Final Report

Supplementary Comparison

Hydrometers Calibrations

AFRIMETS.M. D-S4

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AFRIMETS.M.D-S4 "Hydrometers Calibrations"

1. Introduction

A supplementary comparison for hydrometers calibration using Cuckow's method **[1-2]** was proposed during the 10th AFRIMETS Committee for Mass and Related Quantities (TCM) meeting held on 25th - 26th July 2016 at the NIS, Egypt. Two laboratories only from AFRIMETS, NIS and NMISA, had the capacity to carry out calibration of hydrometer using Cuckow's method and accepted to take part in the comparison. Since no laboratory from AFRIMETS had participated in the CCM.D_K4 **[1]**, a supplementary comparison was therefore decided upon. Generously, Centro Nacional de Metrologia (CENAM) accepted to participate in the comparison. This comparison designated as AFRIMETS. M. D-S4 was coordinated by the National Institute of Standards (NIS, Egypt) supported by all participants. The corrections to be applied to three stated scale readings at 20 °C of different transfer standards in the density range between 0.600 g/cm^3 to 1.500 g/cm^3 was determined. NIS carried out the measurement at the beginning and at the end of the comparison.

Each laboratory was to determine the corrections to be applied to three stated scale readings at 20 °C of different transfer standards in the density range between 0.600 g/cm^3 to 1.500 g/cm^3.

The measurements were completed in January 2019. Draft A of the report was made available in July 2019.

2. Participant laboratories

The participant laboratories are listed in table 2.1.

Table 2.1 List of the participating NMIs and technical contacts.

Laboratory	Country Code	Contact Person
National Institute of Standards (NIS) Tersa St., El-Haram, El-Giza, Egypt P. O.Box 136 Giza-Code 12211	EG	Mohamed Hamdy Mohamed Eng.MohammedHamdi@yahoo.com Tel.: +2 01001929796 Fax: +202 33867452
Centro Nacional de Metrologia (CENAM) Km 4.5 Carretera a los Cués, Mpio El Marqués, Querétaro –MEXICO C.P. 76246	MX	Luis Omar Becerra lbecerra@cenam.mx Tel.: +52 442 2 110573 +52 442 2 110500 to 04 ext. 3602 Fax: +52 442 2 110568
National Metrology Institute of South Africa (NMISA) NMISA Building 5, CSIR campus, Meiring Naude Road, Brummeria, Pretoria, 0184	ZA	Thomas Mautjana tmautjana@nmisa.org Tel.: +27 12 841 4798 Fax: +27 12 841 2131

3. Transfer Standards (Hydrometer samples)

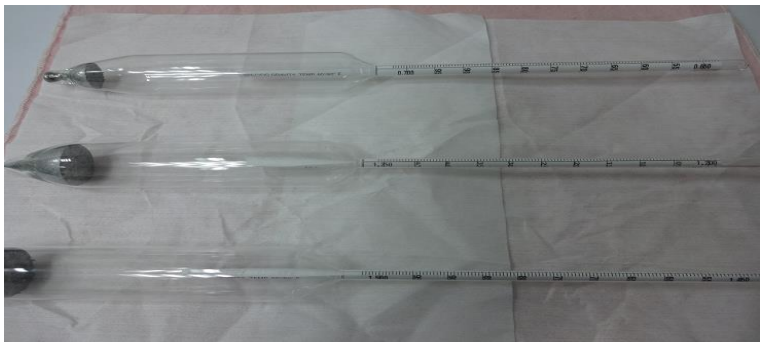
For the comparison NIS supplied a set of three specific gravity hydrometers as artifacts to be used as transfer standards at 20 °C. However, hydrometer 3 broke before CENAM and NMISA could take measurements, while Hydrometer 2 broke before NIS could take the last measurements. Each hydrometer had a scale division of 0.0005 g/cm^3 working within the range 0.600 g/cm^3 to 1.500 g/cm^3.

Table 3.1 Technical information related to the reference hydrometers used as transfer standards (TS) in comparison.

	Hydrometer 1	Hydrometer 2	Hydrometer 3
Range (g/cm^3)	0.6500-0.7000	1.2000-1.2500	1.4500-1.5000
Scale division (g/cm^3)	0.0005	0.0005	0.0005
Manufacture	CHASE-USA	CHASE-USA	CHASE-USA
Nominal values for calibration	0.6950, 0.6750, 0.6550	1.2450, 1.2250, 1.2050	1.4950, 1.4750, 1.4550
Reference surface tension (mN/m)	20, 19, 18	55	
Reference temperature	20 ˚C	20 ˚C	20 ˚C
Cubic thermal expansion coefficient (˚C^{-1})	$25 \times 10^{-6} \pm 2 \times 10^{-6}$		
Hydrometer nominal mass	41 g	83 g	106 g

Photograph 3.1

Transfer Standards (Hydrometers)



4. Circulation and date of measurements

Each laboratory was responsible for receiving the Transfer Packages, calibration then sending them to the next participant according to the schedule.

