**Technical Protocol for the CIPM key comparison**

# CCM.D_K4 "Hydrometer"

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## 1 Outline

A CIPM key comparison concerning the calibration of hydrometers was proposed during the meeting of the Working Group on Density (WG-Density) of the Consultative Committee for Mass and Related Quantities (CCM) held on April 22nd, 2008 at the BIPM with the purpose of linking the regional comparisons previously performed under the auspices of the Regional Metrology Organizations APMP, EURAMET and SIM.

This CIPM key comparison, designated as CCM.D-K4, will be coordinated by the Istituto Nazionale di Ricerca Metrologica (INRIM, Italy) supported by all participants (Appendix A).

For the purpose of this project and to speed up the key comparison, the participating laboratories will be divided into two groups (petals) connected with each other by three independent laboratories INRIM (IT), CENAM (MX) and PTB (DE).

Each laboratory, belonging to an individual petal, 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$^3$ to 2 000 kg/m$^3$.

The linking laboratories INRIM, CENAM and PTB should calibrate all transfer standards involved in the comparison.

The INRIM will carry out the measurement at the beginning and at the end of the comparison.

The measurement are scheduled to end in June 2012. Draft A of the report should be available within December 2012 (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 INRIM supplies two similar sets of four artefacts to be assigned to each petal and used as transfer standards at 20 °C. Each set will be concerned with two hydrometers with a scale division of 0.1 kg/m$^3$ working near 600 kg/m$^3$, and 1 300 kg/m$^3$, an alcoholometer (alcohol hydrometers) cl.1 and an hydrometer with a scale division of 0.2 kg/m$^3$ working near 2 000 kg/m$^3$.

## 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 by courier or 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 laboratories will inform the Pilot Laboratory without delay giving details of the arrival date, the state of the package and its contents (Appendix D).

Each participating laboratory bears its own expenses for the transportation of the travelling set(s) to the next laboratory and any customs charges. The participants are responsible for completing the local customs formalities.

**Note**: An ATA carnet will be issued by INRIM for each package. If it is used, please verify that the carnet is presented to the customs authorities and correctly stamped. Each laboratory must provide all customs formalities in the shortest time to avoid delay in the circulation. A month has been estimated for the transportation, the export and the import formalities.

At the end of October 2011 both packages have to come back to INRIM to arrange, if it is necessary, for two new carnets.

## 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. pressuremeasurements should be made under ambient laboratory conditions close to 20 °C. At, 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.

### 5.2 *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 uncertainty of $2 \cdot 10^{-6}$ °C$^{-1}$, rectangular

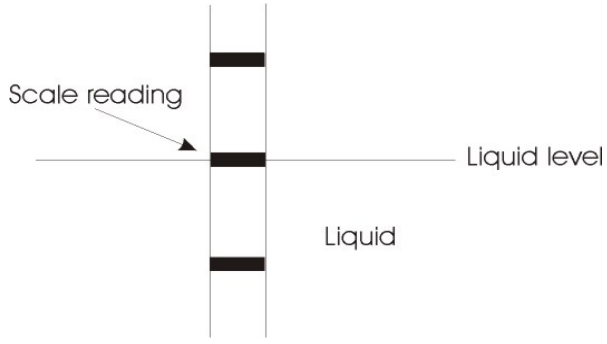


Figure 1. Alignment of the stated scale reading with the horizontal plane of liquid.

distribution.

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 1).

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

As soon as the measurements are over, each laboratory will send 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 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) Company 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 ISO "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 $\nu_{eff}$ of the combined standard uncertainty $u_c$, the t-factor $t_{95}(\nu_{eff})$ taken from the t-distribution for a 95% confidence level and the expanded uncertainty for the corrections as $U_{95} = t_{95}(\nu_{eff}) \cdot u_c$ .

## 7 Deadline

The reports are to be sent to the Pilot Laboratory as soon as possible but six weeks after the measurements are completed at the latest. 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).

## 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.

### 8.3 *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. Only if an additional comparison will be carried out due to breach of the hydrometer/s or due to unexpected delays might a late entry be possible.

## 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] "Guide to the Expression of Uncertainty in Measurement," International Organization for Standardization (ISO), 1995.

Appendices

## A. Participants

| Laboratory | Country code | Contact Person |
|---|---|---|
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| Central Office of Measures - Glówny Urzad Miar (GUM)<br>Physical Chemistry Department,<br>Density, Viscosity and Spectral Analysis Laboratory,<br>ul. Elektoralna 2<br>00-139 Warszawa - POLAND | PL | Elżbieta Lenard,<br>density@gum.gov.pl<br>Tel :+ 48 22 581 9410<br>Fax :+ 48 22 581 9395 |
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## B. Timetable for the comparison

| | |
|---|---|
| September 2010 | Answers to questionnaire |
| December 2010 | Agreement on Technical Protocol |
| December 2010 – January 2011 | Registration of CCM.DK4 key comparison |
| January 2011 | Start of measurements (Pilot Laboratory) |
| March 2011 to June 2012 | Measurements by all participants and Reports from all participants |
| December 2012 | Draft A of comparison report |
| February 2013 | Draft B of comparison report: end of comparison |

## C. Circulation scheme for the comparison

| Group A | | | Group B | |
|---|---|---|---|---|
| **Laboratory** | **Data** | | **Laboratory** | **Data** |
| | | | | |
| INRIM | January 2011 | | INRIM | January 2011 |
| CENAM | March – April 2011 | | CENAM | March – April 2011 |
| LATU | June 2011 | | NMIA | June 2011 |
| NMIJ | August 2011 | | NIST | August 2011 |
| LNE | October 2011 | | KRISS | October 2011 |
| **to INRIM for ATA Carnet formality** | | | | |
| GUM | December 2011 | | MKEH | December 2011 |
| PTB | February – March 2012 | | PTB | February – March 2012 |
| INRIM | May - June 2012 | | INRIM | May - June 2012 |

## 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 telefax to

Salvatore Lorefice
INRIM
Strada delle Cacce, 91 - 73
10135 Torino
ITALY
Fax: +39 011 3977 937
e-mail: S.Lorefice@inrim.it

This report should contain the following information:

Participating laboratory
Contact person
Telephone
Fax

and a text like this:

The package of CMC.D-K4 "Hydrometer" 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

Salvatore Lorefice
INRIM
Strada delle Cacce, 91 - 73
10135 Torino
ITALY
Fax: +39 011 3977 937
e-mail: S.Lorefice@inrim.it

This report should contain the following information:

Participating laboratory
Contact person
Telephone
Fax

and a text like this:

Object: CCM.D-K4 "Hydrometer"

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

Remarks:

Date, signed