# FINAL REPORT

## Bilateral comparison

### CCT-K9.1

# Realizations of the ITS-90 from 273.16 K to 692.677 K

2018 -2019


S. Rudtsch[1], A. I. Pokhodun[2], A. G. Ivanova[2], N. A. Beketov[2]

[1]Physikalisch Technische Bundesanstalt (PTB)
[2]D.I. Mendeleyev Institute for Metrology (VNIIM)


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CONTENT

## Introduction

The bilateral comparison of the ITS-90 realizations at the National Metrological Institutes PTB (Germany) and VNIIM (Russian Federation) in the temperature range from 273.16K to 692.677 K was initiated by the Technical Committee for “Thermometry and thermal physics” TC1.10 of COOMET.
VNIIM was the coordinator of the bilateral comparison.
The purpose of the comparison is to evaluate the differences between the realizations of the national standards in the most essential temperature range with the highest requirements to the measurement uncertainty. This temperature range includes five ITS-90 fixed points: the triple point of water, the melting point of gallium, the freezing points of indium, tin and zinc. Two 25-Ohm Standard Platinum Resistance Thermometers (SPRTs) were used as transfer standards.
The results of the comparison are resistance ratios $W_i$ for both SPRTs including associated uncertainties $U_i$. These were used to calculate differences in temperature $\Delta T_i$ between the fixed-point realizations of the participants for each ITS-90 fixed point ($i$).
This comparison was carried out in a “double blind” manner. This means that neither VNIIM nor PTB knew the results of the other participants before they have submitted their final results. This was ensured by sending the results to an independent third party, i.e. the chair of the CCT-WG-KC (A. Peruzzi). After this point the participants were not allowed to change or correct results. When both NMIs have confirmed that they have provided the final results, the third party submitted all results to the participants for the data evaluation and the common preparation of the Draft A report.

Participating laboratories:

| Name of Laboratory | Contact persons | Address |
|---|---|---|
| Physikalisch-Technische Bundesanstalt (PTB) Germany | Dr. S. Rudtsch steffen.rudtsch@ptb.de | Abbestraße, 2–12, 10587 Berlin, Germany |
| D. I. Mendeleyev Institute for Metrology (VNIIM) Russian Federation | Prof. Pokhodun A.I. a.i.pokhodun@vniim.ru | Moskovsky pr., 19, 190005, St. Petersburg |

## 1. Comparison schedule

| | | |
|---|---|---|
| 01.2018 | Planned | PTB, VNIIM |
| 02.2018 | First draft of the protocol completed | PTB, VNIIM |

| | | |
|---|---|---|
| 03.2018-07.2018 | Review and approval of the protocol | CCT-WG-KC |
| 04.2018-08.2018 | Selection and first calibration of two SPRTs at VNIIM<br>Delivery of the two SPRTs to PTB | VNIIM |
| 08.2018-12.2018 | Calibration of two SPRTs at PTB and return of both SPRTs to VNIIM | PTB |
| 01.2018-03.2019 | Second calibration of the two SPRTs at VNIIM | VNIIM |
| 03.2019 | Delivery of final calibration results and final uncertainty budgets to the chair of CCT-WG-KC (A. Peruzzi) without mutual exchange | VNIIM, PTB |
| 04.2019 -06.2019 | Report in progress. Draft A. | VNIIM, PTB |
| 07.2019-09.2019 | Review of Draft A by CCT-WG-KC | CCT-WG-KC |
| 10.2018 – 12.2019 | Draft B | VNIIM, PTB |
| 01.2020 | Report submitted to CCT WG-KC for final review | VNIIM, PTB |

## 2. Procedures of the comparison

VNIIM selected two SPRTs (type ETS-25, made in Russia): serial number 0002 and 3-95. The SPRTs were calibrated at all ITS-90 fixed points in the comparison range and sent to PTB. PTB calibrated these thermometers at all fixed points and sent the SPRTs back to VNIIM for the back-calibration.

The best measurement practice of PTB and VNIIM should be used for each fixed-point realization. The resistance ratio $W$ was calculated for each fixed point: $W = R_t/R_{TPW}$, where $R_{TPW}$ is the SPRT resistance at the triple point of water obtained directly after measuring $R_t$. The values of $R_t$ and $R_{TPW}$ had to be corrected for self heating due to the measuring current, hydrostatic pressure and pressure in the cell. The calibration cycle had to be repeated at least 3 times.

If after receiving the calibrated SPRTs from VNIIM the measurements of the thermometer resistances at the triple-point of water indicated a change of more than 0,5 mK due to mechanical shock further actions were required. In this case the SPRT resistance had to be stabilized by annealing according to Technical Protocol before the calibration at PTB.

