Int J Thermophys (2008) 29:1644–1651
DOI 10.1007/s10765-008-0426-9

The Humidity Calibration Facility of the National Metrology Institute of South Africa (NMISA)

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Published online: 15 May 2008

Abstract The NMISA Humidity Laboratory maintains the South African national measurement standards for humidity and provides traceability for commercial calibration laboratories. The laboratory performs both relative humidity and dew-point calibrations. The laboratory has been accredited since January 2004 for both parameters by the South African National Accreditation System (SANAS). Industries such as the pharmaceutical industry, power stations, and the food industry make use of the calibration services provided by the laboratory. The facilities and the measurement capabilities of the laboratory are presented in this paper. Two calibration methods are used for the calibration of relative humidity measuring instruments (RH meters). Depending on the type of instrument, they are calibrated against aqueous salt solutions or by comparison with reference RH meters. Measurements are performed over the humidity range 5–95 %rh (Best Measurement Capabilities: 0.4–1.4 %rh). Two chilled-mirror hygrometers are used as reference standards for dew-point calibrations over the range from −75°Cfp to +17°Cdp (Best Measurement Capabilities: 1.5°Cdp to 0.4°Cdp). The laboratory has participated in two international interlaboratory comparisons, one for each accredited parameter. The NMISA Humidity Laboratory is working to reduce its calibration uncertainties for relative humidity instruments: since first accredited in 2004, the best measurement capabilities have been improved from 2.1 %rh to 0.4 %rh for some humidity ranges.

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Keywords Dew point · Measurement capabilities · Relative humidity · National measurement standards

1 Introduction

Prior to 1997, the NMISA Humidity Laboratory used two chilled-mirror hygrometers operating from −15°Cdp to 25°Cdp as reference standards, and could only perform calibrations at ambient conditions (around 20°C and 50 %rh). With the acquisition by mid-1998 of a flow-mixing humidity generator, a chilled-mirror hygrometer operating from −75°Cfp to 20°Cdp, three resistive electrolyte relative humidity meters, 11–90 %rh saturated salt solution capsules and a temperature- and humidity-variable chamber (5–60°C, 10–90 %rh), the calibration capabilities of the laboratory were significantly extended [1]. In 2001, a second −75°Cfp to 20°Cdp chilled-mirror hygrometer and two capacitive polymer RH meters were acquired and, from 2002 to 2005, the addition of unsaturated salt solution ampoules realizing nine relative humidity values in the range from 5 %rh to 95 %rh allowed reduction of the laboratory's relative humidity calibration uncertainties [2,3].

The laboratory has been accredited to ISO/IEC 17025 since January 2004. It is one of three South African laboratories accredited by SANAS (South African National Accreditation System) to perform humidity calibrations, and is the only laboratory in South Africa accredited to calibrate dew-point measuring instruments. The measurement of humidity is important in various industries, such as the paper, pharmaceutical, food, and textile industries. The NMISA Humidity Laboratory supports these industries by developing and maintaining national measurement standards for humidity and by providing a calibration facility for humidity instruments (relative humidity and dew-point hygrometers). Because industry requires increasingly accurate measurements of relative humidity and dew point, the NMISA is working to reduce its calibration uncertainties in these areas.

The Humidity Laboratory calibrates approximately 200 humidity instruments per year. The majority of these instruments are relative humidity hygrometers. The construction of a two-pressure humidity generator is one of the laboratory's current development projects.

2 Laboratory Equipment and Measurement Capabilities

2.1 Dew-Point Measurements

Dew-point calibrations are performed over the range −75°Cfp to +17°Cdp using two chilled-mirror hygrometers as reference standards. The apparatus used for dew-point calibrations is shown in Fig. 1.

Every 2 years, one of the reference dew-point hygrometers is calibrated by a national metrology institute (NMI) that is a signatory of the CIPM "Mutual recognition of national measurement standards" agreement (CIPM MRA), or by an accredited laboratory that is traceable to such an NMI. The hygrometer sent for calibration is alternated. For example, HMS-100 was calibrated in 2001 and 2005, and HMS-110 in 2003.

