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Development of the High-Temperature Dew-Point Generator Over the Past 15 Years

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Abstract At VSL a humidity generator was designed and constructed in the early 1990s. This generator was of the re-circulating-single-pressure type. Over the years, the generator has been thoroughly revised and several critical components have been replaced. Among others the pre-saturator and the change from re-circulation to single-pass mode. Validating experiments showed that the range of the new setup could be extended from 70 °C to 95 °C dew-point temperature, and the last modification allows an uncertainty of 0.048 °C ($k = 2$) at the maximum temperature. In 2009 the setup was used in the Euramet-T-K8 humidity intercomparison at temperatures up to 95 °C. In the period from 2003 to 2015, four state-of-the-art chilled mirror hygrometers were regularly calibrated with the generator. One of these was also calibrated with the primary dew-point standards of several other European National Metrology Institutes, which made it possible to link the VSL generator to the generators used in these institutes. An analysis of the results of these calibrations shows an agreement in calibration capabilities within 0.01 °C with PTB and NPL.

Keywords Calibration · Chilled mirror hygrometer · Dew-point generator · High temperature

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1 Introduction

In the 1990’s VSL designed and constructed a dew-point temperature generator [1,2]. Initially the generator was composed of two separate generators for low and high temperature and a single re-circulation system and covered the range from −60 °C up to 70 °C. This generator was used for the measurements in the CCT-K6 and Euramet-T-K6 Key comparisons in 2003 and 2004, respectively, [3,4].

The generator part for high temperatures had two critical elements, the seal of the circulation pump and the piezo-electric pre-saturator, both needing considerable maintenance. With the introduction of a new type of pre-saturator, the generator was tested as single-pass generator. The generator in this configuration was used in 2009 for the measurements in the Euramet-T.K8 Key comparison (range up to 95 °C).

Over the years VSL purchased three chilled mirror hygrometers (CMH), a MBW DP-30 and two MBW 373 HX, used for internal traceability, which were periodically calibrated using the high-temperature saturator (HTS). The generator was also used for customer calibrations and one of these instruments, a MBW 373 LHX, owned by DTI, was also calibrated by other European National Metrology Institutes (NMI).

The modifications of the saturator and pre-saturator since 2007, the history on the internal chilled mirror hygrometer and the results on the customer instrument are described in this paper.

In this paper we will report on the revision of the high-temperature dew-point generator, with particular emphasis on:

- The design of the pre-saturator and saturator.
- The experiments validating the saturation efficiency.
- The history of the chilled mirror hygrometer used as internal transfer standard.
- The linking of the generator to other primary generators in Europe.

2 Modifications of the Generator over the Past 10 Years

The original dew-point generator was built in the 1990’s as a re-circulating type saturator [1]. A critical component, the ferro-magnetic seal of the re-circulating pump, needed to be refurbished every 2 years, with a time-consuming procedure. The seal was also a potential source of contamination.

In 2007 a new type of pre-saturator, a porous chalk tube, used as oil filter in big ship engines, was tested. The concept was borrowed from the Danish institute DELTA, where such chalk filters were used in a humidifier for big climatic chambers (see Fig. 1). The small pores in the chalk transform the supply gas into small gas bubbles, which are easy to saturate, due to the large surface to volume ratio. Figure 2 shows the principle of the chalk oil filter used in the pre-saturator: a single chalk tube was encapsulated in a glass cylinder, containing the water. The gas from the pre-cooler is pressed through the small chalk tube pores, and the small bubbles then are saturated in the water. Tests showed an improvement in the efficiency of the new pre-saturator, but the generator was still used in re-circulation mode until 2009.

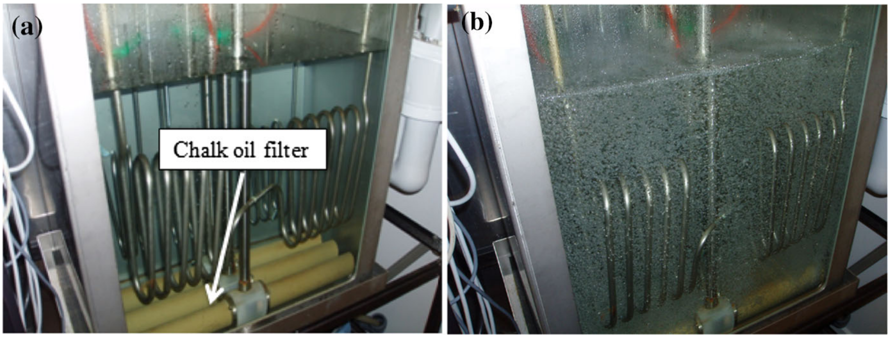

Fig. 1 Saturator for climatic chamber used at DELTA, Denmark, based on chalk oil filters (a), which creates small bubbles due to the small pores in the chalk (b)

