30/6/2014

Report of key comparison CCQM – K105

"Electrolytic conductivity at 5.3 S·m^{-1}"

Final report

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Abstract

The aim of the key comparison CCQM-K105 was to demonstrate the measurement capabilities of the participating institutes with respect to the conductivity of multi-component aqueous salt solutions. Practical salinity results are currently not traceable to metrological references consistent with the International System of Units (SI). Nevertheless, salinity is one of the most important input quantities for oceanographic models, whose measurement data must be accurate on very long time scales. Thus, in order to determine the practical salinity value, there is a strong interest on the part of oceanographic researchers in establishing the traceability to the SI of conductivity measurements. To this end, the conductivity of a standard seawater sample, provided by the support laboratory (PTB), was measured in a way traceable to the SI. The nominal conductivity values of the solution were 5.3 $S\cdot m^{-1}$ at 25 °C and 4.3 $S\cdot m^{-1}$ at 15 °C. Thirteen institutes taking part in the comparison had to measure the conductivity values of the sample at both the temperatures. The median was chosen for both values as an estimator for the KCRV, evaluated on the basis of the Monte Carlo method. An institute requested to be excluded from the determination of the KCRV because of contact problems of its cell. At 25 °C the KCRV is 5.3024 $S\cdot m^{-1}$ with an interval of confidence (at the 95.45 % level of significance) from 5.3005 $S\cdot m^{-1}$ to 5.3044 $S\cdot m^{-1}$. At 15 °C the KCRV is 4.2892 $S\cdot m^{-1}$ with an interval of confidence (at the 95.45 % level of significance) from 4.2877 $S\cdot m^{-1}$ to 4.2907 $S\cdot m^{-1}$. For the "How far the light shines" statement the CMCs can cover the range 1 - 15 $S\cdot m^{-1}$ both for the values 5.3 $S\cdot m^{-1}$ at 25 °C and 4.3 $S\cdot m^{-1}$ at 15 °C. This comparison is a follow-up of the CCQM Pilot Study P111.

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1.1 Coordinating laboratory:
Istituto Nazionale di Ricerca Metrologica – INRiM

1.2 Supporting laboratory:
Physikalisch-Technische Bundesanstalt – PTB

1.3 Metrology Area
Amount of Substance

1.4 Branch
Electrochemistry

1.5 Subject
Determination of the electrolytic conductivity of an unknown sample of standard seawater with nominal value 5.3 $S \cdot m^{-1}$ at 25 °C, and 15 °C.

1.6 Time schedule

September 2012	Technical protocol, approval and solution preparation
October 2012	Dispatch of the samples
November 2012	Measurement period
January 2013	Deadline for receipt of the report
April 2013	(EAWG at BIPM) Discussion of results and Draft A

1.7 Participants
Participants are listed in Table 1.

Table 1: List of Participants

N°	Acronym	Institute	Country	Contact Person
1	BelGIM	Belarussian state institute of metrology	BLR	Alena Zalatarevich, Olga Sevruk
2	CENAM	National Metrology Institute of Mexico	MEX	Aarón Rodríguez Lopez, Jonathan Luevano Sanchez
3	DFM	Danish Fundamental Metrology	DK	Pia Tønnes Jakobsen
4	GUM	Central Office of Measures	PL	Wladyslaw Kozlowski
5	INMETRO	National Institute of Metrology, Quality and Technology	BR	Fabiano Barbieri Gonzaga
6	INPL	The National Physical Laboratory of Israel	IL	Elena Kardash
7	INRIM	Istituto Nazionale di Ricerca Metrologica	I	Francesca Durbiano Elena Orrù
8	MKEH	Hungarian Trade Licensing Office	H	Zsófia Nagyné Szilágyi
9	NMIJ	National Metrology Institute of Japan	J	Igor Maksimov,

				Toshiaki Asakai
10	PTB	Physikalisch-Technische Bundesanstalt	GER	Steffen Seitz
11	SMU	Slovenský metrologický ústav	SK	Leoš Vyskočil
12	SP	SP Technical Research Institute of Sweden	S	Rauno Pyykkö, Bertil Magnusson
13	Ukrcsm	Ukrmetrteststandart Metrologhichna	UKR	Vladymyr Gavrilkin
14	VNIIFTRI	National Research Institute Physicotechnical and Radio Engineering Measurements	RUS	Yury A. Ovchinnikov
15	VNIIM	D.I. Mendeleyev Institute for Metrology	RUS	Leonid A. Konopelko

2. Sample description

2.1 Sample preparation and distribution

The solution used in this comparison was a standard seawater sample from OSIL, P-series batch P154. It is natural seawater taken from the North Atlantic ocean and diluted with water to a Practical Salinity value of 35 (which corresponds to around 5.3 $S\cdot m^{-1}$ at 25 °C and around 4.3 $S\cdot m^{-1}$ at 15 °C). The solution was bought by PTB, who labelled the 200 ml bottles containing the solution with the numbers and K105 identifier and distributed them to the participants without them undergoing any further treatment. The bottles were sealed with tamper-evident closures, which were broken upon opening. Participating laboratories received the number of bottles they had requested. Shipment to all participants was performed at the same time. The bottles were shipped in a cardboard box by courier. The contents were labelled "seawater sample" with no commercial value.

2.2 Checking of the sample integrity

Participants were requested to weigh the received bottles to verify that they had undergone no changes during transportation.

If the discrepancy between bottle mass measured by participants and the mass reported in the datasheet by the supporting laboratory were greater than 0.2 g, the participant would have looked for possible leakage and informed the coordinating laboratory. The coordinating laboratory did not receive any messages, therefore the sample integrity is assumed true for all participants.

