

Humidity measurements at LNE-CETIAT low humidity level

Eric GEORGIN – eric.georgin@cetiat.fr

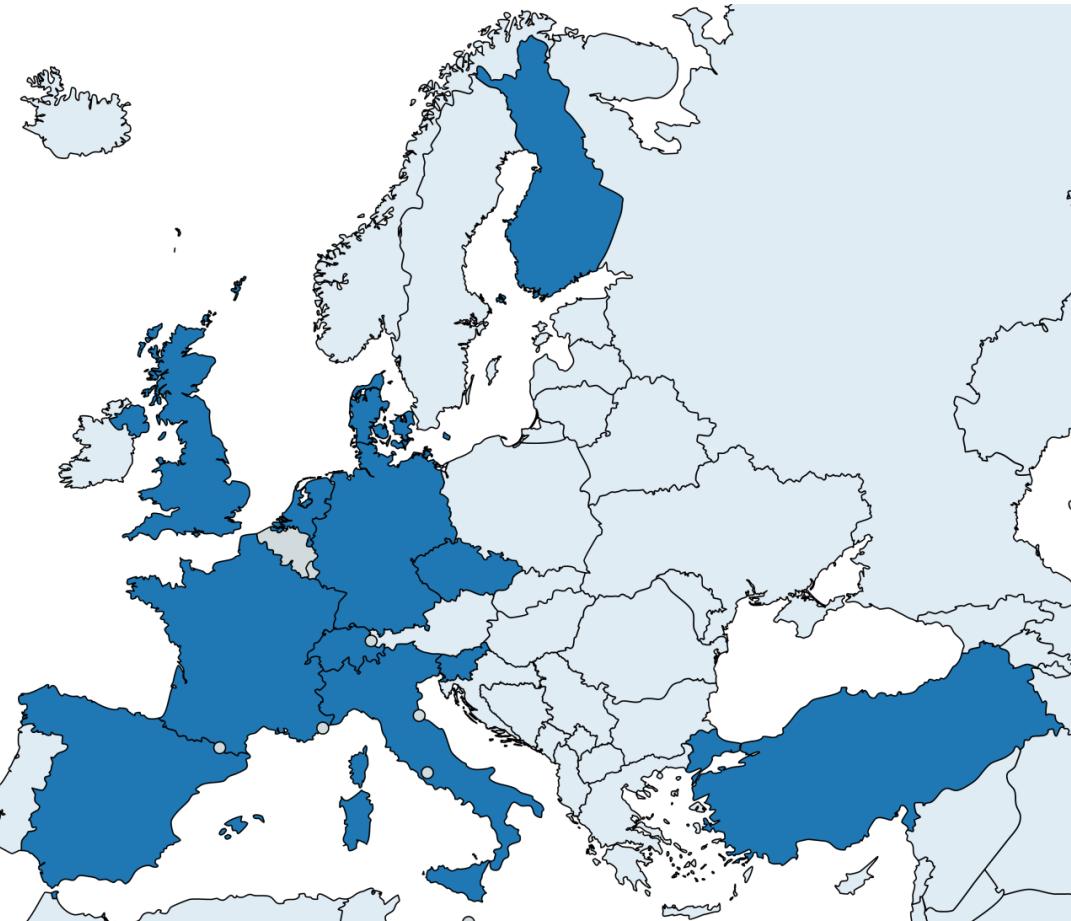


The EMPIR initiative is co-funded by the European Union's Horizon 2020 research and innovation programme and the EMPIR Participating States

Outline

1. JRP PROMETH₂O
2. Humidity at LNE-CETIAT
3. Humid Air Generator – HAG / dilution
4. Results
5. Conclusion

JRP PROMETH2O – the consortium



19 partners from 12 countries – 242 person-months

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



Univerza v Ljubljani



JRP PROMETH2O - Objectives

- New primary standards Ar and H₂ down to 5 K (at temperature) at pressure
- New/improved measurement of oxygen fraction range between 10% and 90% (rel. uncert. 3 % to 8 %)
- New data and correlation enhancement in N₂, O₂ and CO₂ range from -30 °C to +100 °C
- Demonstration at selected sites of real-time measurement
- Provision of a toolkit for robust measurement of UHP process gases, based on standards and measurement

More information

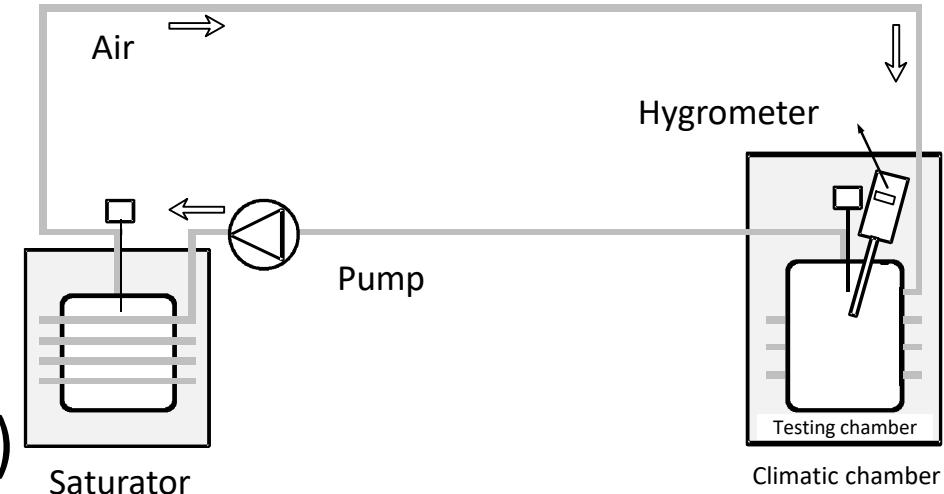
**Session 16 Thermometry, Hygrometry
V. FERNICOLA**

Website
<https://www.prometh2o.eu/>



Humidity at LNE-CETIAT (1/2)

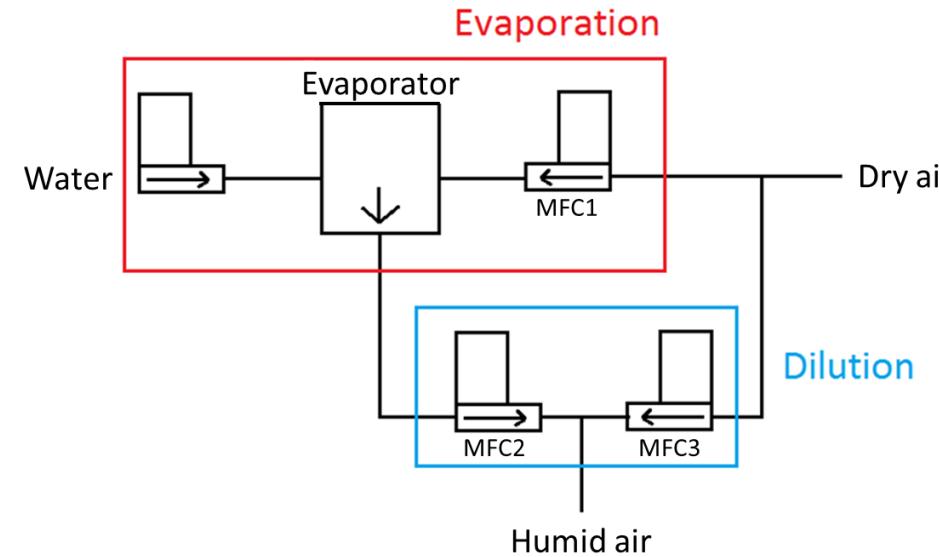
- Designated institute for the French national reference in humidity.
 - Ensures SI traceability for end users
 - \approx 600 COFRAC's calibrations per year
 - Primary realization: Humid Air Generator (HAG)
 - 1T1P / closed loop
 - Dew/frost point
 - Relative humidity



Humidity at LNE-CETIAT (2/2)

■ Secondary realization

- Mixing ratio based on dilution principle



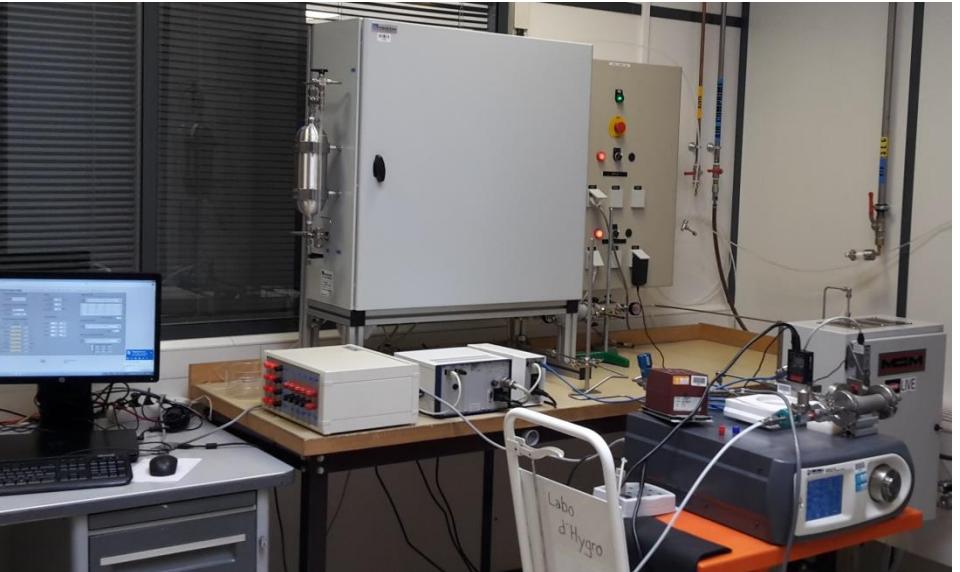
$$r_{evap} = \frac{m_{water}}{m_{MFC\ 1}} = \frac{\dot{m}_{water}}{\dot{m}_{MFC\ 1}}$$

$$r_{dilu} = \frac{\dot{m}_{MFC\ 2} \cdot r_{evap}}{\dot{m}_{MFC\ 2} + \dot{m}_{MFC\ 3}}$$

