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Metrology for trace water in ultra-pure process gases

Lead DTU

WP1 overview and planning

14th of June 2021

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

WP1: Improved trace water measurement methods and techniques (DTU, SUN, Qrometric, MBW, TUBITAK, INRIM, PTB)

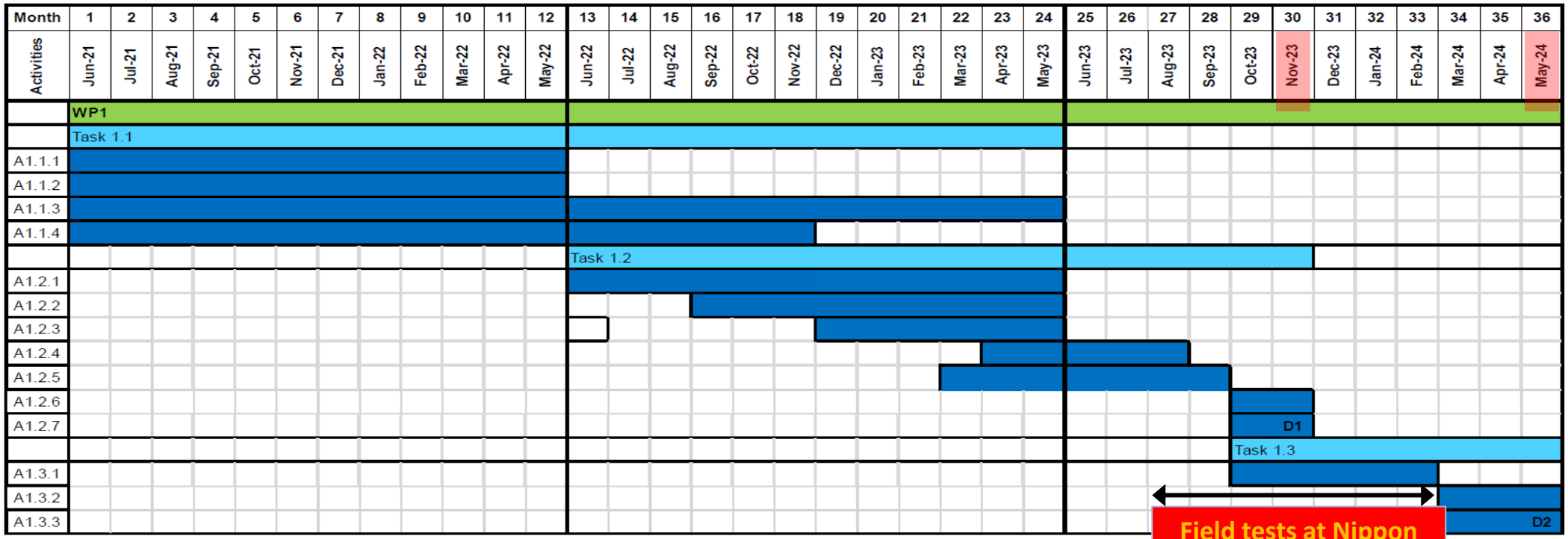
Task 1.1 Development and improvement of optical analyzers (DTU, SUN, Qrometric, TUBITAK)

Task 1.2 Validation of the measurement methods and techniques

Task 1.3 Recommendation of transfer standard for a future CIPM comparison

D1(M30, DTU): Report and recommendations on measurement methods and techniques for trace water measurements in industrial environments

D2(M36, INRIM): Report on the "Recommendation of transfer standards for a future CIPM comparison…"



Task 1.1: Development and improvement of optical analyzers

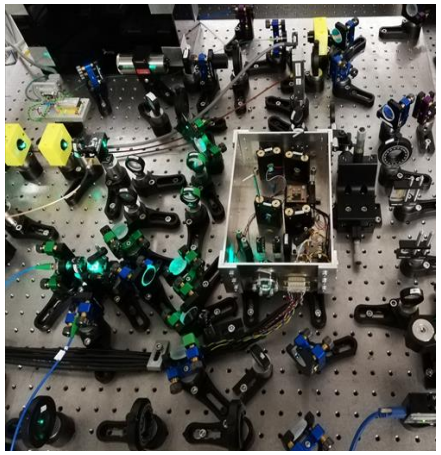
A1.1.1 Development of a compact NIR CC-FS-CRDS spectrometer