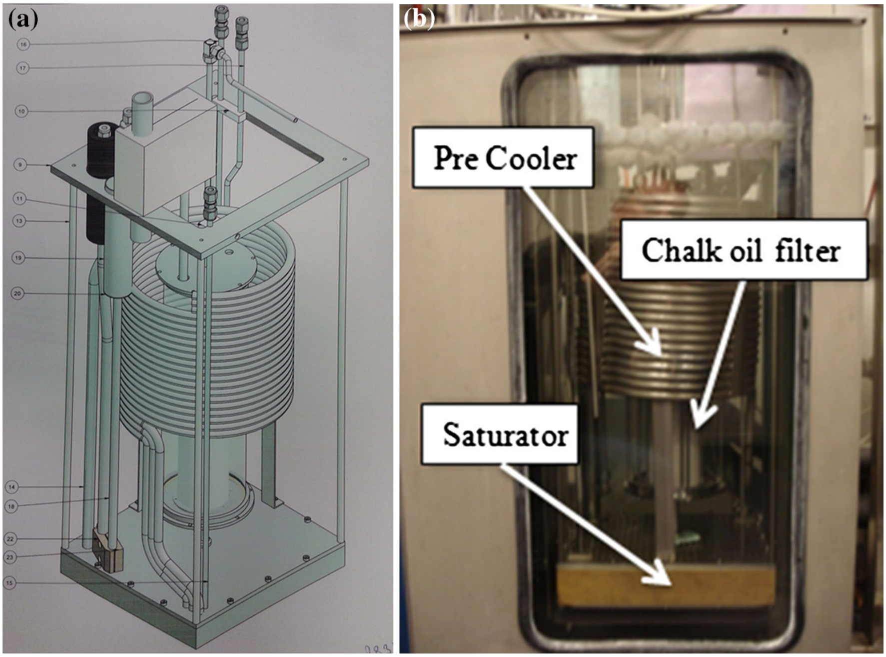

Fig. 2 The HTS with the new pre-saturator based on the chalk oil filters; design (a) and realization (b)

In 2009, before using the generator for the Euramet-T-K8 Key Comparison, the generator was split into two separate single-pass generators; one for high temperature, consisting of the existing generator and one for temperatures below 10 °C, consisting of the newly developed high-pressure dew-point temperature generator, HPDG [5]. The separated HTS was validated as single-pass generator before using it in the comparison in the range from 30 °C up to 95 °C.

The original saturator built in the 1990’s consisted of a channel machined in a copper block with a gold coating to prevent oxidation [1]. During inspection of the saturator in 2009, the gold layer showed dark spots (see Fig. 3a). As gold is normally

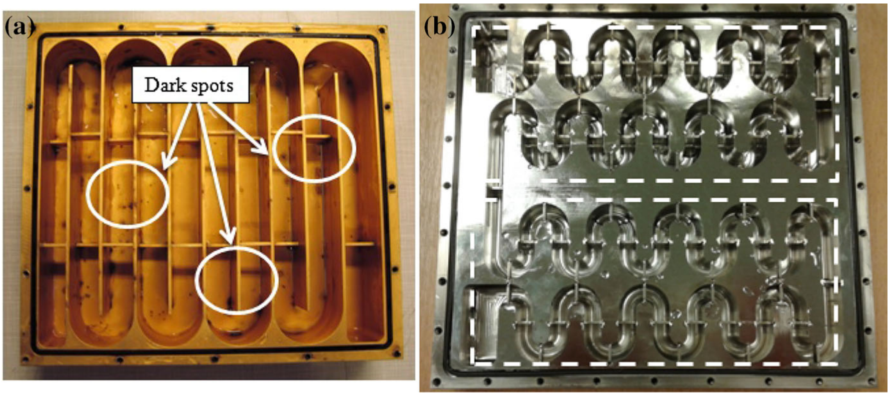

Fig. 3 The original HTS saturator (a) constructed of copper with a gold layer, with dark spots indicating corrosion and the newly built saturator for the HTS based on the design of the HPDG (b). Each section enclosed in a dashed area has the same design (channel length, number of dams and barriers) as the HPDG

not subject to corrosion, it was concluded that the layer at the corroded spots was too thin. In an attempt to re-coat it, the saturator was sandblasted. The sandblasting only partially removed the gold layer. Chemical removal was not possible as the process would have created an uneven surface due to the strong reaction with copper. It was then decided to design and build a new saturator based on the experience gained in the high-pressure dew-point temperature generator, HPDG. As the water bath, Tamson TV7000SP, has a larger operation area than the bath used for the HPDG, the HTS was designed as two HPDG saturators in series (see Fig. 3b) with respect to channel length, dams and barriers. The stainless steel design is explained in more detail in the paper describing the construction of the HPGD [5].

