

20IND06 PROMETH2O

WP3: Demonstration at industrial test beds and facilitation of end-user uptake

Lead VSL

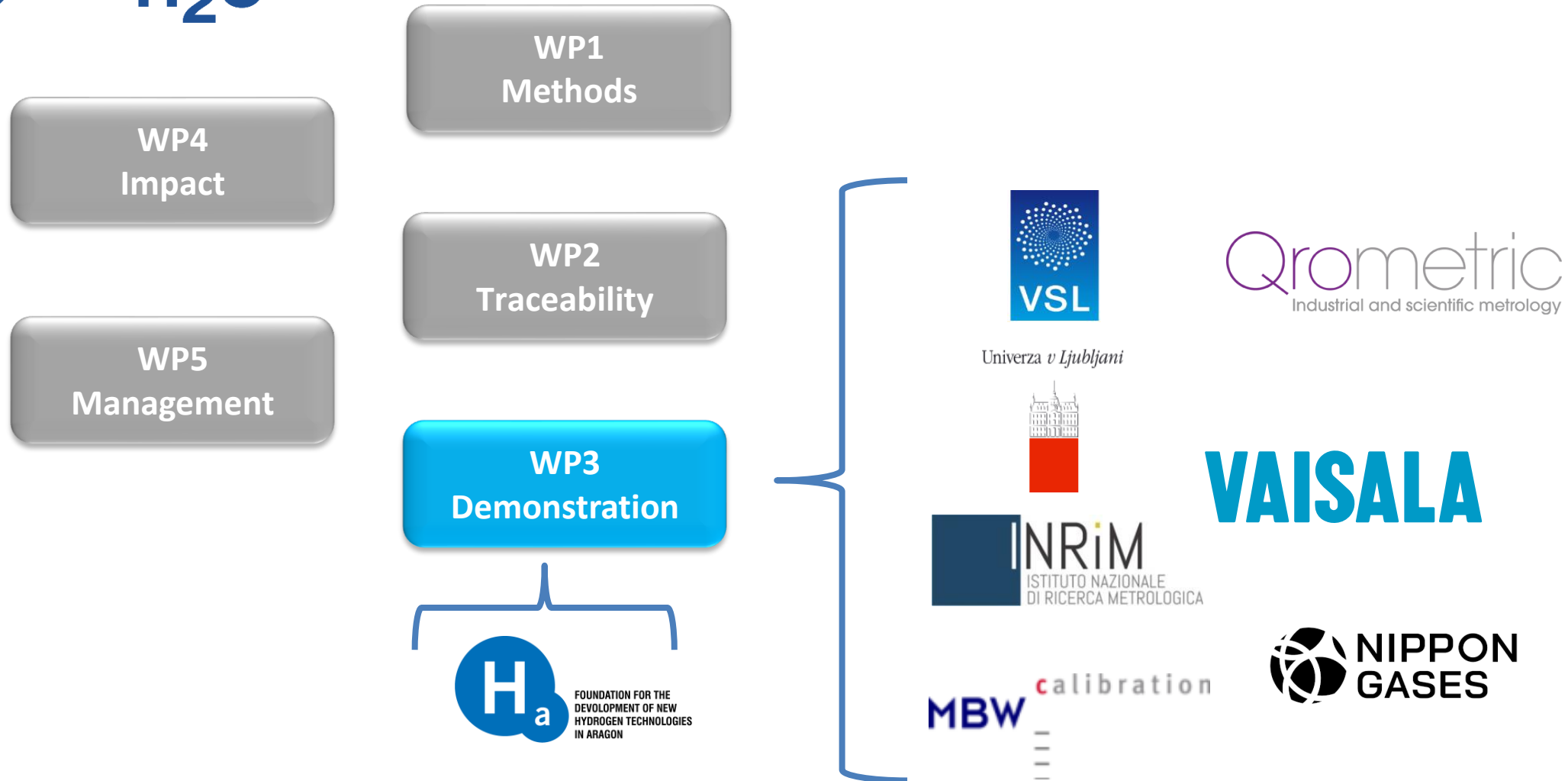
Project M27 meeting, online

27 September 2023

EMPIR



The EMPIR initiative is co-funded by the European Union's Horizon 2020 research and innovation programme and the EMPIR Participating States