CENAM made the measurements after NIS who did the calibration at the beginning of the circulation of the hydrometers and after the hydrometers came back from NMISA.

Table 4.1
Dates of arrival of the travelling standards

National Metrology Institute	Date of arrival
NIS	----
CENAM	April 2018
NMISA	August 2018
NIS	December 2018

5. Traceability of results reported by participants

For the calibration of the hydrometers, all laboratories used their own hydrostatic weighing system. Participant laboratories determined the corrections to the specific indications (at 20 ºC) of the travelling standards by Cuckow's method **[1- 2]**.

Table 5.1 shows the specifications of liquids used by participants and the traceability of the densities as well as the surface tensions reported by the participants.

The table 5.2 shows the Thermostat system, balances, mass standards used, and traceability of weighing reported by the participants.

Table 5.1
Liquids used by participants

Acronym	Liquid	Density and surface tension Standard	Traceability
NIS	Distilled water Density: 998 kg/m^3 Surface Tension: 72 mN/m	Density: Density standard made of Silicon (Sphere shape). Surface tension: Ring method	Density: NIS-Egypt Surface tension: Standard method
CENAM	Pentadecane Density: 769 kg/m^3 Surface Tension: 27 mN/m	Density: Density standard made of zerodur (Sphere shape). Surface tension: Ring Method (Commercial tensiometer)	Density: PTB-Germany Surface tension: Standard method, input parameters calibrated at CENAM
NMISA	Distilled water Density: 998 kg/m^3 Surface Tension: 72 mN/m	Density: Stainless steel weights (OIML shape) Surface tension: Ring method (Commercial tensiometer)	Density: NMISA Surface tension: Standard method

Table 5.2

Thermostat system, balances and mass standard used by participants

Acronym	Thermostat system	Balances and mass standard	Traceability
NIS	Double wall Cylinder Tamson Liquid temperature controller (TLC 15-5) and special designed water bath of maximum capacity: 150 L Temperature range: -15 °C / 60 °C Stability at 20 °C: 0,002 °C	**Balance:** Mettler Toledo Type XP2004S / d=0,1 mg Mass standard: Weights OIML E_2	NIS
CENAM	Tamson Bath Maximum capacity: 70 L Temperature range: 10 °C / 50 °C Stability at 20 °C: 0,002 °C	**Balance:** Mettler Toledo Type AT400 / d=0,1 mg Mass standard: Weights OIML E_2	CENAM
NMISA	Tamson Bath Maximum capacity: 70 L Temperature range: -40 °C / 80 °C Stability at 20 °C: 0,02 °C	**Balance:** Sartorius Type R300S / d=0,1 mg **Balance used for weighing in liquid:** Mettler Toledo XPE205/ d=0,01 mg Mass standard: Weights OIML E_2	NMISA

6. Results and uncertainties as reported by participants

For each hydrometer, the protocol specified three nominal values for which the participants had to report the density corrections and the associated uncertainties at the specific temperature of 20 ºC.

The corrections and their uncertainties reported by participants are shown in the table 6.1, table 6.2 and table 6.3.

Although the reproducibility of each artifact was not known, it is considered that hydrometers are instruments which have long-term stability, for that reason the reproducibility is considered negligible in this comparison.

Table 6.1
Hydrometer 1 (0.6500-0.7000)
Corrections and associated uncertainties reported by participants.

Calibration points	**0.69500**		**0.67500**		**0.65500**	
NMIs	**Correction @ 20 °C g/cm³**	**U, k=2 g/cm³**	**Correction @ 20 °C g/cm³**	**U, k=2 g/cm³**	**Correction @ 20 °C g/cm³**	**U, k=2 g/cm³**
NIS	-0.000458	0.000054	-0.000677	0.000054	-0.000492	0.000054
CENAM	-0.000432	0.000044	-0.000654	0.000044	-0.000447	0.000044
NMISA	-0.000502	0.000119	-0.000692	0.000119	-0.000599	0.000119
NIS	-0.000444	0.000057	-0.000661	0.000057	-0.000479	0.000057

Table 6.2
Hydrometer 2 (1.2000-1.2500)
Corrections and associated uncertainties reported by participants.

