JISSE
ISSN: 2636-4425

Journal of International Society for Science and Engineering
Vol. 3, No. 1, 09-13 (2021)

JISSE
E-ISSN:2682-3438

Verification the Results of the NIS Hydrometers Calibration system

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ARTICLE INFO

Article history:
Received:2021-01-04
Accepted:2021-02-06
Online:2021-02-06

Keywords:
Density of liquids
Hydrometers Calibration System
Hydrostatic weighing technique.

ABSTRACT

A fully automated hydrometer calibration system at the Egyptian National Institute of Standards (NIS) has been established and realized to be used in measuring the density of the liquids and calibration of the hydrometers in the applicable range from 0.5 g/cm^3 up to 2 g/cm^3. The hydrostatic weighing technique based on Cuckow's method was used in the calibration using the hydrometer calibration system. The distilled water density was used as a reference liquid (Density= 998.2 kg/m^3 at 20 ºC). A comparison between the results obtained from the French National Metrology Institute (LNE) system and the (NIS) system was performed. The En-Value analytical technique based on calculating the reference value between the two institute's results and associated uncertainties was used. The (NIS) system results are satisfactory compared to the measurements' reference value. An expanded uncertainty of about 5.5×10^{-5} g/cm^3 ata confidence level of 95% and (K=2) was achieved using the NIS Hydrometer Calibration System.

1. Introduction

Previously, the standard hydrometers of the National Institute of Standards (NIS) have been calibrated in the range only from 0.700 g/cm^3 up to 1.850 g/cm^3 at the National Metrology Institute (NMI) France[1]. The NIS calibrated hydrometers were used to transfer the traceability to thousands of customers' hydrometers used in density measurements in the dairy, distilling, chemical and petroleum industries.

The NIS performs calibrations for the customers' hydrometers by applying the free-floating comparison technique [2], dealing with several liquids and a set of breakable, fragile reference hydrometers. The used liquids can be absolute or a mixture of liquids such as water, sulfuric acid, and volatile organic solvent to verify density measuring points. Such solution mixtures are difficult and hazardous to verify the calibration points located on the hydrometer stem, especially in the density range above the distilled water.

Personal errors, changes in the surrounding temperature during the calibration process and the liquid meniscus which appears around the hydrometer stem give rise to a large uncertainty in density measurements[3] are reached.

To overcome these problems, an automated Hydrometer Calibration System (HCS), which has a range from 0.5 g/cm^3 up to 2g/cm^3 and based on the hydrostatic weighing technique, was designed, fabricated, evaluated and tested at NIS[4] for hydrometer calibration via Cuckow's method[5]. The NIS automated hydrometer calibration system has the advantage of calibrating all the scale marks on the hydrometer stem with any number of repetitions automatically and even overnight. Any calibration point on the hydrometer's stem can be calibrated using only one calibrating fluid, distilled water, which is user-friendly.

2. The Hydrometers Calibration System of NIS

The established system at NIS [4] for hydrometers calibrations was shown in Figure 1.

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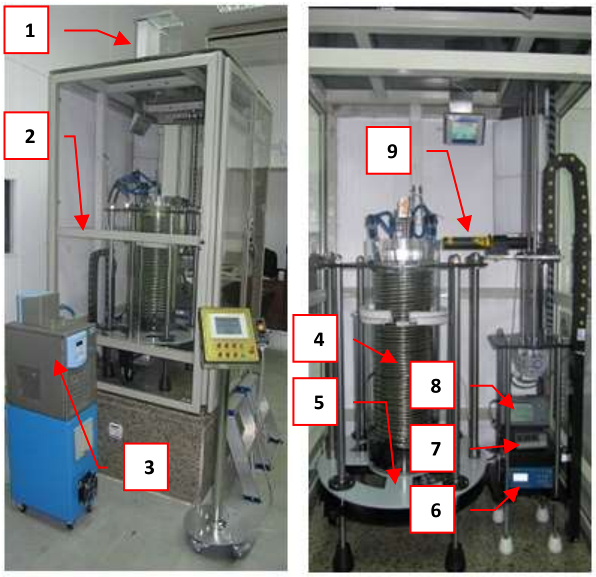

Figure 1: The Hydrometer Calibration System of NIS

The system consists of a high-resolution electronic balance (1) with s capacity of 2.300 kg and readability of 0.0001g placed above the main cabinet (2). For controlling the standard liquid's temperature, a Tamson water bath (3) was used and stability better than ± 0.05K was reached. The double-wall cylinder (4) contains the standard liquid was carried using the mechanical lifter (5). For measuring the ambient condition, a system consisting of; absolute digital pressure indicator (6) with a readability of 0.1 Pa for measuring the ambient pressure. PT100 thermometer with digital temperature indicator (7) of readability 0.001℃ is used for standard liquid temperature measurement. Hygrometer (8)measured the ambient temperature with a readability of 0.0001 ºC and measured the ambient humidity with a readability of 0.01 %for air density calculations [6].A diode laser source (9) is for positioning the standard liquid surface to the under calibration scale mark to be calibrated on the hydrometer stem.

