Laboratory of Metrology and Quality
Faculty of Electrical Engineering
University of Ljubljana, Slovenia

Methanol Bath OB 7/2 ULT

Evaluation report

Approved by *Date: 27. 02. 2012* *Responsible person of LMK* *Date: 27. 02. 2012*

dr. Jovan Bojkovski dr. Janko Drnovšek

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1 Unit under test

The unit under test (UUT) is a methanol bath, model OB 7/2 ULT, serial number 12013928, produced by Kambič laboratory equipment. UUT temperature range is from -90 °C to 10 °C. UUT regulator has user-selectable PID parameters with factory default values Pb=1.25, Ti=120 s, Td=13 s.

Primary usage of the UUT is calibration by comparison of contact thermometers. Maximum immersion depth is 270 mm, the diameter of the working space is 100 mm. The working fluid (methanol or ethanol) enters the working space in the bottom and exits through the overflow holes on the top of the working chamber. Refilling of the working fluid due to contraction is not required even when cooling from room temperature to -90 °C. If working space is overfilled with the working fluid, excessive working fluid is gathered in a container in the service area bellow the working space. The UUT contains approximately 8 litres of fluid.

Figure 1: Kambic OB 7/2 ULT methanol bath with one reference thermometer and several PRTs under calibration

2 Calibration procedure

The aim of the evaluation was to establish the stability and homogeneity of the UUT working space, which are the key parameters for the determination of the uncertainty contribution of the calibration media in the calibration by comparison procedure. Measurements were performed at three temperature setpoints, covering the entire usable temperature range: -90 °C, -50 °C and 1 °C.

Preliminary measurements were performed using eight industrial-grade platinum resistance thermometers (PRTs) distributed over the UUT working space and one reference standard platinum resistance thermometer (SPRT) in the centre of the working space. The calibration uncertainty of PRTs is 20 mK and the calibration uncertainty of the SPRT is 1.5 mK. The results of preliminary measurements indicated that the performance of the UUT is superior to the performance of the used PRTs, so the results were in a large extent reflecting the PRT problems with stability, hysteresis and thermal contact. From the point of view of the determination of the UUT homogeneity, these results were therefore misleading and were discarded.

In the second phase of the evaluation, PRTs were replaced with two additional SPRTs, so in total three SPRTs were used. All three SPRTs are Isotech, model 909, quartz sheath, standard platinum resistance thermometers, calibrated at fixed points with uncertainty 1.5 mK (k=2). Copies of SPRT calibration certificates are provided as attached document. The length of the SPRT sensor is approximately 60 mm, the centre of the SPRT sensor is approximately 40 mm from the end of the SPRT, Figure 2. The SPRT measures the average temperature along the length of the SPRT sensor. The diameter of the SPRT quartz sheath is approximately 7.5 mm. The required immersion depth for these thermometers is between 100 and 150 mm, otherwise errors due to insufficient immersion may occur. SPRTs were connected to the scanner and resistance bridge ASL F700 with measurement uncertainty 1 mK (k=2), figure 2. The combined uncertainty of the SPRTs and measurement system is 2 mK (k=2). The bridge is able to produce a stable reading within 20 seconds (one minute for the complete cycle of three SPRTs).

The characteristic of used equipment assures us that measured inhomogeneities are result of actual bath performance and not characteristic and design of used equipment.

Figure 2: Used SPRT sensor

For the evaluation of the UUT stability, measurements were performed with a single SPRT placed at the bottom of the working space in a central position. UUT was set to three different temperature setpoints and after the temperature has stabilized. Readings were acquired for the period of at least two hours. Stability was determined as the standard deviation of acquired temperature readings.

During the evaluation UUT homogeneity, SPRT 1160 was placed in the centre of the working space, SPRT 1167 was placed in right front side, 30 mm from the centre of the working space, and SPRT 1170 was placed in the left back side, 30 mm from the centre of the working space, as shown in figure 3. Measurements were initially performed with all SPRTs positioned at the bottom of the working space in order to determine UUT stability. Afterwards two edge SPRTs were raised in steps of 20 mm up to the height where immersion effect started to produce significant error. The centre SPRT was kept at the bottom of the working space in order to monitor bath stability. For the measurement at each immersion depth, the UUT was given at least 10 minutes to stabilize and then the average of 10 minutes of stable readings was calculated.

Cooling rate was measured by starting the UUT at room temperature and cooling down to the low range limit of -90 °C. All three SPRTs were positioned at the bottom of the working space.

Hot thermometer immersion test was performed by withdrawing the SPRT from the UUT at -90 °C, warming it to room temperature and reinserting it to the UUT. The dynamic response was observed until the measured temperatures return to the values before the test.

Small setpoint change test was performed by increasing the temperature setpoint from -90 °C to -85 °C. The dynamic response was recorded until the UUT stabilized at the new setpoint. The test was then repeated by reducing the setpoint to -90 °C.

