Report to the CCT on Key Comparison 4

Comparison of Local Realisations of Aluminium and Silver Freezing-Point Temperatures

prepared by
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Contents

1 Introduction

The CCT Key Comparison 4 (K4) is defined as the comparison of the local realisation of ITS-90 temperatures using aluminium and silver freezing-point cells for all participating standard laboratories and is part of the mutual recognition arrangement (MRA), signed in 1999. K4 was initiated by the CCT during its meeting in September 1996. There, the IMGC was invited to be the pilot laboratory of K4. Due to local problems at IMGC, the pilot laboratory was finally transferred to the PTB in Berlin in the summer of 1997. One of the authors (H. G. Nubbemeyer) was authorised to be the coordinator of K4. The experiments for K4 were finally started in January 1998 at NIST and terminated in March 2000 at PTB.

2 Measurements

2.1 Organisation of the K4

The twelve members of K4 were organised in three subgroups (not including the coordinator) according to the geographical regions, with one exception.

American subgroup	3 NMIs
Asian/Australian subgroup	4 NMIs
European subgroup	4 NMIs.

The following table lists all participating standard laboratories together with the names of the main contributors.

PTB	Helmut G. Nubbemeyer (coordinator), Martina Becker
American subgroup	
NIST	Billy W. Mangum, Gregory F. Strouse (subcoordinators)
NRC	Ken Hill
IMGC	Piero Marcarino
Asian/Australian subgroup	
KRISS	Kee Sool Gam (subcoordinator)
NIM	Qiu Ping, Qu Yongmei
*NRLM**	Masaru Arai
NML	John Connolly
European subgroup	
NPL	Richard L. Rusby (subcoordinator), Jayne Gray
NMi/VSL	Martin de Groot, Jaco Dubbeldam
BNM/INM	Georges Bonnier, Eliane Renaot
VNIIM	Anatoly Pokhodun, Natalia Moiseeva, Alina Ivanova

* in the meantime the name of NRLM has changed to NMIJ

The comparisons of aluminium and silver freezing-point cells respectively were first carried out between the pilot‘s and the subcoordinator‘s laboratory successively, followed by round-robin or star comparisons in the subgroups. All aluminium and silver freezing-point cells as well as stable HTSPRTs were hand carried to the next K4 laboratory.

2.2 Additions to the Protocol

The Protocol (cf. Appendix) which is mandatory for all KC’s has been sent to the subcoordinators, who have forwarded it to all subgroup members. The Protocol has been acknowledged by all participants, actively or passively, before the measurements began. It describes in detail the comparison of fixed-point cells, the time schedule for all steps of the key comparisons, the data collection, and the data transfer to the pilot laboratory. The corrected schedule of comparisons is shown below:

PTB circulated cells at NIST	01/10/ - 01/30/1998
PTB circulated cells at KRISS	04/27/ - 05/16/1998
PTB circulated cells at NPL	06/14/ - 06/27/1998
NIST circulated cells at	
NRC	04/1998 - 09/1998
IMGC	10/1998 - 03/1999
KRISS circulated cells at	
NIM	07/1998 -10/1998
NRLM	11/1998 – 02/1999
NML	02/1999 - 06/1999
NPL circulated cells at	
NMi/VSL	08/1998 - 09/1998
BNM/INM	10/1998 - 11/1998
VNIIM	12/1998 - 01/1999
Back comparisons	
KRISS circulated cells at PTB	11/14/ - 11/29/1999
PTB circulated cells at NIST	01/09/ - 01/24/2000
NPL circulated cells at PTB	02/28/ - 03/03/2000

2.3 Laboratory Reports

Table 1 shows the typical parameters of the freezing-point cells used in this experiment. The first two columns show the standard laboratories and the denotations of the fixed-point cells (circulated cells in italics). Column 3 contains the mass and the supplier of the fixed-point metal, while column 4 lists the purity of the metal from the supplier. Column 5 shows the gas pressures inside the fixed-point cells. Finally, the last column gives the full immersion depth which represents the distance between the inside bottom of the quartz well and the upper melt level. For hydrostatic head corrections it has to be reduced by means of the distance between the mid-point of sensor (high temperature standard platinum resistance thermometer (HTSPRT)) to the bottom of the quartz well.

Table 2 lists the main information on the laboratory equipment. Column 1 shows the abbreviation of the standard laboratories, column 2 the resistance bridge model, column 3 the type and value of standard resistor(s), column 4 the bath for the standard resistor and its temperature. Finally, column 5 describes the furnace type for silver freezing-point cells and column 6 the furnace type for aluminium freezing-point cells. Additional information is given below, particularly it is specified whether the measurements were made by direct comparison (simultaneous freezes in identical furnaces) or by indirect comparison (sequential freezes in a single furnace).

BNM/INM

All measurements were made by direct comparisons. The aluminium cell BNM/INM Al 123 is a sealed cell. It was manufactured in January 1998 under the license of BNM/INM by Chauvin-Arnoux Pyrocontrôle. The main impurity in the ingot, according to Johnson Matthey, is Si with 0.3 ppm. The typical duration of the freeze plateau is approximately 10 hours. Calibrations of HTSPRTs or comparisons of fixed-point cells can be carried out during the first 30 % (or 3 h) of freeze after recalescence.

The silver cell BNM/INM Ag 105 is a sealed cell. It was manufactured in November 1995 under the license of Chauvin-Arnoux Pyrocontrôle. The impurities are, according to Johnson Matthey, 0.3 ppm Ca, 0.2 ppm Fe and 0.3 ppm Si. The typical duration of freeze is 10 hours in a pressure controlled heat pipe furnace (thermal enclosure B; ISPRA), it reduces to 4 hours in a classical sodium heat pipe (thermal enclosure A). The BNM/INM and the European subcoordinator's freezing-point cells were alternately compared in both furnaces.

IMGC

All measurements were made by direct comparisons. The open IMGC reference cells IMGC Al Co3 and IMGC Ag JM2 manufactured at IMGC were compared with the corresponding sealed (American coordinator's) circulated cells NIST Al 96-1 and NIST Ag 94-4 in two quasi identical sodium heat-pipe furnaces. The duration of a freeze-plateau for both IMGC cells lasted longer than 10 hours, if the set-point temperature was chosen appropriately (approximately 0.5 K below the freezing-point).

KRISS

All measurements were made by direct comparisons. The freezing-point cells PTB Ag 6 and PTB/NIST Al 94-3 as well as the HTSPRT PTB N 304 were hand-carried to KRISS. All KRISS freezing-point cells used for the key comparisons as KRISS Al, KRISS Al 97-1, KRISS Ag, and KRISS Ag 97-1 were assembled in their thermometry laboratory. The key comparisons were carried out in a Korean double sodium heat-pipe furnace. The resistance measurements were distorted by a much too high noise level on the two bridges (see Table 2), which led to a considerable increase of the uncertainties. The coordinator's HTSPRT PTB N 304 used for the comparisons at KRISS was extremely contaminated to such a high level, that it could no longer be used as a standard thermometer. The source of contamination was probably the KRISS annealing furnace. During the Asian/Australian round-robin the circulated aluminium freezing-point cell KRISS Al 97-1 was broken at NML after the circulation was quasi finished, except for the back calibrations. The circulated silver freezing-point cell KRISS Ag 97-1 was broken at NIM. It was replaced by the NIM silver freezing-point cell NIM Ag 9306, with which the round-robin had to be restarted at KRISS (circulated cell now KRISS/NIM Ag 9306). It was reported, that the circulated HTSPRT, KRISS 91400 became unstable during circulation.

Table 1: Fixed-point cell data collection for all participating standard laboratories. The abbreviations C, JM, SRM, R, and K in column 3 stand for the suppliers of the metal ingot, Cominco American, Johnson Matthey, NIST-SRM, Russian resources, and Koch Chemicals, England.

standard laboratory	fixed-point cell denotation; circulated cells in italics	mass of ingot [g]/ source	purity of the metal from the supplier	gas pressure [kPa]	Immersion depth [mm]
BNM/INM	BNM / INM Ag105	1130 / JM	>6 N	101.3	170
	BNM-INM Al 123	310 / JM	>6N	101.3	170
IMGC	IMGC Ag JM2	1342 / JM	6N	101.3 open	180
	IMGC Al Co3	347 / C	6N	101.3 open	180
KRISS	*KRISS Al 97-1*	410 / JM	6N	101.3	186
	KRISS Al	420 / JM	6N	101.3 open	192
	KRISS Ag 97-1	1500 / JM	6N	101.3	168
	KRISS/NIM Ag 9306	1300	6N	101.3	170
	KRISS Ag	1500 / JM	6N	101.3 open	168
NIM	Al1	350	6N	101.3	167
	NIM Ag 9402	1300	6N	101.3	170
	NIM Ag-9306	1300	6N	101.3	170
NIST	NIST Ag 92-1	1420 / SRM	> 6N	101.3 open	205
	NIST Ag 94-4	1420 / SRM	> 6N	101.3 open	205
	NIST Al 94-2	356 / SRM	> 6N	101.3 open	205
	NIST Al 96-1	356 / SRM	> 6N	101.3 open	205
NMi/VSL	VSL 92T199 (Ag)	2054 / JM	6N	101.3 open	235
	VSL 93T267 (Al)	526 / JM	6N	101.3 open	240
NML	NML Al 98/2	500 / JM	6N	atmospheric pressure	220
	NML Ag 93/1	1995 / JM	< 6N		220
NPL	NPL Ag 8/97	924 / JM	6N	101.3 open	174
	NPL Ag 10/98	930 / JM	6N	101.3 open	174
	NPL Ag 2/97	934 / JM	6N	101.3	174
	NPL Al 89	212 / C	6N	101.3 open	174
	NPL Al 2/97	237 / JM	6N	101.3	174
NRC	NRC Ag-6	1890 / JM	6N	101.3 open	210
	NRC Al-6	471 / JM	6N	101.3 open	210
NRLM	NRLM Ag 98-1	1800 / JM	6N	101.3 open	214
	NRLM Al 97-1	400 / JM	6N	101.3 open	212
PTB	*PTB Ag-6*	1439 / K	6N	95.0	180
	PTB/NIST Al 94-3	356 / JM	> 6N	101.3	205
	PTB Al 97-2	363,2 / SRM	> 6N	101.3	182
VNIIM	VNIIM Ag	410 / R	6N	101.3	180
	VNIIM Al	1422 / R	6N	101.3	180

Table 2: Experimental equipment in the participant's laboratories

standard laboratory	bridge model	standard resistor	resistor bath temperature	furnace type silver	furnace type aluminium
BNM/INM	F.18 Guildline 9975	Tinsley Guildline	Guildline bath	1 Na heat pipe furnace 1 Na heat pipe furnace, pressure controlled	1 Na heat pipe furnace 1 Cs heat pipe furnace, pressure controlled
IMGC	F.18 / GPIB	Tinsley 1 Ω	Tinsley thermostat 36 °C	2 Na heat pipe furnaces	2 Na heat pipe furnaces
KRISS	F.18 / Guildline9975	Tinsley 1 Ω, 10 Ω	oil bath (25±0.01) °C	2 Na heat pipe furnaces in one unit	2 Na heat pipe furnaces in one unit
NIM	F.18 / GPIB 9975/ Guildline	Tinsley 1 Ω	thermostat (20±0.01) °C	1 zone furnace with Ni bloc; 1 Na heat pipe furnace	1 zone furnace with Ni bloc
NIST	F.18 / GPIB	Tinsley 1 Ω, 10 Ω	oil bath (25±0.01) °C	2 Na heat pipe furnaces	2 Na heat pipe furnaces
NMi/VSL	F.18 / GPIB	Tinsley Wilk. Type	oil bath 28°C	2 Na heat pipe furnaces	2 Na heat pipe furnaces
NML	F.18 / GPIB	Guildline 1 Ω	stirred oil bath (20±0.02) °C	3 zone furnace with Na heat pipe	3 zone furnace with Na heat pipe
NPL	F.18 / GPIB	Tinsley model 5684	oil bath (20 ± 0.1) °C	2 Na heat pipe furnaces	2 Na heat pipe furnaces
NRC	F.18 / GPIB	Tinsley	oil bath (25± 0.002) °C	2 Na heat pipe furnaces	2 Na heat pipe furnaces
NRLM	F.18 / GPIB	Tinsley 1 Ω	thermostat 36 °C	2 Na heat pipe furnaces	2 Na heat pipe furnaces
PTB	F.18 / GPIB	Tinsley 1 Ω, 10 Ω	double wall thermostat, 23 °C in air	2 Na heat pipe furnaces +1 zone each	2 Na heat pipe furnaces +1 zone each
VNIIM	Guildline 9975	P-321 Metrologia, Krasnodar	thermostat 25°C	3 zone furnace	3 zone furnace

NIM

All Al measurements were made by direct comparisons, all Ag measurements by indirect comparisons. The Al and Ag fixed-point cells, NIM Al 1 and NIM Ag 9402, were manufactured at NIM. The KRISS circulating cell KRISS Ag 97-1 broke at NIM during the first phase of comparisons. Therefore, a temperature difference between cells NIM Ag and KRISS Ag 97-1 could not be assigned.

NIST

All measurements were made by direct comparisons. The K4 comparisons were started at NIST in January 1998. The PTB freezing-point cells PTB Ag 6 and PTB Al 97-1 together with the HTSPRT 93124 (Chinese origin) were hand-carried to NIST. Due to its lower stability the HTSPRT PTB 93124 was replaced by several HTSPRTs (Al measurements: RS87A-5, 5684-5-1005, 5684-7-1025; Ag measurements: BCT-N004, 5684-7-1025). All K4 measurements at NIST were finally carried out using these HTSPRTs. PTB Al 97-1 was finally replaced by NIST Al 94-3, the future circulated cell PTB/NIST Al 94-3 (sealed cell; originally open). The NIST reference cells NIST Al 94-2 and NIST Ag 92-1 were open cells. The key comparisons were carried out in one aluminium freezing-point furnace with a sodium heat-pipe, one silver freezing-point furnace with a sodium heat-pipe, one spare sodium heat pipe furnace for temperatures up to the silver freezing-point, and an annealing furnace (500°C to 1100°C). All furnaces were supplied with bottom heaters except the annealing furnace, which had a single zone heater. The temperature stability of all sodium heat-pipe furnaces was approximately 10 mK.

NMi/VSL

All measurements were made by direct comparisons. The NMi/VSL reference cells VSL93T267 (Al) and VSL92T199 (Ag) were manufactured at NMi/VSL. Before starting a freeze procedure, 7N Argon was filled into the fixed-point cells up to a pressure (100±1) mbar above ambient air pressure. After consultation with the coordinator the value for the silver freezing point was reconfirmed.

NML

All measurements were made by direct comparisons. The NML reference cells NML Al 98/2 and NML Ag 93/1 were manufactured at NML. They were mounted in quartz tubes with O-ring sealings. Before measurements the freezing-point cells were filled with argon to atmospheric pressure. The freezing-point cell NML Ag 93/1 showed a melting range of 20 mK. It has been reported that this could be due to an oxygen contamination. For both Al and Ag freezing-point cells three zone furnaces with heat-pipes were applied with a uniformity of 0.01 °C/cm over the length of the crucible. The aluminium freeze plateau only lasted two to three hours.

NPL

All measurements were made by direct comparisons. The sealed freezing-point cells NPL Al 2/97 and NPL Ag 2/97 as well as the 0.25 HTSPRT s/n 93094 were circulated in the European round-robin. The open freezing-point cells NPL Al 89, NPL Ag 8/97, and NPL Ag 10/98 (only for back calibrations) were used as reference cells. All freezing-point cells were made at NPL. For comparison the PTB circulated cells PTB/NIST Al 94-3 and PTB Ag 6 (also reference cell) were hand-carried to NPL. Two single zone heat-pipe furnaces were used for the comparison. In one of these furnaces the thermal shunts (shiny platinum) of the circulated cell PTB Ag 6 were coated (over approximately 40 hours) with an irremovable, dark layer, which certainly originated from the furnace. At the end of the European round-robin the sheath of the circulated HTSPRT 93094 broke. It was repaired but was not found to be stable at NPL.

NRC

All measurements were made by direct comparisons. Both open reference cells, NRC Al-6 and NRC Ag-6, were compared with the circulated cells NIST Al 96-1 and NIST Ag 94-4. The comparisons were carried out in two sodium heat-pipe furnaces. The first row of measurements with a 0.25 Ω SPRT yielded very unstable results.

Therefore the measurements were repeated with a NIST 25 Ω SPRT. These results are listed in the Appendix. Four of six silver freeze sets were withdrawn by NRC after consultation with the coordinator. The reason was that the water triple point resistances, which were measured for each freeze set, were only reliable for the third and the fourth set.

NRLM
All measurements were made by direct comparisons. The reference cells NRLM Al 97-1 and NRLM Ag 98-1 were compared with the circulated cells KRISS Al 97-1 and KRISS/NIM Ag 9306. It was reported that the circulated HTSPRT from KRISS, s/n 91400 showed a leakage effect at the silver freezing point as well as a comparatively high instability. The key comparisons were carried out in two sodium heat-pipe furnaces. The value for the aluminium freezing point was recalculated after consultation with the coordinator.

PTB
All measurements were made by direct comparisons. In the beginning of 1998, the PTB had prepared the two reference cells PTB Al 97-2 and PTB Ag 6 and one circulated cell PTB Al 97-1 for K4. At NIST, PTB and NIST decided not to use PTB Al 97-1 as the circulated cell. Instead, the aluminium freezing-point cell NIST Al 94-3 was used as the master circulated cell, which was then called PTB/NIST Al 94-3. For the back calibrations of K4 circulated cells from KRISS, NPL, and also PTB were used. PTB used four DC powered, sodium heat-pipe furnaces of quasi the same design, two for aluminium and two for silver. The temperature stability of the furnaces in the region of the ingots was better than 0.1°C/h. Only HTSPRTs from VNIIM were used at PTB. A special software was developed to run the K4 experiment including the resistance bridge and the furnaces automatically.

VNIIM
All measurements were made by direct comparisons. The reference cells VNIIM Al and VNIIM Ag were manufactured at VNIIM. For K4 two quasi identical three zone furnaces (Al) from a Russian manufacturer and two quasi identical four zone furnaces (Ag) of VNIIM design were installed. VNIIM aluminium and silver freezing-point cells were compared with the circulated freezing-point cells NPL Al 2/97 and NPL Ag 2/97 respectively from the European subgroup, using the VNIIM HTSPRT s/n 0092. A DC Guildline bridge model 9975 was used for resistance measurements.

3 Results

3.1 Immersion Characteristics

All participants of K4 determined the immersion distributions for all local reference and circulated cells of aluminium and silver used for key comparison 4. Most of the graphs in figures 1 and 2 were measured from low to high („down“) immersion depths, i.e. on insertion of the thermometer. In the graphs the temperature variation $T\text{-}T_{\mathrm{bottom}}$ of the SPRTs is plotted versus the distance that the thermometer was raised above full immersion. The PTB results were averaged out of „up“ and „down“ measurements. For NPL both directions were plotted. Due to the limited axis parameters some data of BNM/INM, NMi/VSL, NML, and NPL could not be plotted.

