Newsletter n°4

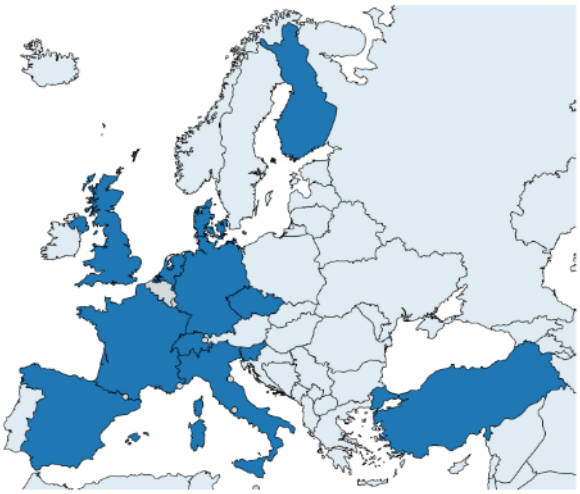
PROMETH$_2$O

Metrology for trace water in ultra-pure process gases

may 2024

Duration	Coordinator	Budget	Amount of work
36 months Start date: 01 June 2021 End date 31 May 2024	Vito Fernicola INRiM	1 798 649,10 €	242 person- months

Consortium partners

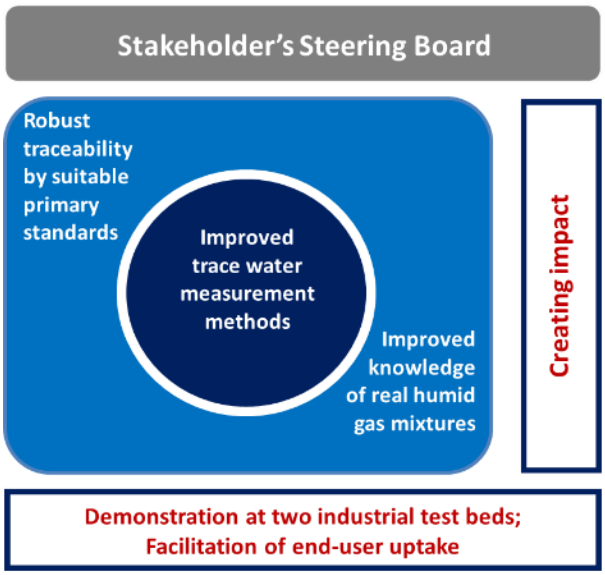


19 partners from 12 countries – 242 person-months

PROMETH2O is supported by world leading manufacturers, international organisations and metrology leaders

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

Organization of the consortium



Steering Board members

Chair: Stephanie Bell - NPL

International Organisations	Instrument Manufacturers	Gas Providers
CIPM CCT WG-Hu	Ball Wave	Air Liquide
IAPWS	Meeco	BOC
JCS	Li-Cor	SOL
KRISS	Baker Hughes	SIAD
NMIJ	PST/Rotronic	SAPIO
ISO/TC 158 WG3	EffecTech Ltd.	FHa
CIPM CCQM GAWG		
UNI CIG		
ACCREDIA		

Overview

Trace water is the single largest matrix contaminant in ultra-high purity (UHP) process gases (e.g. argon Ar, nitrogen N_2 and hydrogen H_2), 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 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.

You can access to publishable summary **here**.

Need

Due to its ubiquity and chemical properties, water vapour is a critical contaminant and one of the most difficult impurities to eliminate. Water contamination effects become relevant when taking into consideration the worldwide production of gases. The global market for industrial gas is expected to reach US$ 149 billion by 2027, with Europe sharing about 16%, owing to rising demand from the electronics, healthcare and pharmaceutical sectors. The semiconductor market alone is expected to reach $ 5.2 billion by 2026. Bulk process gases with ultra-high purity grade (N6.0 or better) need to be manufactured with total impurities below 1 ppm in volume. According to the International Technology Roadmap for Devices and Systems, water vapour measurement techniques need to measure amounts as low as a few parts per billion at the point of use. From 2015 to 2020 these requirements have tightened for some gases (N_2 and Ar) by more than a factor of five. This presents great challenges to both gas producers and analytical instrument makers which aim to improve trace water measurement methods at the part per billion. This would require a metrological infrastructure and measurement technology to provide robust traceability to trace water measurements with a provision of suitable primary standards, improved optically-based methods and improved knowledge of the thermophysical properties of moist gases.