Calibration points	1.2450		1.2250		1.2050	
NMIs	**Correction @ 20 °C g/cm³**	**U, k=2 g/cm³**	**Correction @ 20 °C g/cm³**	**U, k=2 g/cm³**	**Correction @ 20 °C g/cm³**	**U, k=2 g/cm³**
NIS	-0.001546	0.000069	-0.001520	0.000069	-0.001450	0.000069
CENAM	-0.001568	0.000052	-0.001505	0.000052	-0.001475	0.000052
NMISA	-0.001967	0.000064	-0.001564	0.000064	-0.001926	0.000064
	BROKEN					
NIS	//	//	//	//	//	//

Table 6.3
Hydrometer 3 (1.4500-1.5000)
Corrections and associated uncertainties reported by participants.

Calibration points	1.4950		1.4750		1.4550	
NMIs	**Correction @ 20 °C g/cm³**	**U, k=2 g/cm³**	**Correction @ 20 °C g/cm³**	**U, k=2 g/cm³**	**Correction @ 20 °C g/cm³**	**U, k=2 g/cm³**
NIS	-0.001584	0.000074	-0.001394	0.000074	-0.001451	0.000074
	BROKEN					
CENAM	//	//	//	//	//	//
NMISA	//	//	//	//	//	//
NIS	//	//	//	//	//	//

The uncertainty contributions of the density corrections assigned by each NMI are shown in the Annex A, according to the format established in the comparison SIM.M.D-S1 **[3]**.

The Figures from 6.1 to 6.5 presents the graphs of the results as reported by participants

Hydrometer 1 (0.6500-0.7000)

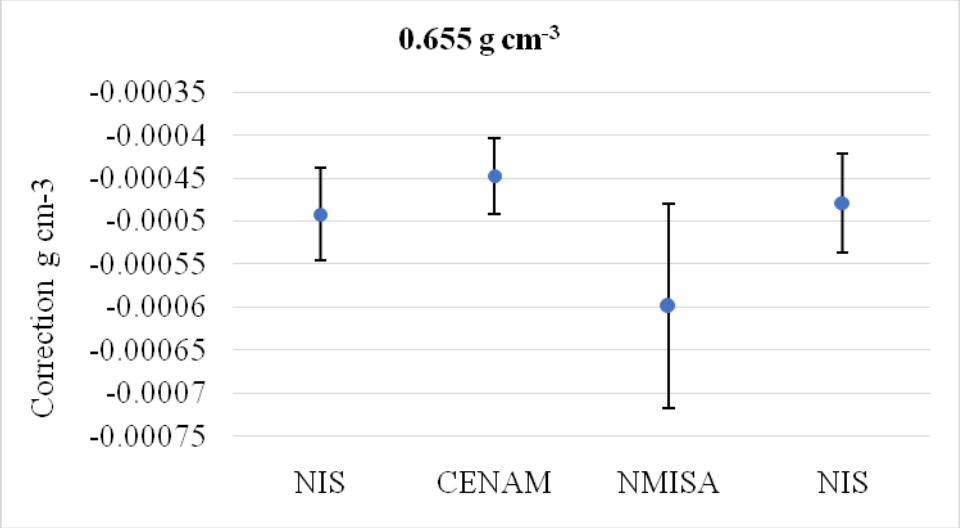


Fig. 6.1 Corrections and associated uncertainties reported by participants for point 0.655 g cm^{-3}