from r_{dilu} to $\theta_{d/f}$

$$x_v = r_{dilution} \cdot \frac{M_{dry\ gas}}{M_{water}}$$

$$e' = x_v \cdot p$$



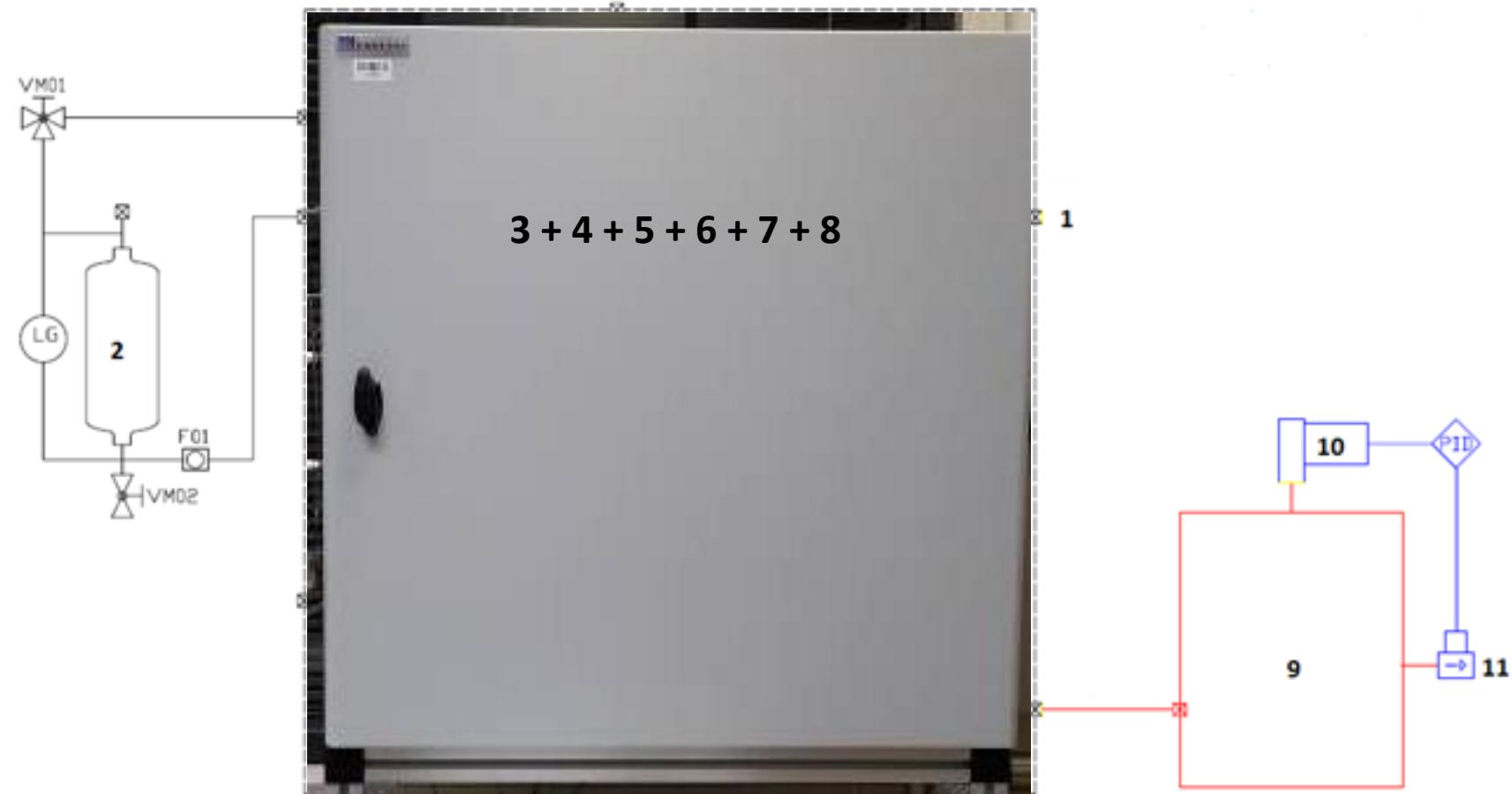
$$e = \frac{e'}{f(p, \theta_{d/f})}$$

$\theta_{d/f}$ D. Sonntag (EIT-90)

HAG / dilution (1/6)

■ Schematic overview

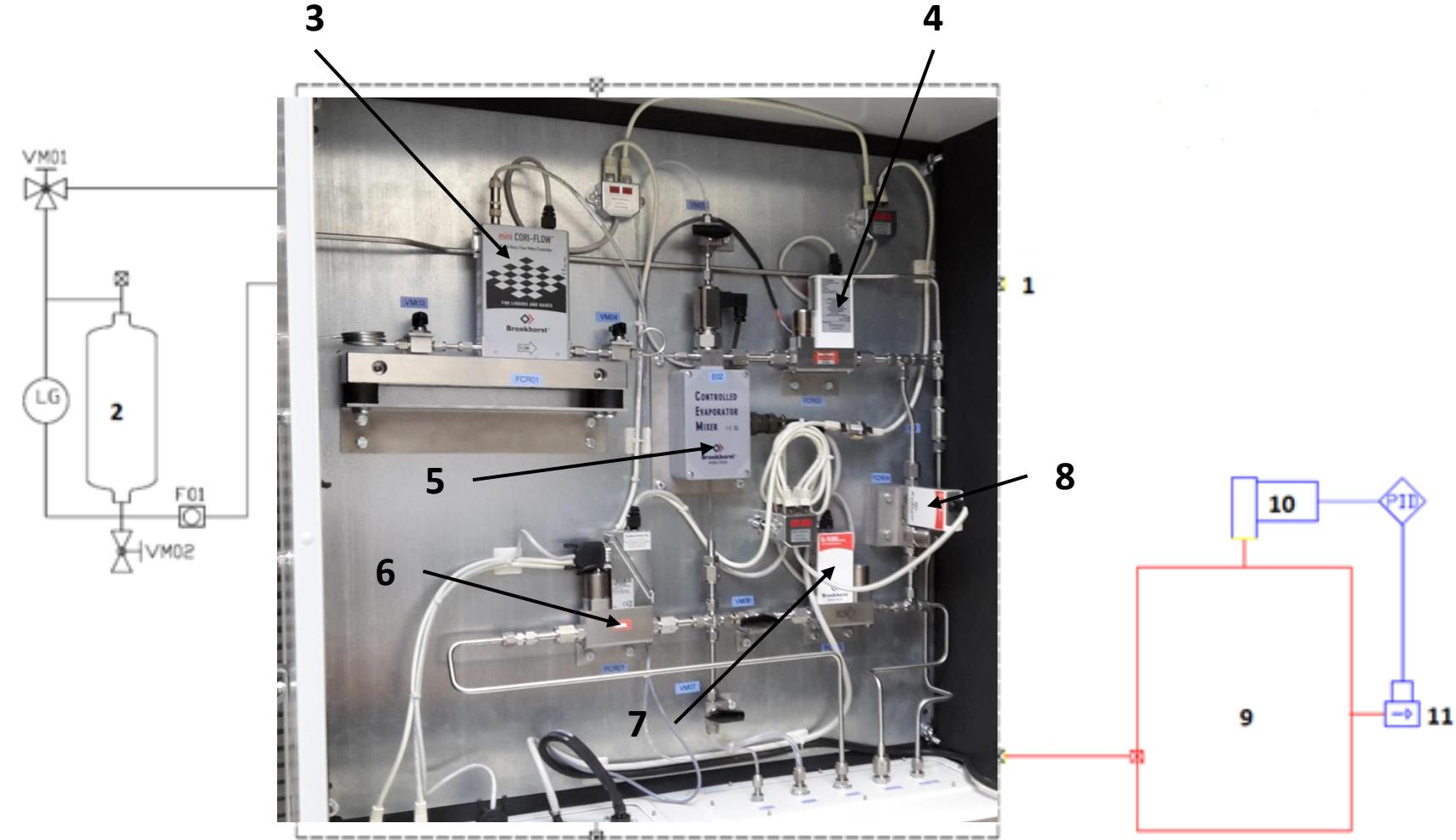
- 1) Clean Dry Air (CDA) inlet,
- 2) pure water reservoir,
- 3) compact Coriolis mass flow controllers for liquids,
- 4) mass flow controllers for gases,
- 5) evaporator,
- 6) pressure controller,
- 7) mass flow controllers for gases,
- 8) mass flow controllers for gases,
- 9) testing chamber or device under test,
- 10) pressure controller,
- 11) outlet



HAG / dilution (1/6)

■ Schematic overview

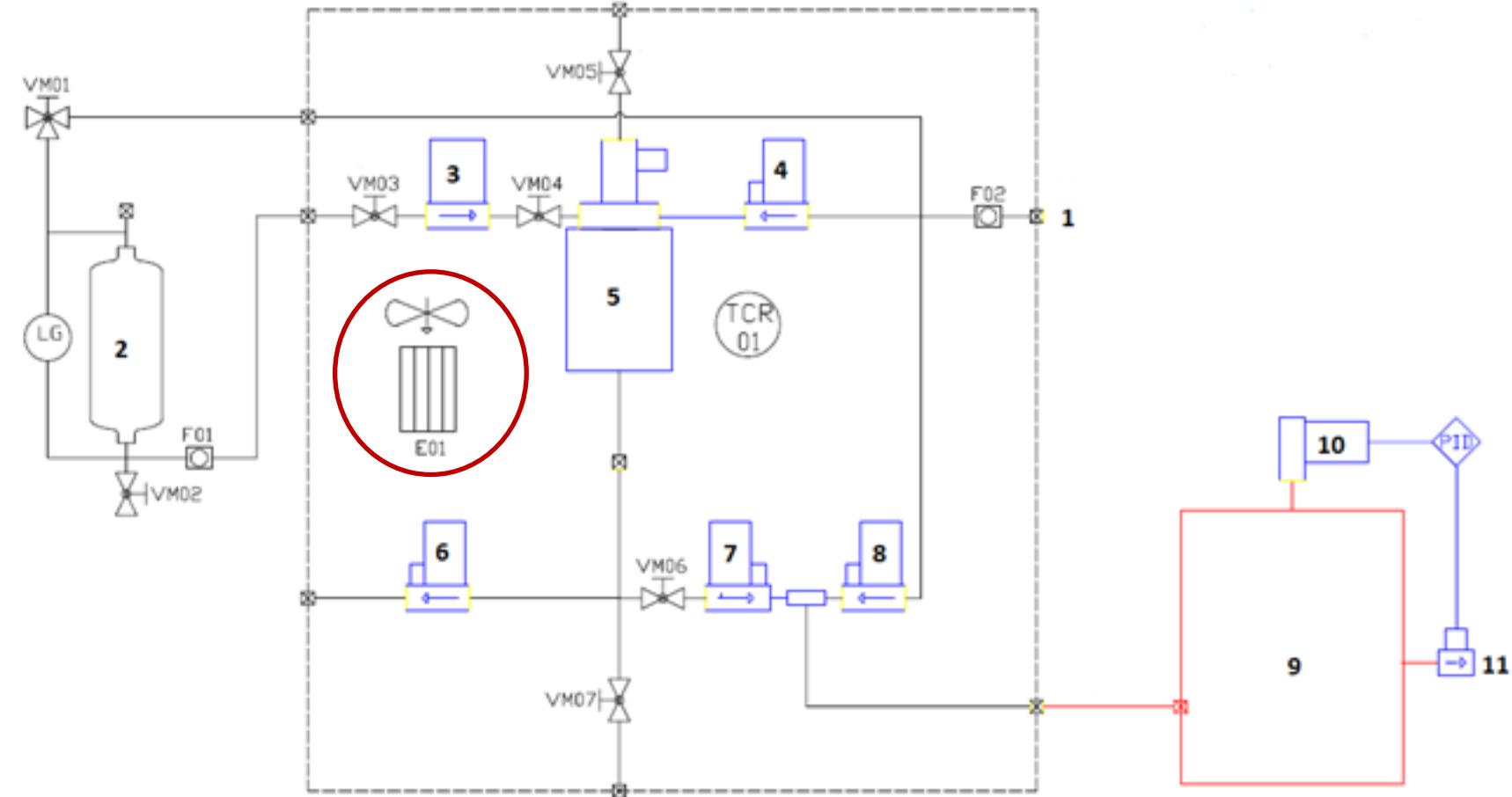
- 1) Clean Dry Air (CDA) inlet,
- 2) pure water reservoir,
- 3) compact Coriolis mass flow controllers for liquids,
- 4) mass flow controllers for gases,
- 5) evaporator,
- 6) pressure controller,
- 7) mass flow controllers for gases,
- 8) mass flow controllers for gases,
- 9) testing chamber or device under test,
- 10) pressure controller,
- 11) outlet



HAG / dilution (1/6)