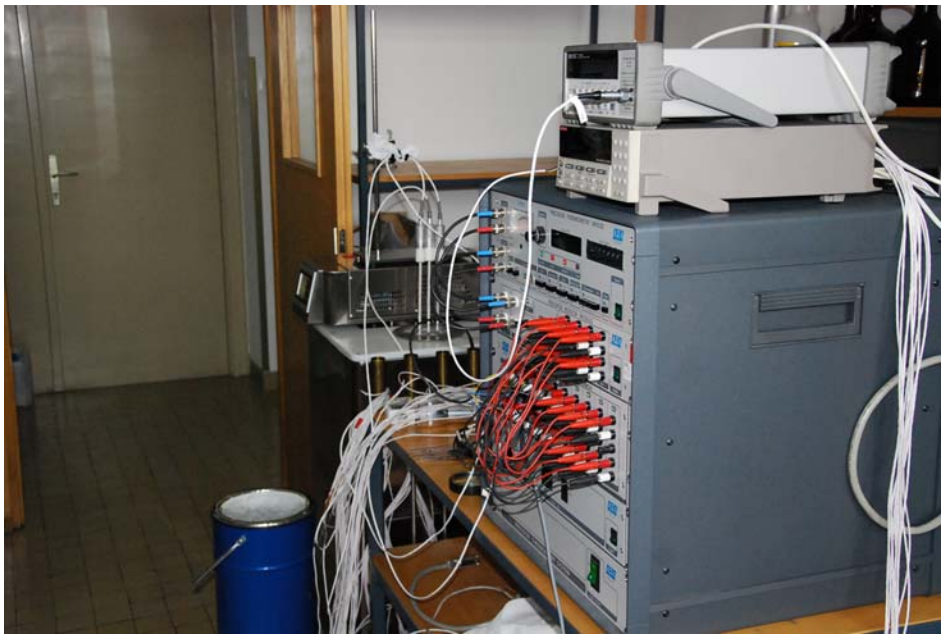

Figure 3: Measurement system with ASL F700 bridge and scanner

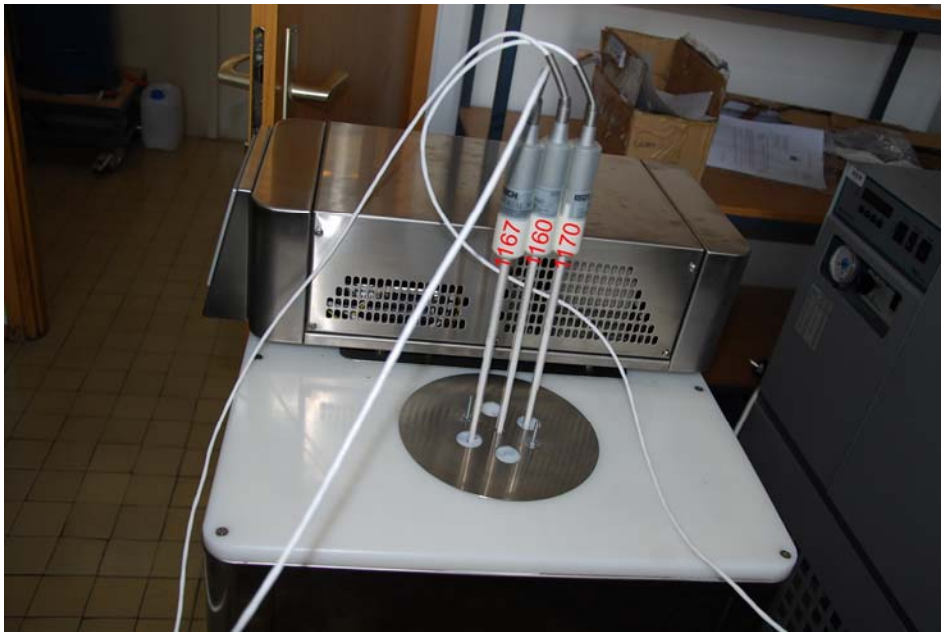

Figure 4: Position of the three SPRTs

3 Environmental conditions

UUT evaluation was performed in laboratory conditions, as specified in the accreditation-approved quality manual. The specified nominal laboratory environmental conditions are temperature within 20 °C ± 5 °C and relative humidity within 15% - 75% RH. Actual environmental conditions during the period of evaluation (February 2012), as measured by the laboratory monitoring system, are presented in Figure 5.

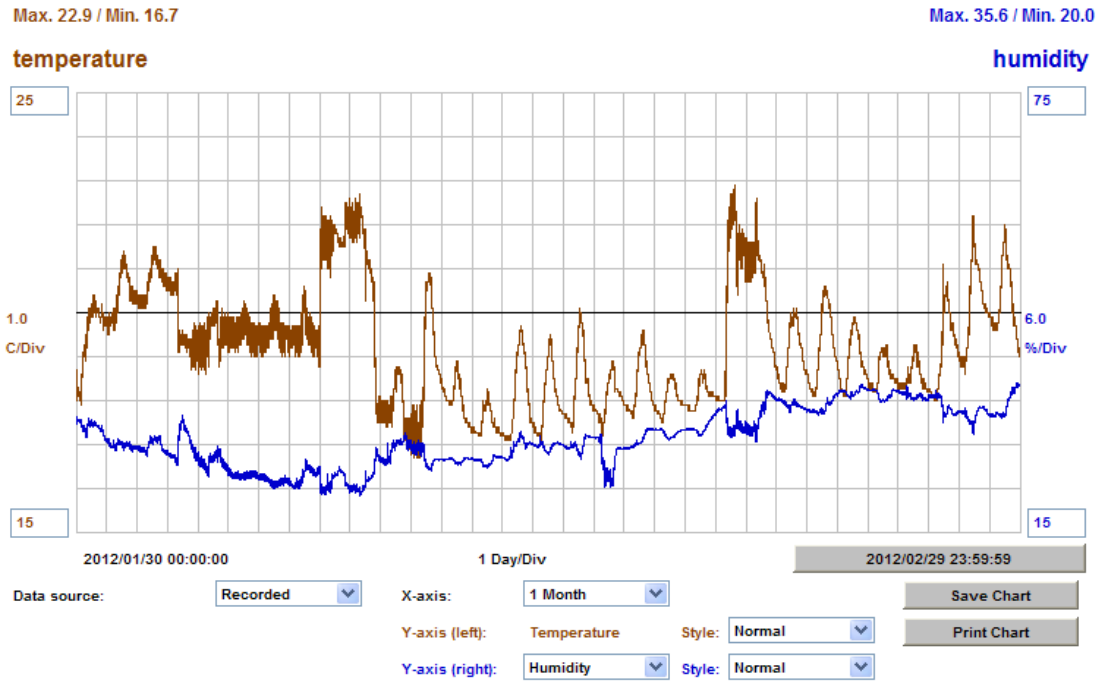

Figure 5: Laboratory environmental conditions in February 2012

4 Results

Results of the UUT evaluation are presented for the measurements of three SPRTs, according to the presented calibration procedure.

4.1 UUT Stability

Stability at -90 °C was measured with one reference SPRT positioned at the bottom in a central position. Standard deviation of readings over 12 hours was 1.7 mK.

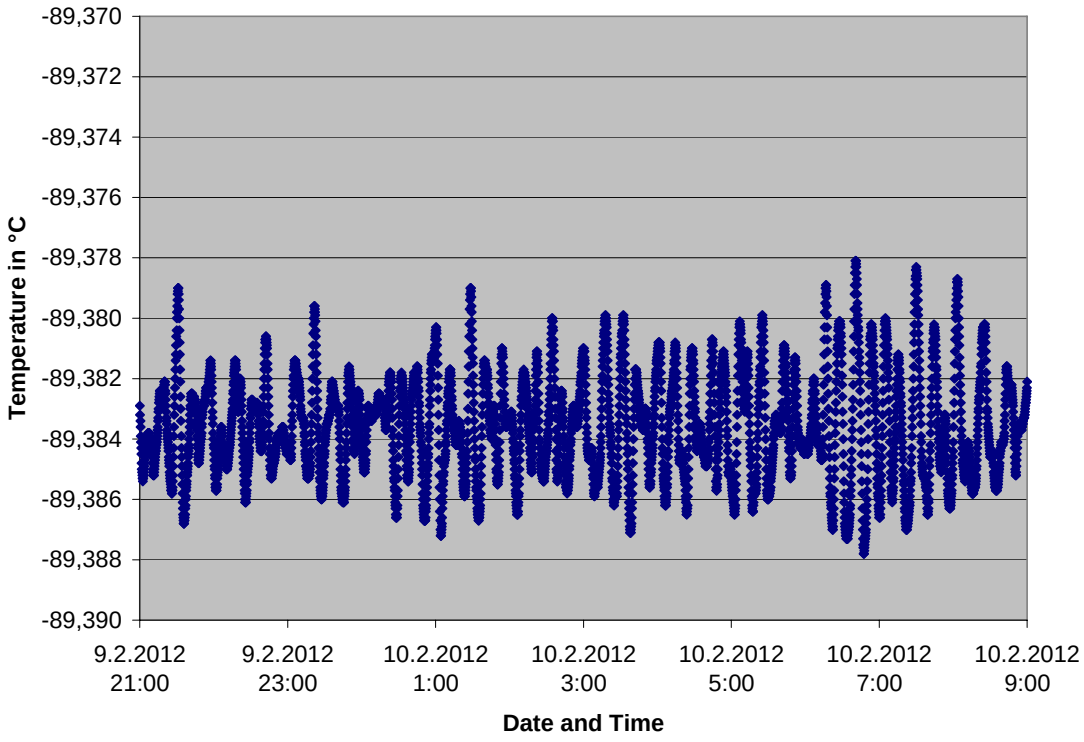

Figure 6: Stability at -90 °C

Stability at -50 °C was measured with one reference SPRT positioned at the bottom in a central position. Standard deviation of readings over 2 hours was 1.0 mK.