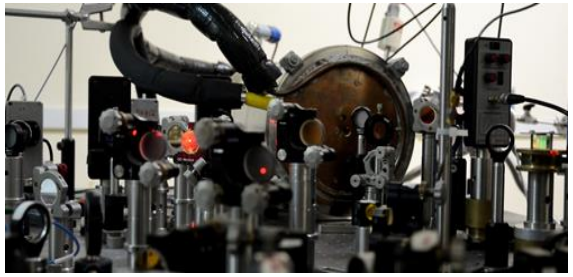
AMP Group – Atoms, Molecules & Precision measurements

Dr. Livio Gianfrani and Co.

Department of Mathematics and Physics Università degli Studi della Campania "Luigi Vanvitelli"

Università degli Studi della Campania *Luigi Vanvitelli*





Task 1.1: Development and improvement of optical analyzers

A1.1.1 Development of a compact NIR CC-FS-CRDS spectrometer

Development of a compact (and transportable) FS-CRDS spectrometer, referenced to an OFCS, for **traceable** measurements of water vapor in H_2 and N_2, from 5 ppm down to 5 ppb.

Upgrades of the existing spectrometer

i. replace the cavity mirrors so as to increase the empty-cavity decay time up to 100 μs

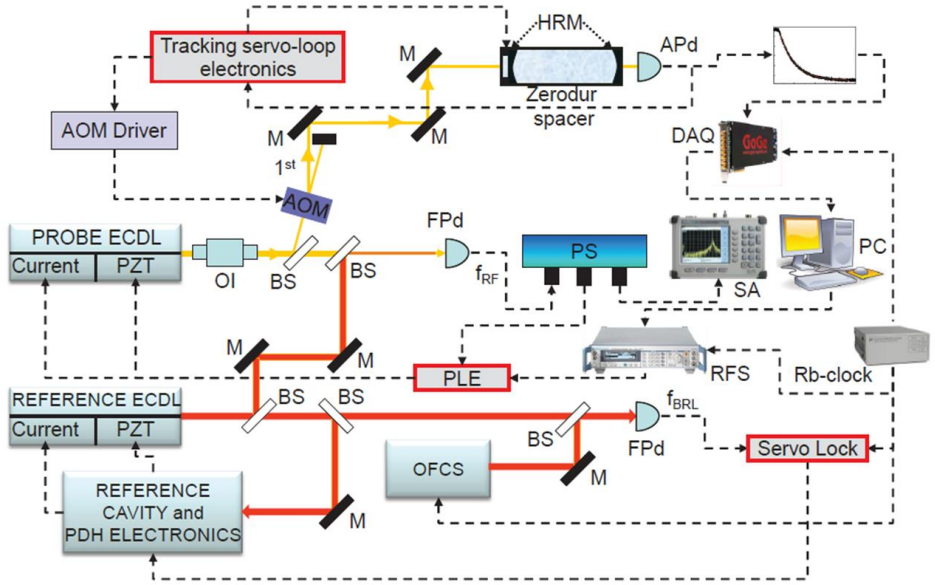
ii. Increase the laser power at 1.39 μm

iii. optimize the electronics so as to improve the measurement precision

iv. make the spectrometer more compact and transportable

v. simplify the optical scheme for comb-referencing

Enhancement of the detection sensitivity to meet Objective 1



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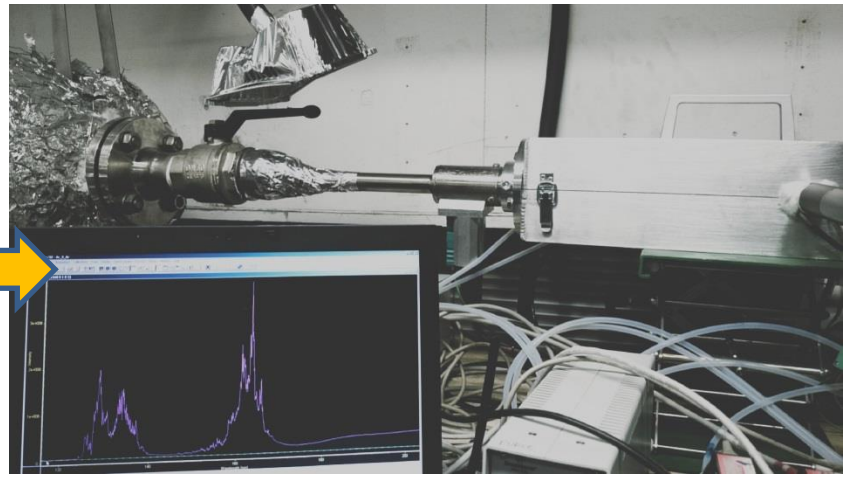
Task 1.1: Development and improvement of optical analyzers

