"COMPARISON ON THE CALIBRATIONS OF HYDROMETERS FOR LIQUID DENSITY DETERMINATION BETWEEN SIM LABORATORIES"

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1 Outline

A supplementary comparison concerning the calibration of hydrometers was proposed during the ANDIMET coordination meeting held on May 31st and Jun 01st 2011 in Bolivia with the purpose of strengthening the national quality infrastructures and cooperation between them to improve regional service availability, internationally recognized and demand-driven besides to know the degree of equivalence between Andean countries. In this regard, according to the requirements of the technical groups of the 4 active ANDIMET members (Colombia, Ecuador, Peru and Bolivia), and after a discussion of priorities, it was decided to make a plan of comparisons between national metrology laboratories of the participating countries. In addition, it was decided to include three SIM NMIs: LACOMET (Costa Rica), INTN (Paraguay) and CENAM (Mexico).

This comparison is carrying out with the support of PTB within the project "*FOMENTO COORDINADO DE LA INFRAESTRUCTURA DE LA CALIDAD EN LA REGIÓN ANDINA, PTB-CAN*", and is following the same protocol of the CCM.D-K4, which is being carried out within the Density Working Group of the CCM [3].

This supplementary comparison will be coordinated by the National Metrology Institute of Peru (SNM-INDECOPI) as Pilot Laboratory and supported by all participants (Appendix A). CENAM accepted to act as co-Pilot Laboratory.

Each laboratory will determine the corrections to be applied to three stated scale readings at 20 °C of different transfer standards in the density range between 600 kg/m^3 and 2 000 kg/m^3.

The linking laboratory CENAM should calibrate all transfer standards involved in the comparison and will carry out the measurement at the beginning and at the end of the comparison.

The measurements are scheduled to end in December 2012. Draft A of the report should be available within March 2013 (Appendix B).

2 Purpose of this document

The purpose of this document is to provide the participating laboratories with instructions for handling the transfer standards (hydrometers) and to report on the measurement results, the measuring procedure and the apparatus.

It is important that all instructions given in this document are followed. This will ensure that the measurement data are obtained under comparable conditions and presented in the same format. Any deviation from the instructions has to be reported to the Pilot Laboratory.

3 Transfer Standards as Artefacts (Hydrometer samples)

For the comparison PTB supplies two similar sets of four artefacts to be used as transfer standards at 20 °C.

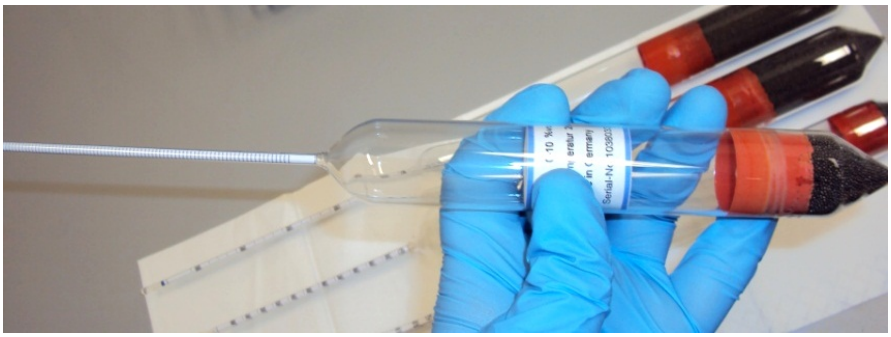

Figure 1. Transfer Standards (Hydrometers)

Figure 2. Some details about the hydrometers

	Hydrometer 1	**Hydrometer 2**	**Hydrometer 3**	**Hydrometer 4**
Manufacturer	Ludwig Schneider	Ludwig Schneider	Ludwig Schneider	Ludwig Schneider
Serial Number	09342350	10380347	11451844	11451847
Range	0,6000 g/cm^3 - 0,6100 g/cm^3	0 - 10 % vol	1,4900 g/cm^3 - 1,5000 g/cm^3	1,9800 g/cm^3 - 2,0000 g/cm^3
Scale division	0,0001 g/cm^3	0,1 % vol	0,0001 g/cm^3	0,0002 g/cm^3
Approximated Mass	89 g	129 g	287 g	294 g
Cubic Expansion Coefficient	25 x 10^{-6} ± 2 x 10^{-6}	25 x 10^{-6} ± 2 x 10^{-6}	25 x 10^{-6} ± 2 x 10^{-6}	25 x 10^{-6} ± 2 x 10^{-6}
Nominal values for calibration	(0,6010 ; 0,6050 ; 0,6090) g/cm^3	(1,0 ; 5,0 ; 9,0) % vol	(1,4910 ; 1,4950 ; 1,4990) g/cm^3	(1,9810 ; 1,9900 ; 1,9990) g/cm^3
Surface Tension	15,2 mN/m	(68,1 ; 57,8 ; 51,8) mN/m	55 mN/m	75 mN/m