■ Schematic overview

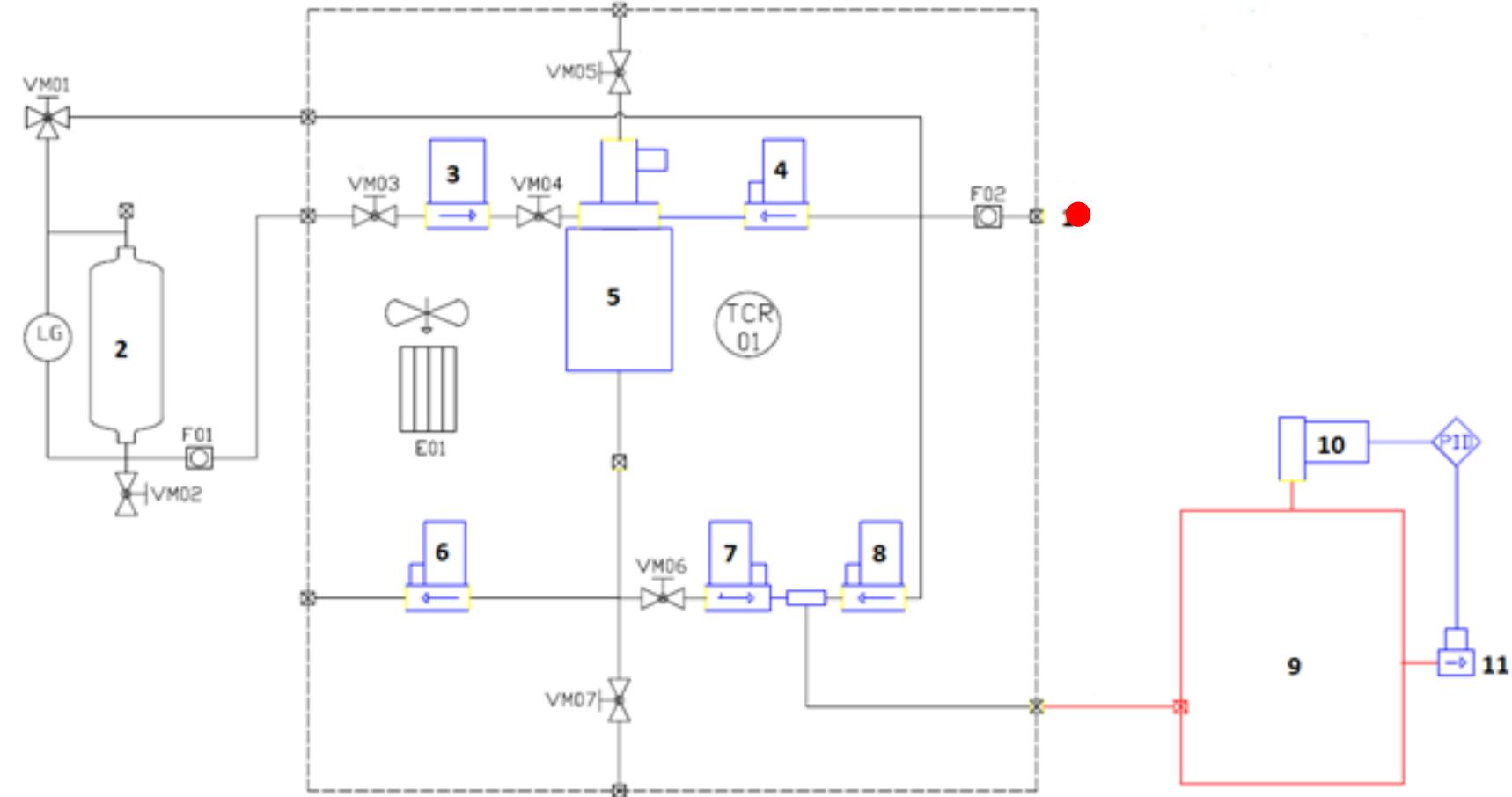
- 1) Clean Dry Air (CDA) inlet,
- 2) pure water reservoir,
- 3) compact Coriolis mass flow controllers for liquids,
- 4) mass flow controllers for gases,
- 5) evaporator,
- 6) pressure controller,
- 7) mass flow controllers for gases,
- 8) mass flow controllers for gases,
- 9) testing chamber or device under test,
- 10) pressure controller,
- 11) outlet



HAG / dilution (1/6)

■ Schematic overview

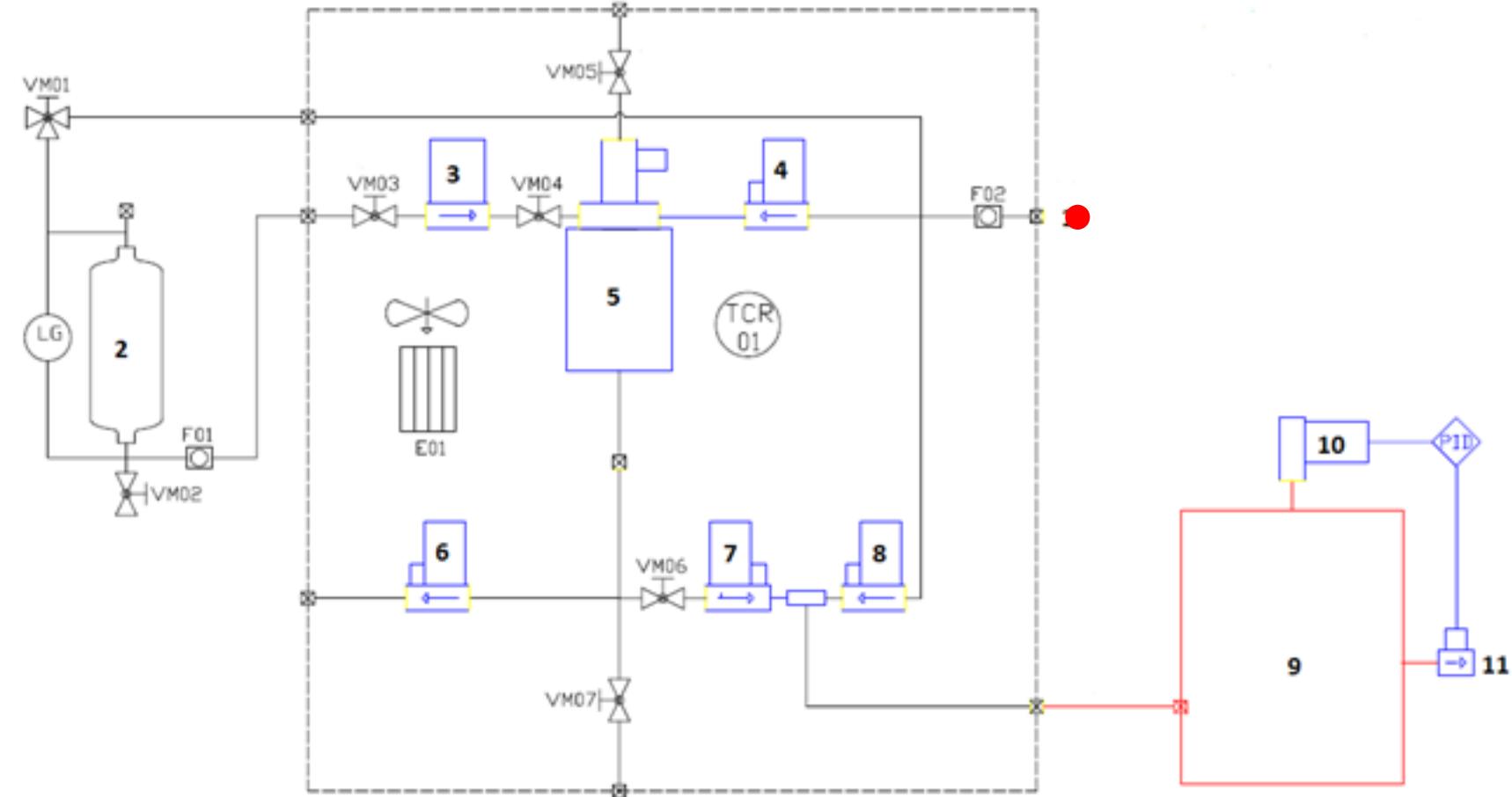
- 1) Clean Dry Air (CDA) inlet,
- 2) pure water reservoir,
- 3) compact Coriolis mass flow controllers for liquids,
- 4) mass flow controllers for gases,
- 5) evaporator,
- 6) pressure controller,
- 7) mass flow controllers for gases,
- 8) mass flow controllers for gases,
- 9) testing chamber or device under test,
- 10) pressure controller,
- 11) outlet



HAG / dilution (1/6)

■ Schematic overview

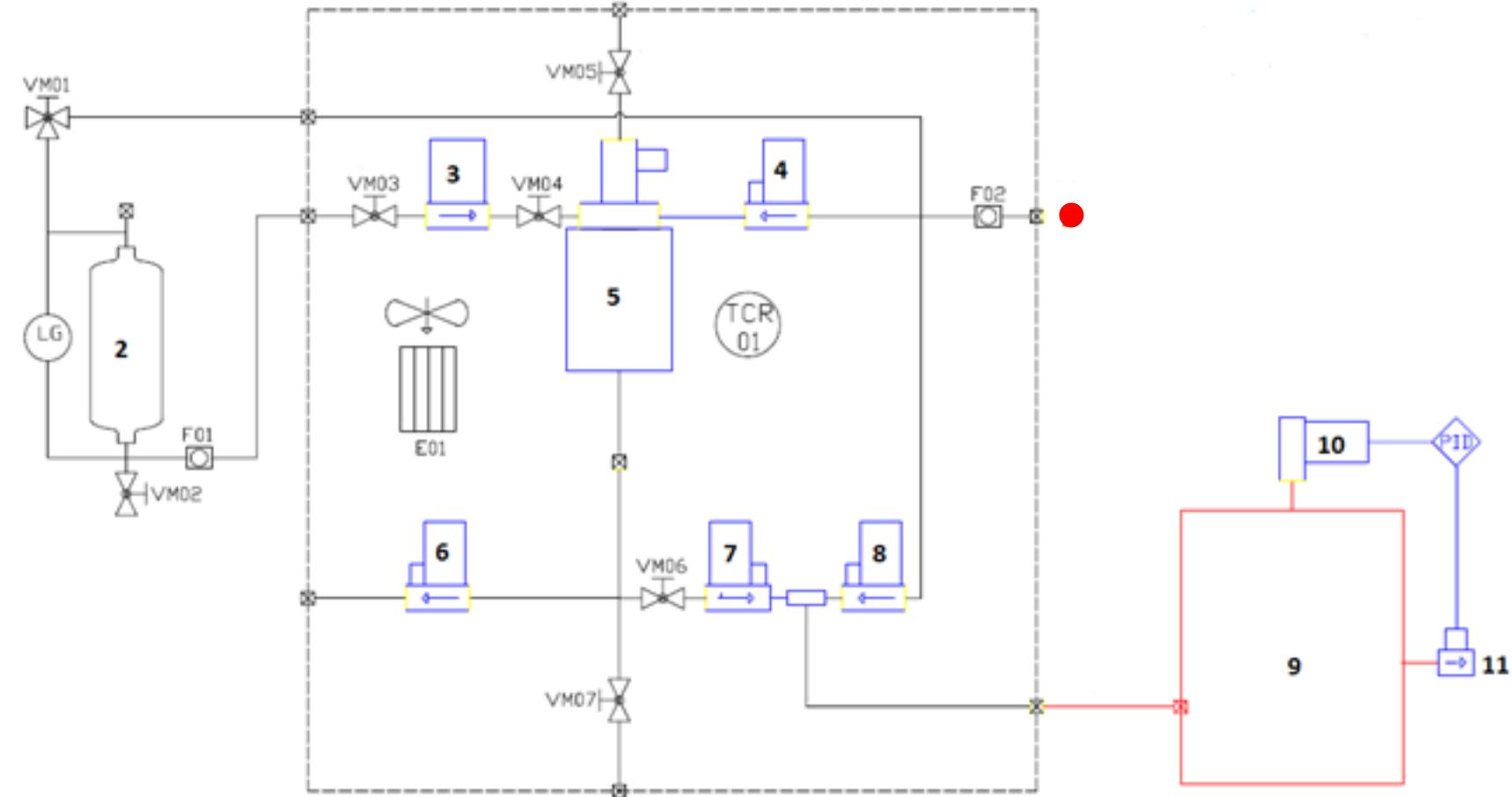
- 1) Clean Dry Air (CDA) inlet,
- 2) pure water reservoir,
- 3) compact Coriolis mass flow controllers for liquids,
- 4) mass flow controllers for gases,
- 5) evaporator,
- 6) pressure controller,
- 7) mass flow controllers for gases,
- 8) mass flow controllers for gases,
- 9) testing chamber or device under test,
- 10) pressure controller,
- 11) outlet