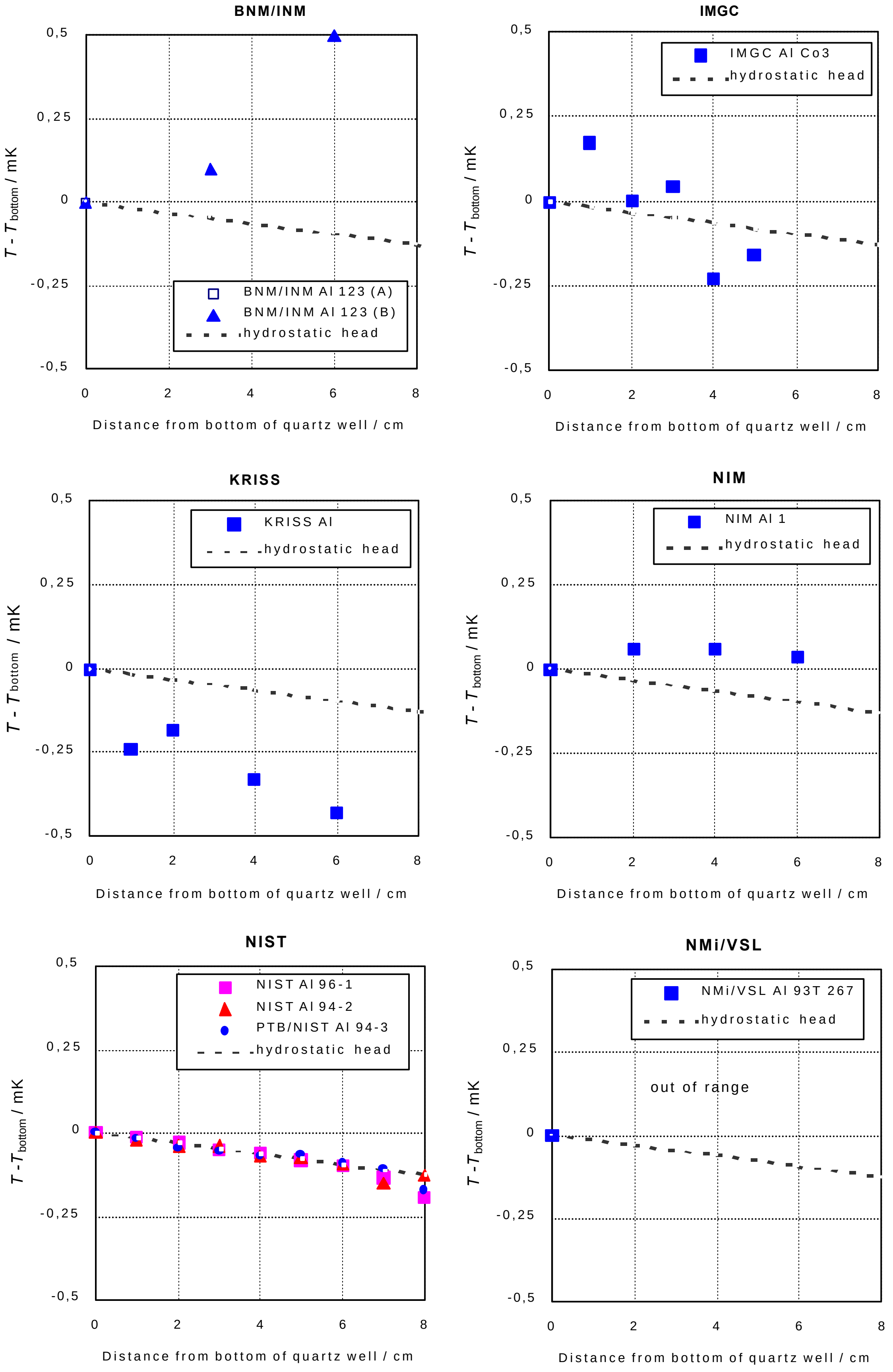

Figure 1: Immersion characteristics of all K4 Al freezing-point cells

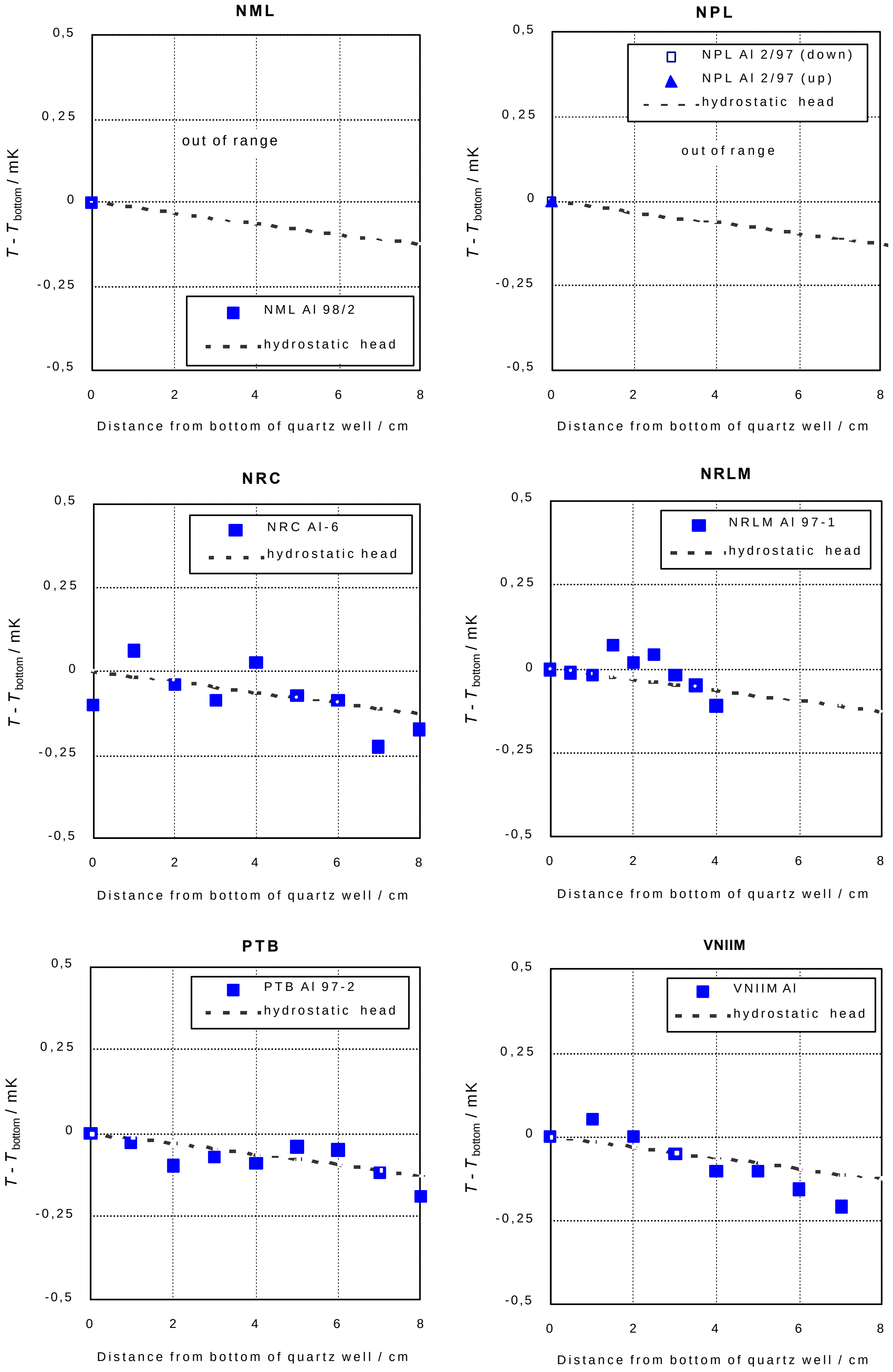

Figure 1: continued

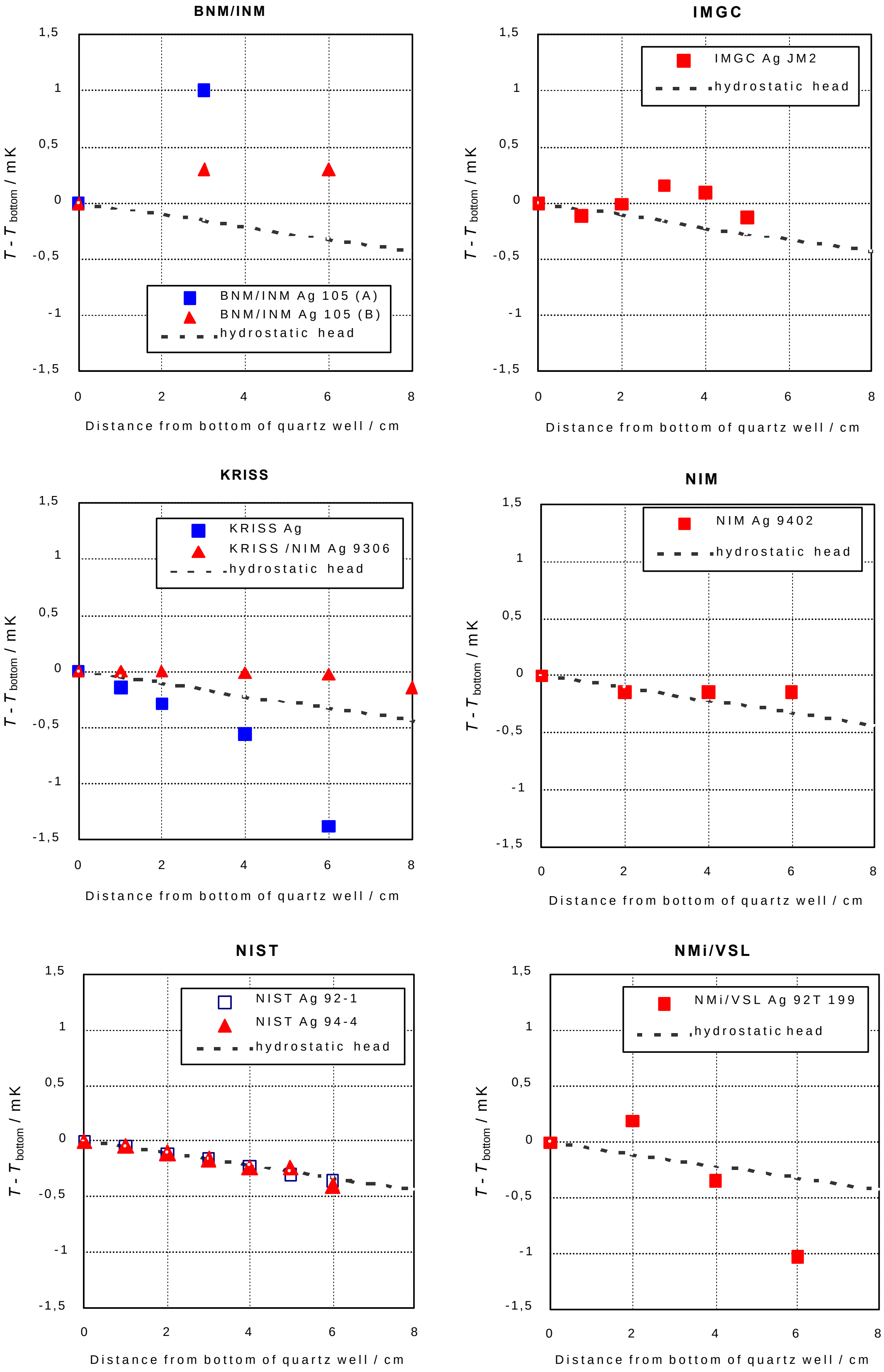

Figure 2: Immersion characteristics of all K4 Ag freezing-point cells

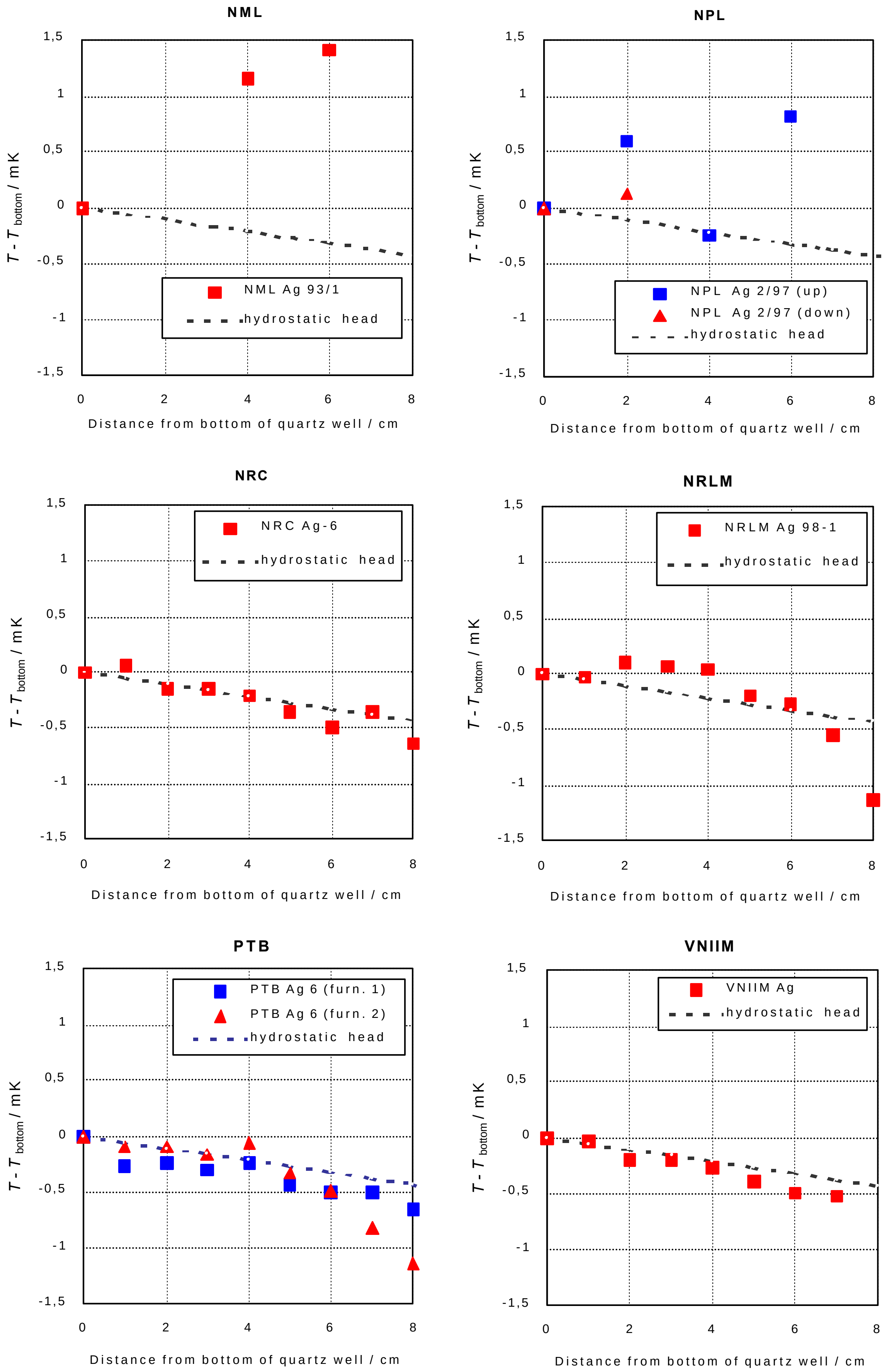

Figure 2: continued

3.2 Subgroup Results

All K4 comparisons of Al and Ag fixed-point cells were carried out in three subgroups, as described in section 2.1. Figures 3 through 8 represent the temperature differences of all aluminium and all silver freezing-point cells, which took part in the K4 comparisons. All figures show the temperature differences local minus circulated fixed-point cells in chronological order within their subgroup versus a measurement number which can also be found in the corresponding tables 3 and 4. Also included in the figures and tables are the comparisons to the circulated cell of the coordinator linking the three subgroups. The error bars indicate the expanded uncertainties (*k*=2) of the host laboratory supplying the corresponding local cell (see section 3.3 for details).

The temperature differences of the American subgroup are shown in figure 3 for aluminium and figure 4 for silver. The plot in figure 3 shows the Al comparison results for NIST Al 96-1 as the circulated cell and the following sequence of "local" cells: PTB/NIST Al 94-3 (master circulated cell; diamonds), NIST Al 94-2 (square), NRC Al-6 (triangle), NIST Al 94-2 (square), IMGC Al Co3 (circle), NIST Al 94-2 (square), and again PTB/NIST Al 94-3. The results show a comparatively small scatter, except for the master circulated cell, which is definitely not a reference cell. The plot in figure 4 shows the Ag comparison results for NIST Ag 94-4 as the circulated cell and the following sequence of "local" cells: PTB Ag 6 (master circulated cell; diamond), NIST Ag 92-1 (square), NRC Ag-6 (triangle), NIST Ag 92-1 (square), IMGC Ag JM2 (circle), NIST Ag 92-1 (square), and again PTB Ag 6. The last measurement was actually made against the cell NIST Ag 92-1 but was referenced to cell NIST Ag 94-4 for this figure. The scatter of data is especially small in this subgroup comparison.

The temperature differences of the Asian/Australian subgroup are shown in figure 5 for aluminium and figure 6 for silver. Figure 5 presents the Al comparison results for KRISS Al 97-1 as the circulated cell, except for the last two comparisons, for which KRISS Al 99-2 was chosen as the circulated cell. The following sequence describes the "local" cells: PTB/NIST Al 94-3 (diamond), KRISS Al (square), NIM Al 1 (triangle), NRLM Al 97-1 (circle), NML Al 98/2 (open square), PTB Al 97-2 (open diamond), and KRISS Al (square). The circulated cell KRISS Al 97-1 broke at NML which prevented a back calibration by means of the master circulated cell PTB/NIST Al 94-3.

The plot in figure 6 shows the Asian/Australian Ag comparison results for KRISS Ag 97-1 as the circulated cell for the first three comparisons, followed by KRISS/NIM Ag 9306 starting with comparison number 11. The following sequence represents the "local" cells: PTB Ag 6 (master circulated cell; diamond), KRISS Ag (square), KRISS Ag (square), NIM Ag 9402 (triangle), NRLM Ag 98-1 (circle), NML Ag 93/1 (open square), KRISS Ag (square), and PTB Ag 6 again. The scatter of data in this subgroup is definitely the highest in K4. During the measurements at NIM the KRISS circulated cell KRISS Ag 97-1 broke. Therefore, a value for the temperature difference between cells NIM Ag and KRISS Ag 97-1 could not be assigned and measurements 8 to 10 were not evaluated (values in brackets in Table 4). The cell has been replaced by a NIM cell later called KRISS/NIM Ag 9306. The comparisons had to be restarted at the beginning with data point 11.

