COMPARISON OF THE CALIBRATION OF HYDROMETERS FOR LIQUID DENSITY DETERMINATION BETWEEN SIM LABORATORIES

Technical Protocol

Coordinating Institutes: Pilot Laboratory: Centro Nacional de Metrología de México CENAM
Co-pilot: Instituto Nacional de Metrología de Colombia INM

1. Outline

Hydrometers are instruments widely used to measure density, specific gravity, alcoholic strength, sugar concentration, milk density etc. of a liquid.

The aim of this SIM comparison is to harmonize the measurements for the hydrometers calibration of NMI laboratories (see Participants List Appendix A), in the density measurement range between 0,600 g/cm^3 and 1,300 g/cm^3 at 20 ºC and to check liquid density measuring instruments used for this purpose.

This protocol is following the guidelines of the Euromet Project 702 “Comparison of the calibration of high resolution hydrometers for liquid density determinations” carried out in 2003-2004 [1].

The comparison is intended to be a regional supplementary comparison accordingly to the CIPM Mutual Recognition Arrangement. It should also support provisional entries for the CMC tables in this sub-field.

CENAM will act as pilot laboratory for this comparison and INM Colombia will act as a co-pilot laboratory. Each participating laboratory, will determine the correction of indication to be applied to three stated scale readings at 20 °C of the four different high precision hydrometers with a scale division of d = 0,000 1 g/cm^3.

The pilot laboratory should calibrate all hydrometers involved in the comparison, at the beginning and at the end of the comparison. In general, the development of this comparison will be carried out taking into account the timetable shown in the Appendix B.

2. Purpose of this document

The purpose of this document is to provide the participating laboratories the guidelines to be followed in the developed of this comparison and it is very important that all laboratories take account these.

Any deviation from the instructions has to be reported to the Pilot and co-pilot Laboratories.

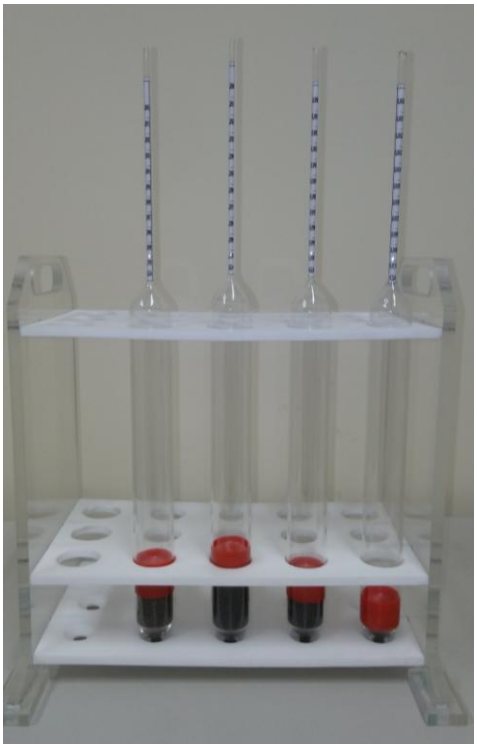
Instruments under Calibration (Hydrometers samples)

For the comparison the pilot and co-pilot laboratories supplies one (1) set of four (4) different hydrometers with the characteristics showed in Table 1 also see the figure 1.

Table 1. Data of the traveling hydrometers for the SIM Density Comparison

Characteristic	Hydrometer 1	Hydrometer 2	Hydrometer 3	Hydrometer 4
Manufacturer	Ludwig Schneider	Ludwig Schneider	Ludwig Schneider	Ludwig Schneider
Serial Number	14550345	12496642	14550350	15607975
Range	0,6000 g/cm^3 - 0,6100 g/cm^3	0,9900 g/cm^3 - 1,0000 g/cm^3	1,0900 g/cm^3 - 1,1000 g/cm^3	1,2900 g/cm^3 - 1,3000 g/cm^3
Resolution	0,0001 g/cm^3	0,0001 g/cm^3	0,0001 g/cm^3	0,0001 g/cm^3
Surface Tension:	15 mN/m	35 mN/m	35 mN/m	35 mN/m
Reference Temperature	20ºC	20ºC	20ºC	20ºC
Hydrometer weight (Approx.):	89 g	144 g	161 g	195 g
Hydrometer Length:	400 mm	400 mm	400 mm	400 mm
Measuring Points:	0,6010 g/cm^3 0,6050 g/cm^3 0,6090 g/cm^3	0,9910 g/cm^3 0,9950 g/cm^3 0,9990 g/cm^3	1,0910 g/cm^3 1,0950 g/cm^3 1,0990 g/cm^3	1,2910 g/cm^3 1,2950 g/cm^3 1,2990 g/cm^3

Figure 1. Set of four Hydrometers



Each participating laboratory should make the corresponding measures in order to calculate the correction of indication of the hydrometers.

After the measurements by the participating laboratory, the set of hydrometers will be sent to the next laboratory in accordance with the Measurement Path (See Appendix C).