HAG / dilution (1/6)

■ Schematic overview

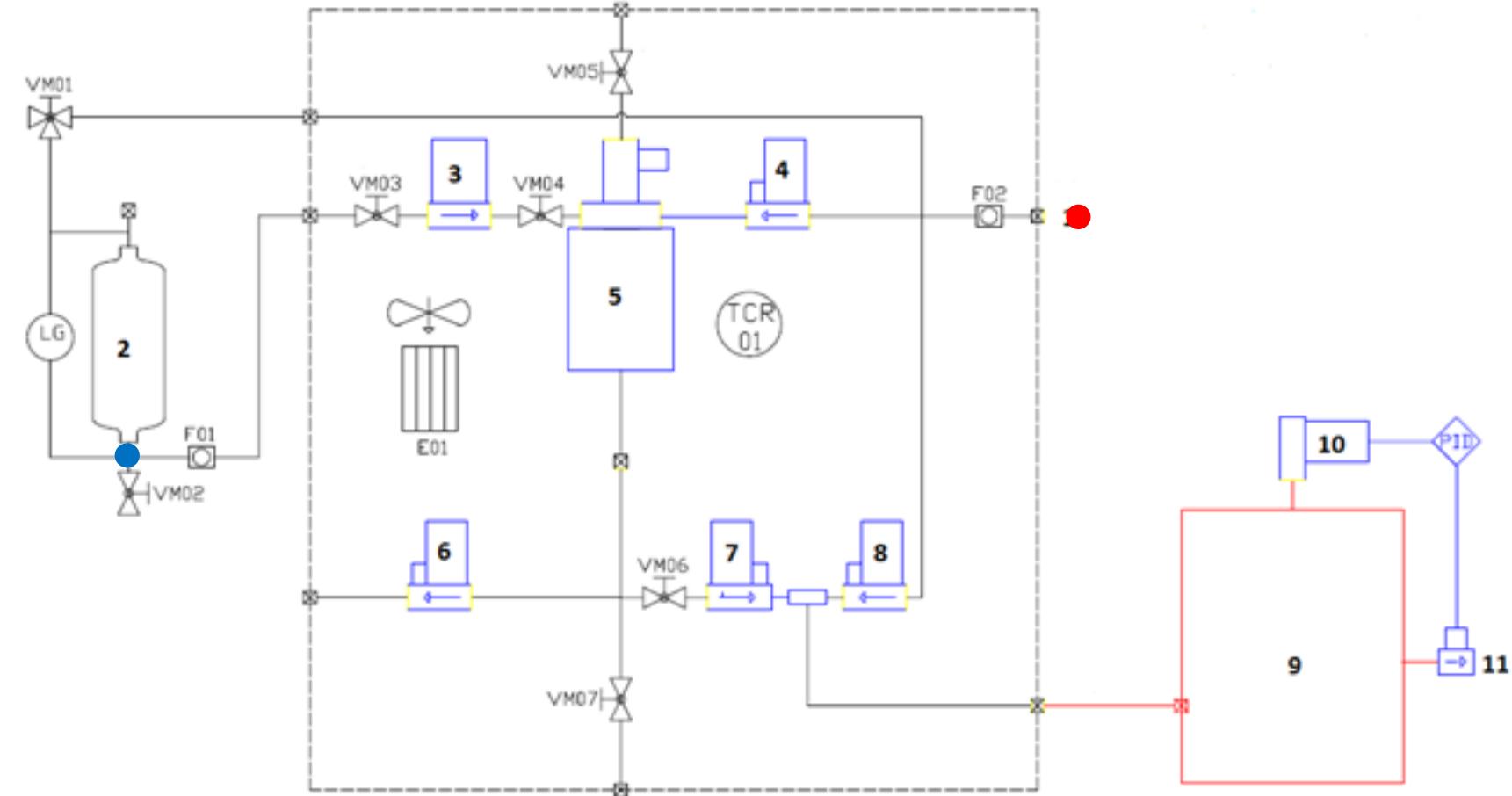
- 1) Clean Dry Air (CDA) inlet,
- 2) pure water reservoir,
- 3) compact Coriolis mass flow controllers for liquids,
- 4) mass flow controllers for gases,
- 5) evaporator,
- 6) pressure controller,
- 7) mass flow controllers for gases,
- 8) mass flow controllers for gases,
- 9) testing chamber or device under test,
- 10) pressure controller,
- 11) outlet



HAG / dilution(1/6)

■ Schematic overview

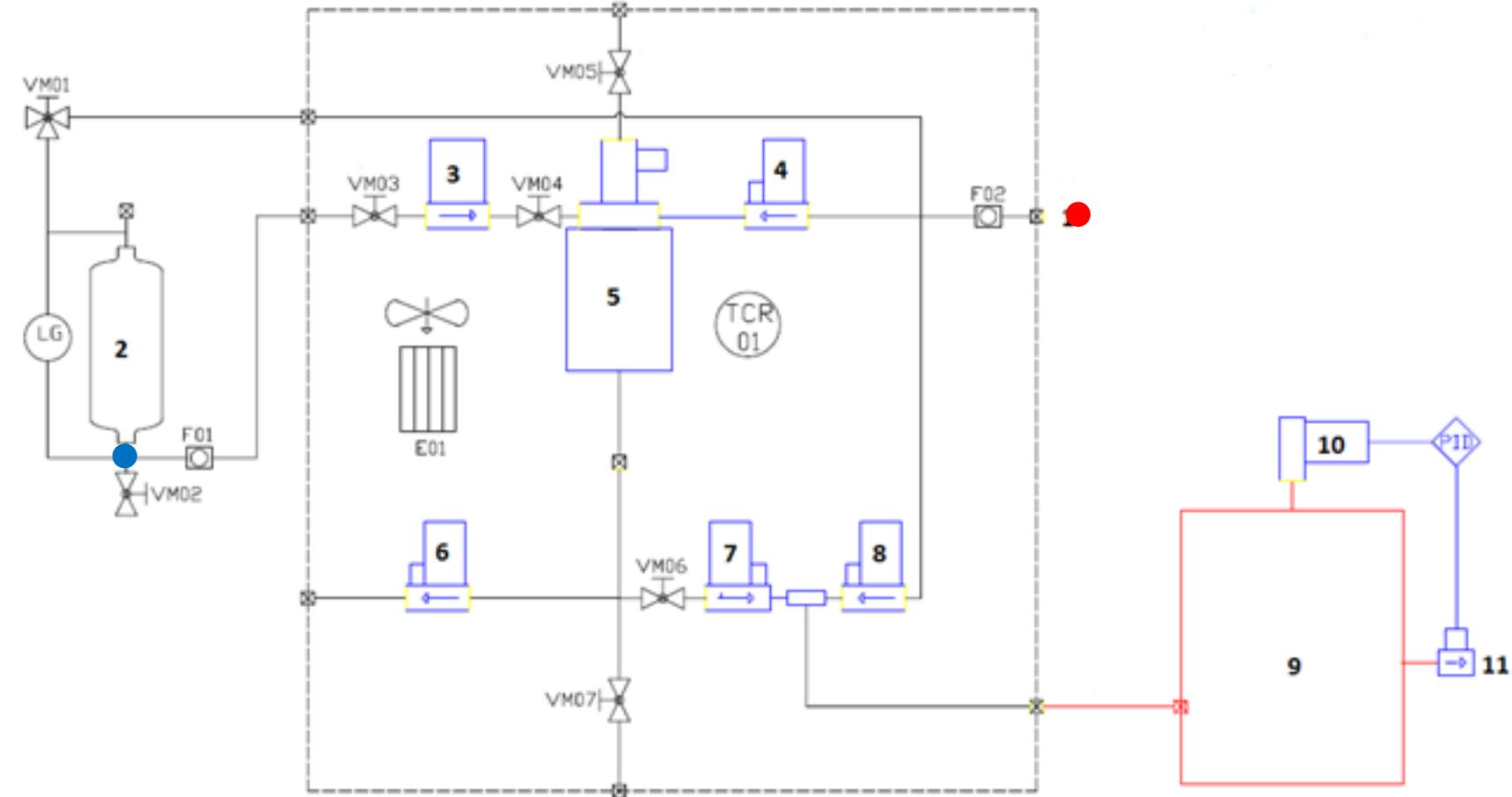
- 1) Clean Dry Air (CDA) inlet,
- 2) pure water reservoir,
- 3) compact Coriolis mass flow controllers for liquids,
- 4) mass flow controllers for gases,
- 5) evaporator,
- 6) pressure controller,
- 7) mass flow controllers for gases,
- 8) mass flow controllers for gases,
- 9) testing chamber or device under test,
- 10) pressure controller,
- 11) outlet



HAG / dilution (1/6)

■ Schematic overview

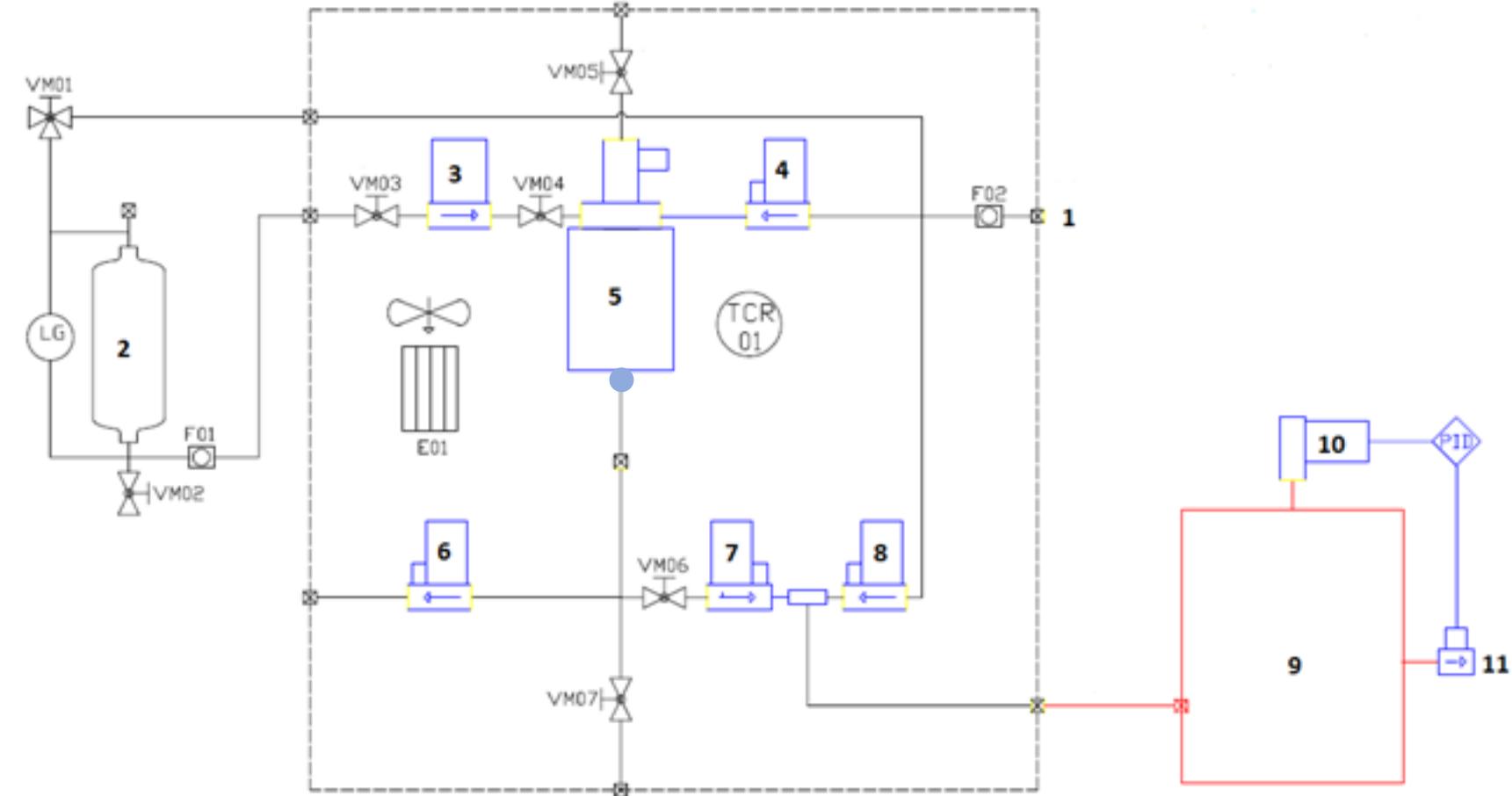
- 1) Clean Dry Air (CDA) inlet,
- 2) pure water reservoir,
- 3) compact Coriolis mass flow controllers for liquids,
- 4) mass flow controllers for gases,
- 5) evaporator,
- 6) pressure controller,
- 7) mass flow controllers for gases,
- 8) mass flow controllers for gases,
- 9) testing chamber or device under test,
- 10) pressure controller,
- 11) outlet