## 3. The measurement results of PTB at the fixed points

Table 1. Average results of 3 cycles for SPRT No. 0002

| | | | | | |
|---|---|---|---|---|---|
| Laboratory name: | | PTB, WG 7.42 | | | |
| $W(t) = R_t/R_{TPW}$, where $R_{TPW}$ – resistance at the TPW after $R_t$ measurement | | | | | |
| Pressure correction in the cell | | | | | |
| Thermometer number | | No. 0002 | | | |
| *R*(1 mA) | *R* (0 mA) | Hydrostatic correction | Pressure correction | $R_{corrected}$ (0mA) | *W* |
| Ohm | Ohm | Ohm | Ohm | Ohm | |
| 24,885264 | 24,885180 | 0,000018 | | 24,885198 | |
| 63,916566 | 63,916438 | -0,000041 | | 63,916397 | **2,5684604** |
| 24,885166 | 24,885083 | 0,000018 | | 24,885101 | |
| 47,096382 | 47,096242 | -0,000033 | | 47,096209 | **1,8925419** |
| 24,885228 | 24,885143 | 0,000018 | | 24,885161 | |
| 40,056057 | 40,055922 | -0,000052 | | 40,055870 | **1,6096271** |
| 24,885253 | 24,885168 | 0,000018 | | 24,885186 | |
| 27,824339 | 27,824246 | 0,000027 | | 27,824273 | **1,1181052** |
| 24,885267 | 24,885183 | 0,000018 | | 24,885201 | |

Table 2. Average results of 3 cycles for SPRT No. 3-95

| | | | | | |
|---|---|---|---|---|---|
| Laboratory name: | | PTB, WG 7.42 | | | |
| $W(t) = R_t/R_{TPW}$, where $R_{TPW}$ – resistance at the TPW after $R_t$ measurement | | | | | |
| Pressure correction in the cell | | | | | |
| Thermometer number | | No.3-95 | | | |
| *R*(1 mA) | *R* (0 mA) | Hydrostatic correction | Pressure correction | $R_{corrected}$ (0mA) | *W* |
| Ohm | Ohm | Ohm | Ohm | Ohm | |
| 24,473745 | 24,473546 | 0,000018 | | 24,473563 | |
| 62,861768 | 62,861559 | -0,000041 | | 62,861519 | **2,5685660** |
| 24,473567 | 24,473374 | 0,000018 | | 24,473391 | |
| 46,318803 | 46,318557 | -0,000033 | | 46,318525 | **1,8926001** |
| 24,473667 | 24,473470 | 0,000018 | | 24,473488 | |
| 39,394496 | 39,394241 | -0,000052 | | 39,394200 | **1,6096671** |
| 24,473684 | 24,473490 | 0,000018 | | 24,473507 | |
| 27,364323 | 27,364132 | 0,000027 | | 27,364159 | **1,1181132** |
| 24,473689 | 24,473496 | 0,000018 | | 24,473513 | |

## 4. The measurement results of VNIIM at the fixed points

Table 3. Average results of 3 cycles for SPRT No. 0002

| Laboratory name: | | VNIIM | | | |
|---|---|---|---|---|---|
| $W(t) = R_t/R_{TPW}$, where $R_{TPW}$ – resistance at the TPW after $R_t$ measurement | | | | | |
| Pressure correction in the cell | | | | | |
| Thermometer number | | No. 0002 | | | |
| *R*(1 mA) | *R* (0mA) | Hydrostatic correction | Pressure correction | $R_{corrected}$ | *W* |
| Ohm | Ohm | Ohm | Ohm | Ohm | |
| Before departure | | | | | |
| 63,916096 | 63,915980 | -0,0000384 | 0,0000075 | 63,915949 | **2,56845927** |
| 24,885009 | 24,88492 | 0,0000178 | 0,0000000 | 24,884938 | |
| 47,096039 | 47,095919 | -0,0000333 | 0,0000069 | 47,095893 | **1,89253974** |
| 24,885093 | 24,885004 | 0,0000178 | 0,0000000 | 24,885022 | |
| 40,055744 | 40,055575 | -0,0000473 | 0,0000035 | 40,055531 | **1,60962489** |
| 24,885077 | 24,884992 | 0,0000178 | 0,0000000 | 24,885010 | |
| 27,824081 | 27,823989 | 0,0000115 | -0,0000023 | 27,823998 | **1,11810519** |
| 24,885025 | 24,884938 | 0,0000178 | 0,0000000 | 24,884956 | |
| After arrival | | | | | |
| 63,917498 | 63,917397 | -0,0000384 | 0,0000075 | 63,917366 | **2,56845965** |
| 24,885552 | 24,885468 | 0,0000178 | 0,0000000 | 24,885486 | |
| 47,096919 | 47,096801 | -0,0000333 | 0,0000069 | 47,096775 | **1,89254020** |
| 24,885551 | 24,885464 | 0,0000178 | 0,0000000 | 24,885482 | |
| 40,056407 | 40,056238 | -0,0000473 | 0,0000035 | 40,056194 | **1,60962443** |
| 24,885496 | 24,885411 | 0,0000178 | 0,0000000 | 24,885429 | |
| 27,824591 | 27,824484 | 0,0000115 | -0,0000023 | 27,824493 | **1,11810554** |
| 24,885465 | 24,885373 | 0,0000178 | 0,0000000 | 24,885391 | |

