New calibration facility developped at LNE-CETIAT

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Abstract. LNE-CETIAT has developed its own primary realization of the unit in humidity. Willing to have a more versatile generator, the laboratory has developed a new humid air generator based on dilution principle. These facilities are presented in this work as well as the results of two comparisons.

1 Introduction

LNE-CETIAT is the designated institute for the French national reference in humidity. It ensures SI traceability for end users needing calibration and about 500 COFRAC's calibrations per year are performed. LNE-CETIAT, has developed its own Humid Air Generator (HAG), [1], [2], [3] and [4]. This primary realization of the unit, in humidity, enables calibration in dew point temperature, relative humidity and other derived quantity. With time and involvement in different projects, ranges and capabilities have been extended with these facilities primary calibration.
Thus, for dew/frost point temperature calibration, the configuration is the following:

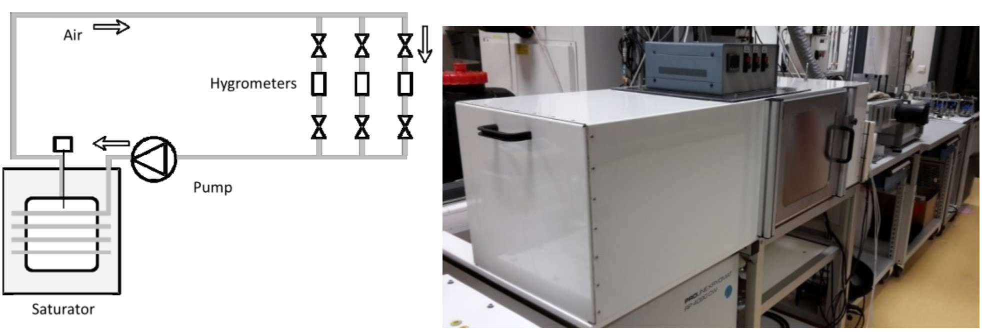

Fig. 1. HAG developed at LNE-CETIAT, 1 pressure 1 temperature closed loop principle

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Table 1. Scope of accreditation in dew/frost point temperature

Measurand	Range	Uncertainty ($U_{k=2}$)
Dew/frost point temperature	-80 °C / -60 °C	0,3 °C
	-60 °C / 0 °C	0,1 °C
	0 °C / +90 °C	0,06 °C

Thus, for relative humidity calibration, the configuration is the following:

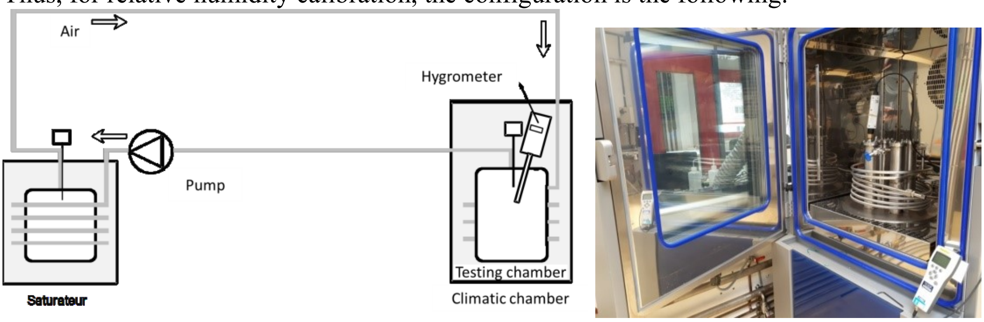

Fig. 2. HAG developed at LNE-CETIAT, 1 pressure 2 temperatures closed loop principle