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Objectives

The overall objective of PROMETH2O is to provide new and improved trace water measurements relevant to the production of pure gases and to demonstrate their impact in improving selected industrial processes and applications.
The specific objectives of this project are:
1. To improve trace water measurement methods in the amount fraction range between 5 parts in 10^6 (5 ppm) and 5 parts in 10^9 (5 ppb) or, equivalently, between -65 °C and -105 °C frost point temperature at 0.1 MPa with a relative standard uncertainty between 3 % and 8 %, from the upper to lower range, respectively.
2. To provide robust traceability to trace water measurements by developing suitable primary standards for the amount fraction range from 5 ppm to 5 ppb (or -65 °C to -105 °C frost point temperature at 0.1 MPa) with a relative standard uncertainty less than 3 % to 8 %, in selected gas matrices of air, N_2, Ar and H_2 at pressures up to 1 MPa.
3. To improve the present knowledge of thermophysical data of real humid gas mixtures, in particular the water vapour enhancement in N_2 and Ar in the temperature range from -30 °C to -90 °C and at pressures from 0.1 MPa to above 1 MPa.
4. To demonstrate improved trace water measurement methods between 5 ppm and 5 ppb or, equivalently, between -65 °C and -105 °C frost point temperature at 0.1 MPa, in two industrially relevant facilities (test beds).
5. To facilitate the take up of the technology and measurement infrastructure developed in the project by the measurement supply chain, standards developing organisations (CIPM, IAPWS, JCS) and end users (instrument manufacturers, gas providers).

Contact us

Project Leader:

Vito FERNICOLA – INRiM

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Project's website:

https://www.prometh2o.eu/

WP leaders

WP1 leader: Alexander FATEEV – DTU (alfa@kt.dtu.dk)

WP2 leader: Rugiada CUCCARO – INRiM (r.cuccaro@inrim.it)

WP3 leader: Stefan PERSIJN – VSL (SPersijn@vsl.nl

WP4 leader: Eric GEORGIN – LNE-CETIAT (eric.georgin@cetiat.fr)

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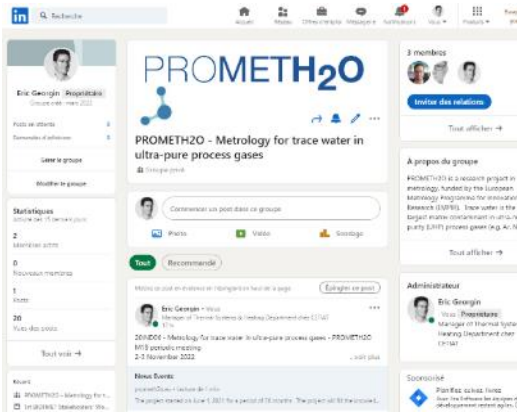
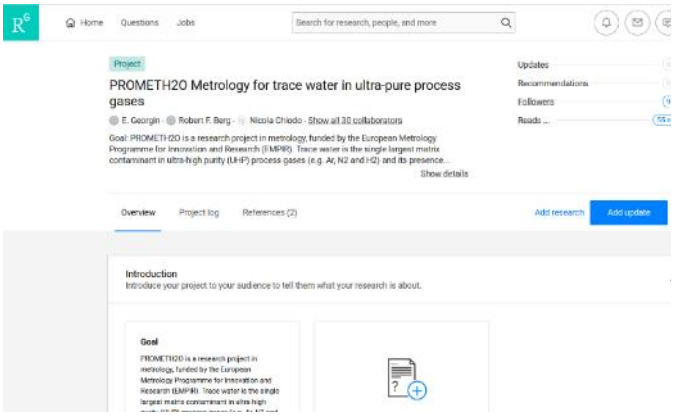
Interested by following project and related special events? Follow us on LinkedIn and Research Gate !





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Training course at NIPPON Gases

In the last months of the project a key event focused on training course has been organized and hosted by NIPPON Gases with the support of JRP partners. The organization of this event was such as the morning was dedicated to theoretical presentation and the afternoon focused on more practical topic with a tour of NIPPON Gases site in Chivasso, as well as a practical demonstration of the Frost Point Generator developed within the project by Qrometric.