The temperature differences of the European subgroup are shown in figure 7 for aluminium and figure 8 for silver. Figure 7 presents the Al comparison results for NPL Al 2/97 as the circulated cell. The following sequence describes the "local" cells: NPL Al 89 (square), PTB/NIST Al 94-3 (diamond), VSL 93T267 (triangle), BNM/INM Al123 (circle), VNIIM Al (open circle), NPL Al 89 (square), and PTB Al 97-2 (open diamond). The data point number 21 represents the back calibration and has been measured at PTB. The plot in figure 8 shows the European Ag comparison results with NPL Ag 2/97 as the circulated cell. The following sequence represents the "local" cells: NPL Ag 8/97 (square), PTB Ag 6 (master circulated cell; diamond), VSL 92T199 (triangle), BNM/INM Ag 105 (circle), VNIIM Ag (open circle), NPL Ag 10/98 (open square), and PTB Ag 6 again. Comparisons of measurements 18 and 23 confirm the stability of the circulated cell NPL Ag 2/97. The circulated HTSPRT NPL 93094 was broken at VNIIM, repaired and became unstable. Therefore, measurements 20 (value in brackets in Table 3) and 22 (value in brackets in Table 4) could not be used for the back comparisons.

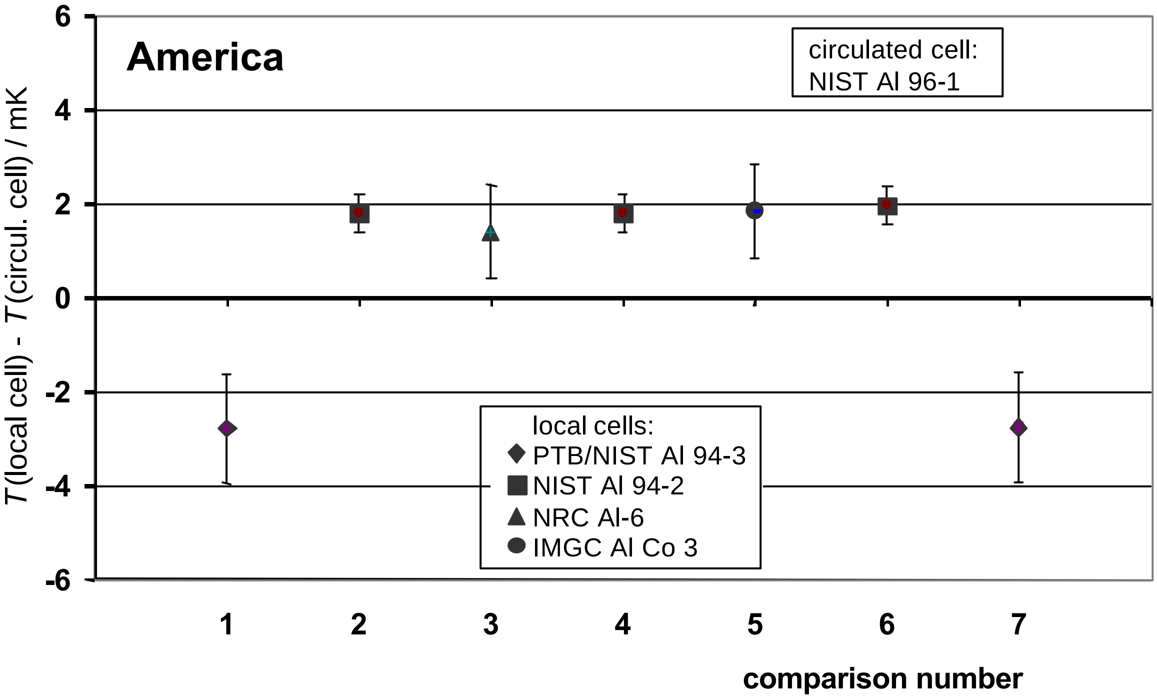

Figure 3: Results of comparisons of local and circulated aluminium fixed-point cells for four standard laboratories in the American subgroup. The error bars indicate the expanded (k=2) uncertainties U_{lab} as given in table 5.

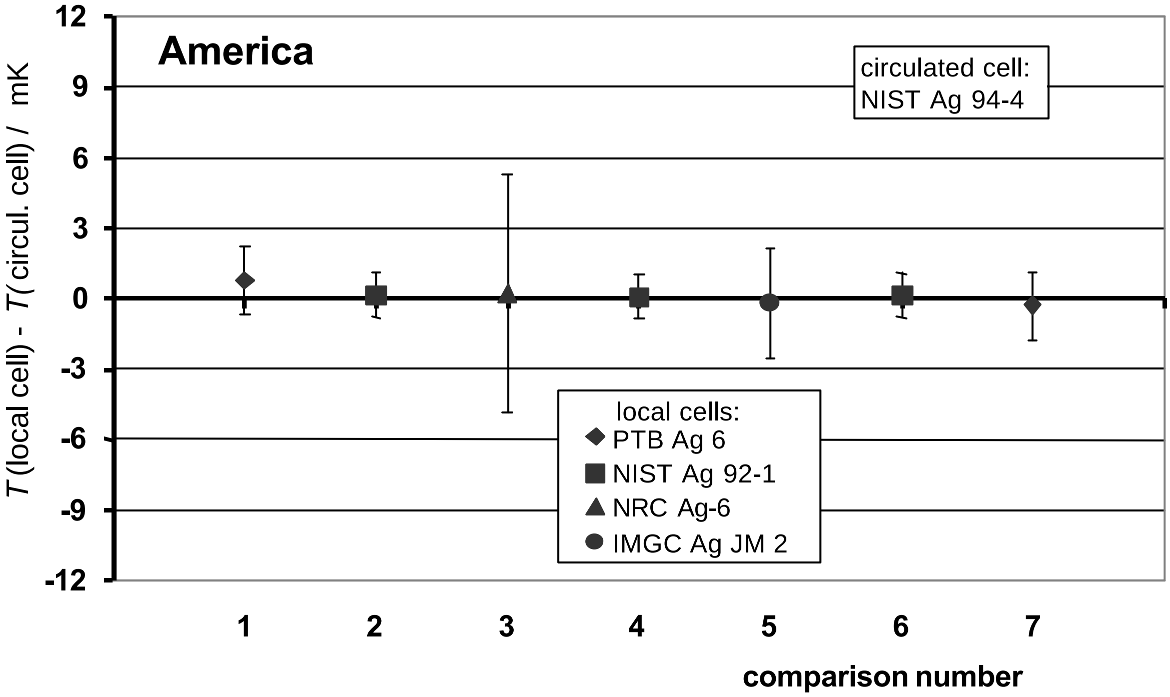

Figure 4: Results of comparisons of local and circulated silver fixed-point cells for four standard laboratories in the American subgroup. The error bars indicate the expanded (k=2) uncertainties U_{lab} as given in table 6.

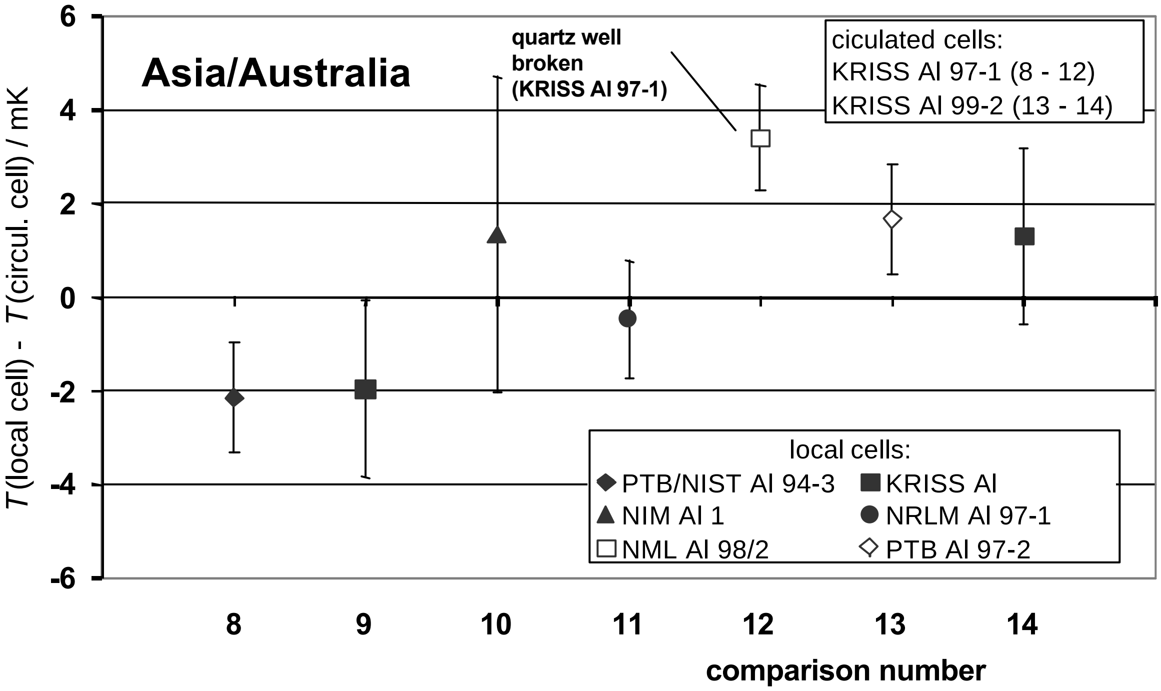

Figure 5: Results of comparisons of local and circulated aluminium fixed-point cells for five standard laboratories in the Asian/Australian subgroup. The error bars indicate the expanded (k=2) uncertainties U_{lab} as given in table 5.

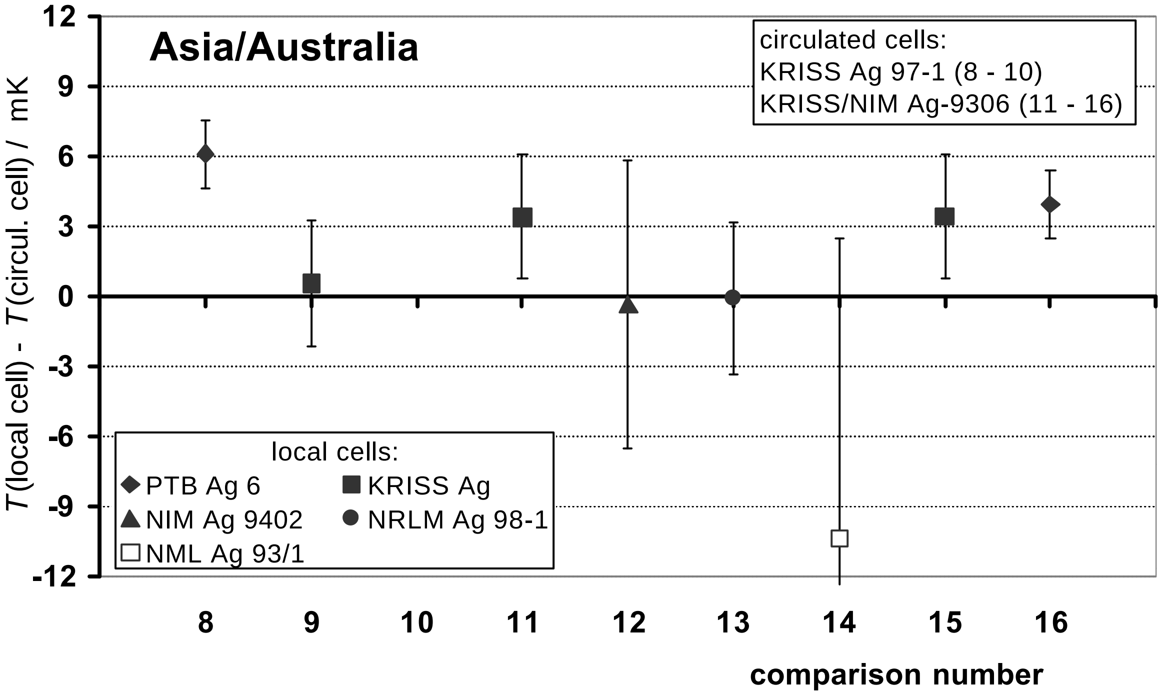

Figure 6: Results of comparisons of local and circulated silver fixed-point cells for five standard laboratories in the Asian/Australian subgroup. The error bars indicate the expanded (k=2) uncertainties U_{lab} as given in table 6.

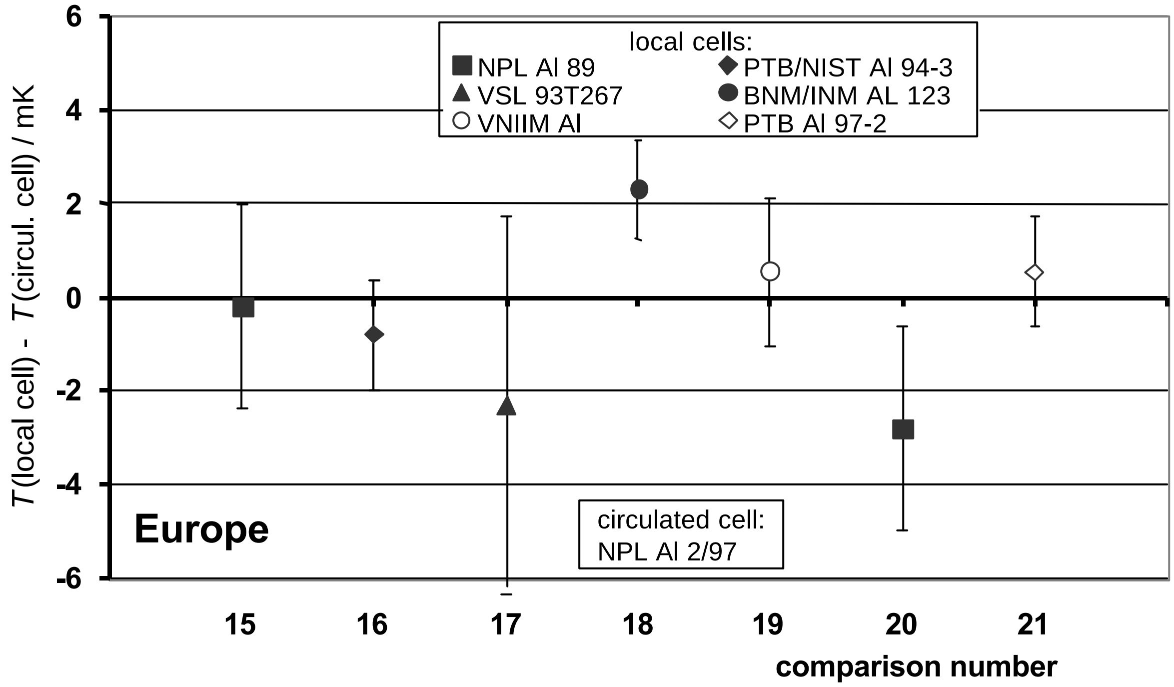

Figure 7: Results of comparisons of local and circulated aluminium fixed-point cells for five standard laboratories in the European subgroup. The error bars indicate the expanded (k=2) uncertainties U_{lab} as given in table 5.

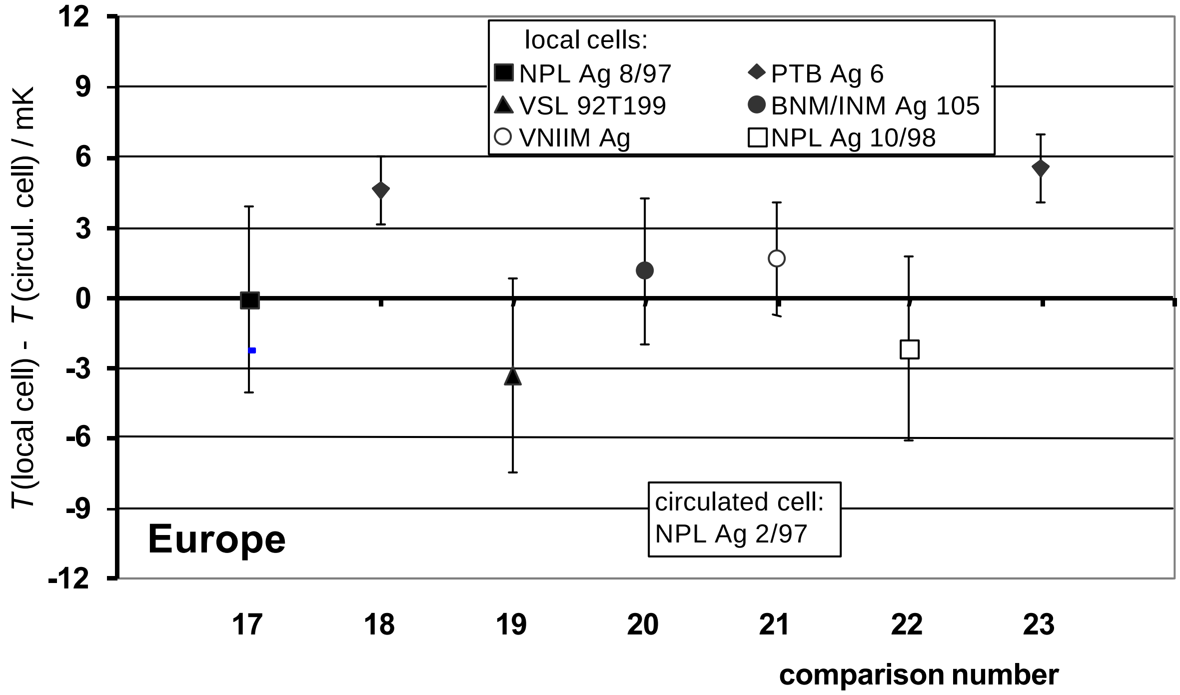

Figure 8: Results of comparisons of local and circulated silver fixed-point cells for five standard laboratories in the European subgroup. The error bars indicate the expanded (k=2) uncertainties U_{lab} as given in table 6.