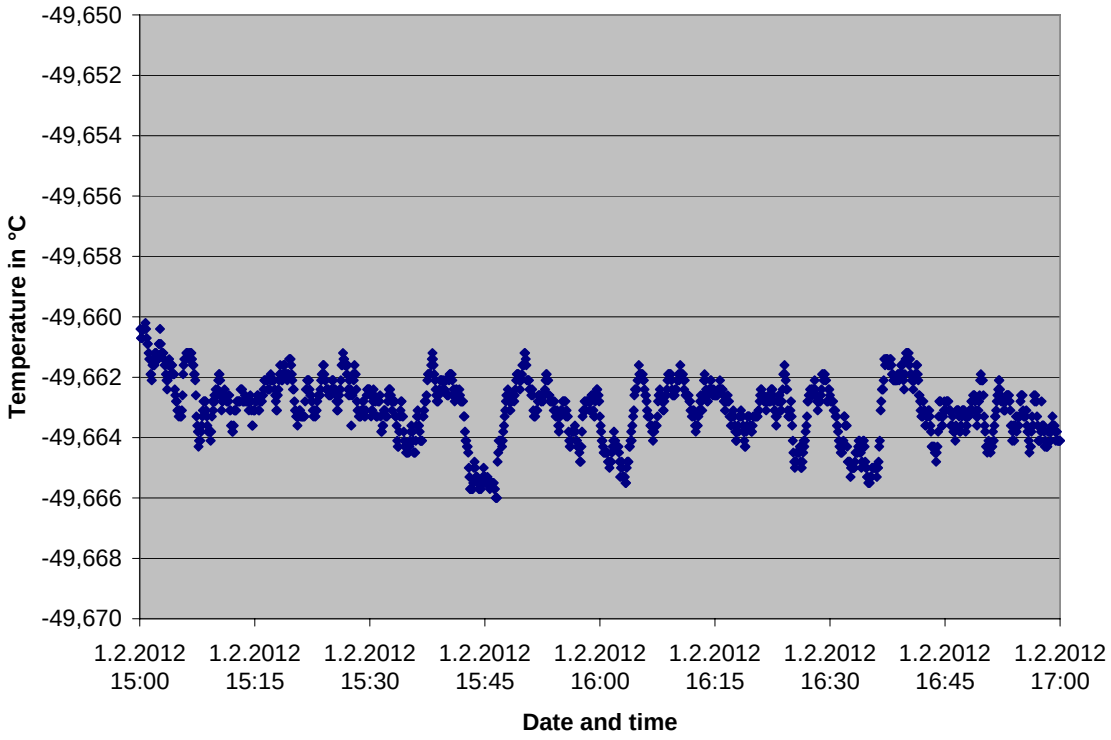

Figure 7: Stability at -50 °C

Stability at 1 °C was measured with one reference SPRT positioned at the bottom in a central position. Standard deviation of readings over 2 hours was 1.7 mK.

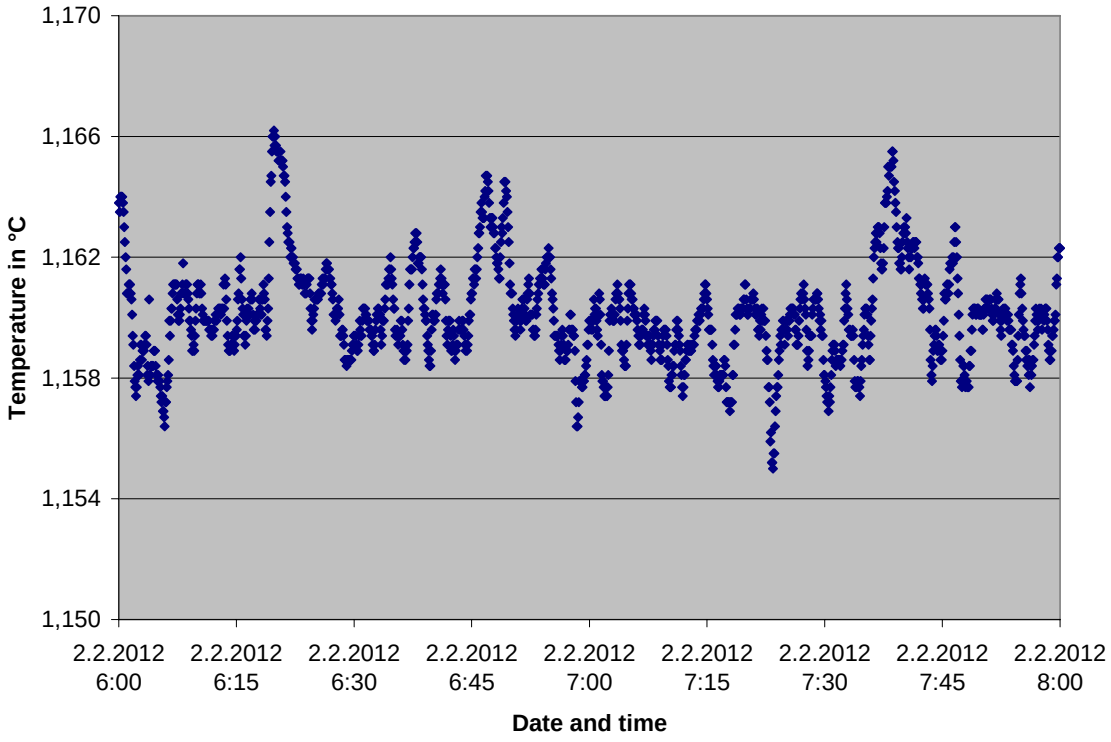

Figure 8: Stability at 1 °C

4.2 UUT Homogeneity

UUT homogeneity was measured using three SPRTs. One of the SPRTs was positioned at the bottom, while the others were lifted in steps of 20 mm.