Based on stakeholders' needs,
WP3 aims to **demonstrate in a *relevant industrial setting*:**

- the improved trace water measurement methods and techniques (WP1)
- the trace water generation methods (WP2)

and to facilitate end-user uptake of the technology.

Task 3.1: inventory will be made using stakeholder needs of the technical features of the test beds, with respect to the technical and logistical requirements for the demonstration.

Demonstration at selected industrial test beds:

Task 3.2, Test bed 1 will deliver a toolkit of metrological solutions to provide measurement traceability in the field.

- Qrometric site (UK) offers a wide range of different generated conditions (N_2 and CO_2 and later on Ar) with humidity levels down to $-75\text{ }^{\circ}C_{fp}$ but with a target of $-90\text{ }^{\circ}C_{fp}$ in development as part of this WP.
- FHa: electrolyzer facility in Spain (collaborator)

Task 3.3, Test bed 2 will assess the production quality of bulk and specialty gases of a major specialty gas company facility.

- The production facility of Nippon Gases (Italy) includes pure & UHP gases and moisture mixtures filling. Access to the company calibration laboratory will enable onsite testing of sensors and instrumentation.

[illegible]

Task 3.1 Inventory of stakeholder needs and protocols for the demonstrations at the test beds

Reported by INRIM at last meeting. Activities in line with stakeholder needs. Strong emphasis on H₂ (this would add extra value to the demonstration at FH_a)



Activity number	Activity description	Partners (Lead in bold)
A3.1.1 M18	INRIM, with support from VSL, UL, Qrometric, DTU, Nippon Gases, and Vaisala will collect the stakeholder needs through the Steering Board. This collection of data will include specifications on gases, dew-point range, pressure range and other relevant information and will be gathered either through Steering Board meetings or mailed questionnaires.	INRIM , VSL, UL, Qrometric, DTU, Nippon Gases, Vaisala
A3.1.2 M21	Qrometric, with support from INRIM, DTU, and UL will do an inventory of logistic, safety and technical requirements of the Test bed 1. The inventory will be used for the demonstration in A3.2.3.	Qrometric , INRIM, DTU, UL
A3.1.3 M21	Nippon Gases, with support from INRIM, DTU, and VSL will do an inventory of logistic, safety and technical requirements of the Test bed 2. The inventory will be used for the demonstration in A3.3.2.	Nippon Gases , INRIM, DTU, VSL
A3.1.4 M27	Using the data from A3.1.1 and the results of the inventory for Test Bed 1 (A3.1.2), Qrometric, with support from INRIM, DTU, and UL will write a protocol specifying the details of the demonstration such as timing, logistics, pressure and temperature range, gases under test and safety issues. Qrometric will update the protocol after the meeting in A3.2.1 (for the demonstration at Test bed 1).	Qrometric , INRIM, DTU, UL
A3.1.5 M27	Using the data from A3.1.1 and the results of the inventory for Test Bed 2 (A3.1.3), Nippon Gases, with support from, INRIM, DTU, and VSL, will write a protocol specifying the details of the demonstration such as timing, logistics, pressure and temperature range, gases under test and safety issues. Nippon Gases will update the protocol after the meeting in A3.3.1 (for the demonstration at Test bed 2).	Nippon Gases , INRIM, DTU, VSL



- A portable generator for dew point sensor calibration [“FPG”]
- Mixed flow system for positive pressure using Nitrogen and CO₂.
- Chilled mirror transfer standard works to -110°Cfp



Status test bed 1 (Qrometric)

A3.1.2 (due Feb 23): Qrometric, with support from INRIM, DTU, and UL will perform an inventory of logistic, safety and technical requirements of the Test bed 1.