Table 3: Temperature differences for aluminium local cells of 12 participating standard laboratories to circulated cells; the reference cells of the coordinator/subcoordinators are underlined. The measurements were carried out from January 1998 through August 2000. Values in brackets were not evaluated for comparison results (see 3.2).

fp cell 1 (local)	**fp cell 2 (circulated)**	**Δt (fp cell 1- fp cell 2), in mK**	**date**	**measurement laboratory**	**fracture *contamination***
1. PTB/NIST Al 94-3	NIST Al 96-1	-2.79	1.98	NIST	
2. NIST Al 94-2	NIST Al 96-1	1.80	2.98	NIST	
3. NRC Al-6	NIST Al 96-1	1.35	8.98	NRC	
4. NIST Al 94-2	NIST Al 96-1	1.80	9.98	NIST	
5. IMGC Al Co3	NIST Al 96-1	1.844	1.99	IMGC	
6. NIST Al 94-2	NIST Al 96-1	1.96	7.99	NIST	
7. PTB/NIST Al 94-3	NIST Al 96-1	-2.76	8.99	NIST	
8. PTB/NIST Al 94-3	KRISS Al 97-1	-2.15	5.98	KRISS	
9. KRISS Al	KRISS Al 97-1	-1.95	6.98	KRISS	
10. NIM Al1	KRISS Al 97-1	1.34	7.-10.98	NIM	
11. NRLM Al 97-1	KRISS Al 97-1	-0.32	11.98-2.99	NRLM	
12. NML Al 98/2	KRISS Al 97-1	3.40	2.-6.99	NML	KRISS Al 97-1
13. PTB Al 97-2	KRISS Al 99-2	1.66	11.99	PTB	
14. KRISS Al	KRISS Al 99-2	1.30	8.00	KRISS	
15. NPL Al 89	NPL Al 2/97	-0.20	5.98	NPL	
16. PTB/NIST Al 94-3	NPL Al 2/97	-0.80	6.98	NPL	
17. VSL 93T267	NPL Al 2/97	-2.33	8.-9.98	NMi/VSL	
18. BNM/INM Al 123	NPL Al 2/97	2.30	10.-11.98	BNM/INM	
19. VNIIM Al	NPL Al 2/97	0.55	12.98-1.99	VNIIM	SPRT NPL 93094
20. NPL Al 89	NPL Al 2/97	[-2.80]	4.99	NPL	
21. PTB Al 97-2	NPL Al 2/97	0.54	2.00	PTB	
22. PTB Al 97-2	PTB/NIST Al 94-3	2.87	12.98	PTB	

Table 4: Temperature differences for silver local cells of 12 participating standard laboratories to circulated cells; the reference cells of the coordinator/subcoordinators are underlined. The measurements were carried out from January 1998 through March 2000. Values in brackets were not evaluated for comparison results (see 3.2).

fp cell 1 (local)	**fp cell 2 (circulated)**	***Dt* (fp cell 1- fp cell 2), in mK**	**date**	**measurement laboratory**	**fracture *contamination***
1. PTB Ag 6	NIST Ag 94-4	0.75	1.98	NIST	
2. NIST Ag 92-1	NIST Ag 94-4	0.13	2.98	NIST	
3. NRC Ag-6	NIST Ag 94-4	0.18	8.98	NRC	
4. NIST Ag 92-1	NIST Ag 94-4	0.07	9.98	NIST	
5. IMGC Ag JM2	NIST Ag 94-4	-0.205	1.99	IMGC	
6. NIST Ag 92-1	NIST Ag 94-4	0.12	7.99	NIST	
7. PTB Ag 6	NIST Ag 92-1	-0.42	1.00	NIST	
8. PTB Ag 6	KRISS Ag 97-1	[6.11]	5.98	KRISS	*SPRT PTB N 304*
9. KRISS Ag	KRISS Ag 97-1	[0.57]	5.98	KRISS	
10. NIM Ag	KRISS Ag 97-1	[-]	6.98	NIM	KRISS Ag 97-1
11. KRISS Ag	KRISS/NIM Ag 9306	3.41	9.98	KRISS	
12. NIM Ag 9402	KRISS/NIM Ag 9306	-0.35	10.98	NIM	
13. NRLM Ag 98-1	KRISS/NIM Ag 9306	-0.06	11.98-2.99	NRLM	
14. NML Ag 93/1	KRISS/NIM Ag 9306	-10.36	2.-6.99	NML	
15. KRISS Ag	KRISS/NIM Ag 9306	3.44	9.99	KRISS	
16. PTB Ag 6	KRISS/NIM Ag 9306	3.95	11.99	PTB	
17. NPL Ag 8/97	NPL Ag 2/97	-0.10	5.98	NPL	
18. PTB Ag 6	NPL Ag 2/97	4.60	6.98	NPL	
19. VSL 92T199	NPL Ag 2/97	-3.33	8.-9.98	NMi/VSL	
20. BNM/INM Ag 105	NPL Ag 2/97	1.10	10.-11.98	BNM/INM	
21. VNIIM Ag	NPL Ag 2/97	1.63	12.98-1.99	VNIIM	SPRT NPL 93094
22. NPL Ag 10/98	NPL Ag 2/97	[-2.20]	4.99	NPL	
23. PTB Ag 6	NPL Ag 2/97	5.52	2.-3.00	PTB	

Table 5: Uncertainty component budget expressed in mK for the comparison of the local temperature realisation of the ITS-90 at the aluminium fixed point against a circulated cell.

	BNM-INM	**IMGC**	**KRISS**	**NIM**	**NIST**	**NMi/ VSL**	**NML**	**NPL**	**NRC**	**NRLM**	**PTB**	**VNIIM**
Type A:												
repeatab. of bridge readings u_A	0.25	0.179	0.38	0.74	0.11	1.02	0.284	0.80	0.30	0.17	0.26	0.50
degrees of freedom ν	*7*	*6*	*6*	*2*	*14*	*8*	*3*	*8*	*8*	*11*	*6*	*9*
Type B:												
hydrostatic head	0.01	0.017	0.02	0.231	0.0166	0.009		0.02	0.003	0.01	0.023	0.02
SPRT self-heating	0.06	0.009	0.11	0.06	0.0368		0.022	0.05	0.020	0.02	0.023	0.05
immersion	0.20	0.196	0.08		0.0032	1.12	0.045	0.40	0.010	0.09	0.009	0.10
gas pressure in f.-p. cell	0.10	0.046	0.05	0.08	4.E-05	0.002	0.011	0.04	0.009	0.01	0.081	0.01
chemical impurities	0.38	0.387	0.67	0.40	0.156	0.387	0.334	0.40	0.40	0.41	0.48	0.49
bridge-measurement errors	0.05	0.058	0.03	0.19		0.015	0.036		0.030	0.051		0.03
choice of freezing-point value		0.173	0.10			1.115			0.050			0.05
SPRT leakage effect/drift							0.056			0.36		0.05
temperature drift propag. from TPW		0.003	0.43	0.12		0.162						
Total type B uncertainty u_B	**0.45**	**0.47**	**0.82**	**0.52**	**0.16**	**1.64**	**0.34**	**0.57**	**0.40**	**0.56**	**0.49**	**0.51**
Total expanded uncertainty U	**1.08**	**1.04**	**1.89**	**3.35**	**0.40**	**4.04**	**1.13**	**2.17**	**1.06**	**1.18**	**1.17**	**1.52**

Table 6: Uncertainty component budget expressed in mK for the comparison of the local temperature realisation of the ITS-90 at the silver fixed point against a circulated cell.

	BNM-INM	**IMGC**	**KRISS**	**NIM**	**NIST**	**NMi/ VSL**	**NML**	**NPL**	**NRC**	**NRLM**	**PTB**	**VNIIM**
Type A												
repeatab. of bridge readings u_A	0.70	0.718	0.47	1.40	0.40	1.28	0.388	1.45	0.50	0.70	0.25	0.90
degree of freedom n	*12*	*6*	*6*	*2*	*13*	*8*	*3*	*8*	*8*	*8*	*8*	*9*
Type B												
hydrostatic head	0.03	0.015	0.02	0.03	0.0194	0.031		0.05	0.011	0.02	0.078	0.05
SPRT self-heating	0.06	0.014	0.11	0.09	0.0414	0.001	0.026	0.12	0.020	0.06	0.069	0.05
immersion	0.90	0.289	0.10		0.0108	0.60	0.038	0.87	0.010	0.25	0.031	0.20
gas pressure in f.-p. cell	0.10	0.040	0.05	0.07	3.E-05	0.002	0.013	0.04	0.008	0.01	0.068	0.01
chemical impurities	1.00	0.648	1.10	0.60	0.167	0.648	6.381	0.58	2.40	0.79	0.65	0.55
bridge-measurement errors	0.05	0.058	0.03	0.25		0.012	0.130		0.030	0.081		0.03
choice of freezing-point value		0.577	0.10			1.115			0.050			0.05
SPRT leakage effect/drift							0.638			1.15		0.10
temperature drift propag. from TPW		0.003	0.43	0.17		0.206						
Total type B uncertainty u_B	**1.35**	**0.92**	**1.20**	**0.68**	**0.17**	**1.44**	**6.41**	**1.05**	**2.40**	**1.42**	**0.66**	**0.60**
Total expanded uncertainty U	**3.10**	**2.55**	**2.66**	**6.17**	**0.93**	**4.13**	**12.88**	**3.95**	**4.94**	**3.27**	**1.44**	**2.36**

3.3 Uncertainties

Each laboratory which participated in key comparisons 4 provided resistance ratios *W*, measured in its own, local fixed-point cells and in the circulated cells. Each participant was requested to supply the uncertainty of the temperature difference of the local realisation of the respective fixed-point and the circulated one. The information provided at each fixed point for each participant included the repeatability of the bridge readings for the difference W_{loc} - W_{circ} across multiple freezes, denoted u_A (standard deviation of the mean), the number of freezes reported for computation of mean temperature differences from which the degrees of freedom $\boldsymbol{n}$ associated with u_A could be calculated, and standard uncertainties for any other sources of uncertainty, denoted u_B. These standard uncertainties are mostly of type B and take into account that two cells have been compared in the same laboratory at the same time with the same transfer thermometer. Therefore additional bridge measurement errors like linearity, the choice of the freezing point value, HTSPRT drift effects, and temperature drifts propagated from the triple point of water affected the uncertainty of the comparison only in some cases and only to a minor amount, except in two cases. The specific sources of uncertainties and the associated uncertainty values can be found in table 5 for the comparison of the local realisation of the aluminium fixed point and in table 6 for the comparison of the local realisation of the silver fixed point.

In order to keep the uncertainty evaluation as transparent and simple as possible and to avoid strange effects when using the Welch-Satterthwaite formula as described in the ISO Guide to the Expression of Uncertainty in Measurement we used an approach similar to that described in the report of the CCT key comparison 3. The expanded uncertainties were computed by using a coverage factor $t_{95}(\boldsymbol{n})$ from the t-distribution and the degrees of freedom $\boldsymbol{n}$ only for the uncertainties u_A. For the components u_B it is assumed to have an infinite number of degrees of freedom. Finally the combined expanded (*k*=2) uncertainties *U* were computed by root-sum-of-squares of type A and type B contributions.

For a comparison of a local realisation of the freezing point temperature against a circulated cell in the laboratory *lab1* the following expanded uncertainty is derived considering that the standard uncertainty of the bridge readings in laboratory *lab2* does not contribute to the uncertainty. As there is a circulated cell involved which had been transported to the site of *lab1* one has to account for instabilities of this cell. Therefore, the additional cell uncertainty U_c is added (see 3.4 for details) :

$$U(T_{lab1} - T_{lab2}) = \sqrt{U_{lab1}^2 + U_{lab2}^2 - t_{95}^2(\boldsymbol{n})\, u_{A,lab2}^2 + U_c^2} \tag{1}$$

This is the basic uncertainty equation which would be valid for the comparison of the local realisations of freezing point temperatures if the circulated cell were also the reference cell of *lab2*. To comply with the more complicated paths of the comparison the following equations have to be used. For a comparison within one subgroup using the circulated cell of a subcoordinator *sub* the following expanded uncertainty applies

$$U(T_{lab1} - T_{lab2}) = \sqrt{U^2(T_{lab1} - T_{sub}) + U^2(T_{lab2} - T_{sub}) - 2\,U_{B,sub}^2} \tag{2}$$

In eq. (2) the uncertainties according to eq. (1) are first combined and then adjusted so that the type B uncertainty $U_{B,sub}$ of the subcoordinator, which affects all of the measurements using the circulated cell of the subcoordinator in the same but unknown way is not included in the uncertainty. For comparisons performed not within one subgroup using two circulated cells of the subcoordinators *sub1* and *sub2* and in addition the master circulated cell *mc* to link the two subgroups the following expanded uncertainty is derived accordingly from eq. (1)

$$U(T_{lab1} - T_{lab2}) =$$
$$\sqrt{U^2(T_{lab1} - T_{sub1}) + U^2(T_{mc} - T_{sub1}) + U^2(T_{mc} - T_{sub2}) + U^2(T_{lab2} - T_{sub2}) - 2\,U^2_{B,sub1} - 2\,U^2_{B,mc} - 2\,U^2_{B,sub2}} \tag{3}$$

From these general formulas all bilateral uncertainties can be derived. Please note that this uncertainty analysis provides the general procedures. In some cases, the uncertainty for a particular comparison had to be handled slightly different than that of the general cases described above.

It is worth noting that the method used to combine the various uncertainty components as detailed in equations (2) and (3) accounts for the correlation in the comparison uncertainty values. By subtracting the type B uncertainties of the suppliers of the circulated cells the main influence of correlation is properly processed and it is avoided to make a posteriori corrections. These were necessary when only pairs of the simple comparison uncertainty values for each laboratory of tables 5 and 6 would have been used. When reporting the laboratory-to-laboratory degree of equivalence in the full-bilateral matrix in tables 9 and 10 consequently correlation effects are included.

3.4 Additional Uncertainty of Circulated Cells

In addition to the uncertainties reported by each laboratory for the comparison of the local realisation of the freezing point temperatures against a circulated cell, uncertainties for possible temperature changes in each circulated cell over the course of the comparison have to be included for each bilateral temperature difference and for the temperature difference to the key comparison reference value. Because they affect only differences in measurements made with the circulated cells over time, this uncertainty component is included only in the uncertainties of the temperature differences, and not in the uncertainties of the measurements made in the individual laboratories given in tables 5 and 6. The most evident way to compute the additional expanded uncertainty U_c of the circulated cells is to analyse the data shown in figures 3 to 8. Possible instabilities of the circulated cells can be derived from the temperature differences to the local cell of the coordinator or subcoordinator at the beginning and the end of a loop in that subgroup. The corresponding expanded (*k*=2) uncertainty U_c is

$$U_c = \frac{\left|(T_{loc} - T_{circ})_{beginn} - (T_{loc} - T_{circ})_{end}\right|}{\sqrt{3}} \tag{4}$$

For aluminium from table 3 values can be derived from measurement numbers 2 and 6 in the American subgroup and from measurement numbers 15 and 20 in the European subgroup. For the Asian/Australian subgroup unfortunately a value covering the complete duration of the measurements could not be derived as this loop was not closed. But from the first two measurements 8 and 9 (9 in combination with 13, 14, and 22) a representative estimate could be made for this subgroup. Finally, the mean value of all three subgroups is calculated as a representative and conservative estimate which resulted in U_c(Al) = 1.0 mK for the overall instability of the circulated aluminium freezing point cells.

For silver from table 4 values in the American subgroup can be derived from the pairs of measurements 1 and 7 in combination with 6, or 2 and 6. To include also the influence of the master circulated cell PTB Ag 6 the higher value was taken as a conservative estimate. A similar value could be derived from measurement numbers 18 and 23 in the European subgroup. In the Asian/Australian subgroup measurements 11 and 15 were used to derive the value. Finally, again the mean value of all three subgroups is calculated as a representative and conservative estimate which resulted in U_c(Ag) = 0.4 mK for the overall instability of the circulated silver freezing point cells. It is surprising that the uncertainty for the aluminium circulated cells is higher than that for silver but this reflects the actual situation during the comparison measurements.

An alternate method to calculate U_c uses equation 4 with the Type A uncertainties (Tables 5 and 6) subtracted to remove the repeatability of the NMI's comparison measurements. This approach is motivated by a desire to avoid double counting of the Type A uncertainties in the combined uncertainty of the comparison.

Using the results of the aluminium comparisons in Table 3, the use of measurement numbers 1, 6, and 7 for the American subgroup; measurement numbers 15 and 20, and measurement numbers 16, 21, and 22 for the European subgroup gives three values of the estimated changes in the circulated cells. No estimate is derived for the Asian/Australian subgroup. After subtracting out the Type A uncertainties, the average change gives a value of 0.69 mK for U_c(Al) (k=2). Application of the alternative U_c(Al) (k=2) to column 4 $(u_{lab}^2 + u_c^2)^{1/2}$ of Table 7 would result in an average reduction of combined uncertainties of 9 %. The largest reduction of 26 % would occur to the NIST uncertainty value.

Using the results of the silver comparisons in Table 4, the use of measurement numbers 1, 6, and 7 for the American subgroup gives a representative value of the estimated change in the circulated cells over the duration of the measurements, for all subgroups. After subtracting out the Type A uncertainties, the apparent change gives a value of 0.46 mK for U_c(Ag) (k=2). Application of the alternative U_c(Ag) (k=2) to column 4 $(u_{lab}^2 + u_c^2)^{1/2}$ of Table 8 would result only in insignificant changes of the combined uncertainties.

As this alternate approach results in most cases in similar uncertainties the more simple calculations according to eq. (4) are used for the final evaluation.

3.5 Key Comparison Reference Value

The key comparison reference value KCRV for both the aluminium and the silver fixed points was formed as the mean of the temperature differences of the local cells of the individual participating laboratories to a corresponding master circulated cell.

This method is very straightforward as the master circulated cells are the backbones of the comparison and all laboratory-to-laboratory bilateral temperature differences can only be calculated on the basis of this concept. The procedure is illustrated in table 7 for aluminium and in table 8 for silver. As far as reasonable the average of multiple paths calculations of the differences had been used. Please see the last column of tables 7 and 8 for details. Forming the KCRV in this way and considering finally only the differences T_{lab} - $KCRV$ the temperature T_{mc} of the chosen master circulated cell completely cancels out. Therefore, the choice of the master circulated cell (PTB/NIST Al 94-3 respectively PTB Ag 6) serves only as a mathematical reference and can not influence the result of the key comparison and consequently could be made considering only practical reasons.

The value of the so-defined KCRV must not be related to the ITS-90 value of the corresponding fixed point. The KCRV has no physical meaning and is used only as a notational shorthand for presenting a common baseline against which all laboratory values can be compared. In contrast the weighting procedure has physical relevance. It was found that weighting with the inverse of the quadratic sum of the uncertainties u_{lab} of the participants and of u_c of the circulated cell gave the result with the best degree of equivalence (eq. (5)). Since this weight includes both the laboratory comparison uncertainty u_{lab} as well as the circulated cell uncertainty u_c , there is no danger that a single laboratory with a much smaller uncertainty than the other participants can dominate the evaluation of the KCRV.

$$KCRV = \frac{\sum_{lab} \frac{T_{lab} - T_{mc}}{u_{lab}^2 + u_c^2}}{\sum_{lab} \frac{1}{u_{lab}^2 + u_c^2}} \qquad (5)$$

By definition this KCRV has no uncertainty. The degree of equivalence of the participant's results with the key comparison reference value is assessed by forming the following expanded (k=2) uncertainty $U_{lab,KCRV}$ for the temperature difference T_{lab} - $KCRV$

$$U_{lab,KCRV} = \sqrt{U_{lab}^2 + U_c^2} \qquad (6)$$

In figures 9 and 10 the error bars represent the expanded uncertainties (k=2) of the differences T_{lab} - $KCRV$ as defined in eq. (6).

3.6 Bilateral Equivalence

In order to assess the degree of equivalence between participants bilateral temperature differences were computed based on the results presented in tables 7 and 8. For the aluminium fixed point the differences T_{lab1}-T_{lab2} between the participants (in mK) can be found in table 9, above the diagonal. Their expanded uncertainties (k=2, in mK) calculated with eqs. (1) to (3) are also listed in table 9 above the diagonal, but in italics. The quantifying equivalence factor $QDE_{lab1,lab2}$ as defined in eq. (7) is given below the diagonal.

$$QDE_{lab1,\,lab2} = \left|T_{lab1} - T_{lab2}\right| + \left\{1.645 + 0.3295 \cdot \exp\left(\frac{-4.05 \cdot \left|T_{lab1} - T_{lab2}\right|}{u\left(T_{lab1} - T_{lab2}\right)}\right)\right\} u\left(T_{lab1} - T_{lab2}\right) \quad (7)$$

The quantifying equivalence factor QDE was defined for bilateral comparisons. It combines both the pair difference of measured fixed-point temperatures and the pair uncertainty into a single value. For details see Ref. [1].

