Newsletter n°2

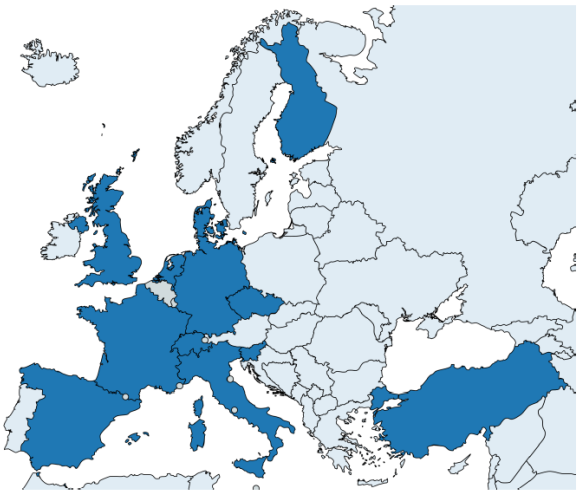
PROMETH$_2$O

Metrology for trace water in ultra-pure process gases

october 2023

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

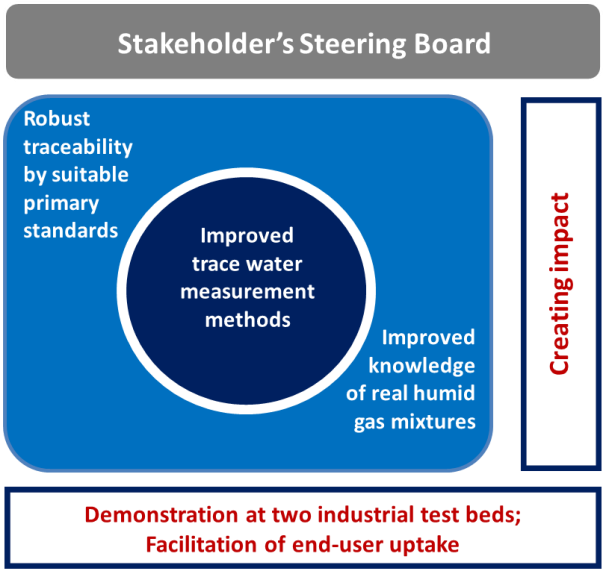


19 partners from 12 countries – 242 person-months

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. 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 still lack 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 the 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 considering the worldwide gas production. 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 (nitrogen and argon) by more than a factor of five. This presents great challenges to both gas producers and analytical instrument makers aiming 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 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 106 (5 ppm) and 5 parts in 109 (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, N2, Ar and H2 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 N2 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)

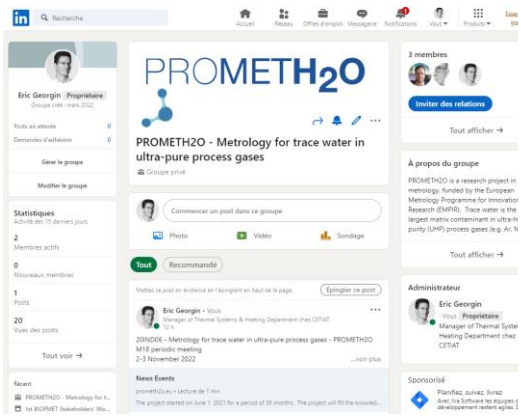
WP2 leader: Rugiada CUCCARO – INRiM (r.cuccaro@inrim.it)

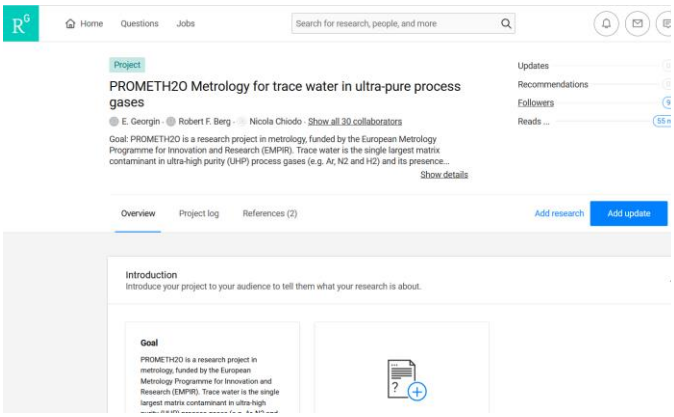
WP3 leader: Stefan PERSIJN – VSL (SPersijn@vsl.nl

