PROMETH$_2$O

A new measurement infrastructure for trace water in ultra-pure process gases

www.prometh2o.eu

INRiM ISTITUTO NAZIONALE DI RICERCA METROLOGICA

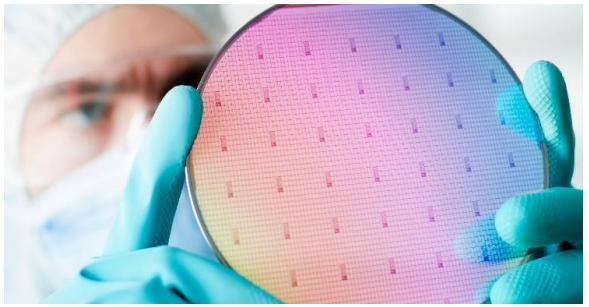
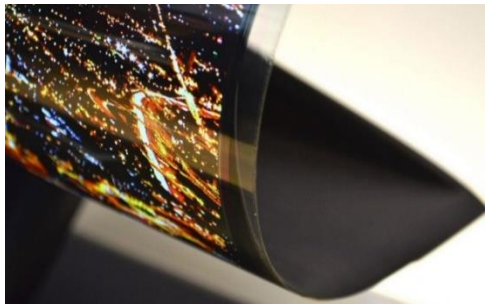
Vito Fernicola (v.fernicola@inrim.it)

on behalf of the PROMETH2O Consortium

EMPIR EURAMET

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

Identified needs

Semiconductor manufacturing - demands for UHP process gases with total impurities as low as few ppb.

Organic electronics - highly moisture-sensitive, needs ultra-dry manufacture and vapour barrier coatings.

UHP bulk process gases - need to be manufactured with total impurities below 1 μmol/mol in volume (grade N6.0 or better).

Utility power generation - needs dry hydrogen (below 5 μmol/mol) to cool down high-efficiency stationary generators.

Instrument manufacturers - need traceable standards to support their product development while end-users rely on them for instrumental testing and calibration.

Project challenges

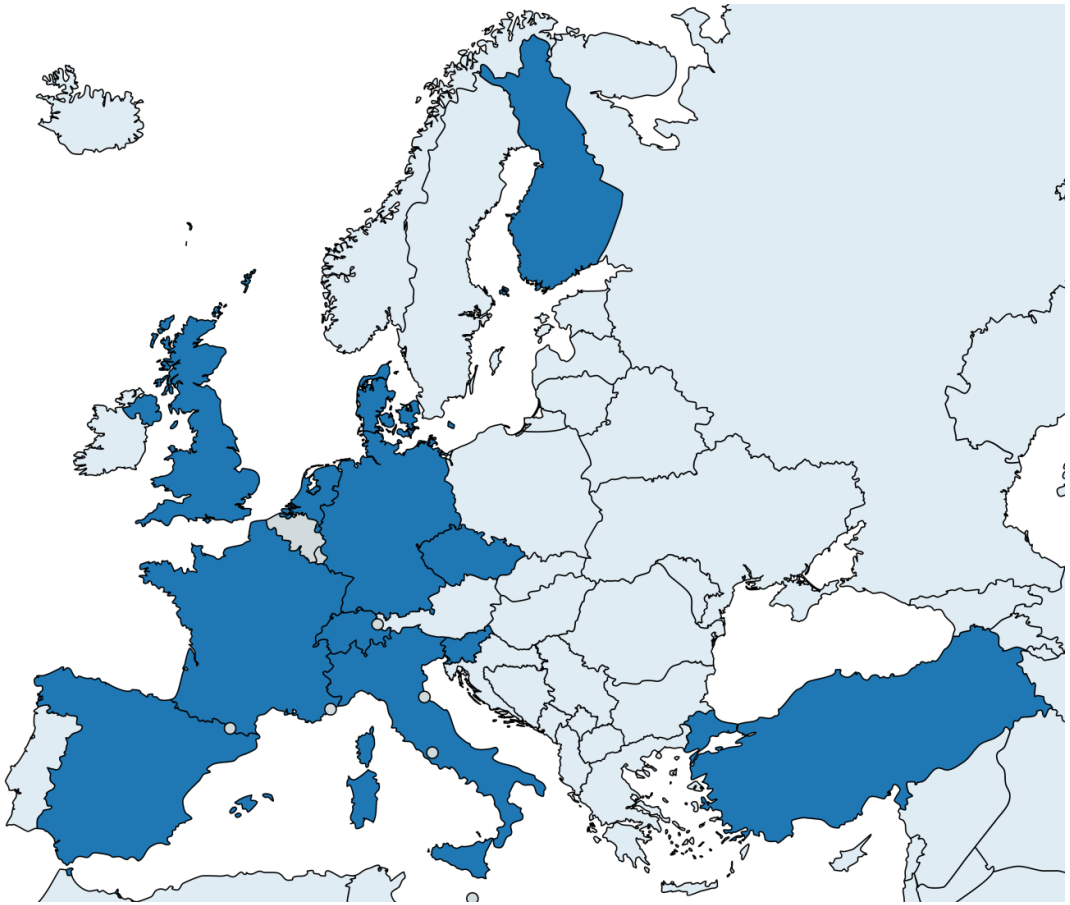
Water vapour is the single largest matrix contaminant in ultra-high purity (UHP) process gases used in key technology areas (e.g. semicon. and optoelectronics).