3. The Hydrometers Calibration System of LNE

The apparatus for calibration of the hydrometers at LNE[7]shown in Figure 2consisting of a comparator balance of 1.1 kg capacity and 10 µg readability (1).A thermostatic bath with water temperature controller (2) and A Pyrex glass vessel contains the reference liquid (Kerdanne oil)isolated from the bathwater (3).a suspension system was used to hang the hydrometer underside the comparator balance (4).A platinum resistance thermometer (Pt25) was used for measuring the temperature of the reference liquid (5).

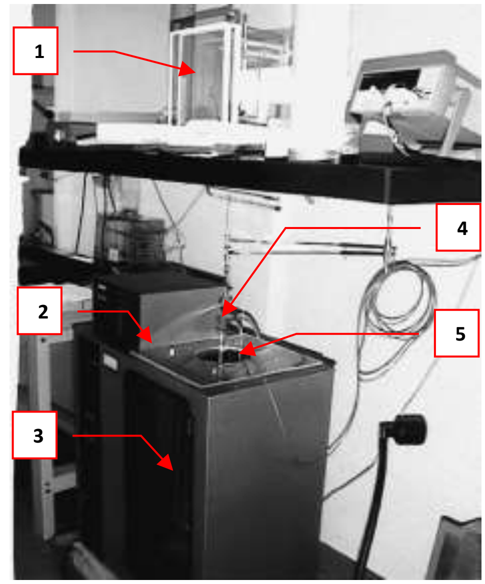

Figure 2: The Hydrometer Calibration System of LNE

4. Method Used in the Hydrometer Calibration

For the calibration laboratories, a method to determine the density of the reference liquid (ρ_L) represented by a given calibration point on the hydrometer range was derived by Cuckow (Cuckow's Method). Forces balance equations are written for a hydrometer in three cases [5]:

Case (1) the hydrometer is freely floating in a liquid of density (ρ_L) at reference temperature T_0,

Case (2) weighing the hydrometer in air and

Case (3) weighing the hydrometer while it is immersed to the same level at the case (1), but in a known density liquid of (ρ_T) at temperature (T_T). It is providing that (T_T) must be different from the hydrometer reference temperature (T_0).

The equation for ρ_L can be driven by solving the three equations simultaneously:

$$\rho_L = \frac{\{\rho_T[1+\beta(T_T-T_0)]-\rho_{a2}\}(\alpha O_2+\pi D Cos\Theta_L/g)}{\alpha O_2-\alpha O_3+\Gamma_T/g+m_s-V_s\rho_T[1+\beta_s(T_T-T_0)]}+\rho_{a2} \qquad (1)$$

When calibrating a hydrometer [8]**,** which has a density measuring range less dense than distilled water, stainless steel ring masses are placed on the hydrometer's bulb to make it sink. The masses have a ring shape of known mass and volume measured using the volume comparator (VC1005). During the hydrometer calibration process, these masses are used to submerge the hydrometer in the reference liquid as in (1)

Calculate the hydrometer corrections at the calibration points to be calibrated on the hydrometer stem (2). The values of A and B can be obtained from equation 3 and 4.

$$C_L = A + B\gamma_L Cos\theta_L \qquad (2)$$

$$A = \frac{\{\{\rho_T[1+\beta(T_T-T_0)]-\rho_{a2}\}(\alpha O_2+\Gamma_L/g)\}}{\alpha O_2-\alpha O_3+\Gamma_T/g+m_s-V_s\rho_T[1+\beta_s(T_T-T_0)]}+\rho_{a2}-R \qquad (3)$$

$$B = (A + R - \rho_{a2})\frac{\pi D}{\alpha O_2 g} \qquad (4)$$

5. Measurement Results

The measurements were performed at a reference temperature of 20 ℃. The reference liquid (Distilled water) was checked in the two coaxial cylinders at six depths (10, 20, 30, 40, 50and 60 cm) from the free surface of the distilled water. A digital temperature indicator (PT100) sensor with a resolution of 0.001 ºC has been used to measure the temperature distribution inside the two coaxial cylinders. The results of the temperature distribution shown in Figure 3.