3. Transportation

For transportation of the set of four hydrometers will be packed into a suitable container. Each individual hydrometer is separately placed into its original case (See Figure 2).

Figure 2. Transportation Package of Hydrometers Set (Case)

The package contains a complete list of the contents with the numbers of all hydrometers and the weight and size of the whole package. This information will be mailed to the participants.

The package only will be transported to the next laboratory by the technical personnel of the laboratory that made the last measurements. The package should be provided with the following warning message *"To be opened only by laboratory personnel".*

After arrival of the package, the participating laboratories will inform to the pilot and co-pilot laboratories about the details of the arrival date, state of the package and its contents; without delay in the established dates (see Appendix D).

Each participant is responsible for completing the local customs formalities, it is also important that the laboratory that made the last measurement establish communication with the receiver laboratory in order to ask for information about the customs requirements in the respective country, previous the corresponding travel.

Note: ***Each participating laboratory bears its own expenses for the transportation of the travelling set to the next laboratory and any customs charges.***

4. Measurements

To determine the errors of indication for each individual hydrometer, the participants will be free to perform all measurements using their own procedure based on the **Cuckow Method** [2]. It is however required that the hydrometers be kept at the laboratory only for the time necessary for calibration and not longer than the planned time.

Before starting the calibration, gently clean the hydrometers with pure Ethanol using gloves.

During the all calibration process, 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 CIPM formula (CIPM 81/2007) is to be used [3]. The mean of this value is to be reported.

Finally, the hydrometers shall be allowed to acclimatize to the weighing conditions for at least 24 hours.

4.1. Mass measurement

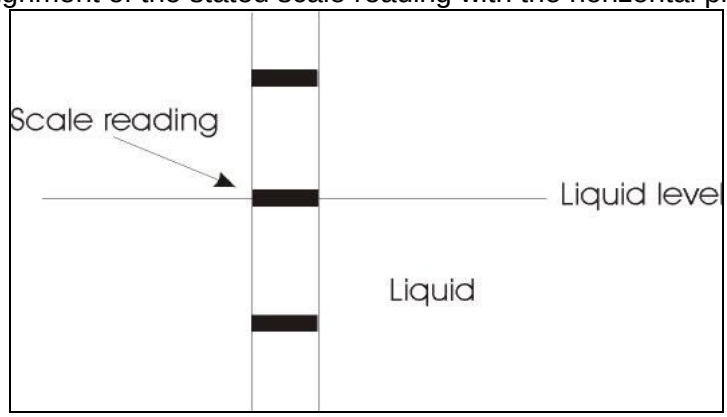
Mass measurements should be made under ambient laboratory conditions close to 20 °C. At least 5 weighing-in-air sequences have to be carried out for the weight determination of each hydrometer and to evaluate the experimental standard deviation.

4.2. Hydrostatic weighing

After the mass measurement, the hydrostatic weighing is to be performed without cleaning. The measurements should be made close to 20 °C.

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 mark is aligned with the horizontal plane of liquid (liquid level), see Figure 3.

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



The cubic expansion coefficient for all hydrometers is assumed to be 25 x 10^{-6} °C^{-1} with an standard uncertainty of 1,2 x 10^{-6} °C^{-1}.

The Pilot and co-pilot laboratories must be informed about the completion of the measurements and the date of the hydrometers dispatch, giving details of the transportation (see, Appendix E).

5. Reports

As soon as the measurements are finished, each laboratory will send to the *Pilot Laboratory* a summary of the procedure used describing the apparatus, including, if possible, any reference, giving the mathematical model equations for calculating the errors of indication 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 (Appendix F).

The MS Excel Report Form consists of two parts:

- ***Report Form 1 (Measurements Instruments):*** It concerns information about the instrumentation used in the measurements. Please add any additional information obtained in your measurements.

- ***Report Form 2 (Measurements Results):*** In this report form will be reported the data related with the measurements, results and its related uncertainties. Please add any additional information obtained 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 ISO "Guide to the Expression of Uncertainty in Measurement" [4].

The reports are to be sent to the *Pilot Laboratory* as soon as possible but three weeks after the measurements and transportation of the hydrometer set are completed at the latest (see 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 all the participants have completed their measurements and all the results have been received (or until the deadline for receipt of results is over).

6. Special problem

Please, do not hesitate to contact the Pilot and co-pilot laboratories for any questions.

6.1. Hydrometer Damage

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

6.2 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.

