PROMETH$_2$O

20IND06 PROMETH2O

Metrology for trace water in ultra-pure process gases

www.prometh2o.eu

Vito Fernicola, INRIM, Italy

Final Workshop

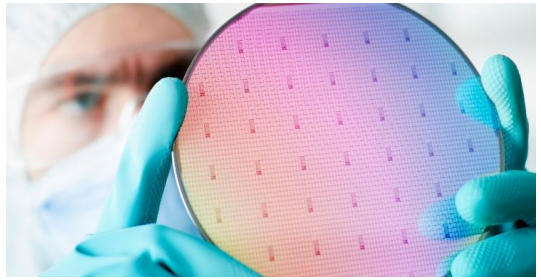
Gas Analysis 2024 Symposium / Porte de Versailles, Paris - France

Tuesday 30th of January 2024

EMPIR EURAMET

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

Project Challenges



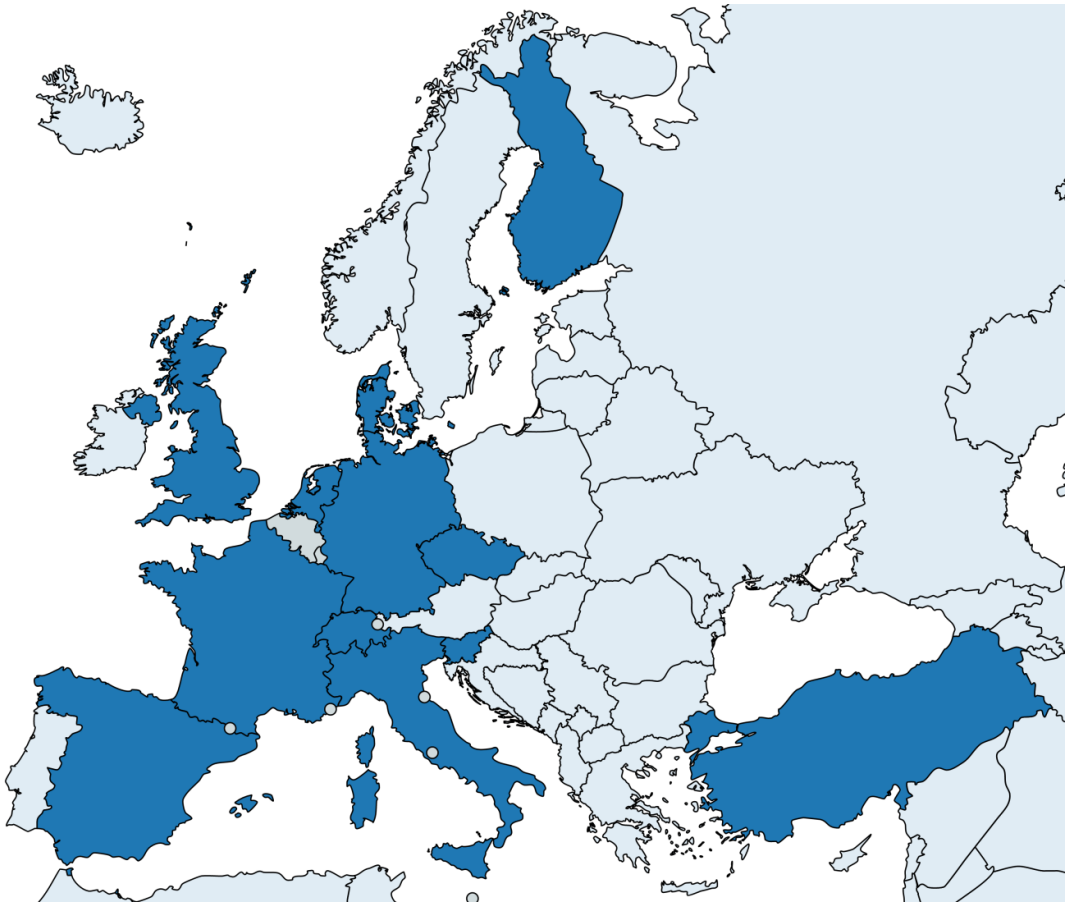
Water vapour is the single largest matrix contaminant in ultra-high purity (UHP) process gases used in key technology areas (e.g. semicon. and optoelectronics).