The same information for the silver fixed point is given in table 10.

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[1] A.G. Steele, B.M. Wood, and R.J. Douglas: Quantifying equivalence for interlaboratory comparisons of fixed-points, Proceedings of TEMPMEKO 99, Delft 1999, 245-250

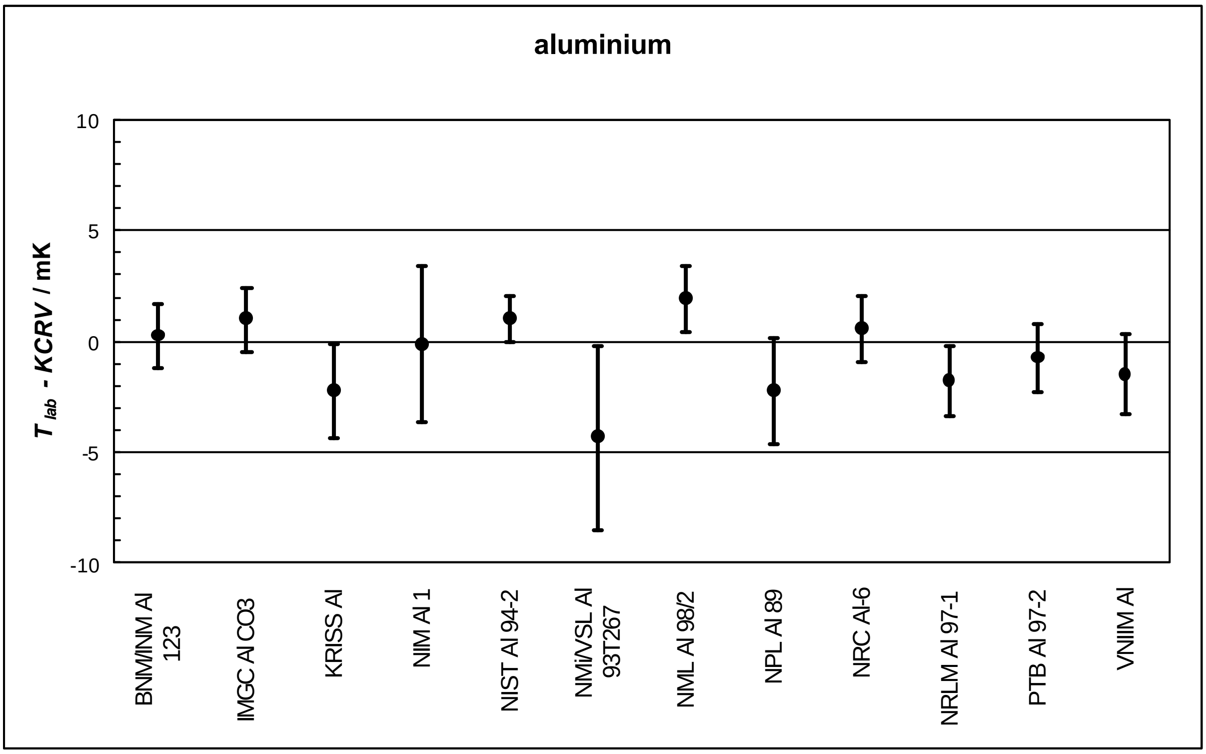


Figure 9 : Differences T_{lab} - *KCRV* for aluminium fixed point cells. The error bars represent the expanded (*k*=2) uncertainties of the differences T_{lab} - *KCRV* as defined in eq. (6).

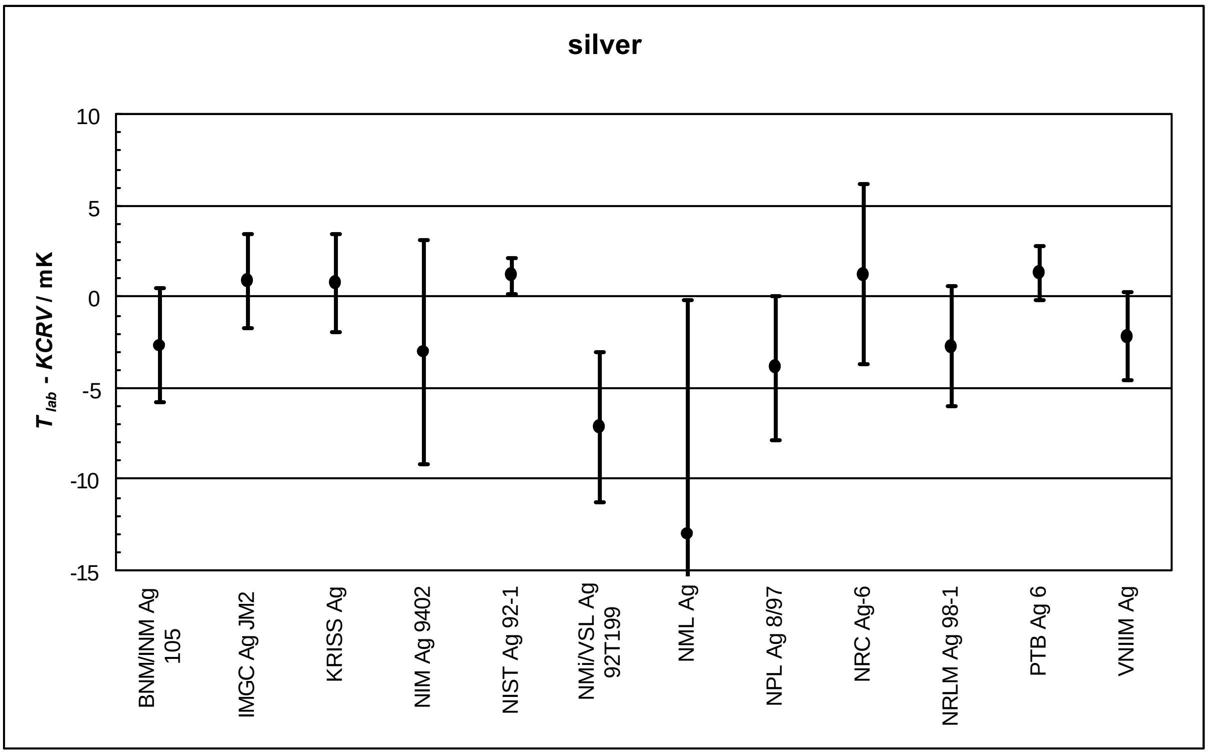


Figure 10 : Differences T_{lab} - *KCRV* for silver fixed point cells. The error bars represent the expanded (*k*=2) uncertainties of the differences T_{lab} - *KCRV* as defined in eq. (6).

Table 7: Differences of aluminium fixed-point temperatures relative to the master circulated cell (PTB/NIST Al 94-3) and to the KCRV; uncertainties, and weight factors; the data were taken from tables 3 and 5.

cell	**T_{lab} - T_{mc} (mK)**	**u_{lab} (mK)**	**$(u_{lab}^2+u_c^2)^{1/2}$ (mK)**	**T_{lab}- KCRV (mK)**	**weight**	**temperature differences contributing to column 2 (comparison number from Table 3)**
BNM/INM Al 123	**3.87**	0.51	0.72	**0.25**	**0.10**	(18) minus average of (16) and ((21)-(22))
IMGC Al Co3	**4.62**	0.50	0.71	**1.00**	**0.10**	(5) minus average of (1) and (7)
KRISS Al	**1.36**	0.90	1.03	**-2.26**	**0.05**	average of ((9) - (8)) and ((22)-(13)+(14))
NIM Al 1	**3.49**	0.90	1.03	**-0.13**	**0.05**	(10) minus (8)
NIST Al 94-2	**4.63**	0.19	0.54	**1.01**	**0.18**	average of (2), (4), and (6); result minus average of (1) and (7)
VSL 93T267	**-0.74**	1.93	1.99	**-4.35**	**0.01**	(17) minus average of (16) and ((21)-(22))
NML Al 98/2	**5.55**	0.44	0.67	**1.93**	**0.12**	(12) minus (8)
NPL Al 89	**1.37**	0.98	1.10	**-2.25**	**0.04**	(15) minus average of (16) and ((21)-(22))
NRC Al-6	**4.18**	0.50	0.71	**0.56**	**0.10**	(3) minus average of (1) and (7)
NRLM Al 97-1	**1.83**	0.61	0.79	**-1.79**	**0.08**	(11) minus (8)
PTB Al 97-2	**2.87**	0.55	0.75	**-0.75**	**0.09**	(22)
VNIIM Al	**2.12**	0.71	0.87	**-1.50**	**0.07**	(19) minus average of (16) and ((21)-(22))

Table 8: Differences of silver fixed-point temperatures relative to the master circulated cell (PTB Ag 6) and to the KCRV, uncertainties, and weight factors; the data were taken from tables 4 and 6.

cell	T_{lab} - T_{mc} (mK)	u_{lab} (mK)	$(u_{\mathrm{lab}}^2+u_c^2)^{1/2}$ (mK)	T_{lab}-*KCRV* (mK)	weight	temperature differences contributing to column 2 (comparison number from Table 4)
BNM/INM Ag 105	**-3.96**	1.52	1.53	**-2.69**	**0.04**	(20) minus average of (18) and (23)
IMGC Ag JM2	**-0.42**	1.17	1.18	**0.85**	**0.07**	(7) plus average of (2), (4) and (6); average result with (1); (5) minus result
KRISS Ag	**-0.53**	1.29	1.30	**0.74**	**0.06**	average of (11) and (15); result minus (16)
NIM Ag 9402	**-4.30**	1.56	1.57	**-3.03**	**0.04**	(12) minus (16)
NIST Ag 92-1	**-0.11**	0.43	0.48	**1.16**	**0.42**	average of (2), (4) and (6) minus (1); average result with - (7)
VSL 92T199	**-8.39**	1.93	1.94	**-7.12**	**0.03**	(19) minus average of (18) and (23)
NML Ag	**-14.3**	6.42	6.42	**-13.03**	**0.00**	(14) minus (16)
NPL Ag 8/97	**-5.16**	1.79	1.80	**-3.89**	**0.03**	(17) minus average of (18) and (23)
NRC Ag-6	**-0.04**	2.45	2.46	**1.23**	**0.02**	(7) plus average of (2), (4) and (6); average result with (1); (3) minus result
NRLM Ag 98-1	**-4.01**	1.58	1.60	**-2.74**	**0.04**	(13) minus (16)
PTB Ag 6	**0**	0.71	0.73	**1.27**	**0.18**	...
VNIIM Ag	**-3.43**	1.08	1.10	**-2.16**	**0.08**	(21) minus average of (18) and (23)

Table 9: Results of CCT-K4 for the aluminium fixed point; the differences T_{lab1}-T_{lab2} between the participants (in mK, above diagonal), their expanded uncertainty (*k*=2, in mK, above diagonal, in italics), and the $QDE_{lab1,lab2}$ (in mK, below diagonal).

lab2 ⇒ lab1 ⇓	BNM/ INM	IMGC	KRISS	NIM	NIST	NMi/ VSL	NML	NPL	NRC	NRLM	PTB	VNIIM
BNM/INM		-0.75 *3.12*	2.51 *3.44*	0.38 *4.54*	-0.76 *2.78*	4.60 *4.41*	-1.69 *3.27*	2.50 *1.86*	-0.31 *3.12*	2.04 *3.31*	1.00 *2.99*	1.75 *2.34*
IMGC	3.6		3.26 *2.77*	1.13 *4.15*	-0.01 *1.48*	5.35 *4.99*	-0.93 *2.70*	3.25 *2.97*	0.44 *2.05*	2.79 *2.75*	1.75 *2.11*	2.50 *3.30*
KRISS	5.5	5.6		-2.14 *3.86*	-3.27 *2.39*	2.09 *5.13*	-4.20 *2.23*	-0.01 *3.22*	-2.82 *2.78*	-0.48 *2.29*	-1.52 *2.35*	-0.76 *3.51*
NIM	4.7	4.9	5.5		-1.14 *3.90*	4.23 *5.98*	-2.06 *3.81*	2.13 *4.45*	-0.69 *4.15*	1.66 *3.84*	0.62 *3.88*	1.38 *4.67*
NIST	3.3	1.5	5.3	4.7		5.36 *4.78*	-0.92 *2.30*	3.26 *2.62*	0.45 *1.49*	2.80 *2.36*	1.76 *1.57*	2.51 *2.98*
NMi/VSL	8.3	9.5	6.7	9.4	9.4		-6.29 *5.09*	-2.10 *4.31*	-4.91 *4.99*	-2.57 *5.11*	-3.61 *4.67*	-2.85 *4.54*
NML	4.6	3.4	6.0	5.4	3.0	10.5		4.19 *3.56*	1.38 *2.71*	3.72 *2.20*	2.68 *2.26*	3.44 *3.45*
NPL	4.0	5.8	3.2	6.1	5.5	5.9	7.2		-2.81 *2.98*	-0.47 *3.18*	-1.51 *2.58*	-0.75 *2.15*
NRC	3.3	2.3	5.2	4.6	1.8	9.1	3.8	5.3		2.35 *2.76*	1.31 *2.12*	2.06 *3.30*
NRLM	4.9	5.1	2.6	5.1	4.8	7.1	5.5	3.5	3.1		-1.04 *2.32*	-0.29 *3.49*
PTB	3.7	3.5	3.6	4.3	3.1	7.6	4.6	3.8	3.1	3.1		0.76 *2.95*
VNIIM	3.8	5.3	4.0	5.6	5.1	6.8	6.3	2.7	3.9	3.6	3.5	

Table 10: Results of CCT-K4 for the silver fixed point; the differences T_{lab1}-T_{lab2} between the participants (in mK, above diagonal), their expanded uncertainty (k=2, in mK, above diagonal, in italics), and the $QDE_{lab1,lab2}$ (in mK, below diagonal).

lab2 ⇒ lab1 ⇓	BNM/ INM	IMGC	KRISS	NIM	NIST	NMi/ VSL	NML	NPL	NRC	NRLM	PTB	VNIIM
BNM/INM		-3.54	-3.44	0.34	-3.85	4.43	10.35	1.20	-3.92	0.05	-3.96	-0.53
		4.22	*4.07*	*7.00*	*3.36*	*5.19*	*13.30*	*3.77*	*5.98*	*4.65*	*3.47*	*3.94*
IMGC	7.1		0.10	3.88	-0.31	7.97	13.89	4.74	-0.39	3.59	-0.42	3.01
			3.71	*6.80*	*2.60*	*5.02*	*13.19*	*3.53*	*5.58*	*4.35*	*3.05*	*3.71*
KRISS	6.9	3.7		3.78	-0.41	7.87	13.79	4.64	-0.49	3.49	-0.53	2.91
				6.63	*2.70*	*4.89*	*13.11*	*3.34*	*5.63*	*4.07*	*2.83*	*3.53*
NIM	7.1	9.8	9.6		-4.19	4.09	10.01	0.86	-4.26	-0.29	-4.30	-0.87
					6.31	*7.51*	*14.29*	*6.61*	*8.01*	*7.01*	*6.36*	*6.71*
NIST	6.7	2.8	2.9	9.6		8.28	14.20	5.05	-0.07	3.90	-0.11	3.32
						4.33	*12.94*	*2.43*	*4.96*	*3.52*	*1.66*	*2.69*
NMi/VSL	8.9	12.1	11.9	10.7	11.9		5.92	-3.23	-8.35	-4.38	-8.39	-4.96
							13.57	*4.65*	*6.57*	*5.39*	*4.41*	*4.79*
NML	21.7	25.0	24.8	22.3	25.1	18.0		-9.15	-14.27	-10.30	-14.31	-10.88
								13.20	*13.86*	*13.30*	*12.97*	*13.14*
NPL	4.6	7.7	7.4	7.1	7.1	7.2	20.5		-5.12	-1.15	-5.16	-1.73
									5.51	*4.03*	*2.58*	*3.19*
NRC	9.1	5.8	5.9	11.3	5.0	13.8	26.0	9.8		3.97	-0.04	3.39
										6.06	*5.21*	*5.63*
NRLM	4.6	7.3	7.0	7.1	6.9	9.0	21.7	4.8	9.2		-4.01	-0.58
											3.62	*4.19*
PTB	6.9	3.3	3.2	9.8	1.7	12.0	25.2	7.3	5.2	7.0		3.43
												2.82
VNIIM	4.3	6.2	5.9	7.2	5.6	9.0	22.1	4.5	8.3	4.5	5.8	

4 Discussion

The practice during the measurements for the K4 led to valuable information for improvements in future comparisons. Some of the obtained results of this project are not fully satisfying and probably did not meet expectations. The reasons for this were partly time limitations during the measurements and/or an underestimation of some uncertainty contributions. The analysis of the results will help the participants to reconsider their uncertainty budgets and to understand better the physics involved in the comparison.

The present key comparison has further demonstrated that it will be necessary for a future K4 to prepare an even more detailed and precise protocol, which then has to be strictly followed by future participants. This will finally facilitate the evaluation of the data of all participating standard laboratories. In the present KC some problems have been reported due to delayed submissions of the participant's final results. It would be very helpful for a future KC if a certain time limit for submission of the final data would be strictly followed. In particular, a future protocol should contain a detailed description how the uncertainties are to be calculated. Again, the key comparison will stimulate the development of methods for better harmonisation of the uncertainty calculations.

The bilateral procedure of the comparisons in the American subgroup was certainly exemplary for the present and future key comparisons. Before and after the measurements at NRC and IMGC, a comparison was carried out at NIST. Thus, an easy drift control for the SPRT and the fixed-point cells could be achieved. Both the European and the Asian/Australian subgroups applied round-robin techniques where a drift control for the circulated fixed-point cells was considerably more difficult and less accurate. However, a suitable cut of the subgroups for a future K4 and more time would allow an application of such a bilateral comparison procedure in all subgroups.

Comparisons before and after the subgroup loops were carried out by the coordinator for each of the subcoordinator's circulated fixed-point cells with one exception, the KRISS circulated cell KRISS Al 97-1, which broke at the end of the Australian comparison measurements at NML. Unfortunately, there is only limited information available about a possible drift of this fixed-point cell. This was taken into account in the estimation of the corresponding uncertainties.

As for the evaluation of the data and the calculation of the uncertainties an attempt was made to find a balance between an easily understandable and transparent presentation of the results and more sophisticated and extremely accurate studies.

5 Appendix

5.1 Protocol of CCT Key Comparison K4

Berlin, June 11, 1997

After the change of coordinators, it seems to be necessary to use the second half of 1997 to get ready for the key comparison number 4. This is the complete schedule of the key comparison number 4. You are certainly requested to submit any changes by July 15, 1997. All statements in this protocol in italics are not mandatory. We have already lost some time in the preparation.