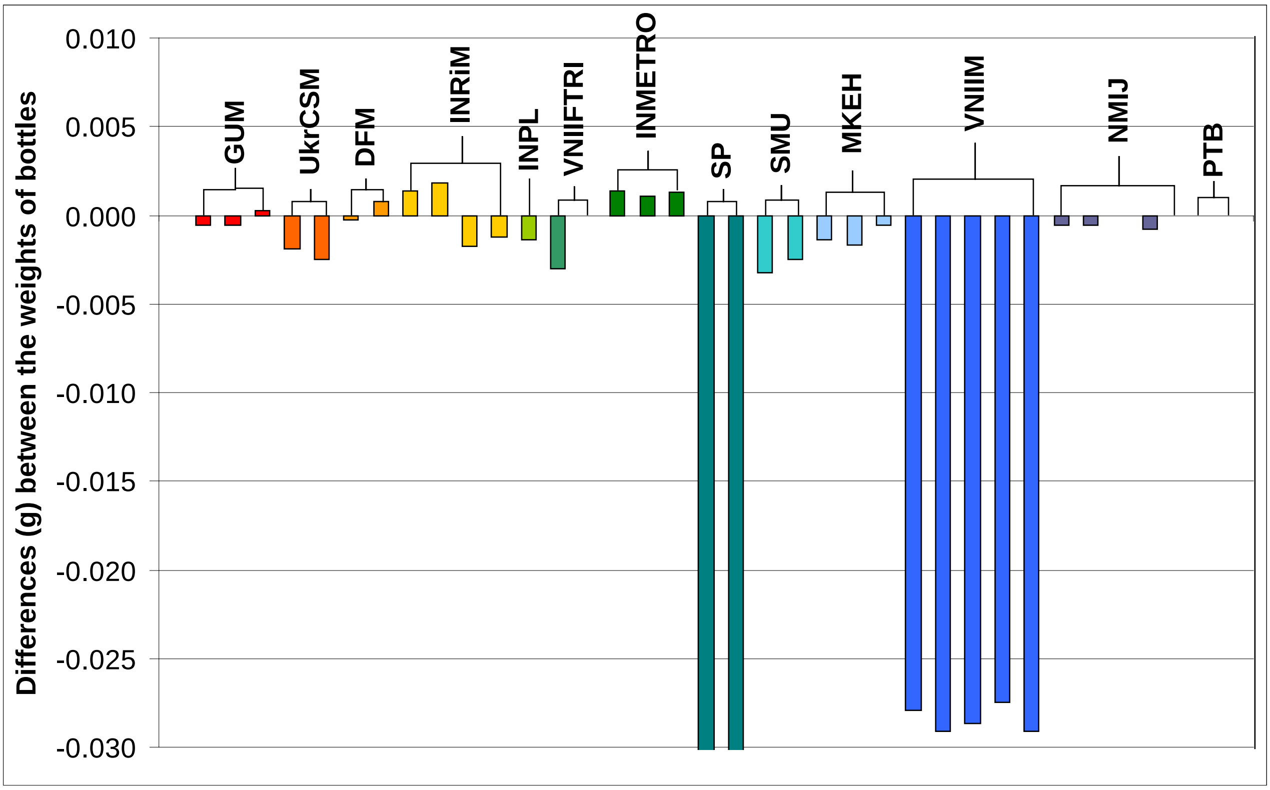

Figure 1: Relative differences between the initial and reported weight of bottles

2.3 Homogeneity and stability

The standard seawater batch P154 used in this comparison was bought from OSIL, which guaranteed its homogeneity [1,2]. Homogeneity and stability tests on the standard seawater batch P154 were also performed by PTB on 27/8/2012 and 2/5/2013 by means of conductance measurements, using a Guildline Autosal 8400B Salinometer. Measurements were performed at 24 °C (nominal set value of the salinometer). Table 2 reports the readings obtained on 2/5/2013 in arbitrary units. The mean of the readings is 1.99965. The standard deviation of the 24 readings, which were considered independent, is 2.49×10^{-5}, which yields a relative homogeneity of 1.25×10^{-5}. The corresponding homogeneity of the conductivity is 6.60×10^{-5} S/m. On the basis of the PTB measurements, reported in Table 2 and 3, a drift coefficient of roughly 10^{-7}/day can be estimated. Since the participating institutes carried out the key comparison measurements from 12/10/2012 to 23/1/2013 (about 100 days), the effect of drift was considered negligible.

The homogeneity of the batch was taken into account in the evaluation of the uncertainty of the KCRV as described in § 4.1.

Table 2. Conductivity measurements in arbitrary units (2/5/2013).

Reading	Bottle 1	Bottle 2	Bottle 3	Bottle 4
1	1.99959	1.99967	1.99965	1.99962
2	1.99963	1.99964	1.99963	1.99962
3	1.99964	1.99965	1.99966	1.99963
4	1.99967	1.99964	1.99967	1.99964
5	1.99966	1.99963	1.99965	1.99965
6	1.99971	1.99966	1.99968	1.99968

Table 3. Conductivity measurements in arbitrary units (27/8/2012).

Reading	Bottle 1
1	1.99959
2	1.99960
3	1.99961
4	1.99961
5	1.99961
Mean	1.99960

3. Results

Nominal value of electrolytic conductivity of seawater sample was 5.3 S·m^{-1} at 25 °C. 15 participating NMIs were requested to measure the conductivity of the sample at the nominal temperature of 25 °C and 15 °C. Afterwards, they were requested to correct the results to exactly 25.000 °C and 15.000 °C respectively.

3.1 Withdrawals

Two laboratories withdrew from the comparison (CENAM and BelGIM).
After the EAWG meeting of the CCQM held at BIPM on 15/4/2013, SMU requested to be excluded from the determination of the KCRV because their cell experienced contact problems.

3.2 Results at 25 °C

The results of electrolytic conductivity values at the temperature of 25 °C are given in Table 4 and Figure 2.