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 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.

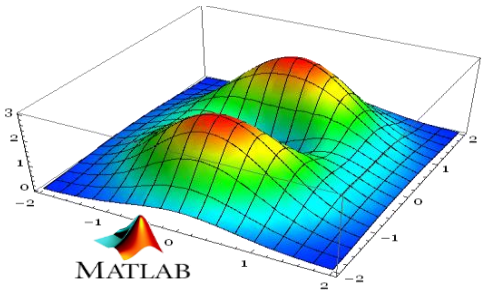
The Consortium

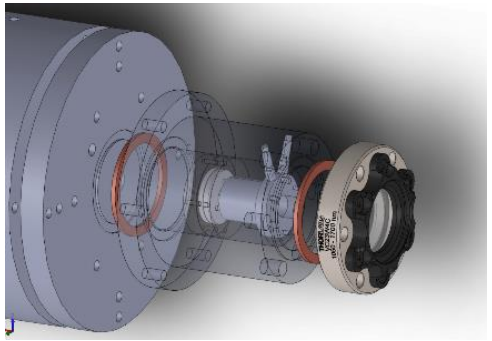


19 partners from 12 countries → 240 person-months

Project Objectives

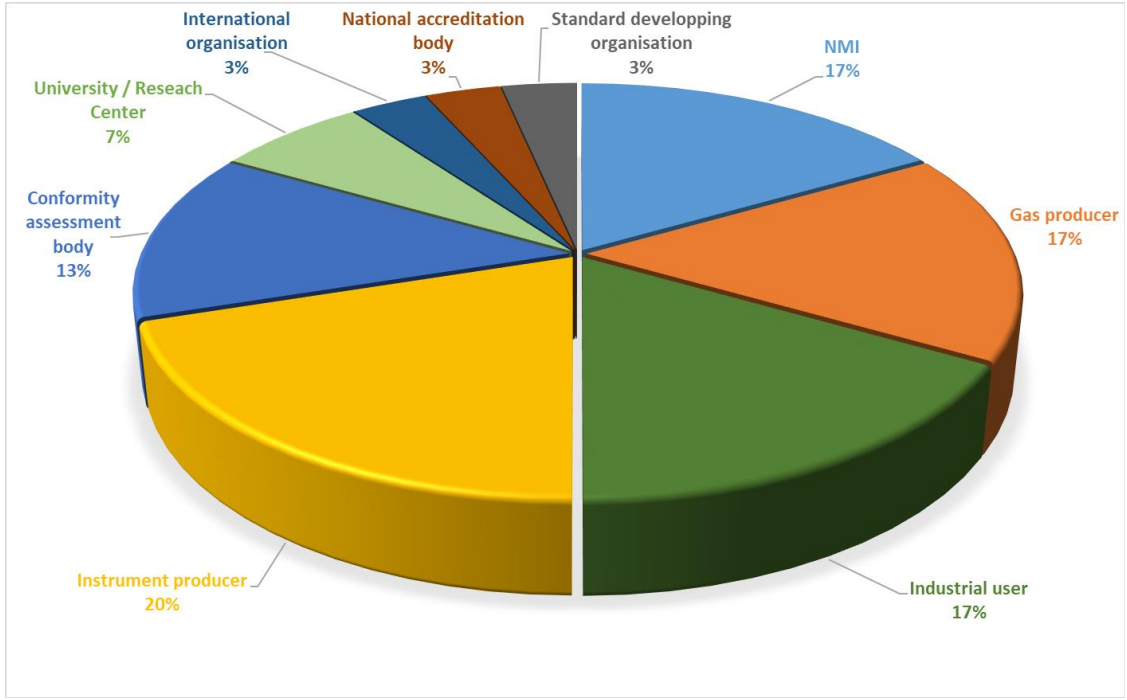
- **New primary standards** for trace water vapour in N_2, Ar and H_2 down to 5 nmol/mol (or -105 °C frost point temperature) at pressures up to 1 MPa.
- **New/improved measurement methods** in the amount fraction range between 5 µmol/mol and 5 nmol/mol (*rel. uncertainty* 3 % to 8 %).
- **New data and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