Table 2. Scope of accreditation in relative humidity

U_k=2($U_{w,i}$)	U_w (%rh)										
Θ_{dry} (°C)	5	10	20	30	40	50	60	70	80	90	95
-30					0,6	0,8	0,9	1,0	1,2	1,3	1,4
-20		0,3	0,4	0,5	0,6	0,7	0,8	1,0	1,1	1,2	1,3
-10	0,3	0,3	0,4	0,5	0,6	0,7	0,8	0,9	1,0	1,1	1,2
0	0,3	0,3	0,3	0,4	0,5	0,6	0,7	0,8	0,9	1,0	1,0
10	0,3	0,3	0,3	0,4	0,5	0,5	0,6	0,6	0,7	0,8	0,8
20	0,3	0,3	0,3	0,3	0,4	0,5	0,5	0,6	0,7	0,7	0,8
30	0,3	0,3	0,3	0,3	0,4	0,4	0,5	0,6	0,6	0,7	0,7
40	0,3	0,3	0,3	0,3	0,4	0,4	0,5	0,5	0,6	0,6	0,7
50	0,3	0,3	0,3	0,3	0,4	0,4	0,4	0,5	0,5	0,6	0,6
60	0,3	0,3	0,3	0,3	0,3	0,4	0,4	0,5	0,5	0,6	0,6
70	0,3	0,3	0,3	0,3	0,3	0,4	0,4	0,5	0,5	0,5	0,6
80	0,3	0,3	0,3	0,3	0,3	0,4	0,4	0,4	0,5	0,5	0,5
90	0,3	0,3	0,3	0,3	0,3	0,4	0,4	0,4	0,5	0,5	0,5
100	0,3	0,3	0,3	0,4	0,4	0,5	0,6	0,7			
110	0,3	0,3	0,3	0,4	0,4	0,5					
120	0,3	0,3	0,3	0,4							
130	0,3	0,3	0,3								
140	0,3	0,3	0,3								

During projects JRP METEOMET 2 – EMRP and JRP HIT – EMPIR, LNE-CETIAT has developed two new calibration rigs enabling secondary realization of the unit in humidity. The principle is mainly based on an accurate dilution of liquid water, which has been evaporated, with dry gas. Whilst traceability to SI unit of the primary realization is ensured through temperature probes calibration, the traceability of the secondary realization is ensured through mass flow controllers calibration. This article presents HAG based on dilution principle: description of the calibration rig, equations used for determining reference in mixing ratio, as well as the characterization of the main uncertainty components or effects and preliminary uncertainty budget. At last, comparison between primary realization and secondary realization is presented and discussed.

2 HAG based on dilution principle

2.1 Overview

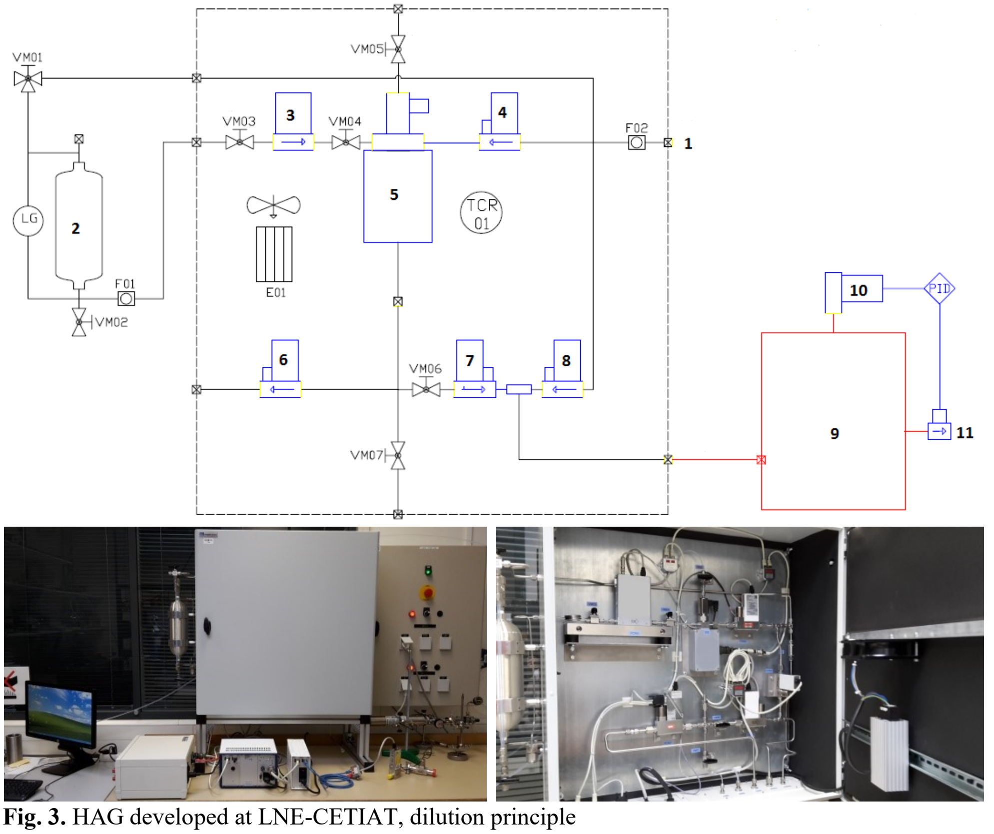

Fig. 3. HAG developed at LNE-CETIAT, dilution principle

On the schematic view: 1) Clean dry air 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.
The facility has been also presented in the reference [5].