Table 4: Results for the electrolytic conductivity (EC) of the seawater sample at 25 °C

N°	Institute	EC S·m^{-1}	*u* S·m^{-1}	$U_{95.45\%}$ S·m^{-1}
1	SMU	5.2779	0.0027	0.0054
2	INRiM	5.2910	0.0191	0.0382
3	NMIJ	5.2959	0.0041	0.0082
4	SP	5.2960	0.0090	0.018
5	INPL	5.2989	0.0045	0.009
6	INMETRO	5.2990	0.0018	0.0052
7	VNIIM	5.3010	0.0008	0.0016
8	GUM	5.3027	0.0018	0.0035
9	PTB	5.3036	0.0017	0.0034
10	VNIIFTRI	5.3040	0.0011	0.0022
11	MKEH	5.3040	0.0015	0.0030
12	DFM	5.3069	0.0017	0.0035
13	UkrCSM	5.3113	0.00135	0.0027

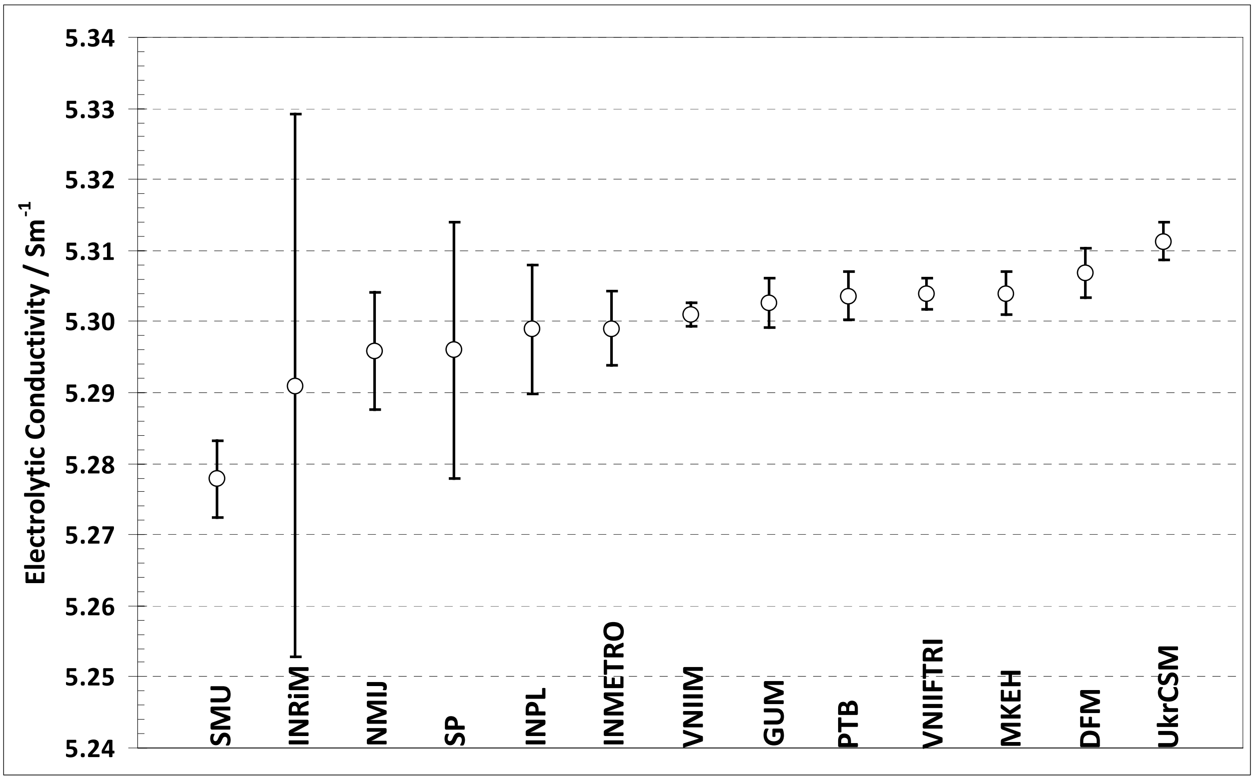

Figure 2: Plot of results for seawater sample at 25 °C and their expanded uncertainties

3.3 Results at 15 °C

The results of electrolytic conductivity values at the temperature of 15 °C are given in Tables 5 and Figure 3.

Table 5: Results for the electrolytic conductivity (EC) of the seawater sample at 15 °C

N°	Institute	EC $S \cdot m^{-1}$	u $S \cdot m^{-1}$	$U_{95.45\%}$ $S \cdot m^{-1}$
1	SMU	4.2766	0.0026	0.0052
2	NMIJ	4.2772	0.0039	0.0078
3	SP	4.2830	0.0077	0.0150
4	INMETRO	4.2849	0.0015	0.0040
5	INPL	4.2854	0.0045	0.0090
6	INRiM	4.2856	0.0131	0.0261
7	PTB	4.2884	0.0009	0.0018
8	VNIIFTRI	4.2891	0.0009	0.0018
9	VNIIM	4.2900	0.0008	0.0017
10	GUM	4.2913	0.0016	0.0031
11	DFM	4.2914	0.0014	0.0029
12	MKEH	4.2918	0.0017	0.0034
13	UkrCSM	4.2919	0.00114	0.0023