HAG / dilution (1/6)

■ Schematic overview

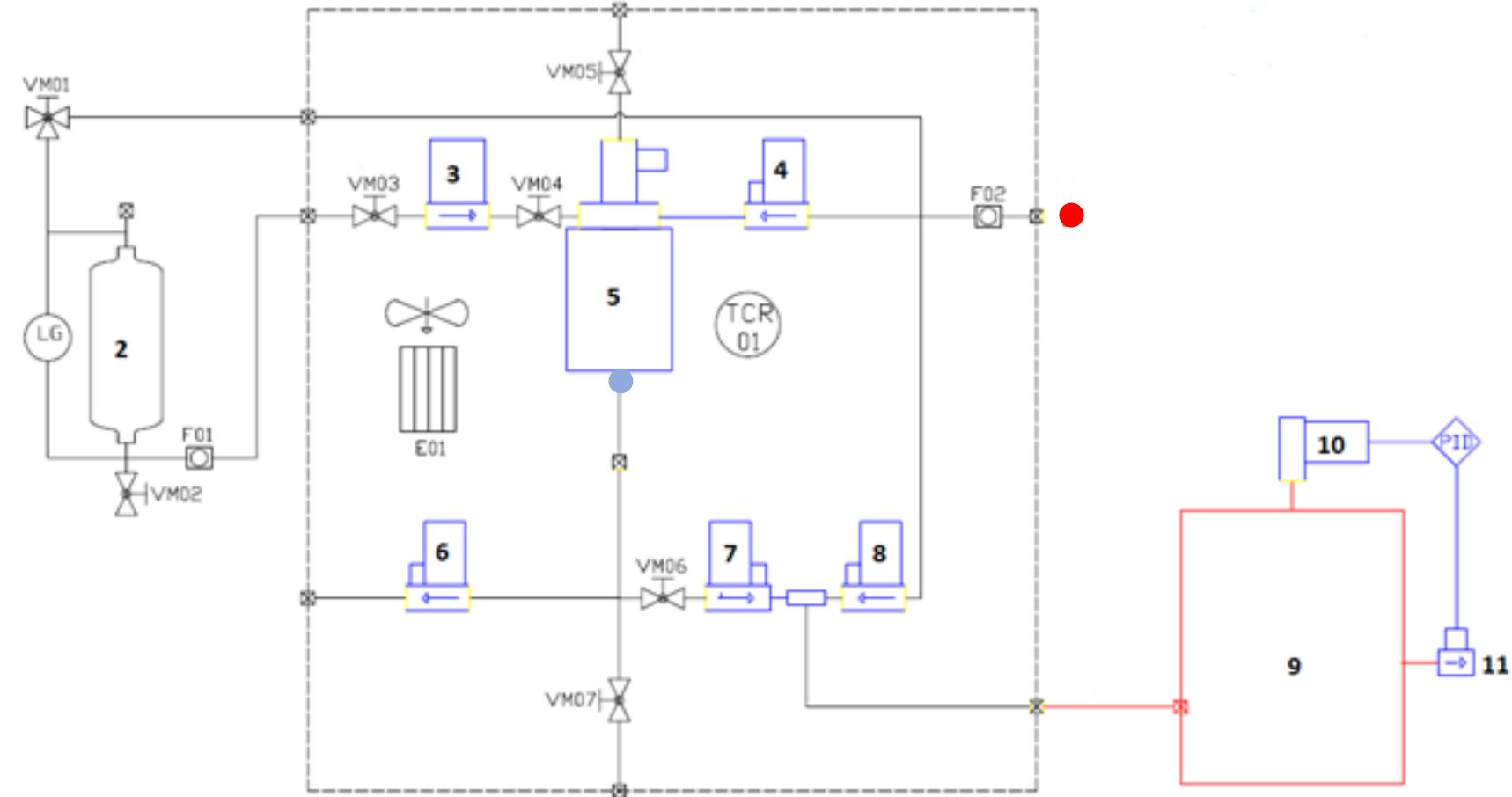
- 1) Clean Dry Air (CDA) inlet,
- 2) pure water reservoir,
- 3) compact Coriolis mass flow controllers for liquids,
- 4) mass flow controllers for gases,
- 5) evaporator,
- 6) pressure controller,
- 7) mass flow controllers for gases,
- 8) mass flow controllers for gases,
- 9) testing chamber or device under test,
- 10) pressure controller,
- 11) outlet



HAG / dilution (1/6)

■ Schematic overview

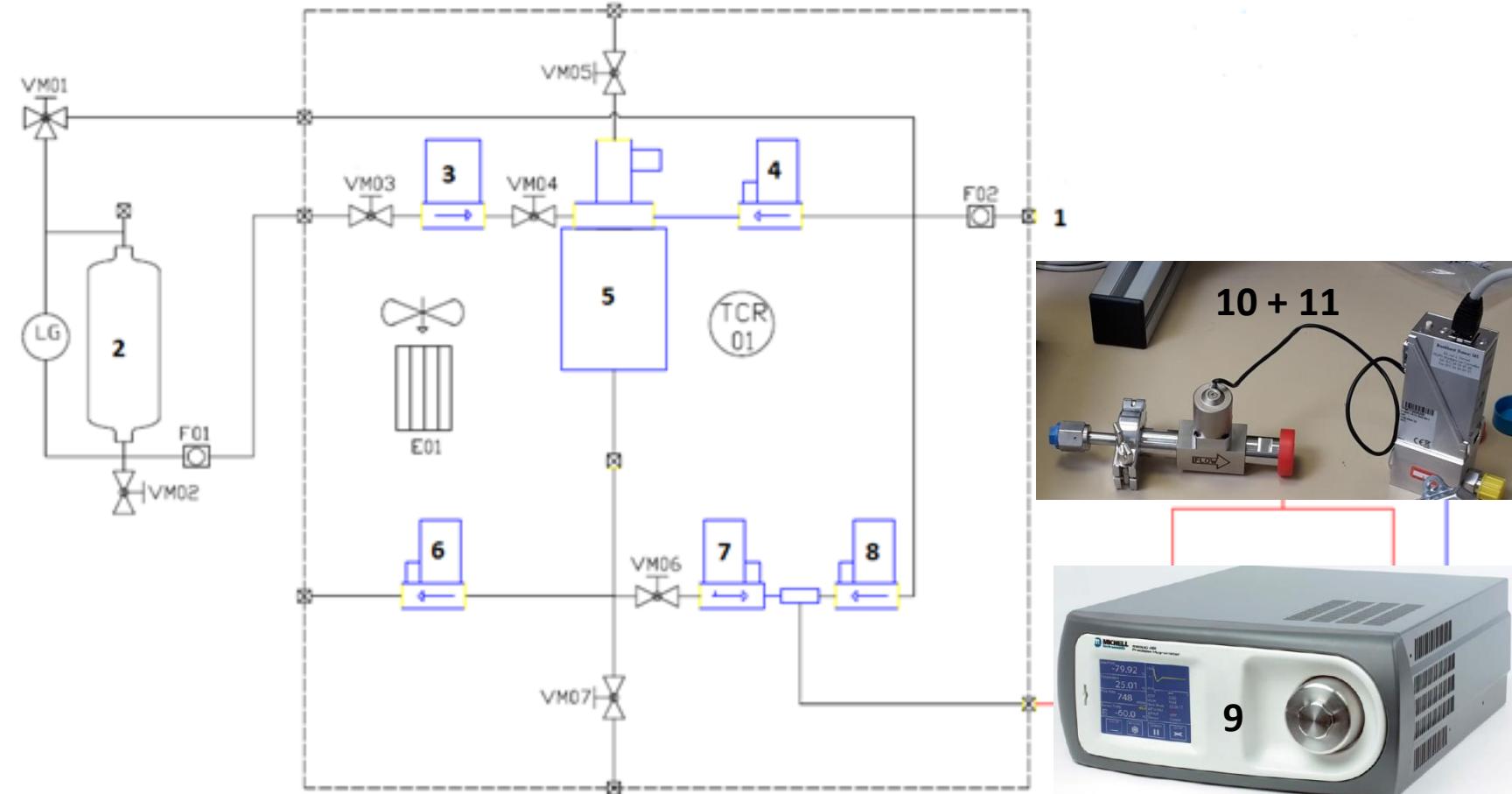
- 1) Clean Dry Air (CDA) inlet,
- 2) pure water reservoir,
- 3) compact Coriolis mass flow controllers for liquids,
- 4) mass flow controllers for gases,
- 5) evaporator,
- 6) pressure controller,
- 7) mass flow controllers for gases,
- 8) mass flow controllers for gases,
- 9) testing chamber or device under test,
- 10) pressure controller,
- 11) outlet



HAG / dilution (1/6)