Table 1. Characteristics of the Hydrometers that will be circulated between participating laboratories

4 Circulation of the Artefacts

Each participating laboratory will send to the subsequent one the assigned set of four hydrometers as soon as calibrated, after that the laboratory, in accordance with the circulation scheme (Appendix C), and the pilot laboratory have been informed. The completion of the measurements and the date of dispatch, giving details of the transportation will be given by the form “Appendix E”.

Each set will be packed into a suitable container. The package will be transported directly by the personnel of the last laboratory. The package will be provided with a warning: To be opened only by laboratory personnel.

After arrival of the package, the participating laboratory will inform the Pilot Laboratory without delay giving details of the arrival date, the state of the package and its contents (Appendix D).

With exception of LACOMET and INTN, the expenses for the hand transportation of the travelling set(s) to the next laboratory will be cover by PTB. The participants are responsible for completing the local customs formalities.

5 Measurements

At least 5 weighing-in-air sequences have to be carried out for the weight determination of each hydrometer and to evaluate the experimental standard deviation. The mean of the parameters contributing to the air density evaluation are to be recorded, i.e. pressure, temperature, relative humidity (or dew point), and CO_2 content (whether measured or assumed). For the calculation of the air density, the revised CIPM formula (2007) is to be used [1]. The mean of this value is to be reported.

5.1 *Mass measurement*

Mass least 5 weighing-in-air sequences have to be carried out for the weight determination of each hydrometer and to evaluate the experimental standard deviation.
The mean of the parameters contributing to the air density evaluation are to be recorded, i.e. pressure measurements should be made under ambient laboratory conditions close to 20 °C. For the calculation of the air density, the revised CIPM formula (2007) is to be used [1]. The mean of this value is to be reported.

5.2 *Hydrostatic weighing*

After the mass measurement, the hydrostatic weighing is to be performed without cleaning.

The measurements should be made close to 20 °C. The cubic expansion coefficient for all hydrometers is assumed to be $25 \cdot 10^{-6}$ °C^{-1} with an interval of rectangular distribution of $2 \cdot 10^{-6}$ °C^{-1},.

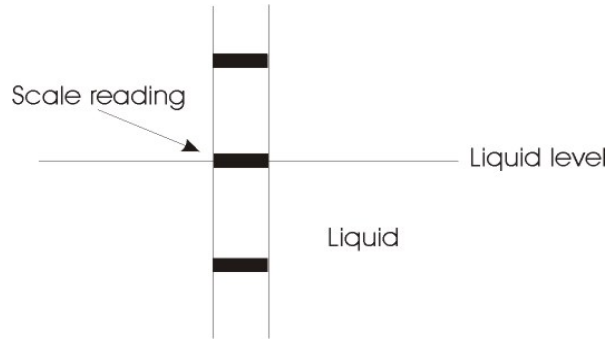

Figure 3. Alignment of the stated scale reading with the horizontal plane of liquid.

At least 5 weighing sequences have to be carried out for the weight determination at each of the three scale readings stated and to evaluate the experimental standard deviation. Adjustment of the scale readings to the liquid level should be made when the middle of the line is aligned with the horizontal plane of liquid (Figure 3).

The mean of the parameters contributing to the air density evaluation are to be recorded, i.e. pressure, temperature, relative humidity (or dew point), and CO_2 content (measured or assumed). The mean value of the air density is to be reported.

6 Reports

Each laboratory will send the Linking Laboratory (CENAM) a summary of the procedure used describing the apparatus, including, if possible, any reference, giving the mathematical model equations for calculating the corrections and how the standard uncertainties of the individual influence quantities are estimated. Besides, information and results should be made up using the enclosed MS Excel Report Form.

