



# PROMETH<sub>2</sub>O

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20IND06 PROMETH2O

**WP2**

## **Provision of robust traceability to trace water measurements in real humid gas mixtures**

**Project Progress Meeting at M9**

Online, hosted by INRIM

**Wednesday 9 March 2022**

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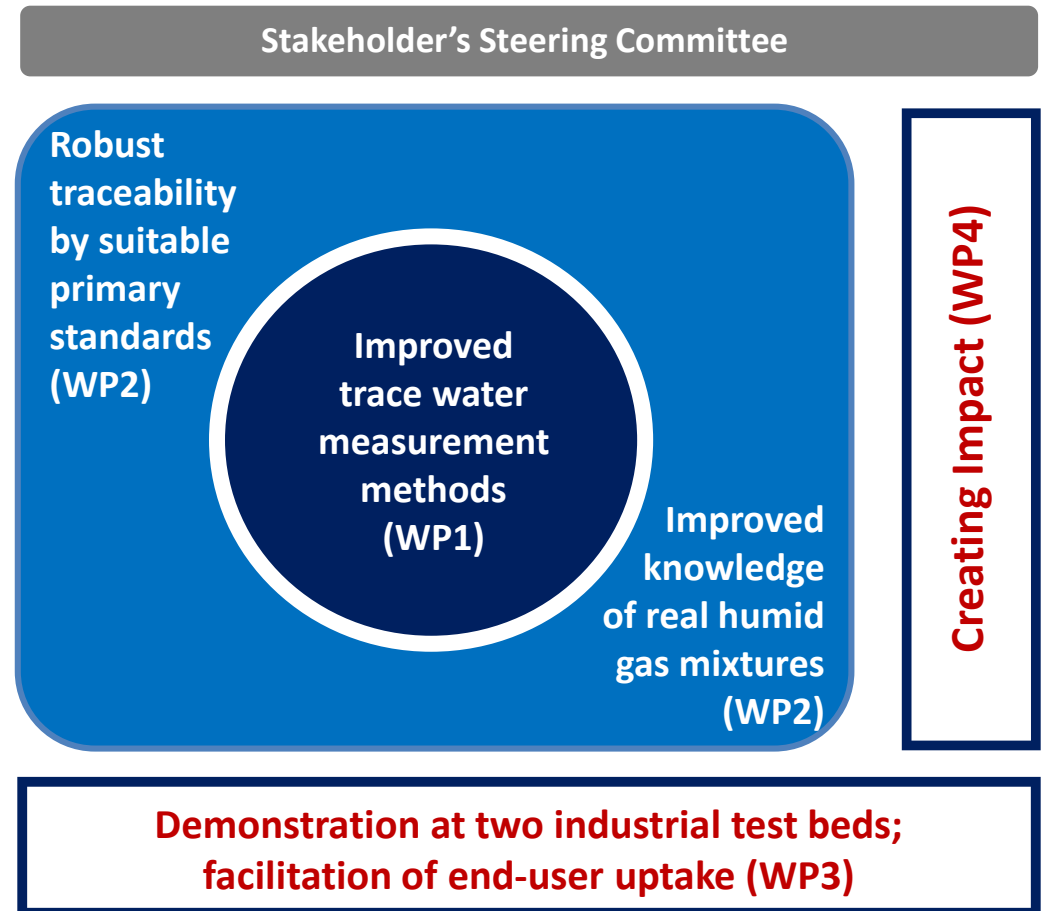
**EMPIR**



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

Development and validation of primary standards for trace water vapour measurements in ultra-pure gases

- by using a variety of **complementary generation techniques (Task 2.1)**
- by improving the knowledge of the **non-ideal behaviour of humid gas mixtures (Task 2.2 - Task 2.3)**



## Task 2.1: Development of primary humidity standards for trace water vapour in an increased range of gas matrices

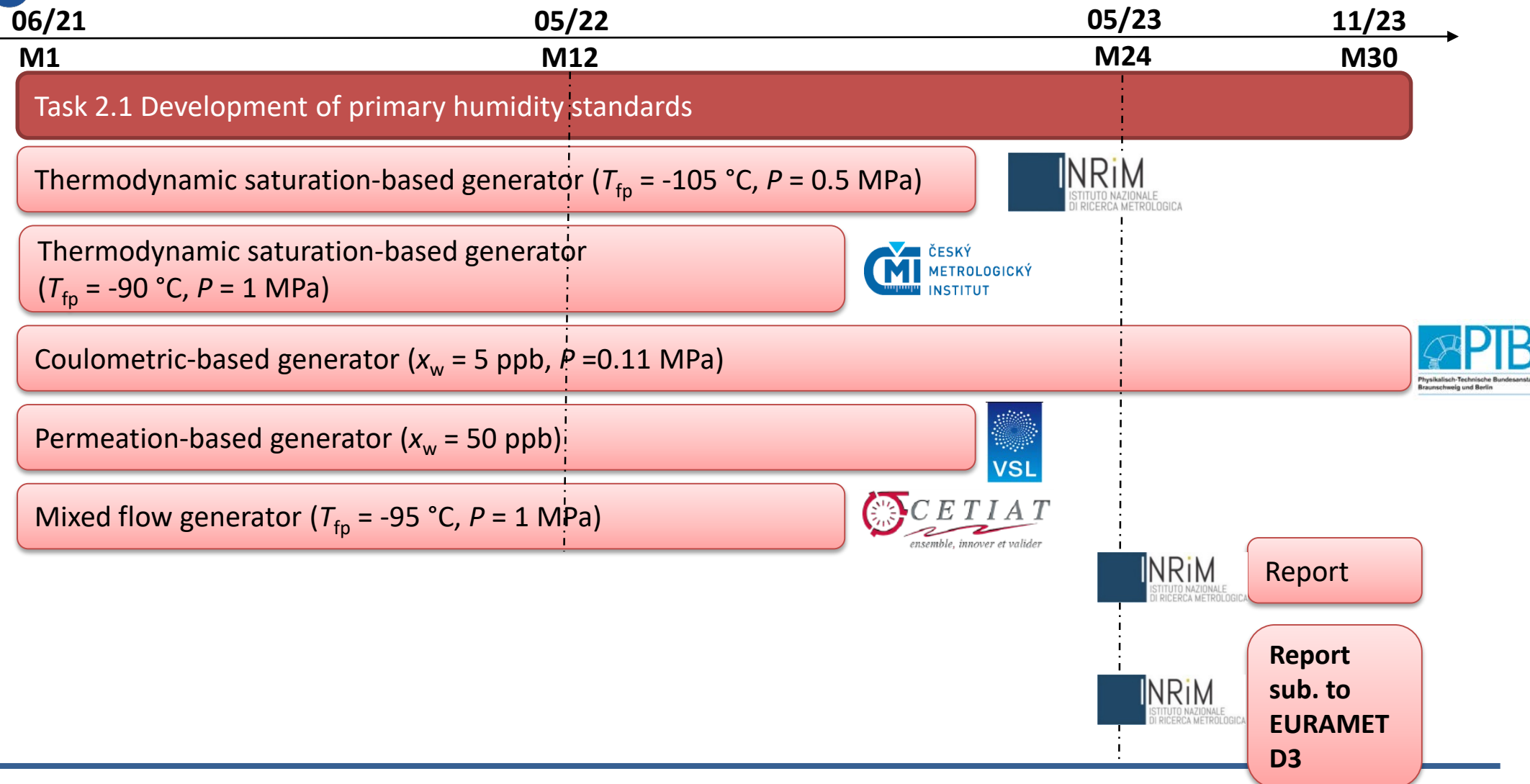
### GOAL

**Development, extension or improvement of primary standards to generate a water vapour amount fraction in the 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<sub>2</sub>, Ar and H<sub>2</sub> at pressures up to 1 MPa.**

### TECHNIQUES

- **Thermodynamic-based standard generators**
- **Water vapour amount fraction generators**