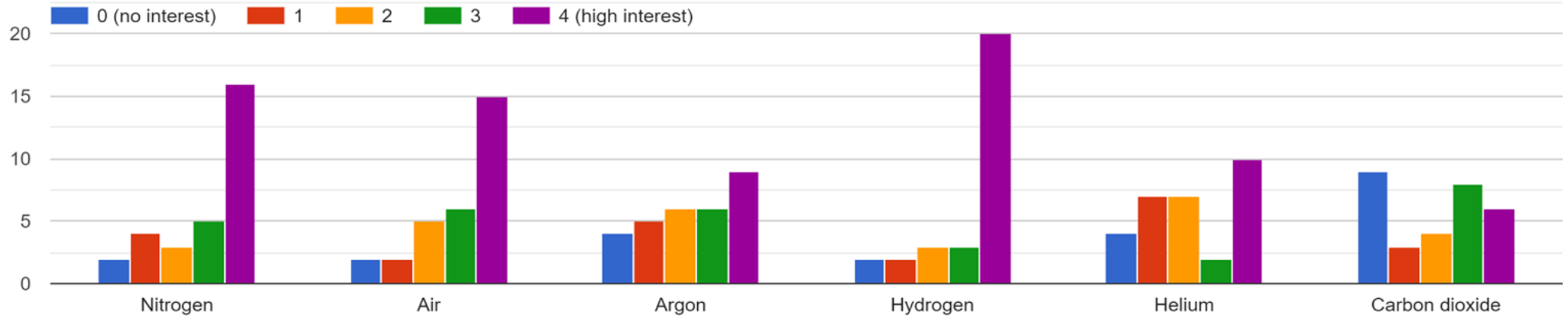
Stakeholder Engagement

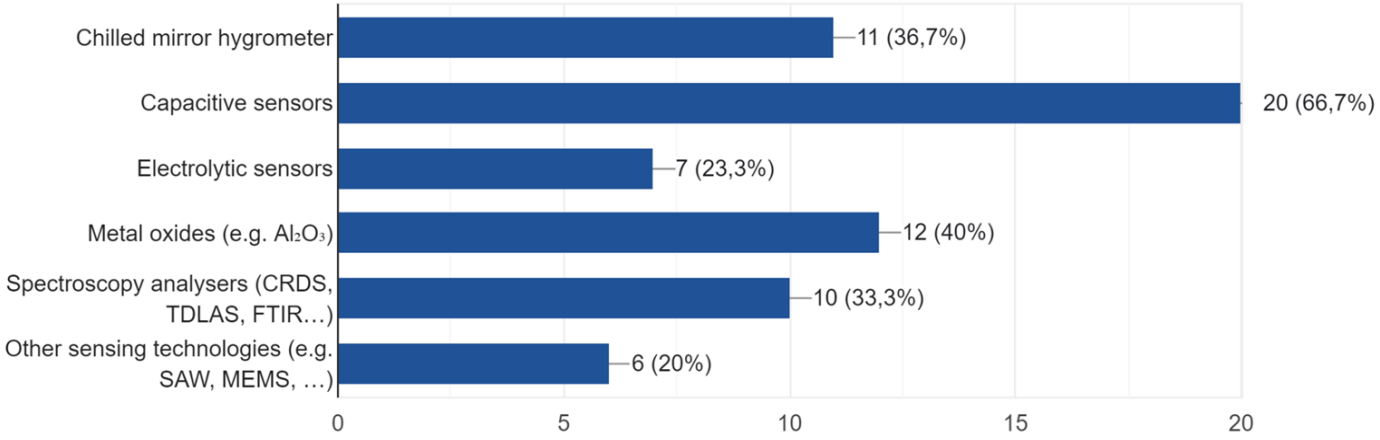
Surveying stakeholders needs and priorities



