EURAMET.M.D-K4.2020
EURAMET Project No. 1496

Technical Protocol for

Hydrometers calibration comparison from 600 kg/m^3 to 2000 kg/m^3

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The following document is a similar version of the original technical protocol for the comparison CCM.D-K4, written by Salvatore Lorefice. The main changed are: the list of the participants (Appendix A), the schedule (Appendix C), the chapter 4 Test Procedure and chapter 5 Cautions.

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.

The CIPM key comparison, designated as CCM.D-K4, was coordinated by the Istituto Nazionale di Ricerca Metrologica (INRIM, Italy). January 2011 to April 2012.

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 were INRIM, CENAM and PTB, they calibrated all transfer standards involved in the comparison: four hydrometers in the range between 600 kg/m^3 to 2 000 kg/m^3.

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

The measurements were performed from January 2011 to April 2012, the Final Report has been published in 2016 [1].

This comparison is the EURAMET Regional Key Comparison of the CCM.D-K4. It is based on a decision at the TC-M Annual Meeting in April 2019 in Budapest (Hungary). The TC-M agreed that INRIM and PTB will organize this key comparison as pilot and co-pilot laboratories. This comparison will be running in 2020.

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

For the comparison INRIM and PTB provide two sets composed of four transfer standards to be assigned to each petal, details are shown in the following table:

Table 1. Hydrometers for petal no. 1(with ATA Carnet)

Range	Resolution	Serial Number
0.600 - 0.610 g/cm^3	0.1 g/cm^3	20731447
0.990 - 1.000 g/cm^3	0.1 g/cm^3	9343118
1.490 - 1.500 g/cm^3	0.1 g/cm^3	9343459
1.980 - 2.000 g/cm^3	0.2 g/cm^3	9346686

Table 2. Hydrometers for petal no. 2

Range	Resolution	Serial Number
0.600 - 0.610 g/cm^3	0.1 g/cm^3	20731448
0.990 - 1.000 g/cm^3	0.1 g/cm^3	9343119
1.490 - 1.500 g/cm^3	0.1 g/cm^3	9343463
1.980 - 2.000 g/cm^3	0.2 g/cm3	6832

4. Test Procedure

The measurand is the correction *C* to be evaluated at each calibration point

$$C = \rho_x - \rho_r$$

where ρ_x is the density of the buoyant liquid in which the hydrometer would freely float at the scale mark ρ_r.

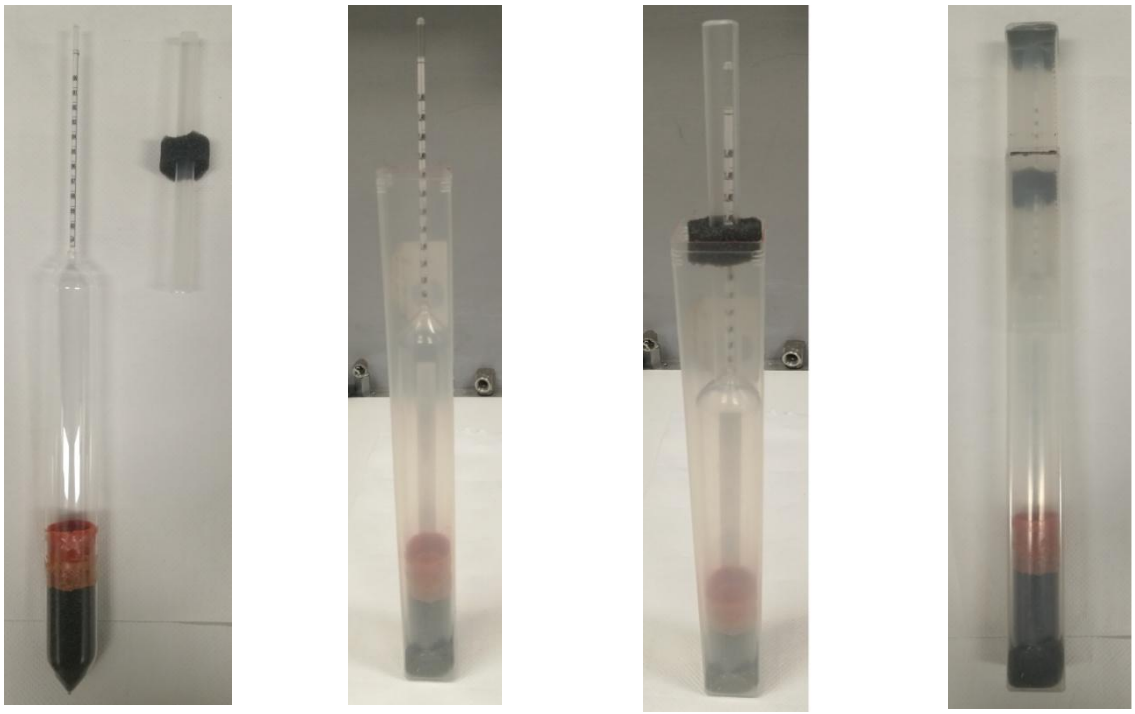
The results have to be given at the reference temperature of 20 ºC, the cubic expansion coefficient for all hydrometers was assumed to be $25 \cdot 10^{-6}$ °C^{-1}, with an uncertainty of $1{,}2 \cdot 10^{-6}$ °C^{-1}.