Training course

Metrology for trace water in ultra-pure process gases

7th of March 2024

VENUE: in person meeting

Morning - *Golf Club Settimo, strada Cebrosa 166 Settimo Torinese (TO)*

Afternoon - *Nippon Gases, via Marie Curie 134/D Chivasso (TO)*

08:45 - 09:00	**Registration, meeting opening and introduction of attendants**
09:00 - 09:30	Quality assurance in the production of pure process gases at Nippon Gases Stefano Boggio - Nippon Gases
09:30 - 09:50	PROMETH2O: a project overview Vito Fernicola - INRiM
09:50 - 10:10	Tips and tricks for traceable humidity measurements Eric Georgin - CETIAT
10:10 - 10:40	**Coffee Break**
10:40 - 11:00	From ideal to real gas mixtures: what we need to know? Shahin Tabandeh - VTT
11:00 - 11:20	Trace water measurements: practical considerations Domen Hudoklin - UL
11:20 - 11:40	Trace water measurements: technology and solutions Thomas Klein - Process Insight
11:40 - 12:00	In-situ dew point measurements: no need to worry about sampling uncertainties? Hannu Sairanen - Vaisala
12:00 - 12:30	Round table about the future of humidity measurements
12:30 - 14:30	**Lunch**
14:30 - 15:30	Group A - Lab tour (Stefano Boggio - Nippon Gases) Group B - FPG & Humidity Sensor demonstration - (Ned Hawes - Qrometric)
15:45 - 16:15	**Coffee Break**
16:15 - 17:45	Group A - FPG & Humidity Sensor demonstration - (Ned Hawes - Qrometric) Group B - Lab tour (Stefano Boggio - Nippon Gases)
17:45 - 18:00	**Meeting closure**

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Kaleidoscopy of the training session

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After an introduction to the day and a presentation on the expression of needs from the gas manufacturer's point of view by Stefano Boggio (NIPPON Gases), Vito Fernicola (INRiM) provided an overview of the project as well as key results and ongoing developments. Eric Georgin (CETIAT) then informed the participants about the difficulties in measuring low humidity. Shahin Tabandeh presented the status of the work performed on the enhancement factor. The morning continued with a focus on trace moisture measurement, including practical considerations presented by Domen Hudoklin (UL) and various measurement technologies available on the market, discussed by Thomas Klein (Process Insights AG). Finally, Hannu Sairanen (Vaisala) shared some considerations about sampling and trace moisture measurements with the audience.

About 30 people have attended this event, predominantly coming from industry and accredited laboratory.

All presentations are available on the project's website (https://www.prometh2o.eu/) or directly by following the link here.

The end of the day was dedicated to a visit to the NIPPON Gases site in Chivasso, Italy. The plant is a Packaged Gas (PAG) facility. Its activities involve the transformation (purification, transfilling, and mixing) of pure products sourced from pipelines, other plants, and suppliers. From a manufacturer's point of view, the analysis of moisture is crucial in our activity because it has significant repercussions on both quality and safety aspects for the gas bottles. The demonstration of the Frost Point Generator, developed within the project, was conducted by Ned Hawes (Qrometric). The generation principle was explained in detail, and the capabilities of the Frost Point Generator were presented live to the attendees.

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PROMETH2O final project (web) meeting

After three years, the project is coming to an end, and the final meeting was held online. On this occasion, the meeting was opened to Steering Board members during the presentation of the technical work packages and the impact work package, while the management discussions were restricted to JRP partners only.

Agenda of the final project meeting (M36)

20IND06 PROMETH2O

Metrology for trace water in ultra-pure process gases

Thursday 30th of May 2024, from 08:50 to 16:30 (UTC + 2)

VENUE: online meeting

30th of May 2024		Lead by
OPEN TO STAKEHOLDERS		
08:50-09:00	**Opening and Introduction** Welcome remarks and Tour of the table	VF
09:00-10:00	**WP1: Improved trace water measurement methods and techniques** - Summary of WP1 and presentation of deliverables D1 and D2. *Partners:* **DTU**, INRIM, MBW, PTB, Qrometric, SUN, TUBITAK	AF
09:00-09:40	D1 – Report on the recommendation on measurement methods and techniques for trace water measurements in industrial environments in the amount fraction range between 5 parts in 10^6 (5 ppm) and 5 parts in 10^9 (5 ppb) (-65 °C and -105 °C frost point) with relative standard uncertainty between 3 % and 8 %. *Partners:* **DTU**, SUN, MBW, INRIM, TUBITAK, Qrometric	AF
09:40-10:00	D2 – Report on the recommendation of transfer standards for a future CIPM comparison in the frost-point temperature range -65 °C to -105 °C (5 ppm to 5 ppb). *Partners:* **INRIM**, PTB, TUBITAK, DTU	VF
10:00-12:00	**WP2: Provision of robust traceability to trace water measurements in real humid gas mixtures** - Summary of WP2 and presentation of deliverables D3, D4 and D5. *Partners:* **INRIM**, CEM, CETIAT, CMI, CNAM, INTA MBW, PTB, UL, UNICAS, UVa, VSL, VTT	RC
10:15-10:45	D3 – Report on the development of primary trace water vapor standards describing the range, the estimated uncertainty and the gas species in which reference values can be generated with a target fraction range from 5 ppm to 5 ppb (-65 °C to -105 °C) with relative standard uncertainty less than 3 % to 8 % in selected gas matrices at pressures up to 1 MPa. *Partners:* **INRIM**, VTT, CMI, INTA, UL, PTB, MBW, VSL, CETIAT	RC