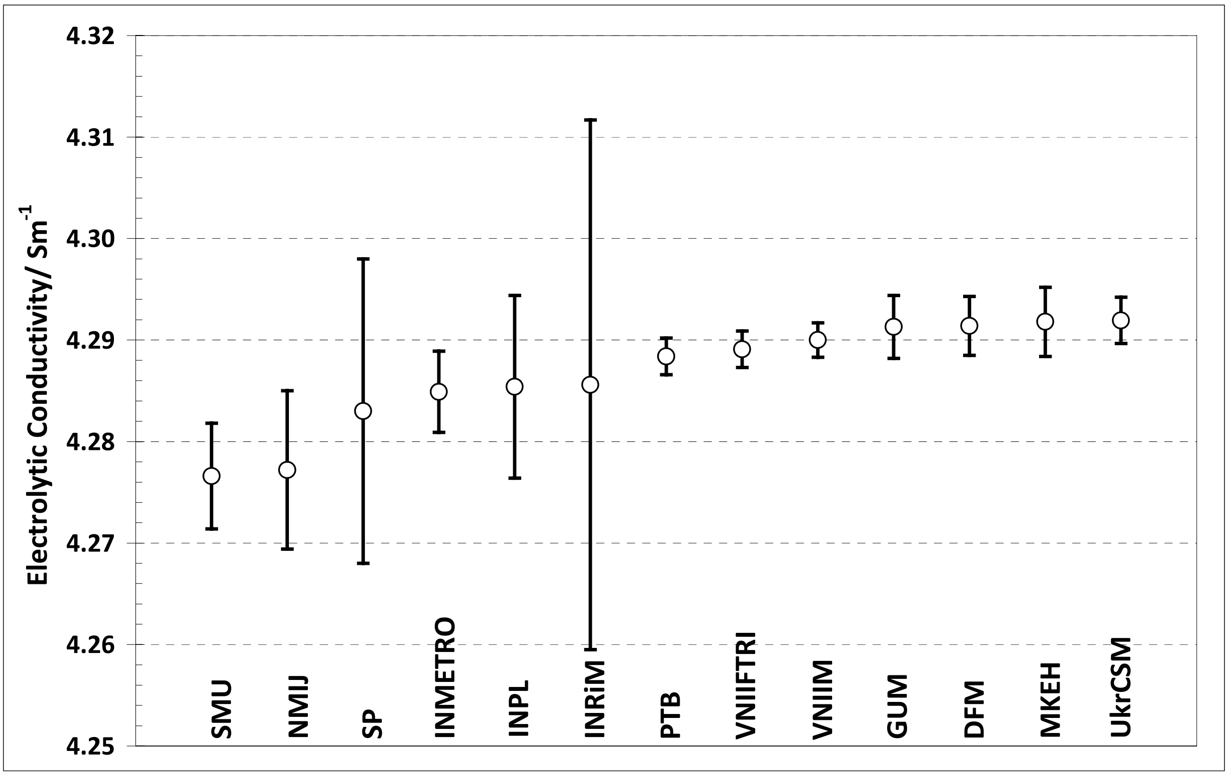

Figure 3: Plot of results for seawater sample at 15 °C and their expanded uncertainties

4. Discussion

To determine the K105 key comparison reference value (KCRV), the associated uncertainty and the degree of equivalence of the measurement made by each participating national institute, the guidelines proposed by Cox in [3] were applied.

4.1 Calculation of the KCRV and its uncertainty at 25 °C

Initially, procedure A reported in [3] was applied and the weighted mean was calculated considering all of the measurements from each institute. However, the chi-square test applied to the results (to carry out an overall consistency check) failed. Consequently, procedure B reported in [3], which is based on the Monte Carlo method, was employed. A random sample of 10^6 trials for each temperature was used. Each random sample is the sum of two other random samples, one associated with the uncertainties of the institutes' results and one associated with the inhomogeneity of the sea water sample. These two random samples were generated according to the following: i) the measurements were considered independent; ii) a Gaussian probability distribution was associated with the measurements of the institutes which declared a coverage factor of 2; iii) a t-student distribution with appropriate degrees of freedom was associated with the measurements of the one institute (INMETRO) which declared a coverage factor of 2.87 at 25 °C (ν = 4) and 2.65 at 15 °C (ν = 5); iv) a Gaussian probability distribution with a standard deviation of 6.60×10^{-5} S/m was associated with the inhomogeneity of the sample.
For the evaluation of the median and the associated uncertainty, the algorithm at points 3-10 of procedure B was applied. d_{i_low} and d_{i_up} are the lower and the upper endpoints of the 95.45% interval of confidence of d_i.
Table 6 and Figure 4 show the results for the degree of equivalence of each institute for the seawater sample at 25 °C using median as an estimator.

Table 6: Results for the degrees of equivalence of institutes for seawater sample at 25 °C. Estimator: median.

Institute	d_i S·m^{-1}	d_{i_low}	d_{i_up}
SMU	-0.024	-0.030	-0.019
INRiM	-0.011	-0.049	0.026
NMIJ	-0.0065	-0.015	0.0012
SP	-0.0064	-0.024	0.011
GUM	-0.0035	-0.012	0.0050
INPL	-0.0034	-0.0088	0.0012
INMETRO	-0.0014	-0.0039	0.00072
VNIIM	0.0003	-0.0033	0.0040
PTB	0.0012	-0.0020	0.0050
VNIIFTRI	0.0016	-0.00085	0.0044
MKEH	0.0016	-0.0013	0.0051
DFM	0.0045	0.00055	0.0084
UkrCSM	0.0089	0.0055	0.012