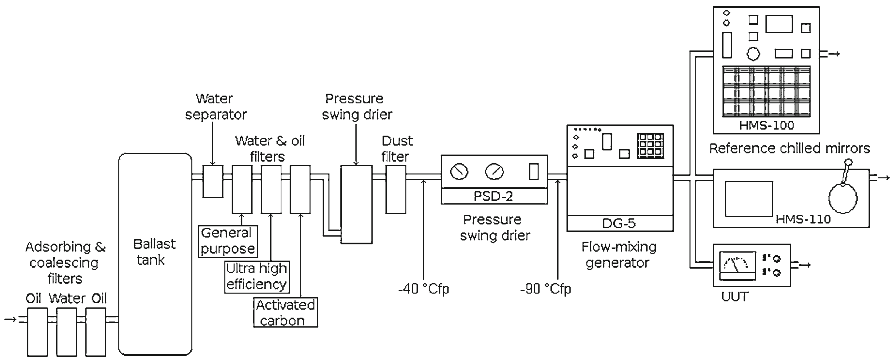

Fig. 1 Air filtering and drying apparatus, flow-mixing humidity generator, and chilled-mirror dew-point hygrometers used at NMISA

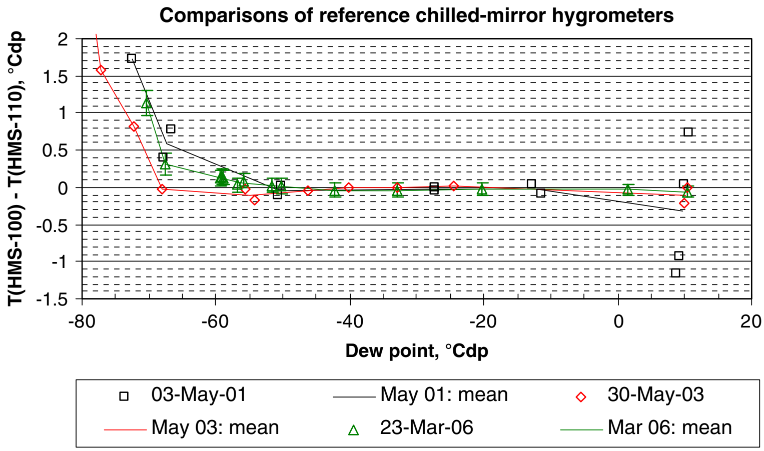

Fig. 2 Results of recent comparisons of NMISA's reference dew-point hygrometers. (Uncertainty bars for the March 2006 comparison show the expanded uncertainties ($k = 2$) of the two hygrometers' calibrations, added in quadrature)

The accuracy of the less recently calibrated hygrometer and the performance of the calibration system as a whole are verified by comparing the two reference hygrometers after one has returned from calibration (Fig. 2).

The increasing difference between HMS-100 and HMS-110 below −60°Cfp is believed to be caused by a leak in the gas path to the sensor of HMS-100. (The effect did not appear when HMS-100 was calibrated in 2005 and 2001, at the same flow rate as is used at NMISA.) The scatter in measured differences at 10°C in May 2001 was caused by instability in the reading of HMS-100 at that temperature (its reading increased by 1.5°C in an hour). The instrument was repaired immediately after this comparison.

NMISA's Best Measurement Capability (BMC) for dew-point calibrations decreases linearly from 1.5°C to 0.4°C in the range −75°Cfp to −60°Cfp and is 0.4°C from −60°Cfp to +17°Cdp.

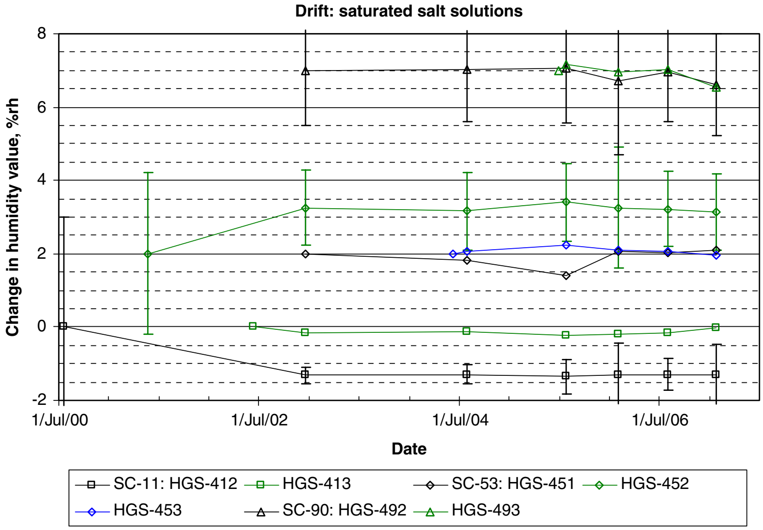

Fig. 3 Calibration history of NMISA's 11 %rh, 53 %rh, and 90 %rh saturated salt solution capsules (53 %rh and 90 %rh values are offset for greater clarity)

2.2 Relative Humidity Measurements

Unsaturated salt solutions (certified by an accredited laboratory traceable to a CIPM MRA signatory) are used as the top-level relative humidity standards. The calibration uncertainties ($k = 2$) of the solutions vary from 0.1 %rh to 1.2 %rh for the ten solutions available in the range 5–95 %rh. They serve as reference standards for the calibration of RH meters and saturated salt solutions (using several of NMISA's calibrated RH meters as transfer standards) (Fig. 3).