Its measurement presents great challenges to both process gas producers and analytical instrument makers.

The project is aiming at

- **filling the gap** between the demand of traceable measurements and the available humidity standards currently limited at ~1 ppm (1 μmol/mol).
- **developing traceable and improved methods** for trace water measurement relevant to the production and use of UHP gases.
- **facilitating the uptake of the technology** by the gas industry supply chain through exploiting knowledge and services developed in an European-wide metrology infrastructure.

The research consortium

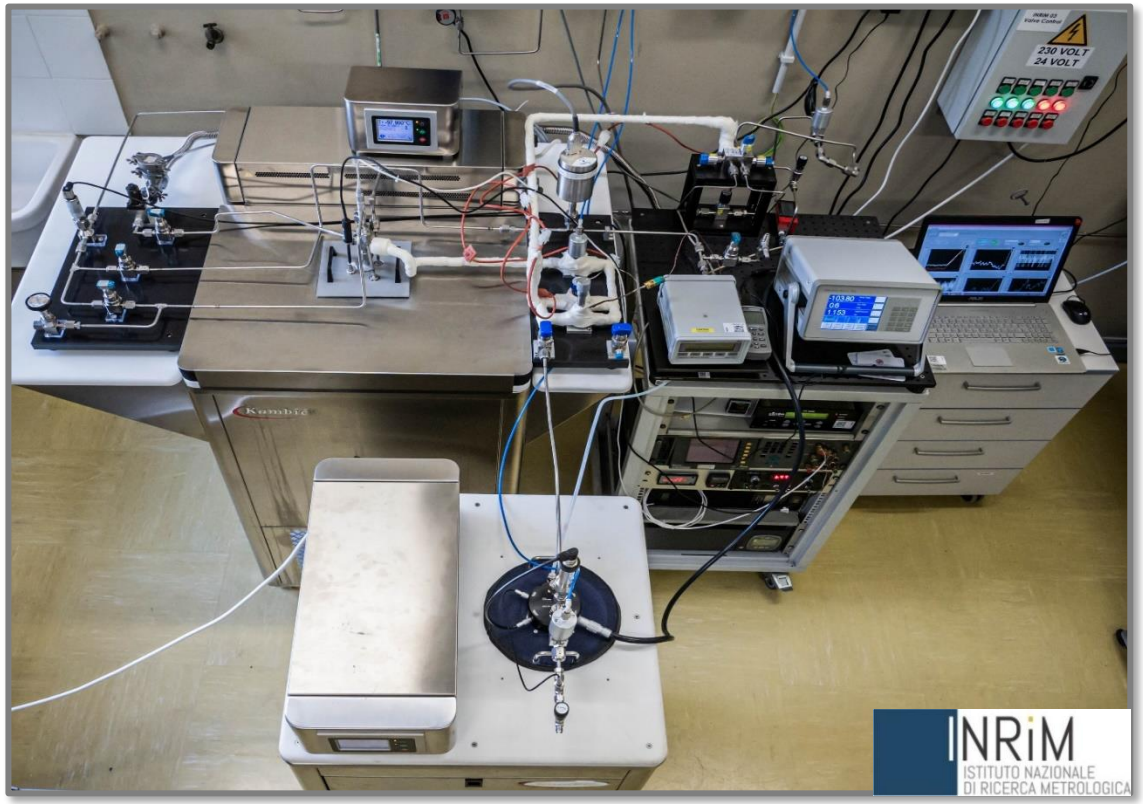


19 partners from 12 countries → 240 person-months

Project objectives

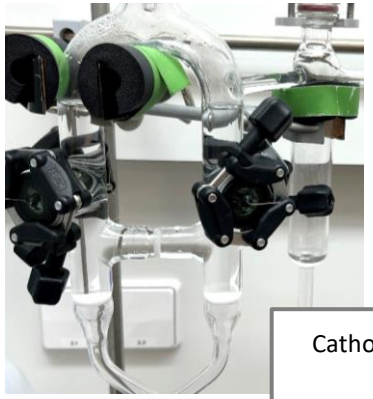
- **New primary standards** for trace water vapour in N_2, Ar and H_2 down to 5 nmol/mol (or -105 °C frost point temperature) at pressures up to 1 MPa.
- **New/improved measurement methods** in the amount fraction range between 5 μmol/mol and 5 nmol/mol (*rel. uncert.* 3 % to 8 %).
- **New data and correlation equations of water vapour enhancement** in N_2, Ar and H_2 in the temperature range from -30 °C to -90 °C and pressures up to 1 MPa.
- **Demonstration at selected industrial facilities** with real-time measurements and on-site calibrations.
- **Provision of a toolkit of metrological solutions** for robust measurement traceability in the production of UHP process gases, by leveraging on improved standards and measurement capabilities.