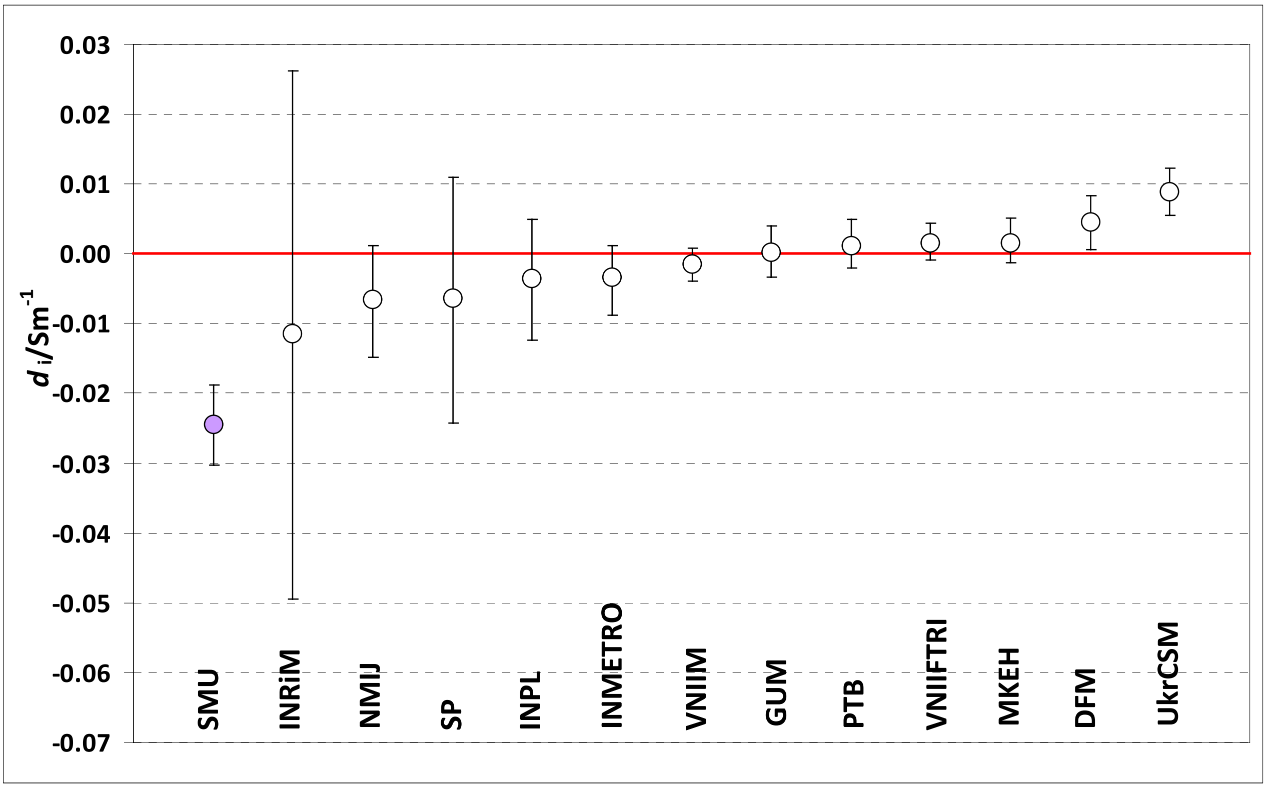

Figure 4. Seawater sample at 25 °C. Plot of the degrees of equivalence and 95.45% interval of confidence according to Cox procedure B.

A summary of the results obtained for the seawater sample at 25 °C is reported in Table 7.

Table 7: Summary of the results for seawater sample at 25 °C.

Participants	Median (S·m^{-1})	u_{Med} (S·m^{-1})	X_{ref_low} (S·m^{-1})	X_{ref_up} (S·m^{-1})
All, SMU excluded	5.3024	0.0010	5.3005	5.3044

4.2 Calculation of the KCRV and its uncertainty at 15 °C

Initially, also in the case of the seawater sample at 15 °C procedure A was applied. However, also in this case the chi-squared test failed. Consequently, procedure B was considered. A random sample of 10^6 trials was used.

Table 8 and Figure 6 show the results for the degree of equivalence of institutes for seawater sample at 15 °C using median as an estimator.

Table 8: Results for the degrees of equivalence of institutes for seawater sample at 15 °C. Estimator: median.

Institute	d_i $S \cdot m^{-1}$	d_{i_LOW}	d_{i_UP}
SMU	-0.013	-0.018	-0.0072
NMIJ	-0.012	-0.020	-0.0041
SP	-0.0062	-0.022	0.0086
INMETRO	-0.0043	-0.0082	-0.000036
INPL	-0.0038	-0.013	0.0048
INRiM	-0.0036	-0.030	0.022
PTB	-0.00082	-0.0031	0.0010
VNIIFTRI	-0.00012	-0.0022	0.0019
VNIIM	0.00078	-0.0010	0.0029
GUM	0.0021	-0.00085	0.0055
DFM	0.0022	-0.00055	0.0052
MKEH	0.0026	-0.00060	0.0062
UkrCSM	0.0027	0.000013	0.0052