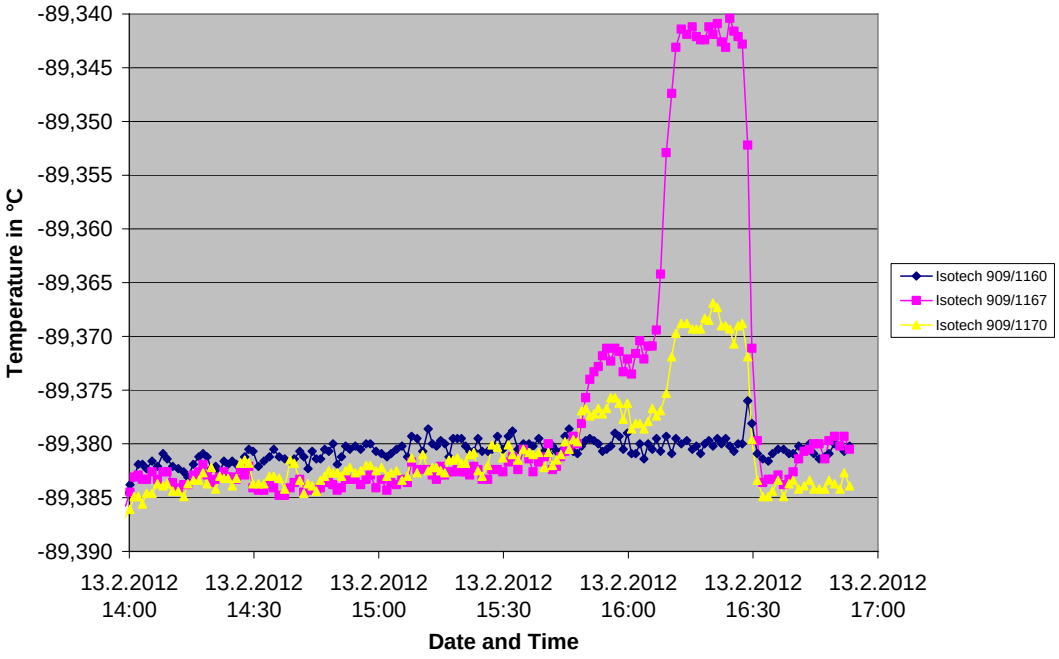

Figure 9: Homogeneity measurements at -90 °C

Table 1: Homogeneity measurements at -90 °C

Start time	End time	Distance to bottom in mm	1160 average temperature in °C	1167 average temperature in °C	1170 average temperature in °C
14:15	14:25	0	-89,3817	-89,3829	-89,3833
14:35	14:45	20	-89,3812	-89,3842	-89,3834
14:50	15:00	40	-89,3806	-89,3836	-89,3824
15:15	15:25	60	-89,3802	-89,3826	-89,3818
15:35	15:45	80	-89,3801	-89,3813	-89,3810
15:55	16:05	100	-89,3802	-89,3718	-89,3772
16:15	16:25	120	-89,3801	-89,3418	-89,3688
16:45	16:55	0	-89,3807	-89,3800	-89,3838