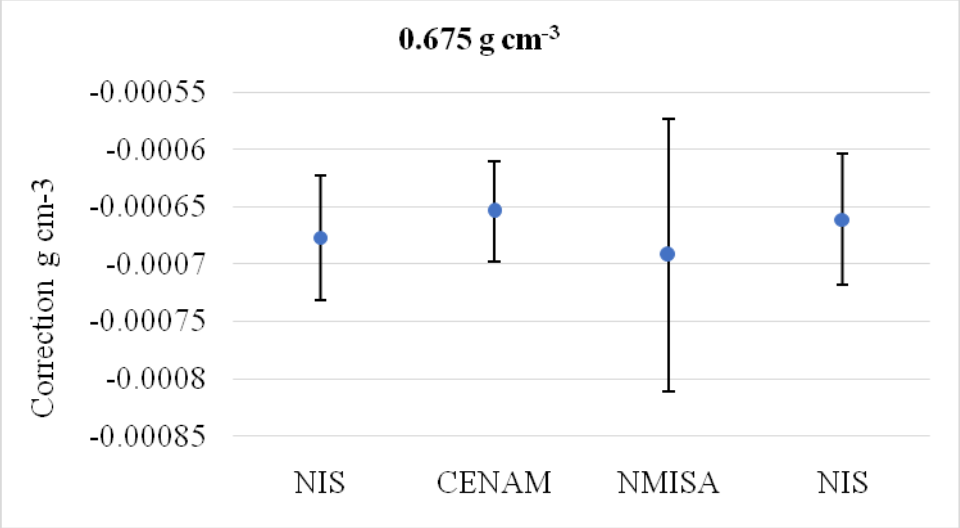


Fig. 6.2 Corrections and associated uncertainties reported by participants for point 0.675 g cm^{-3}

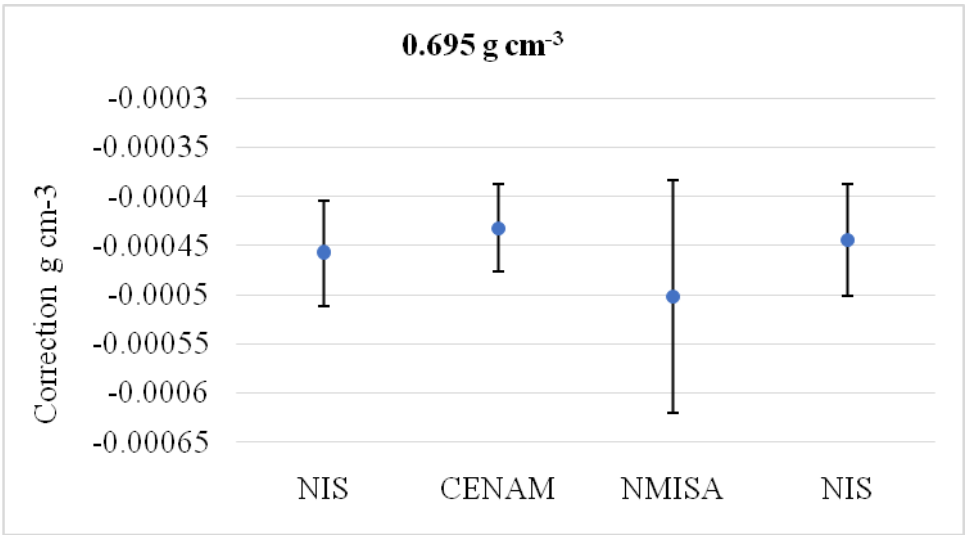


Fig. 6.3 Corrections and associated uncertainties reported by participants for point 0.695 g cm^{-3}

Hydrometer 2 (1.2000-1.2500)

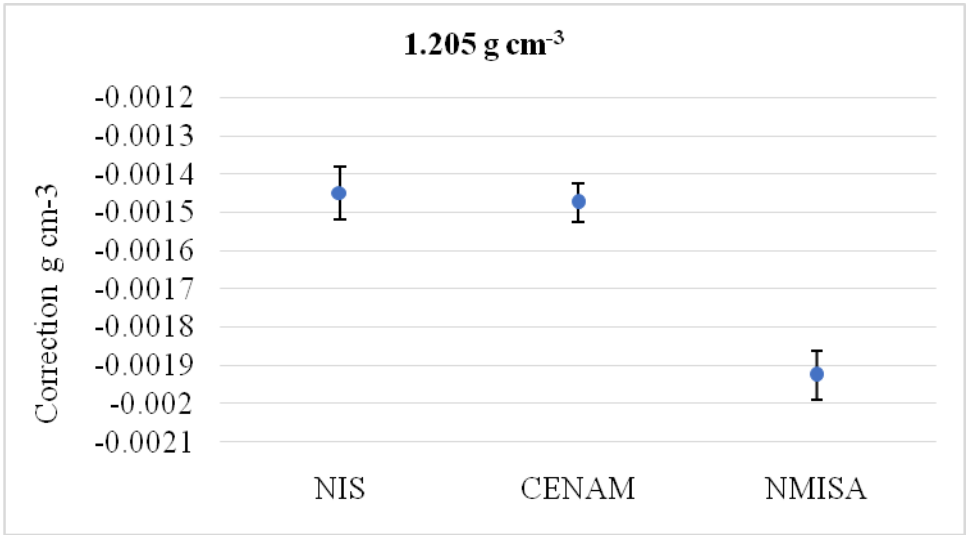


Fig. 6.4 Corrections and associated uncertainties reported by participants for point 1.205 g cm^{-3}