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

Surveying Stakeholder Needs and Priorities



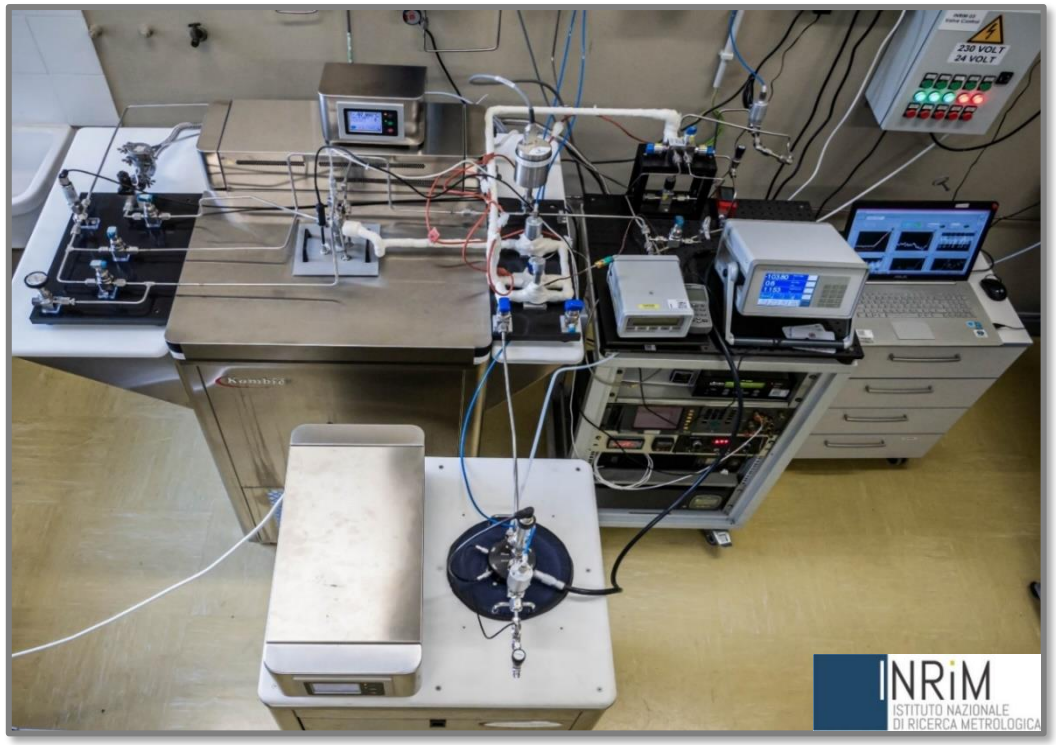


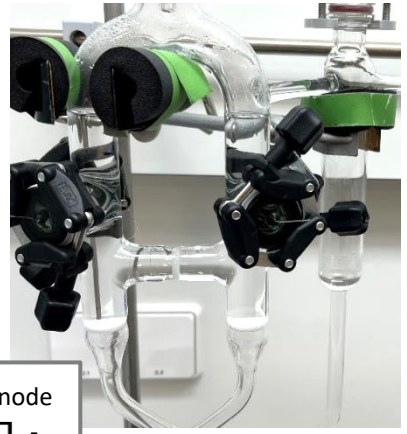
"I need to measure the trace water content in pure process gases as part of the quality assurance process."

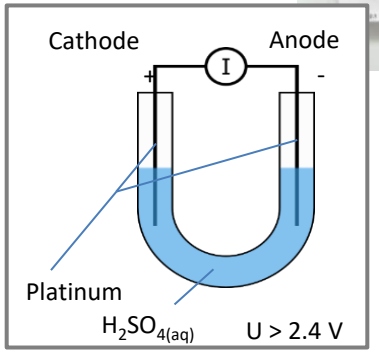
"We focus on hydrogen production by water electrolysis and fuel cell applications."