■ Schematic overview

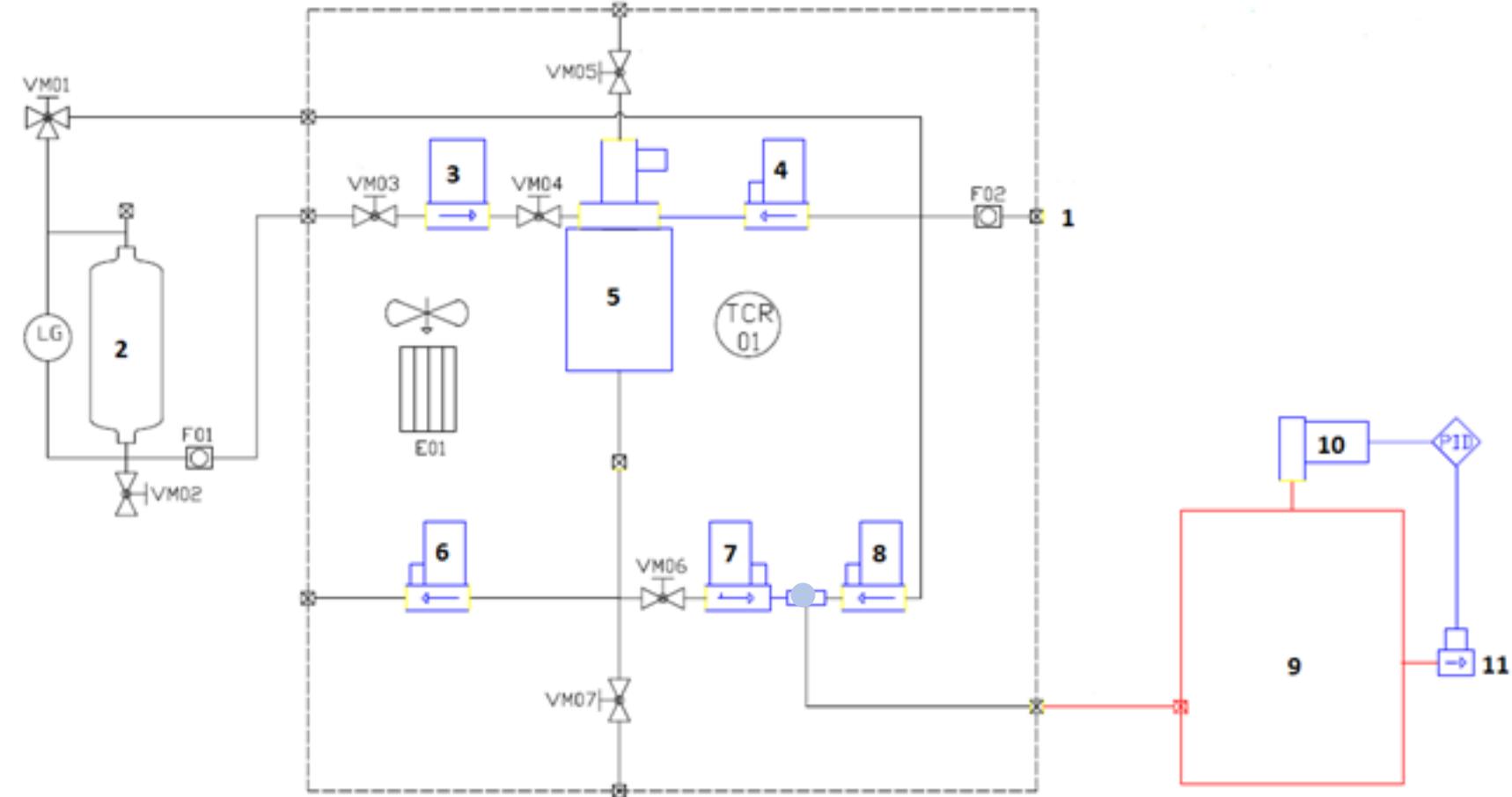
- 1) Clean Dry Air (CDA) inlet,
- 2) pure water reservoir,
- 3) compact Coriolis mass flow controllers for liquids,
- 4) mass flow controllers for gases,
- 5) evaporator,
- 6) pressure controller,
- 7) mass flow controllers for gases,
- 8) mass flow controllers for gases,
- 9) testing chamber or device under test,
- 10) pressure controller,
- 11) outlet



HAG / dilution (1/6)

■ Schematic overview

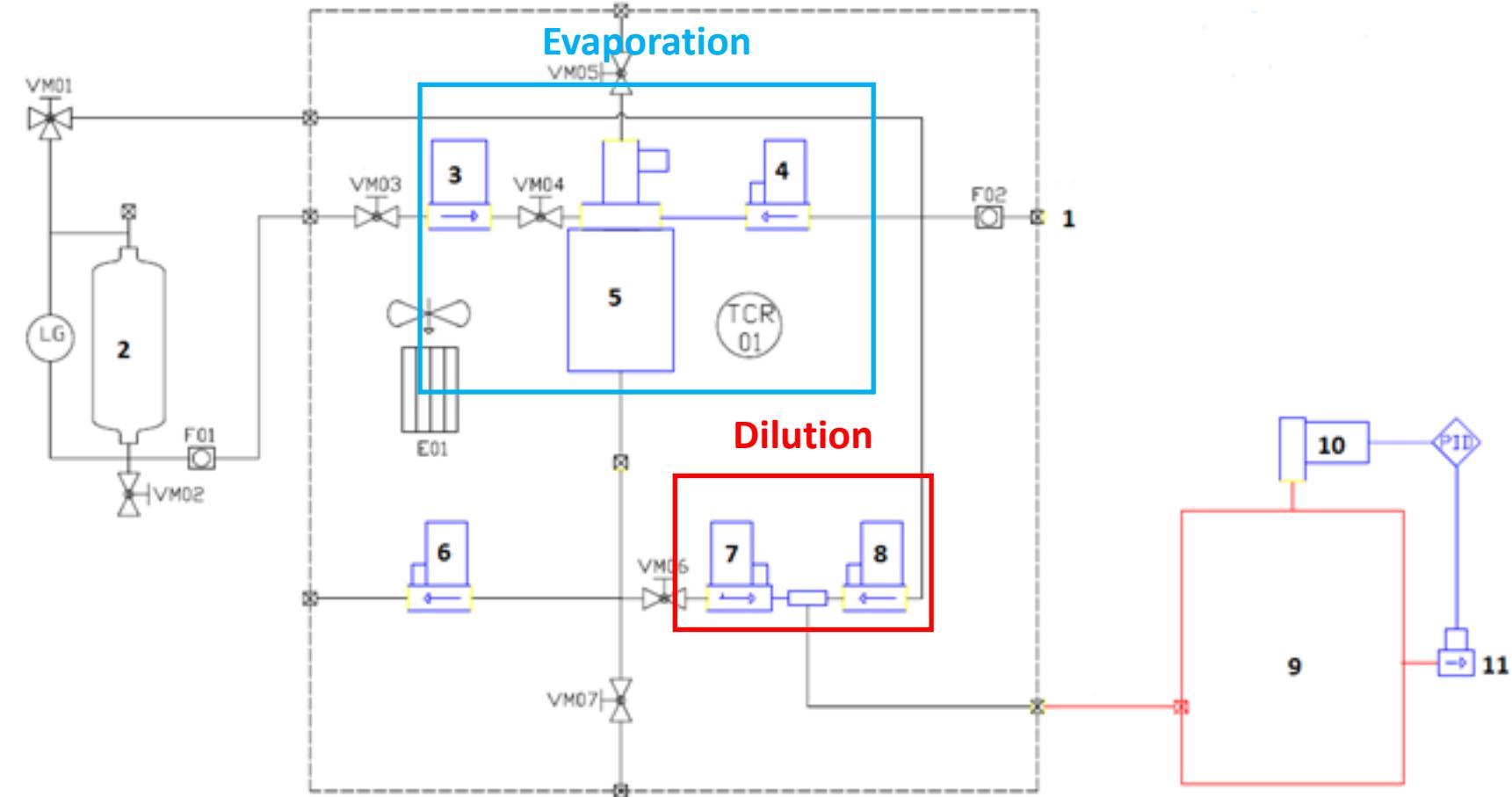
- 1) Clean Dry Air (CDA) inlet,
- 2) pure water reservoir,
- 3) compact Coriolis mass flow controllers for liquids,
- 4) mass flow controllers for gases,
- 5) evaporator,
- 6) pressure controller,
- 7) mass flow controllers for gases,
- 8) mass flow controllers for gases,
- 9) testing chamber or device under test,
- 10) pressure controller,
- 11) outlet



HAG / dilution (1/6)

■ Schematic overview

- 1) Clean Dry Air (CDA) inlet,
 - 2) pure water reservoir,
 - 3) compact Coriolis mass flow controllers for liquids,
 - 4) mass flow controllers for gases,
 - 5) evaporator,
 - 6) pressure controller,
 - 7) mass flow controllers for gases,
 - 8) mass flow controllers for gases,
 - 9) testing chamber or device under test,
 - 10) pressure controller,
 - 11) outlet



HAG / dilution (2/6)

■ Ranges

Component's number	Component's name	Component's function	Range
3)	Coriolis mass flow controller	Injection of liquid water	1 g/h to 5 g/h
4)	Mass flow controller	Control of carrier gas flow	4,8 nl/h to 240 nl/h
6)	Pressure controller	Control of pressure before sampling	200 mbara to 10 bara
7)	Mass flow controller	Control of flow sample	4,8 nm/l/h to 240 nm/l/h
8)	Mass flow controller	Control of dilution gas flow	0,8 nl/h to 40 nl/h

HAG / dilution (3/6)

■ Evaporation step

$$r_{evap} = \frac{m_{water}}{m_{MFC1}} = \frac{\dot{m}_{water}}{\dot{m}_{MFC1}}$$

$$u(r_{evap}) = \sqrt{\left(\frac{1}{\dot{m}_{MFC1}}\right)^2 \cdot u^2(\dot{m}_{water}) + \left(-\frac{\dot{m}_{water}}{(\dot{m}_{MFC1})^2}\right)^2 \cdot u^2(\dot{m}_{MFC1})}$$

Mesurand	Value		$U_{k=2}$
\dot{m}_{water}	5 g/h	$0,5 \cdot 10^{-3}$ kg/h	$4 \cdot 10^{-5}$ kg/h
Q_{MFC1}	240 nl/h	0,31 kg/h	$1,2 \cdot 10^{-3}$ kg/h
r_{evap}	$1,6 \cdot 10^{-3}$ kg v./kg d.a.		$1,61 \cdot 10^{-3}$ kg v./kg d.a.

HAG / dilution (4/6)

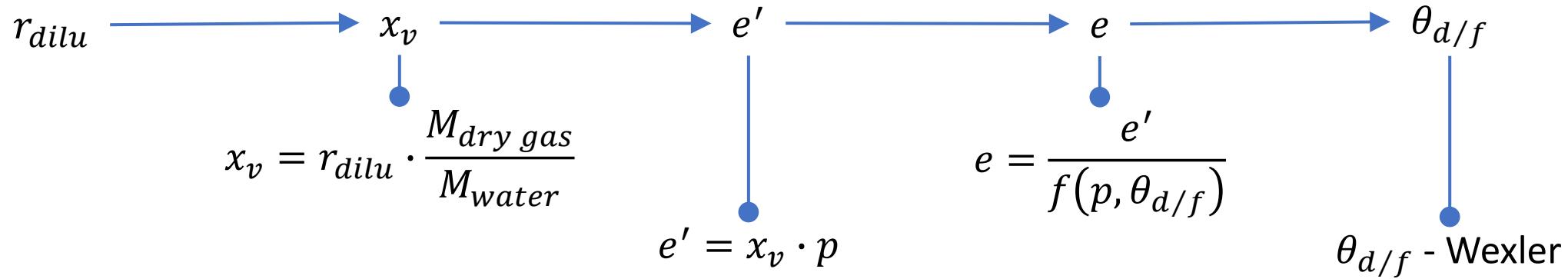
■ Dilution step

$$r_{dilution} = \frac{\dot{m}_{MFC2} \cdot r_{evap}}{\dot{m}_{MFC2} + \dot{m}_{MFC3}}$$

$$\begin{aligned} u^2(r_{dilution}) &= \left(\frac{\dot{m}_{MFC3} \cdot r_{evap}}{(\dot{m}_{MFC2} + \dot{m}_{MFC3})^2} \right)^2 \cdot u^2(\dot{m}_{MFC2}) + \left(-\frac{\dot{m}_{MFC2} \cdot r_{evap}}{(\dot{m}_{MFC2} + \dot{m}_{MFC3})^2} \right)^2 \\ &\quad \cdot u^2(\dot{m}_{MFC3}) + \left(\frac{\dot{m}_{MFC2} \cdot \dot{m}_{MFC3} + \dot{m}_{MFC2}^2}{(\dot{m}_{MFC2} + \dot{m}_{MFC3})^2} \right)^2 \cdot u^2(r_{evap}) \end{aligned}$$

Mesurand	Value		$U_{k=2}$
Q_{MFC2}	8,3 nml/h	$1,07 \cdot 10^{-5}$ kg/h	$1,1 \cdot 10^{-5}$ kg/h
Q_{MFC3}	40 nl/h	$5,17 \cdot 10^{-2}$ kg/h	$2,9 \cdot 10^{-4}$ kg/h
r_{evap}	$1,6 \cdot 10^{-3}$ kg v./kg d.a.		$1,61 \cdot 10^{-3}$ kg v./kg d.a.
r_{dil}	$3,33 \cdot 10^{-7}$ kg v./kg d.a.		$3,33 \cdot 10^{-7}$ kg v./kg d.a.