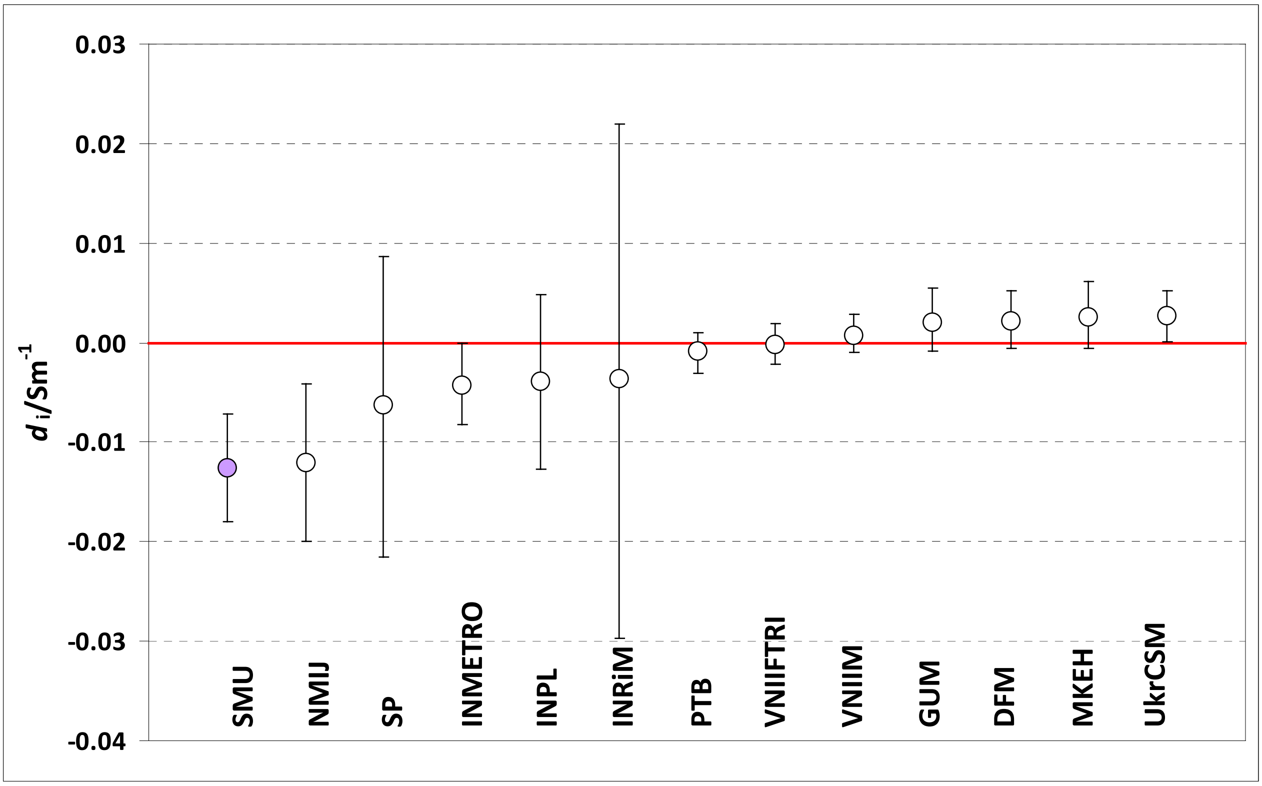

Figure 6: Seawater sample at 15 °C. Plot of the degrees of equivalence and 95.45% interval of confidence according to Cox procedure B.

A summary of the results obtained for the seawater sample at 15 °C is reported in Table 9.

Table 9: Summary of the results for seawater sample at 15 °C.

Participants	Median (S·m^{-1})	u_{Med} (S·m^{-1})	X_{ref_low} (S·m^{-1})	X_{ref_up} (S·m^{-1})
All, SMU excluded	4.28922	0.00074	4.28774	4.29068

4.3 Communication with the participants

- INPL was requested to check its results [30/1/2013]; INPL sent a revised report [10/2/2013].
- SMU was requested to check its results [30/1/2013 and 28/3/2013]; Mariassy explained that at SMU measurements had to be performed in a cell experiencing contact problems because the new fit for purpose cells had not arrived in time. Then, after arriving and calibration of the new cells, one measurement of the remaining K105 sample at 25 °C was carried out that gave a result of conductivity 5.2898 S·m^{-1}, higher than the reported result 5.2779 S·m^{-1}. This new value was not used in the KCRV evaluation, accordingly to the document CIPM MRA-D-05 [4], [31/1/2013 and 4/4/2013]. During the meeting of EAWG of CCQM on 15/4/2013 Mariassy requested to be excluded from the determination of the KCRV because their cell experienced contact problems.
- SP was requested to check its results [17/1/2013]. Since an evident transcription error was reported, a revised report was accepted [22/1/2013].
- NMIJ was requested to check their results; but they decided to keep unchanged their submitted data [1/4/2013].
- INMETRO was requested to check their results [28/3/2013]; but they decided keep unchanged their submitted data [5/4/2013].
- UkrCSM was requested to check their results [28/3/2013]; but they didn't found a reason for changing their submitted data [23/4/2013].
- GUM was requested to check their results [28/3/2013]; GUM sent a revised report [4/4/2013].
- DFM was requested to check their results [28/3/2013]; DFM found no major errors. The only thing they found was that one of the three measurements have not been corrected using the measured values of the standard resistor. Data by DFM were not changed, accordingly to [5], [2/4/2013 and 3/4/2013].
- VNIIM was request to indicate a single value of electrolytic conductivity at each temperature [21/3/2013]; VNIIM sent a revised report [27/3/2013].
- After INRiM has sent a final request by email to CENAM, asking for results [15/3/2013], CENAM, due to technical difficulties with primary system of electrolytic conductivity, withdrew from the comparison [20/3/2013].
- BelGIM has not sent a report of the comparison; after INRiM one last email asking for results [4/4/2013] BelGIM was considered withdrawn from the comparison.

4.4 Reference values and degrees of equivalence: choice of proper estimators

When Procedure A fails, there is no general consensus about the choice of the proper algorithm for the determination of a reference value, its uncertainty and the interval of confidence of the degrees of equivalence. Possible approaches are: the method from Nielsen [6], the Cox Procedure B [3] and the external consistency concept. They are reported in Appendix A. The different KCRV estimators provide similar impact on the degrees of equivalence. Nevertheless, Cox Procedure B [3] is

considered preferable for stating the final comparison result because of the large number of results that need to be purged with other methods [5,6]. Therefore, Cox Procedure B was accepted by the Electrochemical Analysis Working Group of CCQM to be used as a proper estimator for the determination of a reference value, its uncertainty and the interval of confidence of the degrees of equivalence.
However, Cox Procedure B provides asymmetrical uncertainty intervals, but the asymmetry in this comparison is less than 11 % of the uncertainty. Consequently, with regard to CMCs, those supported by this comparison have to be compiled considering the largest interval of the declared uncertainty.

4.5 How far the light shines statement

For the "How far the light shines" statement the document EAWG CMC KC guidelines v9.doc was followed.
As a result of a successful participation in a comparison, a reasonable CMC claim will be made for a range covering one order of magnitude of conductivity, with the measured value in the supporting comparison being nominally in the (logarithmic) centre of this range.
For the nominal values 5.3 $S \cdot m^{-1}$ at 25 °C and 4.3 $S \cdot m^{-1}$ at 15 °C the range will be 1 – 15 $S \cdot m^{-1}$.