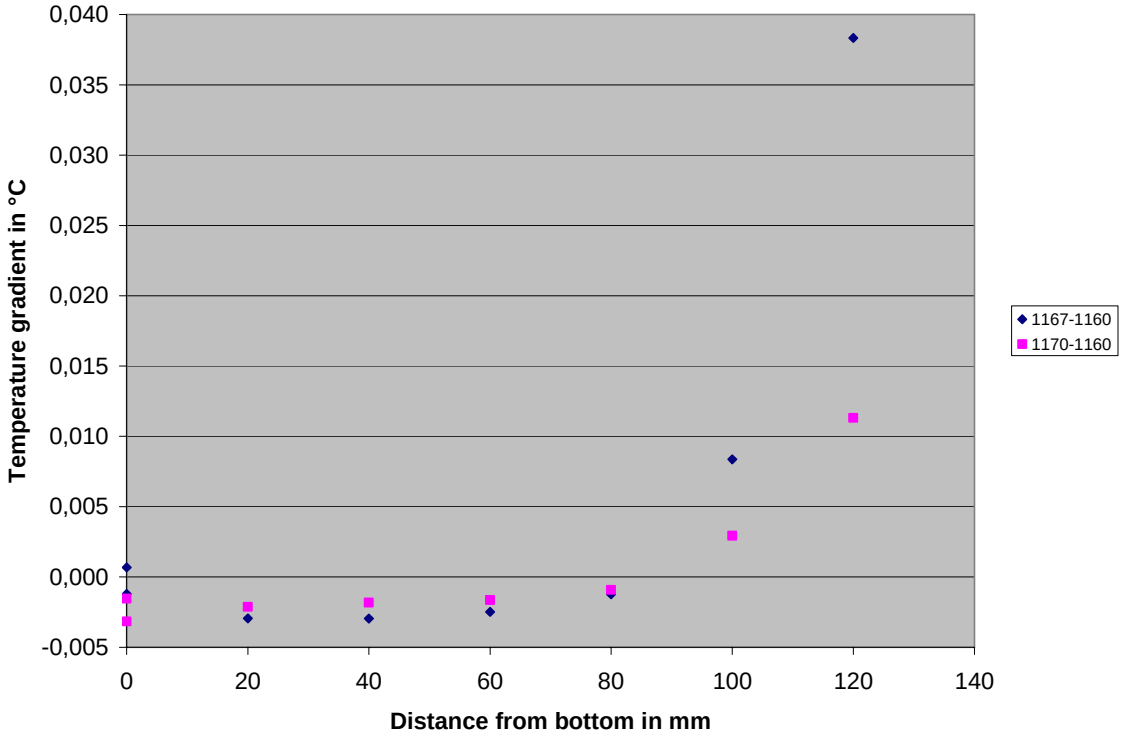

Figure 10: Homogeneity at -90 °C

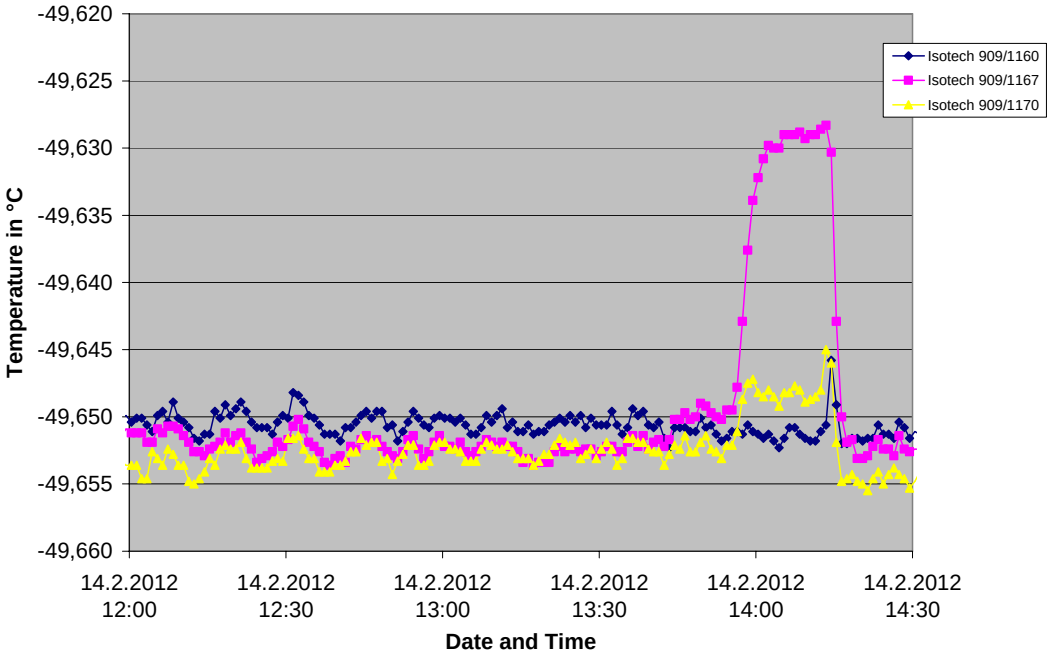

Figure 11: Homogeneity measurements at -50 °C

Table 2: Homogeneity measurements at -50 °C

Start time	End time	Distance to bottom in mm	1160 average temperature in °C	1167 average temperature in °C	1170 average temperature in °C
12:00	12:10	0	-49,6501	-49,6512	-49,6535
12:27	12:37	20	-49,6499	-49,6518	-49,6528
12:47	12:57	40	-49,6505	-49,6524	-49,6530
13:02	13:12	60	-49,6504	-49,6522	-49,6526
13:15	13:25	80	-49,6506	-49,6530	-49,6526
13:29	13:39	100	-49,6503	-49,6522	-49,6524
13:43	13:53	120	-49,6510	-49,6500	-49,6523
14:03	14:13	140	-49,6514	-49,6291	-49,6481
14:20	14:30	0	-49,6512	-49,6524	-49,6547

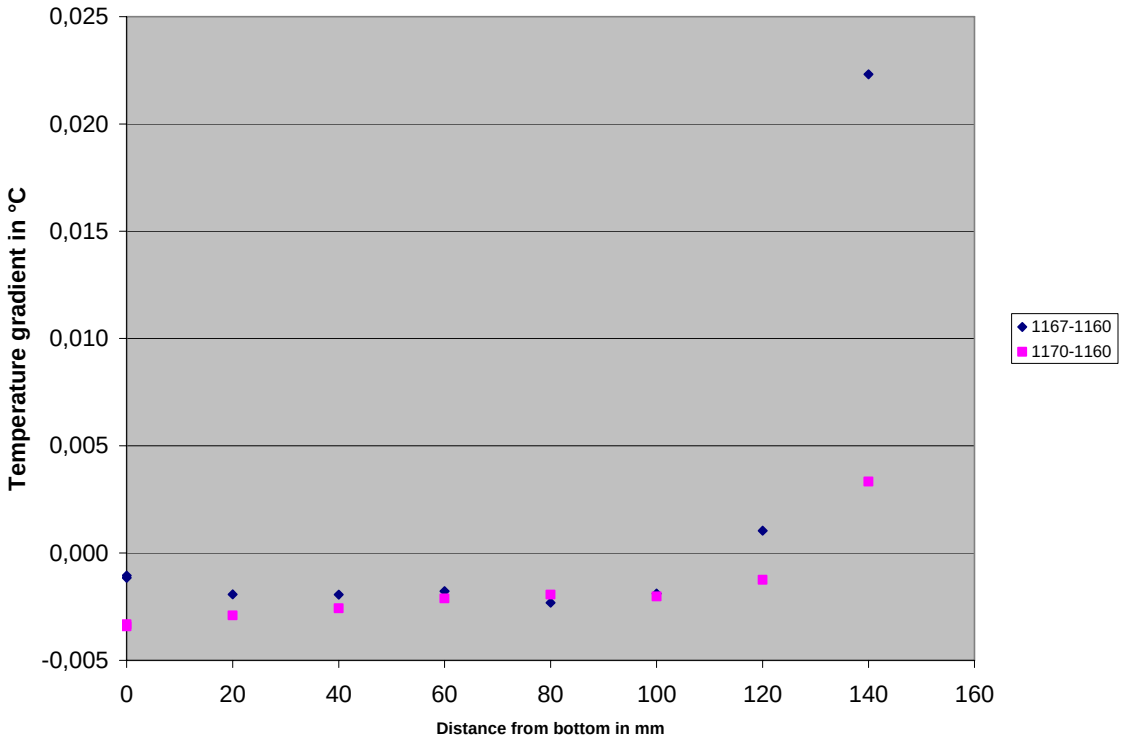

Figure 12: Homogeneity at -50 °C

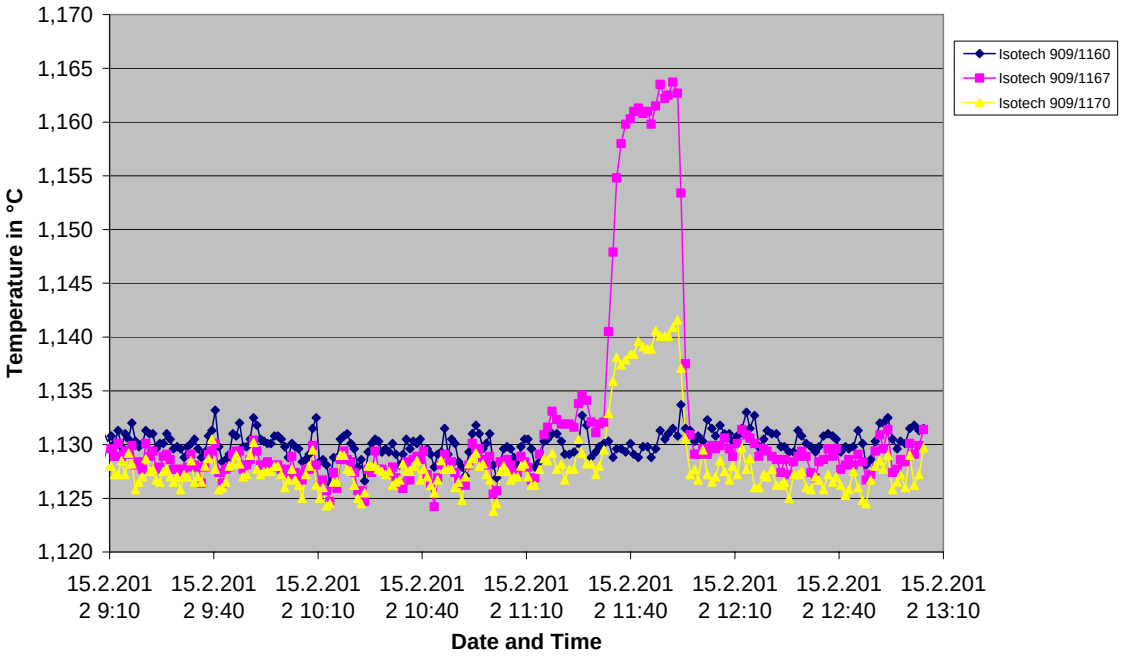

Figure 13: Homogeneity measurements at 1 °C

Table 3: Homogeneity measurements at 1 °C

Start time	End time	Distance to bottom in mm	1160 average temperature in °C	1167 average temperature in °C	1170 average temperature in °C
9:11	9:21	0	1,1307	1,1290	1,1275
9:23	9:33	20	1,1299	1,1281	1,1270
9:35	9:45	40	1,1300	1,1281	1,1275
9:47	9:57	60	1,1307	1,1286	1,1279
10:00	10:10	80	1,1297	1,1277	1,1267
10:11	10:21	100	1,1291	1,1271	1,1266
10:30	10:40	120	1,1296	1,1277	1,1274
11:00	11:10	140	1,1291	1,1277	1,1268
11:19	11:29	160	1,1300	1,1325	1,1282
11:43	11:53	180	1,1303	1,1620	1,1400
12:10	12:20	0	1,1313	1,1300	1,1275