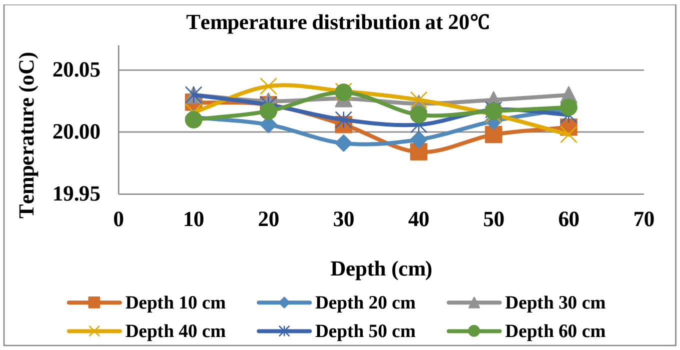

Figure 3: The distilled water temperature distribution at 20℃

The variation was less than ± 0.05 °C at the depth from 10 cm up to 60 cm, which is the weighing region of the hydrometers.

Three standard hydrometers are used for performing this comparative study. These hydrometers were carefully selected to covers wide range of density measurements. The obtained measurements result from the system of NIS as well as the LNE system are given below in Tables 1, 2 and 3. The first hydrometer is ranging from 0.700 – 0.720 g/cm^{3,} the result of which is given in Table 1. The second hydrometer is ranging from1.494 – 1.524g/cm^{3,} the result of which is given in Table 2. The third hydrometer is ranging from1.626 – 1.850g/cm^{3,} the result of which is given in Table 3.

Table 1: The data and the calibration results of the first hydrometer

Measurement Range (g/cm^3)	0.700 – 0.720		
resolution (g/cm^3)	0.0001		
Serial no.	A6		
	Calibrated Points (g/cm^3)	Corrected Value (g/cm^3)	Expanded Uncertainty (g/cm^3)
HCS-NIS Results	0.707000	0.706947	3.0×10^{-5}
	0.711000	0.710953	3.0×10^{-5}
	0.715000	0.714921	3.0×10^{-5}
LNE Results	0.707000	0.706949	3.0×10^{-5}
	0.711000	0.710960	3.0×10^{-5}
	0.715000	0.714927	3.0×10^{-5}

Table 2. The data and the calibration results of the second hydrometer

Measurement Range (g/cm^3)	1.494 – 1.524		
Resolution (g/cm^3)	0.0002		
Serial no.	A43		
	Calibrated Points (g/cm^3)	Corrected Value (g/cm^3)	Expanded Uncertainty (g/cm^3)
HCS-NIS Results	1.496000	1.496040	5.3×10^{-5}
	1.509000	1.509043	5.4×10^{-5}
	1.522000	1.522041	5.5×10^{-5}
LNE Results	1.496000	1.496042	4.0×10^{-5}
	1.509000	1.509051	4.0×10^{-5}
	1.522000	1.522046	4.0×10^{-5}

Table 3. The data and the calibration results of the third hydrometer

Measurement Range (g/cm^3)	1.626 – 1.850		
Resolution (g/cm^3)	0.0002		
Serial no.	A56		
	Calibrated Points (g/cm^3)	Corrected Value (g/cm^3)	Expanded Uncertainty (g/cm^3)
HCS-NIS Results	1.848000	1.848049	3.4×10^{-5}
	1.838000	1.838013	3.4×10^{-5}
	1.828000	1.828035	3.4×10^{-5}
LNE Results	1.848000	1.848038	4.0×10^{-5}
	1.838000	1.838007	4.0×10^{-5}
	1.828000	1.828021	4.0×10^{-5}

6. Comparing the Results of LNE and NIS

The results were compared using the Normalizing Error (E_n-Value) [9]**.**It was used to compare the result values and associated standard uncertainty of the HCS-NIS results and LNE results to the reference value and associated uncertainty.
In inter-laboratory comparisons the reference value method is used to compare participant's measurement results and their associated uncertainty values[10]. The reference value method was used here to evaluate the NIS hydrometer calibration system's performance with respect to LNE Results.