- Covered these points in the User Manual documentation prepared by Qrometric. Now available at <https://www.qrometric.com/wp-content/uploads/2023/07/Qrometric-FPG-User-Manual-V1.0.pdf>

A3.1.4 (due Aug 23): U support from INRIM, I logistics, pressure and

- The protocol for th to be ambient humidit validated in this range
- A production unit i insurance does not cov

A3.2.1 (due Aug 23): C demonstration at Test

- Invited partners to premises
- The MBW SLX is working and will be calibrated by INRIM. This will provide the traceability on FPGs and will be disseminated to the group.



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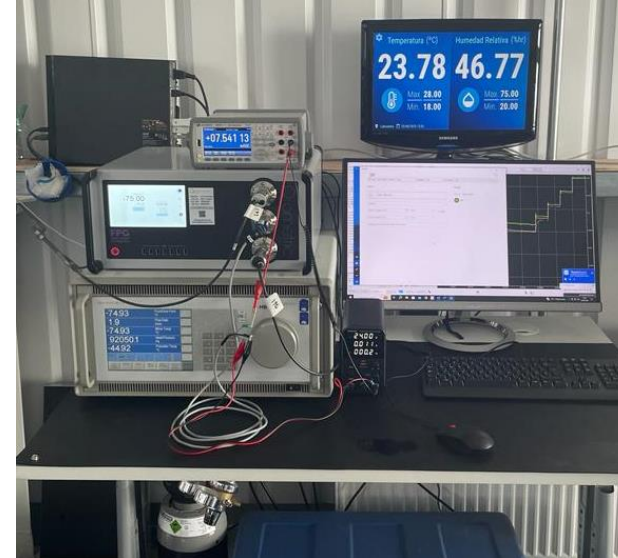
up to 1MPa). Qrometric

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Status test bed 1 (Qrometric)



Calibration of 373LX and Suto DP probe by comparison to SLX with FPG generator



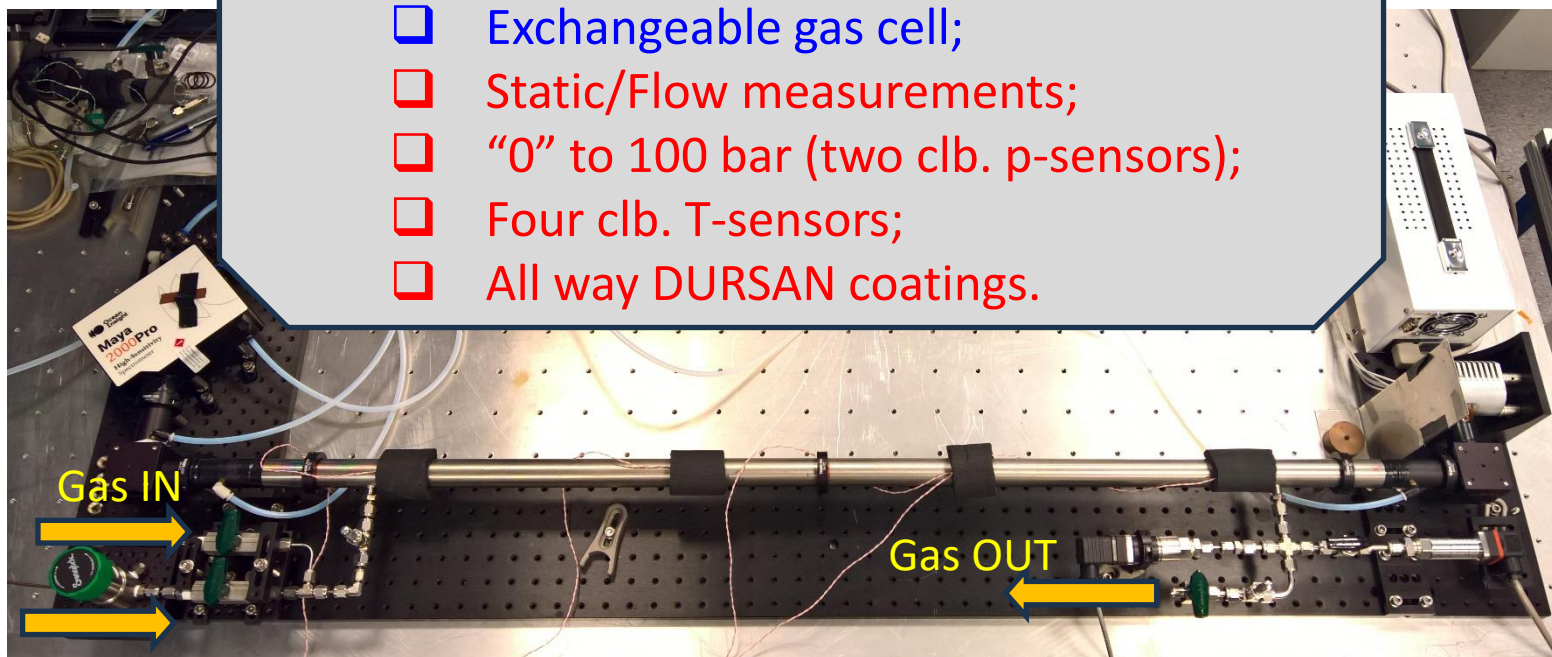
3 x DP probes (1 analogue) vs 373LX in FPG