The last modification on the saturator was a new pre-saturator in 2012 (Fig. 4a) and a new transparent cover in 2015 (Fig. 4b). The chalk pre-saturator was replaced, because at the low flows in the single-pass mode, it used only the top part of the tube and thus only a small part of the water in the pre-saturator. The newly built pre-saturator was a simple container, where gas enters at the bottom of a water column through a small grid. Although the bubbles through the grid are bigger than the bubbles through the pores of the chalk tube, the length of the water column in the container more than compensates for the bubbles size, as was seen in the example validation tests at 50 °C (see Fig. 5). In the validation test, both the dew-point temperature of the pre-saturator and the saturator were measured as function of the gas flow through the generator using a CMH with a constant gas flow of $0.5\,\mathrm{l} \cdot \mathrm{min}^{-1}$. The CMH on the saturator shows no flow dependence, while the CMH on the pre-saturator shows a small flow dependence. The efficiency of the pre-saturator, calculated using the 0.2 °C temperature difference at 50 °C is 99 %.

The transparent cover introduced in 2015 did not directly influence the performance of the (pre-) saturator, but reduced the evaporation of the water in the bath. This makes it possible to use the HTS for longs times at high temperatures, without the need to refill the water in the bath, which immediately disturbs the stability of the HTS.

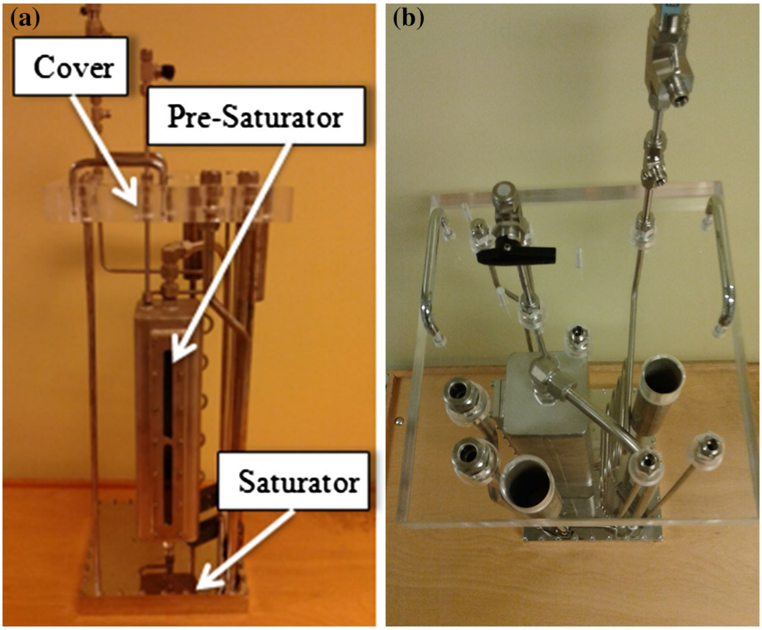

Fig. 4 The HTS with the new pre-saturator (a) and the transparent cover (b)

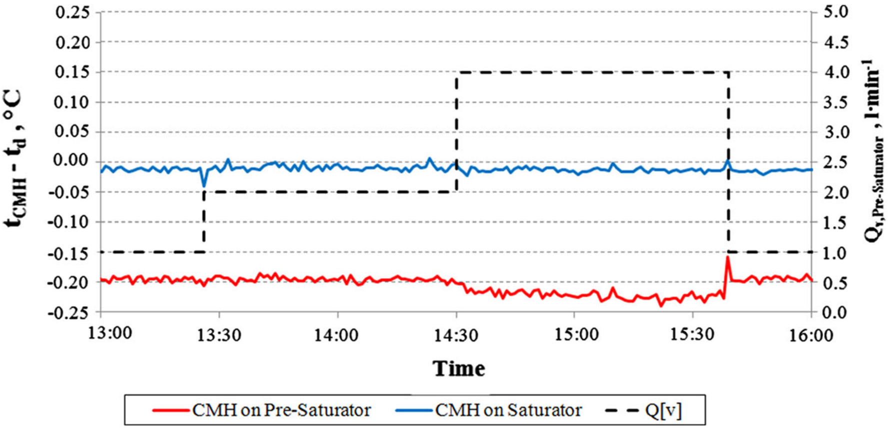

Fig. 5 The efficiency of the 2012 pre-saturator at 50°C measured with two chilled mirror hygrometers. The dew-point temperature indication of the CMH, t_{CMH}, is compared with the dew-point temperature of the generator, t_{d}, at different pre-saturator gas flows, $Q_{\mathrm{v,Pre-Saturator}}$