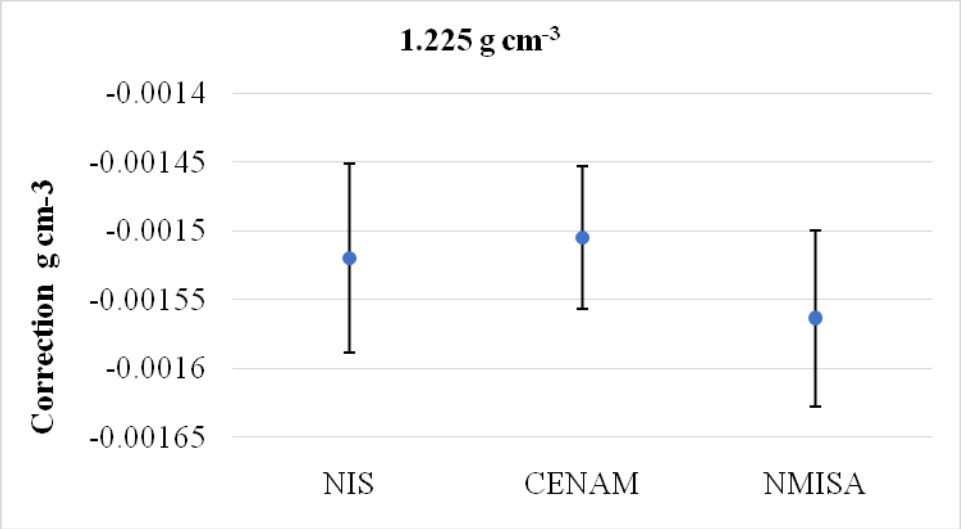


Fig. 6.5 Corrections and associated uncertainties reported by participants for point 1.225 g cm^{-3}

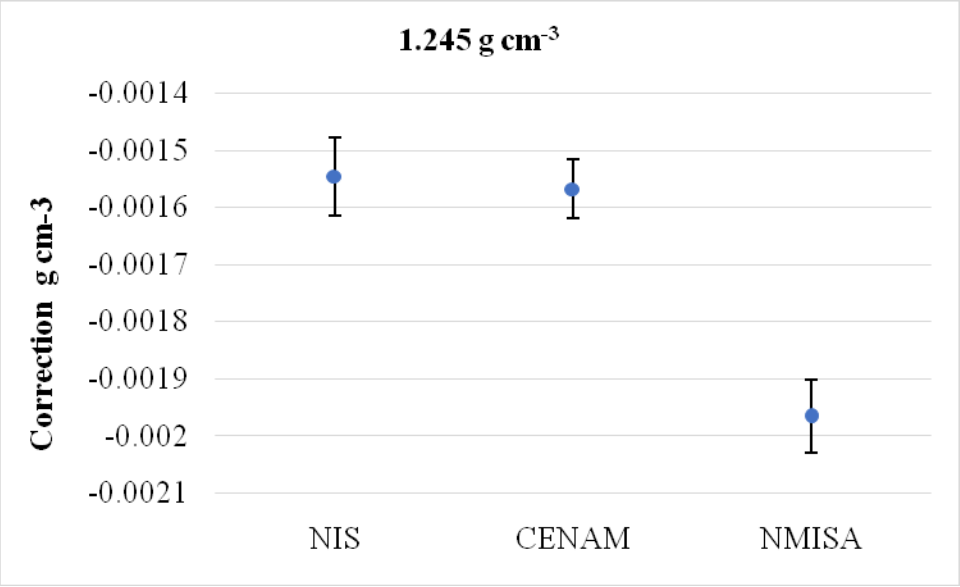


Fig. 6.6 Corrections and associated uncertainties reported by participants for point 1.245 g cm^{-3}

7. Degree of equivalence between participant laboratories:

In order to compare results reported by participants, density differences among results reported by participants were calculated as follows **[4]**,

$$d_{i,j} = X_i - X_j \quad (1$$

were

$d_{i,j}$ is the degree of equivalence between laboratory *i* and laboratory *j*

X_i is the result (correction) reported by laboratory *i*

X_j is the result (correction) reported by laboratory *j*

The expanded uncertainty associated to these differences are calculated as,

$$U(d_{i,j}) = 2\sqrt{u(X_i)^2 + u(X_j)^2} \quad (2$$

$U(d_{i,j})$ is the expanded uncertainty of the difference between laboratory *i* and laboratory *j*

$u(X_i)$ is the standard uncertainty associated to the result (correction) reported by laboratory *i*

$u(X_j)$ is the standard uncertainty associated to the result (correction) reported by laboratory *j*

The normalized error was calculated for each reported value of participants, as follows **[5]**,

$$E_n = \frac{|d_{i,j}|}{U(d_{i,j})} \quad (3$$

With the following criterion,

$$E_n \leq 1$$

If this criterion is accomplished, means that the results are consistent. If not, means that this pair of results are not consistent among them.