## Task 2.1: Development of primary humidity standards for trace water vapour in an increased range of gas matrices



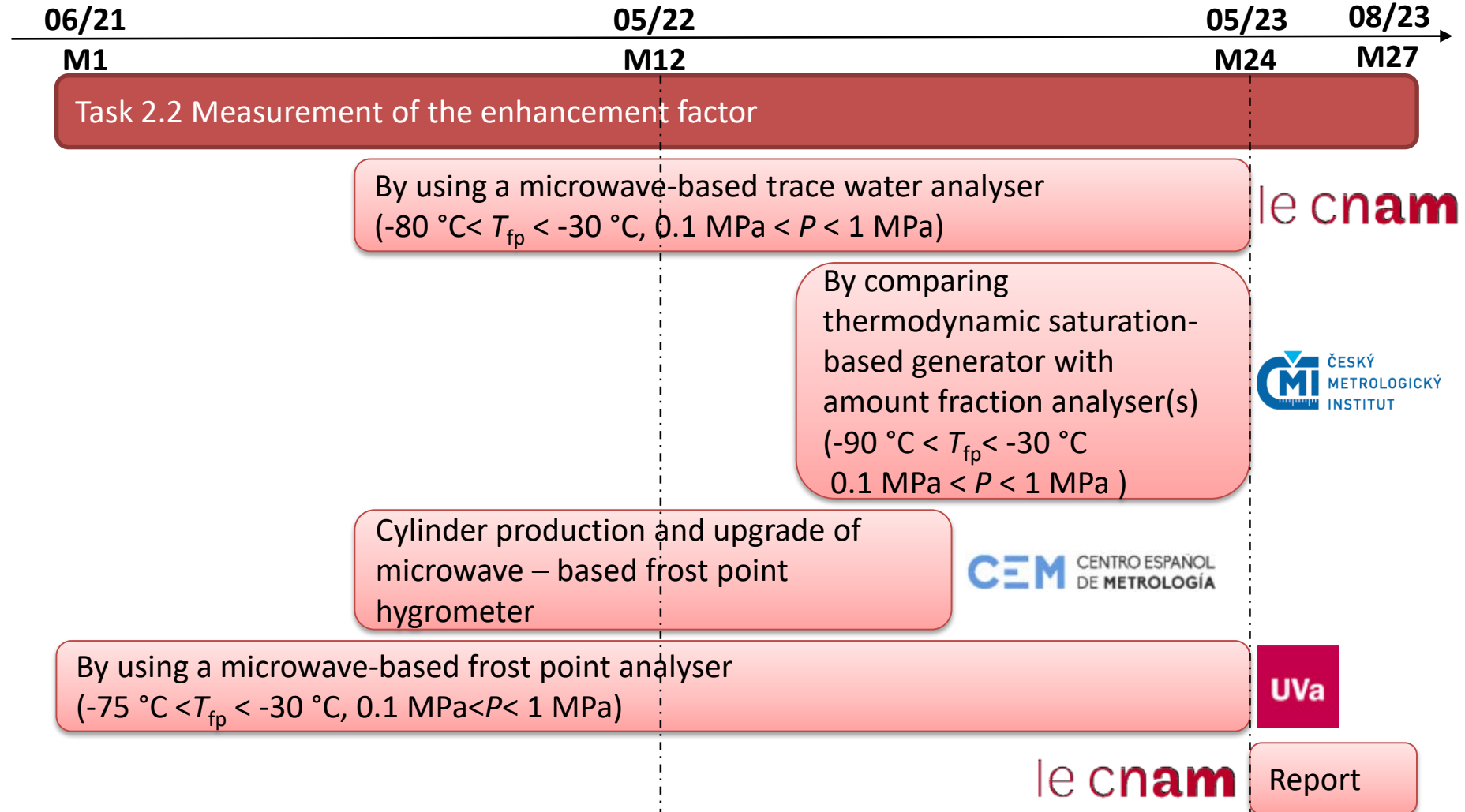
### GOAL

Improving of the data available for water vapour enhancement factor in air, N<sub>2</sub>, Ar and H<sub>2</sub> in the frost-point temperature range between -90 °C and -30 °C and pressures from 0.1 MPa to above 1 MPa.

### METHOD

Cross domain experiments that rely on trace humidity standards developed in Task 2.1 and amount-of-substance measurements methods made available in WP1.

## Task 2.2: Measurement of the enhancement factor in selected humid gas mixtures



## Task 2.3: Development of correlation equations for humid gas mixtures between -30 °C and -90 °C from 0.1 MPa to above 1 MPa

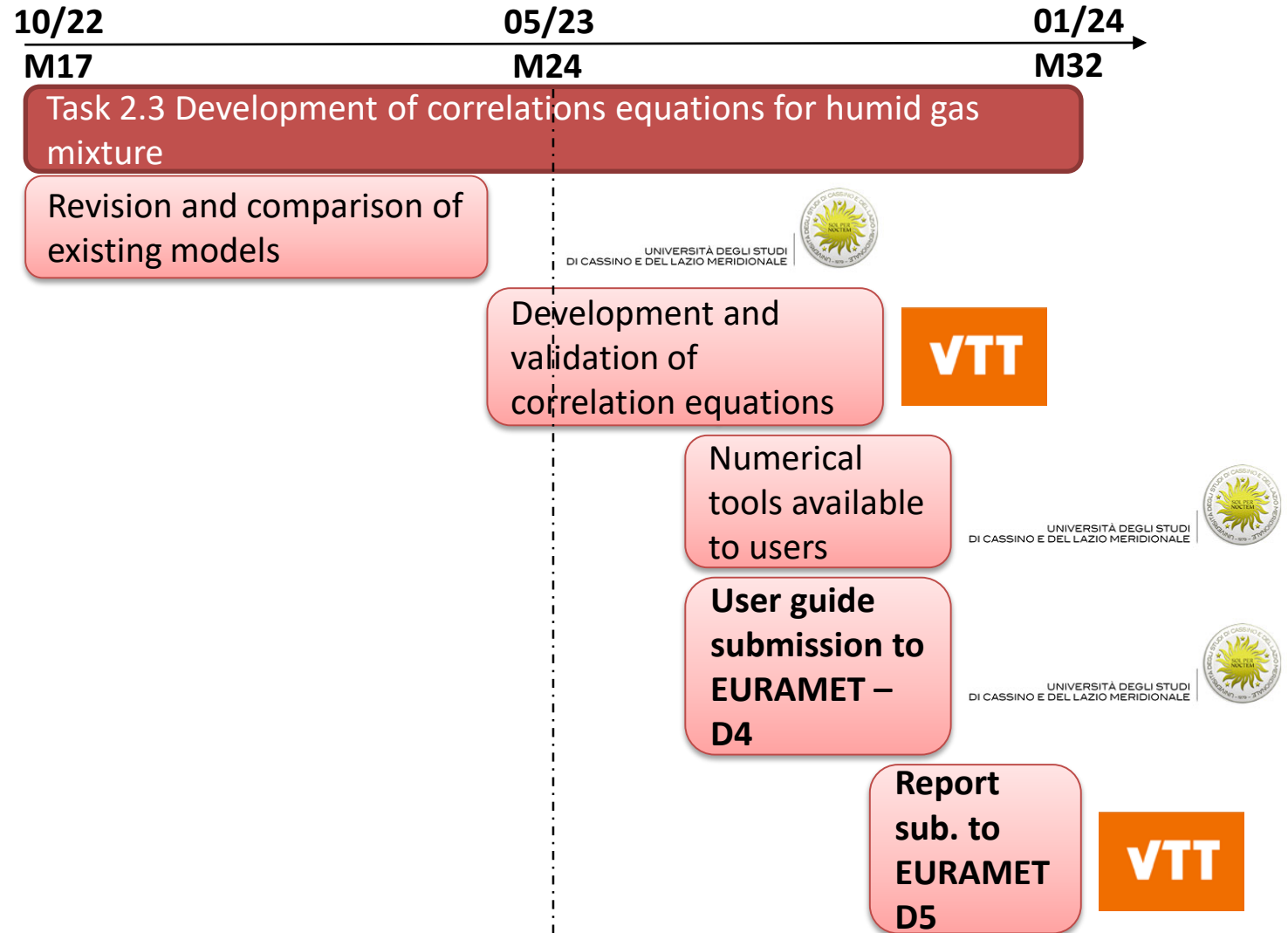
### GOAL

- To improve the humid gas mixtures correlation equations in the temperature range between -30 °C and -90 °C at pressures from 0.1 MPa to above 1 MPa for N<sub>2</sub>, Ar and H<sub>2</sub>.
- To offer a tool to end users for a better comparability of trace humidity measurements based on different principles and gas matrices.

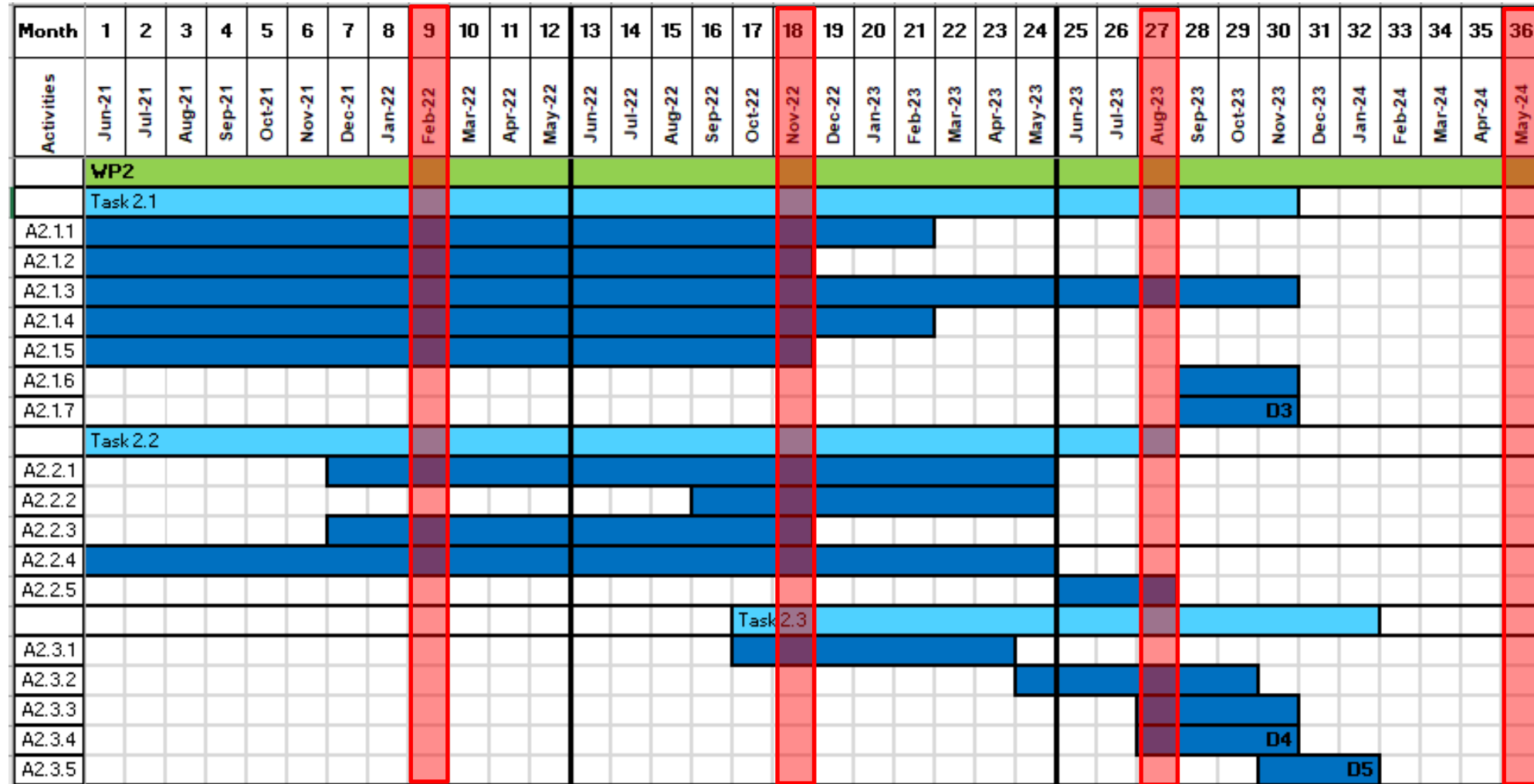
### METHOD

Modelling, simulation and validation of the correlation equations for the water vapour enhancement factor starting from existing non-ideal humid gas mixtures models.