Therefore, we should use the following months to prepare the equipment for the first round of measurements. All sub-coordinator laboratories must prepare (if not available):

1. One sealed aluminium fixed-point cell (reference cell), with an O.D. of approximately 48 mm. The aluminium fixed-point cell has to fit in an Inconel container of 2" O.D. It is necessary to add up to 13 Inconel discs (thermal shunts) on top of the fixed-point cell, which are kept at a 10 mm distance by means of quartz spacers. From the top disc to the top of the Inconel container *washed* Fiberfrax is filled in. *The immersion depth has to be high enough to achieve a reasonable hydrostatic head.*
2. One sealed silver fixed-point cell (reference cell); same conditions as for the aluminium fixed-point cell.
3. One long stem 0.25 high temperature SPRT; it has to be stable during these intense measurements (short time stability). *Suggested short time stability, approximately 0.1 mK. at the TPW .*

B. Travelling to the sub-coordinators begins in January 1998 to compare the aluminium and silver reference fixed-point cells with the PTB standards. I have planned the following visits:

1. NIST	Jan. 10 through Jan. 25, 1998
2. KRISS	Feb. 20 through March 8, 1998
3. NPL	April 1 through April 16, 1998

After this "authorisation", the following schedule applies for the sub-coordinators and all other participants, who transport the aluminium reference fixed-point cell, the silver reference fixed-point cell, and the stable 0.25 high temperature SPRT to the next participating laboratory.

<u>American group/NIST</u>

NIST to NRC,	March 1, 1998
NRC to NIST,	April 15, 1998
NIST to IMGC	June 15, 1998
IMGC to NIST	August 1, 1998

<u>East Asian / Australian group/KRISS</u>

KRISS to CSIRO	April 15, 1998
CSIRO to NRLM	July 1, 1998
NRLM to NIM	August 15, 1998

NIM to KRISS	October 1, 1998

European group/NPL

NPL to BNM/INM	May 1, 1998
BNM/INM to NMi/VSL	June 15, 1998
NMi/VSL to VNIIM	August 1, 1998
VNIIM to NPL	September 15, 1998

According to Piero Marcarino's own wish, he will take part in the American group, in June/July next year, as he told me today. Richard Rusby and Billy Mangum also accept this solution, which makes the size of the groups similar.
Please let me know if this schedule is in conflict with anybody's summer vacation or conferences.

C. Experiments

Two aluminium and two silver furnaces, each pair of the same design, have to be used in all participating laboratories. If you are familiar with the following procedure please forgive me. The local fixed-point cell is in one furnace, the circulated reference cell in the other furnace. All participant's fixed-point cells and the reference fixed-point cells must certainly be compared in the participant's furnaces. The reason for this comparison is not to determine the temperature differences between fixed-point cells, it is to determine the differences between the participant's realisation of the respective fixed-point temperature.

1. a) Measure R (TPW) with the circulated SPRT.
    b) Insert the SPRT in your annealing furnace which is preheated to 500 °C.
    c) Heat the annealing furnace to 675 °C in approximately 1 h.
    d) Anneal the SPRT for 30 minutes.
    e) Lower the temperature of the annealing furnace to 500 °C in approximately 4 hours.
    f) Repeat the measurement of *R*(TPW).
    g) If the difference between *R*(TPW) from step a) to *R*(TPW) from step f) is > 0.2 mK, go through the loop b) - f) again.
    h) If □*R*(TPW) < 0.2 mK, start with step b) and skip the steps e) through h).
    i) Parallel to the above procedure start to heat the aluminium furnace to 2.5 °C above the melting temperature. After stabilisation start the freeze. Thirty minutes after the start of recalescence, quickly but safely remove the SPRT from the annealing furnace and insert it into the ready aluminium furnace.
    j) Measure *R*(FP-Al1) in furnace 1.
    k) The second furnace has to be prepared so that the recalescence has started 30 minutes before the end of step j).
    l) Quickly remove the SPRT from furnace 1 and insert it into furnace 2; measure *R*(FP-Al2)
    m) Carry out steps j) and l) three times without delay.
    n) At the end reinsert the SPRT into the annealing furnace, anneal the SPRT for 30 minutes and then cool down to 500 °C within approximately 4 hours.
    o) Measure *R*(TPW).
2. This schedule has to be repeated three times.

The comparison of the local silver cell with the reference silver cell is identical to the above, except for the annealing temperature (975 °C), the heating rate (max. 500 °C/h), and the fixed-point temperature of 961.78 °C.

D. Report of results

The following data should be sent by all participants to their sub-coordinators, who are supposed to send all data together with a comprehension to the coordinator by November 15, 1998.
Aluminium and silver freezing-point comparisons; the resistance's are corrected for hydrostatic head and gas pressure.

R (TPW)	R (TPW)
R1 (FP-Al1), R1 (FP-Al2)	R1 (FP-Ag1), R1 (FP-Ag2)
R2 (FP-Al1), R2 (FP-Al2)	R2 (FP-Ag1), R2 (FP-Ag2)
R3 (FP-Al1), R3 (FP-Al2)	R3 (FP-Ag1), R3 (TP-Ag2)
R (TPW)	R (TPW)
R (TPW)	R (TPW)
R4 (FP-Al1), R4 (FP-Al2)	R4 (FP-Ag1), R4 (FP-Ag2)
R5 (FP-Al1), R5 (FP-Al2)	R5 (FP-Ag1), R5 (FP-Ag2)
R6 (FP-Al1), R6 (FP-Al2)	R6 (FP-Ag1), R6 (FP-Ag2)
R (TPW)	R (TPW)
R (TPW)	R (TPW)
R7 (FP-Al1), R7 (TP-Al2)	R7 (FP-Ag1), R7 (FP-Ag2)
R8 (FP-Al1), R8 (TP-Al2)	R8 (FP-Ag1), R8 (FP-Ag2)
R9 (FP-Al1), R9 (TP-Al2)	R9 (FP-Ag1), R9 (FP-Ag2)
R(TPW)	R (TPW)

all W's
Hydrostatic head correction for the compared Al and Ag fixed-point cells, including the TPW cell; *Freeze curves of all Al and Ag fixed-point cells with a duration of 10 hours**.
Wlocal cell/Wreference cell

E. Report of uncertainty in measurement

Please calculate the uncertainty budget for the ratios Wlocal cell/Wreference cell. Uncertainties are determined according to "Guide to the Expression of Uncertainty in Measurement, ISO 1993, ISBN 92-67-10188-9". Please list the values of all components of uncertainties in measurement and send them to the pilot lab together with the results in chapter D.

5.2 Original Comparison Data from all Participants of K4

5.2.1 BNM/INM

local fixed-point cells : BNM/INM Al 123, BNM/INM Ag 105
circulated fixed-point cells : NPL Al 2/97, NPL Ag 2/97

BNM/INM Ag 105

$W_{\mathrm{NPL\ Ag\ 2/97}}$	$W_{\mathrm{BNM/INM\ Ag105}}$	$W_{\mathrm{NPL\ Ag\ 2/97}}$ $-W_{\mathrm{BNM/INM\ Ag105}}$	**HTSPRT number**	mean W_{NPL}- $W_{\mathrm{BNM/INM}}$	$t_{\mathrm{NPL\ Ag\ 2/97}}$ - $t_{\mathrm{BNM/INM\ Ag\ 105}}$
4,286 288 2	4,286 279 3	8,82E-06	94821	-3,13E-06	-1,1 mK
4,286 662 2	4,286 667 0	-4,88E-06	93904		
4,286 279 9	4,286 289 8	-9,91E-06	94821		
4,286 635 3	4,286 641 2	-5,87E-06	93904		
4,286 187 4	4,286 175 1	1,23E-05	94821		
4,286 645 4	4,286 654 0	-8,61E-06	93904		
4,286 644 2	4,286 647 2	-3,00E-06	93904		
4,286 543 4	4,286 541 8	1,62E-06	95013		
4,286 656 5	4,286 660 4	-3,87E-06	93904		
4,286 538 6	4,286 544 4	-5,82E-06	95013		
4,286 631 3	4,286 645 1	-1,39E-05	93904		
4,286 558 5	4,286 559 3	-8,12E-07	95013		
4,286 629 1	4,286 635 9	-6,73E-06	93904		

BNM/INM Al 123

$W_{\mathrm{NPL\ Al\ 2/97}}$	$W_{\mathrm{BNM/INM\ Al\ 123}}$	$W_{\mathrm{NPL\ Al\ 2/97}}$ $-W_{\mathrm{BNM/INM\ Al\ 123}}$	**HTSPRT number**	mean W_{NPL} - $W_{\mathrm{BNM/INM}}$	$t_{\mathrm{NPL\ Al\ 2/97}}$ - $t_{\mathrm{BNM/INM\ Al123}}$
3,376 202 57	3,376 206 37	-3,800 9E-06	93094	-7,426 6E-06	-2,3 mK
3,375 941 40	3,375 950 22	-8.820 0E-06	94821		
3,376 205 28	3,376 213 28	-8.001 8E-06	93094		
3,375 950 83	3,375 959 14	-8.308 7E-06	94821		
3,375 947 25	3,375 956 20	-8.947 9E-06	94821		
3,376 201 28	3,376 210 08	-8.802 0E-06	93094		
3,375 951 32	3,375 956 05	-4.729 59E-06	94821		
3,376 201 85	3,376 209 85	-8.001 83E-06	93094		

5.2.2 IMGC

local fixed-point cells : IMGC Ag JM2, IMGC Al Co3
circulated Fixed-point cells : NIST Ag 94-4, NIST Al 96-1

$t_{Ag(JM2)} - t_{Ag(94-4)}$ / mK

	1st plateau	2nd plateau	3rd plateau
1st run	-0,375	0,451	-0,144
2nd run	-0,660	-1,892	
3rd run	0,435	0,814	
mean	-0,200	-0,209	-0,144
Mean	**-0,205**		
Std. Dev.	**0,996**		
Std. Dev. of the mean	**0,407**		

$t_{Al(Co3)} - t_{Al(96-1)}$ / mK

	1st plateau	2nd plateau	3rd plateau
1st run	1,757	1,140	3,082
2nd run	2,019	2,061	
3rd run	1,678	2,407	
mean	1,818	1,869	3,082
Mean	**1,844**		
Std. Dev.	**0,430**		
Std. Dev. of the mean	**0,176**		

5.2.3 KRISS

circulated fixed-point cells : KRISS Ag 97-1 (Ag_K), KRISS/NIM Ag 9306 (Ag_N), KRISS Al 97-1 (Al)

Table: Key comparison 4 (K4) results for Asia-Australia standard laboratories.

	Temperature differen-		**ces**	**(mK)**
FP cell difference	**KRISS**	**NIM**	**NRLM**	**NML**
Ag (local)-Ag_K (circulat-ing)	0.57	8.72		
Ag (local)-Ag_N (circulat-ing)	3.41	9.07*	-0.06	-10.36
Al (local)-Al (circulating)	-1.95	1.34	-0.48	3.4
* Ag_N (local)-Ag_K (circu-lating)				
Fixed -points	**Standard**	**combined**	**uncertainty**	**(k=1)**
	KRISS	**NIM**	**NRLM**	**NML**
KRISS/NIMAg 9306	1.98	1.60	1.58	1.81
KRISS Al 97-1	1.70	0.92	0.61	1.04

Additional PTB/KRISS results:

date	fp cell 1	fp cell 2	Δt (fp cell 1 -fp cell 2), mK
Al:			
05.1998	PTB/NIST Al 9403	KRISS Al 97-1	-2.15
11.1998	PTB Al 97-2	KRISS Al 99-2	1.66
08.2000	KRISS Al	KRISS Al 99-2	1.30
Ag:			
05.1998	PTB Ag 6	KRISS Ag 97-1	6.11
11.1999	PTB Ag 6	KRISS/NIM Ag 9306	3.95
09.1999	KRISS Ag	KRISS/NIM Ag 9306	3.44

Note : the NRLM value for aluminium was changed to -0.32 mK after recalculation

5.2.4 NIM

local cells : NIM Al (Al1), NIM Ag [Ag1(No.4)]
circulated cell : KRISS Al 97-1 (Al2), KRISS NIM Ag9306 (Ag2)

Table 2. Aluminum cells comparison between NIM cell and circulating cell
thermometer: No.91400, Bridge model: 9975

Rtpw (Ω)	R_{Al1} (NIM cell) (Ω)	R_{Al2} (circulating cell) (Ω)		W_{Al1} (NIM cell)	W_{Al2} (circulating cell)	DT(mK) (W_{NIM}-Wcirculating)
0.27999887						
	0.94525040	0.94524937		3.3759022	3.3758985	
	0.94525040	0.94524917		3.3759022	3.3758978	
	0.94525040	0.94524917		3.3759022	3.3758978	
0.27999982				**3.3759022**	**3.3758980**	**1.29**
0.27999977						
	0.94525330	0.94525247		3.3759062	3.3759032	
	0.94525330	0.94525197		3.3759062	3.3759015	
	0.94525330	0.94525197		3.3759062	3.3759015	
0.27999997				3.3759062	**3.3759021**	**1.29**
0.27999997						
	0.94525420	0.94525287		3.3759046	3.3758999	
	0.94525410	0.94525287		3.3759042	3.3758999	
	0.94525420	0.94525287		3.3759046	3.3758999	
0.28000057				**3.3759045**	**3.3758999**	**1.44**
						Average **1.34**

Freezing curves of both NIM and circulating Aluminum fixed point cells are shown in Fig.2 and Fig.3, respectively.

Table 3. Silver cells comparison data between cell No.4 and cell No.9306
thermometer: No.91400, Bridge model: F18

Rtpw Ω	RAg1(No.4) Ω	RAg2(9306) Ω		WAg1(No.4)	WAg2(9306)	DT(mK) 9306-No.4
0.2800067						
	1.2001913	1.2002000		4.2862935	4.2863245	
	1.2001912	1.2001992		4.2862931	4.2863217	
	1.2001914	1.2001979		4.2862938	4.2863170	
0.2800069				**4.2862935**	**4.2863211**	9.72
0.2800072						
	1.2001957	1.2002032		4.2862939	4.2863207	
	1.2001957	1.2002024		4.2862939	4.2863178	
	1.2001954	1.2002026		4.2862928	4.2863185	
0.2800084				**4.2862935**	**4.2863190**	8.97
0.2800084						
	1.2001982	1.2002052		4.2862890	4.2863140	
	1.2001986	1.2002054		4.2862905	4.2863147	
	1.2001989	1.2002054		4.2862915	4.2863147	
0.2800090				**4.2862903**	**4.2863145**	**8.51**
						Average 9.07

Table 4. Silver cells comparison data between cell No.4 and cell No.9402
thermometer: No.91400, Bridge model: F18

Rtpw Ω	RAg1(No.4) Ω	RAg2(9402) Ω		WAg1(No.4)	WAg2(9402)	DT(mK) 9402-No.4
0.2800015						
	1.2001612	1.2001675		4.2862587	4.2862812	
	1.2001603	1.2001677		4.2862555	4.2862819	
	1.2001597	1.2001686		4.2862533	4.2862851	
0.2800026				**4.2862559**	**4.2862827**	9.47
0.2800029						
	1.2001700	1.2001764		4.2862687	4.2862915	
	1.2001700	1.2001769		4.2862687	4.2862933	
	1.2001694	1.2001764		4.2862665	4.2862915	
0.2800040				**4.2862680**	**4.2862921**	**8.51**
0.2800051						
	1.2001823	1.2001889		4.2862713	4.2862948	
	1.2001822	1.2001888		4.2862709	4.2862945	
	1.2001822	1.2001885		4.2862709	4.2862934	
0.2800072				**4.2862710**	**4.2862943**	**8.17**
						Average 8.72

5.2.5 NIST

NIST Al 94-3 is identical to PTB/NIST Al 94-3
aluminium; circulated cell : NIST Al 96-1; local cells : (NIST Al 94-3), NIST Al 94-2

The first of each three W-measurements have been averaged to Δt.

thermometer s/n RS87A-5

date	W NIST Al 96-1	W NIST Al 94-3	Δt (Al 96-1 - Al 94-3), mK	aver. Δt (Al 96-1 - Al 94-3), mK	s.d., mK
Jan. 1998	3.375 729 39	3.375 720 67	2.72	2.79	0.36
	3.375 729 23	3.375 719 69	2.98		
	3.375 728 90	3.375 719 63	2.89		
	3.375 729 03	3.375 721 15	2.46		
	3.375 729 14	3.375 720 80	2.60		
	3.375 728 76	3.375 721 07	2.40		
	3.375 730 75	3.375 720 59	3.17		
	3.375 728 46	3.375 720 91	2.36		
	3.375 729 54	3.375 720 68	2.77		

thermometer s/n RS87A-5

date	W NIST Al 96-1	W NIST Al 94-2	Δt (Al 96-1 - Al 94-2),mK	aver. Δt (Al 96-1 - Al 94-2), mK	s.d., mK
Feb. 1998	3,375 724 18	3,375 730 33	-1,92	-1,80	0,10
	3,375 724 11	3,375 730 21	-1,91		
	3,375 724 08	3,375 729 97	-1,84		
	3,375 723 82	3,375 729 39	-1,74		
	3,375 723 51	3,375 729 14	-1,76		
	3,375 723 34	3,375 728 97	-1,76		
	3,375 723 88	3,375 729 47	-1,75		
	3,375 723 69	3,375 729 28	-1,75		
	3,375 723 43	3,375 729 11	-1,78		

thermometer s/n 1005

date	W NIST Al 96-1	W NIST Al 94-2	Δt (Al 96-1 -Al 94-2), mK	aver. Δt (Al 96-1 - Al 94-2), mK	s.d., mK
Sept. 1998	3,376 171 60	3,376 177 19	-1,74	-1.80	0.11
	3,376 171 25	3,376 177 11	-1,83		
	3,376 170 95	3,376 177 08	-1,92		
	3,376 171 17	3,376 177 33	-1,92		
	3,376 170 92	3,376 177 15	-1,95		
	3,376 170 75	3,376 177 05	-1,97		
	3,376 171 65	3,376 177 18	-1,73		
	3,376 171 53	3,376 177 12	-1,75		
	3,376 171 41	3,376 177 15	-1,79		

thermometer s/n 1025

date	W NIST Al 96-1	W NIST Al 94-2	Δt (Al 96-1 - Al 94-2),mK	aver. Δt (Al 96-1 - Al 94-2), mK	s.d., mK
Jul.1999	3.376 145 12	3.376 151 30	-1.93	-1.96	0.11
	3.376 144 88	3.376 151 17	-1.97		
	3.376 144 65	3.376 151 07	-2.01		
	3.376 145 35	3.376151 35	-1.87		
	3.376 145 24	3.376 151 24	-1.87		
	3.376 145 24	3.376 151 15	-1.85		
	3.376 146 21	3.376 152 91	-2.09		
	3.376 146 16	3.376 152 77	-2.07		
	3.376 145 83	3.376 152 63	-2.12		

thermometer s/n RS87A-5

date	W NIST Al 96-1	W NIST Al 94-3	Δt (Al 96-1- Al 94-3), mK	aver. Δt (Al 96-1 - Al 94-3), mK	s.d., mK
Aug. 1999	3.375 729 33	3,375 720 74	2,68	2,76	0,08
	3.375 729 33	3,375 720 52	2,75		
	3.375 729 13	3,375 720 20	2,79		
	3.375 729 17	3,375 720 29	2,77		
	3.375 729 09	3,375 720 04	2,82		
	3.375 728 97	3,375 719 90	2,83		
	3.375 729 65	3,375 720 55	2,84		
	3.375 729 52	3,375 720 50	2,82		
	3.375 729 40	3,375 720 32	2,84		

silver; circulated cell : NIST Ag 94-4; local cells : (PTB Ag 6), NIST Ag 92-1. The first of each three W-measurements have been averaged to Δt.

