



Final report

CCM Key Comparison CCM.D-K4 “Hydrometer”

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Abstract

This report presents the comparison philosophy, methodology, analysis and the results of the designed CCM.D-K4 key comparison that covered the calibration of high resolution hydrometers for liquid and alcoholometers in the density range 600 kg/m^3 to 2000 kg/m^3 at the temperature of $20 \text{ }^\circ\text{C}$. The main purpose of this comparison was not only to evaluate the degree of equivalence in the calibration of high accuracy hydrometers between NMI participants, but also to link, where it is possible, the results of previous Comparisons to Key Comparison Reference Values (*KCRVs*) of CCM.D-K4. Eleven NMI laboratories took part in the CCM.D-K4 divided in two groups (petals).

With the CCM.D-K4 purpose, two similar sets consisting of three hydrometers for liquid density determinations and an alcoholometer were circulated to the NMI participants as a travelling standard in the time interval from January 2011 to April 2012.

Twelve Key Comparison Reference Values (*KCRVs*) for each petal have been obtained at the density values related to the tested density marks of the transfer standards by the results of participants. The *KCRVs* and corresponding uncertainties were calculated by the weighted mean in the case of consistent results, otherwise the median was used.

The degree of equivalence (*DoE*) with respect to the corresponding *KCRV* was determined for each participant and, in this particular comparison, the Weighted Least Squares (WLS) method was used to link the individual *DoE* of each participant by a continuous function.

Significant drift of the transfer standards was not detected.

This report also gives instructions on calculating pair-wise degrees of equivalence, with the addition of any information on correlations that may be necessary to estimate more accurately as well as the procedure for linking international comparisons to the CCM.D-K4. Finally an example of linkage to the CCM.D-K4 is given by dealing with the results of the bilateral comparison between INRiM and NMIA, which was added to this comparison so that all participants were engaged after the breakage of the 9340171 artefact.

A particularly good agreement was found among the results provided by most of the participants, even if some systematic differences and either underestimated or overestimated uncertainties of the submitted results can be identified with respect to the *KCRVs*. In general the deviations of the laboratory results to the *KCRVs* are within of 1/3rd to 1/4th of a scale division and the uncertainty at 95 % is usually within half a division. During the analysis of the submitted results, a systematic difference between the first and last immersed mark was also noted, possibly due to a temperature gradient along the stem and/or wetting of the stem around the tested mark, and therefore a corrected claimed uncertainty from each laboratory is expected. However this comparison may help the laboratories to solve some residual or marginal problems as well as to better understand the uncertainty components.

The comparison fully supports the calibration measurement capabilities table in the BIPM key comparison database (KCDB). The results can be used to link regional comparisons to this CCM key comparison

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1. PREFACE

A CIPM key comparison concerning the calibration of high resolution hydrometers for liquid density determinations in the range 600 kg/m^3 to 2000 kg/m^3 at the temperature of $20 \text{ }^\circ\text{C}$ 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 to:

- Recognize and compare the different experimental setups and calibration methods applied by the participants;
- Compare the participants’ methods for the uncertainty evaluation and ascertain the consistency of their calibration results;
- Allow linkage of the regional comparisons previously performed under the auspices of the Regional Metrology Organizations APMP, EURAMET and SIM.

This CIPM key comparison program, designated as CCM.D-K4 “Hydrometer”, was coordinated by the Istituto Nazionale di Ricerca Metrologica (INRiM, Italy). The Physikalisch-Technische Bundesanstalt (PTB, Germany) and the Centro Nacional de Metrologia (CENAM, Mexico) supported the pilot to determine the technical protocol for this key comparison. A total of 11 National Metrology Institutes, divided into two groups took part in this comparison.

The Key comparison CCM.D-K4 “Hydrometer”, which covered the density range 600 kg/m^3 to 2000 kg/m^3 at $20 \text{ }^\circ\text{C}$, comes after various comparisons in “Hydrometry” performed everywhere since the 90s. For the purpose of this project two similar sets consisting of three high-accuracy hydrometers for liquid density determinations and an alcoholometer (alcohol hydrometer) cl.1 were arranged and each set of them was circulated to each of the two groups as travelling standards in the time interval from January 2011 to April 2012.

The corrections to be applied to the stated scale readings at $20 \text{ }^\circ\text{C}$ is often the parameter compared in inter-comparisons. Each laboratory, belonging to an individual petal, determined the corrections to be applied to three stated scale readings at $20 \text{ }^\circ\text{C}$ of different hydrometers in accordance with the technical protocol and the report form received in advance [1].

The present report, which is based on the uniform treatment of the results of the participants, describes the organization of the actual project, the method for analysis of the calibration data and the comparison results.

The results also allowed determination of the degrees of equivalence of each participating NMI with the Key Comparison Reference Values (KCRV) of the CCM.D-K4.

The Final report is intended to be a publication for the CIPM Key Comparison Data Base.

Table 1. List of the participating NMIs and technical contacts.

Laboratory	Country code	Contact Person
Istituto Nazionale di Ricerca Metrologica (INRiM) Strada delle Cacce, 91 -73 10135 Torino - ITALY	IT	Salvatore Lorefice s.lorefice@inrim.it Tel: +39 011 3977 929 Fax: +39 011 3977 937
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Central Office of Measures - Główny Urząd Miar (GUM) Physical Chemistry Department, Density, Viscosity and Spectral Analysis Laboratory, ul. Elektoralna 2 00-139 Warszawa - POLAND	PL	Elżbieta Lenard, e.lenard@gum.gov.pl Tel :+ 48 22 581 9410 Fax :+ 48 22 581 9395
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2. ORGANIZATION

2.1 Participants and schedule

Ten NMIs, plus the pilot institute, agreed to participate in the comparison ab-initio.

Table 1 comprises the participating NMIs and the technical contacts.

For the purpose of this project, the participating laboratories were divided into two groups (petal A and petal B) the three laboratories INRiM, CENAM and PTB performed measurements in both loops. The organization of the comparison was changed in the density range of $1\,000\text{ kg/m}^3$ due to the breakage of one alcoholometer, a bilateral comparison between INRiM and NMIA was added to the two scheduled petals so that all participants were engaged.

In addition to the unforeseen difficulty relating to the artefact breakage, transportation, customs and administrative constraints forced changes in the original schedule of the comparison.

Table 2 shows the custom formalities required for movement of goods in international trade and Table 3 shows the final circulation scheme defined for each artefact.

2.2 Transfer standards (hydrometer samples)

INRiM supplied two similar sets of four hydrometers to be assigned to each petal and used as transfer standards (TSs) at $20\text{ }^{\circ}\text{C}$, as shown in Table 4.

Each set consisted of two hydrometers with a scale division of 0.1 kg/m^3 working close to 600 kg/m^3 and 1500 kg/m^3 , an alcoholometer (alcohol hydrometer) cl.1 and a hydrometer with a scale division of 0.2 kg/m^3 working close to 2000 kg/m^3 . The cubic expansion coefficient for all hydrometers was assumed to be $25\cdot 10^{-6}\text{ }^{\circ}\text{C}^{-1}$ with an uncertainty of $2\cdot 10^{-6}\text{ }^{\circ}\text{C}^{-1}$, rectangular distribution.

During the transfer one of the two alcoholometers was broken and it was replaced with another one having the same physical features.

Table 2. Custom formalities required for the movement of the transfer standards through the Laboratory participants

Formalities	Petal A	Petal B
A.T.A Carnet	Japan (NMIJ)	Australia (NMIA), Korea (KRISS), USA (NIST)
Proforma invoice	Mexico (CENAM), Uruguay (LATU)	Mexico (CENAM)
Free circulation	France (LNE), Germany (PTB), Poland (GUM), Italy (INRiM)	Hungary (MKEH), Italy (INRiM)

The pilot laboratory did not detect any significant change in calibration on any of the artefacts after returning from circulation. Although at present these conditions did not allow to determine the stability of each artifact for a long time, they confirm the stability of the transfer standards during the comparison.

2.3 Conditions selected

The corrections to be applied to the stated scale readings at 20 °C is usually the measurand in hydrometry. The participating laboratories were asked to calibrate the assigned hydrometers at three graduations i (1 to 3) of the scale and the correction C_i had to be calculated for each one of them at the reference temperature of 20 °C:

$$C_i = \rho_{x_i} - \rho_{r_i}. \quad (1)$$

The participants were free to perform all measurements using their own procedure as well as determining the density ρ_{x_i} of the buoyant liquid in which the hydrometer would freely float at the scale mark ρ_{r_i} .

The test points and the surface-tension values of the liquid, in which each hydrometer was intended to be used, were stated in advance.

It was, however, required that the hydrometers only stay at the laboratory for the time necessary for calibration and not longer than the allotted time.

When the standards arrived at the participating laboratory, a visual inspection was made and each artefact was allowed to acclimate to the laboratory environment in agreement with the given instruction.

INRiM collected all information concerning the status of the transfer standards, the apparatus used during the comparison and the measurement results of each participant n of the j petal ($j=A$ or B), hereinafter referred to as x_{jni} instead of the symbol C_i of equation (1) at the tested density mark i .

Table 3. Final circulation scheme defined for each artefact.

Id. hydrometer Range in kg/m³ Institute (scheduled period)	9342351	9342348	9340170	9340171	9340172	9343460	9343462	9346684	9346688
600 - 610 600 - 610 990 - 1 000 990 - 1 000 990 - 1 000 1 490 - 1 500 1 490 - 1 500 1 980 - 2 000 1 980 - 2 000	600 - 610	600 - 610	990 - 1 000	990 - 1 000	990 - 1 000	1 490 - 1 500	1 490 - 1 500	1 980 - 2 000	1 980 - 2 000
INRiM (29/12/10 – 20/01/11)	X	X	X	X	X	X	X	X	X
NMIA (04/03 – 17/03/11)		X	X				X		X
CENAM (29/03 – 26/04/11) (06/06 – 05/07/11)	X	X		X	X	X	X	X	X
LATU (03/08 – 31/08/11)	X				X	X		X	
NMIJ (14/10 – 24/10/11)	X				X	X		X	
KRISS (06/01 – 14/01/12)	X				X	X		X	
NIST (28/07 – 03/08/11)		X		X			X		X
LNE (04/10 – 16/11/11)		X		X			X		X
GUM (01/12/11 – 26/01/12)		X		X			X		X
MKEH (06/02 – 20/02/12)		X		X			X		X
PTB (12/03 – 17/04/12)	X	X		X	X	X	X	X	X

Table 4. Technical information and a picture related to the reference hydrometers used as transfer standards (TS) in this key comparison.

Petal A	Petal B	Scale div.	Φ of body [mm]	Length of body [mm]	Φ of stem [mm]	Length of stem [mm]	Weight [g]
0.600–0.610 g/cm ³	0.600–0.610 g/cm ³	0.000 1 g/cm ³	28	260	5.5	140	90
0 – 10 % Vol.	0 – 10 % Vol.	0.1 % Vol	28	240	4	230	130
1.490 – 1.500 g/cm ³	1.490 – 1.500 g/cm ³	0.000 1 g/cm ³	32	270	4.5	140	290
1.980 – 2.000 g/cm ³	1.980 – 2.000 g/cm ³	0.000 2 g/cm ³	29	260	4	170	295



2.4 Procedure and method of measurement

Table 5 summarizes the differences in the equipment used at each NMI and in their calibration procedures.

All participants carried out their task by adopting the hydrostatic weighing in a single liquid, the density of which was known for the test temperature (Cuckow’s method).

Four laboratories: KRISS, NMJJ, NMIA and LATU used two different balances for weighing the tested hydrometer in air and immersed in the buoyant liquid, respectively. The weighing method was the direct reading of the balance for INRiM, MKEH, NIST, NMJJ and LATU while the laboratories CENAM, KRISS, PTB, GUM, LNE and NMIA used the balance as a comparator and the substitution weighing by means of calibrated weights was performed to achieve the balance readings within a narrow electronic range.

Straight chain alkanes such as *n*-Pentadecane, *n*-Nonane, *n*-Tridecane and *n*-Tetradecane were used by the different laboratories as buoyant liquids. Each participant measured the density of the used liquid by hydrostatic weighing, oscillating-type density meter, and sensitive standard hydrometers. However each laboratory monitored the stability of the density of the buoyant liquid at least before and after the hydrometer calibration by using the same instruments and standards used to determine the density of the buoyant liquid.

The majority of participants measured the surface tension of the buoyant liquid by commercial available tensiometers by applying different measuring methods, CENAM used the stem measurement with the hydrostatic weighing and the MKEH knew the values of interest from reference data.

The mean of the parameters contributing to the air-density calculation were recorded during calibration, i. e. pressure, temperature, relative humidity (or dew point). With the exception of KRISS, all laboratories assumed a constant value of 0.04% for the CO₂ content. The mean of the air-density values was calculated by the CIPM formula (CIPM2007) [2] and reported.