6.1. Calculating the reference value (y)

The reference value(y)[11] is calculated for the density measurements obtained from the two techniques, using the inverses of the squares of the associated standard uncertainties as in (5). The reference value and associated uncertainty are tabulated in Table 6.

$$y = \frac{\sum_{i=1}^{i=2} \frac{x_i}{u^2(x_i)}}{\sum_{i=1}^{i=2} \frac{1}{u^2(x_i)}} \qquad (5)$$

The reference value uncertainty *u(y)* is calculated using as in **(6)**:

$$\frac{1}{u^2(y)} = \sum_{i=1}^{i=2} \frac{1}{u^2(x_i)} \quad (6)$$

Table 4. The results of the reference value (y)

	Calibrated Points (g/cm^3)	Reference value (y) (g/cm^3)	u_y (g/cm^3)
Hyd. (1)	0.707	0.706948	2.1×10^{-05}
	0.711	0.710957	2.1×10^{-05}
	0.715	0.714924	2.1×10^{-05}
Hyd. (2)	1.496	1.496041	3.2×10^{-05}
	1.509	1.509048	3.2×10^{-05}
	1.522	1.522044	3.2×10^{-05}
Hyd. (3)	1.8480	1.848044	2.6×10^{-05}
	1.8380	1.838010	2.6×10^{-05}
	1.8280	1.828029	2.6×10^{-05}

6.2. Performance Evaluation using Normalizing Error (En)

The results obtained from the hydrometer calibration system were compared to the reference value obtained from both the HCS-NIS results and LNE results based on normalizing error (En) [9]. E_n value was calculated using as in (7) and the results are shown in Figure 4.

$$E_n = \frac{x_{Mean} - x_{ref}}{\sqrt{(U_{Mean})^2 + (U_{ref})^2}} \quad (7)$$

Evaluation criteria when:

- |En| <= 1 the results are satisfactory.
- |En| > 1 the results are unsatisfactory.

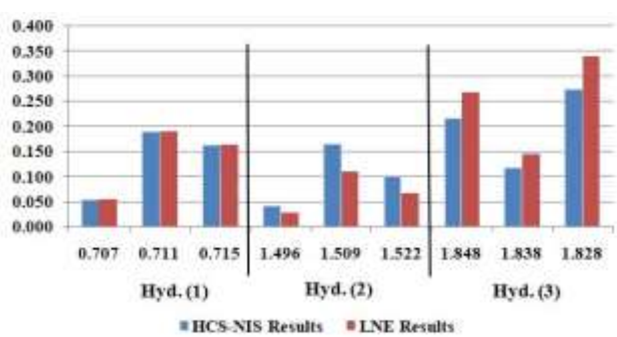

Figure 4: En-Values between HCS-NIS Results and LNE Results

All the results of HCS-NIS are satisfactory compared to the reference value of the measurements.

7. Conclusions

The hydrometer calibration system (HCS) at the (NIS) Egypt has been established for the hydrometers calibrations range from 0.5g/cm^3 up to 2 g/cm^3.A comparison for the measurement performance of the HCS has been carried out by comparing the obtained results versus LNE's results. The stability of the reference liquid (Distilled water) used in the HCS was checked at six different heights in the two coaxial cylinders and found that the variation was about ± 0.05 ℃. The En-Value analytical method was used to compare both NIS and LNE results in the calculated reference value. An expanded uncertainty (K=2) of about 0.55 ppm was achieved using the NIS Hydrometer Calibration System.

Acknowledgment

The authors thank their various collaborators, especially those who are authors of references cited here.

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Abbreviation and symbols

NIS	The National Institute of Standards
LNE	Laboratoire National de Metrologie et d'Essais
NMI	National Metrology Institute
ρ_T	The distilled water density (998.2 kg/m3 at 20 °C)
β	Volumetric thermal expansion coefficient of the hydrometer material
T_T	The distilled water temperature
T_0	Hydrometer reference temperature
ρ_{a2}	Density of the air at the weighting time in air
α	The balance calibration coefficient,
O_2	The hydrometer weight in air,
D	Hydrometer stem diameter at the calibration point,
γ_L	surface tension of the liquid in which the hydrometer is used,
O_3	Output from the balance for the hydrostatic weighing.
γ_T	Surface tension of the distilled water.
ms	Mass of the stainless-steel ring masses added in order to sink the hydrometer,
Vs	volume of the stainless-steel weight, and
βs	Volumetric thermal expansion coefficient of the stainless-steel ring masses.
R	The hydrometer reading at the calibrated scale mark
x_i	The obtained values by the two institutes.
$u(x_i)$	The estimated standard uncertainty for the obtained value by the institutes.
x_{Mean}	The mean value for the results
x_{ref}	The reference value (assigned value) for the results
U_{Mean}	The reported expanded uncertainty of the mean value
U_{ref}	The reported expanded uncertainty of the reference value