, blue: H_2O, green: N_2, light blue: O_2.

2. Measurement

2.1. Quantitative measurements

2.1.1. Dry gas concentrations measurement

Figure 2 presents a typical raw Raman spectrum acquired using the analyzer for a dry gas mixture containing 4% H_2, 19.6% O_2, and 76.4% N_2. Each spectrum was recorded with an acquisition time of 3.5 seconds. Distinct peaks corresponding to each gas are clearly recognizable, and their intensities directly correlate with concentration levels.

Clean Hydrogen Partnership
Co-funded by the European Union
The project is supported by the Clean Hydrogen Partnership and its members under the GA 101192075. Co-funded by the European Union. Views and opinions expressed are however those of the author(s) only and do not necessarily reflect those of the European Union or the Clean Hydrogen Partnership. Neither the European Union nor the granting authority can be held responsible for them.

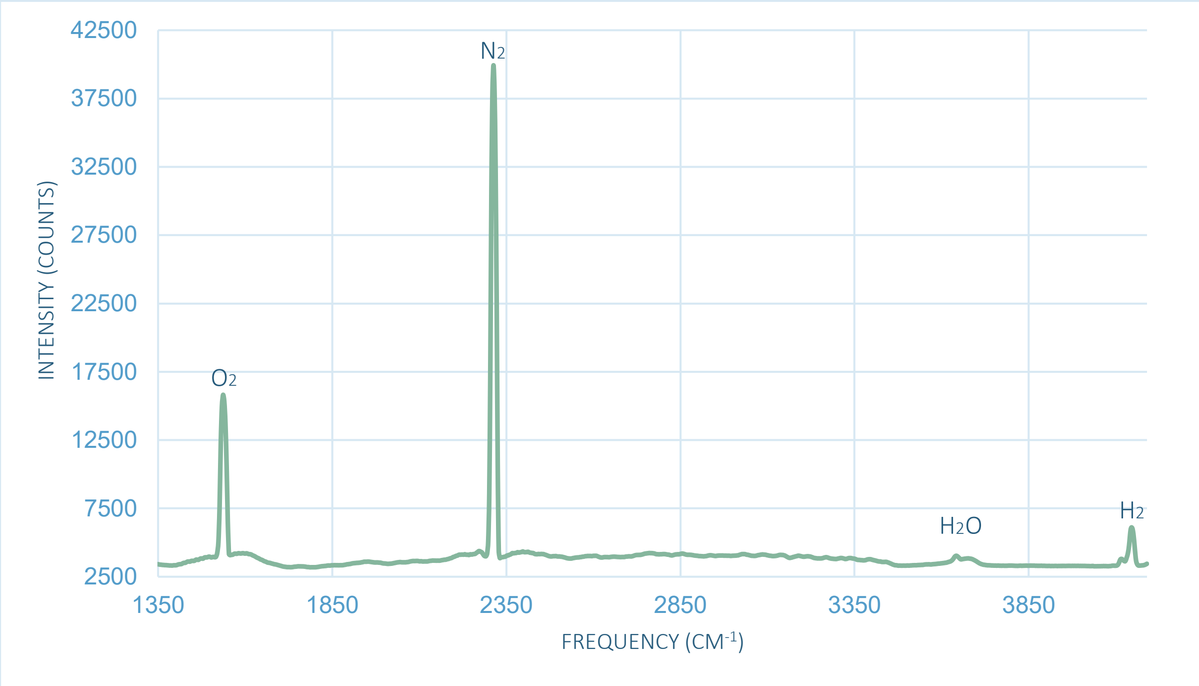

Figure 2: Raw Raman spectrum of the gas mixture (O_2, N_2, H_2)

Table 1 summarizes the average concentrations (30min continuous acquisition) measured by two probes (HEAD 1 and HEAD 2), along with their full-scale (FS) and percent errors relative to the calibrated values. These concentrations are determined after baseline correction of the spectra using automated algorithms provided by the Astra H2 software.

Despite the dry nature of the gas mixture, a small humidity reading (~0.1%) was detected. This is attributed to the prototype's current detection limit (LOD), which is governed by the signal-to-noise ratio of the Raman peaks. While longer acquisition times improve precision (since signal to noise ratio gets bigger), they must be balanced against the risk of detector saturation. Optimal acquisition time for this prototype is around 3–4 seconds, enabling accurate quantitative measurements via Astra H2.

	HEAD 1	Error FS (%)	Error (%)	HEAD 2	Error FS (%)	Error (%)
N_2 (%)	76	0.3	0.4	76.4	0.0	0.0
O_2 (%)	19.8	-0.2	-1.2	19.3	0.2	1.0
H_2 (%)	3.9	0.0	0.8	4	-0.1	-1.9
H_2O (%)	0.1	-0.1	100.0	0.1	-0.1	100.0

Table 1: Measured average concentrations for each gas using the data acquired via the Raman gas analyzer software. Head1 and Head2 stand for the two Raman probes, FS stands for full-scale.

Clean Hydrogen Partnership
Co-funded by the European Union
The project is supported by the Clean Hydrogen Partnership and its members under the GA 101192075. Co-funded by the European Union. Views and opinions expressed are however those of the author(s) only and do not necessarily reflect those of the European Union or the Clean Hydrogen Partnership. Neither the European Union nor the granting authority can be held responsible for them.