3 Validation of the HTS

After each modification the performance of the HTS was checked using the internal transfer standards, one MBW DP-30 and two MBW 373 HX chilled mirror hygrometers. The checks showed no deviating behaviour of the HTS as the change of the measured error of the transfer standards was within the calibration uncertainty.

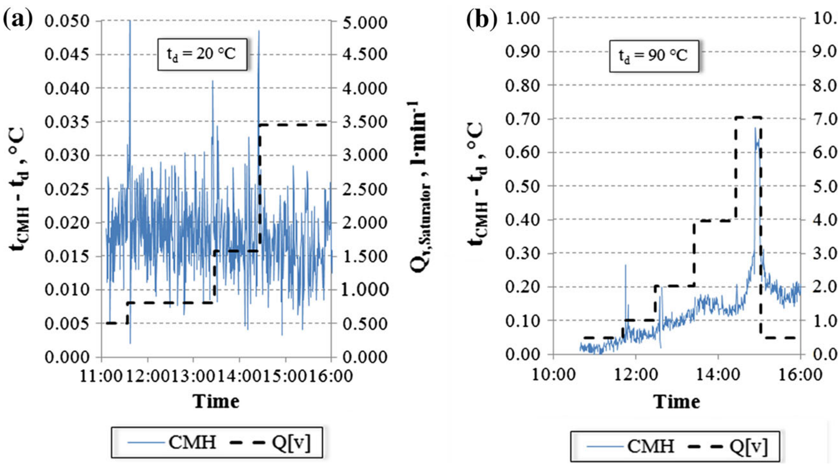

Fig. 6 The validation of the saturator efficiency of the HTS in 2007 at 20 °C (a) and 90 °C (b) during the measurements performed for the Euramet-K8 Key Comparison

A full validation of the generator was performed at the end of the Euramet-T-K8 Key Comparison measurements in September 2009 and after the last modification in January 2016. In the validations, the output of the HTS was monitored with an internal CMH. The flow through the CMH was kept constant at 0.5 l·min^{-1} and the flow through the generator, $Q_{\mathrm{v,Saturator}}$, was increased in steps up to 7 l · min^{-1}. The indication of the CMH, t_{CMH}, was compared with the reference temperature, t_{d}, measured with a Standard Platinum Resistance Thermometer (SPRT). The sensing element of the SPRT was in close thermal contact with the exit of the saturator. Figure 6 shows the offset, $t_{\mathrm{CMH}} - t_{\mathrm{d}}$, at 20°C and 90 °C during the validation in 2009 and Fig. 7 shows the offset at 15 °C and 90 °C during the validation in 2016. The results at low temperature are comparable, but the results at high temperature show a large improvement. Most likely this is caused by the new cover, which reduces the evaporation in the bath (dropping water level) and provides stable readings at high temperatures.

The uncertainty component for the efficiency, $u(t_{\mathrm{d,eff}})$, was calculated in 2009 as the difference between the average offset at 0.5 l · min^{-1} and the average offset at the maximum flow tested divided by $2 \cdot \sqrt{3}$. The measurement at 7 l · min^{-1} and 90 °C in 2009 is not used in the calculation for the efficiency as the point is clearly indicating a bad saturation. Later measurements showed that the saturator can only be used up to 5 l · min^{-1}, as with higher flow water droplets appear in the output flow. In 2016 the uncertainty component was calculated using a linear fit in the offset versus saturator flow data; the dotted lines in Fig. 7b, d show the linear fits. The efficiency uncertainty component was calculated as the difference between the fit result at 0 l · min^{-1} and at 4 l · min^{-1} divided by $2 \cdot \sqrt{3}$. The validation results of the efficiency tests are given in Table 1 and the new uncertainty budget for the modified HTS is presented in Table 2. With the new validation results, the CMC at 95 °C can be reduced from 0.10 °C to 0.048 °C ($k = 2$), because of the improved temperature stability and reduced efficiency contribution (a lower limit of 6 mK is used over the whole range; higher temperatures showed lower value but with a 6 mK uncertainty).