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

Achievements: Primary Standards for Trace Water







PTB
Physikalisch-Technische Bundesanstalt
Nationales Metrologieinstitut

LFP primary humidity generator - Mark 2

- ❑ Frost-point temperature: -105 °C to -20 °C
- ❑ Water vapour mole fraction: 5 ppb to 1000 ppm
- ❑ Pressure: 200 hPa to 0.68 MPa (N_2 and Ar)

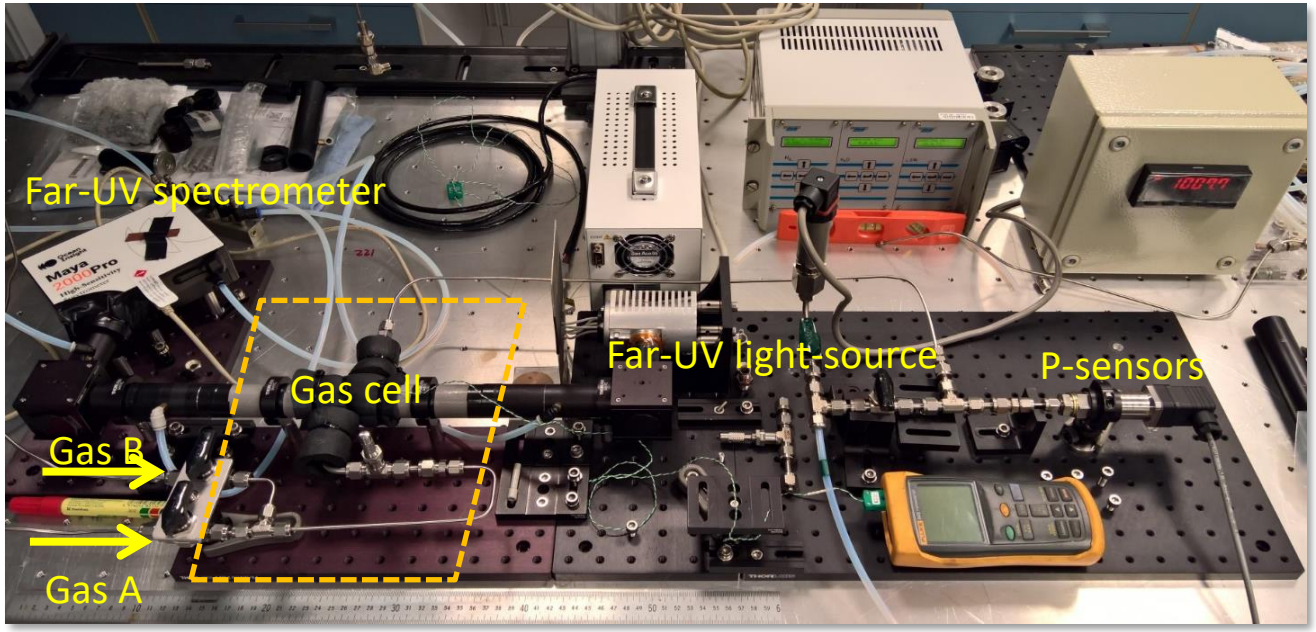
Coulometric Trace Water Generator (CTWG)

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

Permeation system based on a passivated MSB

- ❑ Range: 50 ppb to 5 ppm

Achievements: Improved Trace Water Analysers and Enhancement Factor Measurements

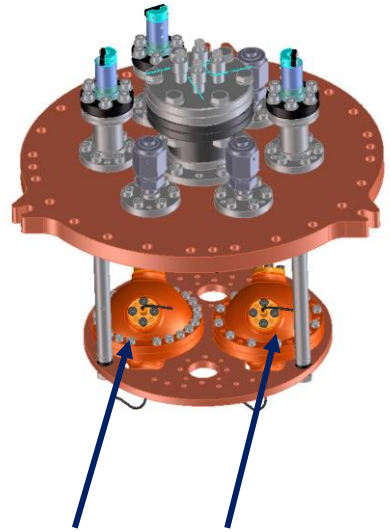


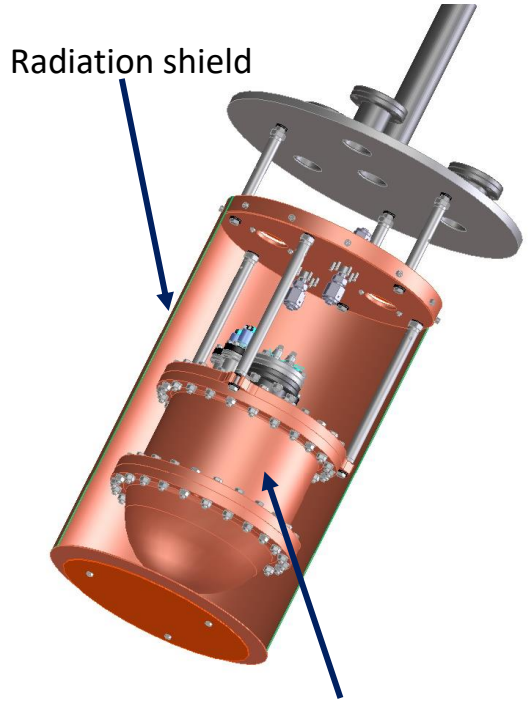
Far-UV spectroscopy system

- 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”