7. References

[1] R. S. Davis: "Revised formula for the Density of moist air (CIPM-2007)", Metrología, 2008, vol. 45, pp. 149-155.
[2] "Guide to the Expression of Uncertainty in Measurement," International Organization for Standardization (ISO), 2008.
[3] Lorefice S., Bettin H., Vámossy C., Technical Protocol for EUROMET Project 702 “Comparison of the calibrations of high resolution hydrometers for liquid density determinations”, 2003.
[4] Cuckow W. “A new method of high accuracy for the calibration of the reference standard hydrometers”, J.S.C.L. 68, February of 1949

APPENDIX A.
PARTICIPANTS LIST

National Institute of Metrology	Acronym	Technical Contact
Centro Nacional de Metrología km. 4,5 Carretera a los Cués, Mpio. El Marqués Querétaro, México	CENAM	Luis Omar Becerra lbecerra@cenam.mx Tel: +52 442 211 05 00 ext. 3602,
Instituto Nacional de Metrología Av. Carrera 50 No.2755 Int. 2, Bogotá, Colombia	INM	Luis Carlos Castro lcastro@inm.gov.co Tel: +57 1 2542222 Ext. 1618
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Instituto Nacional de Tecnología Industrial. Av. Gral. Paz 5445, B1650KNA, San Martín, Prov. de Buenos Aires, Argentina	INTI	Jorge Sánchez sanchezj@inti.gob.ar Tel: +54 11 4724 6200/300/400 Interno 64370800 444 4004
Instituto Boliviano de Metrología Av. Camacho No. 1488 La Paz, Bolivia	IBMETRO	Elisa Santalla esantalla@ibmetro.gob.bo Tel:+ 591 2 237 20 46 + 591 2 214 79 45 Int. 350
Instituto Ecuatoriano de Normalización. Autopista "General Rumiñahui, Sector Conocoto, puente peatonal No. 5", Quito-Ecuador	INEN	Victor Guevara vguevara@normalizacion.gob.ec Tel: + 593 3931010 al 1019
Laboratorio Costarricense de Metrología 500 m N, 50 m O del Supermercado Muñoz & Nanne, Ciudad de la Investigación, Universidad de Costa Rica, San Pedro de Montes de Oca, Costa Rica	LACOMET	Francisco Sequeira fsequeira@lacomet.go.cr Tel: 506 283 65 80 ext 111-112, 506 253 20 48 ext 111-112

APPENDIX B.
TIMETABLE FOR THE COMPARISON

April, 2017	Agreement on Technical Protocol
June, 2017	Registration of the SIM Density Comparison
November, 2016	Start of measurements (Pilot Laboratory)
June, 2017	Start of measurements by all participants and Reports from all participants
2018	End of measurements (Pilot Laboratory)
2018	Draft A of comparison report: End of Comparison
2018	Final Report

APPENDIX C.
MEASUREMENT PATH FOR THE COMPARISON

Participants	Date of Measurements	Date of transportation of the travelling standards	Date of sending results of the measurements
CENAM	November, 2016	December, 2016	
INM	June 12 – June 30, 2017	July 3 - July 7, 2017	Until July 28, 2017
CESMEC	July 10 – July 28, 2017	July 30 - August 4, 2017	Until August 25, 2017
INTI	August 7 – August 25, 2017	August 28 – September 1, 2017	Until September 22, 2017
IBMETRO	September 4 – September 22, 2017	September 25 – September 29, 2017	Until October 20, 2017
INEN	October 2 – October 20, 2017	October 23 – October 27, 2017	Until November 17, 2017
LACOMET	October 30 – November 17, 2017	November 20 – November 24, 2017	Until December 15, 2017
CENAM	2018		

APPENDIX D.
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 e-mail to:

Pilot Laboratory
Luis Omar Becerra
CENAM
Km 4,5 Carretera a los Cués, Municipio el Marqués
Querétaro, México
Teléfono: +52 442 2 11 05 68
e-mail: lbecerra@cenam.mx

Co-pilot Laboratory
Luis Carlos Castro Camacho
INM Colombia
Avenida Carrera 50 No. 26-55 Int 2
Bogotá, Colombia
Teléfono: +57 1 254 22 22 Ext. 1618
e-mail: lcastro@inm.gov.co

This report should contain the following information:

Participating laboratory
Contact person
Telephone
E-mail

and a text like this:

The package of SIM Hydrometer Calibration was received on (date).
The package and its content seems, after short inspection, (not) to be damaged.
If it is damaged is necessary to specificate if the damage is not serious or is not suitable for use.

Remarks:

Date, signed

APPENDIX E.
E-MAIL: PROGRESS REPORT

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

Pilot Laboratory	**Co-pilot Laboratory**
Luis Omar Becerra CENAM Km 4,5 Carretera a los Cués, Municipio el Marqués Querétaro, México Fax: +52 442 2 11 05 68 e-mail: lbecerra@cenam.mx	Luis Carlos Castro Camacho INM Colombia Avenida Carrera 50 No. 26-55 Int 2 Bogotá, Colombia Teléfono: +57 1 254 22 22 Ext. 1618 e-mail: lcastro@inm.gov.co

This report should contain the following information:

Participating laboratory
Contact person
Telephone
E-mail

and a text like this:

SIM Hydrometer Calibration

The measurements were completed on (date).

The comparison package was shipped on (date) (Please give a brief detail about the transportation)
The results were sent to the Pilot Laboratory on (date).

Remarks:

Date, signed

APPENDIX F.
REPORT FORMS

Excel File with Report form 1 and Report form 2 are part of this protocol.
Report Form 1. – Measuring Instruments
Report Form 2. – Measurement Results.

End of the document.