## Task 2.3: Development of correlation equations for humid gas mixtures between -30 °C and -90 °C from 0.1 MPa to above 1 MPa







Reporting period



# PROMETH<sub>2</sub>O

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## Task 2.1

# Development of primary humidity standards for trace water vapour in an increased range of gas matrices

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Activity number	Activity description	Partners (Lead in bold)
<b>A2.1.1</b> M21	<p>INRIM will improve its thermodynamic saturation-based primary standard generator to generate the humid gas mixtures standard in nitrogen and argon at pressures up to 0.5 MPa and to extend the lower limit of frost-point temperature to -105 °C with a standard uncertainty of 0.35 °C. VTT will extend its saturation-based primary standard generator to -100 °C at 0.11 MPa to generate humid gas mixtures in nitrogen and air.</p> <p>INRIM and VTT will use such primary humidity standards to provide traceability to trace water analysers, such as the CC-FS-CRDS spectrometer (A1.1.1), high-quality CMH, and CE-FM spectroscopy hygrometer (A1.1.4) and underpin their validation in A1.2.1 to A1.2.3.</p>	<b>INRIM</b> , VTT

The design of the extended-range generator was completed; parts were ordered and they will be delivered in the next months.

New trace water analysers (low-frost-point CMH and CRDS) were purchased and are under commissioning:

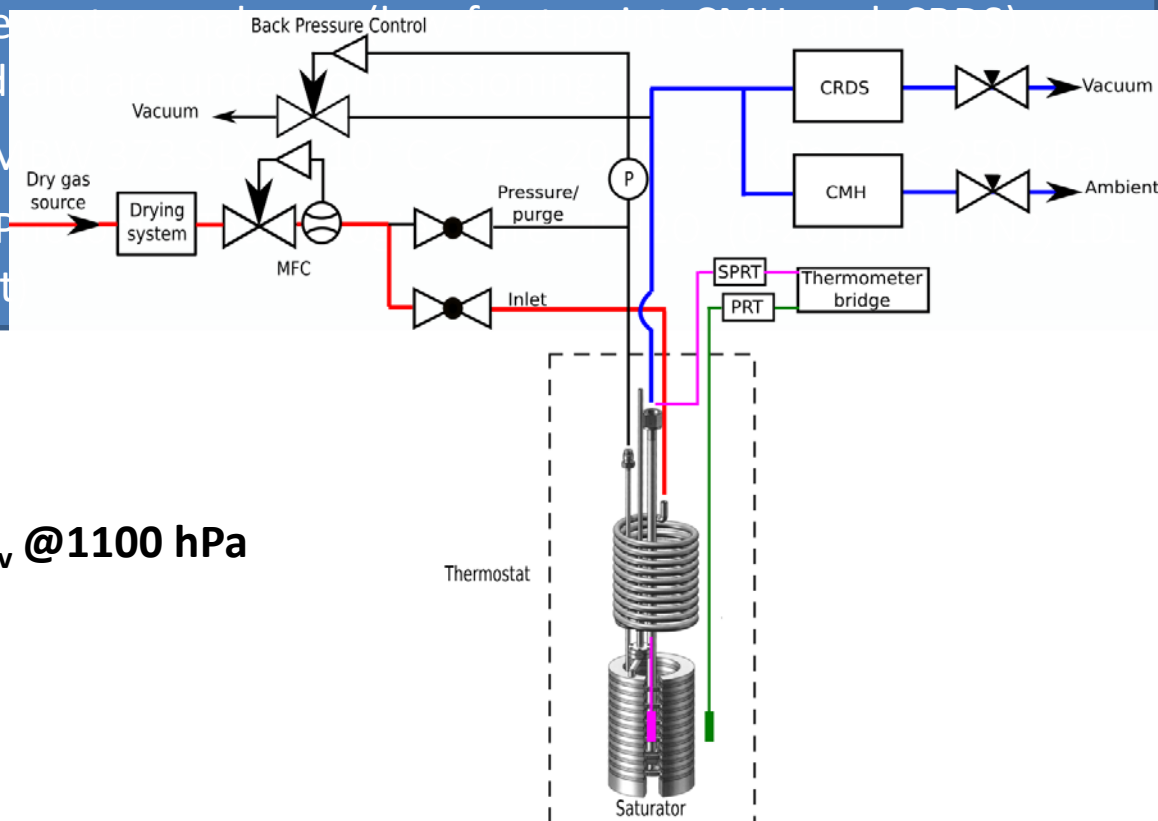
- CMH: MBW 373-SLX ( $-110\text{ °C} < T_{fp} < 20\text{ °C}$  ;  $50\text{ kPa} < P < 250\text{ kPa}$ )
- CRDS: Photonics Technologies Pure<sup>n</sup>-T H<sub>2</sub>O (0-20 ppm in N<sub>2</sub>, LDL 200 ppt)

Activity number	Activity description
A2.1.1 M21	INRIM will improve its thermodynamic saturation-based primary standard to generate the humid gas mixtures standard in nitrogen and argon at 0.5 MPa and to extend the lower limit of frost-point temperature standard uncertainty of 0.35 °C. VTT will extend its saturation-based generator to -100 °C at 0.11 MPa to generate humid gas mixtures in INRIM and VTT will use such primary humidity standards to provide water analysers, such as the CC-FS-CRDS spectrometer (A1.1.1), and CE-FM spectroscopy hygrometer (A1.1.4) and underpin their validity to A1.2.3.

The design of the extended-range generator was completed; parts were ordered and they will be delivered in the next months.

New trace gas

- CMH: M
- CRDS: F
- 200 ppt

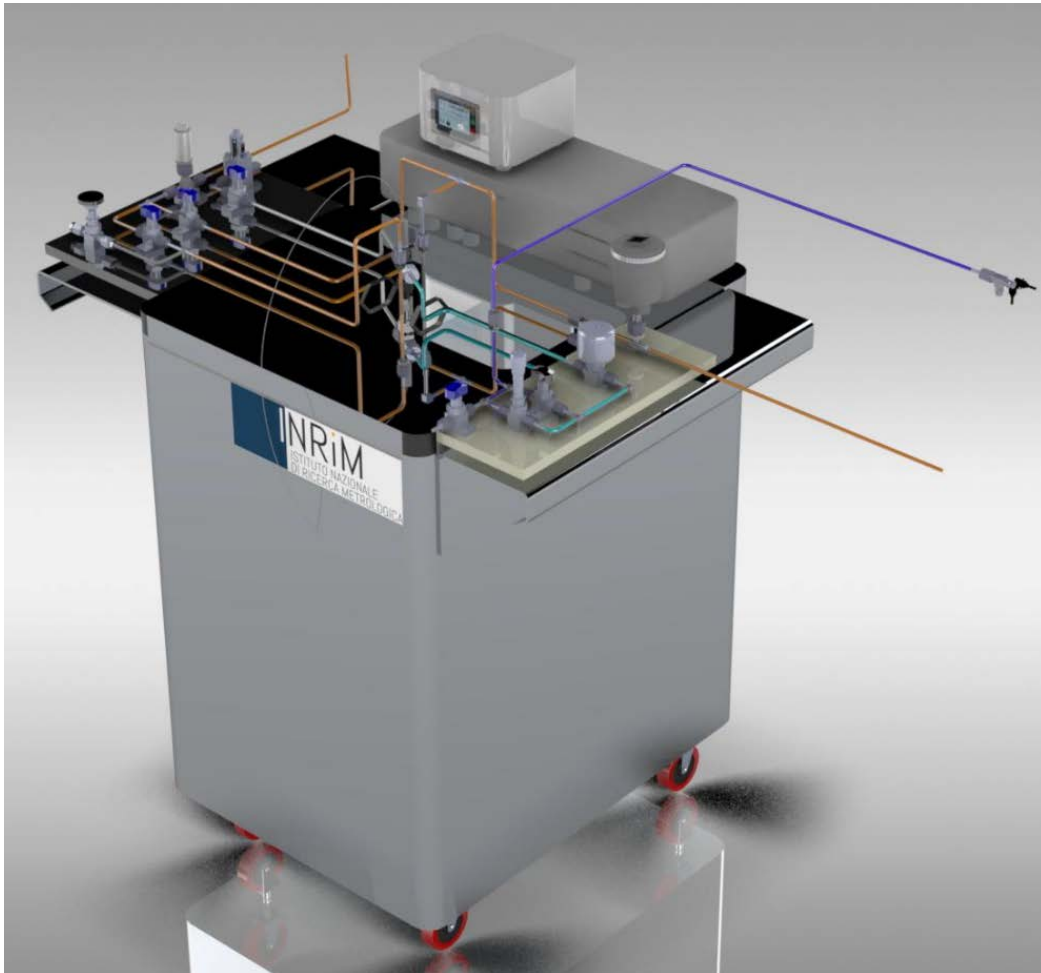


## LFP HUMIDITY GENERATOR

- Single-pressure, single-pass humidity generator
- **Frost-point temperature between -99 °C and -20 °C**
- **Water vapour mole fraction between 15 ppb<sub>v</sub> and 945 ppm<sub>v</sub> @1100 hPa**
- Pressure: 200 hPa to 2400 hPa
- Carrier gas: Nitrogen