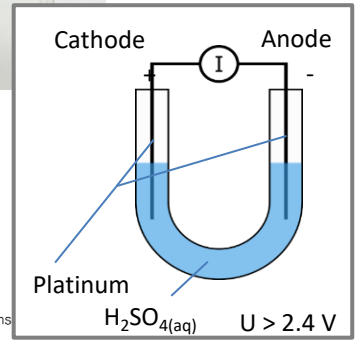
Achievements: primary standards for trace water



Low-frost point primary humidity generator - Mark 2

- Frost-point temperature: -105 °C to -20 °C
- Water vapour mole fraction: 5 nmol/mol to 0.1 cmol/mol
- Pressure: 200 hPa to 0.68 MPa (N_2 and Ar)





PTB
Physikalisch-Technische Bundesanstalt
Nationales Metrologieinstitut

Details of the electrolysis cell

Coulometric Trace Water Generator (CTWG)

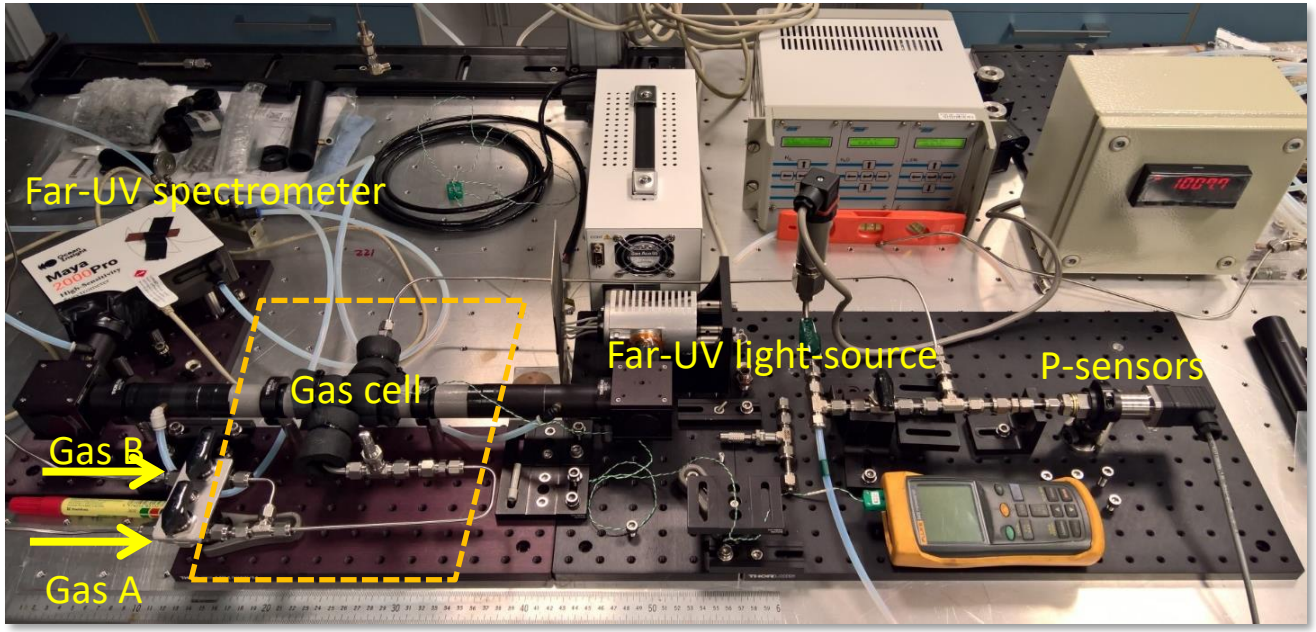
- Amount fraction > 5 ppb
- Rel. uncertainty: 8 %

Permeation system based on a passivated magnetic-suspension balance

VSL

- Range: 50 ppb to 5 ppm

Achievements: improved trace water analysers and enhancement factor measurements

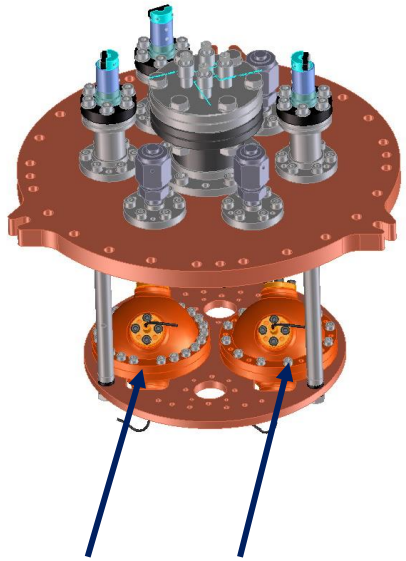


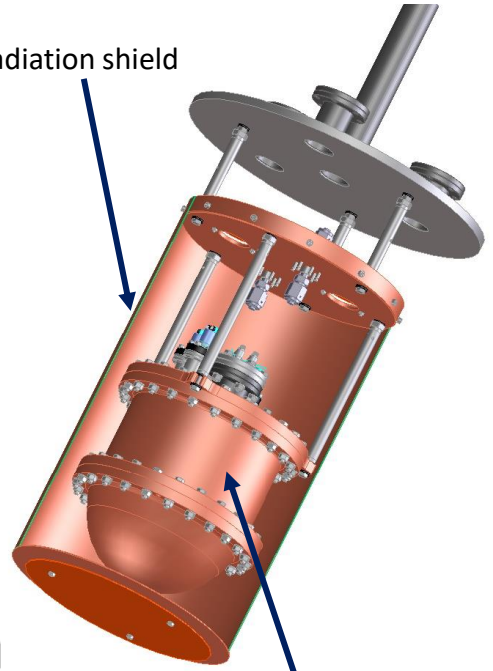
Far-UV system

- Highly-modular gas cells: (0.5 to 100) cm;
- Flow or static measurement: (0 to 100) bar;
- 100-cm cell with DURSAN® coatings;
- Two-way spectra analysis: “full” and “DOAS”