Demonstration at Qrometric (Portslade, UK)

- Qrometric recently moved to new facilities
- Set dates: Monday 23-Wednesday 25 October.
- Travel to Gatwick is easiest. Pick-up from the airport can be arranged.
- Details of suggested hotels for attendees will be shared.
- No special safety requirements.
- Present at the demonstration: Qrometric, UL (Domen till maximum noon on Wednesday), Process Insights (Thomas), possible also Vaisala (to be checked internally). DTU present at later dates (27 Nov -1 Dec).

- ☐ Modular system;
- ☐ Exchangeable gas cell;
- ☐ Static/Flow measurements;
- ☐ "0" to 100 bar (two clb. p-sensors);
- ☐ Four clb. T-sensors;
- ☐ All way DURSAN coatings.



- Gases: Ar, N₂, H₂, CH₄, etc.
- Two gas inlets;
- Ar or N₂ purge;
- Measurement time per 1×data set can be in ms;
- Spectral range limited by cut off sapphire windows;
- Can be used with other spectrometer, windows and light-source: extended spectral range version.

Preliminary plan (week 48):

- ☐ Nov. 28th(can also be 27th): arrival to the Qrometric (afternoon), install the system. The system will be purged during night.
- ☐ Nov. 29th : measurements with Ar or N₂ with ppm-H₂O at 1 bar;
- ☐ Nov. 30th: measurements with Ar or N₂ with ppm-H₂O at X(?) bar;
- ☐ Dec. 1st: measurements Ar or N₂ with ppb-H₂O at X(?) bar, pack the van in afternoon/evening;
- ☐ Dec. 2nd Leave back to DK.