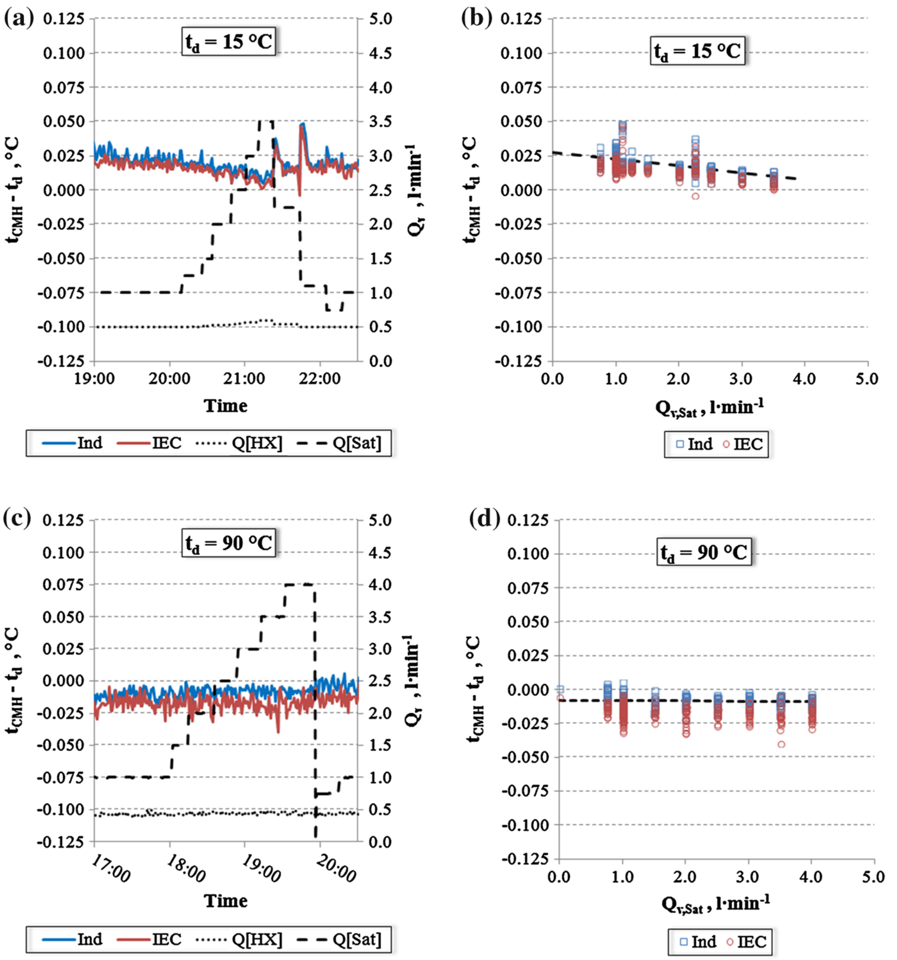

Fig. 7 The validation the saturator efficiency of the HTS in 2016 at 15 °C (a, b) and 90 °C (c, d) using the display temperature indication (Ind) and resistance measurement of the rear mirror output (IEC) of a MBW 373 HX

Table 1 The results of the validation of the HTS in 2007 and 2015 with the efficiency component, $u(t_{d,\mathrm{eff}})$, at dew-point temperatures, t_d, between 15 °C and 90°C and associated experimental standard deviation, s

t_d °C	$u(t_{d,\mathrm{eff}})$ 2009 K	s K	t_d °C	$u(t_{d,\mathrm{eff}})$ 2015 K	s K
			15	0.0058	0.0056
20	0.0009	0.005			
50	0.0092	0.009	50	0.0005	0.0054
			75	0.0005	0.0058
90	0.0344	0.014	90	0.0007	0.0052