20IND06 PROMETH2O M36 Meeting Agenda_v3 - 30 May 2024.docx

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10:45-11:00	Coffee Break	
11:00-11:20	D4 – User guide related with the software tool(s) of a web-based application to estimate the enhancement factor and uncertainty in water vapour in N_2, Ar and H_2 in the temperature range between -30 °C and -90 °C and pressure range from 0.1 MPa to above 1 MPa. *Partners*: UNICAS, VTT	FA
11:20-12:00	D5 – Report on the development and validation of correlation equations for the enhancement of water vapour in N_2, Ar and H_2 in the temperature range from -30 °C to -90 °C and at pressures from 0.1 MPa to above 1 MPa. *Partners*: VTT, CNAM, CETIAT, CMI, VSL, UL, INTA, CEM, UVa, UNICAS	ST
12:00-13:15	Lunch Break	
13:15-14:15	**WP3: Demonstration at industrial test beds and facilitation of end-user uptake** - Summary of WP3 and presentation of deliverables D6 and D7. *Partners:* VSL, DTU, INRIM, MBW, Nippon Gases, Qrometric, UL, Vaisala	SP
13:15-13:45	D6 - Report on a) the delivery of a tested toolkit of metrological solutions at an equipment developer to provide measurement traceability in the field with a target of improved trace water measurement methods between 5 ppm and 5 ppb and b) discussion with stakeholder needs on the compliance and lessons learned *Partners*: Qrometric, UL, MBW, INRIM, Vaisala, VSL	NH
13:45-14:15	D7 - Report on assessing the production quality of bulk and specialty gases at a specialty gas company facility to demonstrate improved trace water measurement methods between 5 ppm and 5 ppb in an industrially relevant facility. *Partners*: Nippon Gases, INRIM, VSL, DTU, Vaisala	SB
14:15-14:45	**WP4: Creating impact** - Summary of WP4 and presentation of deliverable D8. *Partners:* CETIAT, all partners	EG
14:15-14:45	D8 - Evidence of contributions to or influence on new or improved international guides, recommendations, and standards with a focus on selected committees. *Partners*: CETIAT, all partners.	EG
14:45-15:30	**Feedback from the Stakeholder Board (SB)** - Open discussion, questions and suggestions from the SB.	SB
15:30-15:45	Coffee Break	
RESTRICTED TO PARTNERS		

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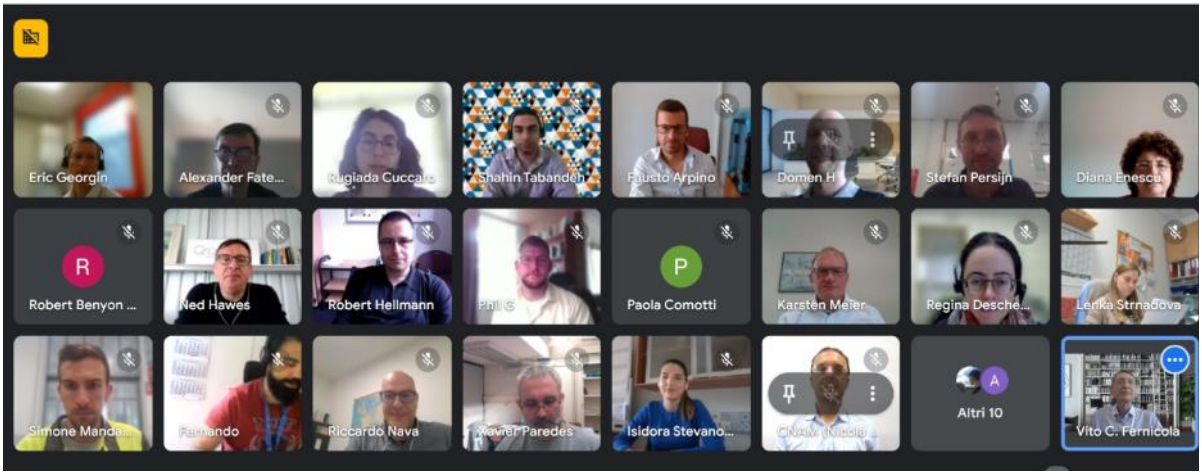
15:45-16:00	**WP5: Management and coordination** *All partners*	VF, DE
15:45-16:00	Presentation and discussions of the periodic (P2) reporting for the M36 (31 May 2024 + 60 days): - Technical Report (or Progress report) - Output and Impact Report - Data Management Plan (DMP) - Publishable Summary - Horizon 2020 Questionnaire - P2 Project Financial Report - Final Publishable Report - the consortium shall provide this document, covering the whole project - Complete set of Financial Statements - Certificates on the Financial Statements	VF, DE; PC
16:00-16.30	**Conclusions** - Project lifecycle, key achievements, and lessons learned. - Acknowledgements and appreciation to all contributors and partners. - Future collaborations and next steps.	ALL
16:30	**Meeting closure**	