Table 4. Average results of 3 cycles for SPRT No. 3-95

| Laboratory name: | | VNIIM | | | |
|---|---|---|---|---|---|
| $W(t) = R_t/R_{TPW}$, where $R_{TPW}$ – resistance at the TPW after $R_t$ measurement | | | | | |
| Pressure correction in the cell | | | | | |
| Thermometer number | | No.3-95 | | | |
| $R$(1 mA) | $R$ (0mA) | Hydrostatic correction | Pressure correction | $R_{corrected}$ | $W$ |
| Ohm | Ohm | Ohm | Ohm | Ohm | |
| Before departure | | | | | |
| 62,861695 | 62,861508 | -0,0000390 | 0,0000070 | 62,861476 | **2,56856347** |
| 24,473585 | 24,47338 | 0,0000180 | 0,0000000 | 24,473398 | |
| 46,318764 | 46,318527 | -0,0000338 | 0,0000067 | 46,318500 | **1,89259757** |
| 24,473697 | 24,473489 | 0,0000180 | 0,0000000 | 24,473507 | |
| 39,394458 | 39,394184 | -0,0000490 | 0,0000057 | 39,394141 | **1,60966544** |
| 24,473679 | 24,473478 | 0,0000180 | 0,0000000 | 24,473496 | |
| 27,364249 | 27,364049 | 0,0000121 | -0,0000027 | 27,364058 | **1,11811311** |
| 24,473601 | 24,473408 | 0,0000180 | 0,0000000 | 24,473426 | |
| After arrival | | | | | |
| 62,861877 | 62,861686 | -0,0000390 | 0,0000070 | 62,861654 | **2,56856423** |
| 24,473630 | 24,473442 | 0,0000180 | 0,0000000 | 24,473460 | |
| 46,318800 | 46,318575 | -0,0000338 | 0,0000067 | 46,318548 | **1,89259822** |
| 24,473695 | 24,473506 | 0,0000180 | 0,0000000 | 24,473524 | |
| 39,394486 | 39,394197 | -0,0000490 | 0,0000057 | 39,394154 | **1,60966525** |
| 24,473685 | 24,473489 | 0,0000180 | 0,0000000 | 24,473507 | |
| 27,364267 | 27,364064 | 0,0000121 | -0,0000027 | 27,364073 | **1,11811335** |
| 24,473612 | 24,473416 | 0,0000180 | 0,0000000 | 24,473434 | |

## 5. Comparison results

The differences of the temperature values $\Delta T_i$ and $U_i$ ($\Delta T_i$) of each fixed point was calculated according to:

$$\Delta T_i = \frac{W_{PTB} - (W_{VNIIM,before} + W_{VNIIM,after})/2}{dW_i / dT_i} \qquad (1)$$

$$U_i(\Delta T_i) = \sqrt{U^2_{PTB} + U^2_{VNIIM}} \qquad (2)$$

In addition to the uncertainties reported by the laboratories the uncertainty of possible changes in the transfer SPRTs over the course of the comparison has to be taken into account for the determination of the uncertainty of temperature difference. The uncertainty of the measurement results due to the instability of the transfer standard, $u_{\mathrm{PRT}}$ was calculated under the assumption that the distribution of the thermometer drift in time was rectangular and symmetrical:

$$u_{prt} = \frac{[(W_{VNIIM})_{end} - (W_{VNIIM})_{start}]}{\sqrt{3}} \cdot \frac{\partial T}{\partial W} \qquad (3)$$

The evaluations of the SPRTs instability during the VNIIM- PTB-VNIIM cycle are presented in Tables 5 and 6.

Table 5. SPRT No. 0002

| **Fixed point** | **$(W_{\mathrm{VNIIM}})_{\mathrm{start}}$** | **$(W_{\mathrm{VNIIM}})_{\mathrm{end}}$** | **$\Delta T$ mK** | **$u_{\mathrm{PRT}}$, mK ($k$=1)** |
|---|---|---|---|---|
| Ga | 1,11810519 | 1,11810554 | 0,09 | 0,05 |
| In | 1,60962489 | 1,60962443 | -0,12 | 0,07 |
| Sn | 1,89253974 | 1,89254020 | 0,12 | 0,07 |
| Zn | 2,56845927 | 2,56845965 | 0,11 | 0,06 |

Table 6. SPRT No. 3-95

| **Fixed point** | **$(W_{\mathrm{VNIIM}})_{\mathrm{start}}$** | **$(W_{\mathrm{VNIIM}})_{\mathrm{end}}$** | **$\Delta T$ mK** | **$u_{\mathrm{PRT}}$, mK ($k$=1)** |
|---|---|---|---|---|
| Ga | 1,11811311 | 1,11811335 | 0,06 | 0,03 |
| In | 1,60966544 | 1,60966525 | -0,05 | 0,03 |
| Sn | 1,89259757 | 1,89259822 | 0,18 | 0,10 |
| Zn | 2,56856347 | 2,56856423 | 0,22 | 0,13 |

The differences $\Delta T_i$ (PTB-VNIIM) for the fixed points and their uncertainties $U_i(\Delta T)$ are presented in tables 7 and 8 for each SPRT and their mean values in Table 9.