Table 2 The uncertainty budget for the CMC of the new HTS

Saturator temperature Quantity	15 °C u_i, °C	50 °C u_i, °C	75 °C u_i, °C	95 °C u_i, °C
Thermometer				
Calibration uncertainty (sensor and indicator unit)	0.00 050	0.00 050	0.00 050	0.00 050
Long-term stability (sensor)	0.00 017	0.00 017	0.00 017	0.00 017
Self-heating and residual heat fluxes (sensor)	0.00 100	0.00 100	0.00 100	0.00 100
Calibration uncertainty (linearity)	0.00 049	0.00 050	0.00 050	0.00 051
Resolution (indicator unit)	0.00 007	0.00 007	0.00 007	0.00 007
Resistor	0.00 036	0.00 041	0.00 045	0.00 048
Saturator				
Temperature homogeneity	0.002	0.004	0.007	0.009
Temperature stability	0.001	0.004	0.004	0.005
Saturation efficiency	0.006	0.006	0.006	0.006
UUT				
Pressure drop between point of realization and UUT	0.008	0.011	0.013	0.015
Experimental standard deviation of UUT	0.010	0.010	0.010	0.010
Resolution of UUT	0.000 029	0.000 029	0.000 029	0.000 029
Reproducibility	0.010	0.010	0.010	0.010
Combined uncertainty	0.018	0.020	0.022	0.024
Expanded uncertainty	0.037	0.040	0.044	0.048

4 History of the VSL Transfer Standards

For test purposes and for internal traceability to relative humidity calibration facilities [6,7], 3 chilled mirror hygrometers were purchased for use as transfer standard; in 1995 a MBW DP-30 and in 2003 and 2010 a MBW 373 HX. A transfer standard is used to provide traceability to other facilities, but can also provide information about the performance of the generator. Deviations between transfer standard and generator during test measurements, larger than the calibration uncertainty of the transfer standard, can indicate problems with either the generator and/or transfer standard. This analysis increases the reliability of the generator.

The CMH's were calibrated against the HTS and provide a long history on the performance of the instrument and the dew-point temperature generator. The CMH's resistance output, converted to temperature according to IEC 60751 Ed. 2.0:2008, t_{IEC}, and/or the display indication, t_{ind}, was used for the calibration history. Figures 8, 9 and 10 show the history of the three CMH; the DP-30 was only operational up to 2010. The MBW 373 HX, with two separate PRT's behind the sensing mirror, one used for the internal operation and indicated on the display, t_{ind}, and one accessible via the rear resistance output, t_{IEC}, have two history graphs. Most calibration points

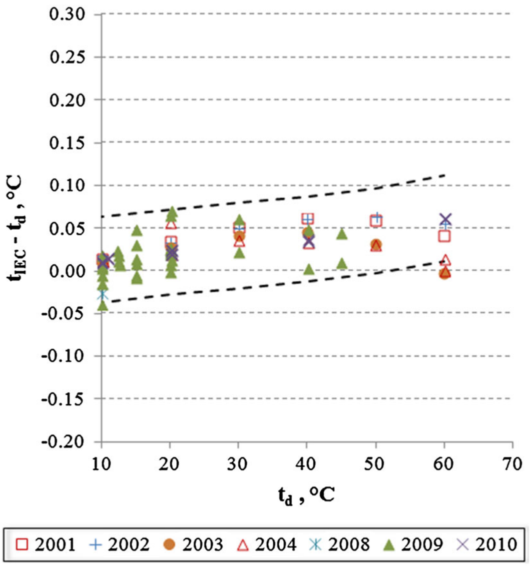

Fig. 8 The calibration results of the MBW DP-30, S/N 95-0103 rear mirror output over the period 2001–2010. The *dotted line* is the calibration uncertainty of 2010 ($k = 1$)

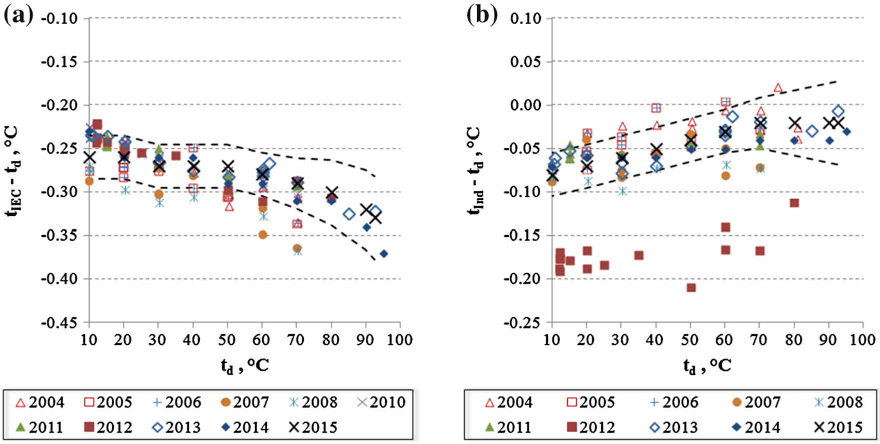

Fig. 9 The calibration results of the MBW 373 HX, S/N 03-0731 rear mirror output (a) and the display temperature indication (b) over the period 2004–2015. The *dotted line* is the calibration uncertainty of 2015 ($k = 1$)

are within the uncertainty boundaries of the last calibration, and all points are within the expanded uncertainty boundaries. The only exception is the 2012 calibration of the display temperature indication from MBW 373 HX S/N 03-0731. Limited time, and correct performance of the second output used for internal traceability, prevented a thorough investigation of the deviation. In 2013, 2014 and 2015, the calibration results were again in line with previous history. The three instruments confirm the correct operation of the HTS over period from 2003 to 2015.