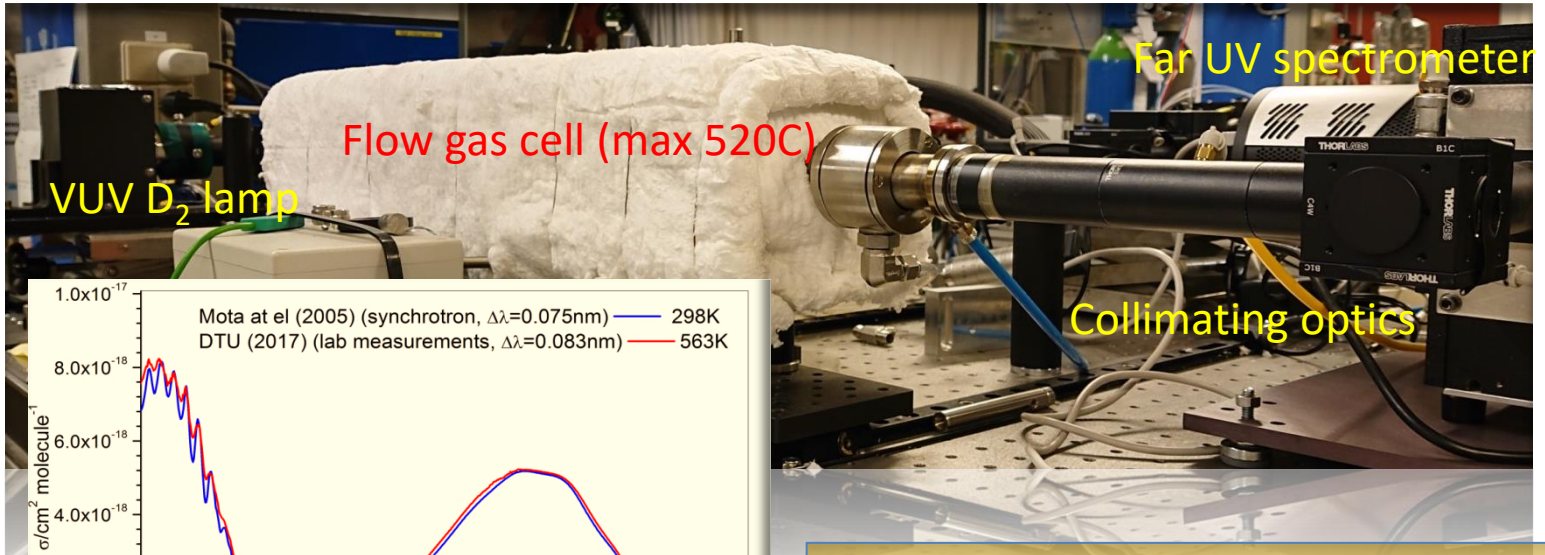
A1.1.2 Development of a compact and transportable far-UV system

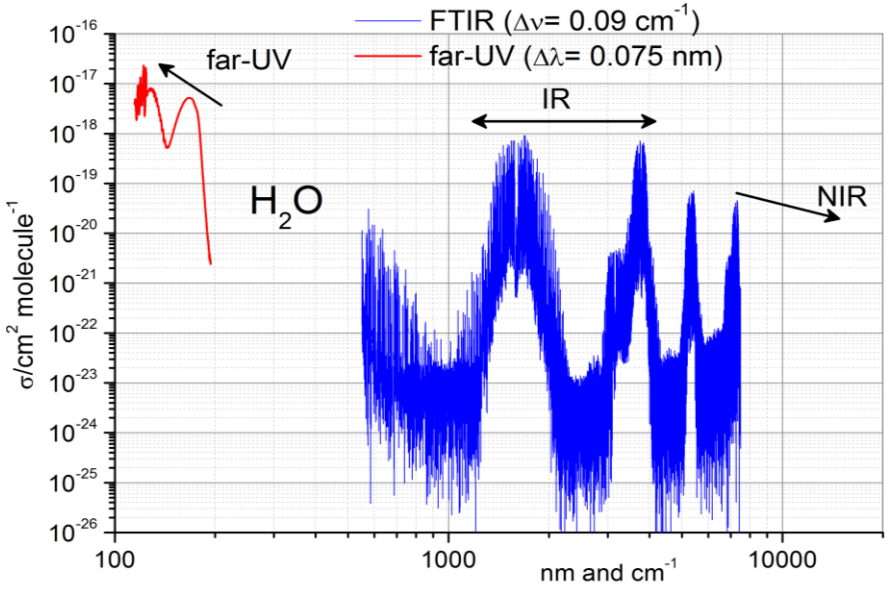
Far-UV on-line measurements in gasification gas at about 300°C