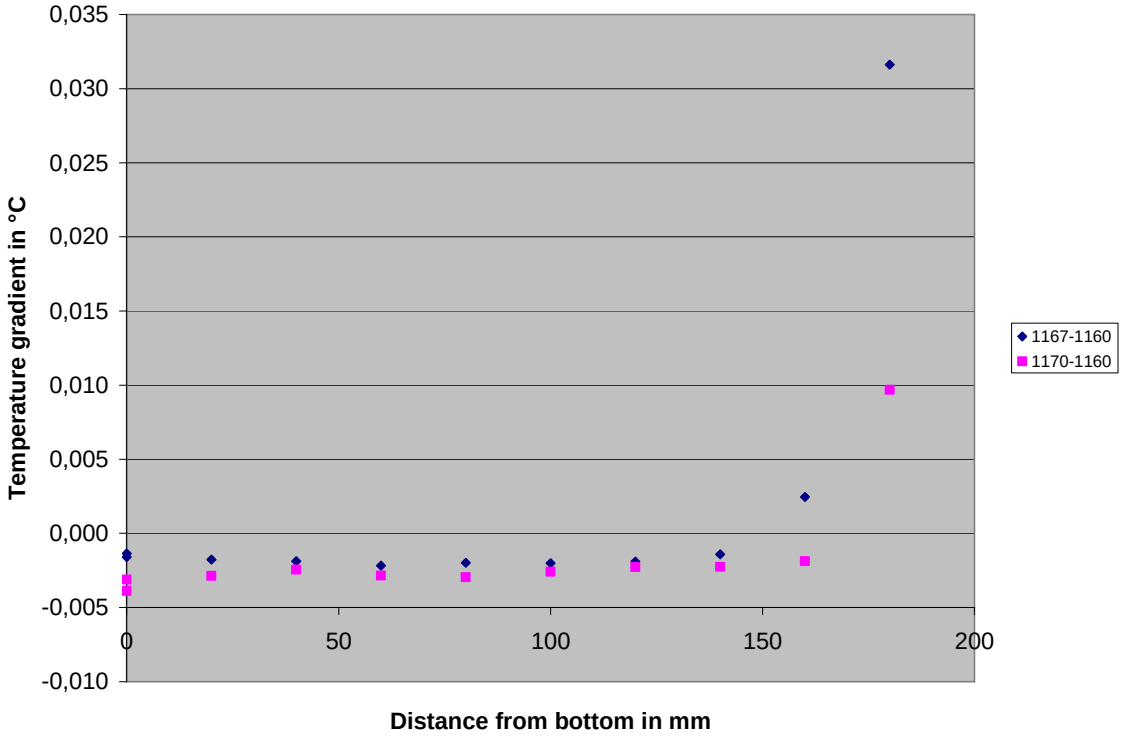

Figure 14: Homogeneity at 1 °C

4.3 UUT Cooling Rate

UUT cooling rate was measured by measuring the time it takes to cool down from room temperature to -90 °C. The temperature stabilized at -90 °C after approximately 3.5 hours, so the average cooling rate is approximately 30 °C per hour. Note that the cooling rate decreases at lower temperatures. The UUT is capable of cooling down to -90 °C setpoint and normally stabilizing. From the shape of the cooling curve it can be assumed that the UUT is capable of cooling even a few °C bellow -90 °C, but this also depends on the purity of methanol, which may degrade over time due to humidification.

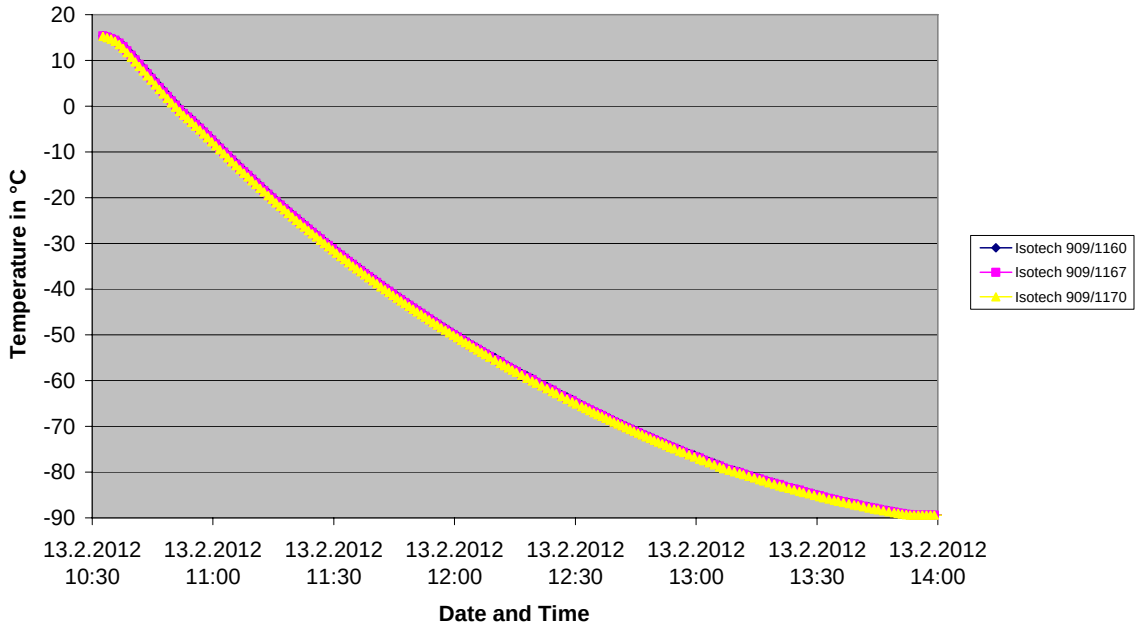

Figure 15: Cooling from room temperature to -90 °C

4.4 UUT Small Setpoint Change

UUT setpoint was increased from -90 °C to -85 °C, allowed to stabilize and decreased back to -90 °C. The dynamic response was observed.

The bath is faster when heating up. The bath took approximately 5 minutes to achieve a new setpoint and another ten minutes to stabilize.