- From r_{dilu} to $\theta_{d/f}$



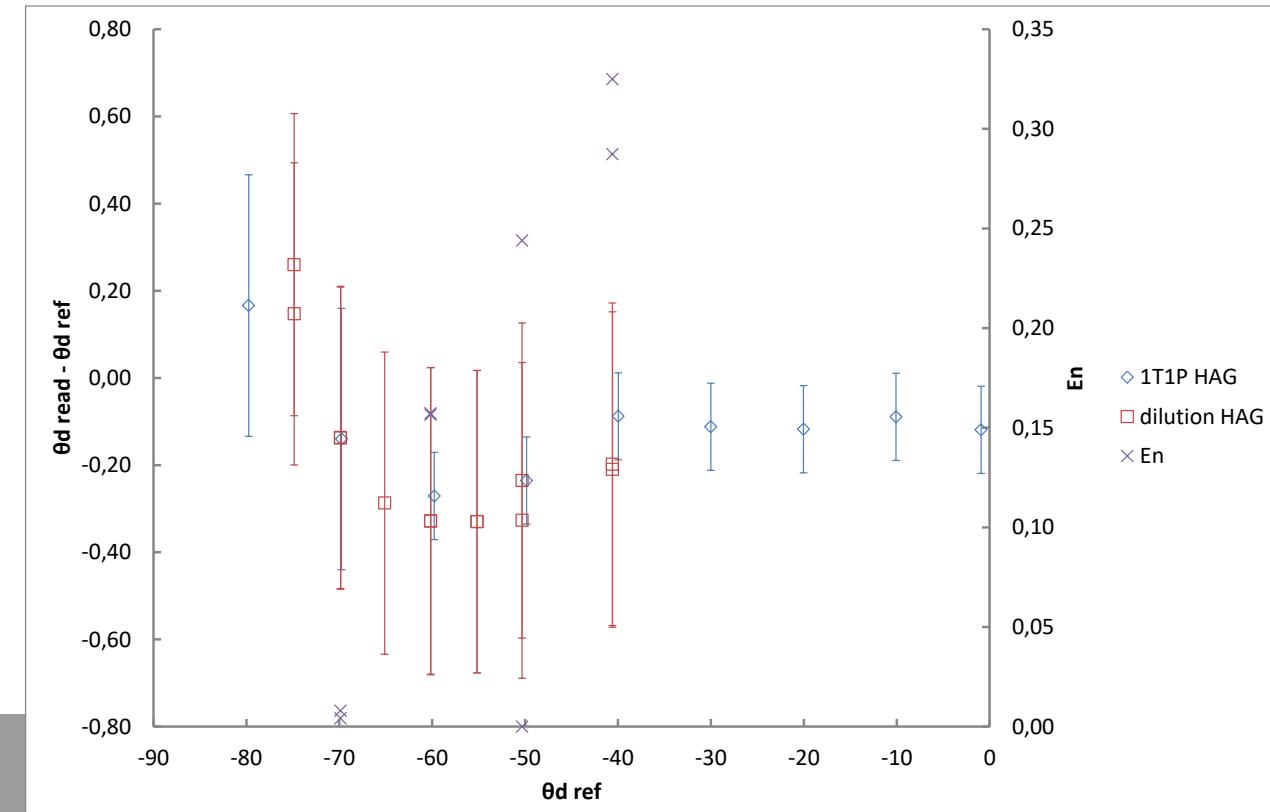
HAG / dilution (6/6)

- Expanded uncertainty

Mesurand	Value		$U_{k=2}$
\dot{m}_{water}	5 g/h	$0,5.10^{-3} \text{ kg/h}$	4.10^{-5} kg/h
Q_{MFC1}	240 nl/h	0,31 kg/h	$1,2.10^{-3} \text{ kg/h}$
Q_{MFC2}	8,3 nmol/h	$1,07.10^{-5} \text{ kg/h}$	$1,1.10^{-5} \text{ kg/h}$
Q_{MFC3}	40 nl/h	$5,17.10^{-2} \text{ kg/h}$	$2,9.10^{-4} \text{ kg/h}$
p	101325 Pa		300 Pa
r_{evap}	$1,6.10^{-3} \text{ kg v./kg d.a.}$		$1,61.10^{-3} \text{ kg v./kg d.a.}$
r_{dil}	$3,33.10^{-7} \text{ kg v./kg d.a.}$		$3,33.10^{-7} \text{ kg v./kg d.a.}$
θ_{ref}	$-80 \text{ }^{\circ}\text{C}$		0,35 $^{\circ}\text{C}$

Results (1/3)

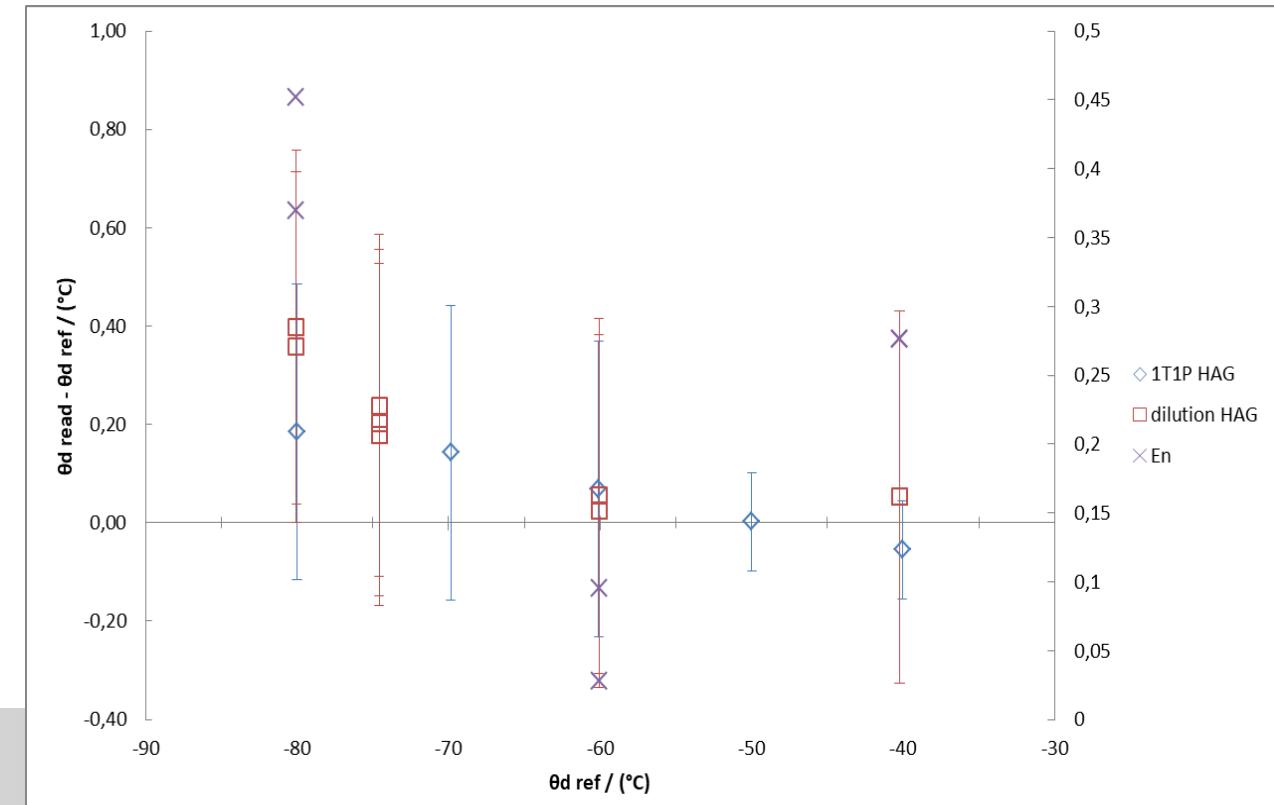
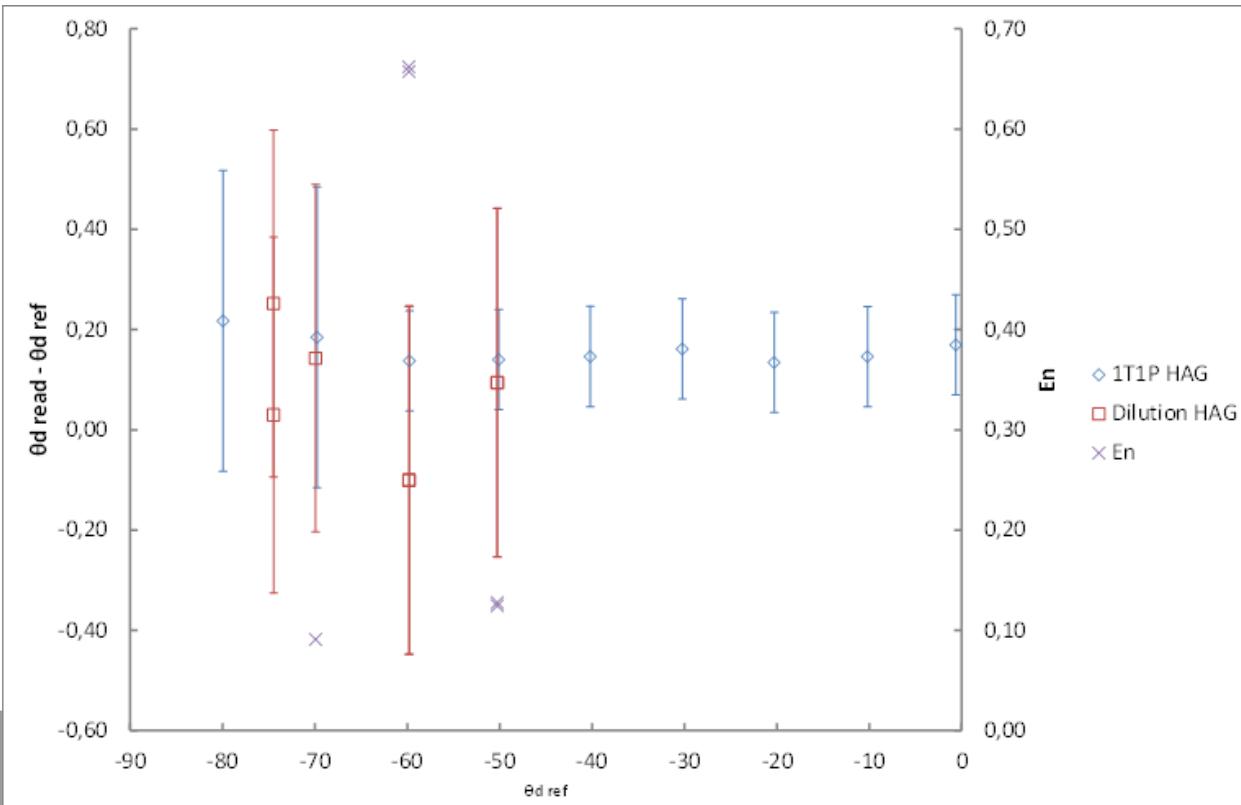
- Intra-laboratory comparison: 1T1P HAG vs HAG/dilution
 - Travelling standards:
 - MBW DP30