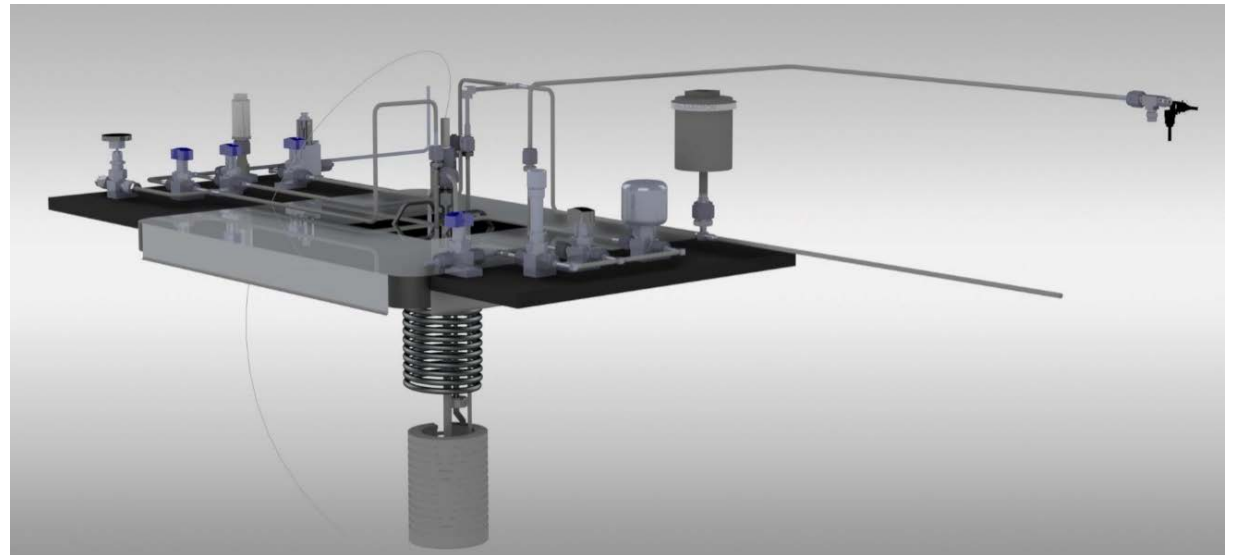
Reference: R Cuccaro et al 2018 Meas. Sci. Technol. 29 054002  
<https://doi.org/10.1088/1361-6501/aaa785>

# Range expansion of the LFP generator

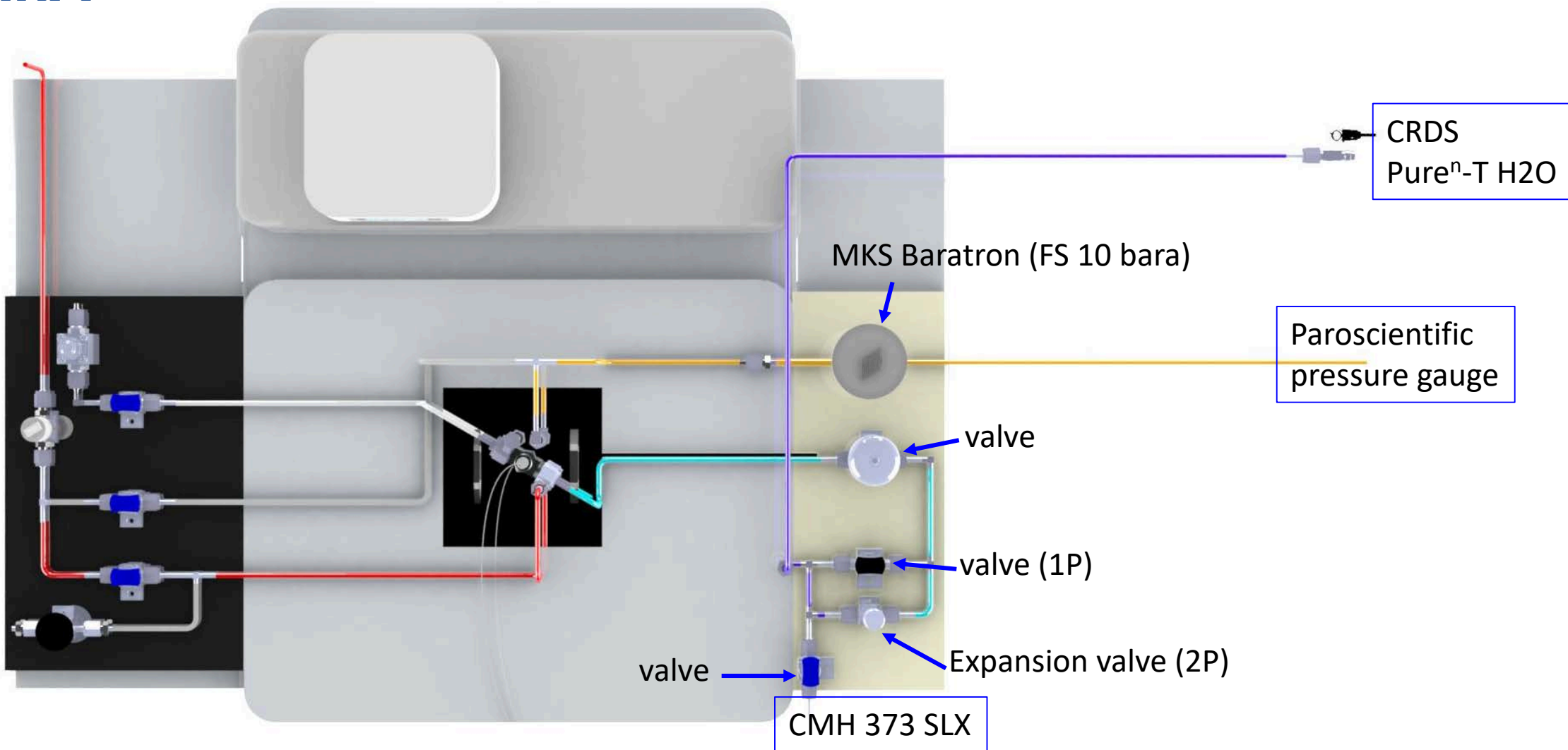


## LFP HUMIDITY GENERATOR - new version

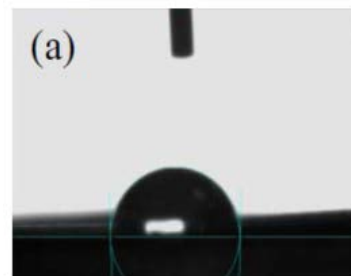
- 2-pressure, single-pass humidity generator
- Frost-point temperature between  $-105\text{ }^{\circ}\text{C}$  and  $-20\text{ }^{\circ}\text{C}$
- Water vapour mole fraction between 5 ppb<sub>v</sub> and 1038 ppm<sub>v</sub> @1000 hPa
- Pressure: 200 hPa to 5000 hPa
- Carrier gas: Nitrogen, Argon



# Schematic of the new generator



- The generator was operating at the beginning of the project.
- Currently the thermostatic bath has some malfunctioning and is transported for the repairment.
- Adsorption/desorption contributes to the uncertainty budget by 27%
- Temperature gradient contributes by 4%.
- Our target is to shrink the uncertainty budget possibly by 20-25%



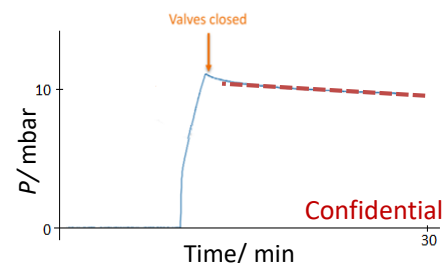
(a)  
Droplet of water on highly polished SS 316L

Coating techniques based on Chemical Vapor Deposition (CVD) :

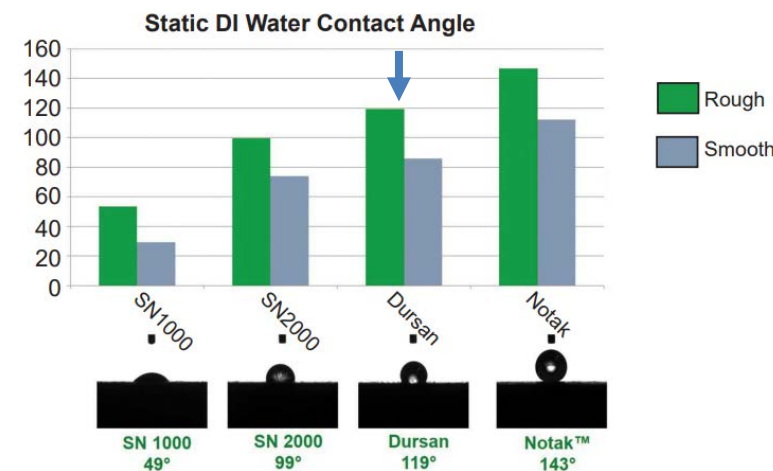
## NON-WETTING

SilcoNert 2000 doubles the hydrophobicity of stainless steel and is commonly specified in moisture analyzer applications.

In-house characterization:



Pressure stabilization time is reduced significantly by introducing Dursan coating





## C2.a Task 2.1: Development of primary humidity standards for trace water vapour in an increased range of gas matrices

The aim of this task is to develop primary humidity standards for ultra-trace water vapour in an increased range of gas matrices (nitrogen, air, argon and hydrogen) based on a range of principles providing traceability through different routes.