NMISA's BMCs for the calibration of salt solution capsules increase from 0.4 %rh to 1.4 %rh in the range 11–90 %rh. The BMCs for the calibration of RH meters against unsaturated salt solutions range from 0.5 %rh to 1.4 %rh from 10 %rh to 95 %rh (Fig. 4).

Relative humidity measuring instruments that cannot be calibrated against the salt solutions, because the sensor is too big to fit into the salt solution capsule or chamber, are calibrated in a large temperature- and humidity-variable chamber by comparison with calibrated RH meters. The calibration uncertainty is then of the order of 3.0 %rh ($k = 2$). The chamber can operate over a humidity range of 10–90 %rh and a temperature range of (5–60)°C, but relative humidity calibrations are only performed at temperatures between (10 and 30)°C at present, because of the unknown uncertainty in the temperature coefficients of the unsaturated salt solutions (and therefore unknown uncertainty in any temperature coefficient measurements performed on NMISA's reference RH meters over a wide temperature range).

3 Development Work Performed in the Laboratory

The Humidity Laboratory has carried out various development projects to improve the quality of the calibrations performed in the laboratory.

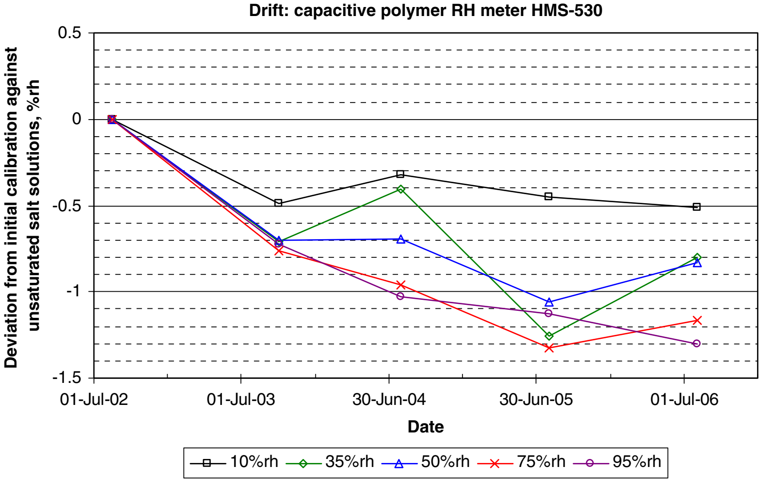

Fig. 4 Calibration history of one of NMISA's relative humidity hygrometers

In 2001, the laboratory's saturated salt solution capsules were compared to the chilled mirror of the temperature- and humidity-variable chamber [4]. The results are shown in Fig. 5. The uncertainties of calibration of the chamber's PRT (measuring air temperature) and chilled mirror, combined with the temperature gradients in the chamber, result in larger combined uncertainties in relative humidity than those of measurements against salt solutions. For this reason, NMISA uses salt solutions as the starting point of traceability for relative humidity.

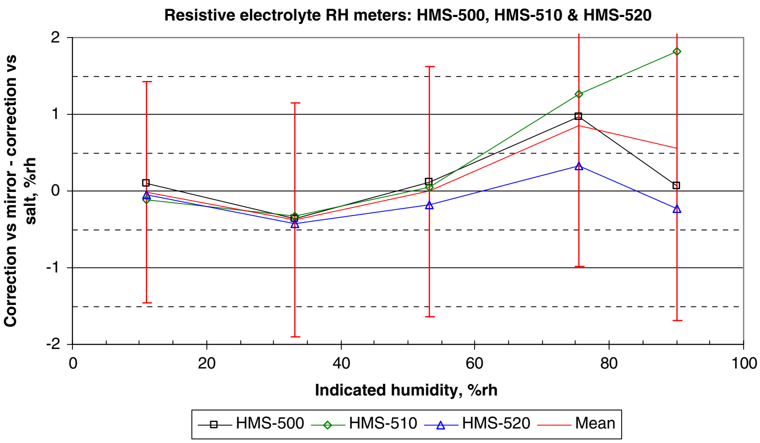

Fig. 5 Comparison of saturated salt solution capsules with a chilled-mirror hygrometer, using three RH meters as transfer artifacts. (Uncertainty bars include the expanded uncertainty of calibration of the chamber's chilled mirror and PRT, and the ESDM (experimental standard deviation of the mean) of the three results at each humidity, but exclude the effect of temperature gradients in the chamber)

In 2006, components of the uncertainty in the calibration of RH meters against unsaturated salt solutions or reference RH meters were evaluated [3]. The effect of leaks in the test chamber was measured and the temperature coefficients, long-term stability, and non-linearity of the reference RH meters were evaluated. As a result, the laboratory's relative humidity BMCs could be reduced.