Task 1.1: Development and improvement of optical analyzers

A1.1.2 Development of a compact and transportable Far-UV system





Beyond state of art:

- Far-UV spectroscopy in 120-200 nm for laboratory and field measurements
- Compact Far-UV spectrometer: 153 nm < λ – is sufficient
- No need for high spectral resolution
- Absorption cross-sections higher in far-UV
- O_2, CO_2 can be measured at the same time

Example: comparison of the far-UV H_2O absorption cross-sections at 298 K (synchrotron) and 563 K (laboratory)

Task 1.1: Development and improvement of optical analyzers

A1.1.3 FTIR-based system sensitivity improvement

THERMODYNAMIC METROLOGY LABORATORY

Dr. Hümbet NASİBLİ
Dr. Seda OĞUZ AYTEKİN

TÜBİTAK UME

Task 1.1: Development and improvement of optical analyzers

A1.1.3 FTIR-based system sensitivity improvement

TUBUTAK will

- ✓ upgrade the existing high-resolution FTIR system by adding a new multi-pass gas cell and related interfaces;
- ✓ remove residual water in the optical path as much as possible a water removal system will be established;
- ✓ do trace water vapor measurements in N_2 and Ar from 5 ppm to 50 ppb with standard relative uncertainty between 3 % and 8 % and operation pressure up to 1 MPa are targeted within the scope of this WP.

Task 1.1: Development and improvement of optical analyzers

A1.1.4 Development of CE-FM spectroscopy hygrometer

Qrometric
Industrial and scientific metrology

Dr. Ned Hawes

QROMETRIC – WORLD CLASS METROLOGY

- ✓ Qrometric is a UK metrology company specializing in product and system innovation. Our specialty is the measurement and calibration of temperature and humidity.
- ✓ We have partnered with manufacturers who offer ‘best in class’ equipment that deliver best measurement capability, optimum calibration performance, stability and reliability.

Task 1.1: Development and improvement of optical analyzers

A1.1.4 Development of CE-FM spectroscopy hygrometer

Development of a NIR cavity-enhanced frequency-modulated (CE-FM) laser spectroscopic instrument for measurement of sub ppb water vapour concentration

To meet project objectives:

- Change macro optical components for fibre-coupled versions;
- House in robust transportable case;
- Further development of FPG generator (FPG=portable generator for dew point sensor calibration)
- To lower the range of operation of FPG from -75 to -90°Cfp and perhaps further. (The -75°Cfp version exists as a production prototype now and is close to market readiness)

Qrometric
Industrial and scientific metrology

Please note, at present this development is not public knowledge. Your discretion is appreciated.

Task 1.2: Validation of the measurement methods and techniques

== Start from M13 (June 2022) ==

The aim of this task is to validate improved and new measurement methods and techniques developed in **Task 1.1**:

- NIR CC-FS-CRDS spectrometer (**SUN** ↔ INRIM);
- High-quality CMH (new commercial instrument developed by MBW) (**INRIM** ↔ MBW ↔ Qrometric);
- NIR CE-FM spectroscopy hygrometer (**Qrometric** ↔ INRIM ↔ MBW);
- Far-UV system (**DTU** ↔ Qrometric)
- FTIR system (**TUBUTAK**, in house)

on various humidified gases (e.g. N_2, Ar or H_2).