Table 7. SPRT No. 0002

| **Fixed point** | **PTB** | **VNIIM** | **$\Delta T_i$(PTB-VNIIM)** | **$U_i(\Delta T_i)$ ($k$=2)** |
|---|---|---|---|---|
| | ***W*** | ***W*** | **mK** | **mK** |
| Zn | 2,5684604 | 2,5684595 | 0,29 | 1,6 |
| Sn | 1,8925419 | 1,8925400 | 0,51 | 1,1 |
| In | 1,6096271 | 1,6096247 | 0,64 | 1,0 |
| Ga | 1,1181052 | 1,1181054 | -0,04 | 0,3 |

Table 8. SPRT No. 3-95

| Fixed point | PTB | VNIIM | $\Delta T_i$ (PTB-VNIIM) | $U_i(\Delta T_i)$ ($k$=2) |
|---|---|---|---|---|
| | *W* | *W* | mK | mK |
| Zn | 2,5685660 | 2,5685638 | 0,60 | 1,6 |
| Sn | 1,8926001 | 1,8925979 | 0,59 | 1,1 |
| In | 1,6096671 | 1,6096654 | 0,47 | 1,0 |
| Ga | 1,1181132 | 1,1181132 | <0,01 | 0,3 |

Table 9.

| Fixed point | $\Delta T_i$ (PTB-VNIIM) | $U_i(\Delta T_i)$ ($k$=2) |
|---|---|---|
| | mK | mK |
| Zn | 0,44 | 1,6 |
| Sn | 0,55 | 1,1 |
| In | 0,55 | 1,0 |
| Ga | -0,02 | 0,3 |

## 6. Conclusions

The differences between the values of temperatures of the fixed-point realizations at PTB and VNIIM ($T_{PTB}$ - $T_{VNIIM}$), and their uncertainties are shown on Figure 1. The results confirm the consistency of the ITS-90 realizations of PTB and VNIIM within the stated uncertainties.

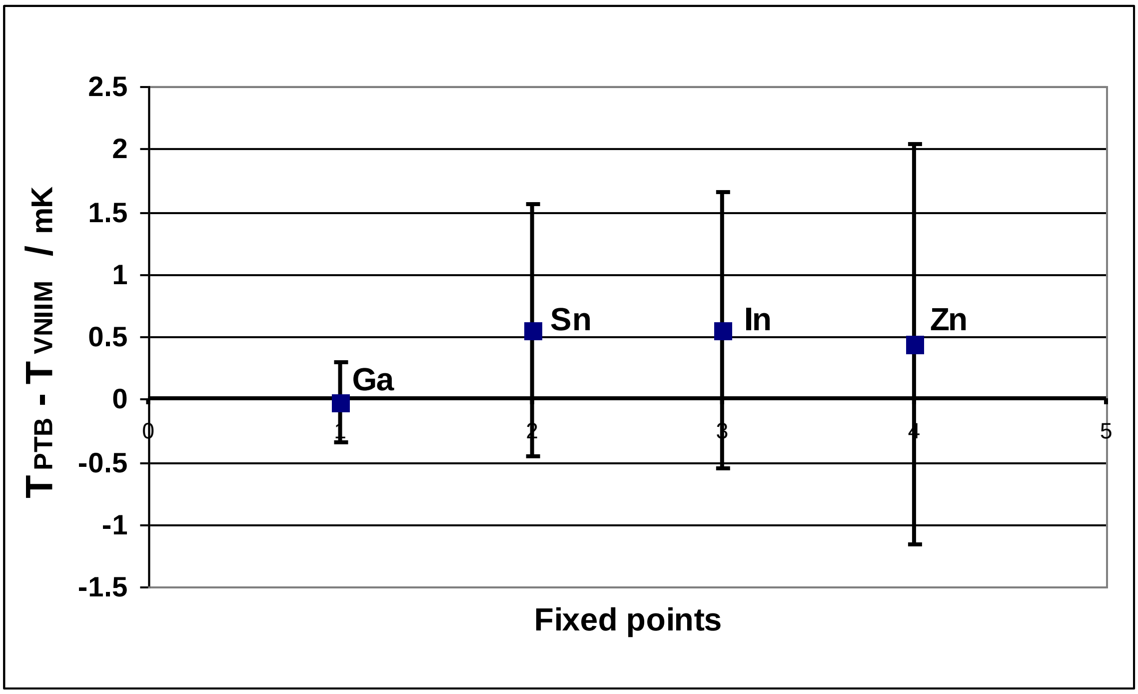


Fig. 1 The differences between temperature values: PTB – VNIIM for the fixed points with their expanded uncertainties ($k$=2).