Accurate calipers or suitable instruments with a resolution between 0.01 and 0.1 mm were used to measure the diameter of the stem of the hydrometer to be calibrated. PTB used an automatic measuring device by means of which the separation between graduation mark and the stem diameter throughout the whole scale of each hydrometer to be calibrated were measured.

In general, the alignment of the liquid horizontal plane with the selected scale-mark is identified by CCD camera servo-assisted with a desktop computer or by monitor, a few laboratories used a magnifier. The alignment principles by CCD camera were independently based on [3, 4, 5] although technical modifications were made by the users.

Table 5. Summary of the experimental facilities operated at the different NMIs.

Institute	Balance Max capacity [g]/readability [g]		Buoyant liquid	Thermostat type, capacity	Thermometer for liquid temperature	Alignment	Surface tension method
	Weighing in air	Hydrostatic weighing					
INRiM	505 / 0.000 01		n-Nonane	Double-walled glass vessel, 30 litre	Pt100 Ohm, ASL F17	CCD camera PC aided	Plate
CENAM*	405 / 0.000 1		n-Pentadecane	Tamson TV7000, 70 litre	Pt100 Ohm, ASL F250	CCD camera PC aided	Stem method, hydrostatic weighing
KRISS*	405 / 0.000 1	230 / 0.000 01	n-Tridecane	Lauda RP1840, 21 litre	Pt100 Ohm, ASL F300	CCD camera PC aided	Ring
PTB*	1 109 / 0.000 01		n-Tridecane	Tamson TVBX 70, 70 litre	SPRT 25 Ohm, PAAR MKT25	CCD camera PC aided	Plate
MKEH	1 000 / 0.000 1		n-Nonane	Tamson TMV70, 70 litre	Pt100 Ohm, Hart Sci Black Stack 1560	Magnifier hand-operated	Reference data
NIST	205 / 0.000 01		n-Tridecane	Hart 7011, 20 litre	YSI Thermistor, Keithley 195A	laser sheet + laser power meter	Du Noüy & Wilhelmy ring
NMIJ	520 / 0.000 1	520 / 0.000 1	n-Tridecane	Double-walled PVC and aluminium vessel, 30 litre	Pt100 Ohm, ASL F200	Magnifier hand-operated	Plate
GUM*	1 109 / 0.000 01		n-Nonane	Tamson TV7000, 70 litre	25 Ohm PRT, ASL F700B	Magnifier electronically controlled support	Ring
LNE*	405 / 0.000 1		n-Tetradecane	Tamson TV7000, 70 litre	Pt100 Ohm, HP34420A	Magnifier hand-operated	Du Noüy & Wilhelmy ring
NMIA*	1 000 / 0.000 1 160 / 0.000 01	205 / 0.000 01	n-Nonane	Colora NB33498, 25 litre	Pt100 Ohm, Hewlett Packard	CCD camera hand-operated	Ring
LATU	1200 / 0.001	204 / 0.000 1	n-Tridecane	Tamson TV7000, 70 litre	Pt100 Ohm	Magnifier hand-operated	Ring

* **Weighing procedure:** By substitution method

Mechanical devices were used for sinking the tested hydrometer or additionally, sinkers adjustable in height were used in order that the liquid level corresponds to the scale mark concerned.

2.4.1 Uncertainty claims of NMIs

In addition to the uncertainty contributions proposed in the worksheet by the coordinating laboratories [1], several laboratories have included the contribution due to standard deviation of the mean of corrections or reproducibility of measurements. Moreover PTB also took into account the contribution due to the influence of the temperature distribution in the bath, NMIJ the contributions due to the weighing value of suspension and the density of used weights and NIST considered tridecane density changing, surface tension, and positioning the liquid surface at the mark to be calibrated (contribution due to the contact angle of the buoyant liquid on the hydrometer stem).

3. RESULTS

After the participating laboratory completed its own measurements, all information concerning the calibration was submitted to the pilot laboratory using the sheets Report Form 1 and Report Form 2 annexed to [1].

3.1 Data received from participants

The INRiM collected and analysed anything related to:

- a) Details of the instrumentation used by each participant in the project, including the origin of their traceability to the SI.
- b) Details of the relevant information on the measurements and parameters used for the comparison such as local gravity, mass measurements, density of working fluid and, finally, the ambient conditions including data on air density, air temperature, air pressure, humidity and CO₂ content.
- c) Calculated values of the three corrections for each transfer standard at the specified reading marks and surface tension values.
- d) Uncertainty budget of the twelve calculated corrections, which were estimated and combined following GUM [6] under the responsibility of each participating institute. Each laboratory also reported the uncertainty of all measured quantities 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 $U_{95} = t_{95}(\nu_{\text{eff}}) \cdot u_c$.

- e) Analysis of reported data, INRiM elaborated all laboratory data as soon as they were available. Some participants were also invited to check for results that appeared to be anomalous before the Draft A was sent to them in agreement with the CIPM MRA-D-05, March 2014 [7].

3.2 Communication from the participating laboratories

The alcoholometer S/N 9340170 broke during the circulation and as a result, it was decided that the calibration results obtained by NMIA and INRiM would be presented as a bilateral comparison.

LATU detected some problems with their apparatus during the calibration of the three hydrometers in the range from 1 000 kg/m³ to 2 000 kg/m³; the expected results of the transfer standards identified with the Serial Numbers 9340172, 9343460 and 9346684 were not submitted.

After the first issue of the draft A circulated among the participants, the MKEH and the NIST submitted new results.

Before the issue of the draft A the MKEH staff suspected that the building reconstruction in the laboratory during the comparison measurements could have affected its results. The MKEH was able to detect the possible cause after the conclusion of the draft. The influence of temperature changing in the room during the immersion was the main problem. A density correction because of the temperature influence has been made.

NIST made a mistake about the units used for the uncertainties, as reported in in the data report (Form2). NIST thought the units were parts per million when they were actually g/cm³·10⁻⁶. In addition the laboratory suspected that density changes of the buoyant liquid over time due to evaporation of less dense impurities affected the own measurements and gave communication to pilot laboratory that:

“NIST results show differences between 39×10^{-6} g/cm³ and 77×10^{-6} g/cm³ from the KCRVs. Note that the currently approved NIST CMC uncertainty is 100 ppm, but smaller, customized uncertainty values were used for this KC. SIM.M.D-K4 2007 – 2008 covered density range 601 kg/m³ to 1 300 kg/m³ and NIST’s degrees of equivalence with the reference values ranged from -15 ppm to 30 ppm. Moreover, NIST suspects that density changes of their tridecane over time due to evaporation of less dense impurities is the source of the larger degrees of equivalence in this comparison.”

Therefore NIST wrote that ”NIST will:

- employ a higher resolution check standard to better reveal changes in their Cuckow’s apparatus. (Presently, a check standard is used before each batch of customer calibrations and the results must agree with prior results within 100 parts in 10^6 , the NIST CMC uncertainty).
- measure the properties of the tridecane more frequently to track changes due to evaporation.
- not seek a lower CMC than our current value of 100 ppm until future comparisons support such reductions.”

3.3 Interpretation of the comparison: the degree of equivalence *DoE*

According to the CIPM MRA, the degree of equivalence (*DoE*) must be established for each comparison, which can provide traceability for a calibration measurement capability announced in the Key Comparison Data Base, KCDB, Appendix C, by the laboratory.

DoE must be calculated as the deviation of the value reported by the laboratory from the key comparison reference value evaluated in the framework of an internationally accepted key comparison.

Twelve Key Comparison Reference Values ($i=1$ to 12) have been obtained at the tested density mark of the four transfer standards for each petal by the results of participants in the range 600 kg/m^3 to 2000 kg/m^3 . To quantify the discrepancies, tests of equivalence of the measurements are based on a recommended method used for key comparisons [8].

The results from each of the n laboratories of the j petal ($j=A$ or B) have then been characterized in terms of a ‘degree of equivalence’ representing the deviation D_{jni} of its result x_{jni} from the estimated Key Comparison Reference Value ($KCRV_{ji}$) at the tested density mark i as

$$D_{jni} = x_{jni} - KCRV_{ji} \quad (2)$$

with the associated uncertainty

$$u(D_{jni}) = \left(u^2(x_{jni}) + u^2(KCRV_{ji}) - 2u(x_{jni}, KCRV_{ji}) \right)^{\frac{1}{2}}. \quad (3)$$

In equation (3), the $u(KCRV_{ji})$ is interpreted as an estimate of the reference value uncertainty made on the basis of the measurements provided by the participating institutes, and $u(x_{jni}, KCRV_{ji})$ is the possible covariance term between the laboratory results and the reference value.

The Reference value firstly was determined by weighted mean of the institutes’ measurements (Procedure A), using the inverses of the squares of the associated standard uncertainties as the weights:

$$KCRV_{ji} = \left(\sum_n \frac{x_{jni}}{u^2(x_{jni})} \right) \left(\sum_n \frac{1}{u^2(x_{jni})} \right)^{-1}. \quad (4)$$

The individual result x_{jni} of each of the n laboratory results was considered consistent with the “Reference value” at the 95 % of the level of significance if

$$|D_{jni}| \leq 2u(D_{jni}). \quad (5)$$

Such a reference value, however, is not applicable if some of the institutes’ measurements appear to be anomalous or discrepant.

To identify the overall consistency of the claimed results, a chi-squared test was then applied considering the consistency check as failing if

$$\Pr\{\chi^2(\nu) > \chi^2_{obs}\} < 0.05 \quad (6)$$

where $\Pr$ denotes “probability of”, $\nu = n - 1$ is the number of degrees of freedom and

$$\chi^2_{obs} = \frac{(x_1 - x_{ref})^2}{u^2(x_1)} + \dots + \frac{(x_n - x_{ref})^2}{u^2(x_n)}$$

is the observed chi-squared value.

If the test was not satisfied, the $KCRV_{ji}$ is the median value amongst the submitted measurement results (Procedure B).

By means of a Monte Carlo simulation, 100 000 random samples were generated, each made of N values drawn from the distributions representing the results from each laboratory (N , here, is the number of the laboratories of the relevant petal). In this way, 100 000 values for the median of the drawn samples were obtained. The mean of such values was taken as the $KCRV_{ji}$ of the single tested mark i . Also the corresponding simulated deviation terms of the degrees of equivalence were obtained for each laboratory and used to determine a 95% coverage interval for the laboratories’ deviations from the $KCRV_{ji}$.

3.4 Key Comparison Reference Values (KCRVs) and degrees of equivalence

The current results of the key comparison corresponding to the involved A and B “j” petals are reported in the **Appendix A1** relating to each tested hydrometer.

The measurement results with the corresponding uncertainties claimed by the participants are listed in the odd table “A” or “B” which also provides the $KCRVs$ with their own extended uncertainty, the lower and upper limits of the coverage interval if procedure B was applied and the χ^2_{obs} value at each tested mark. The even table “A” or “B” for the same artefact shows at

each tested mark of each “ n ” laboratory of the petal the degree of equivalence to the $KCRV$ with the coverage interval. Moreover the table reports the arithmetic mean of the three degree of equivalence corresponding to the “ k ” tested hydrometer $\Delta_{jnk} = \frac{\sum_i D_{jni}}{3}$ with its estimated standard uncertainty $u_{\Delta_{jnk}} = \left(u_{D_{jni}}^2 + \frac{(D_{jnimax} - D_{jnimin})^2}{12} \right)^{\frac{1}{2}}$. Corresponding to the same tested artefact, the figure also shows the degree of equivalence with respect to the $KCRV$ of each participant of the relevant petal related to each calibrated mark and their arithmetic mean with their own expanded uncertainties.

The average of the three degrees of equivalence to the $KCRVs$, Δ_{jnk} and the corresponding estimated standard uncertainties are a good indicator of the ability of the laboratory n in the calibration of hydrometers, because it shows the laboratory repeatability. The estimated standard uncertainty $u_{\Delta_{jnk}}$ takes into account the associated uncertainty of the individual degree of equivalence $u_{D_{jni}}$ calculated by (3), and the reproducibility in the calibration of the laboratory by assuming a rectangular distribution from the dispersion of the individual degrees of equivalence with respect to the three $KCRVs$ for each transfer standard.

Table 6 shows the Δ_{jnk} at the corresponding petal j of each k tested hydrometer for each n participant to the comparison, its uncertainty, and finally information about the consistency of each claimed result from the laboratory, according to equation (5): “A” the result is consistent, “B” the result is not consistent.