Participating laboratories should use their routine calibration procedure based on the Cuckow's method.

In Table 3 the calibration points and the reference surface tension of the liquid, in which each hydrometer was intended to be used, are shown.

Table 3. Calibration points and reference surface tensions

Range	Reading scale/ g/cm^3	Ref. surface tension/ mN/m
0.600 - 0.610 g/cm^3	0.601 0	15.1
	0.605 0	15.3
	0.609 0	15.5
0.990 - 1.000 g/cm^3	0.991 0	75.0
	0.995 0	75.0
	0.999 0	75.0
1.490 - 1500 g/cm^3	1.491 0	55.0
	1.495 0	55.0
	1.499 0	55.0
1.980 - 2.000 g/cm^3	1.981 0	75.0
	1.990 0	75.0
	1.999 0	75.0



a) b) c) d)

Figure 1. The system of packaging: a) hydrometer with the stem protector, b) hydrometer into the container, c) position the stem protector, d) close the container firmly.

5. Cautions

The stem of the hydrometers used in this comparison are very fragile, so care must be taken when handling them.

- When not under calibration, the hydrometers should be stored in their storage container.
- When possible, handle the hydrometer by the bulb.
- Take the hydrometer for the stem only when it is in the vertical position.
- Extract the hydrometer from the container keeping the container in a perfectly vertical position, or extract the hydrometer by placing the container horizontally on a table.
- Insert the hydrometer in the container keeping the container in a perfectly vertical position.
- Take great care in preparing the hooking system for the hydrostatic weighing
- Before calibration, the hydrometers should be cleaned (e.g with ethanol) and then kept in the laboratory for an appropriate time for temperature stabilization.
- After calibration, the hydrometers should be cleaned.

- Before sending the hydrometers, they must be carefully placed in their storage container (see Figure 1), other types of containers are not allowed.

6. Circulation of the Artefacts

Each participating laboratory (Appendix A), as soon as calibrated, will send to the next laboratory the set of the four assigned hydrometers, according to the circulation scheme (Appendix C), informing the receiving laboratory and the pilot laboratory (Appendix E).

Each travelling standard is placed in the appropriate container, provided by the pilot laboratory. The packaging system is shown in the Figure 1, other methods are not allowed.
The four hydrometers have to be placed in cardboard box with packaging material, provided by the pilot laboratory. The total weight of the transportation case together with the travelling standards is 3 kg in total, dimension is about 60x30x40 cm.
It should be ensured by the sending institute that the travelling standards will be delivered to the next participating institute on time according to the planned schedule given in Appendix C.
The package will be transported by courier or directly by hand by the laboratory staff.