The normalized errors are listed in Tables 7.3 to 7.18.

The correction values and associated uncertainty of CENAM were taken as reference values. NIS and NMISA were compared to these values.

Hydrometer 1 (0.6500-0.7000)

Nominal value 0.655 g cm^{-3}

Table 7.1

$d_{i,j}$ Degree of equivalence for 0.655 g cm^{-3}

NMIs	NIS	NMISA
CENAM	0.00005	-0.00015

Table 7.2

$U(d_{i,j})$ expanded uncertainty of the difference between laboratories in g cm^{-3} for 0.655 g cm^{-3}

NMIs	NIS	NMISA
CENAM	0.00007	0.00013

Table 7.3

*E*n-Values between the participants for 0.655 g cm^{-3}

NMIs	NIS	NMISA
CENAM	0.65	1.20

Nominal value 0.675 g cm^{-3}

Table 7.4

$d_{i,j}$ Degree of equivalence for 0.675 g cm^{-3}

NMIs	NIS	NMISA
CENAM	0.00002	-0.00004

Table 7.5

$U(d_{i,j})$ expanded uncertainty of the difference between laboratories in g cm^{-3} for 0.675 g cm^{-3}

NMIs	NIS	NMISA
CENAM	0.00007	0.00013

Table 7.6

*E*n-Values between the participants for 0.675 g cm^{-3}

NMIs	NIS	NMISA
CENAM	0.33	0.30

Nominal value 0.695 g cm^{-3}

Table 7.7

$d_{i,j}$ Degree of equivalence for 0.695 g cm^{-3}

NMIs	NIS	NMISA
CENAM	0.00003	-0.00007

Table 7.8

$U(d_{i,j})$ expanded uncertainty of the difference between laboratories in g cm^{-3} for 0.695 g cm^{-3}

NMIs	NIS	NMISA
CENAM	0.00007	0.00013

Table 7.9

*E*n-Values between the participants for 0.695 g cm^{-3}

NMIs	NIS	NMISA
CENAM	0.37	0.55

Hydrometer 2 (1.2000-1.2500)

Nominal value 1.205 g cm^{-3}

Table 7.10

$d_{i,j}$ Degree of equivalence for 1.205 g cm^{-3}

NMIs	NIS	NMISA
CENAM	-0.00003	-0.00045

Table 7.11

$U(d_{i,j})$ expanded uncertainty of the difference between laboratories in g cm^{-3} for 1.205 g cm^{-3}

NMIs	NIS	NMISA
CENAM	0.00009	0.00008

Table 7.12

*E*n-Values between the participants for 1.205 g cm^{-3}

NMIs	NIS	NMISA
CENAM	0.29	5.47

Nominal value 1.225 g cm^{-3}

Table 7.13

$d_{i,j}$ Degree of equivalence for 1.225 g cm^{-3}

NMIs	NIS	NMISA
CENAM	0.00002	-0.00006

Table 7.14

$U(d_{i,j})$ expanded uncertainty of the difference

between laboratories in g cm^{-3} for 1.225 g cm^{-3}

NMIs	NIS	NMISA
CENAM	0.00009	0.00008

Table 7.15

*E*n-Values between the participants for 1.225 g cm^{-3}

NMIs	NIS	NMISA
CENAM	0.17	0.72

Nominal value 1.245 g cm^{-3}

Table 7.16

$d_{i,j}$ Degree of equivalence for 1.245 g cm^{-3}

NMIs	NIS	NMISA
CENAM	-0.00002	-0.00040

Table 7.17

$U(d_{i,j})$ expanded uncertainty of the difference between laboratories in g cm^{-3} for 1.245 g cm^{-3}

NMIs	NIS	NMISA
CENAM	0.00009	0.00008

Table 7.18

*E*n-Values between the participants for 1.245 g cm^{-3}

NMIs	NIS	NMISA
CENAM	0.25	4.84

8. Conclusions:

The main objectives of this AFRIMETS comparison were the evaluation of the degree of equivalence between AFRIMETS NMIs (NIS and NMISA) and SIM NMI (CENAM) in the calibration of hydrometers within the range of 0.6000 g/cm^3 to 1.5000 g/cm^{3}at 20 °C.