Each work package leader summarized the key achievements and the status of each task and activity. Special focus was given to the progress of each deliverable, which are mandatory for the conclusion of the project.



In the previous e-newsletter, special focus was given to WP1 “Improved Trace Water Measurement Methods and Techniques” and WP2 “Provision of Robust Traceability to Trace Water Measurements in Real Humid Gas Mixtures.” The editorial choice for this issue is to focus on the last technical work package, WP3: “Demonstration at Industrial Test Beds and Facilitation of End-User Uptake.”.

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

The goal of this work package was to demonstrate, in a real industrial setting, the enhanced methods for measuring trace amounts of water developed in WP1, and the methods for generating trace water from WP2, based on the needs of our stakeholders. This helped end-users adopt the technology more effectively.

First, an inventory has been done of the technical features required for the test beds based on stakeholder needs, considering both technical and logistical requirements for the demonstration. The demonstration was then conducted at two selected industrial test sites:

Test Bed 1: This provided a toolkit of metrological solutions to ensure measurement traceability in the field. Qrometric’s site in the UK was used, which offered a range of generated conditions (initially nitrogen and carbon dioxide, with plans to include argon). Humidity levels ranged from ambient down to a frost point of -

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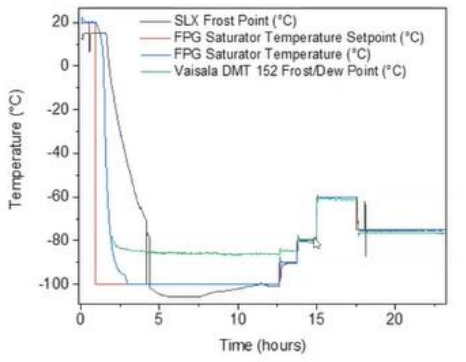
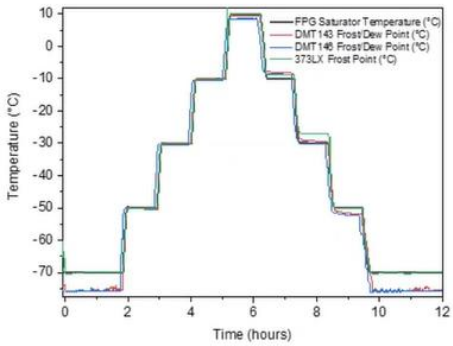
75°C, with a target of -90°C. The site had in-house measurement capabilities using a chilled-mirror hygrometer down to -100°C.





Test Bed 2: This evaluated the production quality of bulk and specialty gases at a major specialty gas company's facility. Nippon Gases Italia's production site in Chivasso, Italy, was used. This facility produced pure and ultra-high purity (UHP) gases and moisture mixtures. Nippon Gases offered a variety of pure gases under different conditions and mixtures with varying humidity levels. Access to their calibration laboratory allowed for onsite testing of sensors and instrumentation.

The project showcased the innovative features of the first portable dew-frost point calibrator, addressing an existing need highlighted by stakeholders. Collaboration between companies and National Metrology Institutes (NMIs), as well as inter-company partnerships, resulted in a win-win situation.

This work package successfully demonstrated the capabilities of Qrometric and Vaisala sensors, facilitated knowledge transfer—particularly regarding uncertainty—and provided direction for NMIs on future measurement standards. A unique aspect of this work package was the opportunity to access the facilities of a major specialty gas company, NIPPON Gases, offering invaluable insights.

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Key lessons learned included understanding the effects of dead volumes on response time and the equilibration time required to achieve accurate measurements. These insights will be crucial for future advancements in the field.

Thank you all for your participation and commitment during this project !!!

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