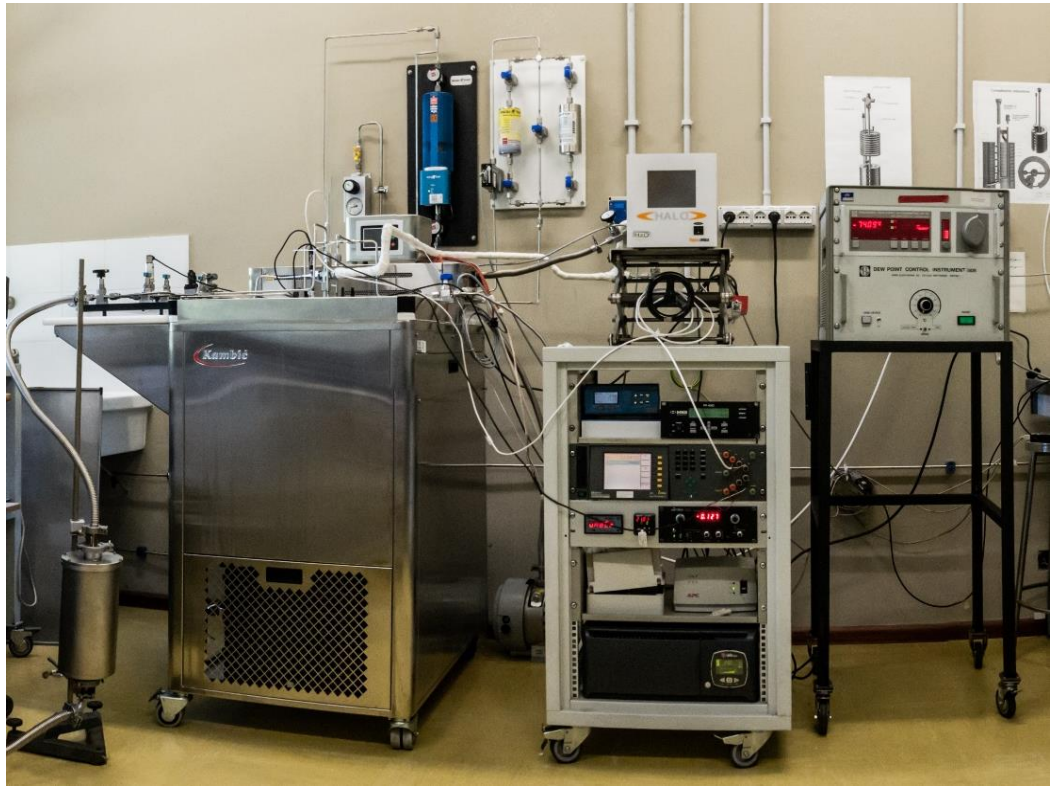
High-level D1 (M30, November 2023): "***Report*** *and recommendations on measurement methods and techniques for trace water measurements in industrial environments in the amount fraction range between 5 parts in 106 (5 ppm) and 5 parts in 109 (5 ppb) (-65 °C and -105 °C frost point) with relative standard uncertainty between 3 % and 8 %, from upper to lower range, respectively"*

Task 1.2: Validation of the measurement methods and techniques

== Start from M13 (June 2022) ==

With its thermodynamic saturation-based primary standard generator (WP2), **INRIM** will support the validation of the following instruments for trace water measurement based on different techniques.

- ✓ CC-FS-CRDS spectrometer developed by SUN;
- ✓ CMH for ultra-low frost-point measurements by MBW;
- ✓ Cavity-enhanced frequency modulated (CE-FM) spectroscopy hygrometer developed by Qrometric.



Task 1.3: Recommendation of transfer standard(s) for future CIPM comparison in the trace water range -65°C to -105°C (5ppm to 5ppb)

== Start from M29 (October 2023) (INRIM, PTB, TUBITAK, DTU) ==

The aim of this task is to summarize the results of the **Tasks 1.2** in a recommendation report containing a recommendation of transfer standards in the frost-point temperature range -65 °C to -105 °C (5 ppm to 5 ppb) addressed to a key project stakeholder, the CIPM, to help shaping a future comparison in trace water measurements.

High-level D2 (M36, May 2024: "***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)"*

Beyond WP1: Field tests at Nippon Gases (WP3, Task 3.3)

== Start from M27 (August 2023) ==

- High-quality CMH (MBW) and Far-UV system (DTU) will be used in Test Bed 2 (Nippon Gases, IT) in selected gas matrices at pressures up to 1 MPa

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Thank you

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