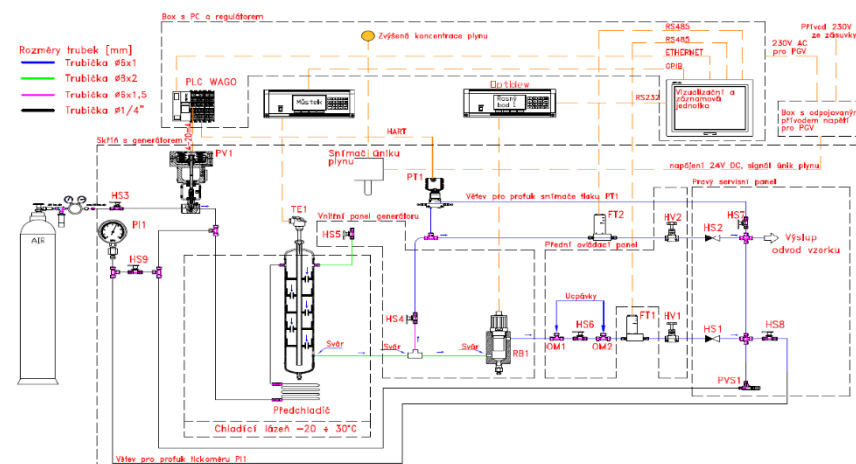
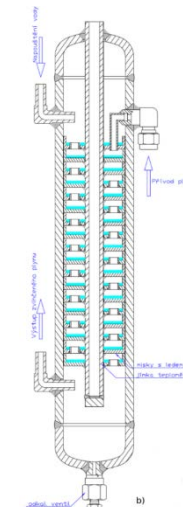
Plan:  
**03/2022**  
temperature bath measurement (stab., homogeneity, saturator),  
Pt100 calibration finished,  
installation into the “old” system  
  
**04/2022**  
Validation of the new system into  $-90^{\circ}\text{C}_{\text{fp}}$

	Activity number	Activity description	Partners (Lead in bold)
12/2022	A2.1.2 M18	CMI, INTA and UL will <b>upgrade their saturation-based generators</b> to produce humid gas mixtures in nitrogen and argon to extend the lower limit of reference frost-point temperatures <b>to <math>-90^{\circ}\text{C}</math></b> and at <b>pressures up to 1 MPa and above</b> , with standard uncertainty of $0.25^{\circ}\text{C}$ at $-90^{\circ}\text{C}$ . Only for INTA the pressure will go to 0.5 MPa.	<b>CMI</b> , INTA, UL
12/2023	A2.1.6 M30	INRIM, VTT, CMI, INTA, UL, PTB, MBW, VSL, and CETIAT using the results from A2.1.1 to A2.1.5 will write a summary report on the development of the trace water vapour standards describing the range and uncertainty achievable and the gas species in which reference humidity values can be generated.	<b>INRIM</b> , VTT, <b>CMI</b> , INTA, UL, PTB, MBW, VSL, CETIAT
	A2.1.7 M30	INRIM, VTT, CMI, INTA, UL, PTB, MBW, VSL, and CETIAT will review the report from A2.1.6 and will send it to the coordinator.  Once the report has been agreed by the consortium, the coordinator on behalf of INRIM, VTT, CMI, INTA, UL, PTB, MBW, VSL, and CETIAT will then submit it to EURAMET as <b>D3: ‘Report on the development of primary trace water vapour 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 (<math>-65^{\circ}\text{C}</math> to <math>-105^{\circ}\text{C}</math>) with relative standard uncertainty less than 3 % to 8 % in selected gas matrices at pressures up to 1 MPa’.</b>	<b>INRIM</b> , VTT, <b>CMI</b> , INTA, UL, PTB, MBW, VSL, CETIAT



- **Primary humidity generator, 1P1T type, thermodynamic saturation-based principle, in Prague:**

- **Pressure: 15 MPa max.**
- **Humidity range:  $(-80 \text{ to } 30) ^\circ\text{C}_{\text{dp/fp}}$**
- **Gas matrix: Air,  $\text{N}_2$ , Ar,  $\text{CH}_4$ , natural gas**
- **Flow rate: up to approx.  $2 \text{ L}_\text{N}/\text{min}$**
- **$U (k=2)$  from  $-80 ^\circ\text{C}_{\text{fp}}$  to  $-30 ^\circ\text{C}_{\text{fp}}$ :**
  - ✓ at **1 MPa:**  $0.11\text{-}0.23 ^\circ\text{C}$
  - ✓ at **0.1 MPa:**  $0.09\text{-}0.15 ^\circ\text{C}$  or less



Kambič Calibration  
 bath OB-22/2 ULT  
 to -90 °C...



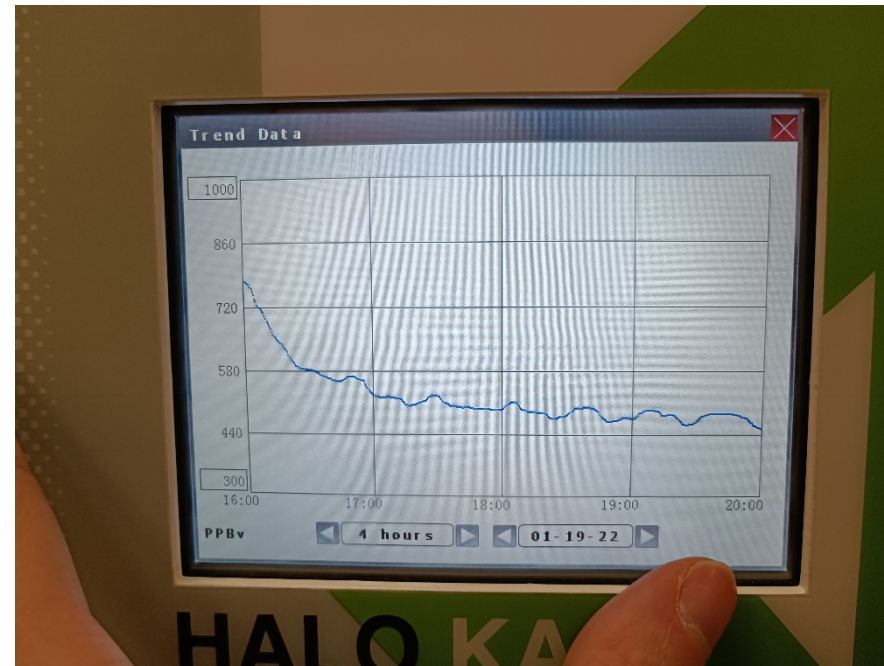
Thermometry bridge MI 6242T +  
 resistance etalon  
 - improved temperature  
 measurement precision

New SPRT glass  
 temperature resistance  
 probe...



Thermodynamic saturator  
 (expected maintaining  
 function without  
 modification)

New fraction humidity analyzer at CMI:  
Tiger Optics, HALO KA H<sub>2</sub>O – laser absorption CRDS hygrometer



traceability (NIST)

**Tiger Optics**  
Certificate of Calibration

Customer: ELECTRA A.S.  
Purchase Order: 01773  
Call Page: 0400 KA  
Part Number: 17500-S  
Serial Number: 1278  
Date Issued: 11/17/2024

This is to certify that the instrument described above has been assembled, inspected and tested in accordance with specifications set by Tiger Optics, LLC.

On the date tested, the output values of this instrument were compared against known reference standards and were found to be within the manufacturer's published operating specifications as stated in the user manual.

Reference	Reference Standard	HALO KA Reading
Q-Gas Dry Gas	< 1 ppb H <sub>2</sub> O in N <sub>2</sub>	< 500 ppb
Q-100 Wet Gas	1000 ppb H <sub>2</sub> O in N <sub>2</sub>	1002 ppb
Unit: 100		± 100% Relative H <sub>2</sub> O

The accuracy and reliability of this instrument are based on the reference standards that are compared, at planned intervals, to national standards maintained by the National Institute of Standards and Technology (NIST), by comparison to national physical constants or other existing metrology measurements.

The instrument standards that support these results are well known and strictly set intervals to ensure full repeatable accuracy.

*[Signature]*  
Authorized Signature

Tiger Optics, LLC • 219 Oriental Road • Hingham, MA 01944 • 253-656-8800

SW





New fraction humidity analyzer at CMI:  
Tiger Optics, HALO KA H<sub>2</sub>O

## HALO KA H<sub>2</sub>O Ultra-High Purity Gas Analyzer



Performance	
Operating range	See table on next page
Detection limit (LDL, 3σ/24h)	See table on next page
Precision (1σ, greater of)	± 0.75% or 1/3 of LDL
Accuracy (greater of)	± 4% or LDL
Speed of response	< 2 minutes to 95%*
Environmental conditions	10°C to 40°C 30% to 80% RH (non-condensing)
Storage temperature	-10°C to 50°C
Gas Handling System and Conditions	
Wetted materials	316L stainless steel (corrosive gas version optional)
	10 Ra surface finish
Gas connections	1/4" male VCR inlet and outlet
Leak tested to	1 x 10 <sup>-9</sup> mbar l / sec
Inlet pressure	10 – 125 psig (1.7 – 9.6 bara)
Flow rate	0.05 – 1.8 slpm
Sample gases	Most inert, toxic, passive and corrosive matrices
Gas temperature	Up to 60°C

Dimensions	
H x W x D [in (mm)]	
Standard sensor	8.73 x 8.57 x 23.6 (222 x 218 x 599)
Sensor rack	8.73 x 19.0 x 23.6 (222 x 483 x 599)
(fits up to two sensors)	
Weight	
Standard sensor	28 lbs (12.7 kg)
Electrical and Interfaces	
Platform	Max series analyzer
Alarm indicators	2 user programmable 1 system fault
	Form C relays
Power requirements	90 – 240 VAC, 50/60 Hz
Power consumption	40 Watts max.
Signal output	Isolated 4–20 mA per sensor
User interfaces	5.7" LCD touchscreen 10/100 Base-T Ethernet USB, RS-232, RS-485 Modbus TCP (optional)
Data storage	Internal or external flash drive
Certification	CE Mark