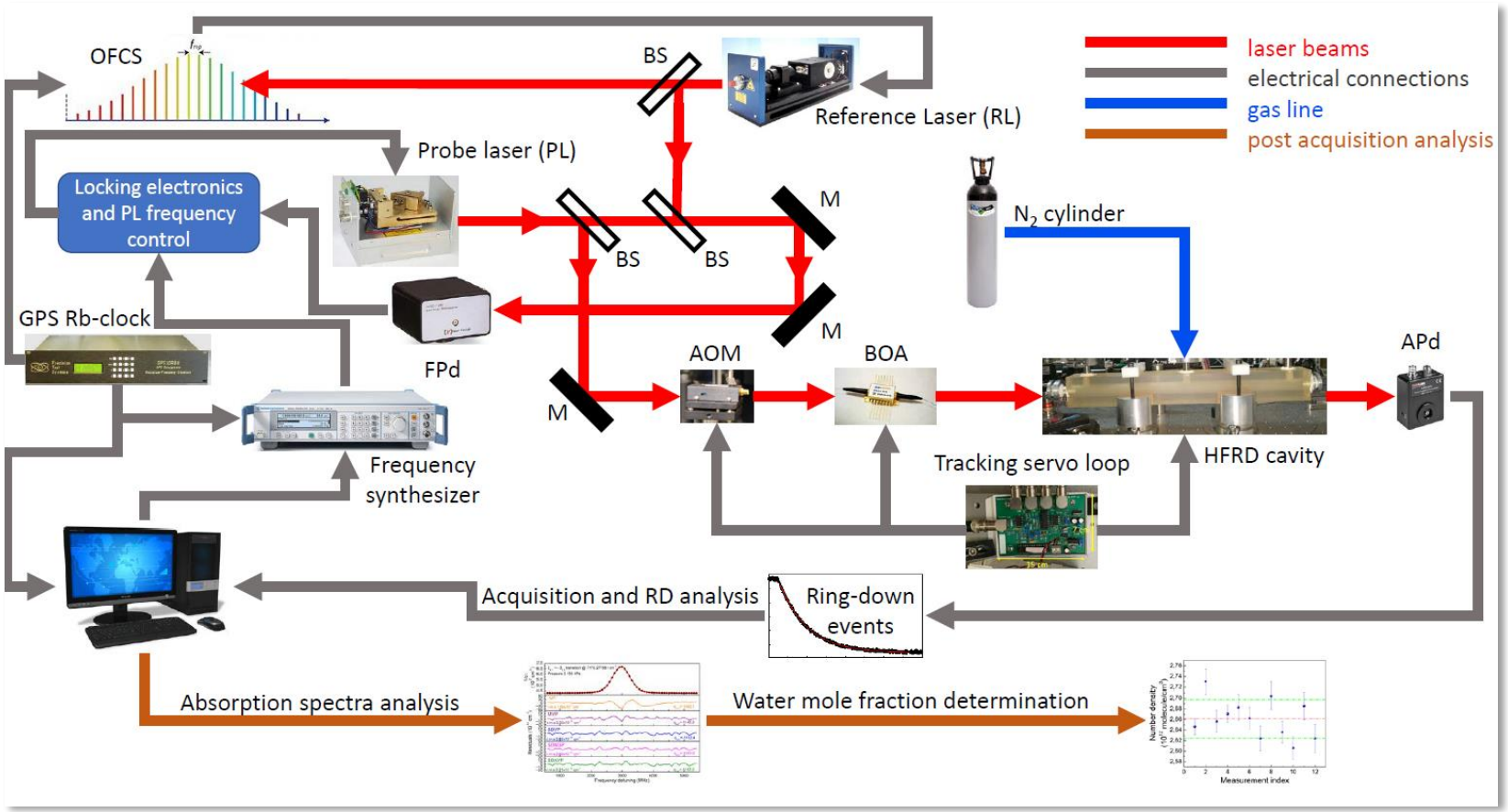
Microwave-based trace water hygrometers

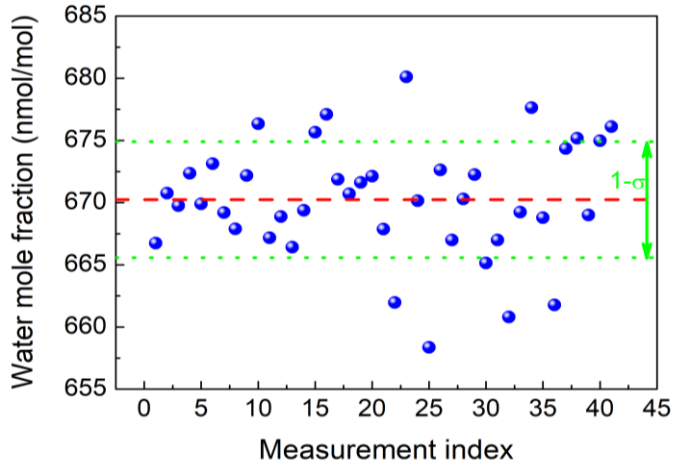
to measure the enhancement factor of H_2O vapour in N_2, Ar, and H_2