Task 3.2 Provision of measurement traceability in the field

Activity number	Activity description	Partners (Lead in bold)
A3.2.1 M27	Qrometric, with support from INRIM, UL, MBW, and Vaisala will prepare a meeting for the demonstration at Test bed 1. This meeting will provide updates (e.g. facility access, logistics, and safety issues) to the measurement protocol generated in A3.1.4. Qrometric will record lessons learned during this activity.	Qrometric , INRIM, UL, MBW, Vaisala
A3.2.2 M24	Qrometric, with support from MBW, will extend the temperature range of its portable generator of Test bed 1 down to -90 °C frost point. The methods developed in WP1 such as high-quality CMH (A1.2.2) and cavity-enhanced frequency modulated (A1.2.3) will be used in Test bed 1.	Qrometric , MBW
A3.2.3 M30	UL, with support from Qrometric, MBW, and Vaisala will use the measurement protocol developed in A3.1.4, to perform the validation of extended-range to -90 °C portable generator of Test bed 1. UL will also provide advice for the validation steps and will evaluate the performed measurements. All the measurements will be traceable to the standards developed in WP2 such as thermodynamic saturation-based standards (A2.1.1 and A2.1.2). UL will record lessons learned during this activity.	UL , Qrometric, MBW, Vaisala
A3.2.4 M33	Qrometric will perform the onsite calibration of a trace-water process sensor in hydrogen flow. For this demonstration, the consortium will use the facilities at FHa hydrogen production facility. Vaisala will provide current and recent prototype capacitive trace water sensors. Qrometric will record lessons learned during this activity.	Qrometric , VSL, Vaisala
A3.2.5 M36	VSL, Qrometric and Vaisala will prepare a report using the results from A3.2.4 on the demonstration at the FHa hydrogen facility.	VSL , Qrometric, Vaisala
A3.2.6 M32	Qrometric with support from UL, MBW, INRIM, Vaisala and VSL, will write a report on the portable generator (range-extension, validation and field demonstration) results (A3.2.2 to A3.2.4) and discuss compliance with stakeholder needs (A3.1.1) and lessons learned (A3.2.1 to A3.2.4). Qrometric, UL, MBW, INRIM, Vaisala and VSL, will review the report and will send it to the coordinator. Once the report has been agreed by the consortium, the coordinator on behalf of Qrometric, UL, MBW, INRIM, Vaisala, and VSL, will then submit it to EURAMET as D6:	Qrometric , UL, MBW, INRIM, Vaisala, VSL