In order to reach such objectives, one set of three hydrometers were circulated between three NMIs. All measurements were carried out from April 2018 to December 2018.

The Hydrometer 3 (1.450-1.500) was broken and it could not be measured by participants, which are the range of the comparison was only from 0.600 g/cm3 to 1.250 g/cm3 at 20 °C. Although hydrometer 2 was measured by all the participants, it could not be remeasured by NIS at the end as it was also broken.

For the measurements, each laboratory used their own hydrostatic weighing system with their own respective standard liquid such as: Distilled water and Pentadecane.

The traceability of the density standard liquids is either from PTB's density standard for CENAM, or from different formulae to calculate the density of water (Tanaka's formula and other formulae).

The calibration values and its associated uncertainty obtained by CENAM were taken as a reference values.

As result of this comparison, results of NIS and CENAM in both ranges 0.655 to 0.695 g/cm^3 and 1.205 to 1.245 g/cm^3 were consistent among them.

NMISA results were consistent with the reference values of CENAM in the nominal values of 0.675 g/cm^3, 0.695 g/cm^3 and 1.225 g/cm^3 but, NMISA and CENAM

results were not consistent in the nominal values of 0.655 g/cm^3 (normalized error of 1.2), 1.205 g/cm^3 (normalized error of 4.84), and 1.245 g/cm^3 ((normalized error of 5.47).

The authors would like to acknowledge the kind assistance of all the colleagues in the participating laboratories for helping this comparison to run so smoothly.

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- Bongani Ndlovu, NMISA
- Daniel Mabena, NMISA
- Arturo A. Daued, CENAM
- Víctor A. Servin, CENAM

References:

[1] Lorefice, S. - Key comparison CCM.D_K4 ―Hydrometer‖ Project: Comparison of the calibration of high-resolution hydrometers for liquid density determinations. 2011-2012.

[2] Cuckow F W - A new method of high accuracy for the calibration of reference standard hydrometers J. Soc. Chem. Indust. 68 44–9, 1949.

[3] Becerra L. and Lorefice, S. – Supplementary comparison SIM.M.D-S1, Comparison of the calibration of hydrometers for liquid density determination (bilateral CENAM - INRIM). 2007.

[4] Cox M.G., The evaluation of key comparison data, Metrologia, 2002, 39, 589-595.ANNEX A

[5] ISO13528," Statistical methods for using proficiency testing by interlaboratory comparison", 2015

A.1 Uncertainty contributions values reported by the NMIs for the hydrometers

Table A1.1

Uncertainty contributions reported by the NMIs for the hydrometer 1 (0.6500-0.7000)

INFLUENCE QUANTITY	Unit	NIS	CENAM	NMISA	NIS
Weighing value of hydrometer in air	g	1.00E-04	1.11E-04	1.44E-04	2.00E-04
Weighing value of hydrometer in buoyant liquid (1st point)	g	1.51E-05	1.12E-04	2.50E-04	3.51E-05
Weighing value of hydrometer in buoyant liquid (2nd point)	g	1.24E-05	1.12E-04	2.61E-04	3.24E-05
Weighing value of hydrometer in buoyant liquid (3rd point)	g	1.21E-05	1.12E-04	2.48E-04	3.21E-05
Cubic thermal expansion coefficient of glass	1/K	1.20E-06	1.15E-06	1.15E-06	1.20E-06
Diameter of stem of hydrometer	mm	5.00E-04	1.40E-01	2.50E-02	5.00E-04
Density air	g/cm^3	7.80E-07	7.80E-07	1.11E-06	7.80E-07
Density buoyant liquid (1st point)	g/cm^3	1.00E-06	6.48E-06	1.26E-05	1.00E-06
Density buoyant liquid (2nd point)	g/cm^3	1.00E-06	6.48E-06	1.26E-05	1.00E-06
Density buoyant liquid (3rd point)	g/cm^3	1.00E-06	6.48E-06	1.25E-05	1.00E-06
Temperature of liquid at hydrometer	°C	2.50E-03	1.00E-02	6.00E-02	2.50E-03
Cubic thermal expansion coefficient of liquid	$g/(cm^3K)$	2.07E-05	7.00E-06	2.07E-05	2.07E-05
Compressibility of liquid	Pa^{-1}	4.48E-11	8.50E-11		4.48E-11
Surface tension of liquid	mN/m	1.00E-04	1.00E-04	6.00E-01	1.00E-04
Gravitation acceleration	m/s^2	2.00E-05	2.00E-05	5.00E-07	2.00E-05
Gradient of gravitational acceleration	m^{-1}	5.02E-07	3.09E-07	2.50E-08	//
Height difference of weights and hydrometer	m	//	5.00E-03	1.00E-02	//
Reading's error	mm	1.00E-04	5.00E-04	4.00E-01	//
Reproducibility	g/cm^3	1.17E-04	1.20E-05	//	//
Repeatability of the measurement		1.73E-04	//	1.68E-06	2.31E-04