Achievements: improved comb-assisted CRDS optical analysers





$$x_w = (671 \pm 4)\ nmol/mol$$

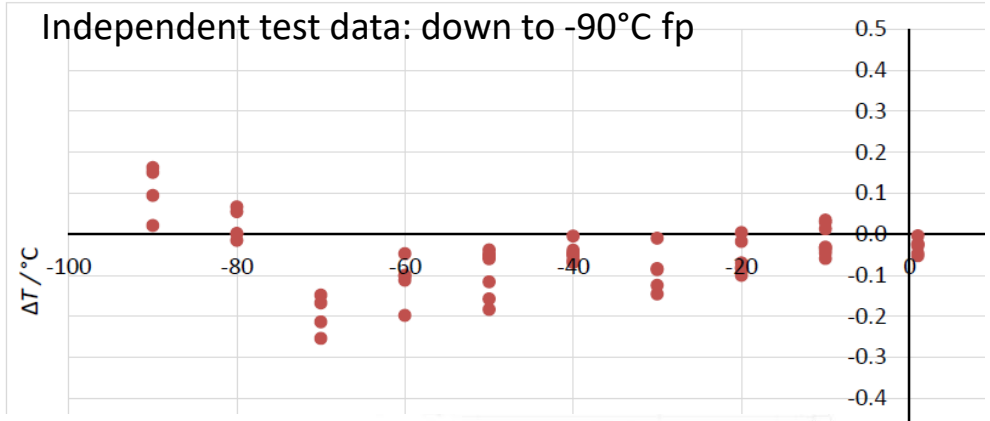
Contribution (k=1)	Type A (%)	Type B (%)
Statistical	**0.5**	
Line strength		**0.3**
Frequency scale		**Negl.**
Line shape model (SDVP)		**0.1**
RD per point & frequency step		**< 0.2**
Laser scan width		**< 0.2**
Gas temperature		**0.05**
Partition function		**0.04**
Pressure		**0.05**
Overall combined uncertainty = 0.7 %		

Università degli Studi della Campania *Luigi Vanvitelli*

Achievements: provision of (on-site) traceability

Accelerated development and validation of a portable frost point generator (FPG)

- Generated conditions: down to -100°C fp
- Independent test data: down to -90°C fp



Traceable assessment of state-of-the-art hygrometers and transmitters

down to -105 °C (5 nmol/mol)

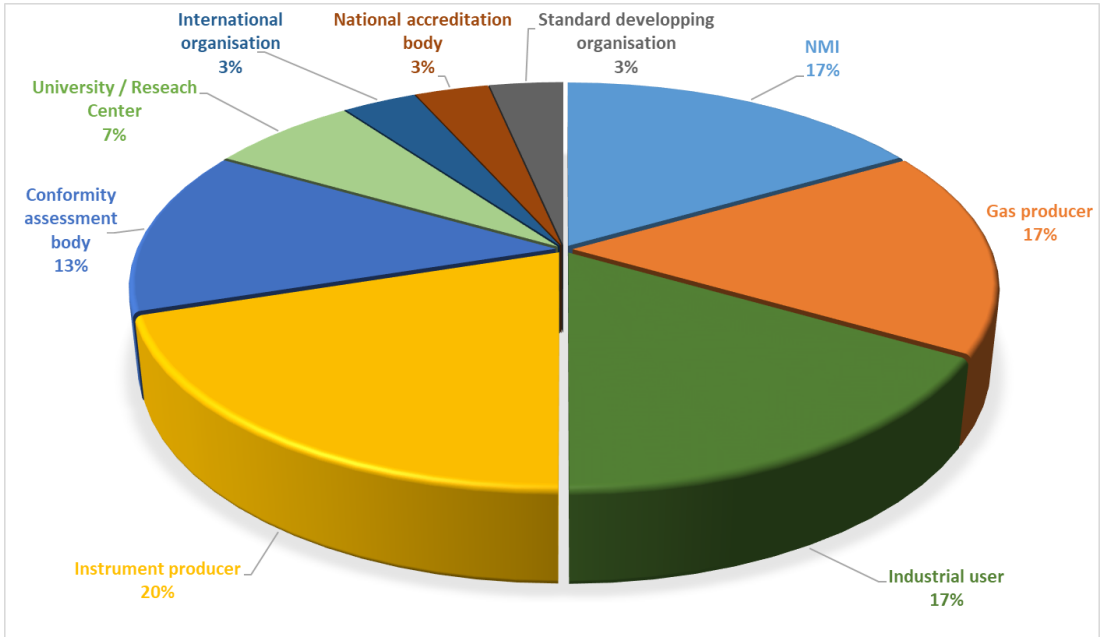
Test bed demonstration

Industrial production facilities of speciality gases that include pure gases, UHP gases and humid gas mixtures