The MS Excel Report Form consists of two parts:

6.1 Report Form 1

It concerns information about the instrumentation used in the project. Please add any additional information obtained in your measurements.

For the balance(s) used to determine the weighing value in air/liquid, the following information is to be given:

a) Manufacturer and type of balance,
b) Maximum capacity, electronic range, resolution,
c) Standard deviation, maximum non-linearity, out-of-centre error,
d) Calibration uncertainty, method and frequency.

If mass standards are used as "substitutional weight" for the comparison of mass of hydrometer to the mass of weights, the following information is to be given:

a) Manufacturer, name and material of weights,
b) Identification of weights, their masses and standard uncertainties (k=1),
c) Date of last calibration and traceability.

For the thermostat system used to stabilize the temperature of the buoyant liquid, the following information is to be given:

a) Manufacturer and type of thermostat,
b) Capacity,
c) Temperature stability and uniformity at 20 °C.

For the alignment system used for alignment the scale readings, the following information is to be given:

a) Type of magnifier,
b) Method of alignment,
c) Uncertainty of alignment.

For the instruments used for the determination of the density of air (air pressure, temperature, humidity and CO_2 content), the following information is to be given:

a) Manufacturer, type,
b) Resolution,
c) Frequency of measurement,
d) Calibration uncertainty, date and traceability.

For the instruments used for the measurement of the liquid temperature, the following information is to be given:

a) Manufacturer and type of sensor,
b) Manufacturer and type of resistance bridge and standard resistor (if applicable),
c) Resolution of temperature measurement,
d) Calibration uncertainty, date and traceability of thermometer (whole temperature range).

For the apparatus used for determining the surface tension of the buoyant liquid, the following information is to be given:

a) Manufacturer and measuring method,
b) Calibration uncertainty and traceability.

For the reference buoyant liquid the following information is to be given:

a) Manufacturer,
b) product name,
c) Molecular formula and assay.

6.2 *Report Form 2(ID Hydrometer)*

For each hydrometer, general information and results of measurements are to be given.
General information is to be given in:

Table 1-1: Movement of the travelling standard

a) Date of arrival of the hydrometer at the laboratory,
b) Condition of package and of the individual hydrometer,
c) Date of shipment of hydrometers,
d) Responsible for transportation.

Table 1-2: Measurement period

a) Date for hydrometer mass measurement,
b) Date for hydrostatic weighing.

Measurement information is to be given in:

Table 1-3: Mass determination

a) Substitutional weights, if they are used, and mean value of the hydrometer mass for five weights in air and its standard deviation of the mean.
b) Ambient conditions during mass measurements including data about air density, air temperature, air pressure, humidity and CO_2 content.

Table 1-4: Hydrostatic weighing

a) Mean value of the additional weight (ballast) if a stainless steel weight is added to the hydrometer to cause it to sink, and the standard deviation for the mean.

b) Mean values of the hydrometer weighing value at the indicated scale reading. The standard deviation for the mean and the property of buoyant liquid during the hydrostatic weighing.

c) Ambient conditions during hydrostatic weighing, including data about air density, air temperature, air pressure, humidity and CO_2 content.

Table 1-5: Results

a) Corrections at each scale reading as determined at 20 °C. In the last column the reference surface tension is given.

Table 1-6: Uncertainty budget for the hydrometer corrections

A list of main components of the uncertainty budget is given. Please add any additional component occurring in your measurements.

The uncertainty evaluation should include a list of all influence quantities, values, their degrees of freedom and their combined standard uncertainty. This is obtained by combining the individual standard uncertainties obtained from Type A and Type B evaluations, according to "Guide to the Expression of Uncertainty in Measurement" [2].

A pooled experimental standard deviation characterizing the hydrometer weighing value in buoyant liquid is to be given [2 (H.3.6)].

Table 1-7: Uncertainty of the hydrometer corrections

The uncertainty of measurement for the corrections is calculated here with the Excel sheet from the above given data, as well as the effective degrees of freedom ν_{eff} of the combined standard uncertainty u_c, the t-factor $t_{95}(\nu_{\text{eff}})$ taken from the t-distribution for a 95% confidence level and the expanded uncertainty for the corrections as $U_{95} = t_{95}(\nu_{\text{eff}}) \cdot u_c$.