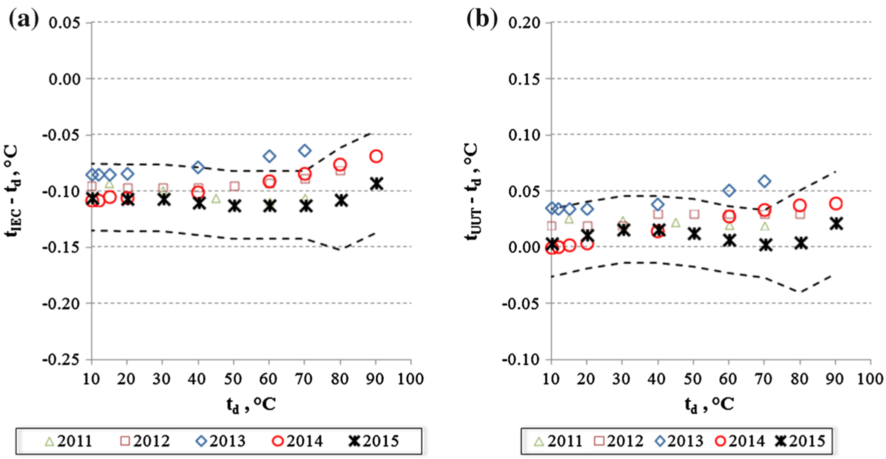

Fig. 10 The calibration results of the MBW 373 HX, S/N 10-0345 rear mirror output (a) and the display temperature indication (b) over the period 2011–2015. The *dotted line* is the calibration uncertainty of 2015 ($k = 1$)

Table 3 The calibration results of the MBW 373 LHX, S/N 06-1236 chilled mirror hygrometer at VSL in 2010 and 2015 (uncertainty, u, stated with k = 1)

Nominal	t_d VSL, 2010	$t_{IEC} - t_d$	u	t_d VSL, 2015	$t_{IEC} - t_d$	u	*Drift*
°C	°C	K	K	°C	K	K	*K*
10	10.027	−0.102	0.020				
20	20.020	−0.105	0.020	19.997	−0.114	0.030	−0.009
30				29.991	−0.113	0.030	
40	39.959	−0.099	0.020	39.943	−0.112	0.030	−0.013
50	49.933	−0.095	0.020	49.940	−0.104	0.030	−0.009
60				59.935	−0.108	0.030	
70	69.944	−0.105	0.025	69.908	−0.091	0.030	0.014

5 Linking of the Generator to NMI’s

The HTS is used to calibrate internal transfer chilled mirror hygrometers, but also for customer instruments. One of these instruments is owned by DTI in Denmark and this instrument, a MBW 373 LHX, S/N 06-1236, was calibrated at VSL in 2010 and 2015. The result of the calibration is shown in Table 3, and the two calibrations show the good stability of the instrument, as the drift over 5 years is less than 0.02 °C.

The DTI instrument was also calibrated at three other institutes of which two are National Metrology Institutes: PTB, Germany and NPL, UK. The third institute is the manufacturer of the CMH, MBW, presently the DI for humidity in Switzerland. The results of their calibration together with the uncertainty are given in Table 4, and all calibration data are displayed in Fig. 11.

Table 4 The calibration results of the MBW 373 LHX, S/N 06-1236 chilled mirror hygrometer over the period 2009–2014 by PTB and NPL and the manufacturer MBW (uncertainty, u, stated with k = 1)

Nominal °C	t_d PTB, 2009 °C	$t_{IEC} - t_d$ K	u K	t_d NPL, 2013 °C	$t_{IEC} - t_d$ K	u K	t_d MBW, 2014 °C	$t_{IEC} - t_d$ K	u K
10							10.004	−0.085	0.025
20	20.010	−0.123	0.018	20.240	−0.115	0.020	20.026	−0.097	0.025
30									
40	40.000	−0.105	0.018	39.640	−0.111	0.020	39.962	−0.091	0.025
50									
60							60.121	−0.071	0.025
70	70.000	−0.089	0.018	70.120	−0.089	0.025	70.109	−0.060	0.035