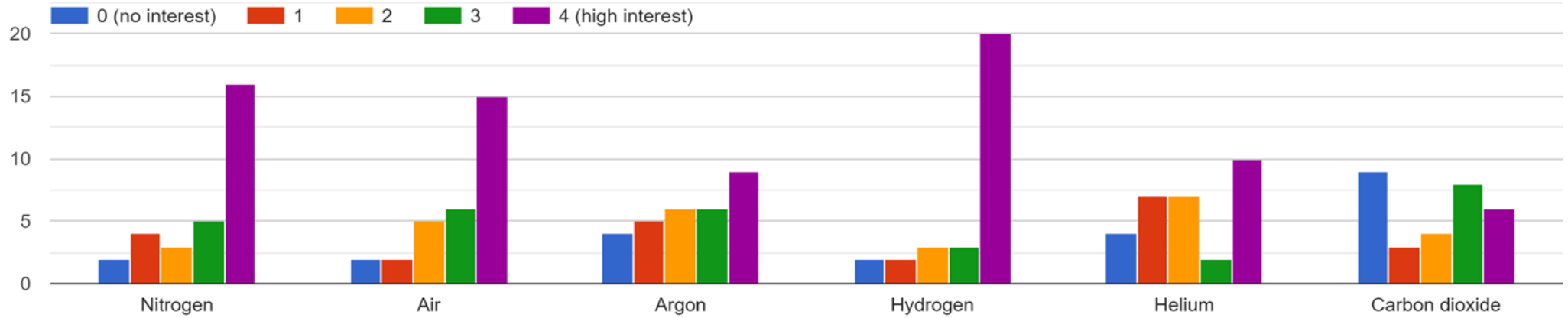
Stakeholder engagement for early impact of research

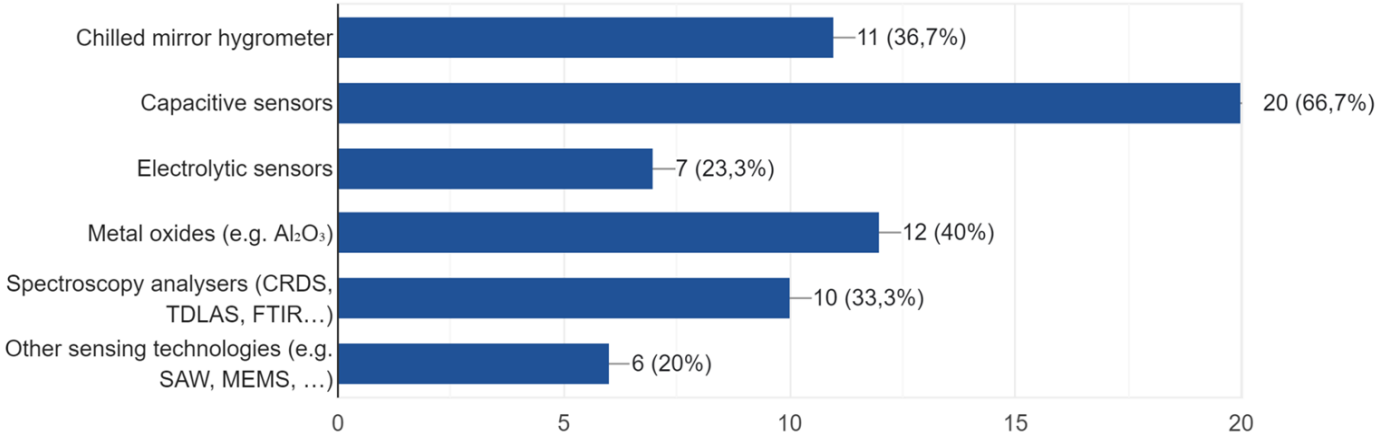
Steering Board made of 21 key stakeholders among gas producers, instrument manufacturers, and international scientific organisations.

Surveying stakeholders needs and priorities



Surveying stakeholder needs and priorities



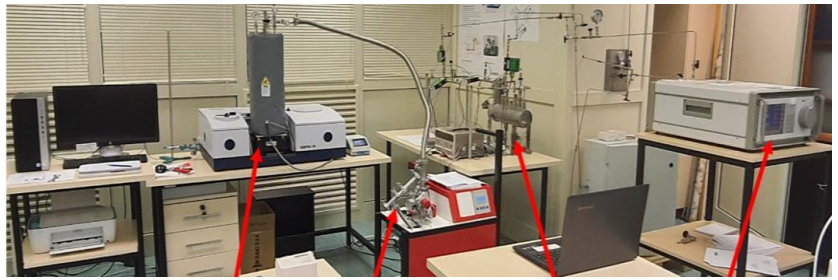


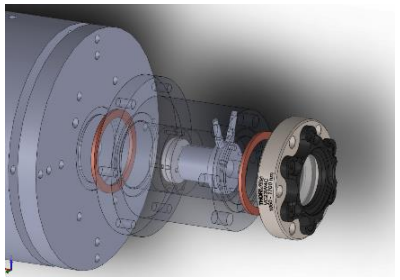
"I need to measure the trace water content in pure process gases as part of the quality assurance process."

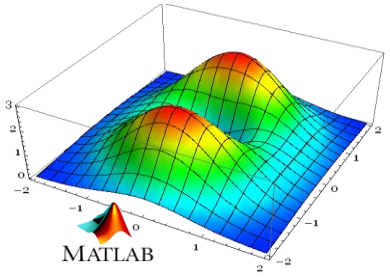
"We focus on hydrogen production by water electrolysis and fuel cell applications."

"We need an international traceable system for calibration at under 0.1 ppm."