Performance, H <sub>2</sub> O:	Range	LDL (3σ)	Precision (1σ) @ zero
INERT/ PASSIVE GASES	In Nitrogen	0 – 20 ppm	300 ppt
	In Helium	0 – 4 ppm	100 ppt
	In Argon	0 – 9 ppm	130 ppt
	In Hydrogen	0 – 16 ppm	200 ppt
	In Deuterium (D <sub>2</sub> )	0 – 14 ppm	900 ppt
OXYGENATED GASES	In Oxygen	0 – 10 ppm	150 ppt
	In Clean Dry Air (CDA)	0 – 18 ppm	300 ppt
	In CO	0 – 24 ppm	600 ppt
	In CO <sub>2</sub>	0 – 25 ppm	800 ppt
	In COS	0 – 23 ppm	4 ppb
RARE GASES	In Neon	0 – 5 ppm	100 ppt
	In Krypton	0 – 11 ppm	160 ppt
	In Xenon	0 – 13 ppm	250 ppt
COR- ROSIVE GASES	In Cl <sub>2</sub> *	0 – 25 ppm	650 ppt
	In HCl†	0 – 50 ppm	1200 ppt
	In HBr*	0 – 50 ppm	12 ppb
FLUORINATED GASES	In SF <sub>6</sub>	0 – 15 ppm	400 ppt
	In NF <sub>3</sub>	0 – 20 ppm	600 ppt
	In CF <sub>4</sub>	0 – 15 ppm	800 ppt
	In C <sub>2</sub> F <sub>6</sub>	0 – 15 ppm	1200 ppt
	In C <sub>3</sub> F <sub>8</sub>	0 – 20 ppm	1200 ppt
	In C <sub>4</sub> F <sub>6</sub>	0 – 25 ppm	150 ppb
	In C <sub>4</sub> F <sub>8</sub>	0 – 20 ppm	1200 ppt
	In C <sub>5</sub> F <sub>8</sub>	0 – 32 ppm	8 ppb
HY- DRIDE GASES	In 1% GeH <sub>4</sub> /99% H <sub>2</sub> mixture	0 – 16 ppm	7 ppb
	In 10% GeH <sub>4</sub> /90% H <sub>2</sub> mixture	0 – 16 ppm	35 ppb

\*Corrosive gas version required.  
†Corrosive gas version recommended for H<sub>2</sub>O concentration that could exceed 1 ppm.  
Contact us for additional analytes and matrices.  
U.S. Patent # 7,277,177

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**Tiger Optics**  
A Process Insights Company  
3/2021

### A2.1.2 M18

CMI, INTA and UL will upgrade their saturation-based generators to extend the lower limit of reference frost-point temperatures to -90 °C and at pressures up to 1 MPa and above (INTA to 0.5 MPa) with standard uncertainty of 0.25 °C at -90 °C. Such trace water generators in nitrogen and argon will perform/support the investigation of water vapour enhancement factor in Task 2.2. Participation in the pilot study described in Task 1.3 requires these standards to be available.

INTA

- INTA has recalibrated the temperature and pressure sensors of the frost-point two pressure-two temperature saturation-based generator, an upgraded THUNDER SCIENTIFIC 4500 model. Calibration of the transfer standard hygrometer to be provided to UVA in M11 has been initiated. Currently INTA is awaiting the repair of the water cooling system for the 4500 in order to continue measurements of the chilled-mirror and CRDS transfer standards are necessary for the characterization of the new generator in the overlapping range down to -75 °C.
- The sonic nozzles, regulators and accessories necessary for the modification of the new generator have been ordered. Maintenance and testing of the getter N2 purifier has been accomplished. Calibration of the SPRTS has been completed.

- upgrade frost-point generator
- frost-point temperatures to -90 °C and at pressures up to 1 Mpa
- standard uncertainty of 0.25 °C at -90 °C
- in nitrogen and argon
- perform/support the investigation of water vapour enhancement factor
- by november 2022



## STATUS

- design of the saturator about to end
- starting with the preparations for construction
- some delay (staff, covid)

- **Task 2.1.3 – M30 (Development/Testing):**

Development of a coulometric principal standard to generate water vapour in nitrogen and argon. Tests of selected instruments with the generator.

(**PTB**, MBW)

- **Task 2.1.6 – M30 (Report):**

Summary report on the development of the trace water vapour standards.

(INRIM, VTT, CMI, INTA, UL, **PTB**, MBW, VSL, CETIAT)

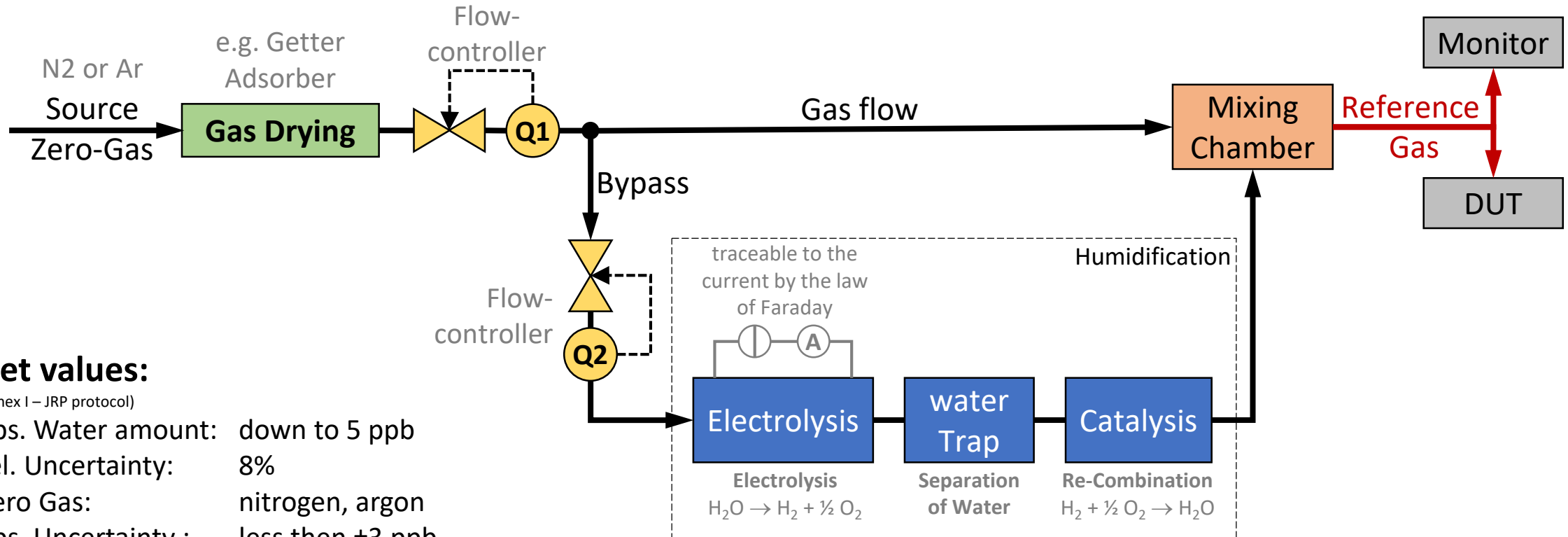
- **Task 2.1.7 – M30 (Review):**

Review of the report (A2.1.6) and send it to the coordinator.

(INRIM, VTT, CMI, INTA, UL, **PTB**, MBW, VSL, CETIAT)



### Basic setup of the Coulometric Trace Water Generator (CTWG)



#### Target values:

(from Annex I – JRP protocol)

- abs. Water amount: down to 5 ppb
- rel. Uncertainty: 8%
- Zero Gas: nitrogen, argon
- abs. Uncertainty : less than  $\pm 3$  ppb

## Steps already in progress

- Development of overall concept in process
- Acquisition of hardware in process
- Preparation for test of catalyst for recombination
- Test of gas purification systems using high sensitivity H<sub>2</sub>O CRDS detection

## Schedule for major steps

- |                                  |           |
|----------------------------------|-----------|
| • Elaboration of design          | 21/22     |
| • Acquisition of hardware        | end 21/22 |
| • Construction of the apparatus  | 22/23     |
| • Commissioning and measurements | 23        |

Set up a permeation system based on a passivated magnetic suspension balance to generate primary standard of water amount fractions following ISO 6145-10 and ISO 6145-7.

Target range: in the range of 50 nmol/mol up to 5  $\mu$ mol/mol

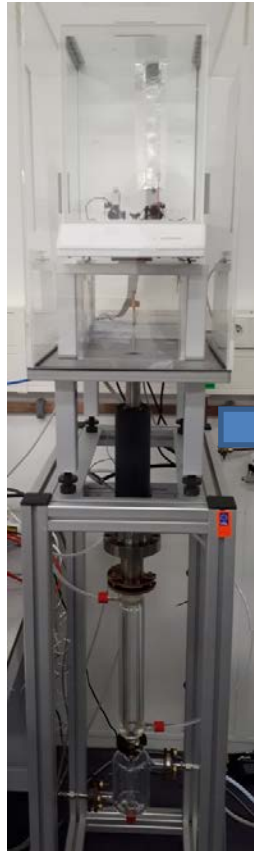
Matrix gases: N<sub>2</sub> and H<sub>2</sub>

**Initial steps taken:**

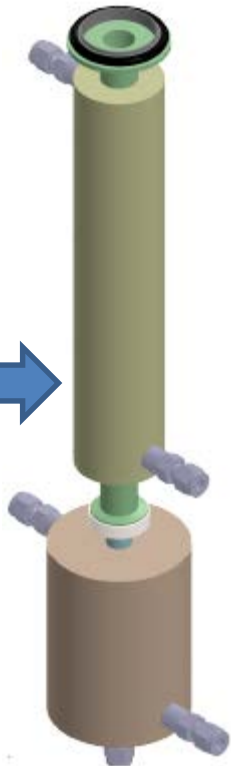
- 1) Completed design. External company manufactured metal chamber with less connections compared to glass chamber
- 2) Ordered new purifier
- 3) Started testing