Cooling down is slower. It took approximately 25 minutes to achieve the setpoint and another ten minutes to stabilize. Note that cooling down at -90 °C is the worst case scenario; cooling rates at higher temperatures are higher.

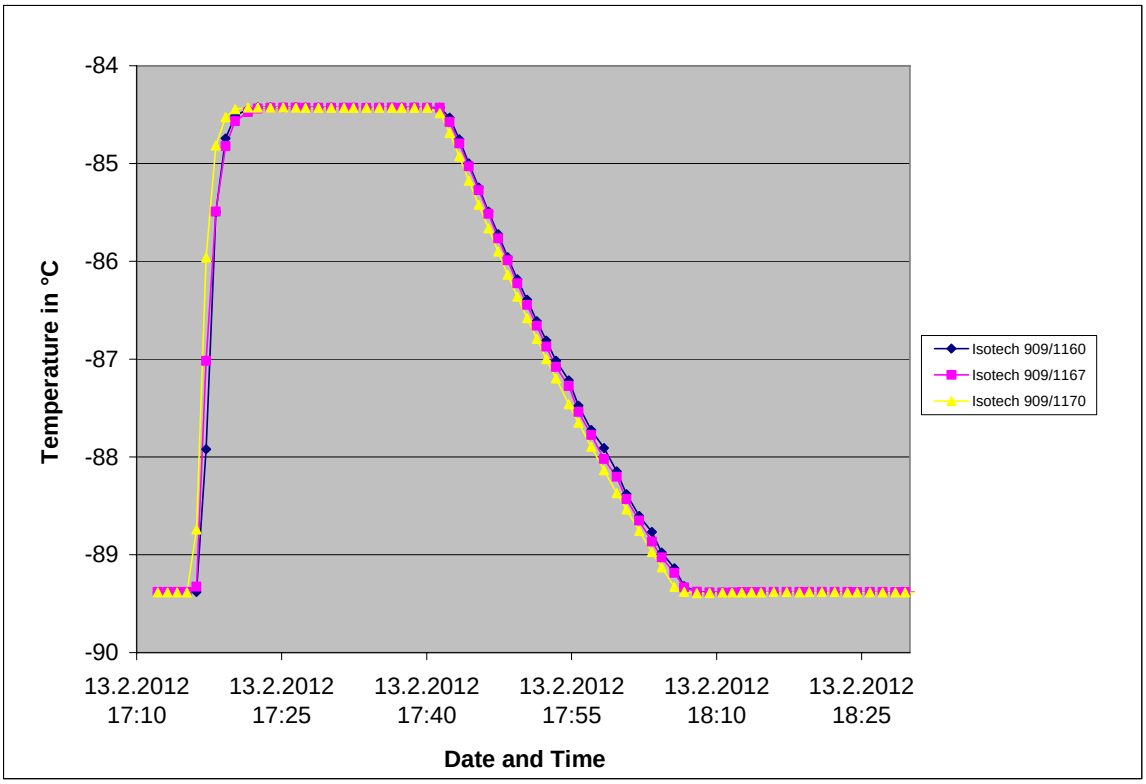

Figure 16: Small setpoint change at -90 °C

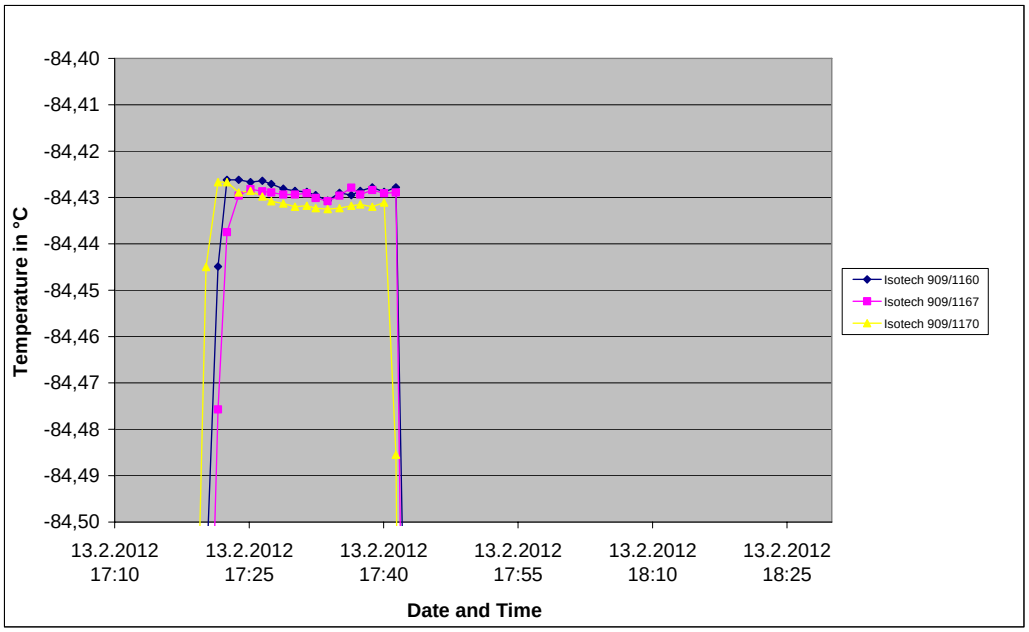

Figure 17: Stabilization at -85 °C after increase from -90 °C

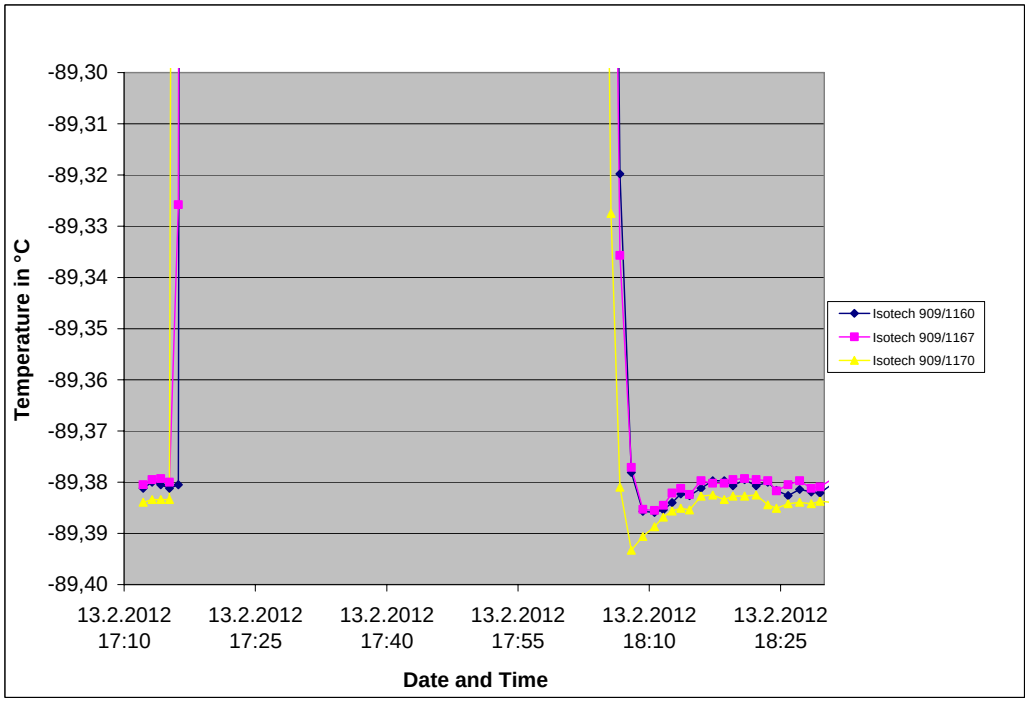

Figure 18: Stabilization at -90 °C after decrease from -85 °C

4.5 UUT Hot Thermometer Insertion Test

Thermometer 1160 was withdrawn from the bath and cooled to room temperature. After that, thermometer was reinserted in the bath and bath dynamic response was observed.