thermometer s/n BTC-4

date	W NIST Ag 94-4	W PTB Ag 6	Δt (Ag 94-4 -Ag 6), mK	aver. Δt (Ag 94-4 - Ag 6), mK	s.d., mK
Jan. 1998	4.285 970 30	4.285 982 38	(-4.25)	-0.75	1.47
	4.285 980 94	4.285 980 71	0.08		
	4.285 980 96	4.285 979 93	0.36		
	4.285 964 56	4.285 963 18	0.48		
	4.285 963 23	4.285 962 96	0.09		
	4.285 961 87	4.285 964 47	-0.92		
	4.285 950 38	4.285 956 03	-1.99		
	4.285 955 04	4.285 956 38	-0.47		
	4.285 954 74	4.285 956 17	-0.50		

thermometer s/n BTC-4

date	W NIST Ag 94-4	W NIST Ag 92-1	Δt (Ag 94-4 - Ag 92-1), mK	aver. Δt (Al 94-4 - Al 92-1), mK	s.d., mK
Feb.1998	4.285 968 72	4.285 969 09	-0.13	-0.13	0.06
	4.285 968 32	4.285 968 78	-0.16		
	4.285 968 30	4.285 968 57	-0.09		
	4.285 968 67	4.285 968 87	-0.07		
	4.285 968 34	4.285 968 71	-0.13		
	4.285 968 01	4.285 968 90	-0.31		
	4.285 967 62	4.285 968 18	-0.20		
	4.285 967 21	4.285 967 81	-0.21		
	4.285 966 96	4.285 967 45	-0.17		

thermometer s/n 1025

date	W NIST Ag 94-4	W NIST Ag 92-1	Δt (Ag 94-4 - Ag 92-1), mK	aver. Δt (Al 94-4 - Al 92-1), mK	s.d., mK
Sep.1998	4.286 639 52	4.286 639 59	-0.03	-0.07	0.08
	4.286 639 45	4.286 639 47	-0.01		
	4.286 639 11	4.286 639 40	-0.10		
	4.286 638 92	4.286 639 38	-0.16		
	4.286 638 65	4.286 639 06	-0.14		
	4.286 638 32	4.286 638 69	-0.13		
	4.286 638 89	4.286 639 98	-0.03		
	4.286 639 65	4.286 639 76	-0.04		
	4.286 639 49	4.286 639 52	-0.01		

thermometer s/n 1025

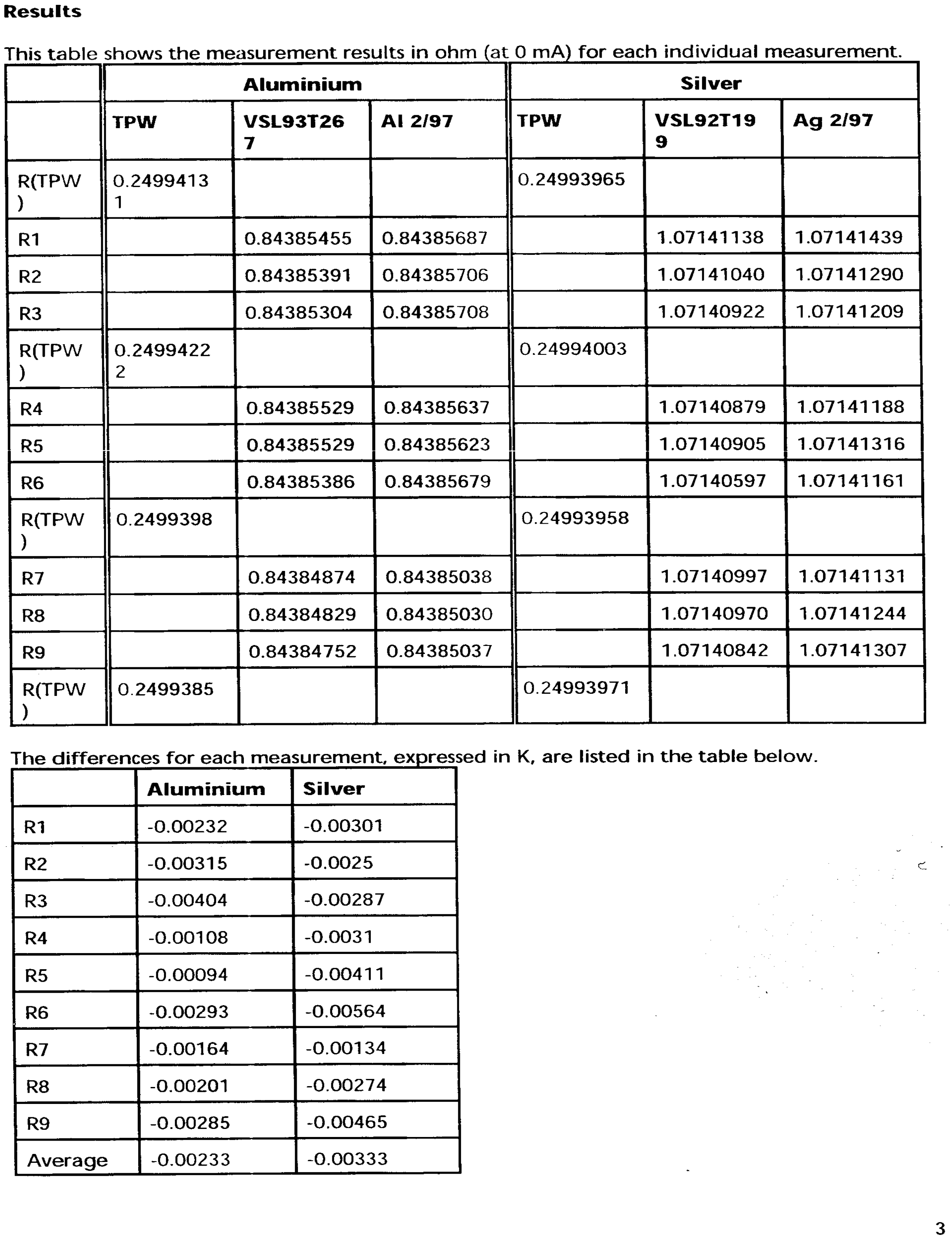
date	W NIST Ag 94-4	W NIST Ag 92-1	Δt (Ag 94-4 - Ag 92-1), mK	aver. Δt (Al 94-4 - Al 92-1), mK	s.d., mK
July 1999	4.286 638 89	4.286 639 21	-0.11	-0.12	0.08
	4.286 638 45	4.286 638 92	-0.17		
	4.286 638 30	4.286 638 62	-0.11		
	4.286 639 35	4.286 639 92	-0.20		
	4.286 639 23	4.286 639 77	-0.19		
	4.286 638 76	4.286 639 39	-0.22		
	4.286 639 42	4.286 639 53	-0.04		
	4.286 639 66	4.286 639 85	-0.07		
	4.286 639 44	4.286 639 73	-0.10		

thermometer s/n 1025

date	W PTB Ag 6	W NIST Ag 92-1	Δt (Ag 6 - Ag 92-1), mK	aver. Δt (Ag 6 - Ag 92-1), mK	s.d., mK
Jan. 2000	4.286 638 09	4.286 638 71	-0.22	-0.42	0.23
	4.286 637 14	4.286 638 13	-0.35		
	4.286 635 51	4.286 637 63	-0.75		
	4.286 638 50	4.286 637 43	-0.38		
	4.286 638 02	4.286 637 41	-0.21		
	4.286 636 39	4.286 635 40	-0.35		
	4.286 637 55	4.286 639 46	-0.67		
	4.286 637 09	4.286 639 12	-0.72		
	4.286 636 43	4.286 638 96	-0.89		

5.2.6 NMi/VSL

local cells : VSL 93T26 (Al), VSL 92T19 (Ag)
circulated cells : NPL Al 2/97, NPL Ag 2/97



Results

This table shows the measurement results in ohm (at 0 mA) for each individual measurement.

	Aluminium			**Silver**		
	TPW	**VSL93T267**	**Al 2/97**	**TPW**	**VSL92T199**	**Ag 2/97**
R(TPW)	0.24994131			0.24993965		
R1		0.84385455	0.84385687		1.07141138	1.07141439
R2		0.84385391	0.84385706		1.07141040	1.07141290
R3		0.84385304	0.84385708		1.07140922	1.07141209
R(TPW)	0.24994222			0.24994003		
R4		0.84385529	0.84385637		1.07140879	1.07141188
R5		0.84385529	0.84385623		1.07140905	1.07141316
R6		0.84385386	0.84385679		1.07140597	1.07141161
R(TPW)	0.2499398			0.24993958		
R7		0.84384874	0.84385038		1.07140997	1.07141131
R8		0.84384829	0.84385030		1.07140970	1.07141244
R9		0.84384752	0.84385037		1.07140842	1.07141307
R(TPW)	0.2499385			0.24993971		

The differences for each measurement, expressed in K, are listed in the table below.

	Aluminium	**Silver**
R1	-0.00232	-0.00301
R2	-0.00315	-0.0025
R3	-0.00404	-0.00287
R4	-0.00108	-0.0031
R5	-0.00094	-0.00411
R6	-0.00293	-0.00564
R7	-0.00164	-0.00134
R8	-0.00201	-0.00274
R9	-0.00285	-0.00465
Average	-0.00233	-0.00333

3

5.2.7 NML

local cells : NML Al 98/2, NML Ag 93/1
circulated cells : KRISS Al 97-1, KRISS/NIM Ag 9306

date	W NML Al 98/2	W KRISS Al 97-1	aver. (Al 98/2 - Al 97-1)	Δt (Al 98/2 - Al 97-1), mK
May 1999	3.374 693 14	(3.374 641 86)	1.089 3E-05	3.40
	3.374 692 61	3.374 661 19		
	3.374 686 36	3.374 671 79		
	(3.374 684 46)	3.374 706 45		

date	W NML Ag 93/1	W KRISS/NIM Ag 9306	aver. (Ag 93/1 - Ag 9306)	Δt (Ag 93/1 - Ag 9306), mK
May 1999	4.286 101 89	4.286 138 11	-2.941 5E-05	-10.36
	4.286 110 19	4.286 139 56		
	4.286 104 82	4.286 131 97		
	4.286 105 51	4.286 141 36		
	4.286 114 31			
	4.286 113 29			

5.2.8 NPL

local cells : NPL Al 89, NPL Ag 10/98
circulated cell : NPL Al 2/97, NPL Ag 2/97

	NPL May 1998	**PTB (at NPL) June 1998**	**NMi/VSL Aug-Sep 1998**	**BNM-INM Oct-Nov 1998**
Al	Al 89 - Al 2/97 -0.2 ± 0.55	Al 98-1 - Al 2/97 -0.8 ± 1.1	VSL93T267 - Al 2/97 -2.3 ± 1.0	Al 123 - Al 2/97 2.3 ± 0.5
Ag	Ag 8/97 - Ag 2/97 0.0 ± 1.4	Ag 6- Ag 2/97 4.6 ± 1.7	VSL92T199 - Ag 2/97 -3.3 ± 1.3	Ag 105 - Ag 2/97 1.1 ± 1.5

	VNIIM Dec 1998-June 1999	**NPL April 1999**
Al	VNIIM - Al 2/97 0.55 ± 0.20	Al 89 - Al 2/97 -2.8 ± 0.6
Ag	VNIIM - Ag 2/97 1.63 ± 0.27	Ag 10/98 - Ag 2/97 -2.2 ± 1.1

5.2.9 NRC

local cells : NRC Al-6, NRC Ag-6
circulated cells : NIST Al 96-1, NIST Ag 94-4

date	W NRC Al-6	W NIST Al 96-1	ΔW (Al-6 - Al 96-1), mK	Δt, mK
Aug. 1998	3,375 733 144	3,375 729 613	4.335E-06	1.35
	3,375 732 547	3,375 728 301		
	3,375 731 832	3,375 726 869		

date	W NRC Ag-6	W NIST Ag 94-4	Δt (Ag-6 - Al 94-4), mK	aver. Δt, mK
Aug. 1998	4,286 388 7	4,286 395 447	-1,69	
	4,286 387 0	4,286 394 011		
	4,286 387 2	4,286 387 856		
	4,2863920	4,286391549	-0.87	
	4,2863920	4,286398936		
	4,2863920	4,28639278		
	4,2863936	4,286394044	0.05	0.18
	4,2863940	4,286394454		
	4,2863932	4,286391992		
	4,2863942	4,286393633	0.31	
	4,2863932	4,286391787		
	4,2863918	4,286390966		
	4,2863916	4,286390966		
	4,2863916	4,286401471	-3.23	
	4,2863908	4,286399953		
	4,2863908	4,286399173		
	4,2863986	4,286405534	-2.12	
	4,2863994	4,286405534		
	4,2864002	4,286405124		

5.2.10 NRLM

local cells : NRLM Al, NRLM Ag
circulated cells : KRISS Al 97-1, KRISS/NIM Ag 9306

Al point comparison measurement

$\mathrm{Dw}_{\text{NRLM Al 97-1 - KRISS Al 97-1}}$	average	Dt / mK
-1,96E-06	-1,02E-06	-0,32
-1,32E-06 -6,34E-07		
-1,99E-06		
-6,52E-07		
-4,95E-07		
-1,62E-06		
-1,22E-06		
-4,12E-07		
-1,96E-06		
-5,22E-07		
5,71E-07		

Ag point comparison measurement

$\mathrm{Dw}_{\text{NRLM Ag 98-1 - KRISS/NIM Ag 9306}}$	average	Dt / mK
3,25E-06	-1,8E-07	-0,06
-1,44E-06		
-3,90E-06		
2,29E-06 1,92E-06		
7,8E-07		
-1,47E-06 8,9E-07		
-3,94E-06		

5.2.11 PTB

local cell : PTB Al 97-2, PTB Ag 6
circulated cell : PTB Ag 6, PTB/NIST Al 94-3

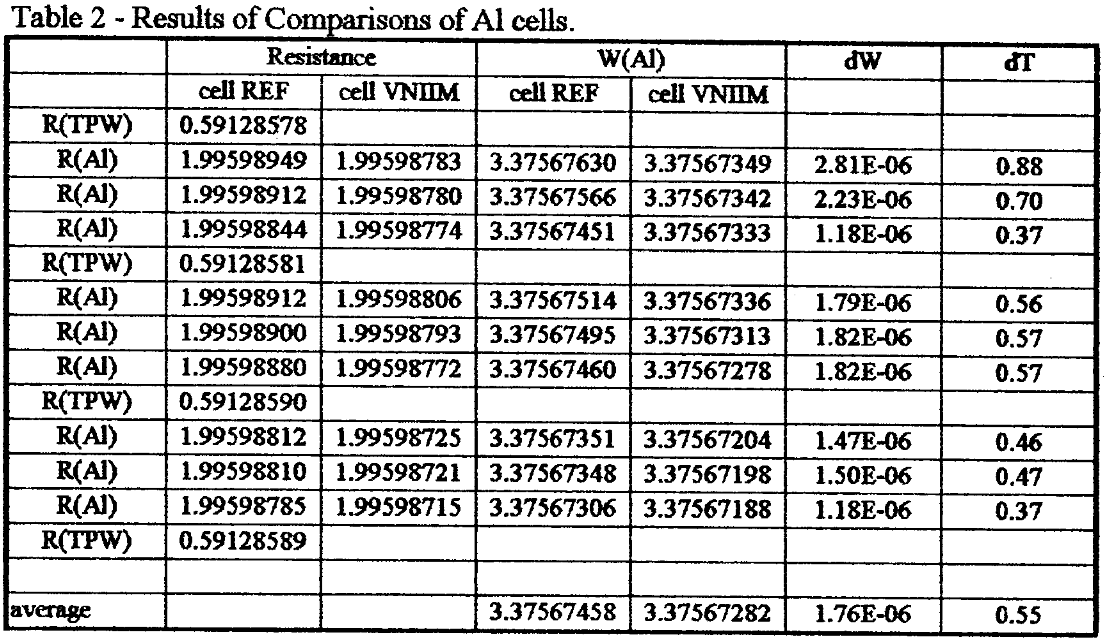
date	W PTB Al 97-2	W PTB/NIST Al 94-3	ΔW (Al 97-2 - Al 94-3), mK	aver. Δt (Al 97-2 - Al 94-3), mK	s.d., mK
Dec. 1998	3.375 660 49	3.375 652 29	8.20E-06	2.87_2	0.35
	3.375 656 33	3.375 650 24	6.09E-06		
	3.375 657 11	3.375 646 63	10.48E-06		
	3.375 659 27	3.375 650 27	9.00E-06		
	3.375 660 41	3.375 650 92	9.49E-06		
	3.375 659 77	3.375 648 04	11.73E-06		
	3.375 657 98	3.375 646 45	11.53E-06		
	3.375 661 60	3.375 653 23	8.37E-06		
	3.375 660 90	3.375 652 26	8.64E-06		
	3.375 660 79	3.375 652 26	8.53E-06		

All other comparison results of the PTB are listed in tables 5.2.3, 5.2.5, and 5.2.8.

5.2.12 VNIIM

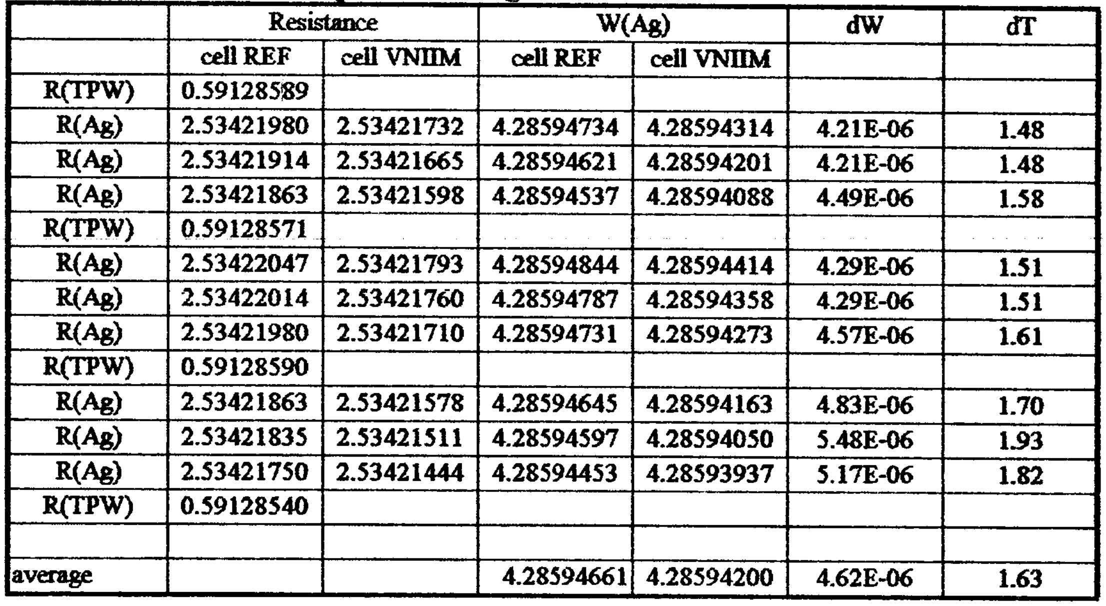
local cells : VNIIM Al, VNIIM Ag
circulated cells : NPL Al 2/97, NPL Ag 2/97

Table 2 - Results of Comparisons of Al cells.