Task 3.3 Demonstration at a speciality gases production facility

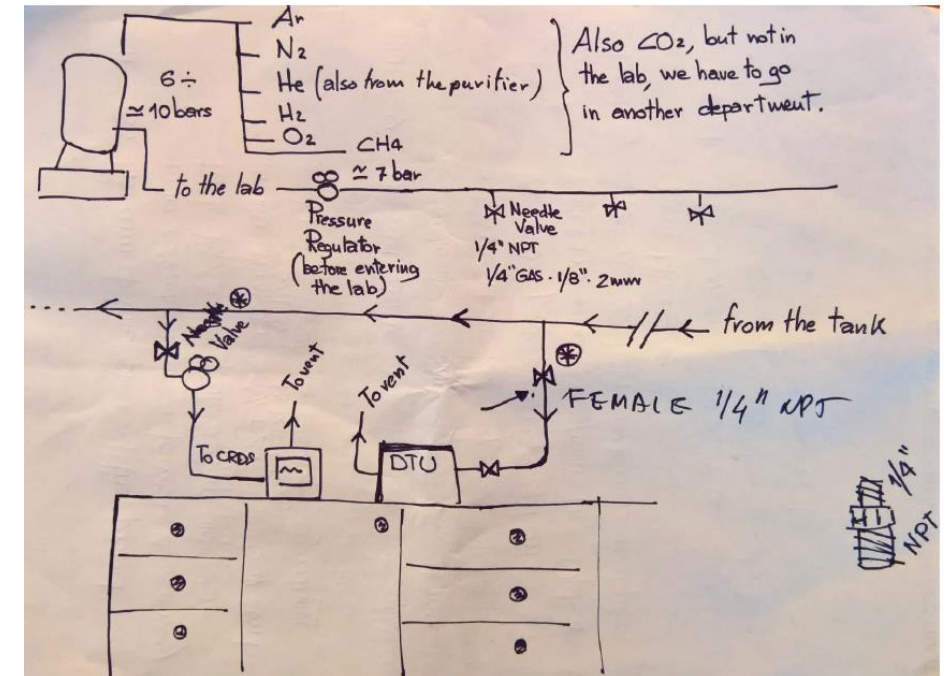
Activity number	Activity description	Partners (Lead in bold)
A3.3.1 M27	Nippon Gases, with the support from INRIM, VSL, DTU, and Vaisala will prepare a meeting for the demonstration at Test Bed 2. This meeting will provide updates (e.g. facility access, logistics, and safety issues) to the measurement protocol developed in A3.1.5. Nippon Gases will record lessons learned during this activity.	Nippon Gases , INRIM, VSL, DTU, Vaisala
A3.3.2 M33	Nippon Gases, with the support from INRIM, VSL, DTU, and Vaisala and using in-house equipment (e.g. CRDS analyser from Tiger Optics and electrolysis-based analyser from Meeco) will follow the measurement protocol developed in A3.1.5, to measure the trace water contamination in N₂ and Ar production in Test bed 2. Additionally, the methods developed in WP1 such as high-quality CMH (A1.2.2) and far-UV system (A1.2.4) will be used in Test bed 2. A prototype capacitive trace water sensor made available from Vaisala will be deployed in the field by Nippon Gases. All the measurements will be traceable to the standards developed in WP2 such as thermodynamic saturation-based standards (A2.1.1) and permeation system (A2.1.4) with a target fraction range from 5 ppm to 5 ppb with relative standard uncertainty less than 3 % to 8 % in selected gas matrices at pressures up to 1 MPa (A2.1.7). Nippon Gases will record lessons learned during this activity.	Nippon Gases , INRIM, VSL, DTU, Vaisala
A3.3.3 M36	Nippon Gases, with the support from INRIM, VSL, DTU, and Vaisala will prepare a report integrating information from the demonstration results at Test Bed 2 (A3.3.2) and discuss compliance with stakeholder needs and lessons learned (A3.3.1-A3.3.2).	Nippon Gases , INRIM, VSL, DTU, Vaisala
A3.3.4 M36	Nippon Gases, INRIM, VSL, DTU, and Vaisala will review the report from A3.3.3 and will send it to the coordinator. Once the report has been agreed by the consortium, the coordinator on behalf of Nippon Gases, INRIM, VSL, DTU, and Vaisala, will then submit it to EURAMET as D7:	Nippon Gases , INRIM, VSL, DTU, Vaisala

- Nippon Gases is one of the major gas company in Europe, and its business comprehend the production of bulk cryogenic liquid, industrial, medical and specialty gases.
- The production of speciality gases is located in Chivasso (TO) that include pure and ultra-high pure gases and moisture mixtures filling. Nippon Gases can so provide a wide range of pure gases at different conditions (matrix gases, pressure) and a wide range of mixtures with different humidity levels. The company offer also the availability to its laboratories, with its instruments and equipment. for all the tests.



Status test bed 2 (NIPPON Gases)

- **A3.1.3 (due February 2023):** Nippon Gases, with support from INRIM, DTU, and VSL will perform an inventory of logistic, safety and technical requirements of the Test bed 2.
- Online discussion with group in December 2022
- Measurements will be made from gas cylinders and from gas tanks
- Capacity tanks: Ar 15 m³, O₂ 20m³, N₂ 20 m³
- No control on the filling level of the tank (possible refill during testing period)
- Length pipelines 30-60 m
- Discussed the available connections (no issues expected due to use standard connections in the facility)
- Safety issues seem limited



Demonstration at NIPPON Gases (**Chivasso, Italy**)

- Set dates: week 45 (Monday 30 Oct - Friday 3 Nov). DTU and NIPPON already agreed on the DTU visit.
- At the end of September, Stefan Boggio will provide information regarding safety etc.
- Present at the demonstration: Nippon Gases, DTU (Alexander), INRIM (1 person), possibly also Vaisala (to be checked internally, possibly 2-3 Nov) and VSL (to be checked internally).