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

Follow us

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





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Regarding the project's lifespan

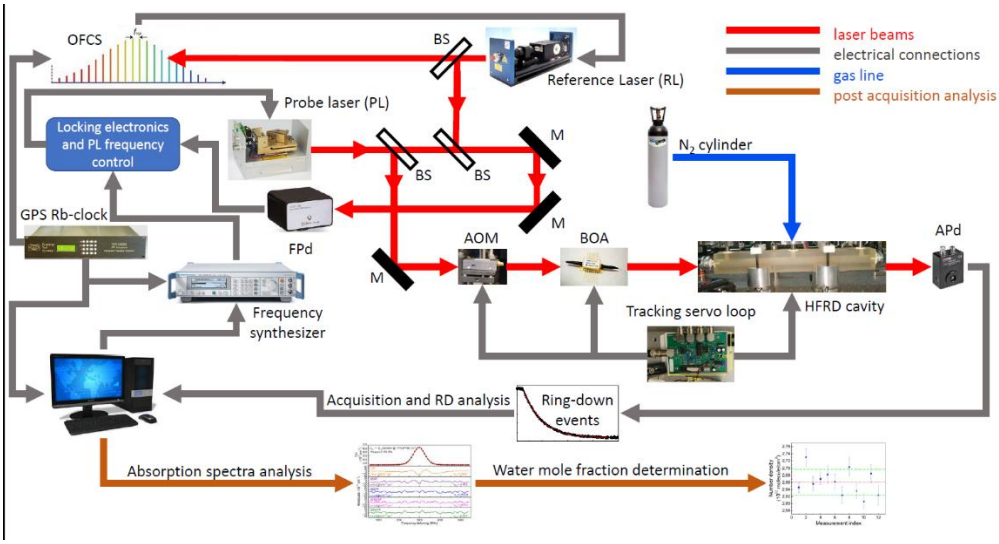
The beginning of 2023 was important for the project due to the mid-term review of the project. We are glad to announce the success of this review and the approval of the M18 interim report.

The publishable summary has been released and is available here: https://www.prometh2o.eu/en/documents

Key achievements at a glance

Among the various tasks undertaken by the consortium, one of them is to enhance trace water measurement methods and techniques within the water fraction range of 5 ppm to 5 ppb, which is equivalent to frost point temperatures between -65 °C and -105 °C at 0,1 MPa. The target relative uncertainty for these measurements is set between 3% and 8% for water concentrations of 5 ppm and 5 ppb, respectively. The project will provide sensors capable of managing potential interferences while maintaining their high selectivity for the target species. In this 2nd newsletter, we take the opportunity to present a brief overview of these developments carried out within WP1, with a focus on the instruments developed.

WP1: Improved trace water measurement methods and techniques: Development and improvement of optical analysers



SUN is developing a compact NIR CC-FS-CRDS spectrometer to enhance sensitivity, thereby reducing the limit of detection. This spectrometer is referenced to an optical frequency comb for traceable measurements of water vapor in H_2 and N_2, ranging from 5 ppm down to 50 ppb, with a standard relative uncertainty between 3% and 8% and an operational pressure as low as 10 kPa.

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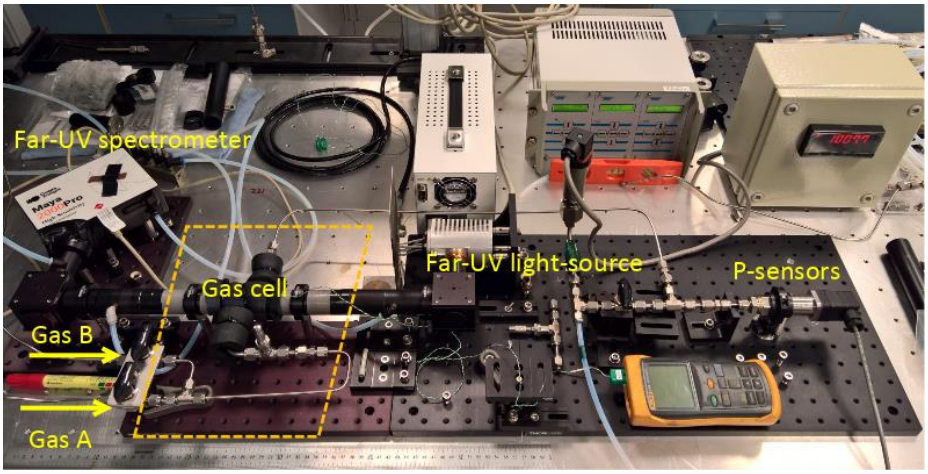

DTU is developing a compact and transportable far-UV system for trace water vapor measurements in Ar, N_2, and H_2, ranging from 5 ppm to 5 ppb, with a standard relative uncertainty between 3% and 8%, and an operating pressure of up to 1 MPa.