References

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Appendix A

1. Alternative evaluation by searching a consistent subset at 25 °C

On the basis of an alternative procedure proposed by Nielsen [6], the weighted mean of a consistent subset of data was calculated. In this process procedure A [3] was iteratively applied by excluding for each iteration the more discrepant datum, and by recomputing the weighted mean and performing a chi-square test on the remaining result subset until a consistent subset was obtained.

The results to be removed from the dataset to achieve this condition are those of SMU, UkrCSM and DFM. Table 10 and Figure 7 show the results for the degree of equivalence of institutes for seawater sample at 25 °C using weighted mean as an estimator.

Table 10: Results for the degrees of equivalence of institutes for seawater sample at 25 °C. Estimator: weighted mean (excluding SMU, UkrCSM and DFM)

Institute	d_i $S \cdot m^{-1}$	$U(d_i)$ $S \cdot m^{-1}$
SMU	-0.024	0.0055
INRiM	-0.011	0.0382
NMIJ	-0.0061	0.0081
SP	-0.0060	0.0180
INPL	-0.0031	0.0089
INMETRO	-0.0030	0.0035
VNIIM	-0.0010	0.0012
GUM	0.00065	0.0035
PTB	0.0015	0.0032
VNIIFTRI	0.0019	0.0019
MKEH	0.0019	0.0028
DFM	0.0048	0.0035
UkrCSM	0.0092	0.0029

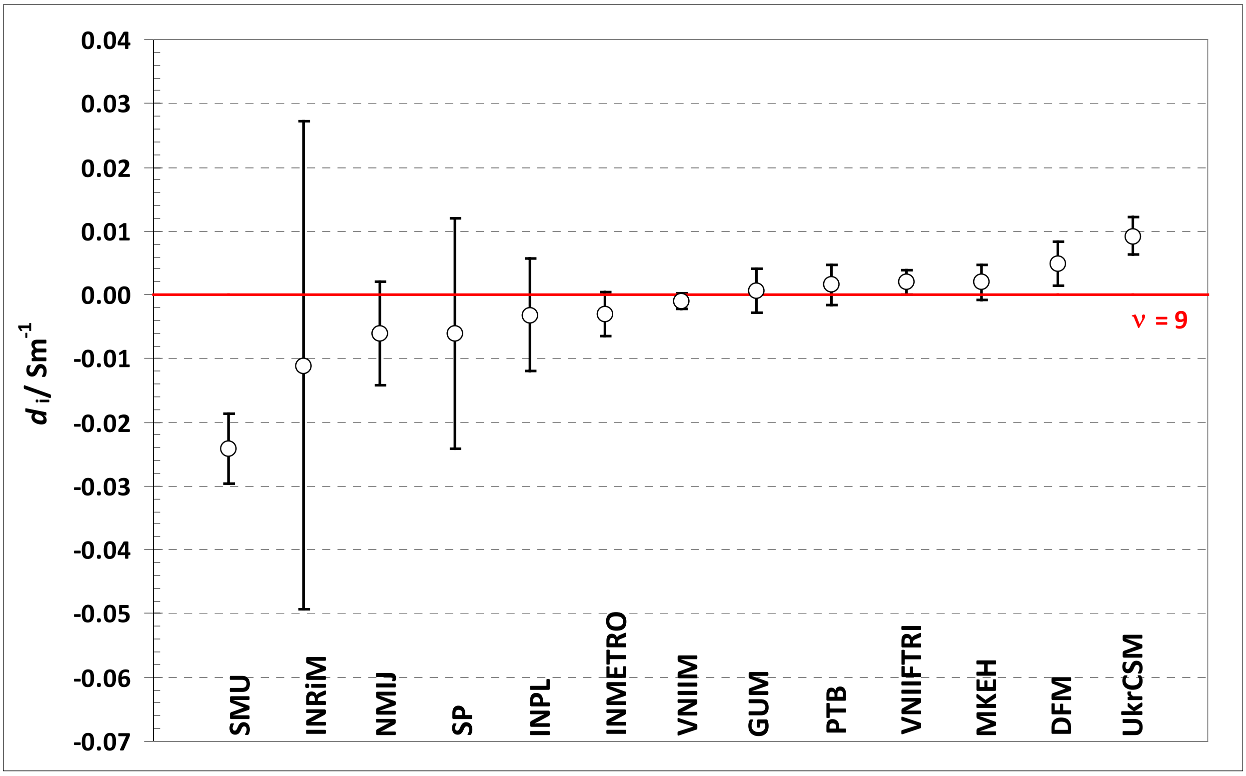

Figure 7: Seawater sample at 25 °C. Plot of the degrees of equivalence and 95% interval for the consistent subset obtained according to Appendix A § 1. Estimator: weighted mean.

A summary of the results obtained for the seawater sample at 25 °C is reported in Table 11.

Table 11: Summary of the results for seawater sample at 25 °C.

Participants	Degrees of freedom, ν	χ^2_{obs}	χ^2_{tab} 95% probability	Weighted mean (S·m^{-1})	Standard Uncertainty (S·m^{-1})
All	12	146.6	21.0	5.30275	0.00045
SMU excluded	11	59.5	19.7	5.30345	0.00046
SMU, **UkrCSM** excluded	10	21.4	18.3	5.30244	0.00048
SMU, **UkrCSM**, **DFM** excluded	9	13.9	16.9	5.30205	0.00051

2. Alternative evaluation by searching a consistent subset at 15 °C

The same procedure of Appendix A § 1 is applied. The results to be purged from the dataset to achieve this condition are those of SMU, NMIJ, INMETRO. Table 12 and Figure 8 show the results for the degree of equivalence of institutes for seawater sample at 15 °C using weighted mean as an estimator.