2.1.2. Humid gas concentrations measurement

Figure 3 shows the typical spectra for a humid complex gas mixture, namely, 70.4% N_2, 18.4% O_2, 3.7% H_2, and 7.6% H_2O. As we can observe, a marked peak appears on the spectra corresponding to the Raman response of the water vapor.

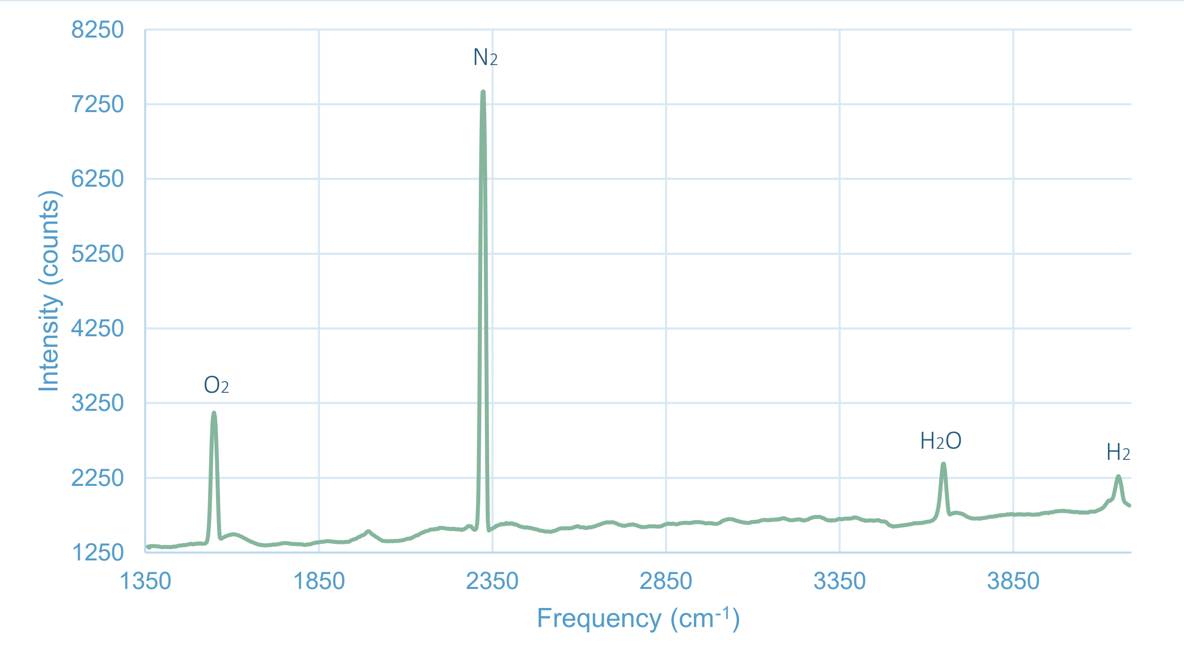

Figure 3: Raw Raman spectrum of the gas mixture (O_2, N_2, H_2, H_2O)

Table 2 summarized the average concentrations measured by two probes (HEAD 1 and HEAD 2) along with their full-scale (FS) and percent (Pt) errors relative to the calibrated values. As we can observe in Table 2, the measurement uncertainty stays <1%, however, the relative error becomes bigger for lower concentrations (see the measurement for H2 at 4.3%).

	HEAD 1	Error FS (%)	Error (%)	HEAD 2	Error FS (%)	Error (%)
N_2 (%)	70.2	0.1	0.2	70.8	-0.4	-0.6
O_2 (%)	19.3	-0.9	-4.8	18.4	-0.1	-0.4
H_2 (%)	4.3	-0.6	-13.1	4.0	-0.3	-8.0
H_2O (%)	6.2	1.3	21.5	6.8	0.8	11.8

Table 2: Measured average concentrations for the humid gas with the following composition: 70.4% N_2, 18.4% O_2, 3.7% H_2, and 7.6% H_2O

The project is supported by the Clean Hydrogen Partnership and its members under the GA 101192075. Co-funded by the European Union. Views and opinions expressed are however those of the author(s) only and do not necessarily reflect those of the European Union or the Clean Hydrogen Partnership. Neither the European Union nor the granting authority can be held responsible for them.

2.2. Qualitative measurements

The Raman probe can also be used for qualitative detection of concentration components at <1%. Raw spectral data can be processed by algorithms to identify the presence of gases that produce Raman signals within the detector’s bandwidth (1350–4200 cm^{-1}).

Particularly, besides measuring the four main above-mentioned components, it could be interesting to monitor the presence of carbon dioxide (CO_2) which can come from ambient air contamination during the stack deterioration. The Raman peak of CO_2 is positioned at 1388cm-1. Other trace components could be present as well, such as hydrocarbons or ammonia (NH_3), however these concentrations will be challenging to detect with the current prototype performances.

Clean Hydrogen Partnership
Co-funded by the European Union
The project is supported by the Clean Hydrogen Partnership and its members under the GA 101192075. Co-funded by the European Union. Views and opinions expressed are however those of the author(s) only and do not necessarily reflect those of the European Union or the Clean Hydrogen Partnership. Neither the European Union nor the granting authority can be held responsible for them.