The package will be equipped with a warning: To be opened only by laboratory staff.

After the arrival of the package, the laboratory will inform without delay the pilot laboratory indicating the date of arrival, the status of the package and its contents (Appendix D).

Each participating laboratory bears its own costs for the transportation of the travelling standards to the next laboratory and any customs charges. The participants are responsible for completing the local customs formalities.

ATA Carnet will be released by INRIM for the petal no. 1 (Appendix C), if used, please ensure that the carnet is presented to the customs authorities and stamped correctly.
Each laboratory must take care of all customs formalities as soon as possible to avoid delays in circulation.
If some hydrometer should break, it can be replaced using a new hydrometer, and a new circulation schema will be set up.

7. Measurements required

Mass measurement

At least five weighing sequences in air shall be performed to determine the weight of each hydrometer and to evaluate the experimental standard deviation.

The averages of the parameters contributing to the evaluation of air density shall be recorded, the measurements shall be carried out under laboratory ambient conditions at a temperature close to 20 °C. For the calculation of air density, the CIPM 2007 formula [2] shall be used.

Hydrostatic weighing

After the mass measurement, the hydrostatic weighing has to be carried out.
The measurements should be made close to 20 °C.
At least five weighing sequences have to be carried out for the weight determination at each of the three scale readings stated in Table 3, 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 2).

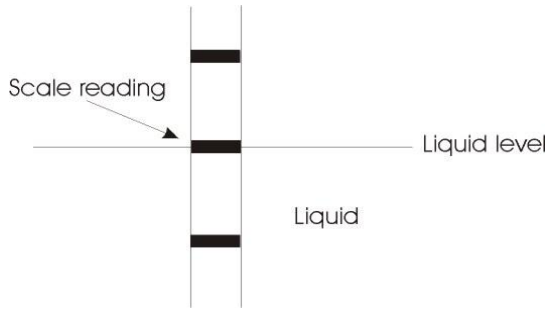


Figure 2. Alignment of the stated scale reading with the horizontal plane of liquid.

8. Reports

As soon as the measurements are completed, each laboratory will send the pilot laboratory a summary of the procedure used and a description of the apparatus (Appendix F). In addition, information and results should be compiled using the Excel Report Form, provided by the pilot laboratory.

The Excel Report Form consists of two parts:

Report Form 1

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

For the balance/s used to determine the weighing value in air/liquid, the following information have 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 weights" for the comparison of mass of hydrometer to the mass of weights, the following information is to be given:

a) Manufacturer, type and material of the weights.
b) Identification of weights, their values and standard uncertainties.
c) Date of last calibration and traceability.

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

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

Report Form 2(ID Hydrometer)

For each hydrometer, general information and results of measurements have to be given.
General information have 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 have 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) Value of the additional weight (ballast) if a stainless steel weight is added to the hydrometer to cause it to sink, measurement correction and uncertainty.
b) Mean values of the hydrometer weighing value at the indicated scale reading. The standard deviation for the mean and the temperature and density 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" [3].
A pooled experimental standard deviation characterizing the hydrometer weighing value in buoyant liquid have to be given [3 (H.3.6)].

Table 1-7: Uncertainty of the hydrometer corrections

The uncertainty of measurement for the corrections is calculated 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 $t_{95}(\nu_{\mathrm{eff}})$ taken from the t-distribution for a 95% confidence level and the expanded uncertainty for the corrections as $U_{95} = t_{95}(\nu_{\mathrm{eff}}) \cdot u_{\mathrm{c}}$.

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 the results).