To make impact, PROMETH2O will …

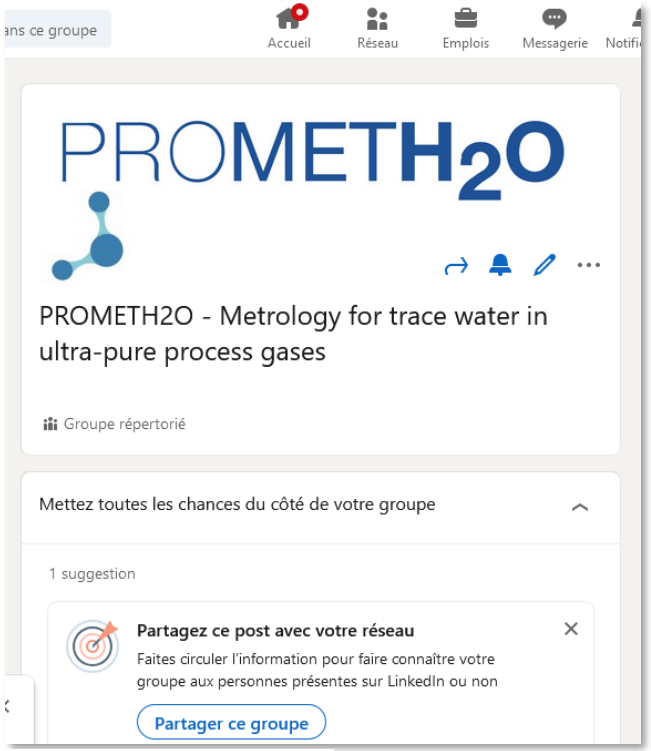






- Make available **primary national standard** to provide traceability for instruments measuring trace water vapour to serve the industry and support the accreditation scope of CABs.
- Recommend **suitable transfer standards** to support future international comparisons in the range from -65 °C to -105 °C (or 5 μmol/mol to 5 nmol/mol).
- Contribute to new or improved international guides, standards and recommended values of **thermo-physical properties of water-gas mixtures**.
- Develop validated software tools to **estimate the enhancement of water vapour** in N_2, Ar and H_2 and its uncertainty in the range between -30 °C and -90 °C and up to 1 MPa.
- Demonstrate **on-site traceable calibration and measurement** of water contamination in UHP process gases at industrially-relevant facilities.