Results (2/3)

■ Intra-laboratory comparison: 1T1P HAG vs HAG/dilution

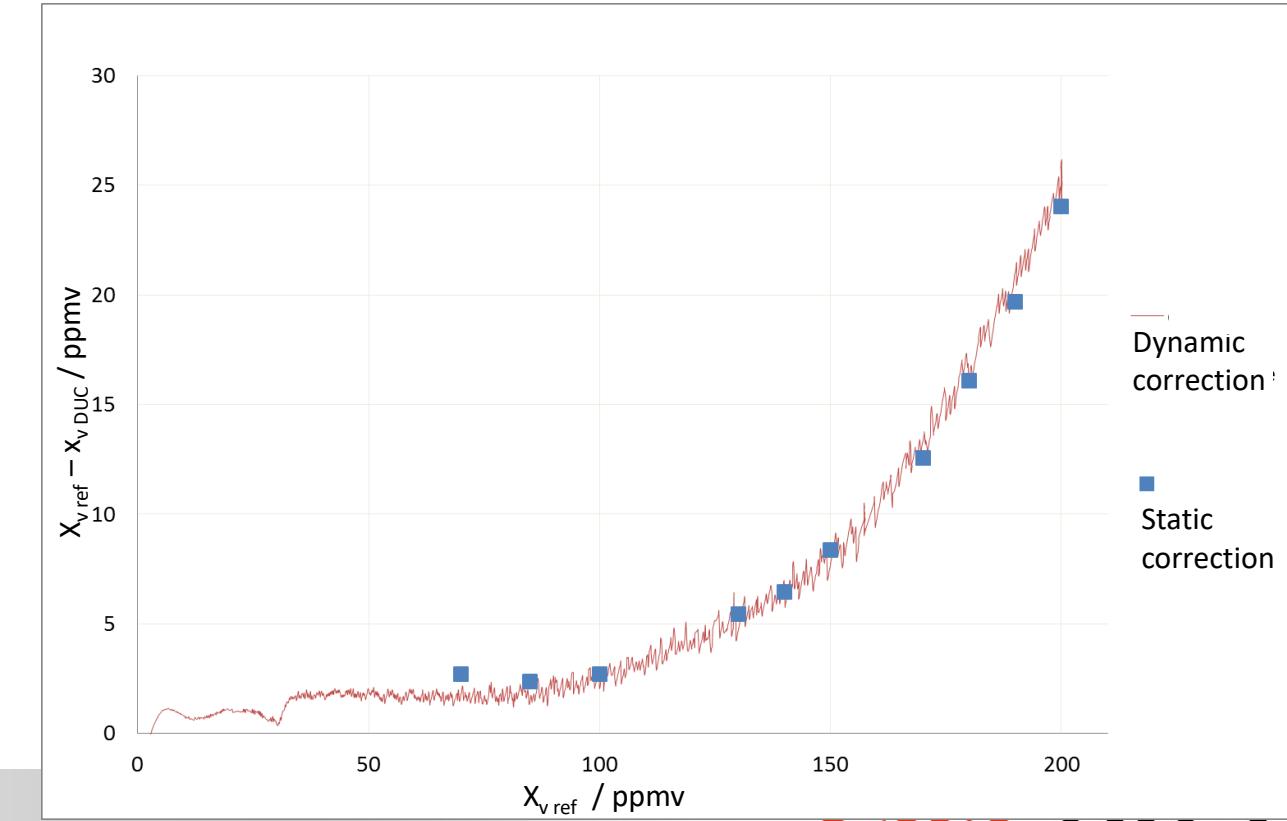
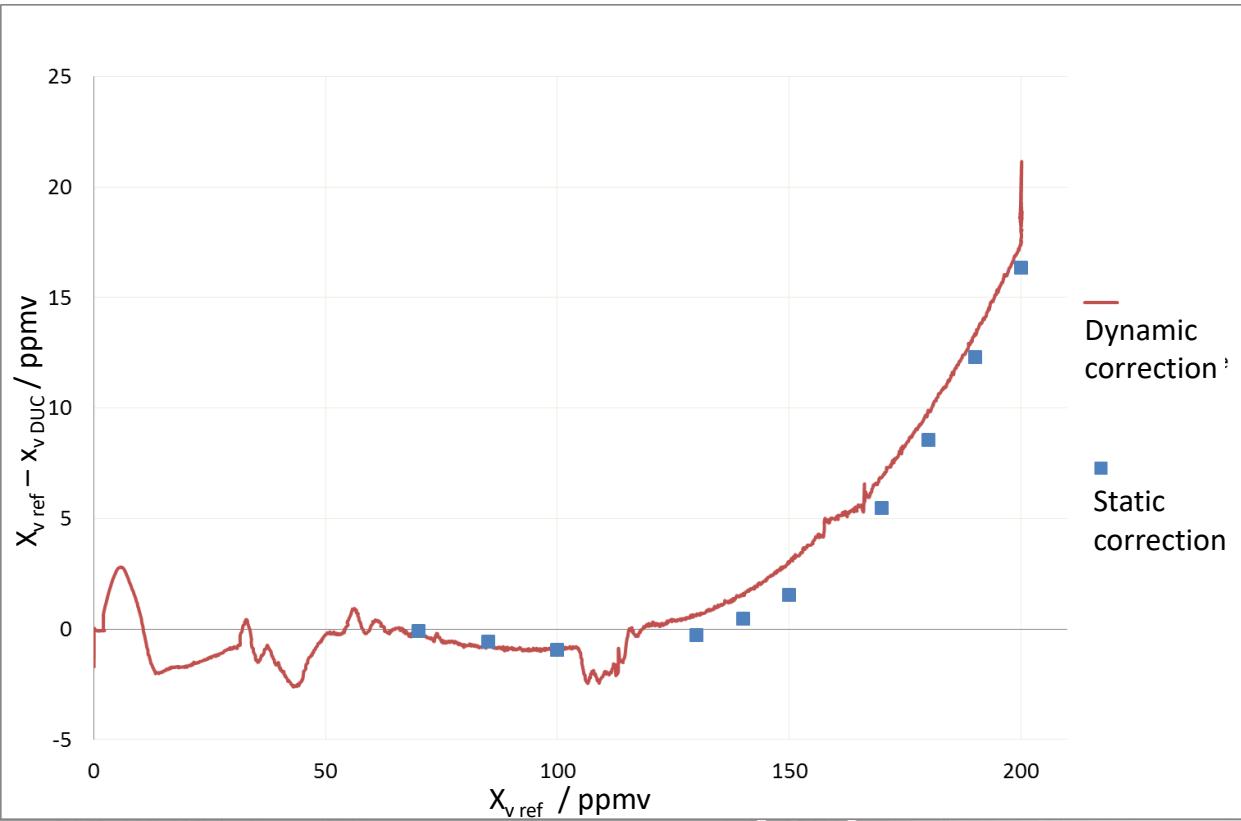
- Travelling standards:
 - MICHELL Instruments S8000 RS
 - MICHELL Instruments S8000 -100



Results (3/3)

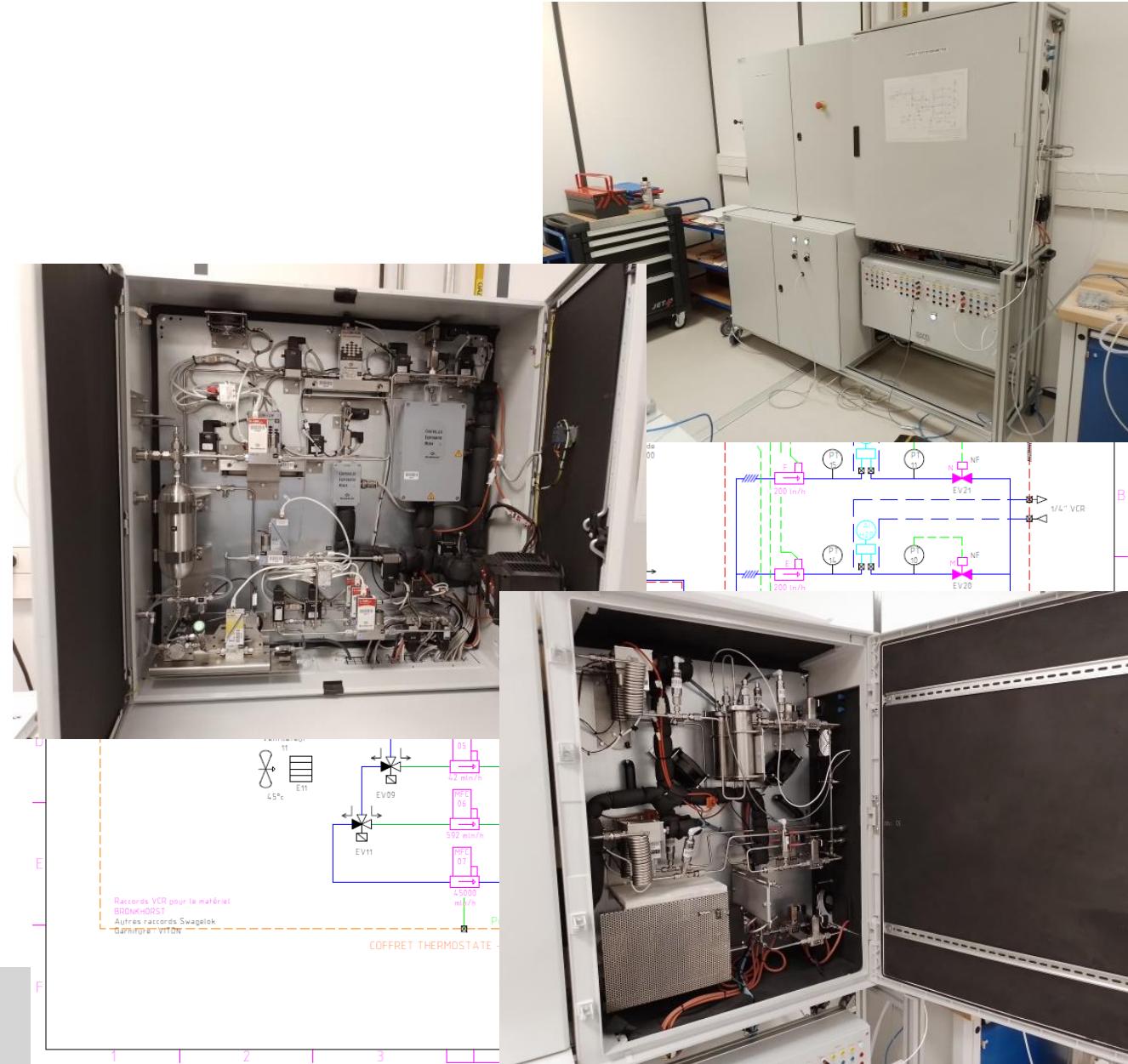
■ Dynamique calibration

- Based on the idea proposed by VTT-MIKES (JRP 14IND11 HIT)
- Static calibration vs Dynamic calibration



Conclusion and next steps

- Validation has been done
 - Up to -80 °C
 - At atmospheric pressure
- Investigation of dynamic calibration
 - Promissing perspectives have been observed
- Validate the evaporation HAG
 - Below -80 °C
 - Pressure up to 10 bar abs.
- Increase the calibration capabilities with HAG/dilution



Acknowledgments

■ Institutions

- EMPIR initiative, co-funded by European Union's Horizon 2020 research and innovation programme and the EMPIR participating States
- French Metrology



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

■ Providers

- Bronkhorst
- 2MProcess
- MICHELL Instruments

Thank you for your attention