**Still to do:**

- 3) Complete testing and validation of the new system



previous



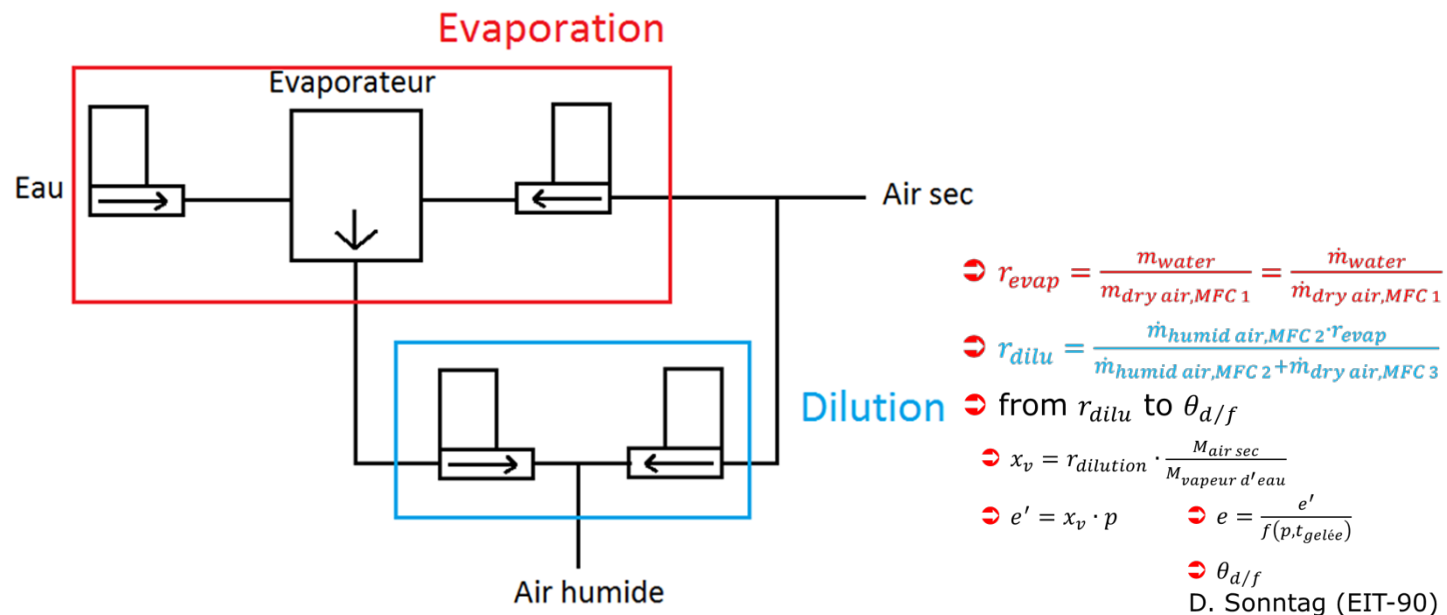
design



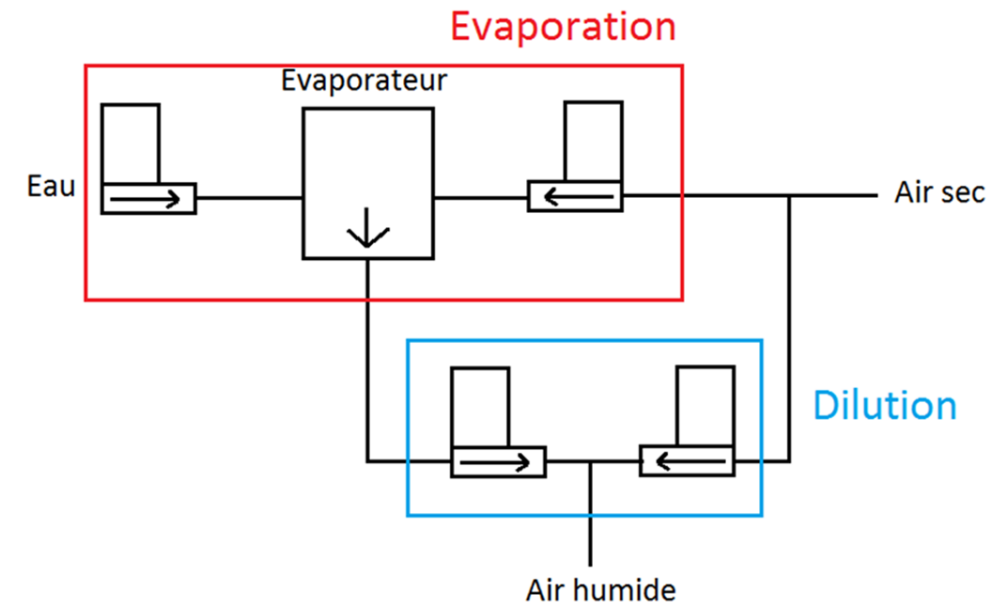
new chamber

Activity number	Activity description	Partners
A2.1.5 M18	CETIAT will upgrade its mixed flow generator in pressure, from 0.1 MPa up to 1 MPa, and in frost point temperature, from -85 °C down to -90 °C, possibly -95 °C, with a standard uncertainty of 0.25°C.	CETIAT

- Upgrade of the mixed flow generator / JRP METEOMET 2



- Upgrade of the mixed flow generator / JRP METEOMET 2
  - Pressure range from 40 kPa to 100 kPa
    - Upgrade up to 1 000 kPa
      - Change of pressure controller (hardware + software)
  - Frost point range from -85 °C to +10 °C
    - Upgrade down to -90 °C (-95 °C)
      - Change molecular sieve
      - Add one supplementary dilution step
  - Uncertainty  $U_{k=2}=0,35\text{ °C}$ 
    - Update the uncertainty budget according to the upgrades





# PROMETH<sub>2</sub>O

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20IND06 PROMETH2O

## Task 2.2

# Measurement of the enhancement factor in selected humid gas mixtures

**Project Progress Meeting at M9**

Online, hosted by INRIM

**Wednesday 9 March 2022**

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**EMPIR**

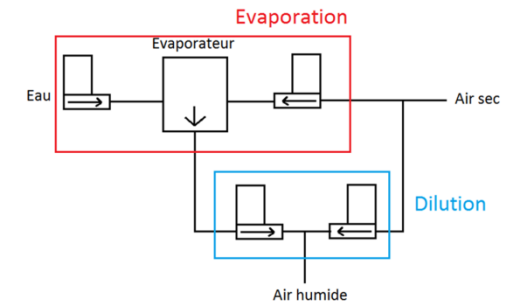


The EMPIR initiative is co-funded by the European Union's Horizon 2020 research and innovation programme and the EMPIR Participating States

Activity number	Activity description	Partners
A2.2.1 M24	CNAM and CETIAT using the facility developed in A2.1.5, will upgrade CNAM microwave-based trace water analyser to perform measurements of the enhancement of water vapour in nitrogen and argon in the frost-point temperature range between -80 °C and -30 °C at selected pressures from 0.1 MPa to above 1 MPa.	<b>CNAM,</b> CETIAT

### CETIAT

- Upgrade of the mixed flow generator (cf A2.1.5)
  - Providing humid gas to microwave-based trace water analyser
- Technical support related to humidity



CNAM, contribution to the project (first 9 months):

- Conception of the new hygrometer (Activity A2.2.1)
- Publication of the article:

Berg, R. F., Chiodo, N., and Georgin, E.: **Silicone tube humidity generator**, Atmos. Meas. Tech., 15, 819–832, <https://doi.org/10.5194/amt-15-819-2022>, 2022.

The project is in the financial support. CNAM developed a compact humidity generator.



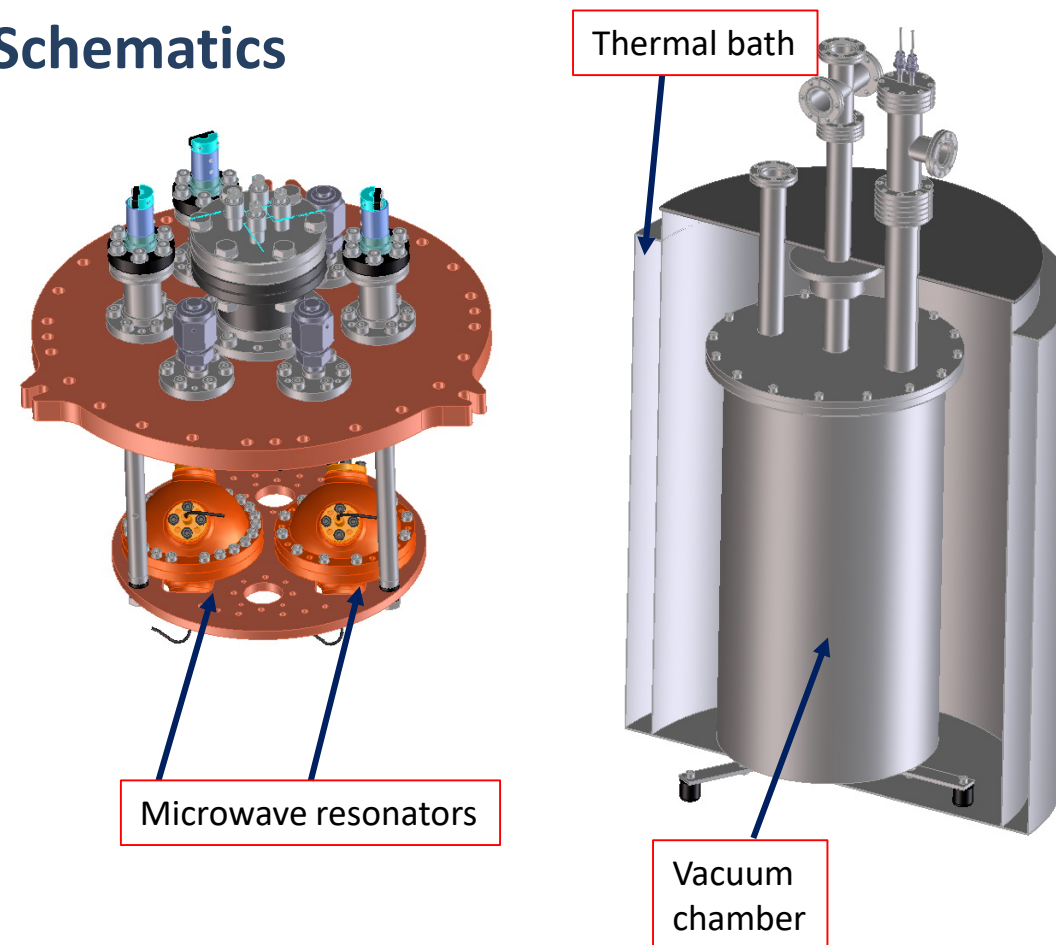
## New hygrometer (activity A2.2.1)

Design of the new hygrometer system operating up to a pressure of 7-10 bar: we have completed the design of the new system (microwave resonators, the pressure vessel, the thermal shield and the vacuum chamber).