Table A1.2

Uncertainty contributions reported by the NMIs for the hydrometer 2 (1.2000-1.2500)

INFLUENCE QUANTITY	Unit	NIS	CENAM	NMISA
Weighing value of hydrometer in air	g	1.00E-05	8.53E-08	2.06E-04
Weighing value of hydrometer in buoyant liquid (1st point)	g	1.51E-05	Not reported	3.66E-05
Weighing value of hydrometer in buoyant liquid (2nd point)	g	1.24E-05	Not reported	2.91E-05
Weighing value of hydrometer in buoyant liquid (3rd point)	g	1.21E-05	Not reported	2.95E-05
Cubic thermal expansion coefficient of glass	1/K	1.20E-06	1.15E-06	1.15E-06
Diameter of stem of hydrometer	mm	5.00E-04	1.40E-01	2.50E-03
Density air	g/cm^3	7.80E-07	7.80E-07	1.11E-06
Density buoyant liquid (1st point)	g/cm^3	1.00E-06	6.48E-06	1.26E-05
Density buoyant liquid (2nd point)	g/cm^3	1.00E-06	6.48E-06	1.26E-05
Density buoyant liquid (3rd point)	g/cm^3	1.00E-06	6.48E-06	1.26E-05
Temperature of liquid at hydrometer	°C	2.50E-03	1.00E-02	6.00E-02
Cubic thermal expansion coefficient of liquid	$g/(cm^3K)$	2.07E-05	7.00E-06	2.07E-05
Compressibility of liquid	Pa^{-1}	4.48E-11	8.50E-11	
Surface tension of liquid	mN/m	1.00E-04	1.00E-04	6.00E-01
Gravitation acceleration	m/s^2	2.00E-05	2.00E-05	5.00E-07
Gradient of gravitational acceleration	m^{-1}	5.02E-07	3.09E-07	2.50E-08
Height difference of weights and hydrometer	m	//	5.00E-03	1.00E-02
Reading's error	mm	1.00E-04	5.00E-04	4.00E-01
additional uncertainty component 1	g/cm^3	//	1.59E-05	//
Repeatability of the measurement		1.73E-04		1.41E-06

Table A1.3

Uncertainty contributions reported by the NMIs for the hydrometer 3 (1.4500-1.5000)

INFLUENCE QUANTITY	Unit	NIS	CENAM	NMISA
Weighing value of hydrometer in air	g	1.00E-04	//	//
Weighing value of hydrometer in buoyant liquid (1st point)	g	1.51E-05	//	//
Weighing value of hydrometer in buoyant liquid (2nd point)	g	1.24E-05	//	//
Weighing value of hydrometer in buoyant liquid (3rd point)	g	1.21E-05	//	//
Cubic thermal expansion coefficient of glass	1/K	1.20E-06	//	//
Diameter of stem of hydrometer	mm	5.00E-04	//	//
Density air	g/cm^3	7.80E-07	//	//
Density buoyant liquid (1st point)	g/cm^3	1.00E-06	//	//
Density buoyant liquid (2nd point)	g/cm^3	1.00E-06	//	//
Density buoyant liquid (3rd point)	g/cm^3	1.00E-06	//	//
Density buoyant liquid (4th point)	g/cm^3	2.50E-03	//	//
Temperature of liquid at hydrometer	°C	2.07E-05	//	//
Cubic thermal expansion coefficient of liquid	$g/(cm^3K)$	4.48E-11	//	//
Compressibility of liquid	Pa^{-1}	1.00E-04	//	//
Surface tension of liquid	mN/m	2.00E-05	//	//
Gravitation acceleration	m/s^2	1.00E-04	//	//
Gradient of gravitational acceleration	m^{-1}	5.02E-07	//	//
Height difference of weights and hydrometer	m	//	//	//
Reading's error	mm	1.00E-04	//	//
Reproducibility	g/cm^3	1.17E-04	//	//
Repeatability of the measurement		1.15E-04	//	//