In general it was observed that:

- **Petal A** shows that all data are rather close to the weighted mean values. The experimentally observed χ_{obs}^2 values are always lower than 11.07 with 5 degrees of freedom and 9.47 with 4 degrees of freedom. The LATU results were not considered in the $KCRV$ calculations with the exception in the range 0.600 g/cm³ and 0.610 g/cm³.
- **Petal B** shows that some laboratories exhibit a largest deviation related to the reference values. In such inconsistent case, the mean of medians was taken as the $KCRV_{ji}$. In particular that was due to the results of NIST and MKEH at 0.601 g/cm³, LNE at 0.985 g/cm³ and 0.991 g/cm³, and again MKEH at 1.981 g/cm³. The NMIA results were not considered in the reference value calculations in the range 0.985 g/cm³ and 0.998 g/cm³.

Table 6. Arithmetic mean value of the three degrees of equivalence of the NMIs with respect to the $KCRVs \Delta_{jnk}$, at the corresponding petal j of each k tested hydrometer for each n participant to the comparison and the relevant uncertainty. Table also shows information about the consistency of each laboratory result i with the individual *Key Comparison Reference Value* in the range of the tested hydrometer: “A” the result is consistent, “B” the result is not consistent, the different colours give a level of attention (**orange: medium attention, yellow: low attention**)

Range (k) / g cm ⁻³		0.600 0 - 0.610 0			0.985 9 - 0.996 7			1.490 0 - 1.500 0			1.980 0 - 2.000 0		
	Laboratory (n)	$\Delta_{jnk} \times 10^{-6}$	$U(\Delta_{jnk}) \times 10^{-6}$	$ D_{jni} \leq 2u(D_{jni})$	$\Delta_{jnk} \times 10^{-6}$	$U(\Delta_{jnk}) \times 10^{-6}$	$ D_{jni} \leq 2u(D_{jni})$	$\Delta_{jnk} \times 10^{-6}$	$U(\Delta_{jnk}) \times 10^{-6}$	$ D_{jni} \leq 2u(D_{jni})$	$\Delta_{jnk} \times 10^{-6}$	$U(\Delta_{jnk}) \times 10^{-6}$	$ D_{jni} \leq 2u(D_{jni})$
Petal A (j)	INRiM	-6	14	AAA	-9	21	AAA	-7	36	AAA	-8	34	AAA
	CENAM	5	16	AAA	8	33	AAA	8	38	AAA	12	50	AAA
	LATU	-34	33	ABB									
	NMIJ	8	11	AAA	-6	26	AAA	-1	30	AAA	-9	54	AAA
	KRISS	1	24	AAA	11	34	AAA	5	51	AAA	8	71	AAA
	PTB	-2	14	AAA	5	21	AAA	-1	18	AAA	3	30	AAA
Petal B (j)	INRiM	-1	15	AAA	1	25	AAA	2	33	AAA	-11	44	AAA
	NMIA	-6	8	AAB				-9	13	AAA	-13	27	AAA
	CENAM	2	16	AAA	-10	32	AAA	3	39	AAA	-6	46	AAA
	NIST	46	46	BAA	45	57	AAA	46	76	AAA	63	111	AAA
	LNE	-10	18	AAA	-28	28	BBA	-16	29	BAA	-15	39	AAA
	GUM	-1	12	AAA	-4	21	AAA	1	25	AAA	1	36	AAA
	MKEH	16	14	BBA	7	28	AAA	16	27	BAA	16	49	BAA
	PTB	2	14	AAA	7	19	AAA	13	21	AAA	27	38	AAA

Table 7. Differences of the average of the degree of equivalence of the INRiM, CENAM and PTB who measured all Transfer standards.Division of the scale 0.0001 g cm^{-3} in the density range 0.600 g cm^{-3} and 1.500 g cm^{-3} .Division of the scale 0.0002 g cm^{-3} in the density range 1.980 g cm^{-3} and 2.000 g cm^{-3} .

	Range / g cm^{-3}	$d_{jk} \times 10^{-6}$		
		INRiM-CENAM	INRiM-PTB	CENAM-PTB
Petal A	0.600 - 0.610	-11	-4	7
	0.986 - 0.997	-17	-13	3
	1.490 - 1.500	-15	-6	8
	1.980 - 2.000	-21	-12	9
Petal B	0.600 - 0.610	-2	-2	0
	0.986 - 0.997	12	-5	-17
	1.490 - 1.500	-1	-10	-9
	1.980 - 2.000	-5	-38	-33

Furthermore the stability of the circulated standards was confirmed from the difference of the average between the INRiM, CENAM and PTB measurements who tested them in both petals and can be estimated as approximately one tenth (1/10) of a division of the scale, Table 7.

3.5 Continuous functions of *DoEs*

Individual degrees of equivalence *DoEs* between the values reported by participants and the corresponding *KCRVs*, as well as the average value related to the range of each tested hydrometer for both petals, have been introduced in the above sections. As each participating laboratory reported a total of 12 density values for the four hydrometers, it is better to express a set of *DoEs* of each participant as a function of nominal density values ρ_i , such as 600 kg/m^3 , 1000 kg/m^3 , 1500 kg/m^3 , etc. A regression curve (i.e. first order curve) for corresponding *DoE* is therefore proposed for each participant n from its own set of *DoEs*.

In order to introduce all sets of values, a matrix equation takes the following form

$$\mathbf{D}_n = \boldsymbol{\rho}_i \boldsymbol{\beta}_n + \boldsymbol{\varepsilon}_n \quad (7)$$

where $\mathbf{D}_n$ is the column vector of the degrees of equivalence of the participant n , $\boldsymbol{\rho}_i$ is the matrix of the i tested nominal values of density, $\boldsymbol{\varepsilon}_n$ the residual vector of the fitting and, $\boldsymbol{\beta}_n$ is the column vector which contains the slope m_n and the intercept b_n of the proposed straight line fitting:

$$\mathbf{D}_n = \begin{bmatrix} D_{n1} \\ D_{n2} \\ \vdots \\ D_{n11} \\ D_{n12} \end{bmatrix} ; \quad \boldsymbol{\rho}_i = \begin{bmatrix} \rho_1 & 1 \\ \rho_2 & 1 \\ \vdots & \vdots \\ \rho_{11} & 1 \\ \rho_{12} & 1 \end{bmatrix} ; \quad \boldsymbol{\beta}_n = \begin{bmatrix} m_n \\ b_n \end{bmatrix} ; \quad \boldsymbol{\varepsilon}_n = \begin{bmatrix} \varepsilon_{n1} \\ \varepsilon_{n2} \\ \vdots \\ \varepsilon_{n11} \\ \varepsilon_{n12} \end{bmatrix} .$$

The weighted least square method (WLS) can be used for calculating the regression curves, the solution of (7) by WLS is

$$\hat{\beta}_n = (\mathbf{p}_i^T \Psi_{D_n}^{-1} \mathbf{p}_i)^{-1} \mathbf{p}_i^T \Psi_{D_n}^{-1} \mathbf{D}_n \quad (8)$$

where concerning the participant n the weighting matrix $\Psi_{D_n}^{-1}$, is formed by the variance (and covariance) of the corresponding *DoEs* uncertainties given in Table 6 and the term $\Psi_{\hat{\beta}_n} = (\mathbf{p}_i^T \Psi_{D_n}^{-1} \mathbf{p}_i)^{-1}$ is assumed to be the variance-covariance matrix of the best fit parameters $\hat{\beta}_n$. For the correlation coefficient 0.9 for *DoEs* corresponding to the calibration of same hydrometer can be assumed and 0.3 for *DoEs* corresponding to the calibration of different hydrometers. An additional test, i.e chi-square test, can be used to indicate agreement between the observed and predicted values as well as between the estimated variance of the fit and the input uncertainties.

3.6 Degree of equivalence between pairs of NMIs

According to the 14th CCM meeting (February, 2013) pair-wise degrees of equivalence should no longer be published in the KCDB. Information on pair-wise degrees of equivalence published in KC reports should be limited to the equations needed to calculate them, with the addition of any information on correlations that may be necessary to estimate them more accurately.

In brief, mathematically the pair-wise degree of equivalence for each pair of laboratories p and q is the difference d_{pq} of the comparison results of the two laboratories:

- Both laboratories p and q are in the same loop. The degree of equivalence $d_{pq}(\rho_i)$ of each petal at the stated density value or range of ρ_i is the difference between the comparison results x_i of the two independent laboratories

$$d_{pq}(\rho_i) = (D_p - D_q) = (x_p - x_q) \quad (9)$$

Its standard uncertainty is

$$u_{d_{pq}} = (u_{x_p}^2 + u_{x_q}^2)^{\frac{1}{2}} \quad (10)$$

with the expanded uncertainty is $U_{d_{pq}} = 2u_{d_{pq}}$.

Note. The implication of this condition is that there is no mutual dependence of the institute's measurements.

- Anyway, in the usual cases in which the laboratories are shifted in separate loops and different TSs are used, equation (9) should take into account an offset given by the difference

between the set of the reference values of the different loops. Defining by the continuous relations $f_{KCRV_A}(\rho)$ and $g_{KCRV_B}(\rho)$ the offset to be considered, the d_{pq} is

$$d_{pq}(\rho_i) = (x_p - x_q) + (f_{KCRV_A}(\rho_i) - g_{KCRV_B}(\rho_i)) = (D_p(\rho_i) - D_q(\rho_i)) \quad (11)$$

and the uncertainty is

$$u_{d_{pq}} \approx \left(\sum_{k=D_p, D_q | \rho_i} u_k^2 - 2u(D_p, D_q) \right)^{\frac{1}{2}} \quad (12)$$

where $u(D_p, D_q)$ is the correlation term of the *DoEs* with respect to the *KCRVs* of the corresponding loops.

The solution of $u(D_p, D_q)$ can be resumed as follows:

- The independent laboratories p and q worked respectively in the A and B petals, contributing to determine each one the reference values $KCRV_A$ and $KCRV_B$ by means of the two different set of transfer standards. The degree of equivalence between two laboratories can be obtained for a stated density value or range of ρ_i ; the correlation term is given by

$$\begin{aligned} u(D_p, D_q) &= u(KCRV_A, KCRV_B) \\ &= \left(\sum_k \frac{1}{u^2(x_k)} \right) \left(\sum_A \frac{1}{u^2(x_i)} \sum_B \frac{1}{u^2(x_j)} \right)^{-1}, \end{aligned} \quad (13)$$

where i and j represent the laboratories participating in the two loops A and B, respectively.

Note. The three laboratories k (INRiM, CENAM and PTB) make an important contribution in the correlation determination since they worked in both loops. **Appendix A2** explains how to evaluate the degrees of equivalence and their uncertainties between the two institutes, p and q , who participated in the two different loops, A and B.

3.7 Linkage of international comparisons to the CCM.D-K4

A number of linked comparisons have already been published in the KCDB using different methods. The procedure described in [9] to link SIM NMIs to the EURAMET key comparison of EURAMET.M.D-K4 is suitable for linking the results of regional, supplementary and bilateral comparisons to the *KCRVs* of CCM.D-K4 “Hydrometer” through the *DoEs* between the results reported by the joint participation of each participant at this CCM comparison and in the concerning comparisons. In such cases, degrees of equivalence are computed for the participants in the previous and subsequent comparison with respect to all other participants and to the previous key comparison reference value.

By the continuous function $D'(\rho)$ the individual *DoE* to the *KCRV* are computed for the participants in the supplementary comparison

$$D'(\rho) = d_{pq(RMO)}(\rho) + d_{bil}(\rho) + D_{link}(\rho) \quad (14)$$

where

$D_{link}(\rho)$ is the continuous function concerning the *DoEs* between results reported by the linking laboratory (laboratories) with respect to the *KCRVs* in the CCM.D-K4;

$d_{pq(RMO)}(\rho)$ is the continuous function concerning the degree of equivalence between pairs of NMI laboratories, in the Regional comparison;

$d_{bil}(\rho)$ is the continuous function concerning the difference between the result measurement of the two laboratories, the linking laboratory and the laboratory that took part in the Regional and/or supplementary comparison.

In order to calculate the uncertainty of equation (14), numerical simulations by the Monte Carlo method can be performed for each estimated value of $D'(\rho)$ [10].

3.8 The Bilateral comparison INRiM - NMIA and its linkage to CCM.D-K4

During the circulation of the transfer standards, the alcoholometer S/N 9340170, belonging to petal B, was broken and replaced with the S/N 9340171, having the same characteristics in the density range of 1 000 kg/m³. Anyway the calibration results obtained by NMIA and INRiM concerning the broken standard S/N 9340170 were handled as a bilateral comparison between the two participants inside this key comparison. Table 8 shows the measurement results of the two laboratories, the reference values determined by the weighted means with uncertainties at 95 % of the level of confidence and finally the normalized error ratio E_n which, if results between -1 and +1, generally indicates that the measured values are considered consistent.