TUBITAK is improving the existing FTIR-based trace water measurements in N2 and Ar from 5 ppm to 50 ppb with a standard relative uncertainty between 3% and 8% and an operating pressure of up to 1 MPa. The existing high-resolution FTIR system is being upgraded with a new pump system and a new multi-pass gas cell to enable water vapor measurements down to 50 ppb and operating pressure in the cell of up to 1 MPa.

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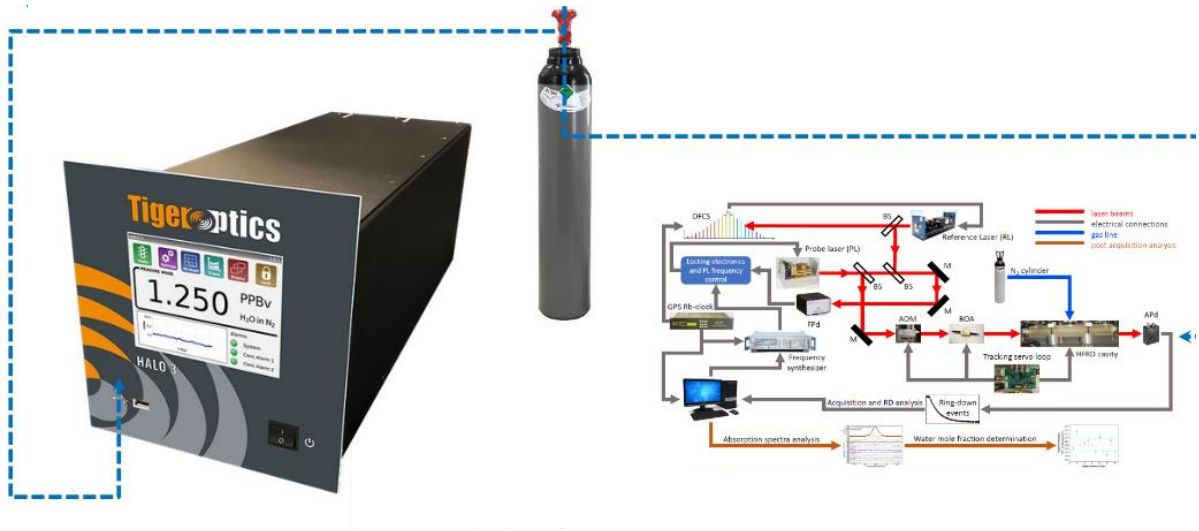
WP1: Improved trace water measurement methods and techniques: Validation of the measurement methods and techniques



SUN validates and performs an inter-comparison of the CC-FS-CRDS spectrometer using a reference humidity generator. SUN, with the support of INRIM, assesses the performance, potential effects of gas matrices on the measurements, and measurement uncertainties of the CC-FS-CRDS spectrometer in the amount fraction range between 5 parts in 10^6 (5 ppm) and 5 parts in 10^9 (5 ppb) with relative standard uncertainties between 3% and 8%, from the upper to the lower range, respectively.

This work has been presented at two conferences, namely:

- C-PASS 2023 Conference on Photonics for Advanced Spectroscopy and Sensing, 3-8 September 2023, Castellaneta M. (ITALY).
  Presentation (invitedspeaker): «Comb-assisted frequency-stabilized cavity ring-down spectroscopy: application to ultra-sensitive detection of water vapor».
- •EGAS54 Conference of the European Group on Atomic Systems, 19-22 June 2023, Strasbourg (France)
  Poster: «Lamb-dip cavity ring-down spectroscopy of acetylene at 1.4 μm».