The manufacturing procedure and the purchase process are in progress.

Possible risks: shortage of raw materials (copper, aluminum) and bureaucracy could lengthen manufacturing times by several months.

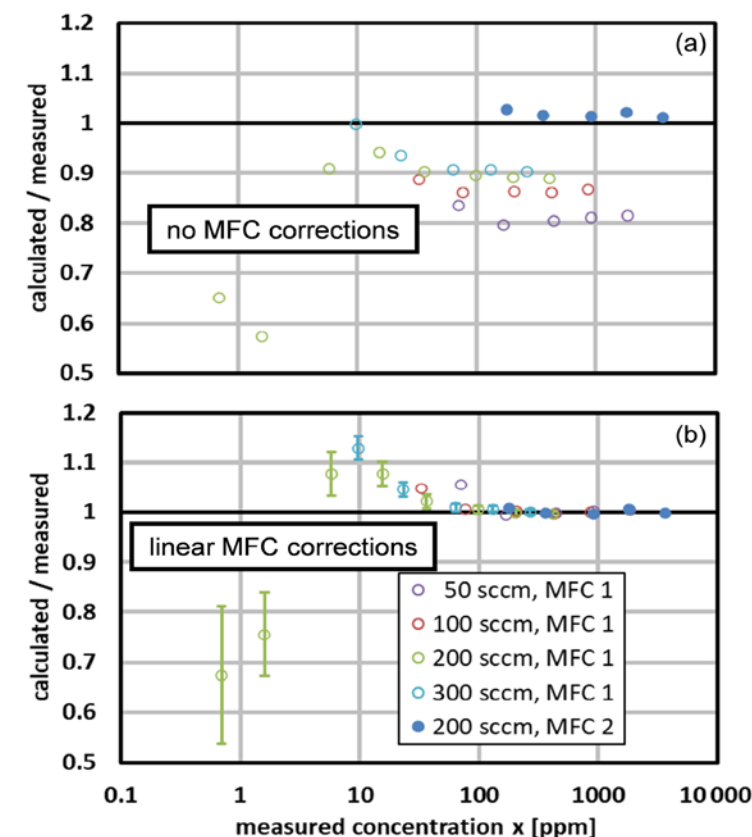
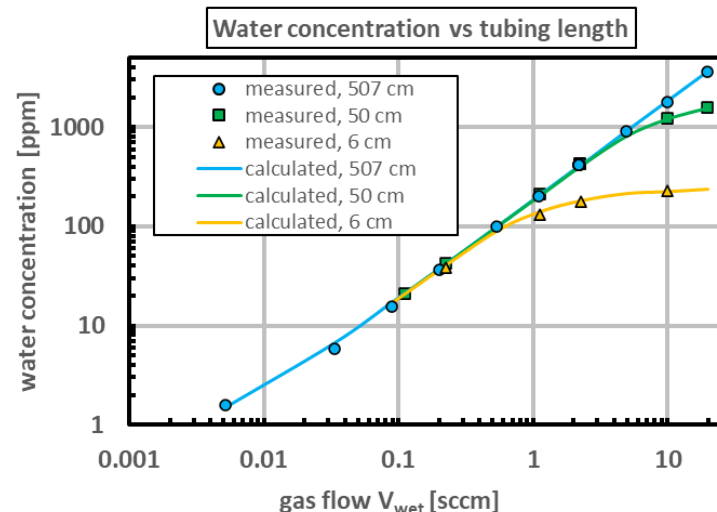
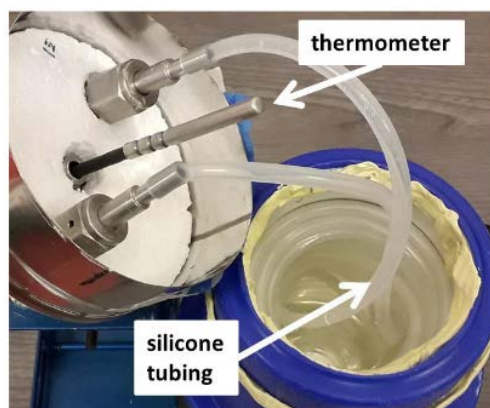
## Schematics



The article describes the model and construction of a two-flow (or divided-flow) humidity generator, developed at CNAM, that uses mass flow controllers to mix a stream of dry gas with a stream of humid gas saturated at 28 °C. It can generate a wide range of humidity, with mole fractions in the range between 0.7 ppm and 5000 ppm.

The generator's novel feature is a saturator that comprises 5 m of silicone tubing immersed in water.

The model required corrections for the humidity of the input “dry” gas, the permeation of argon through silicone, the diffusion of water vapor through argon, and the pressure drops caused by flow through capillaries due to mass-flow controllers (MFC).



## C2.b Task 2.2: Measurement of the enhancement factor in selected humid gas mixtures

The aim of this task is to improve the measurements available for water vapour enhancement factor in nitrogen, argon and hydrogen at selected temperatures and pressures, in the frost-point temperature range between -90 °C and -30 °C and pressure range from 0.1 MPa to above 1 MPa.

	Activity number	Activity description	Partners (Lead in bold)	Plan:
6/2023	A2.2.2 M24	CMI and UL, using the upgraded saturation-based generators from A2.1.2, will perform independent measurements of the enhancement of water vapour in <b>nitrogen and argon</b> in the frost-point temperature range between <b>-90 °C and -30 °C</b> . VSL, using its existing standard, will confirm the measurements to -80 °C at selected pressures from <b>0.1 MPa to above 1 MPa</b> .  These independent measurements will evaluate the non-ideality of gas mixtures (i.e., enhancement factor) with trace amount of water by comparing humid gas mixtures generated by frost-point temperature standards with corresponding humidity quantities as measured by amount-of-substance fraction analyser(s).	<b>CMI</b> , VSL, UL	<b>03/2022</b> – preparation (possible start of validation)  <b>04/2022</b> – slot for unrealeted measurement (customers)  <b>05/2022</b> – validation -90 °C <sub>fp</sub>
9/2023	A2.2.5 M27	CNAM, using the results from A2.2.1 to A2.2.4 will prepare a report stating the improved measurements for water vapour enhancement factor in nitrogen, argon and hydrogen at selected temperatures and pressures, in the frost-point temperature range between -90 °C and -30 °C and pressure range from 0.1 MPa to above 1 MPa.  CNAM, CETIAT, CMI, VSL, UL, INTA, CEM, UVa will review the report and provide feedback.	<b>CNAM</b> , CETIAT, <b>CMI</b> , VSL, UL, INTA, CEM, UVa	<b>06/2022</b> – potential start of enhancement factor measurement (N <sub>2</sub> )  ... Ar

## A2.2.2: Enhancement factors of nitrogen and argon

Perform measurements of the enhancement factors of water vapour in different carrier gasses.

**Target frost point range:** 500 ppb / -80 °C , 1 ppm / -75 °C, 10 ppm / -60 °C , 127 ppm / -40 °C, 376 ppm / -30 °C

**Pressure range:** 0.1, 0.2, 0.5, 0.7, 0.9, and 1 MPa.

**Carrier gas:** N<sub>2</sub> and Ar.

In progress:

- 1) Training of scientist
- 2) Testing and revalidation of the two-pressure system
- 2) Validation of system with the enhancement factors for air:  
CMH pressure at saturator pressure or atmospheric pressure



current setup



A2.2.3 M18	CEM, will produce cylinders containing static, pressurised humid gas reference mixtures in matrices of nitrogen, argon and hydrogen with amount fractions of water vapour down to 1 $\mu\text{mol mol}^{-1}$ . The target standard uncertainty for the cylinders is 3 % of value. The gas cylinders will be used by UVa and will further develop and upgrade its microwave-based frost point hygrometer.	CEM, UVa
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UVa and CEM agreed to start with H<sub>2</sub>O in matrix N<sub>2</sub> mixture with concentration level of 500  $\mu\text{mol}\cdot\text{mol}^{-1}$

CEM: Expected date first mixture ready by May 2022

<b>A2.2.4 M24</b>	<p>UVa and INTA, using the upgraded saturation-based generator from A2.1.2 and upgraded microwave-based frost point hygrometer in A2.2.3, will perform measurements of the enhancement of water vapour in nitrogen, argon and hydrogen in the frost-point temperature range between -75 °C and -30 °C at selected pressures from 0.1 MPa to above 1 MPa.</p> <p>These measurements will evaluate the non-ideality of gas mixtures (i.e., enhancement factor) with trace amount of water by comparing humid gas mixtures generated by trace humidity standards (saturator-based generators) with corresponding humidity quantities as measured by amount-of-substance fraction analyser.</p>	<b>UVa, INTA</b>
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- UVa has completed a set of water vapour in nitrogen measurements using the existing microwave cylindrical resonator, and the humidity generator and reference hygrometer loaned by INTA. The aim of those measurements is to tune the systems and their coupling, upgrade the thermostat and control systems, and develop new data acquisition and modelling software.
- UVa designed and ordered the construction of a 5 cm diameter golden-plated quasi-spherical microwave resonator (QSMWR), 15 µm gold thickness. The new hygrometer is held in TermoCal UVa's premises.
- The new QSMWR has been tested. Antennas have been tuned offering high quality resonant modes. Software has been upgraded and fully setup.
- The construction of a new thermostat for the new QSMWR is almost completed.
- INTA has recalibrated the temperature and pressure sensors of the frost-point two pressure-two temperature saturation-based generator, an upgraded THUNDER SCIENTIFIC 4500 model. Calibration of the transfer standard hygrometer to be provided to UVa in M11 has been initiated. Currently INTA is awaiting the repair of the water cooling system for the 4500.
- The integration of the hygrometer, thermostat, QSMWR, control and software is being undertaken as per planned schedule.





# PROMETH<sub>2</sub>O

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## Thank you for your attention

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**EMPIR**



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