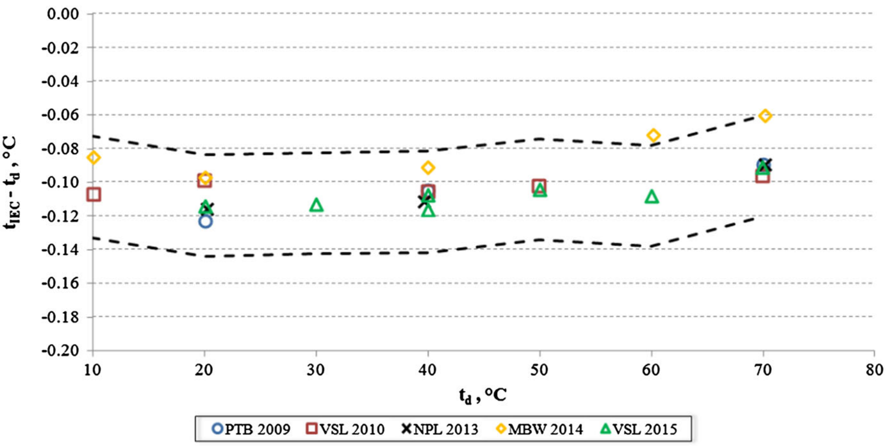

Fig. 11 The calibration results of the MBW 373 LHX, S/N 06-1236 rear mirror over the period 2009–2015 from three NMI's and the manufacturer of the instrument. The *dotted line* is the VSL calibration uncertainty of 2015 ($k = 1$)

Using the 4 data points of the NMI-measurements in 2009, 2010, 2013 and 2015 at 20°C, 50°C and 70°C an average offset, $(t_{\mathrm{IEC}} - t_{\mathrm{d}})_{\mathrm{Avg}}$ was calculated. These results and their experimental standard deviation, s, are shown in Table 5. For each institute the difference the average offset, Δt_{d}, and the normalized error, E_{n}, were obtained via:

$$\Delta t_{\mathrm{d}} = (t_{\mathrm{IEC}} - t_{\mathrm{d}})_{\mathrm{i}} - (t_{\mathrm{IEC}} - t_{\mathrm{d}})_{\mathrm{Avg}}$$
$$E_{\mathrm{n}} = |\Delta t_{\mathrm{d}}|/(2 \cdot \sqrt{(u_{\mathrm{i}}^2 + s^2)})$$

where i = PTB, NPL, VSL and MBW.

For VSL the average of the 2010 and 2015 results was used in the calculation. For all institutes, the E_{n} value is smaller than 1, indicating a good agreement between them.

6 Conclusions

The high-temperature dew-point generator of VSL was originally built in the 1990's and changed from re-circulating mode to single-pass mode. During the years several modifications were made, which improved the uncertainty of the generator. A new validation from January 2016 proves that the CMC uncertainty at 95 °C can be reduced from the present 0.10°C to 0.048°C ($k = 2$).

VSL uses multiple CMH's to confirm the proper operation of both the primary generator and the CMH's within their uncertainties. Investigation of the calibration history of the CMH's showed that these instruments have had an extremely stable performance over more than 1 decade.

Table 5 The results of the comparison between four institutes based on the calibration data of the MBW 373 LHX, S/N 06-1236

Nominal °C	$(t_{\mathrm{IEC}} - t_{\mathrm{d}})_{\mathrm{Avg}}$ K	s K	Δt_{d} PTB K	E_{n} $\mathrm{K{\cdot}K^{-1}}$	Δt_{d} VSL K	E_{n} $\mathrm{K{\cdot}K^{-1}}$	Δt_{d} NPL K	E_{n} $\mathrm{K{\cdot}K^{-1}}$	Δt_{d} MBW K	E_{n} $\mathrm{K \cdot K^{-1}}$
20	−0.114	0.007	−0.009	0.2	0.005	0.1	−0.001	0.03	0.017	0.3
40	−0.106	0.006	0.002	0.04	0.001	0.03	−0.004	0.1	0.015	0.3
70	−0.093	0.008	0.004	0.1	−0.004	0.1	0.004	0.1	0.034	0.5

Using the stable performance of a CMH owned by DTI and calibrated at VSL, PTB, NPL and MBW a link could be made with primary generators in other NMI's. This comparison showed an agreement well within the uncertainty.

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