It is hoped that the NMISA's relative humidity standards may obtain traceability directly from temperature and pressure standards in the future, with the development of a humidity generator. A previously constructed flow-mixing generator using a saturator coil and permeation tube as sources of moist air exhibited instability of the order of 0.2°Cdp at some dew points [5]. It is hoped that a two-pressure generator being studied at present will perform better [6]. An accurate chilled-mirror hygrometer capable of operating from −30°Cfp to +60°Cdp will also be required (to cover the range 5–95 %rh, 5°C to 60°C), to verify the accuracy of the generator.

4 Interlaboratory Comparisons

In 1997, the NMISA participated in a bilateral comparison of dew-point measurement capabilities with INTA (Spain), using a silicon dioxide dew-point hygrometer as the transfer instrument. The results are shown in Fig. 6. The comparison uncertainties were significantly larger than the NMISA's BMCs (0.4°Cfp in this range), resulting from the characteristics of the transfer hygrometer. NMISA participated in EUROMET key comparison P621 in 2006, using two chilled-mirror hygrometers as transfer artifacts over the range −50°Cfp to +20°Cdp. It is hoped that the results of this comparison will allow a reduction in NMISA's dew-point BMCs.

The NMISA participated in a European Cooperation for Accreditation comparison of relative humidity calibration capabilities in 1998 [7]. Two RH meters were mea-

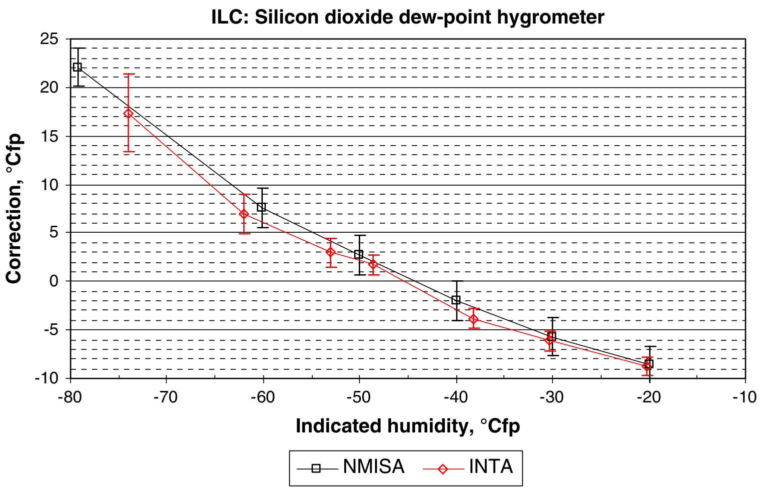

Fig. 6 1997 bilateral comparison of dew-point calibration capabilities (actual dew-point range: −57°Cfp to −29°Cfp)

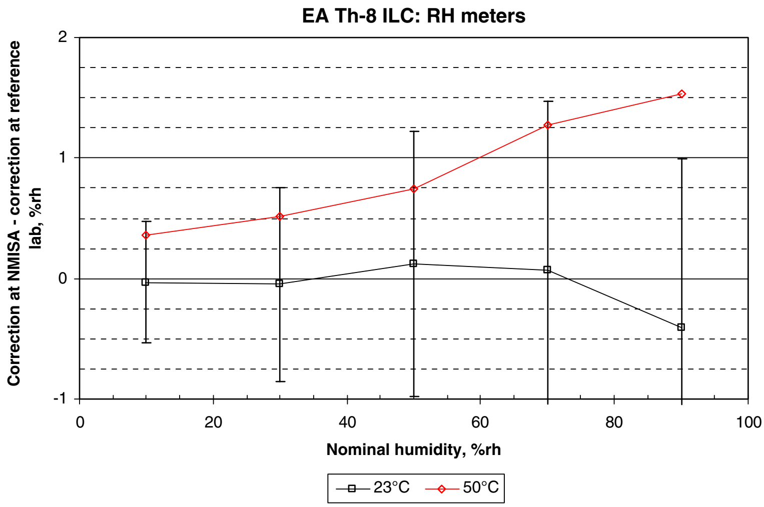

Fig. 7 NMISA's (previously NML) results in the 1998 EA comparison of relative humidity calibration capabilities (Uncertainty bars show NMISA's current BMCs for the calibration of RH meters)

sured from 10 %rh to 90 %rh, one at 23°C and the other at 50°C. NMISA's results are shown in Fig. 7.

5 Future Development

In future, it is hoped that traceability for both relative humidity and dew point can be obtained from temperature and pressure measurements, using humidity generators as primary standards. A generator to be used for relative humidity calibrations is being developed [6]; with the experience gained from this project, it is hoped that an accurate generator working to lower dew points can be developed for use as a dew-point standard.

Acknowledgment The authors wish to thank the South African Department of Trade and Industry for funding the work performed by the laboratory.

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