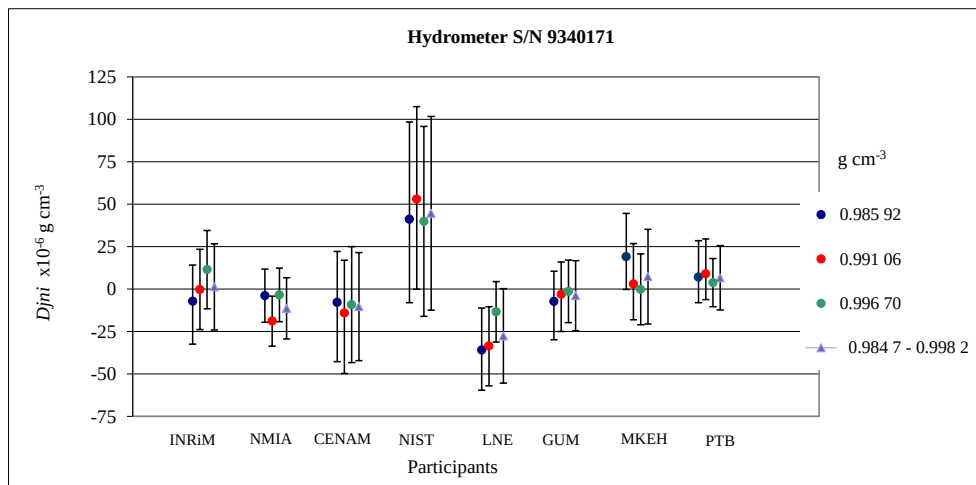
Alcoholometer S/N 9340170		x10 ⁻⁶ / g cm ⁻³	INRiM	NMIA			
Range (0 - 10) % vol	g/cm3				RV (weighted mean)	U (RV)	E _n
1.0	0.996 70		-28	-43	-40	12	0.6
5.0	0.991 06		-51	-70	-66	12	0.7
9.0	0.985 92		-46	-43	-44	12	0.1
Combined std uncertainty of corrections u _c			12	7			
Expanded uncertainty of corrections U ₉₅ = t ₉₅ u _c			24	13			
Student t-factor t ₉₅			2	2			

Table 8. Measurements results as reported by INRiM and NMIA concerning the alcohol hydrometer S/N 9340170. Last column reports the normalized error for each calibrated graduation marks

Table 9, *DoEs* of the NMIA with respect to the CCM. D-K4 *KCRVs* of the Petal B at the three tested density marks with the corresponding expanded uncertainties. Moreover the table shows the mean average of the three tested values in the range 0.9847 and 0.9982 with its uncertainty at the 95 % of the confidence level.

ρ g cm ⁻³	$d_{bil}(\rho) \times 10^{-6}$	$D_{INRiM}(\rho) \times 10^{-6}$	$D'_{NMIA}(\rho) \times 10^{-6}$	$UD'_{NMIA}(\rho) \times 10^{-6}$
0.985 92	3	-7	-4	16
0.991 06	-19	-0,3	-19	15
0.996 70	-15	11	-3	16
0.984 7 - 0.998 2			-11	18

Figure 1. Degrees of equivalence respect to the *KCRV* of each laboratory of the relevant petal identified by the alcohol hydrometer 9340171. The lengths of the bars show the expanded uncertainty of the degree of equivalence related to each calibrated mark and of their average. This Figure replaces the Figure B2 in Appendix.



In order to link the NMIA results to *KCRV* values of the CCM.D-K4 “Hydrometer” for each result at the three tested marks in the range of 1 000 kg/m³, equation (14) is re-written as

$$D'_{NMIA}(\rho) = d_{bil}(\rho) + D_{INRiM}(\rho) \quad (15)$$

where

$d_{bil}(\rho) = x_{(NMIA)}(\rho) - x_{(INRiM)}(\rho)$ is the difference of the measurands as reported by INRiM and NMIA at each tested mark in the bilateral comparison;

$D_{INRiM}(\rho)$ is the individual *DoE* of INRiM with respect to the *KCRV* concerning the CCM.D-K4 at the density related to tested mark.

The variance of each *DoE* of NMIA at the density related to the tested mark result

$$u^2_{D'_{NMIA}}(\rho) = u^2_{d_{bil}}(\rho) + u^2_{D_{INRiM}}(\rho) + 2u(x_{INRiM}, D_{INRiM}) \quad (16)$$

where the term of correlation is assumed to be

$$\begin{aligned}
u(x_{INRiM}, D_{INRiM}) &= -u(x_{INRiM}, x_{D_{INRiM}}) + u(x_{D_{INRiM}}, KCRV_B) \\
&= -u^2(x_{INRiM}) + \left(\sum_j \frac{1}{u^2(x_j)} \right)^{-1} \approx -0.9u_{x_{INRiM}}^2
\end{aligned} \tag{17}$$

where $u(x_j)$ is the uncertainty claimed by each participant belonging to petal B.

The linkage results concerning the NMIA with respect to the *KCRV* values of the CCM.D-K4 “Hydrometer” in the range 1 000 kg/m³, as they were calculated by equations (15) and (16) are shown in Table 9. The Table also shows the average of the three NMIA *DoE* values and the uncertainty at the 95 % level of significance, value which takes into account the repeatability of the calibration results on the three tested marks.

Figure 1 replaces the Figure B2 in Appendix. It shows the degree of equivalence with respect to the *KCRVs* of the concerned NMIs including the determined values of NMIA.

4. DISCUSSION and CONCLUSION

The Key comparison CCM.D-K4 “Hydrometer”, which covered the density range 600 kg/m³ to 2000 kg/m³ at 20 °C, comes after various comparisons in “Hydrometry” performed since the 90s by regional metrology organizations. So, the main purpose of it was not only to evaluate the degree of equivalence between NMI participants in the calibration of hydrometers of high accuracy, but also to establish a base to link, where it is possible, the results of previous comparisons to the Key Comparison Reference Values (*KCRVs*) of CCM.D-K4.

In order to reach such objectives, two similar sets of three high-accuracy hydrometers for liquid density determinations and an alcoholometer were circulated to the NMI participants as a travelling standard in the time interval from January 2011 to April 2012.

The eleven participating NMIs were divided into two groups which to form two loops (petals). The three density laboratories of INRiM, CENAM and PTB performed calibrations in both petals. The calibration measurements of each hydrometer were carried out at three specified division marks at atmospheric pressure. Each laboratory used their own hydrostatic weighing system with their own respective standard liquid such as: n-pentadecane, n-nonane, n-tridecane and n-tetradecane.

Two sets concerned twelve *KRCVs*, for each petal, have been obtained at the tested density marks by the results of participants. The *KRCVs* and corresponding uncertainties were calculated by the weighted mean in the case of consistent results, otherwise the median was used.

The *DoE* with respect to the corresponding *KCRV* was determined for each participant and also, in this particular comparison, the Weighted Least Squares (WLS) method to link the individual *DoE* of each participant by a continuous function has been proposed. Furthermore, information on pair-wise degrees of equivalence was provided: the equations needed to calculate them, and any information on correlations that may be necessary to estimate them more accurately. We also explained the procedure for linking international comparisons to the CCM.D-K4. Finally an example of linkage to the CCM.D-K4 is given by dealing with the results of the bilateral comparison between INRiM and NMIA, which was added to this comparison so that all participants were engaged after the breakage of the 9340170 artefact.

The overall results have shown a very satisfying agreement among the results provided by most of the participants. Technical improvements can be made in some laboratories and, in general, on the uncertainty evaluation. With the exception of few cases, the deviations of the laboratory results to the *KCRVs* are within of 1/3 to 1/4 of a division of scale and the uncertainty at 95% is usually within half a division. Anyway some systematic differences and either underestimated or overestimated uncertainties of the submitted results have been identified with respect to the *KCRVs* which had the responsibility to fail the consistency test of the Reference value of this comparison. During the analysis of the submitted results, a systematic difference between the first and last immersed mark was also noted, possibly due to a temperature gradient along the stem and/or wetting of the stem close to the tested mark [11]. A corrected claimed uncertainty from each laboratory is expected according Table 6. However this comparison may help the laboratories to solve some residual or marginal problems as well as to better understand the uncertainty components.

The CCM.D-K4 “Hydrometer” key comparison fully supports the calibration measurement capabilities table in the BIPM key comparison database (KCDB). The results can be used to link regional comparisons to this CCM key comparison.

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Appendix A1

This section deals with the measurement results and the standard uncertainties as reported by the participants. For each artefact, the calculated CCM *Key-Comparison Reference Value* (*KCRV*) at each calibrated mark concerning with each of the two petals, with the related uncertainty or the lower (U_l) and upper (U_r) limits of the confidence interval if procedure B was applied [8]. Moreover, for each petal and for each artefact the three degrees of equivalence to the *KCRVs* and their average with the corresponding estimated standard uncertainties concerning each of the NMIs are listed and shown.

Appendix A1 – Petal A

Hydrometer S/N 9342351

The *KCRVs* for the petal A, identified by the hydrometer 9342351, have been calculated by applying the “*weighted mean*” method according to the results of the consistency check $\Pr\{\chi^2(\nu) > \chi^2_{obs}\} < 0.05$ of the measurement results and standard uncertainties of the participants reported in

Table A.1. The **Table** shows also each calculated *KCRV* with the expanded uncertainties and the χ^2_{obs} values.

Table A.2 and **Figure A.1** present the degrees of equivalence to the *KCRVs* and their average of the concerned NMIs.

Table A.1. Measurements results as reported by the participants for the petal identified by the hydrometer 9342351 and the *KCRV* with its expanded uncertainty and the χ^2_{obs} value at each calibrated mark.

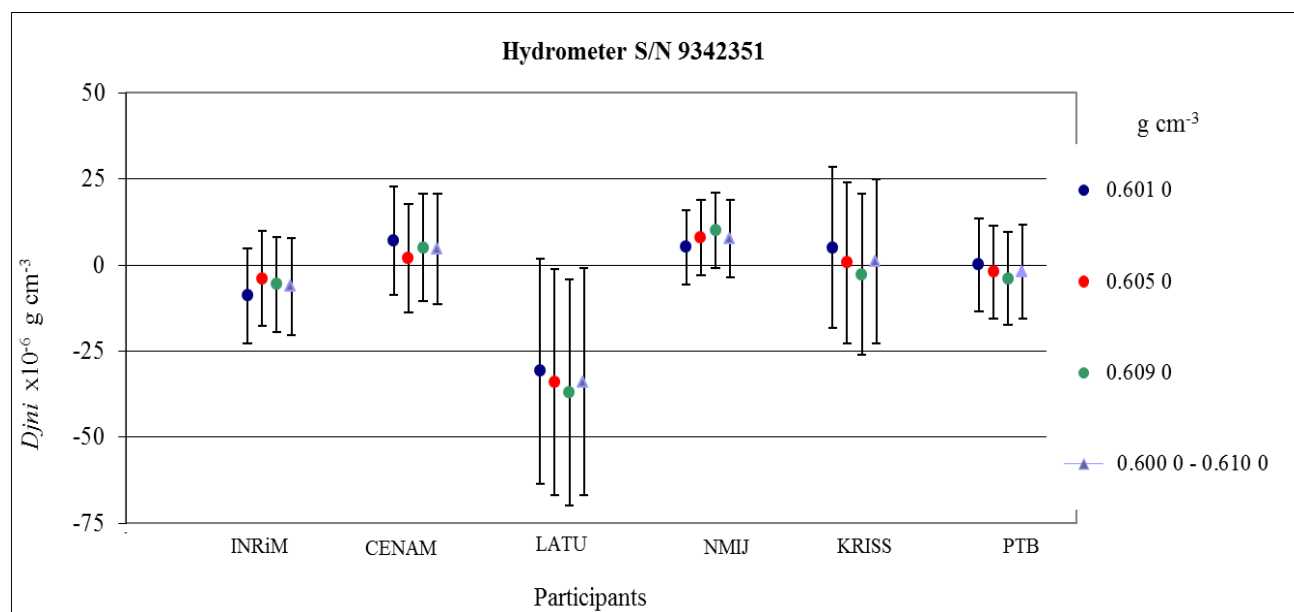
Hydrometer S/N 9342351		x10 ⁻⁶ / g cm ⁻³	INRiM	CENAM	LATU	NMIJ	KRISS	PTB		
Range (0.600 0 - 0.610 0) g/cm ³									<i>KCRV</i> _A (weighted mean)	<i>U</i> (<i>KCRV</i> _A)
0.601 0			-104	-88	-126	-90	-90	-95	-95	7
0.605 0			-102	-96	-132	-90	-97	-100	-98	7
0.609 0			-96	-85	-127	-80	-93	-94	-90	7
									Pr{χ ² (ν)>χ ² _{obs} }<0,05 ? χ ² (5)=11.07>χ ² _{obs} = (6.21; 6.03; 8.47) The consistency test doesn't fail: procedure A	
Combined std uncertainty of corrections <i>u</i> _c			8	9	17	6	12	8		
Expanded uncertainty of corrections <i>U</i> ₉₅ = <i>t</i> ₉₅ <i>u</i> _c			15	17	30	13	24	15		
Student t-factor <i>t</i> ₉₅			1.98	1.97	1.97	2.07	1.97	1.96		

Table A.2. Degree of equivalence to the *KCRV* and expanded uncertainty at each tested mark of each laboratory and average of the petal identified by the hydrometer 9342351.