2.2 Principle

Liquid water, coming from the Coriolis mass flow controller, is evaporated and mixed with clean dry air, coming from mass flow controller n°1, in the evaportator.

$$r_{evap} = \frac{m_{water}}{m_{dry\ air,MFC\ 1}} = \frac{\dot{m}_{water}}{\dot{m}_{dry\ air,MFC\ 1}} \qquad (1)$$

Then, the humid air generated is either sent to the vent via a pressure controller, or sampled with a second mass flow controller n°2. This sample is then mixed with clean dry air, coming from mass flow controller n°2, and leading to:

$$r_{dilu} = \frac{\dot{m}_{humid\ air,MFC\ 2} \cdot r_{evap}}{\dot{m}_{humid\ air,MFC\ 2} + \dot{m}_{dry\ air,MFC\ 3}} \quad (2)$$

At last the humid air generated is delivered to the device under test where the pressure is measured and controlled by a pressure controller associated to a discharge valve.
Here the measurand generated a mixing ratio and the traceability to SI unit is ensured by calibration in mass and time of the mass flow controllers.

2.3 Discussion

In order to have a better handling of any thermal disturbance on mass flow controllers, pressure controller and Coriolis mass flow controller, the surrounding environment is temperature controlled at the nominal value of 42 °C ±1 °C. For this purpose, all the instruments are set in a box, see **Fig. 3**, thermally insulated. A temperature controller, associated with Pt100 probe and an electrical heater, ensures temperature handling while a fan ensures temperature homogeneity in the volume.
Due to the principle of the Coriolis mass flow controller, a great care has been taken about vibrations and tare value. For this purpose, the Coriolis mass flow controller has been set on massive socket and fixed on silent blocks, see **Fig. 3**. Indeed any vibration equal or close to the eigenfrequency of the Coriolis mass flow controller could affect precision and uncertainty of the instrument. For a better instrument-zeroing, two isolation valves has been integrated upstream and downstream the instrument, see **Fig. 3**. This will be also helpful for controlling the drift of the instrument during measurement campaign. The tare value is checked before every calibration point.
The typical calibration uncertainty that can be obtained, with this type of instrument, with the French national reference for micro-flows, is 0,2% precision and a calibration uncertainty of about 0,1 % (k=2) from 1 g/h to 10 g/h.
All the instruments are kept inside the thermostated box during their calibration
The dryness of the clean dry air is checked every week. Indeed this air is delivered from a compressed dry air facility and an additional dryer is used in the laboratory prior the dilution HAG. Usually the dryness is below -90 °C at ambient pressure.

Table 3. Scope of accreditation in dew/frost point temperature

Measurand	Range	Uncertainty ($U_{k=2}$)
Dew/frost point temperature	-75 °C / -40 °C	0,35 °C

In order to validate the development of this new calibration facility, two comparisons have been done inside the laboratory. Two units, one MBW DP30 and one Michell Instrument S8000, have been calibrated with the 1T1P HAG presented in the introduction and then these two units have been calibrated with the dilution HAG. Results are presented below as well as the normalized error *En*.