9. Reference

[1] S. Lorefice et al "CCM key comparison CCM.D-K4 'Hydrometer'", Metrologia, 2016, 53 07003

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

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

Appendices

A. Participants

List of Participants				
NMI	**Country**	**Contact Person**	**E-mail**	**Address**
INRIM	Italy	Andrea Malengo	a.malengo@inrim.it	Istituto Nazionale di Ricerca Metrologica (INRIM) Strada delle Cacce, 73 10135 Torino Italy
PTB	Germany	Daniela Eppers	daniela.eppers@ptb.de	Physikalisch-Technische Bundesanstalt (PTB) AG 1.13 Bundesallee 100 38116 Braunschweig Germany
UME	Turkey	Ümit Y. AKÇADAĞ	umit.akcadag@tubitak.gov.tr	TÜBİTAK UME (UME) TÜBİTAK Gebze Yerleşkesi Barış Mah. Dr.Zeki Acar Cad. No:1 41470 Gebze / KOCAELİ Turkey
IPQ	Portugal	Andreia Furtado	afurtado@ipq.pt	Instituto Português da Qualidade (IPQ) Rua António Gião, 2 2829-513 Caparica Portugal
BEV	Austria	Markus Schiebl	markus.schiebl@bev.gv.at	Bundesamt für Eich-u. Vermessungswesen (BEV) Arltgasse 35 1160 Wien Austria
BFKH	Ungary	Csilla Vámossy	vamossy.csilla@bfkh.gov.hu	Budapest Főváros Kormányhivatala (BFKH) 1124 Budapest Németvölgyi út 37-39. Hungary
DMDM	Serbia	Jelena Bebić	jelenabebic@dmdm.rs	Directorate of Measures and Precious Metals (DMDM) Mike Alasa 14, 11000 Belgrade Serbia
DZM	Croatia	Mladen Bezjak	mladen.bezjak@dzm.hr	State Office for Metrology (DZM) Ibrišimovićeva 11 10000 Zagreb Croatia
NMI	China	Wang Jintao	wangjt@nim.ac.cn	Liquid volume and density lab, Building 14, Room 218 Division of Mechanical and Acoustics, National Institute of Metrology of China (NMI) Bei san huan dong lu No18, Chao Yang District, 100029 Beijing, P. R. China

B. Timetable for the comparison

March 2020	Start of measurements (pilot laboratory)
March 2020 to November 2020	Measurements by all participants and Reports from all participants
December 2020	Draft A of the comparison
February 2021	Draft B of the comparison: end of comparison

C. Circulation scheme for the comparison

Petal no. 1 (With ATA Carnet)		
2nd March	16th March	INRIM
16nd March	14th April	PTB
14th April	18th May	UME
18th May	15th June	DMDM
15th June	6th July	INRIM
6th July	24th August	NMI
24th August	21 September	BFKH
21 September	9th October	INRIM

Petal no. 2		
2nd March	16th March	INRIM
16nd March	14th April	PTB
14th April	8th June	IPQ
8th June	29th June	BEV
29th June	20th July	DZM
20th July	21th September	BFKH
21th September	9th October	INRIM

D. Receipt of the standards

To monitor the progress of the comparison, it is required, upon receipt of the package, to kindly send an e-mail to:

- pilot laboratory

Andrea Malengo

e-mail: a.malengo@inrim.it

Object: EURAMET.M.D-K4 comparison on hydrometers
This e-mail should contain the information about the status of the hydrometers and their packaging.

E. End of measurements and shipping

To monitor the progress of the comparison, it is required, upon completion of the measures, before shipment, to kindly send an e-mail to:

- pilot laboratory
Andrea Malengo
e-mail: a.malengo@inrim.it
- the contact person of the next laboratory

Object: EURAMET.M.D-K4 comparison on hydrometers
This e-mail should contain the following information:
- date of the end of measurements
- shipping date and tracking number (if possible)

F. Results

Calibration results should be sent within three weeks after the end of the measurements.
it is required to kindly send an e-mail to:

- pilot laboratory
Andrea Malengo
e-mail: a.malengo@inrim.it

Object: EURAMET.M.D-K4 comparison on hydrometers
This e-mail should contain the following information:
- Calibration results (Excel Report Form)
- A summary of the procedure used and a description of the apparatus