In addition, two articles have been published and are available in open access:

- A. Castrillo et al. "On the $^{12}C_2H_2$ near-infrared spectrum: absolute transition frequencies and an improved spectroscopic network at the kHz accuracy level", Phys. Chem. Chem. Phys., 25, 23614-23625 (2023),
  https://doi.org/10.1039/D3CP01835K
- E. Fasci et al. "Water vapor concentration measurements in high purity gases by means of comb assisted cavity ring down spectroscopy", Sensors and Actuators A: Physical, 362, 114632, (2023), https://doi.org/10.1016/j.sna.2023.114632

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DTU and Qrometric validate and perform an inter-comparison of the far-UV system developed against a traceable transfer standard water analyzer (e.g., CE-FM or CMH). DTU and Qrometric assess the performance, possible gas matrix effects on the measurements, and measurement uncertainties in the amount fraction range between 5 parts in 10^6 (5 ppm) and 5 parts in 10^9 (5 ppb) with a relative standard uncertainty between 3% and 8%, from the upper to the lower range, respectively.

Additional information: WP1 leader, Alexander FATEEV – DTU, alfa@kt.dtu.dk

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Scientific dissemination

From the latest e-newsletter, the consortium has undertaken numerous achievements and scientific dissemination activities. While it is not possible to provide detailed reports of all these actions, a brief selection is presented here.

Key figures

As of this date, the project has 4 articles published in open-access peer-reviewed journals and has presented 20 oral presentations or posters at 16 international or national events.

ITS10 – April 3/7, 2023

The ITS10 conference brought together hundreds of researchers from around the world across all disciplines where temperature-related research or technology development was a foremost concern.

The Technical Program covered five plenary sessions, seven parallel oral sessions arranged in four tracks, and three poster sessions. The parallel sessions were: “Temperature Metrology, Scales, and Fixed Points”; “Radiation Thermometry and Remote Sensing of Temperature”; “Industrial Sensors and Applications”; and “Special Scientific Applications and New Technologies, including climate research, biological temperature measurements, and novel sensor technologies”.

ITS10 Program Plan

Start Times	Monday April 3	Tuesday April 4	Wednesday April 5	Thursday April 6	Friday April 7
8:30	Registration / CCT Committee Meetings -AM	Opening Session A : James F Schooley Plenary Lecture	Plenary Session E : Trends in Industrial Temperature Measurement	Plenary Session I : Temperature, Climate, and Human Health	Parallel Sessions M1-M5
10:00		Coffee Break	Coffee Break	Coffee Break	Coffee Break
10:45		Parallel Sessions B1-B5	Parallel Sessions F1-F5	Parallel Sessions J1-J5	Poster Sessions N1-N4
12:15		ITS10 Luncheon	ITS10-MSC Luncheon	ITS10-MSC Luncheon	ITS10-MSC Luncheon
13:45	Registration / CCT Committee Meetings -PM	Plenary Session C : Frontiers in Temperature Measurement	Parallel Sessions G1-G5	Parallel Sessions K1-K5	Session P Closing Plenary
15:15		Coffee Break	Coffee Break	Coffee Break	
16:00		Parallel Sessions D1-D5	Poster Sessions H1-H4	Poster Sessions L1-L4	Unscheduled
17:30					
18:00	ITS10 Welcome Reception	Exhibitors Reception	ITS10 Banquet	President's Reception	Unscheduled

On this occasion, 1 oral presentation related to work performed within the project was presented:

- Uncertainty in trace moisture measurements by adsorption/desorption phenomena Mr Sairanen – VAISALA / Finland

The total participation in this congress was approximately 180 attendees from 30 countries. The audience was predominantly from the scientific community: universities, NMI and public research organisations.

Further information are available here: https://www.prometh2o.eu/en/documents and here: https://its10.msc-conf.com/