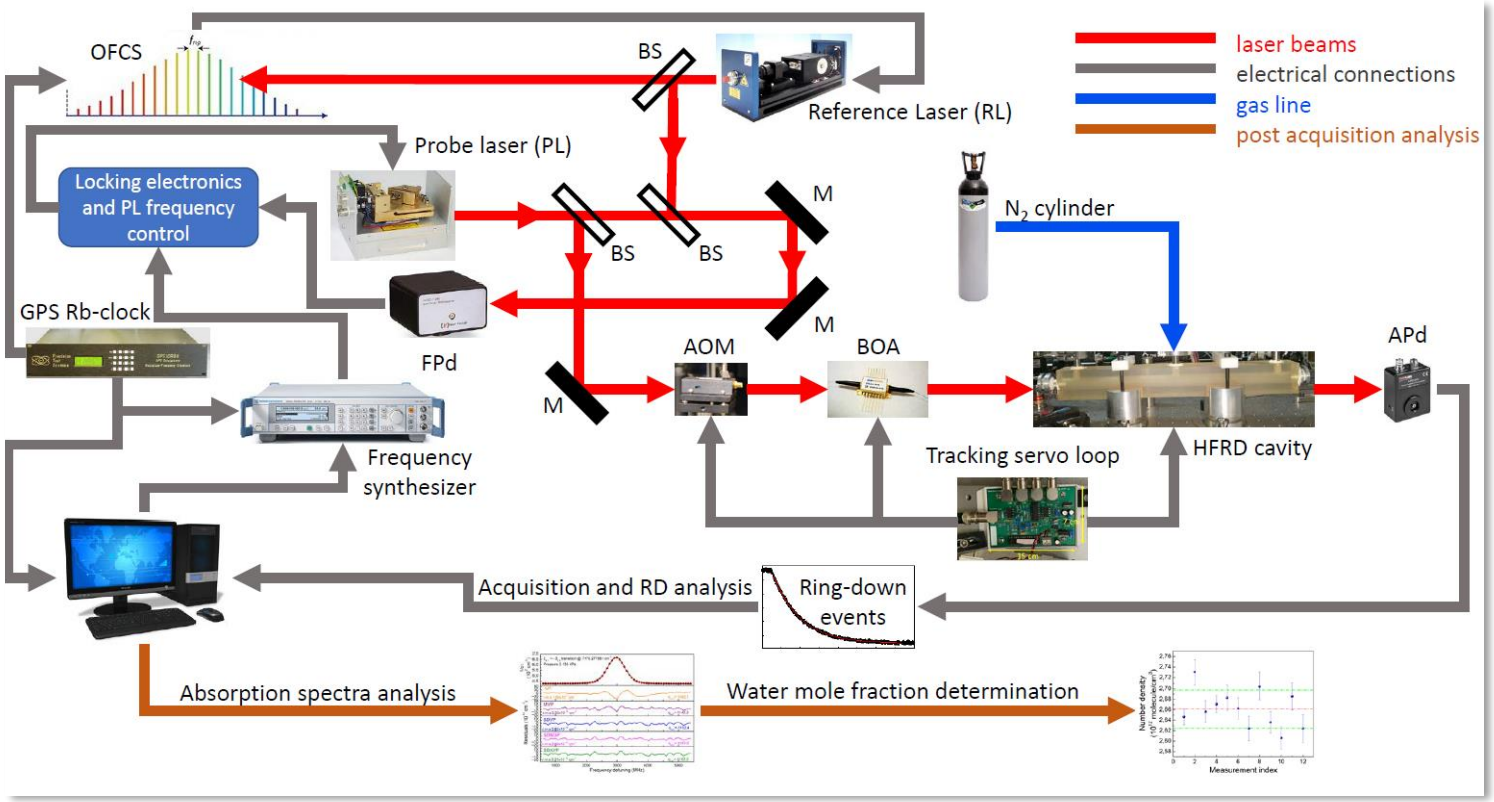
Microwave-based trace water hygrometers

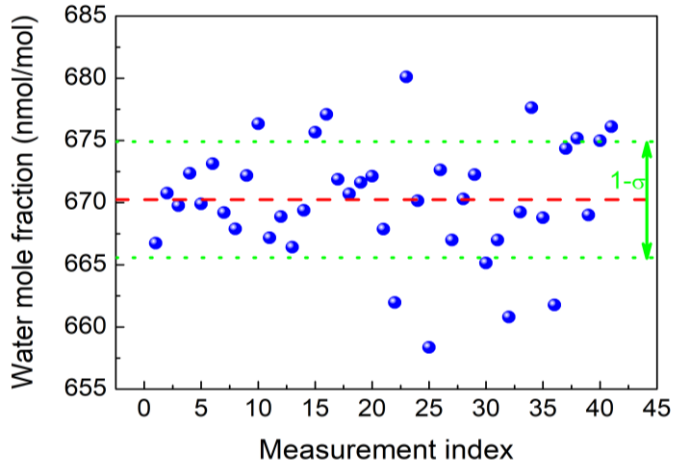
to measure the enhancement factor of H_2O vapour in N_2, Ar, and H_2





Achievements: Improved Comb-assisted CRDS Optical Analyser





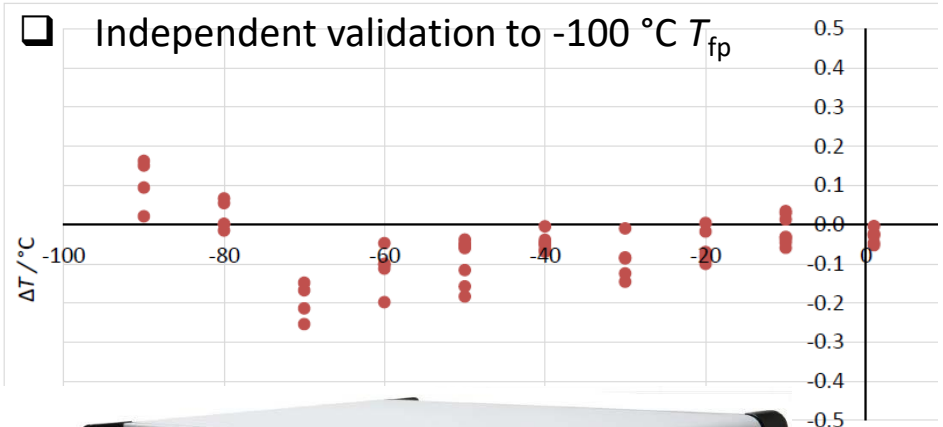
$$x_w = (671 \pm 4)\ nmol/mol$$

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)



VAISALA

Qrometric
Industrial and scientific metrology

MBW calibration

Traceable assessment of state-of-the-art commercial hygrometers
down to -105 °C (5 ppb)

NIPPON GASES

Test bed demonstrators
Industrial production facility of speciality gases that includes pure and UHP gases and humid gas mixtures.

Making an Impact …

- Availability of **primary standard** to provide traceability, also via calibration services, to instruments measuring trace water between 5 μmol/mol to 5 nmol/mol.
- Recommendation on **suitable transfer standards to support future international comparisons** in the trace water regime, either -65 °C to -105 °C or 5 μmol/mol to 5 nmol/mol.
- Contributions to new or improved **guides and recommended values** of thermo-physical properties of water-gas mixtures.
- **Documented and 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 at pressure up to 1 MPa.
- Establishment of an **European-wide measurement infrastructure**.
- **Early impact on UHP gas manufacturing**: on-site traceable calibration and measurement of water contamination demonstrated at industrially-relevant facilities.

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Thank you for your attention

and

enjoy the PROMETH2O workshop!

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