Follow us on …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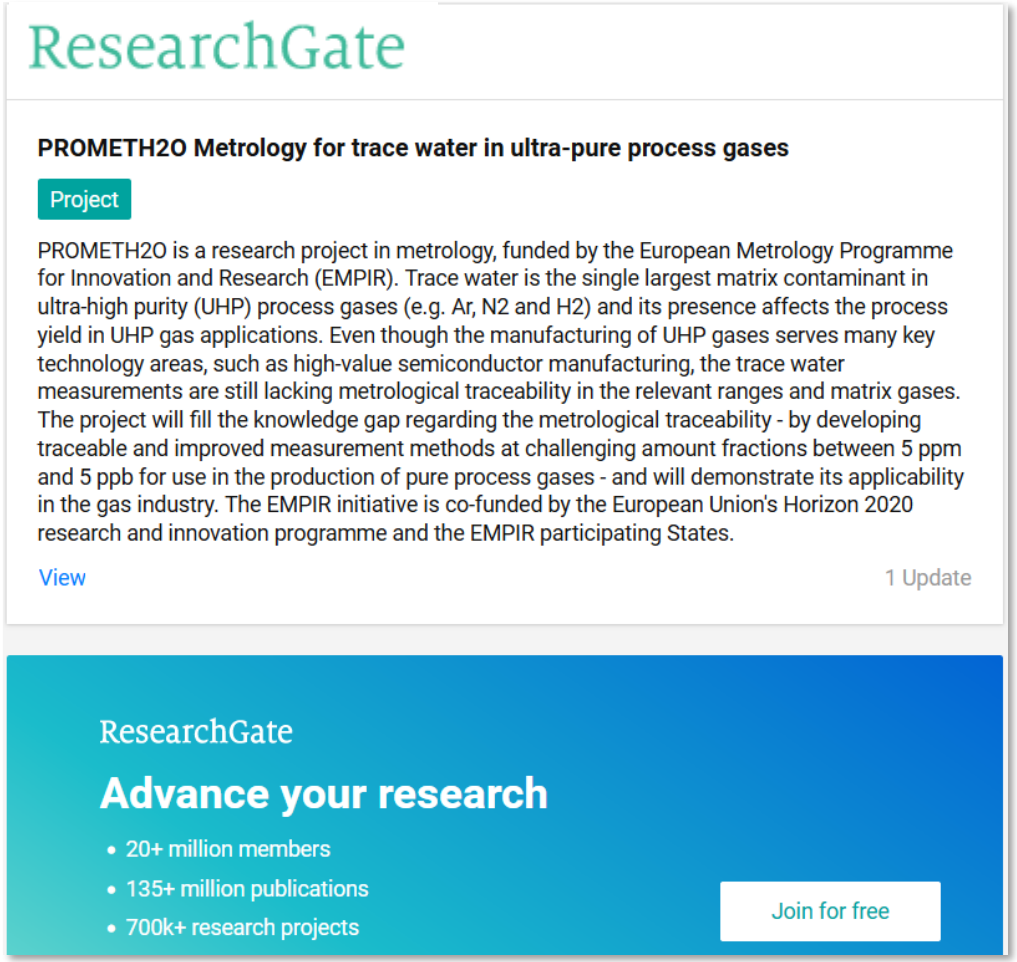
ResearchGate
PROMETH2O Metrology for trace water in ultra-pure process gases
Project
PROMETH2O is a research project in metrology, funded by the European Metrology Programme for Innovation and Research (EMPIR). Trace water is the single largest matrix contaminant in ultra-high purity (UHP) process gases (e.g. Ar, N2 and H2) and its presence affects the process yield in UHP gas applications. Even though the manufacturing of UHP gases serves many key technology areas, such as high-value semiconductor manufacturing, the trace water measurements are still lacking metrological traceability in the relevant ranges and matrix gases. The project will fill the knowledge gap regarding the metrological traceability - by developing traceable and improved measurement methods at challenging amount fractions between 5 ppm and 5 ppb for use in the production of pure process gases - and will demonstrate its applicability in the gas industry. The EMPIR initiative is co-funded by the European Union's Horizon 2020 research and innovation programme and the EMPIR participating States.
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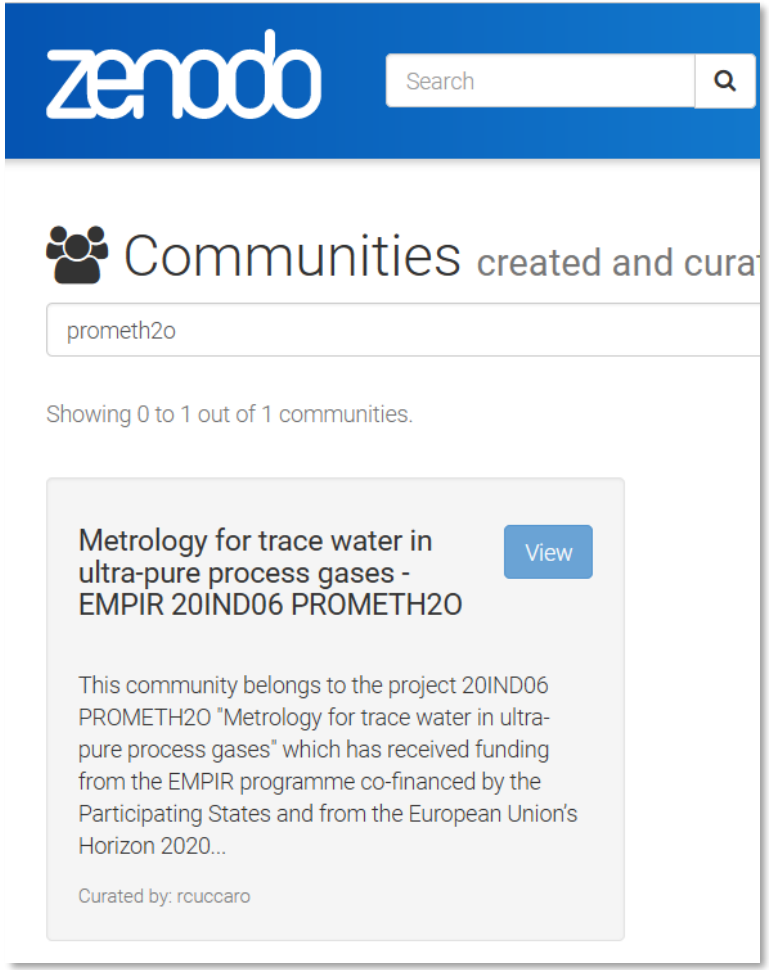
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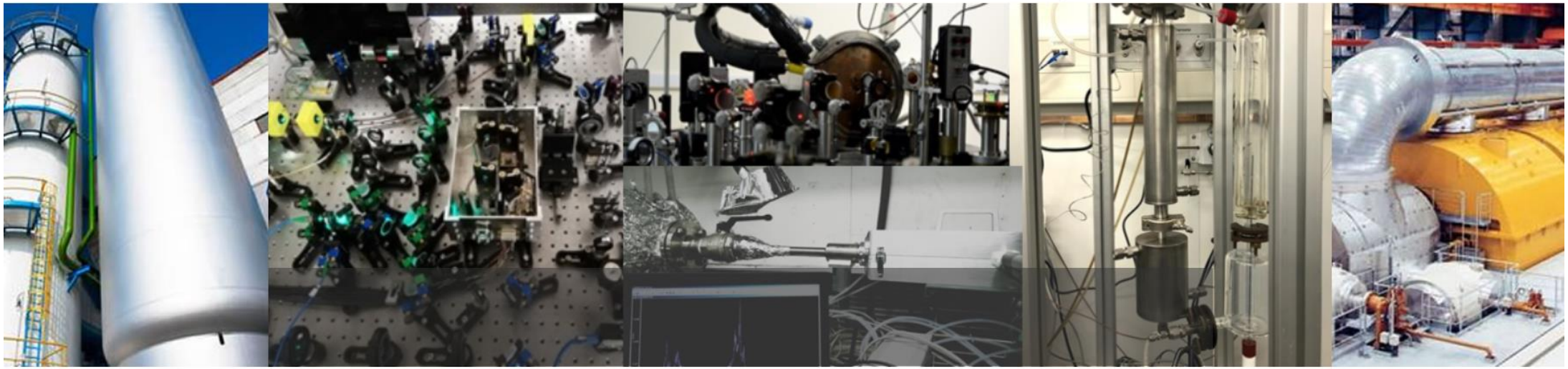

THE PROJECT

Metrology for trace water in ultra-pure process gases

Overview

Trace water is the single largest matrix contaminant in ultra-high purity (UHP) process gases. Even though the manufacturing of UHP gases serves many of the key technology areas, such as high-value semiconductor manufacturing, trace water measurements are still lacking measurement traceability in the relevant ranges and matrix gases.

READ MORE →

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Thank you for your attention!

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