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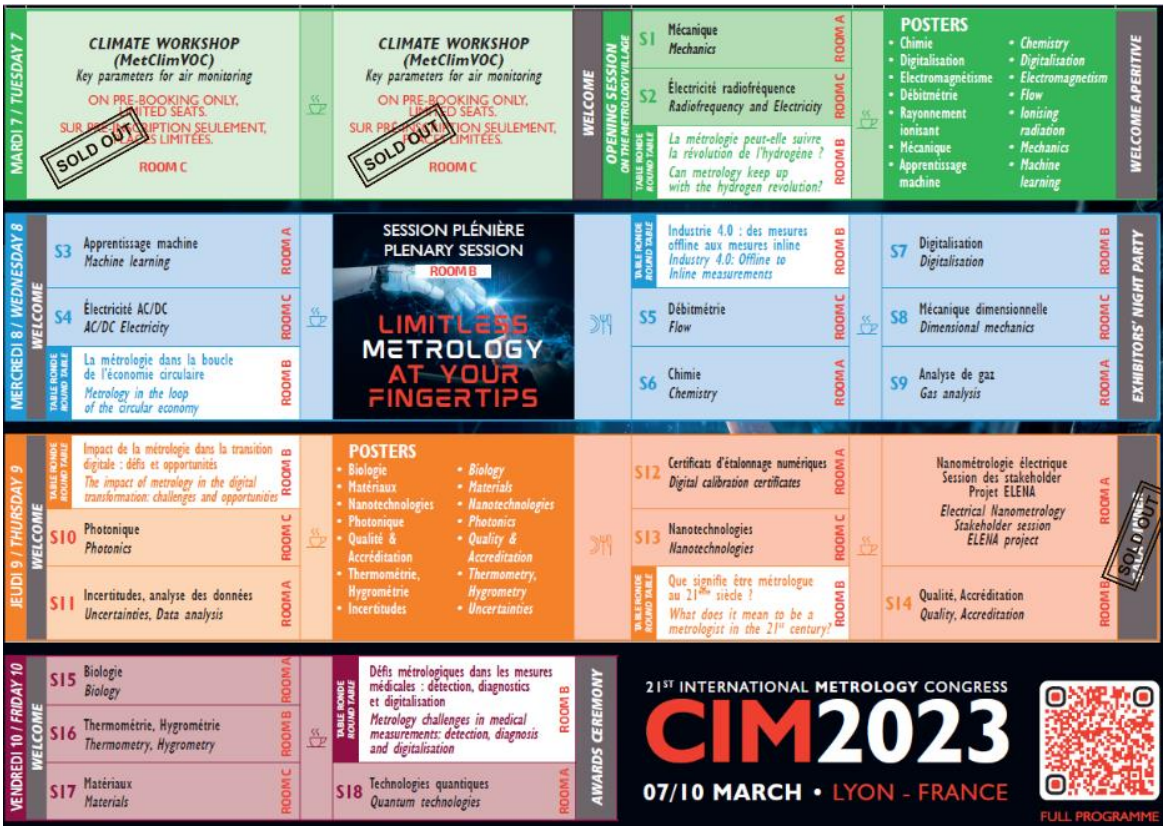
21st International Metrology Congress – March 07/10, 2023

The CIM is the premier event where metrology intersects with science, industry, and quality infrastructure organizations. It serves to foster the development of solutions to address global challenges, particularly by ensuring the reliability of measurements. It has been organized by the Collège Français de Métrology – CFM, in partnership with Global Industrie from March 7 to 10 in Lyon – FRANCE.

The congress revolves around three key applications: Industry 4.0, Health, and Climate Challenges. It comprises 200 conferences that are categorized into technical sessions, poster presentations, and round tables.



At this occasion, 2 oral presentations and 2 posters related to work performed within the project were presented:

- Presentation
  - A new measurement infrastructure for trace water in ultra-pure process gases
    Mr FERNICOLA – INRiM / Italy
  - Upgrade of low frost-point humidity range at LNE-CETIAT
    Mr GEORGIN – LNE-CETIAT / France
- Poster
  - Performance Qualification of a Novel Transportable Dew Point Calibrator
    Mr FARLEY – QROMETRIC / United Kingdom
  - Determining water-vapour enhancement factors in ultra-high pure process gases at VSL
    Mr PANMAN – VSL / The Netherlands

The total participation in this congress was approximately 550, including exhibitors, speakers, and attendees from 40 countries. Approximately 45% of the attendees represented the industry, while the remaining 55% came from universities, NMIs, or the research field.

Further information are available here: https://www.prometh2o.eu/en/documents and here: https://www.cim2023.com/en/

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Next steps and announcements

The upcoming e-newsletter will provide you with information about the other tasks and developments carried out within the project, including those in the other work packages.

Save the date !!

The consortium organize a workshop in the framework of the Gas Analysis Conference 2024. It will be held on January 30th 2024, at Porte de Versailles, Hall 4 – Paris, France.

The programme is available here: https://www.gasanalysisevent.com/images/gasanalysis/docs/GAS2024-Fullprogramme.pdf

Do not hesitate to register for this event; we will be delighted to welcome you and present our project!

JRP partners will also be presenting their work during the Gas Analysis 2024 conference. Come and see us!

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