## Appendix A

**Uncertainty budget** **VNIIM**

| **Uncertainty components** | **mK (*k*=1)** | | | |
|---|---|---|---|---|
| | **Zn** | **Sn** | **In** | **Ga** |
| **Type A** | | | | |
| Repeatability of values, $W_t - u_1$ | 0.1 | 0.1 | 0.08 | 0.05 |
| **Type B** | | | | |
| Chemical impurities, $u_{sie}$ | 0.42 | 0.25 | 0.22 | 0.035 |
| Hydrostatic head, $u_h$ | 0.01 | 0.01 | 0.01 | 0.004 |
| SPRT self-heating, $u_i$ | 0.04 | 0.04 | 0.04 | 0.01 |
| Heat flux, $u_q$ | 0.15 | 0.12 | 0.10 | 0.01 |
| Gas pressure, $u_p$ | 0.001 | 0.001 | 0.001 | 0.001 |
| Bridge non-linearity, $u_r$ | 0.05 | 0.05 | 0.05 | 0.05 |
| Reference resistance temperature, $u_R$ | 0.0001 | 0.0001 | 0.0001 | 0.0001 |
| Reference resistance stability, $u_t$ | 0.0001 | 0.0001 | 0.0001 | 0.0001 |
| Propagated TPW, $u_T$ | 0.064 | 0.045 | 0.037 | 0.025 |
| | | | | |
| Combined uncertainty | 0.47 | 0.30 | 0.26 | 0.084 |
| Expanded uncertainty (*k*=2) | 0.93 | 0.61 | 0.53 | 0.168 |

| **Component of $R_{TPW}$ measurement, type B, mK (*k*=1)** | |
|---|---|
| Purity and isotopic composition | 0,01 |
| Hydrostatic head | 0.002 |
| SPRT self-heating | 0.015 |
| Heat flux | 0.013 |
| | |
| Total uncertainty, $u$(TPW) | 0.022 |

# PTB

## Uncertainty budget for the determination of the *W*-Value of SPRTs

| | Ga | | In | | Sn | | Zn | | |
|---|---|---|---|---|---|---|---|---|---|
| | **mK** | **df** | **mK** | **df** | **mK** | **df** | **mK** | **df** | **Type A or B (*)** |
| Phase Transition Realization Repeatability | 0.18 | 3 | 0.19 | 3 | 0.29 | 4 | 0.29 | 4 | A |
| Bridge (repeatability, non-linearity, AC quadrature) | 0.05 | ∞ | 0.05 | - | 0.05 | ∞ | 0.05 | ∞ | B |
| Reference resistor stability | 0.02 | ∞ | 0.03 | - | 0.03 | ∞ | 0.03 | ∞ | B |
| Chemical Impurities | 0.07 | ∞ | 0.09 | - | 0.15 | ∞ | 0.28 | ∞ | B |
| Hydrostatic-head | 0.01 | ∞ | 0.03 | - | 0.02 | ∞ | 0.03 | ∞ | B |
| Propagated TPW | 0.06 | ∞ | 0.08 | - | 0.10 | ∞ | 0.15 | ∞ | B |
| SPRT self-heating | 0.02 | ∞ | 0.02 | - | 0.03 | ∞ | 0.03 | ∞ | B |
| HeatFlux | 0.10 | ∞ | 0.70 | - | 0.70 | ∞ | 1.00 | ∞ | B |
| Insulation leakage | 0.00 | ∞ | 0.00 | - | 0.00 | ∞ | 0.00 | ∞ | B |
| SPRT Pt Oxydation | 0.00 | ∞ | 0.00 | - | 0.00 | ∞ | 0.00 | ∞ | B |
| Gas pressure | 0.02 | ∞ | 0.05 | - | 0.03 | ∞ | 0.04 | ∞ | B |
| Combined Standard Uncertainty | **0.13** | | **0.43** | | **0.45** | | **0.65** | | |
| Expanded Uncertainty (*k*=2 level, using effective df) | **0.27** | | **0.86** | | **0.90** | | **1.30** | | |

(*) write A or B depending on the

**Appendix B**

## PTB

## Information on fixed-point cells, measurement instruments and furnace/maintenance systems

| **Laboratory** | |
|---|---|
| | |
| Bridge, potentiometer | |
| Manufacturer | ASL |
| Type | F900 |
| | |
| A.C. or D.C. | AC |
| A.C. frequency | 25 Hz |
| D.C. period | |
| | |
| Normal measurement current | 1 mA |
| Self-heating current | 1,41421 mA |
| | |
| Bridge non-linearity | $5 \cdot 10^{-8}$ |
| | |
| Reference resistor | |
| Manufacturer | Tinsley |
| Type | 100 Ohm |
| Temperature control of reference resistor (yes/no) | yes |
| | |
| TPW cell | 1517 |
| Manufacturer | ISOTECH |
| Outer diameter | 50 mm |
| Thermometer well, inner diameter | 11 mm |
| Immersion depth of the middle of the SPRT sensing element | 250 mm |
| Thermostat for maintaining the TPW | ISOTECH |
| | |
| Zn fixed point cell | 326 |
| Manufacturer | ISOTECH |
| Open or closed | closed |
| Outer diameter (crucible container) | 50 mm |
| Thermometer well, inner diameter | 8 mm |
| Metal purity | 6N |
| Immersion depth of the middle of the SPRT sensing element | 175 mm |
| | |
| Furnace for the fixed point of Zn | ITL17703 |
| Manufacturer | ISOTECH |
| Type (1,2 or 3 zones) | 3 zone |
| Freezing plateau duration | 14 h |
| | |