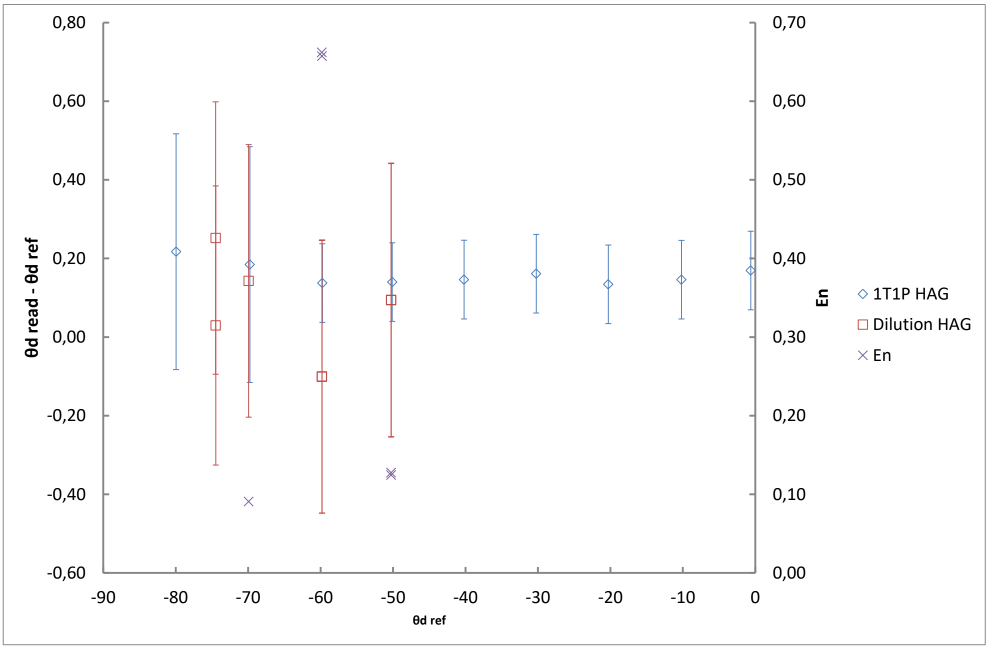

Fig. 4. Comparison 1T1P HAG and dilution HAG with S8000 unit

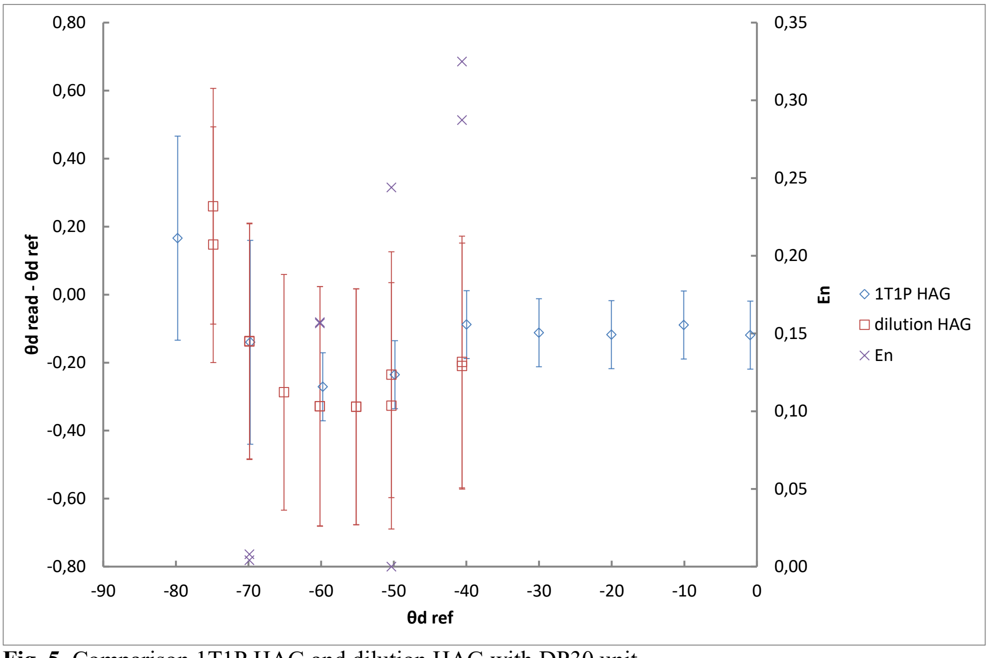

Fig. 5. Comparison 1T1P HAG and dilution HAG with DP30 unit

In both cases the normalized error, En, is below 0,7 and 0,35 respectively.

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3 Conclusion

LNE-CETIAT has developed during METEOMET project a dilution HAG, this generator has been slightly improved during HIT project. The principle relies on the evaporation of liquid water, mixed with clean dry air and then diluted with clean dry in dilution step.
During this work different some key contributions, in the overall uncertainty, have been identified and discussed. The detailed uncertainty budget will be presented in separated work; nevertheless the range, as well as the final uncertainty have been presented and validated with a comparison inside the laboratory with our 1T1P HAG.

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References

1. Gonin, F., B. Blanquart, Improvement of CETIAT humid air generator in low-range, proceedings of 5th ISHM, (2006)
2. Cretinon, B., The CETIAT standard humidity generator operating from -60°C to +70°C in dewpoint temperature, proceedings of 3rd ISHM, 1998
3. Georgin, E., Blanquart, B., Humid air generator in low range: qualification, proceedings of TEMPMEKO & ISHM, (2010)
4. Georgin, E. et al., Vers un principe de dilution pour la génération d'air humide au LNE-CETIAT, proceedings of 16th International Congress of Metrology, Paris, 2013
5. Georgin *et al.*, Enhancement factor measurements under atmospheric conditions, proceedings of 18th International Congress of Metrology, Paris – France (2017)