NMI (n)	mark (i)/range (k)	0.601 0		0.605 0		0.609 0		0.600 0 - 0.610 0	
		<i>Dj_{ni}</i> x10 ⁻⁶	<i>U</i> (<i>Dj_{ni}</i>) x10 ⁻⁶	<i>Dj_{ni}</i> x10 ⁻⁶	<i>U</i> (<i>Dj_{ni}</i>) x10 ⁻⁶	<i>Dj_{ni}</i> x10 ⁻⁶	<i>U</i> (<i>Dj_{ni}</i>) x10 ⁻⁶	Δ_{jnk} x10 ⁻⁶	<i>U</i> (Δ_{jnk}) x10 ⁻⁶
INRiM	g cm ⁻³	-9	14	-4	14	-6	14	-6	14
CENAM		7	16	2	16	5	16	5	16
LATU		-31	33	-34	33	-37	33	-34	33
NMIJ		5	11	8	11	10	11	8	11
KRISS		5	23	1	23	-3	23	1	24
PTB		0	13	-2	13	-4	13	-2	14

Appendix A1 – Petal A

Figure A.1. Degree of equivalence with respect to the *KCRV* of each laboratory of the relevant petal identified by the hydrometer 9342351. The lengths of the bars show the expanded uncertainty of the degree of equivalence related to each calibrated mark and of their average.



Appendix A1 – Petal A

Hydrometer S/N 9340172

The *KCRVs* for the petal A, identified by the alcohol hydrometer 9340172, have been calculated by applying the “*weighted mean*” method according to the results of the consistency check $\Pr\{\chi^2(\nu) > \chi^2_{obs}\} < 0.05$ of the measurement results and standard uncertainties of the participants reported in **Table A.3**. The **Table** shows also each calculated *KCRVs* with the expanded uncertainties and the χ^2_{obs} values.

Table A.4 and **Figure A.2** present the degrees of equivalence to the *KCRV* and their average of the concerned NMIs.

Table A.3. Measurement results as reported by the participants for the petal identified by the alcohol hydrometer 9340172 and value of the *KCRV* with its expanded uncertainty and the χ^2_{obs} value at each calibrated mark.

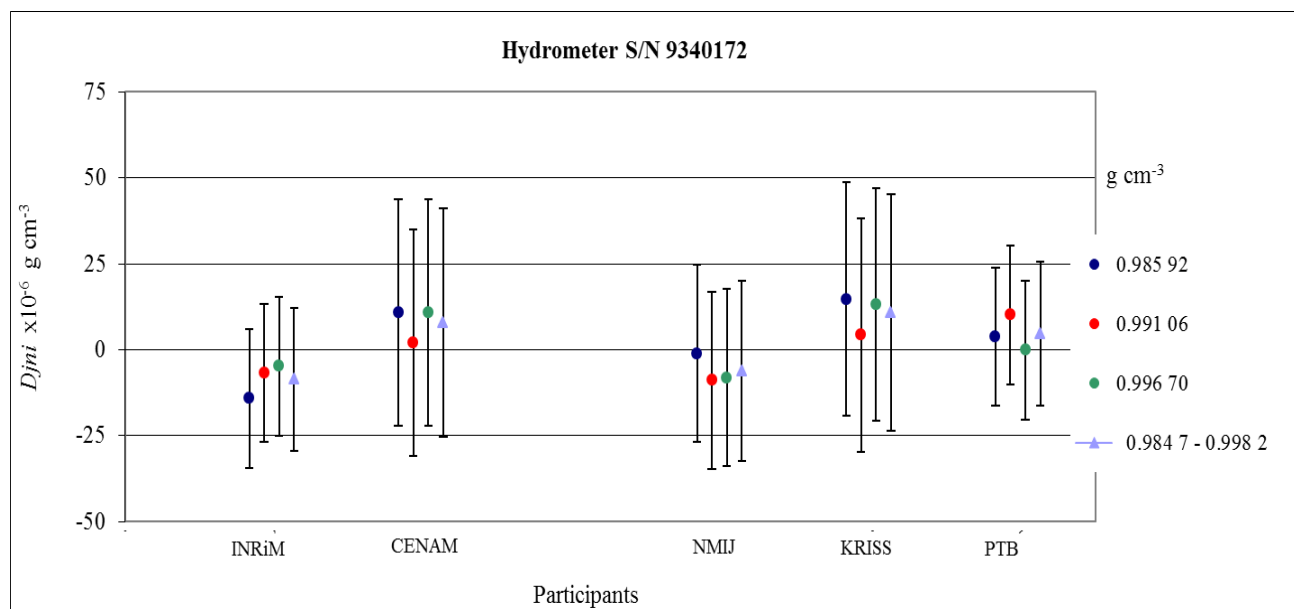
Alcoholometer S/N 9340172		x10 ⁻⁶ / g cm ⁻³	INRiM	CENAM		NMIJ	KRISS	PTB			
Range (0 - 10) % vol	g/cm ³								KCRV _A (weighted mean)	U (KCRV _A)	
1,0	0.996 70		-27	-11		-30	-9	-22	-22	13	
5,0	0.991 06		-48	-39		-50	-37	-31	-41	13	
9,0	0.985 92		-43	-18		-30	-14	-25	-29	13	
										Pr{χ ² (ν)>χ ² _{obs} } <0,05 ? χ ² (4)=9.49>χ ² _{obs} = (1.39; 1.51; 2.58) The consistency test doesn't fail: procedure A	
Combined std uncertainty of corrections u _c			12	18		14	18	12			
Expanded uncertainty of corrections U ₉₅ =t ₉₅ u _c			24	35		31	35	24			
Student t-factor t ₉₅			1.98	1.97		2.14	1.96	2.00			

Table A.4. Degree of equivalence to the *KCRV* and expanded uncertainty at each tested mark of each laboratory and average of the petal identified by the hydrometer 9340172.

NMI (n)	mark (i)/range (k)	0.985 92		0.991 06		0.996 70		0.984 7 - 0.998 2	
		<i>Dj_{ni}</i> $\times 10^{-6}$	<i>U</i> (<i>Dj_{ni}</i>) $\times 10^{-6}$	<i>Dj_{ni}</i> $\times 10^{-6}$	<i>U</i> (<i>Dj_{ni}</i>) $\times 10^{-6}$	<i>Dj_{ni}</i> $\times 10^{-6}$	<i>U</i> (<i>Dj_{ni}</i>) $\times 10^{-6}$	Δ_{jnk} $\times 10^{-6}$	<i>U</i> (Δ_{jnk}) $\times 10^{-6}$
INRiM	g cm^{-3}	-14	20	-7	20	-5	20	-9	21
CENAM		11	33	2	33	11	33	8	33
NMIJ		-1	26	-9	26	-8	26	-6	26
KRISS		15	34	4	34	13	34	11	34
PTB		4	20	10	20	0	20	5	21

Appendix A1 – Petal A

Figure A.2. Degree of equivalence to the *KCRV* of each laboratory of the petal identified by the alcohol hydrometer 9340172. The lengths of the bars show the uncertainty of the degree of equivalence related to each calibrated mark and of their average.



Appendix A1 – Petal A

Hydrometer S/N 9343460

The *KCRVs* for the petal A, identified by the hydrometer 9343460, have been calculated by applying the “**weighted mean**” method according to the results of the consistency check $\Pr\{\chi^2(\nu) > \chi^2_{obs}\} < 0.05$ of the measurement results and standard uncertainties of the participants reported in

Table A.5. The **Table** shows also each calculated *KCRVs* with the expanded uncertainties and the χ^2_{obs} values.

Table A.6 and **Figure A.3** present the degrees of equivalence to the *KCRVs* and their average of the concerned NMIs.

Table A.5. Measurements results as reported by the participants for the petal identified by the hydrometer 9343460 and the *KCRV* with its expanded uncertainty and the χ^2_{obs} value at each calibrated mark.

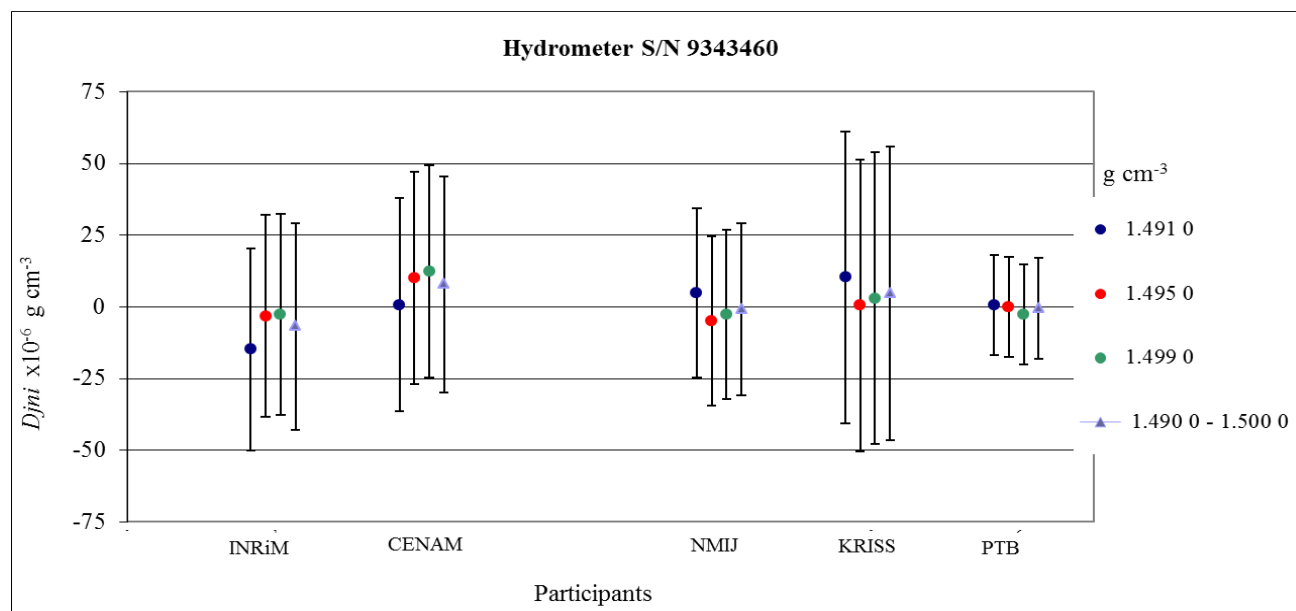
Hydrometer: 9343460		x10 ⁻⁶ / g cm ⁻³	INRiM	CENAM		NMIJ	KRISS	PTB			
Range(1.490 0 - 1.500 0) g/cm ³									KCRV _A (weighted mean)	U(KCRV _A)	
1.491 0			60	76		80	86	76	75	15	
1.495 0			82	95		80	86	85	85	15	
1.499 0			70	85		70	76	70	73	15	
										Pr{χ ² (v)>χ ² _{obs} } <0,05 ? χ ² (4)=9.49>χ ² _{obs} =(0.84; 0.37; 0.49) The consistency test doesn't fail: procedure A	
Combined std uncertainty of corrections u _c			17	20		17	27	11			
Expanded uncertainty of corrections U ₉₅ =t ₉₅ u _c			33	39		35	52	23			
Student t-factor t ₉₅			1.98	1.98		2.09	1.96	1.97			

Table A.6. Degree of equivalence to the *KCRV* and expanded uncertainty at each tested mark of each laboratory and average of the petal identified by the hydrometer 9343460.

NMI (n)	mark (i)/range (k)	1.491 0		1.495 0		1.499 0		1.490 0 - 1.500 0	
		<i>Dj_{ni}</i> x10 ⁻⁶	<i>U(Dj_{ni})</i> x10 ⁻⁶	<i>Dj_{ni}</i> x10 ⁻⁶	<i>U(Dj_{ni})</i> x10 ⁻⁶	<i>Dj_{ni}</i> x10 ⁻⁶	<i>U(Dj_{ni})</i> x10 ⁻⁶	Δ_{jnk} x10 ⁻⁶	<i>U(Δ_{jnk})</i> x10 ⁻⁶
INRiM	g cm ⁻³	-15	35	-3	35	-3	35	-7	36
CENAM		1	37	10	37	12	37	8	38
NMIJ		5	29	-5	29	-3	29	-1	30
KRISS		10	51	1	51	3	51	5	51
PTB		1	17	0	17	-3	17	-1	18

Appendix A1 – Petal A

Figure A.3. Degree of equivalence to the *KCRV* of each laboratory of the petal identified by the hydrometer 9343460. The lengths of the bars show the expanded uncertainty of the degree of equivalence related to each calibrated mark and of their average.