| | |
|---|---|
| Sn fixed point cell | 284 |
| Manufacturer | ISOTECH |
| Open or closed | closed |
| Outer diameter (crucible container) | 50 mm |
| Thermometer well, inner diameter | 8 mm |
| Metal purity | 6N |
| Immersion depth of the middle of the SPRT sensing element | 175 mm |
| | |
| Furnace for the fixed point of Sn | ITLM17701 |
| Manufacturer | ISOTECH |
| Type (1,2 or 3 zones) | 3 zone |
| Freezing plateau duration | 8 h |
| | |
| In fixed point cell | 211 |
| Manufacturer | PTB |
| Open or closed | open |
| Outer diameter | 50 mm |
| Thermometer well, inner diameter | 8 mm |
| Metal purity | 7N |
| Immersion depth of the middle of the SPRT sensing element | 166 mm |
| | |
| Furnace for the fixed point of In | 9114 |
| Manufacturer | Hart Scientific |
| Type (1,2 or 3 zones) | 3 zone |
| Freezing plateau duration | 5 h |
| | |
| Ga fixed point cell | 546 |
| Manufacturer | ISOTECH |
| Open or closed | closed |
| Outer diameter | 38 mm |
| Thermometer well, inner diameter | 12 mm |
| Metal purity | 7N |
| Immersion depth of the middle of the SPRT sensing element | 230 mm |
| | |
| Furnace for the fixed point of Ga | ITL-M-17402B |
| Manufacturer | ISOTECH |
| Type (1,2 or 3 zones) | not specified |

# VNIIM

## Information on fixed-point cells, measurement instruments and furnace/maintenance systems

| Laboratory | VNIIM |
|---|---|
| | |
| Bridge, potentiometer | |
| Manufacturer | |
| Type | ASL |
| | F900 |
| A.C. or D.C. | |
| A.C. frequency | AC |
| D.C. period | 25 Hz |
| | |
| Normal measurement current | 1 mA |
| Overheating current | 1.414 mA |
| | |
| | |
| Reference resistor | |
| Manufacturer | ZIP, RF |
| Type | MC3020 |
| Temperature control of reference resistor (yes/no) | yes |
| | |
| TPW cell | No.0/41 |
| Manufacturer | VNIIM |
| Outer diameter | 50 mm |
| Thermometer well diameter | 11 mm |
| Immersion depth of the middle of the SPRT sensing element | 260 mm |
| Thermostat for maintaining the TPW | Hart Scientific model 7312 |
| | |
| Zn fixed point cell | No.109 |
| Manufacturer | VNIIM |
| Open or closed | open |
| Outer diameter | 52 mm |
| Thermometer well diameter | 8 mm |
| Metal purity | 6N |
| Immersion depth of the middle of the SPRT sensing element | 160 mm |
| | |
| Furnace for the fixed point of Zn | |
| Manufacturer | VNIIM |
| Type (1, 2 or 3 zones) | 3 zones |
| Melting plateau duration | |
| Freezing plateau duration | 10 hours |
| | |
| | |
| Sn fixed point cell | No.30 |
| Manufacturer | VNIIM |
| Open or closed | open |

| | |
|---|---|
| Outer diameter | 52 mm |
| Thermometer well diameter | 12 mm |
| Metal purity | 6N |
| Immersion depth of the middle of the SPRT sensing element | 160 mm |
| | |
| Furnace for the fixed point of Sn | |
| Manufacturer | VNIIM |
| Type (1, 2 or 3 zones) | 3 zones |
| Melting plateau duration | |
| Freezing plateau duration | 11 hours |
| | |
| In fixed point cell | No.1 |
| Manufacturer | VNIIM |
| Open or closed | open |
| Outer diameter | 42 mm |
| Thermometer well diameter | 10 mm |
| Metal purity | 6N |
| Immersion depth of the middle of the SPRT sensing element | 173 mm |
| | |
| Furnace for the fixed point of In | |
| Manufacturer | VNIIM |
| Type (1, 2 or 3 zones) | 3 zones |
| Melting plateau duration | |
| Freezing plateau duration | 12 hours |
| | |
| Ga fixed point cell | No.1 |
| Manufacturer | VNIIM |
| Open or closed | open |
| Outer diameter | 42 mm |
| Thermometer well diameter | 10 mm |
| Metal purity | 7N |
| Immersion depth of the middle of the SPRT sensing element | 167 mm |
| | |
| Furnace for the fixed point of Ga | |
| Manufacturer | VNIIM |
| Type (1, 2 or 3 zones) | 2 zones |
| Melting plateau duration | 17 hours |
| | |