Table 12: Results for the degrees of equivalence of institutes for seawater sample at 15 °C. Estimator: weighted mean (excluding SMU, NMIJ, INMETRO).

Institute	d_i S·m^{-1}	$U(d_i)$ S·m^{-1}
SMU	-0.013	0.0053
NMIJ	-0.013	0.0078
SP	-0.0070	0.0154

INMETRO	-0.0051	0.0031
INPL	-0.0046	0.0090
INRiM	-0.0044	0.0262
PTB	-0.0016	0.0016
VNIIFTRI	-0.00087	0.0016
VNIIM	2.5E-05	0.0014
GUM	0.0013	0.0031
DFM	0.0014	0.0027
MKEH	0.0018	0.0033
UkrCSM	0.0019	0.0021

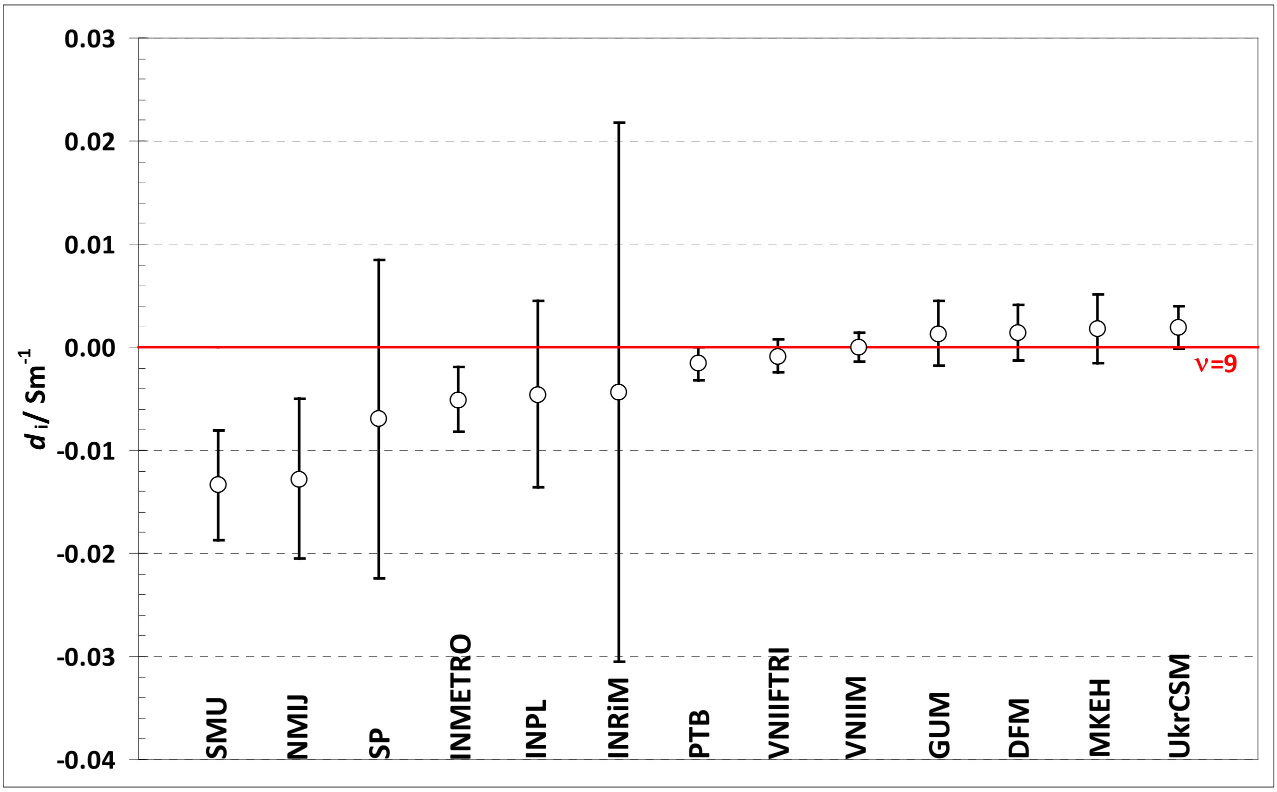

Figure 8: Seawater sample at 15 °C. Plot of the degrees of equivalence and 95% interval of confidence of the consistent subset found according to Appendix A § 1. Estimator: weighted mean.

A summary of the results obtained for the seawater sample at 15 °C is reported in Table 9.

Table 13: Summary of the results for seawater sample at 15 °C.

Participants	Degrees of freedom, ν	χ^2_{obs}	χ^2_{tab} 95% probability	Weighted mean ($S\cdot m^{-1}$)	Standard Uncertainty ($S\cdot m^{-1}$)
All	12	56.5	21.0	4.28923	0.00038
SMU excluded	11	32.4	19.7	4.28951	0.00039
SMU, **NMIJ** excluded	10	22.4	18.3	4.28963	0.00039
SMU, **NMIJ**, **INMETRO** excluded	9	11.7	16.9	4.28998	0.00040

3. Alternative evaluation by considering the external consistence concept
In general the assumptions for the external consistency evaluation that have to be taken into account are [1,2]:
1) The travelling standard has to be stable;
2) The measurement of each NMI has to be realised independently of the measurements of other NMIs;
3) For each NMI a Gaussian distribution has to be assigned to the declared value;
4) Lab data-set of each NMI should be available for the analysis;
5) The uncertainty budget of each NMI should include only Type A uncertainty.

In CCQM-K105 case the assumptions for the external consistency evaluation on which this estimator is based are not met:
1) Data-sets of the NMIs are not available;
2) One NMI implicitly declared a non-Gaussian distribution;
3) The uncertainties of di should be evaluated taking into account the correlations between xi and the weighted mean.
Therefore, the external consistency evaluation can not be used for the KCRV estimation.

Appendix A References

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Appendix B

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