Appendix A1 – Petal A

Hydrometer S/N 9346684

The *KCRVs* for the petal A, identified by the hydrometer 9346684, have been calculated by applying the “**weighted mean**” method according to the results of the consistency check $\Pr\{\chi^2(\nu) > \chi^2_{obs}\} < 0.05$ of the measurement results and standard uncertainties of the participants reported in

Table A.7. The **Table** shows also each calculated *KCRVs* with the expanded uncertainties and the χ^2_{obs} values.

Table A.8 and **Figure A.4** present the degrees of equivalence with respect to the *KCRV* and their average of the concerned NMIs.

Table A.7. Measurements results as reported by the participants for the petal identified by the hydrometer 9346684 and the *KCRV* with its expanded uncertainty and the χ^2_{obs} value at each calibrated mark.

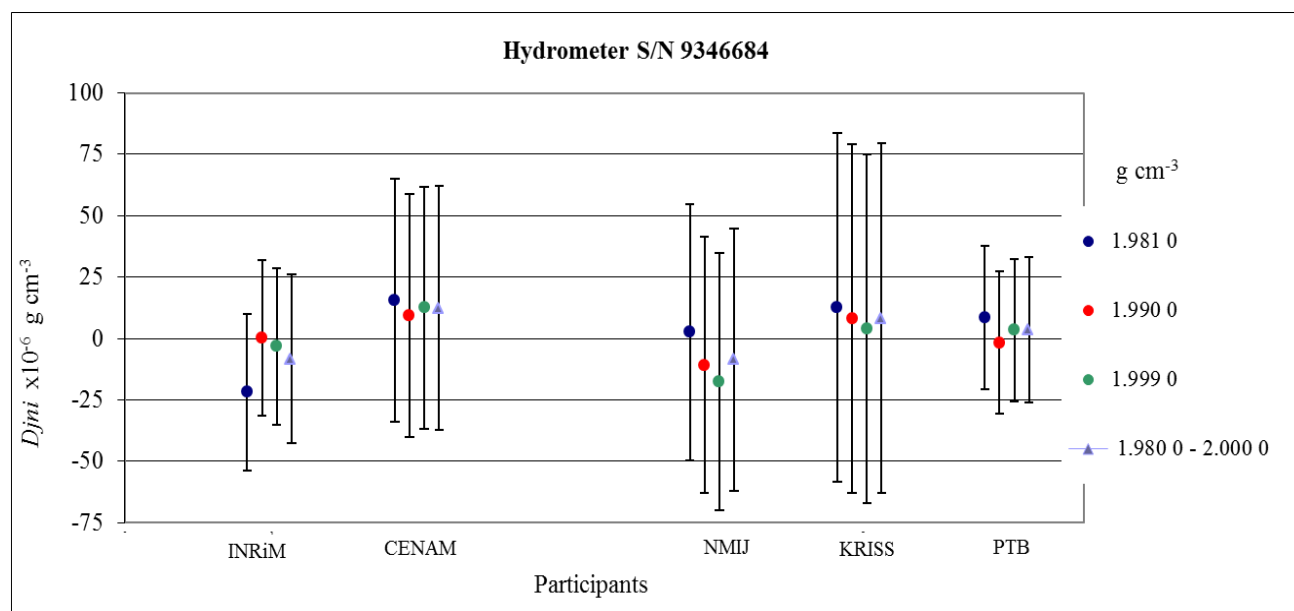
Hydrometer: 9346684 Range (1.980 0 - 2.000 0) g/cm ³		INRiM	CENAM		NMIJ	KRISS	PTB	<i>KCRV_A</i> (weighted mean)	<i>U</i> (<i>KCRV_A</i>)
1.981 0	$\times 10^{-6} / \text{g cm}^{-3}$	86	123		110	120	116	108	21
1.990 0		91	100		80	99	89	91	21
1.999 0		144	160		130	151	151	148	21
Combined std uncertainty of corrections u_c		19	27		28	37	18	$\Pr\{\chi^2(\nu) > \chi^2_{obs}\} < 0,05$? $\chi^2(4) = 9.49 > \chi^2_{obs} = (2.02; 0.32; 0.68)$ The consistency test doesn't fail: procedure A	
Expanded uncertainty of corrections $U_{95} = t_{95} u_c$		38	53		59	73	35		
Student t-factor t_{95}		1.98	1.98		2.09	1.96	1.97		

Table A.8. Degree of equivalence to the *KCRV* and expanded uncertainty at each tested mark of each laboratory and average of the petal identified by the hydrometer 9346684.

NMI (n)	mark (i)/range (k)	1.981 0		1.990 0		1.999 0		1.980 0 - 2.000 0	
		$D_{jni} \times 10^{-6}$	$U(D_{jni}) \times 10^{-6}$	$D_{jni} \times 10^{-6}$	$U(D_{jni}) \times 10^{-6}$	$D_{jni} \times 10^{-6}$	$U(D_{jni}) \times 10^{-6}$	$\Delta_{jnk} \times 10^{-6}$	$U(\Delta_{jnk}) \times 10^{-6}$
INRiM	g cm^{-3}	-22	32	0	32	-3	32	-8	34
CENAM		15	49	9	49	12	49	12	50
NMIJ		2	52	-11	52	-18	52	-9	54
KRISS		13	71	8	71	4	71	8	71
PTB		8	29	-2	29	3	29	3	30

Appendix A1 – Petal A

Figure A.4. Degree of equivalence to the *KCRV* of each laboratory of the petal identified by the hydrometer 9346684. The lengths of the bars show the expanded uncertainty of the degree of equivalence related at each calibrated mark and of their average.



Appendix A1 – Petal B

Hydrometer S/N 9342348

The *KCRVs* for the petal B, identified by the hydrometer 9342348, have been calculated at the density value of 0.601 g cm^{-3} by applying the “*median*” method according to the results of the consistency check $\Pr\{\chi^2(\nu) > \chi^2_{obs}\} < 0.05$ of the measurement results and standard uncertainties of the participants reported in **Table B.1**. The source of inconsistency have been identified in the Institutes data of NIST and MKEH. For the remaining two density values the consistency test didn’t fail, the *KCRVs* were calculated by the “*weighted mean*” method. **Table B.1** also shows each calculated *KCRVs* with the lower (U_l) and upper (U_r) limits of the interval containing the median when the procedure B was applied, the expanded uncertainties and the χ^2_{obs} values.

Table B.2 and **Figure B.1** present the degrees of equivalence to the *KCRV* and their average of the concerned NMIs.

Table B.1. Measurements results as reported by the participants for the petal identified by the hydrometer 9342348 and the *KCRV* with the lower and upper limits of the interval containing the median when the procedure B was applied, the expanded uncertainty and the χ^2_{obs} value at each tested mark.

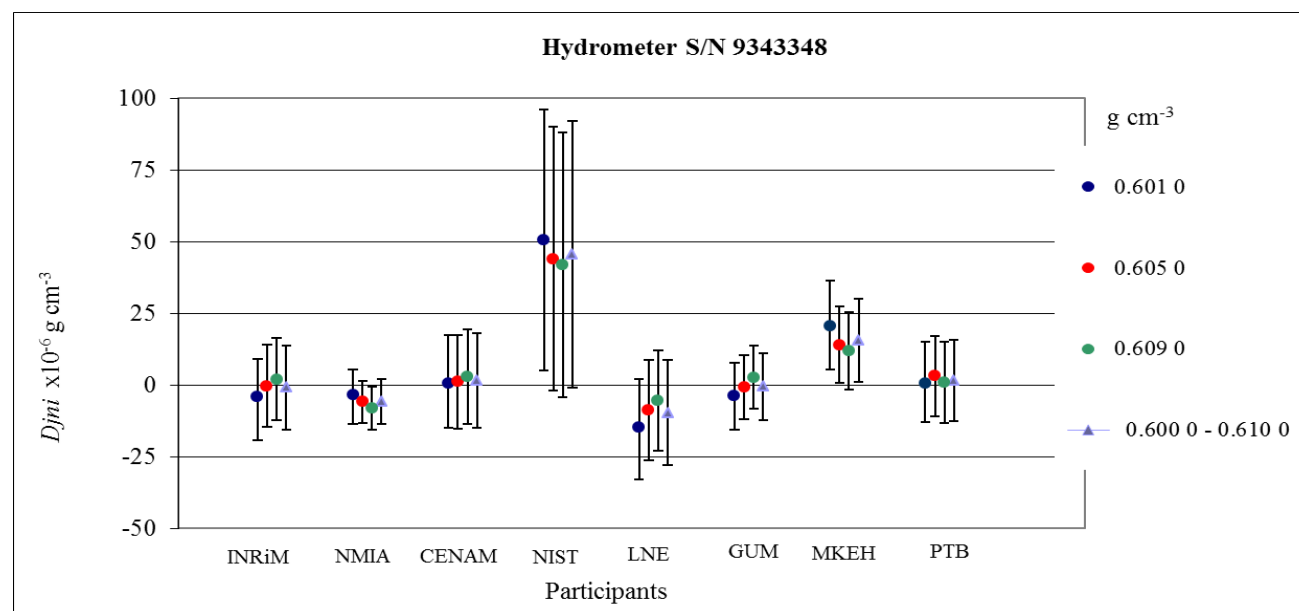
Hydrometer S/N 9342348 Range (0.600 0 - 0.610 0) g/cm ³		INRiM	NMIA	CENAM	NIST	LNE	GUM	MKEH	PTB					
										<i>KCRV_B</i> (weighted mean)	<i>KCRV_B</i> (median)	$U_l(KCRV_B)$	$U_r(KCRV_B)$	$U(KCRV_B)$
0.601 0	$\times 10^{-6} / \text{g cm}^{-3}$	-75	-74	-70	-20	-86	-74	-50	-70		-71	-77	-64	7
0.605 0		-64	-70	-63	-20	-73	-65	-50	-61	-64				5
0.609 0		-60	-70	-59	-20	-68	-59	-50	-61	-62				5
Combined std uncertainty of corrections u_c		8	4	9	23	9	6	7,2	7,5	$\Pr\{\chi^2(\nu) > \chi^2_{obs}\} < 0,05$? $\chi^2(7) = 14.07 < \chi^2_{obs} = 17.04$ The consistency test fails: procedure B $\chi^2(7) = 14.07 > \chi^2_{obs} = (10,31; 10,04)$ The consistency test doesn't fail: procedure A				
Expanded uncertainty of corrections $U_{95} = t_{95} u_c$		15	9	17	46	20	12	14	15					
Student t-factor t_{95}		1,98	1,97	1,97	1,98	2,14	1,98	1,97	1,96					

Table B.2. Degree of equivalence to the *KCRV* and coverage interval at each tested mark of each laboratory and average of the petal identified by the hydrometer 9342348.

NMI (n)	mark (i)/range (k)	0.601 0				0.605 0		0.609 0		0.600 0 - 0.610 0	
		$D_{jni} \times 10^{-6}$	$U_l(D_{jni}) \times 10^{-6}$	$U_r(D_{jni}) \times 10^{-6}$	$U(D_i) \times 10^{-6}(-6)$	$D_{jni} \times 10^{-6}$	$U(D_{jni}) \times 10^{-6}$	$D_{jni} \times 10^{-6}$	$U(D_{jni}) \times 10^{-6}$	$\Delta_{jnk} \times 10^{-6}$	$U(\Delta_{jnk}) \times 10^{-6}$
INRiM	g cm^{-3}	-4	15	13	14	0	14	2	14	-1	15
NMIA		-3	10	9	7	-6	7	-8	7	-6	8
CENAM		1	16	17	16	1	16	3	16	2	16
NIST		51	46	45	46	44	46	42	46	46	46
LNE		-15	18	17	18	-9	18	-6	18	-10	18
GUM		-4	12	11	11	-1	11	3	11	-1	12
MKEH		21	15	16	13	14	13	12	13	16	14
PTB		1	14	14	14	3	14	1	14	2	14

Appendix A1 – Petal B

Figure B.1. Degree of equivalence to the *KCRV* of each laboratory of the petal identified by the hydrometer 9342348. The lengths of the bars show the uncertainty or coverage interval of the degree of equivalence related to each calibrated mark and of their average.



Appendix A1 – Petal B

Hydrometer S/N 9340171

The *KCRVs* for the petal B, identified by the alcohol hydrometer 9340171, have been calculated at the density value of $0.985\,92\text{ g cm}^{-3}$ and 0.99106 g cm^{-3} by applying the “*median*” method according to the results of the consistency check $\Pr\{\chi^2(\nu) > \chi^2_{obs}\} < 0.05$ of the measurement results and standard uncertainties of the participants reported in **Table B.3**. The source of inconsistency have been identified in the Institute data of LNE. For the remaining density value the consistency test didn’t fail, the *KCRVs* were calculated by the “*weighted mean*” method. **Table B.3** also shows each calculated *KCRVs* with the lower (U_l) and upper (U_r) limits of the interval containing the median when the procedure B was applied, the expanded uncertainties and the χ^2_{obs} values.