**Appendix C**

# Technical protocol

## Bilateral comparison
**CCT-K9.1**

**Realizations of the ITS-90 from 273.16 K to 692.677 K**
**2018**

Prepared by Prof. A. I. Pokhodun (VNIIM) and Dr. S. Rudtsch (PTB)

## Introduction

The bilateral comparison of the ITS-90 realizations at the National Metrological Institutes PTB (Germany) and VNIIM (Russian Federation) in the temperature range from 273.16 K to 692.677 K was initiated by the Technical Committee on "Thermometry and thermal physics" TC1.10 of COOMET.

VNIIM (RF) is the coordinator of the bilateral comparison.

The purpose of the comparison is to evaluate the differences between the realizations of the national standards in the most essential temperature range with the highest requirements to the measurement uncertainty. This temperature range includes five ITS-90 fixed points: the triple point of water, the melting point of gallium, the freezing points of indium, tin and zinc. Two 25-Ohm Standard Platinum Resistance Thermometers (SPRT) will be used as transfer standards.

The results of the comparisons will be the differences of the temperature values $\Delta T_i$ and the evaluations of the uncertainties $U_i$ for each fixed-point temperature.

Participating laboratories:

| Name of Laboratory | Contact persons | Address |
|---|---|---|
| Physikalisch-Technische Bundesanstalt (PTB)<br>Germany | Dr. S. Rudtsch<br>steffen.rudtsch@ptb.de | Abbestraße, 2–12,<br>10587 Berlin, Germany |
| D. I. Mendeleyev Institute for Metrology (VNIIM)<br>Russian Federation | Prof. Pokhodun A.I.<br>a.i.pokhodun@vniim.ru | Moskovsky pr., 19, 190005,<br>St. Petersburg |

## Comparison schedule

| | | |
|---|---|---|
| 01.2018 | Planned | PTB, VNIIM |
| 02.2018 | First draft of the protocol completed | PTB, VNIIM |
| 03.2018-07.2018 | Review and approval of the protocol | CCT-WG-KC |
| 04.2018-08.2018 | Selection and first calibration of two SPRTs at VNIIM<br>Delivery of the two SPRTs to PTB | VNIIM |
| 08.2018-12.2018 | Calibration of two SPRTs at PTB and return of both SPRTs to VNIIM | PTB |
| 01.2018-03.2019 | Second calibration of the two SPRTs at VNIIM | VNIIM |
| 03.2019 | Delivery of final calibration results and final uncertainty budgets to the heat of CCT-WG-KC (A. Peruzzi) without mutual exchange | VNIIM, PTB |
| 04.2019 -06.2019 | Report in progress. Draft A. | VNIIM, PTB |
| 07.2019-09.2019 | Review of Draft A by CCT-WG-KC | CCT-WG-KC |
| 10.2018 – 12.2019 | Draft B | VNIIM, PTB |
| 01.2020 | Report submitted to CCT WG-KC for final review | VNIIM, PTB |

## Procedures of the comparison

VNIIM will select and calibrate two SPRTs (type ETS-25, made in Russia) at all ITS-90 fixed points in the comparison range and send the SPRTs to PTB. PTB will calibrate these thermometers at all fixed points and send the SPRTs back to VNIIM for the back-calibration.

The differences of the temperature values $\Delta T_i$ and $U_i$ $(\Delta T_i)$ of each fixed point will be calculated according to:

$$\Delta T_i = \frac{W_{PTB} - (W_{VNIIM,before} + W_{VNIIM,after})/2}{dW_i / dT_i} \qquad (1)$$

$$U_i(\Delta T_i) = \sqrt{U_{PTB}^2 + U_{VNIIM}^2} \qquad (2)$$

If the measurements of the thermometer resistances at the triple-point of water indicate a significant change due to mechanical shock the participants might agree on a modified evaluation procedure. After receiving the calibrated SPRTs from VNIIIM and before the calibration at PTB the SPRT resistance will be stabilized by annealing in a certain sequence:

a) Measurement of the SPRT resistance at the TPW

b) The SPRT is inserted into the annealing furnace at a temperature of 500 $^{o}$C,

c) The temperature in the annealing furnace is raised to 675 $^{o}$C,

d) The SPRT is kept in the furnace for two hours,

e) The temperature in the furnace is reduced to 450 $^{o}$C within 3.5 hours,

f) The SPRT is quickly removed from the annealing furnace and cools down (in air) to room temperature.

If the SPRT resistance at the triple point of water after annealing is changed by less than 0.5 mK in the temperature equivalent, it is possible to start its calibration at the fixed points.

If the SPRT resistance at the triple point of water after annealing is changed by 0.5 mK or more in the temperature equivalent, annealing should be repeated.