7 Deadline

The reports with the results of measurements should be sent to the Linking Laboratory (CENAM) at most in the date indicated in the table of Appendix C. A result will not be considered complete unless an associated uncertainty supported by a complete uncertainty budget is given. The results are confidential until Draft B is approved.

8 Special problem

Please, do not hesitate to contact the Pilot Laboratory for any questions.

8.1 *Breach of travelling standard*

In case a hydrometer is broken, its substitution will be decided at that time.

8.2 *Late entry of a participant*

Due to the tight timetable, it is not possible for any additional participant to join after the circulation has started.

9 References

[1] A. Picard, R.S. Davies, M. Glaser, and K. Fujii : "Revised formula for the density of moist atr (CIPM 2007)," Metrologia, 2008, 45, pp. 149 - 155.

[2] "Evaluation of measurement data – Guide to the expression of uncertainty in measurement" JCGM 100:2008.

[3] S. Lorefice : Technical Protocol for the CIPM key comparison CCM.D_K4 “Hydrometer”, 2010

Appendices

A. Participants

Laboratory	Acronym	Contact Person
Centro Nacional de Metrologia Km 4.5 Carretera a los Cués, Mpio El Marqués, Querétaro México	CENAM	Luis Omar Becerra lbecerra@cenam.mx Tel.: +52 442 2 110573 +52 442 2 110500 to 04 ext. 3602
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Instituto Ecuatoriano de Normalización Autopista general Rumiñahui pasando el puente peatonal 5, pasando 20 metros en la dirección del trébol al valle de los chillos, Quito Ecuador	INEN	Manuel Salazar msalazar@inen.gob.ec Tel.: +593 2 2343379, 2343116, 2344394
Instituto Nacional de Defensa de la Competencia y de la Protección de la Propiedad Intelectual Calle de la Prosa 104 – San Borja – Lima Perú	INDECOPI	Abed Morales amorales@indecopi.gob.pe Tel.: +511 2247800 ext. 1661
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Instituto Nacional de Tecnología, Normalización y Metrología Av. Artigas 3973 - Asunción Paraguay	INTN	Diana Cantero dcantero@intn.gov.py Tel +59521295408
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B. Timetable for the comparison

March 2012	Workshop in La Paz – Bolivia. Agreement on Technical Protocol
March 2012 to April 2012	Start of measurements (Linking Laboratory: CENAM)
May 2012 to November 2012	Measurements by all participants and Reports from all participants
December 2012	End of measurements (Linking Laboratory: CENAM)
March 2013	Draft A of comparison report
May 2013	Draft B of comparison report: End of comparison

C. Circulation scheme for the comparison

	National Metrology Institute	Arrival	Sending of results
1	CENAM-Mexico		
2	IBMETRO-Bolivia	2012-05-01	2012-06-19
3	INTN-Paraguay	2012-05-28	2012-07-16
4	INEN-Ecuador	2012-07-02	2012-08-20
5	INDECOPI-Peru	2012-08-06	2012-09-24
6	INM-Colombia	2012-09-10	2012-10-29
7	LACOMET-Costa Rica	2012-10-15	2012-12-03
8	CENAM-Mexico	2012-11-19	2013-01-07

D. Fax/e-mail: Receipt of a comparison package

To monitor the progress of the comparison, we ask on receipt of the package to kindly send a report by fax/e-mail to

Abed Morales
INDECOPI
Calle de La Prosa 104, San Borja
Lima
PERU
Fax: +511-2247800 ext. 1264
e-mail: amorales@indecopi.gob.pe

This report should contain the following information:

Participating laboratory
Contact person
Telephone
Fax

and a text like this:

The package of the hydrometers was received on (date).

The package seems, after short inspection, (not) to be damaged.

If damaged: (not) seriously.

The contents is probably (not) suitable for use.

Remarks:

Date, signed

E. Fax/e-mail: Progress report

To monitor the progress of the comparison, we ask to kindly send a report by fax/e-mail to

Abed Morales
INDECOPI
Calle de La Prosa 104, San Borja
Lima
PERU
Fax: +511-2247800 ext. 1264
e-mail: amorales@indecopi.gob.pe

This report should contain the following information:

Participating laboratory
Contact person
Telephone
Fax

and a text like this:

Object: Package of the hydrometers

The measurements were completed on (date).
[The comparison package was shipped on (date).]
[The results were sent to the Pilot Laboratory on (date).]

Remarks:

Date, signed