Table B.4 and **Figure B.2** present the degrees of equivalence respect to the *KCRV* and their average of the concerned NMIs.

Table B.3. Measurements results as reported by the participants for the petal identified by the hydrometer 9340171 and the *KCRV* with the lower and upper limits of the interval containing the median when the procedure B was applied, the expanded uncertainty and the χ^2_{obs} value at each tested mark.

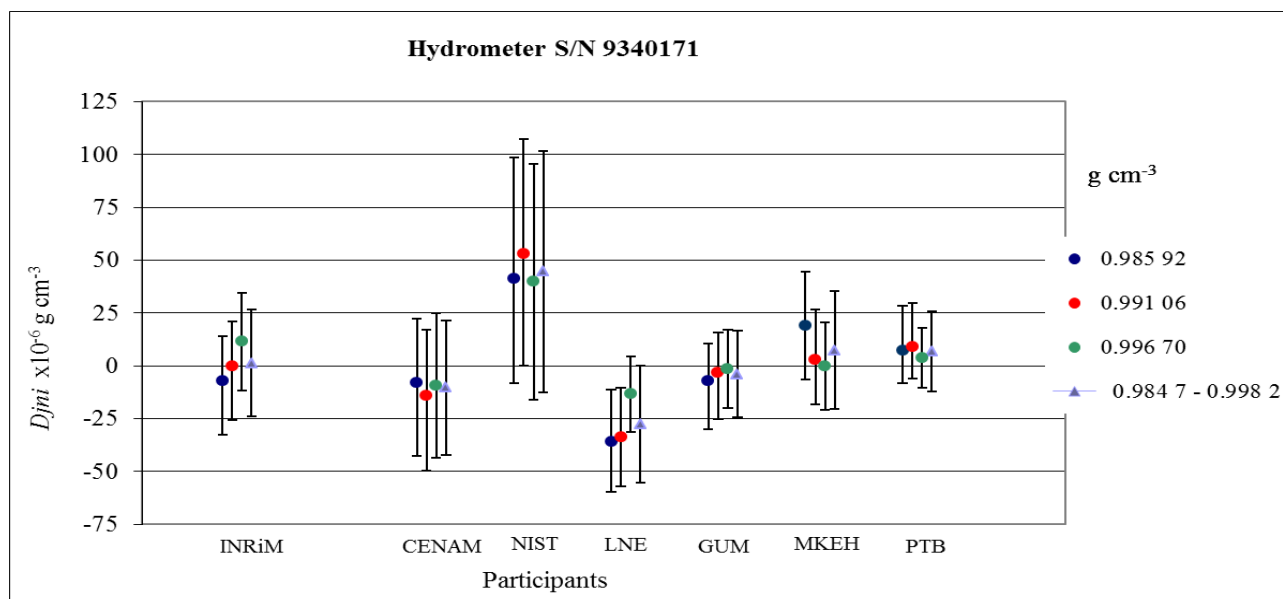
Alcoholometer S/N 9340171 Range (0 - 10) % vol					INRiM		CENAM	NIST	LNE	GUM	MKEH	PTB					
g/cm3		x10 ⁻⁶ / g cm ⁻³											KCRV _B (weighted mean)	KCRV _B (median)	U _l (KCRV _B)	U _r (KCRV _B)	U (KCRV _B)
1,0	0.996 70		-18		-39	10	-43	-31	-30	-26	-30						9
5,0	0.991 06		-33		-47	20	-67	-36	-30	-24			-33	-46	-20		12
9,0	0.985 92		-28		-29	20	-57	-28	-2	-14			-21	-36	-7		14
Combined std uncertainty of corrections u _c			12		18	28	10	10	11	8	Pr{χ ² (ν)>χ ² _{obs} } <0,05 ? χ ² (6)=12.59>χ ² _{obs} = 5.17 The consistency test doesn't fail: procedure A χ ² (6)=12.59<χ ² _{obs} =(19.02; 16.17) The consistency test fails: procedure B						
Expanded uncertainty of corrections U ₉₅ = t ₉₅ u _c			24		35	56	20	20	22	16							
Student t-factor t ₉₅			1.98		1.97	1.97	2.02	1.97	1.96	1.97							

Table B.4. Degree of equivalence to the *KCRV* and coverage interval at each tested mark of each laboratory and average of the petal identified by the hydrometer 9340171.

NMI (n)	mark (i)/range (k)	0.985 92				0.991 06				0.996 70		0.984 7 - 0.998 2	
		$D_{jni} \times 10^{-6}$	$U_l(D_{jni}) \times 10^{-6}$	$U_r(D_{jni}) \times 10^{-6}$	$U(D_{jni}) \times 10^{-6}$	$D_{jni} \times 10^{-6}$	$U_l(D_{jni}) \times 10^{-6}$	$U_r(D_{jni}) \times 10^{-6}$	$U(D_{jni}) \times 10^{-6}$	$D_{jni} \times 10^{-6}$	$U(D_{jni}) \times 10^{-6}$	$\Delta_{jnk} \times 10^{-6}$	$U(\Delta_{jnk}) \times 10^{-6}$
INRiM	g cm^{-3}	-7	25	21	23	0	24	24	22	11	23	1	25
CENAM		-8	35	30	32	-14	36	31	34	-9	34	-10	32
NIST		41	49	57	57	53	53	54	57	40	56	45	57
LNE		-36	24	25	25	-34	23	23	24	-13	18	-28	28
GUM		-7	23	18	20	-3	22	19	19	-1	18	-4	21
MKEH		19	19	25	26	3	21	24	21	0	21	7	28
PTB		7	15	21	19	9	15	21	19	4	14	7	19

Appendix A1 – Petal B

Figure B.2. Degree of equivalence with respect to the *KCRV* of each laboratory of the petal identified by the hydrometer 9343171. The lengths of the bars show the uncertainty or coverage interval of the degree of equivalence related to each calibrated mark and of their average.



Appendix A1 – Petal B

Hydrometer S/N 9343462

The *KCRVs* for the petal B, identified by the hydrometer 93443462, have been calculated by applying the “**weighted mean**” method according to the results of the consistency check $\Pr\{\chi^2(\nu) > \chi^2_{obs}\} < 0.05$ of the measurement results and standard uncertainties of the participants reported in

Table B.5. The **Table** shows also each calculated *KCRVs* with the expanded uncertainties and the χ^2_{obs} values.

Table B.6 and **Figure B.3** present the degrees of equivalence respect to the *KCRV* and their average of the concerned NMIs.

Table B.5. Measurement results as reported by the participants for the petal identified by the hydrometer 9343462 and the *KCRV* with its expanded uncertainty and the χ^2_{obs} value at each calibrated mark.

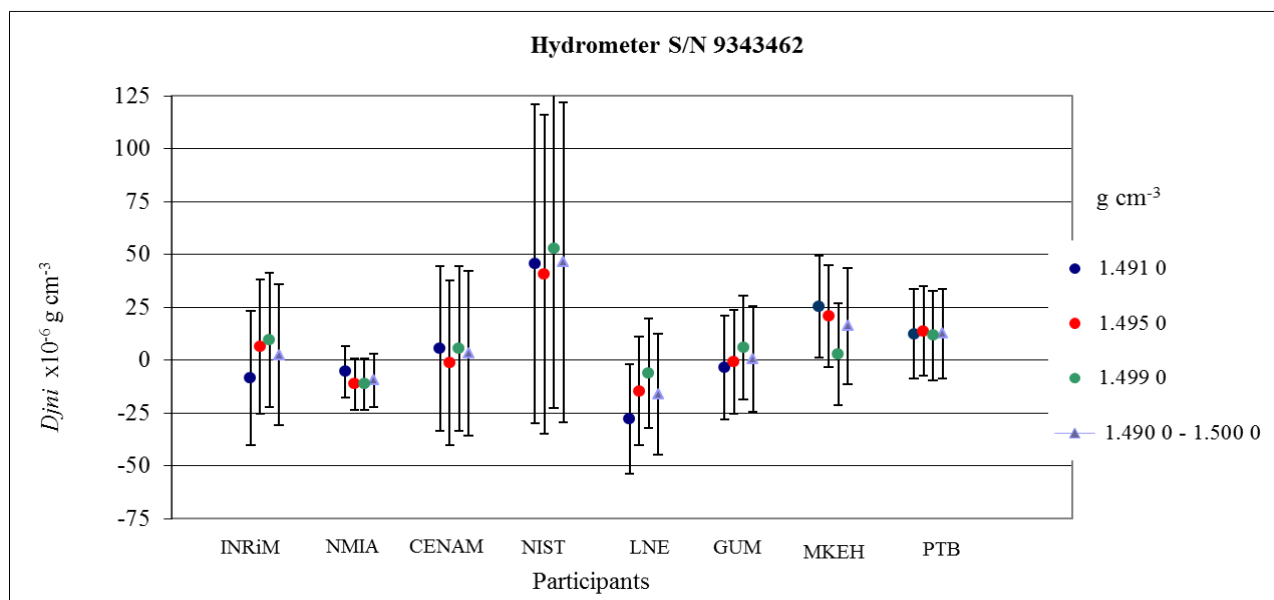
Hydrometer: 9343462 Range: (1.490 0 - 1.500 0) g/cm ³		INRIM	NMIA	CENAM	NIST	LNE	GUM	MKEH	PTB	<i>KCRV_B</i> (weighted mean)	<i>U</i> (<i>KCRV_B</i>)
1.491 0	$\times 10^{-6} / \text{g cm}^{-3}$	-4	-1	10	50	-23	1	30	17	4	9
1.495 0		26	8	18	60	5	18	40	33	19	9
1.499 0		-3	-24	-7	40	-19	-7	-10	-1	-13	9
Combined std uncertainty of corrections u_c		17	8	20	38	14	13	13	11	$\Pr\{\chi^2(\nu) > \chi^2_{obs}\} < 0,05$? $\chi^2(7) = 14,07 > \chi^2_{obs} = (11.63; 8.67; 6,07)$ The consistency test doesn't fail: procedure A	
Expanded uncertainty of corrections $U_{95} = t_{95} u_c$		33	15	39	75	28	26	25	23		
Student t-factor t_{95}		1,98	1,98	1,98	1,98	2,04	1,99	1,96	1,97		

Table B.6. Degree of equivalence respect to the *KCRV* and expanded uncertainty at each calibrated mark of each laboratory and average of the petal identified by the hydrometer 9343462.

NMI (n)	mark (i)/range (k)	1.491 0		1.495 0		1.499 0		1.490 0 - 1.500 0	
		<i>Dj_{ni}</i> $\times 10^{-6}$	<i>U</i> (<i>Dj_{ni}</i>) $\times 10^{-6}$	<i>Dj_{ni}</i> $\times 10^{-6}$	<i>U</i> (<i>Dj_{ni}</i>) $\times 10^{-6}$	<i>Dj_{ni}</i> $\times 10^{-6}$	<i>U</i> (<i>Dj_{ni}</i>) $\times 10^{-6}$	Δ_{jnk} $\times 10^{-6}$	<i>U</i> (Δ_{jnk}) $\times 10^{-6}$
INRiM	g cm ⁻³	-8	32	6	32	10	32	2	33
NMIA		-5	12	-11	12	-11	12	-9	13
CENAM		6	39	-1	39	6	39	3	39
NIST		46	75	41	75	53	75	46	76
LNE		-28	26	-14	26	-6	26	-16	29
GUM		-4	24	-1	24	6	24	1	25
MKEH		26	24	21	24	3	24	16	27
PTB		13	21	14	21	12	21	13	21

Appendix A1 – Petal B

Figure B.3. Degree of equivalence with respect to the *KCRV* of each laboratory of the petal identified by the hydrometer 9343462. The lengths of the bars show the expanded uncertainty of the degree of equivalence related to each calibrated mark and of their average.



Appendix A1 – Petal B

Hydrometer S/N 9346688

The *KCRVs* for the petal B, identified by the hydrometer 9346688, have been calculated at the density value of 1.981 g cm^{-3} by applying the “*median*” method according to the results of the consistency check $\Pr\{\chi^2(\nu) > \chi^2_{\text{obs}}\} < 0.05$ of the measurement results and standard uncertainties of the participants reported in **Table B.7**. The source of inconsistency have been identified in the Institutes data of MKEH. For the remaining two density values the consistency test didn’t fail, the *KCRVs* were calculated by the “*weighted mean*” method. **Table B.7** shows also each calculated *KCRVs* with the lower (U_l) and upper (U_r) limits of the interval containing the median when the procedure B was applied, the expanded uncertainties and the χ^2_{obs} values.