	Resistance		W(Al)		dW	dT
	cell REF	cell VNIIM	cell REF	cell VNIIM		
R(TPW)	0.59128578					
R(Al)	1.99598949	1.99598783	3.37567630	3.37567349	2.81E-06	0.88
R(Al)	1.99598912	1.99598780	3.37567566	3.37567342	2.23E-06	0.70
R(Al)	1.99598844	1.99598774	3.37567451	3.37567333	1.18E-06	0.37
R(TPW)	0.59128581					
R(Al)	1.99598912	1.99598806	3.37567514	3.37567336	1.79E-06	0.56
R(Al)	1.99598900	1.99598793	3.37567495	3.37567313	1.82E-06	0.57
R(Al)	1.99598880	1.99598772	3.37567460	3.37567278	1.82E-06	0.57
R(TPW)	0.59128590					
R(Al)	1.99598812	1.99598725	3.37567351	3.37567204	1.47E-06	0.46
R(Al)	1.99598810	1.99598721	3.37567348	3.37567198	1.50E-06	0.47
R(Al)	1.99598785	1.99598715	3.37567306	3.37567188	1.18E-06	0.37
R(TPW)	0.59128589					
average			3.37567458	3.37567282	1.76E-06	0.55

Table 3 - Results of Comparisons of Ag cells.



	Resistance		W(Ag)		dW	dT
	cell REF	cell VNIIM	cell REF	cell VNIIM		
R(TPW)	0.59128589					
R(Ag)	2.53421980	2.53421732	4.28594734	4.28594314	4.21E-06	1.48
R(Ag)	2.53421914	2.53421665	4.28594621	4.28594201	4.21E-06	1.48
R(Ag)	2.53421863	2.53421598	4.28594537	4.28594088	4.49E-06	1.58
R(TPW)	0.59128571					
R(Ag)	2.53422047	2.53421793	4.28594844	4.28594414	4.29E-06	1.51
R(Ag)	2.53422014	2.53421760	4.28594787	4.28594358	4.29E-06	1.51
R(Ag)	2.53421980	2.53421710	4.28594731	4.28594273	4.57E-06	1.61
R(TPW)	0.59128590					
R(Ag)	2.53421863	2.53421578	4.28594645	4.28594163	4.83E-06	1.70
R(Ag)	2.53421835	2.53421511	4.28594597	4.28594050	5.48E-06	1.93
R(Ag)	2.53421750	2.53421444	4.28594453	4.28593937	5.17E-06	1.82
R(TPW)	0.59128540					
average			4.28594661	4.28594200	4.62E-06	1.63

5.3 Comments by participants on the Key Comparison Reference Value

After a long e-mail discussion all participants or their representatives met on June 20, 2001 at the TEMPMEKO 2001 conference in Berlin. During a vote eight participants decided for the use of a KCRV in CCT K4 calculated as the un-weighted average of the individual deviations from the master circulated cell, while four participants were against it. However, all participants decided to accept the majority vote for the use of a KCRV (voters in italics). It was decided in the following discussion that al methods of calculating a KCRV should be explored by the pilot. Finally, all participants agreed to the use of a weighted mean for the reasons described in section 3.5.

Participants (representatives) in favour of the use of a KCRV

BNM/INM (*Bonnier*)
IMGC (*Marcarino*)
KRISS (*Gam*)
NMi/VSL (*de Groot*)
NML (*Ballico*)
NPL (*Rusby*)
NMIJ (*Arai*)
PTB (*Nubbemeyer*)

Participants (representatives) opposed the use of a KCRV

NIM (*Duan Yuning*)
NIST (*Strouse*)
NRC (*Hill*)
VNIIM (*Pokhodun*)

The next four sections (5.3.1 - 5.3.4) represent the authentic opinions of all four participants who opposed the use of a KCRV.

5.3.1 NIM position on the use of a KCRV in CCT K4

Following is our reason why we opposed to a KCRV:
We think KCRV for K4 has neither physical nor scientific backgrounds, and unbiased KCRV and its uncertainty are difficult to obtain. Moreover, if there is a KCRV , we are worry that the KCRV can be misleading and the used of such values will tend to cause labs to attempt to attain the KCRV rather than the assigned values of the ITS-90.

5.3.2 NIST position on the use of a KCRV in CCT K4

The reasons that NIST does not agree with the use of a KCRV in CCT KC4 are:

a) Different methods of deriving a KCRV (e.g. the use of equally- or unequally-weighted averages) will yield qualitatively different results for apparent agreement between particular laboratories and the KCRV with no clear basis for preference of one KCR value over another.

b) In order to make valid comparisons in a key comparison in which different laboratories have made measurements using non-identical transfer standards, the data must first be normalized to a common basis. The normalization of the data usually introduces different levels of uncertainty and correlations between different laboratories' results, which complicates the computation of the uncertainties of the pair-wise differences between the laboratories. Under these conditions, which are present in KC4 (and KC3), determination of the uncertainties usually requires the use of a variance-covariance matrix (or equivalent non-matrix calculations) to be used in determining the bilateral difference uncertainties. Therefore, unless the uncertainties and correlations introduced by the normalization of the data to accommodate the use of different transfer instruments happens to be the same for all comparisons between laboratories, the KCRV approach to expressing the degree of equivalence cannot be easily implemented in the usual way.

5.3.3 NRC position on the use of a KCRV in CCT K4

I have reviewed the July 13, 2001 version of the CCT-K4 report as requested by the coordinator. Since I am of the opinion that CCT-K4 is better reported without a KCRV and because the majority (8 of 12) of the participants voted in favour of a KCRV, I offer the following commentary as the NRC portion of the minority opinion to be included within the report. First, let me state that the easiest way to explain the difficulties associated with a particular choice of KCRV statistic is to compare the results with those that would have been obtained using a different KCRV statistic. For simplicity, I will compare the weighted average with the simple average since both have been proposed as candidate KCRVs for CCT-K4.

For aluminium, the weighted average leads to a reference value "hotter" by 0.69 mK than the simple average. There are 5 "technical failures", with respect to the weighted average, where the value of the KCRV is not contained within the laboratory's uncertainty at the 95% level. (This is the usual "overlapping error bars" criterion for agreement.) Choosing the simple average leads to 3 "technical failures". One laboratory (NML) fails both criteria. Five laboratories (BNM, NIM, NRC, PTB, and VNIIM), out of the 12 participants, satisfy both criteria. The standard deviation of the reported results is 1.8 mK, a value that is larger than the laboratory uncertainty of all but 1 of the 12 participants.

For silver, the weighted average leads to a reference value that is 2.35 mK "hotter" than the value obtained by the simple average. There are 3 technical failures (at the 95% level) with respect to the weighted average and 5 failures with respect to the simple average. Two laboratories, NIST and VSL, fail both criteria. Six laboratories (BNM, NIM, NML, NRC, NRLM, and VNIIM) satisfy both criteria. The standard deviation of the reported results is 4.24 mK, a value that is larger than the laboratory uncertainty of all but 1 of the 12 participants.

At the end of this commentary, I have included two tables containing the calculations relevant to the preceding discussion. Included in the tables are two columns with the heading QDC (Quantified Demonstrated Confidence). The calculations were performed using the QDE Toolkit distributed by Wood, Douglas and Steele of NRC. The QDC columns contain the probabilities (in percent) that the KCRV will be included within the interval defined by the laboratory's distribution at the 95% interval and centered on the laboratory's reported value. Note that the QDC values for the technical failures identified above never exceed 50%.

Why am I against the calculation of a KCRV? Basically, I see the K4 results as a failure to demonstrate compatibility amongst the world's best laboratories. Clearly, much work needs to be done if we wish to achieve such interoperability. It is disconcerting that the identification of the failing laboratories is so sensitive to the method used to calculate the KCRV. This represents, to me, a sufficient technical reason to avoid defining a KCRV.

Further, it has been suggested that statistics which "move around" as much as the weighted and simple averages do in CCT-K4 are not suitable choices for a KCRV. They may be indicative of a data set that is unworthy of such detailed statistical scrutiny. In K3, for instance, it was decided that no KCRV could be chosen to represent the aggregate behaviour of all comparison participants. Similar reasoning seems to apply here, particularly as I would characterize the results of K4 as "worse" than K3 from this perspective.

Recently, Alan Steele and I have taken a different approach in looking at the results of key comparisons. A note on the method, accompanied by the calculations for all of the CCT-K2 and CCT-K3 results, has been prepared as one of the working documents for the 21st meeting of the CCT. The method is quite straightforward and involves summing the various distributions as represented by each laboratory’s value and uncertainty to obtain the pooled distribution representative of our expectations of what would occur on repeated measurement comparisons. The results of applying this technique to CCT-K4 are included here as Figures 1 and 2.

The distributions in Figures 1 and 2 are clearly bimodal and asymmetric. In the case of aluminium, one mode peaks 0.8 mK above the weighted average (which serves as the zero for the horizontal axis) and the second mode peaks 1.4 mK below the weighted average. In the case of silver, one mode peaks 1.2 mK above the weighted average, while the second peaks 2.4 mK below the weighted average. Since the distributions do not appear “normal” (i.e. Gaussian), I believe that further statistical analysis is unwarranted. Rather, CCT-K4 is better reported without recourse to a KCRV and further consideration of the experimental vagaries exposed in this comparison is required.

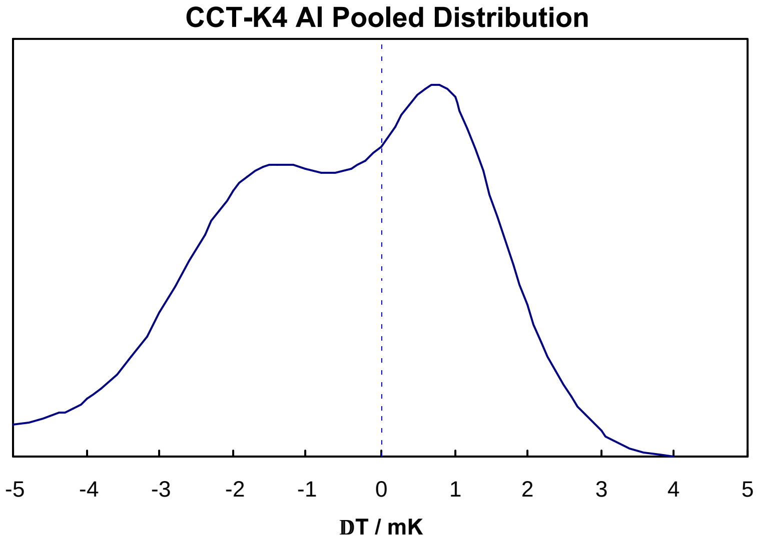


Figure 1. The pooled distributions at the freezing point of aluminium based on data from the CCT-K4 report (July13, 2001). The weighted average is the zero on the horizontal axis.

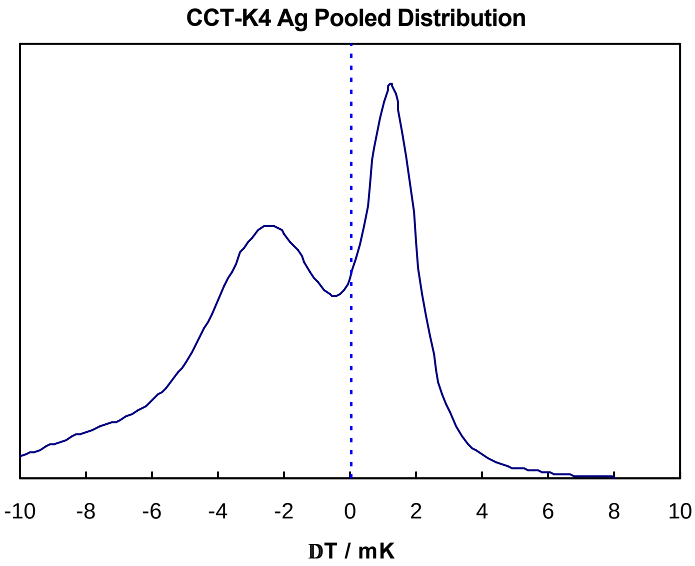


Figure 2. The pooled distributions at the freezing point of silver based on data from the CCT-K4 report (July13, 2001). The weighted average is the zero on the horizontal axis.

The next question that we must consider is whether our actions in choosing to report a KCRV and deciding on its method of calculation are consistent with paragraph T.3 of the Technical supplement to the arrangement (the MRA):
"Although the key comparison reference value is normally a close approximation to the corresponding SI value, it is possible that some of the values submitted by individual participants may be even closer. In a few instances, for example in some chemical measurements, there may be difficulty in relating results to the SI. Nevertheless, the key comparison reference value and deviations from it are good indicators of the SI value. For this reason, these values are used to express the degree of equivalence between the standards of participating laboratories. In some exceptional cases, a Consultative Committee may conclude that for technical reasons a reference value for a particular key comparison is not appropriate; the results are then expressed directly in terms of the degrees of equivalence between pairs of standards."

Have we satisfied the text of paragraph T.3 in selecting the weighted average from K4 as the KCRV? I do not believe that we have. Had the pooled distributions appeared Gaussian, then we might have had some justification to consider some summary statistic from the distribution to be representative of the realization of the particular fixed point. We might further be willing to discuss the significance of the width of the distribution as a measure of the reliability with which the fixed point is realized in the various national metrology institutes. Instead, we are left with a result that is best described as disappointing. The results are unworthy of a KCRV-style analysis. Could we have simply summarized the results of CCT-K4 as the bilateral differences alluded to in the final sentence of paragraph T.3? Undoubtedly!

Finally, it may be worth remembering that the choice of summary statistic, when one is selected, speaks to the degree of faith in the reported values and their associated uncertainties. The simple average implies confidence in only the reported values. The weighted average implies confidence in both the values and the uncertainties. The median provides a mechanism to suppress the influence of outliers. As it is a lack of confidence in the reported values that troubles me in selecting a summary statistic for CCT-K4, I find that none of the proposed methods of calculating a KCRV fill me with confidence in the result thus obtained.

Table 1. Analysis of the CCT-K4 data at the freezing point of aluminium.

Participant	**T_{lab} - T_{mc} (mK)**	**u (mK)**	**T_{lab} - $T_{average}$ (mK)**	**T_{lab} - $T_{weighted\ average}$ (mK)**	**QDC** (Average)	**QDC** (Weighted average)
BNM-INM	3.87	0.72	0.94	0.25	76%	94%
IMGC	4.62	0.71	1.69	1.00	35%	72%
KRISS	1.36	1.03	-1.57	-2.26	68%	42%
NIM	3.49	1.03	0.56	-0.13	92%	95%
NIST	4.63	0.54	1.70	1.01	13%	55%
VSL	-0.74	1.99	-3.67	-4.36	56%	42%
NML	5.55	0.67	2.62	1.93	3%	19%
NPL	1.37	1.10	-1.56	-2.25	72%	48%
NRC	4.18	0.71	1.25	0.56	59%	89%
NRLM	1.83	0.79	-1.10	-1.79	73%	39%
PTB	2.87	0.75	-0.06	-0.75	95%	84%
VNIIM	2.12	0.90	-0.81	-1.50	86%	63%
Standard deviation	1.80					
Median	3.18					
Average	2.93		3 failures	5 failures		
Weighted average	3.62					

Table 2. Analysis of the CCT-K4 data at the freezing point of silver.

Participant	**T_{lab} - T_{mc} (mK)**	**u (mK)**	**T_{lab} - $T_{average}$ (mK)**	**T_{lab} - $T_{weighted\ average}$ (mK)**	**QDC** (Average)	**QDC** (Weighted average)
BNM-INM	-3.96	1.55	-0.24	-2.59	95%	63%
IMGC	-0.42	1.20	3.30	0.95	23%	88%
KRISS	-0.53	1.32	3.19	0.84	34%	91%
NIM	-4.30	1.59	-0.58	-2.93	94%	56%
NIST	-0.11	0.53	3.61	1.26	0%	35%
VSL	-8.39	1.95	-4.67	-7.02	35%	5%
NML	-14.30	6.43	-10.58	-12.93	64%	50%
NPL	-5.16	1.82	-1.44	-3.79	88%	47%
NRC	-0.04	2.47	3.68	1.33	69%	92%
NRLM	-4.01	1.61	-0.29	-2.64	95%	64%
PTB	0.00	0.77	3.72	1.37	0%	59%
VNIIM	-3.43	1.14	0.29	-2.06	95%	58%
Standard deviation	4.24					
Median	-3.70					
Average	-3.72		5 failures	3 failures		
Weighted average	-1.37					

5.3.4 VNIIM position on the use of a KCRV in CCT K4

We voted for KCRV of KC4. We should understand this act is not scientific result but only the bow to MRA.

It is clear from KC4 results that obtained KCRV may be just only for the concrete set of the national laboratory cells (omitting the discussion of the method of KCRV calculation and uncertainties) because it can be seen from KC4 that the cells of one national laboratory differ considerably: for NIST – to 4,62 mK, for KRISS – to 3,25 mK, for PTB – to 3,67 mK. It is clear for us that the cells are fragile and received KCRV will lose its meaning after the change of any cell of KC4.

Besides that a presence of the differences between the cells of one laboratory about 3-4 mK proposes the demonstration of a reason of that in components of uncertainty budget of the real cell. We did not see these data in KC4. If one laboratory have the cells with difference above 3 –4 mK without the known reasons and the cells of 12 laboratories differ on 6-7 mK, then what the meaning of KCRV is? At large differences between the cells the questions came up about plateau constancy, its slope and etc.

Let us compare the bilateral differences between national laboratories in the Al point in KC3 and KC4. We can see that their signs are opposite and their divergences amount to 4-5 mK when the maximum difference for all laboratories equal to 6-7 mK. Please, pay attention at the curious situation; in Al point (BNM-IMGC) = - 0,75 mK in KC4 at the cell comparison, but (BNM-IMGC) = +4,69 mK in KC3 at the realization Al point.

The choice between simple and weighted means for KCRV can be made after acceptation of uniformity for the uncertainty budget calculation. Now the difference between the combined uncertainties of the national laboratories amount to 10 times at using metals of 6N purity, proposed method of realization and practically the same measuring apparatus.