If after the second annealing the SPRT resistance at the triple point of water is changed by more than 0.5 mK in the temperature equivalent, the SPRT should be replaced.

The best measurement practice accepted at PTB and VNIIM should be used for each fixed-point realization. The resistance ratio $W$ is calculated for each fixed point: $W = R_t/R_{TPW}$, where $R_{TPW}$ is the SPRT resistance at the triple point of water obtained directly after measuring $R_t$. The values of $R_t$ and $R_{TPW}$ should be corrected for self heating due to the measuring current, hydrostatic pressure and pressure in the cell. The calibration cycle must be repeated at least 3 times

**Submission of information**

a) Measurement results of $R_{TPW}$(0 mA) before departure at VNIIM, after arrival and before departure at PTB and after arrival at VNIIM to check the stability during transportation. Further measurement will be carried out before and after each annealing procedure.

b) Measurement results of $W = R_t/R_{TPW}$, where $R_t$ is the ETS-25 resistance at the fixed-point temperatures and $R_{TPW}$ is ETS-25 resistance at the TPW, obtained after $R_t$ measurement, according to Appendix A.

c) Uncertainty budget for each fixed-point according to Appendix B, additional components of uncertainty can be added.

d) Characteristics of the fixed-points cells, furnaces, measuring instruments, used in the comparisons, according to the form in Appendix C.

**Transportation of the transfer standard**

VNIIM will send 2 SPRTs type ETS- 25 (made in Russia) in a suitable transportation box with the designation by large bold type 'This artifact is FRAGILE. It must only be handled by a qualified person', in English, German and Russian languages.

The SPRTs will be selected by VNIIM based on the results of previous investigations (stability, reproducibility).

To comply with customs regulations when crossing state borders the declaration – the Carnet form – must be completed carefully, indicating the recipient country and dates of import and export of the thermometer. VNIIM pays for preparing of «ATACornet» document.

For customs declaration:

The following two artifacts will be sent for an intercomparison with PTB (Germany)SPRT type: ETS-25 and serial number:

The artifact has no commercial value (it is not for sale). It is meant solely for the calibration of national standards and will be reexported immediately after the calibration is complete.

We request that the device is not handled or removed from the container/package.

If a Customs inspection is required then pleasure contact the relevant person so that he/she can be present and help you unpack it.

PTB is responsible for transferring the ETS-25 back to VNIIM, its return and customs registration for import to the Russian Federation and export in accordance with the «ATACarnet» document.

## Linkage to CCT Key Comparison Reference Value and reporting

At the time this protocol was created two options for the linkage were possible, i.e. to the key comparison reference values of CCT-K3 and CCT-K9. VNIIM and PTB were/are both participants of CCT-K3 and CCT-K9.

Whereas the final report of CCT-K3 was issued in February 2002 it is currently not foreseeable when Draft B of CCT-K9 will be available. For several reasons a linkage to CCT-K3 would be unsatisfactory. Instead we assume that Draft B of CCT-K9 will be available at the time when Draft A of this comparison (CCT K9.1) will be prepared.

It is expected that VNIIM and PTB will have comparable measurement uncertainties. Therefore, it is intended to use the results of both institutes to establish the link to CCT-K9.

The deviations between the results of VNIIM and PTB to CCT-K9 will be determined as follows.

The results will be represented as differences $\Delta T_i = [T(NMI_i) - KCRV]$, with associated uncertainties $\boldsymbol{U}_{\mathbf{i}}$ for each fixed point and NMI.

The linkage of the bilateral comparisons results: $NMI_1$, and $NMI_2$, with the results of **K9.1** can be defined using bilateral measurement results of the differences $[T(NMI_1) - T(NMI_2)]$ and the link one of them with results of **K9.1.**

$$\Delta T_1 = [\, T(NMI_1) - KCRV_{K9}] = [T(NMI_1) - T(NMI_2)] + [T(NMI_2) - KCRV_{K9}]$$

$$\Delta T_2 = [\, T(NMI_2) - KCRV_{K9}] = [T(NMI_2) - T(NMI_1)] + [T(NMI_1) - KCRV_{K9}]$$

and the uncertainty of $U(\Delta \boldsymbol{T_i})$

$$U^2(\Delta T_1) = U^2[T(NMI_1) - T(NMI_2)] + U^2[T(NMI_2) - KCRV_{K9}]$$

$$U^2(\Delta T_2) = U^2[T(NMI_2) - T(NMI_1)] + U^2[T(NMI_1) - KCRV_{K9}]$$

Because of the above described constellation this comparison will be carried out in a “double blind” manner. This means that neither VNIIM nor PTB will know the results of the other participants before they have submitted their final results. This is ensured by sending the results to an independent third party, i.e. the head of the CCT-WG-KC (A. Peruzzi). After this point the participants are not allowed to change or correct results. When both NMIs have confirmed that they have provided the final results, the third party will submit all results to the participants for the data evaluation and the common preparation of the Draft A report.