Table B.8 and **Figure B.4** present the degrees of equivalence with respect to the *KCRVs* and their average of the concerned NMIs.

Table B.7. Measurement results as reported by the participants for the petal identified by the hydrometer 9346688 and the *KCRV* with the lower and upper limits of the interval containing the median when the procedure B was applied, the expanded uncertainty and the χ^2_{obs} value at each tested mark.

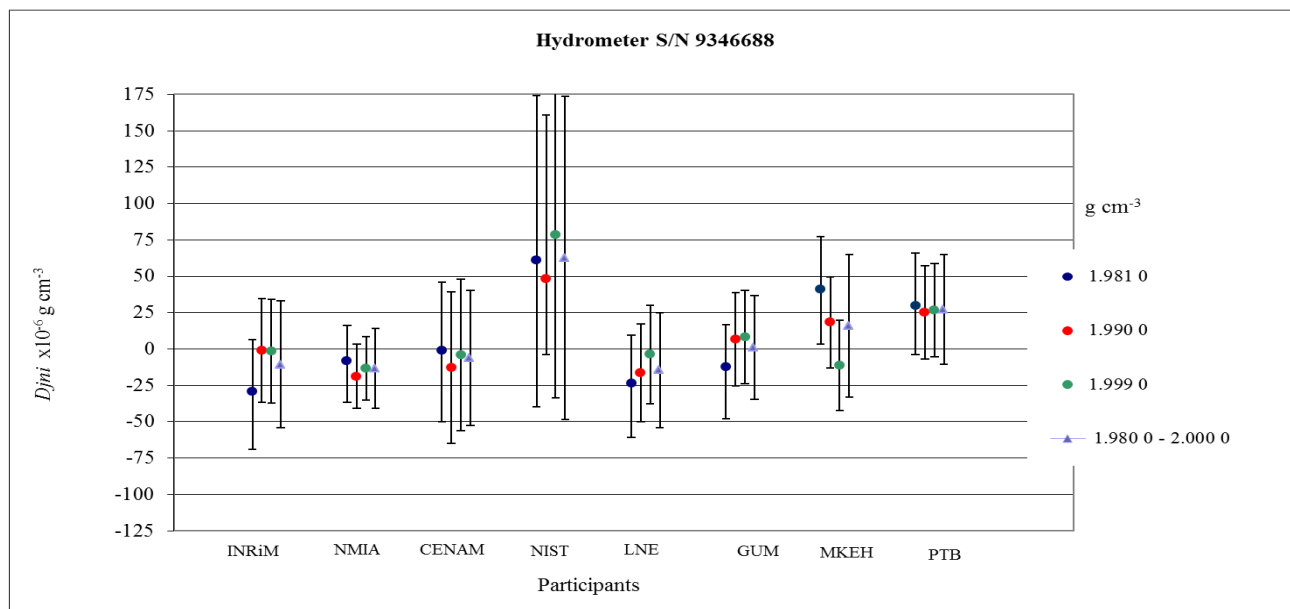
Hydrometer: 9346688 Range (1.980 0 - 2.000 0) g/cm ³		x10 ⁻⁶ / g cm ³	INRiM	NMIA	CENAM	NIST	LNE	GUM	MKEH	PTB	KCRV _B (weighted mean)	KCRV _B (median)	U _l (KCRV _B)	U _r (KCRV _B)	U(CCM KCRV _B)
1.981 0	10		31	38	100	16	27	80	69		39	20	59	20	
1.990 0	1		-17	-11	50	-15	9	20	27	2				13	
1.999 0	50		38	47	130	47	60	40	78	51				13	
											Pr{χ ² (ν)>χ ² _{obs} } <0,05 ?				
Combined std uncertainty of corrections u _c			19	13	27	57	18	17	17	17	χ ² (7)=14.07<χ ² _{obs} =15.01 The consistency test fails: procedure B				
Expanded uncertainty of corrections U ₉₅ =t ₉₅ u _c			38	25	53	112	36	34	33	34	χ ² (7)=14.07>χ ² _{obs} =(7.41; 6.18) The consistency test doesn't fail: procedure A				
Student t-factor t ₉₅			1.98	1.97	1.98	1.97	1.99	1.99	1.97	1.97					

Table B.8. Degree of equivalence with respect to the *KCRV* and the uncertainty at each calibrated mark of each laboratory and average of the petal identified by the hydrometer 9346688.

NMI (n)	mark (i)/range (k)	1.981 0				1.990 0		1.999 0		1.980 0 - 2.000 0	
		Djni x10 ⁻⁶	U _l (Djni) x10 ⁻⁶	U _r (Djni) x10 ⁻⁶	U(Djni) x10 ⁻⁶	Djni x10 ⁻⁶	U(Djni) x10 ⁻⁶	Djni x10 ⁻⁶	U(Djni) x10 ⁻⁶	Δ_{jnk} x10 ⁻⁶	U(Δ_{jnk}) x10 ⁻⁶
INRiM	g cm ⁻³	-29	40	36	40	-1	36	-2	36	-11	44
NMIA		-8	28	24	27	-19	22	-13	22	-13	27
CENAM		-1	49	47	46	-13	52	-4	52	-6	46
NIST		61	101	113	110	48	113	79	113	63	111
LNE		-24	37	33	38	-16	34	-4	34	-15	39
GUM		-12	36	29	33	7	32	8	32	1	36
MKEH		41	37	37	39	18	31	-11	31	16	49
PTB		30	34	36	38	25	32	27	32	27	38

Appendix A1 – Petal B

Figure B.4. Degree of equivalence with respect to the *KCRV* of each laboratory of the petal identified by the hydrometer 9346688. The lengths of the bars show the uncertainty or coverage interval of the degree of equivalence related to each calibrated mark and of their average.



Appendix A2

This appendix explains how to evaluate the degrees of equivalence and their uncertainties between the two institutes, p and q , who participated in the two different loops, A and B, respectively. Assuming that the measurement results of all participants are independent with each other, the degrees of equivalence of the two institutes from $KCRV_A$ and $KCRV_B$ and their uncertainties are expressed as follows [8]:

$$D_p = x_p - KCRV_A, \quad (A1)$$

$$D_q = x_q - KCRV_B, \quad (A2)$$

$$u^2(d_p) = u^2(x_p) - u^2(KCRV_A) = u^2(x_p) - \frac{1}{1/u^2(x_{A1}) + \dots + 1/u^2(x_{AM})}, \quad (A3)$$

$$u^2(d_q) = u^2(x_q) - u^2(KCRV_B) = u^2(x_q) - \frac{1}{1/u^2(x_{B1}) + \dots + 1/u^2(x_{BN})}, \text{ and} \quad (A4)$$

$$\begin{aligned} d_{pq} &= D_p - D_q = (x_p - KCRV_A) - (x_q - KCRV_B) \\ &= x_p - \frac{x_{A1}/u^2(x_{A1}) + \dots + x_{AM}/u^2(x_{AM})}{1/u^2(x_{A1}) + \dots + 1/u^2(x_{AM})} - x_q + \frac{x_{B1}/u^2(x_{B1}) + \dots + x_{BN}/u^2(x_{BN})}{1/u^2(x_{B1}) + \dots + 1/u^2(x_{BN})}, \end{aligned} \quad (A5)$$

where the two loops A and B contain M and N participants, respectively.

In this key comparison, the three institutes, $k = 1$ to $k = 3$, participated in both loops as link laboratories. In order to estimate the uncertainty of $(D_p - D_q)$ in this condition, the coefficient of partial derivative of equation (A5) for each variable is expressed as follows:

$$x_p: 1 - \frac{1/u^2(x_p)}{1/u^2(x_{A1}) + \dots + 1/u^2(x_{AM})}, \quad (A6)$$

$$x_q: -1 + \frac{1/u^2(x_q)}{1/u^2(x_{B1}) + \dots + 1/u^2(x_{BN})}, \quad (A7)$$

$$x_i \text{ for other participants in loop A: } -\frac{1/u^2(x_{Ai})}{1/u^2(x_{A1}) + \dots + 1/u^2(x_{AM})}, \text{ and} \quad (A8)$$

$$x_j \text{ for other participants in loop B: } \frac{1/u^2(x_{Bj})}{1/u^2(x_{B1}) + \dots + 1/u^2(x_{BN})}. \quad (A9)$$

Since the link laboratory k participated in the both loops, the coefficient for x_k is given as

$$x_k: -\frac{1/u^2(x_k)}{1/u^2(x_{A1}) + \dots + 1/u^2(x_{AM})} + \frac{1/u^2(x_k)}{1/u^2(x_{B1}) + \dots + 1/u^2(x_{BN})}. \quad (A10)$$

The variance of $D_p - D_q$ is therefore given from equation (A5) as

$$\begin{aligned}
u^2(D_p - D_q) &= \frac{1/u^2(x_{A1})}{(1/u^2(x_{A1}) + \dots + 1/u^2(x_{AM}))^2} + \dots + \left(1 - \frac{1/u^2(x_p)}{1/u^2(x_{A1}) + \dots + 1/u^2(x_{AM})}\right)^2 u^2(x_p) + \dots \\
&+ \frac{1/u^2(x_{AM})}{(1/u^2(x_{A1}) + \dots + 1/u^2(x_{AM}))^2} \\
&+ \frac{1/u^2(x_{B1})}{(1/u^2(x_{B1}) + \dots + 1/u^2(x_{BN}))^2} + \dots + \left(1 - \frac{1/u^2(x_q)}{1/u^2(x_{B1}) + \dots + 1/u^2(x_{BN})}\right)^2 u^2(x_q) + \dots \\
&+ \frac{1/u^2(x_{BN})}{(1/u^2(x_{B1}) + \dots + 1/u^2(x_{BN}))^2} \\
&- 2 \frac{\sum_{k=1}^3 1/u^2(x_k)}{(1/u^2(x_{A1}) + \dots + 1/u^2(x_{AM}))(1/u^2(x_{B1}) + \dots + 1/u^2(x_{BN}))} \\
&= \frac{1/u^2(x_{A1})}{(1/u^2(x_{A1}) + \dots + 1/u^2(x_{AM}))^2} + \dots + \frac{1/u^2(x_p)}{(1/u^2(x_{A1}) + \dots + 1/u^2(x_{AM}))^2} + \dots \\
&+ \frac{1/u^2(x_{AM})}{(1/u^2(x_{A1}) + \dots + 1/u^2(x_{AM}))^2} + u^2(x_p) - 2 \frac{1}{1/u^2(x_{A1}) + \dots + 1/u^2(x_{AM})} \\
&+ \frac{1/u^2(x_{B1})}{(1/u^2(x_{B1}) + \dots + 1/u^2(x_{BN}))^2} + \dots + \frac{1/u^2(x_q)}{(1/u^2(x_{B1}) + \dots + 1/u^2(x_{BN}))^2} + \dots \\
&+ \frac{1/u^2(x_{BN})}{(1/u^2(x_{B1}) + \dots + 1/u^2(x_{BN}))^2} + u^2(x_q) - 2 \frac{1}{1/u^2(x_{B1}) + \dots + 1/u^2(x_{BN})} \\
&- 2 \frac{\sum_{k=1}^3 1/u^2(x_k)}{(1/u^2(x_{A1}) + \dots + 1/u^2(x_{AM}))(1/u^2(x_{B1}) + \dots + 1/u^2(x_{BN}))} \\
&= u^2(x_p) - \frac{1}{1/u^2(x_{A1}) + \dots + 1/u^2(x_{AM})} + u^2(x_q) - \frac{1}{1/u^2(x_{B1}) + \dots + 1/u^2(x_{BN})} \\
&- 2 \frac{\sum_{k=1}^3 1/u^2(x_k)}{(1/u^2(x_{A1}) + \dots + 1/u^2(x_{AM}))(1/u^2(x_{B1}) + \dots + 1/u^2(x_{BN}))} \\
&= u^2(x_p) - u^2(KCRV_A) + u^2(x_q) - u^2(KCRV_B) - 2u^2(KCRV_A)u^2(KCRV_B) \sum_{k=1}^3 1/u^2(x_k) \\
&= u^2(D_p) + u^2(D_q) - 2u^2(KCRV_A)u^2(KCRV_B) \sum_{k=1}^3 1/u^2(x_k). \tag{A11}
\end{aligned}$$

As the variance of $D_p - D_q$ is expressed as $u^2(D_p - D_q) = u^2(D_p) + u^2(D_q) - 2u(D_p, D_q)$, the last term in equation (A11) is therefore equal to the correlation term $2u(D_p, D_q)$, proving that

$$u(D_p, D_q) = u^2(KCRV_A)u^2(KCRV_B) \sum_{k=1}^3 1/u^2(x_k) = \frac{\sum_{k=1}^3 1/u^2(x_k)}{\sum_A \frac{1}{u^2(x_i)} \sum_B \frac{1}{u^2(x_j)}}. \tag{A12}$$