The bath regulation was able to fully stabilize the bath in approximately 4 minutes. The inserted thermometer was fully stabilized after approximately 10 minutes.

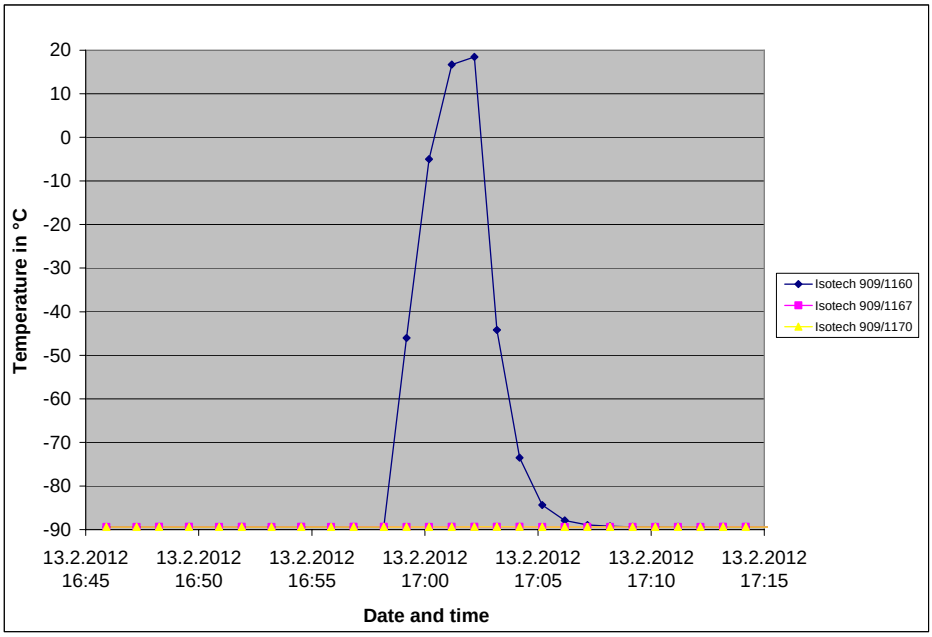

Figure 19: Withdrawal and reinsertion of a thermometer at -90 °C

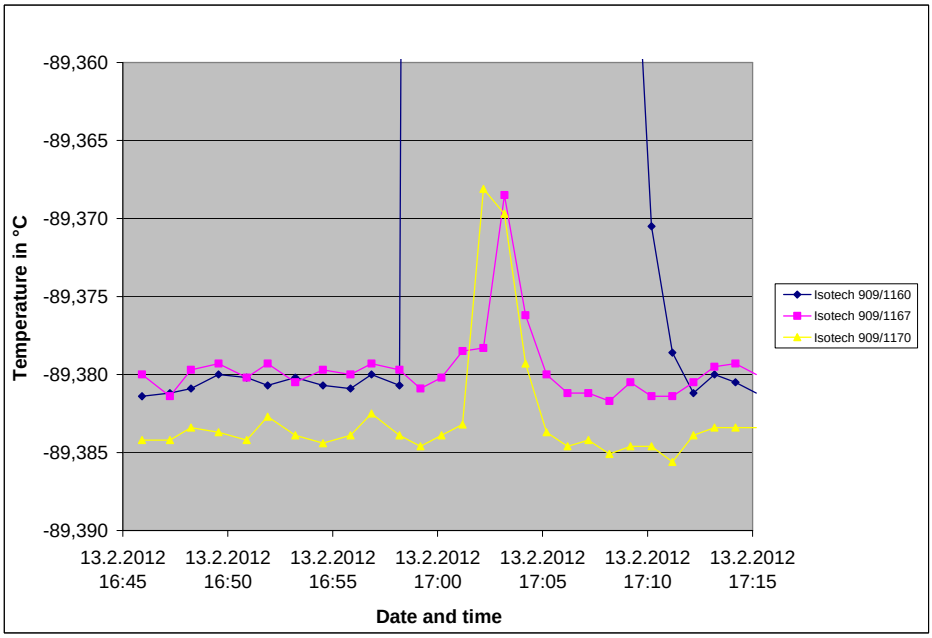

Figure 20: Dynamic response and stabilization after thermometer insertion at -90 °C

5 Conclusions

UUT was found to be adequate for the purpose of calibration by comparison of thermometers at industrial and secondary level. UUT has excellent cooling rate (approximately 30 °C/hour, approximately 3.5 hours from room temperature to -90 °C) and is able to stabilize at a new temperature setpoint very quickly and without significant overshoots. The time to reach a new setpoint depends on the temperature, cooling or heating, ambient temperature and temperature difference, but in most practical cases the UUT is stabilized at the next setpoint in 15 to 30 minutes. This enables the calibration of a thermometer to be practically performed over the UUT temperature range in a single working day. UUT has medium immersion depth (270 mm maximum), which is sufficient for calibration by comparison of most practical thermometers. Working space is sufficient for simultaneous calibration of at least four thermometers under test and placement of a reference thermometer. If some basic precautions are taken, even more thermometers can be calibrated simultaneously, but they should be placed at least 20 mm from the walls of the working space. True temperature during calibration should always be measured with a calibrated reference thermometer, relying on the UUT indication may result in very large errors.
Evaluation results are summarized in the following tables:

Table 4: Homogeneity

Temperature setpoint	Distance from the bottom	Homogeneity (confidence level 95%)	Remark
1 °C	150 mm	5 mK	Depends on thermometer immersion characteristic
-50 °C	120 mm	5 mK	Depends on thermometer immersion characteristic
-90 °C	100 mm	5 mK	Depends on thermometer immersion characteristic

Table 5: Stability

Temperature setpoint	Evaluation period	Standard deviation *s*	2*s* (confidence level 95%)
1 °C	2 hours	1.7 mK	3.4 mK
-50 °C	2 hours	1.0 mK	2.0 mK
-90 °C	12 hours	1.7 mK	3.4 mK
1 °C	10 minutes	1.0 mK	2.0 mK
-50 °C	10 minutes	0.6 mK	1.2 mK
-90 °C	10 minutes	0.8 mK	1.6 mK