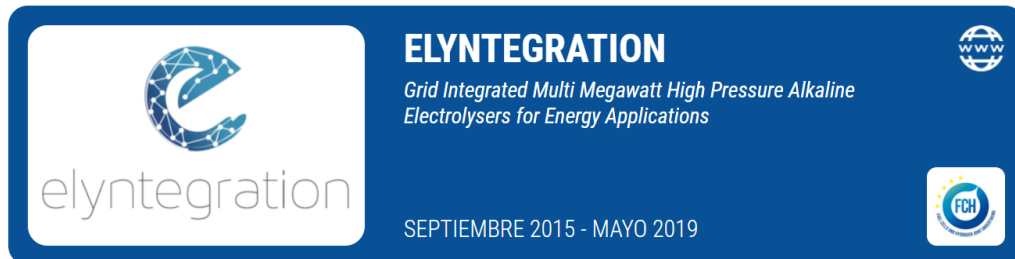
Preliminary plan (week 44):

- ☐ Oct. 29th, evening: arrival to Chivasso (2 days drive from DK);
- ☐ Oct. 30th : meeting at the site, formalities, systems installation, purge connection, trial measurements;
- ☐ Oct. 31st : measurements. we can start from e.g. gas cylinders (Ar, N₂, CH₄, H₂);
- ☐ Nov. 1st: day off, National Holiday (work from the hotel);
- ☐ Nov. 2nd: measurements (from tank: Ar and N₂ as a “must”);
- ☐ Nov. 3rd: measurements (from tank/cylinders: Ar and N₂ as a “must”, CH₄ and H₂ optional) and pack the van in the afternoon;
- ☐ Nov. 4th: leave to DK.
- ☐ Possible adds on: FTIR (MIR, NIR) and extended range far-UV measurements.

Electrolyzer facility of FHA

- **Green Hydrogen Production by alkaline water electrolyzers (AELWE)**
ELYNTEGRATION Project: Goal → Development of new AELWE components for stack assembling and demonstration in dynamic operation (30 bars).
Some of relevant FHa tasks related to Balance of plant (BoP) and high pressure testing.
 - Design, development and implementation of BoP and test benches (pilot scale 10-25 kW, up to 60 bar) for high dynamic operation and accelerated stress tests.

Visit: www.elyntegration.eu



INRIM and VSL had a discussion with Guillermo Figueruelo and Laura Abadia Albas from Fha (22 June). FHa presented the different electrolyser systems, including:

	Principle	H ₂ production (Nm ³ /h)	Pressure (barg)	purity	sensor	Anticipated impurities
1	Alkaline	2.5-10	10-30	Depending on testing condition	Teledyne 8800A Al ₂ O ₃ (pressure regulated to 1.5 bara)	O ₂ , KOH, H ₂ O
2	Alkaline (McPhy)	2.5-10	8	99.5 ± 0.3%		
3	PEM (H2GREEM)	1	6	99.995%		H ₂ O ≤ 600 ppm
4	AEM (ENAPTER)	1.2-2	30	(manufacturer specs dryer: ISO14687 compliant for H ₂ O and O ₂)		

Important issue: ATEX requirement for the measurement systems! What are the possibilities?

- 1) Suggestion Domen: Easidew PRO XP (Michell Instruments) (Specs: -100°C ... $+20^{\circ}\text{C}$ Cdp, accuracy $\pm 1^{\circ}\text{C}$... $\pm 2^{\circ}\text{C}$) Deviations outside -40°C ... -80°C anticipated (feedback Hannu)
- 2) MEECO (next week I hope to get an update on this)
- 3) Process insights: Spark H₂O (CRDS) wit ATEX Shoebox (costly, takes time)
- 4) Manalytical http://www.manalytical.com/products/atex_certified_analysers/
- 5) Vaisala (high dew points, down to -20°C)
- 6) Discuss with FHa possibilities for using non ATEX system





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Thank you for your attention

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