EURAMET TC-T Bilateral comparison SMU – BEV
No. 1429

1 Objective

This comparison is designed to compare the realization of the ITS-90 fixed point cells through the calibration of SPRTs. The fixed point cells covered in this comparison concerns the triple point fixed pointof Hg (234.3156 K). The transfer standards used will be long-stem SPRTs.

The purpose of the bilateral comparison was to validate improved measurement methods and systems, consistent with the results of previous comparisons that the participating NMIs attended (EURAMET.T-K9 andCCT-K3, see Appendix A).

2 Topology of the comparison

The presented comparison will include two national metrological laboratories (MNIs). More specifically, the Slovak Institute of Metrology (SMU) and Bundesamt für Eich- und Vermessungswesen (BEV). The comparison presented due to this fact is bilateral by nature. The scheme of the comparison is presented in the following figure 1.

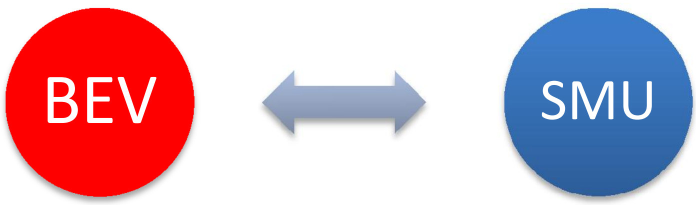


Figure 1: Pictorial view of the comparison topology with pilots and other participants.

3. Participants

- Participant (coordinator) 1: **SMU** (SLOVAKIA)
- Participant 2: **BEV** (AUSTRIA)

4. Projected Timeline

Protocol Agreement	**Jan, 2018**
Transfer Standards Measurements at BEV	**Feb, 2018**
Transfer Standards Measurements at SMU	**March, 2018**
Final Report Completed	**March, 2019**

5. Duties of the participants

Each participant will provide the following:

- ITS-90 calibrated SPRT:
  SPRT will be measured at the triple point of water and Hg, and at the melting point of Ga (the Ga point measurements purpose is to check the purity of platinum of the selected SPRT according to the ITS-90 recommendations). Measurements in both participants laboratories will be done by the respected home organization.

  - Coordinator will select his own SPRT based on his own criteria for suitability

  - Coordinator will calibrate the selected SPRT within the MP-Ga (melting point of gallium), TP-W (triple point of water)and TP-Hg(triple point of mercury) at home NMI,

  - Participant 2will calibrate the selected SPRT within the TP-W (triple point of water)and TP-Hg(triple point of mercury) at hosting NMI,

- Calibration results supplied in $\overline{W}(FP)$ for each Fixed Point (FP) with all corrections applied by the NMI such that the $\overline{W}(FP)$ values are equivalent to the ITS-90 assigned temperature values for 0 mA. Uncertainties, $u(\overline{W})$, may be specific to each SPRT.

  - Appendix A gives a reporting worksheet

- The measurement equation used to compute each calibration result with an indication of which inputs vary randomly for each realized equilibrium and which inputs are systematic across all equilibriums for each fixed point within this comparison
  - Any quantities in the measurement equation that are a mixture of random and systematiceffects for each SPRT should be broken into constituent parts that are either purely random or purely systematic within this comparison.

  - An example of an SPRT measurement is given in Appendix B

- Uncertainty budget compliant with CCT WG3 excluding the degrees of freedom associated to each component

  - A template for the uncertainty budget is given in Appendix C
  - Sources of uncertainty may be added or deleted as needed
  - An NMI/DI may choose to supply their own uncertainty budget (CMC and WG3 compliant) excludingthe degrees of freedom for each source of uncertainty

(NMI/DI are encouraged to use the template, but if it prefers it may supply its own, taking care to not forget any uncertainty components (for this point, the suggested budget can be a help)

- Heat Flux (Immersion) profile for each fixed-point cell using the SPRTs of this comparison
  - [*R*(FP), 0 mA] and corresponding [immersion depth (sensor midpoint), cm]
- All results and required information will be e-mailed sheared between the coordinator and participants.

6. Duties of the coordinator (SMU):

- To prepare the protocol
- To send the protocol to all the participants for agreement
- To register the comparison and to keep contact with EURAMET
- To receive all the results of measurements
- To write the draft A and draft B, using the entries fromparticipants and with the help of the participants.
- To exchange with all the participants concerning draft A and draft B.

7. Duties of the participants:

- To liaise with each other regarding receipt and return of the SPRT
- To check the initial value of *R*(TPW) of the SPRT delivered by each participant. In the case of significant discrepancy, to consult with the participant and/or the coordinator before proceeding with the calibration
- To carry out the calibration of the SPRT using the appropriate equipment that will make possible to deliver the best possible measurement uncertainty and wont put the used SPRT at risk
- To prepare tables of results and uncertainties for the coordinator

8. Other instructions

- **Advice on handling the SPRTs:** The SPRT must be handled with care and only by qualified metrology personnel. Hand-carriage of the SPRT from the participant lab to the pilot lab and vice versa is recommended and under the responsibility (and costs) of the participant

- **Instructions for reporting the results:** The templates in Appendices A, B and C should be used for reporting the results

- **Timetable for communicating the results to the pilot:** The participants must report the results of their measurements to the coordinator

- **Financial aspects of the comparison:** each participating institute is responsible for its own costs for the measurements and transport of the SPRTs to and back from the pilot laboratory.

9. Conclusion:

The conducted bilateral comparison of two triplepoint cells of Hg realized by a SPRT transfer standard was considered as satisfactory. This is based on the measured data that has shown an agreement of the realized values of the two fixed point cells within the measurement uncertainty of each involved partner (BEV, SMU). The transfer standard used has shown compliance with the platinum purity based on the W values measured in melting point of Ga and Hg set by the ITS-90. Furthermore the SPRT has shown an acceptable level of stability that was 0,11 mK.

Appendix A: Measurement Reporting Worksheet

Participating NMI/DI

BEV (participant NMI), SMU (home NMI)

Measurements performed by BEV

On Receipt *R*(TPW) 25,53787083

	$\overline{W}(FP)$	$u[\overline{W}(FP)], mK$	n (*)	Comments (if any)
Gallium	1,118113758	1.1	1	This W values purpose is to check that the SPRT is suitable according the ITS-90 and meets the condition *W*(29,7646 °C) ≥ 1,118 070
Mercury	0,84415761	0,46	1	This *W* values purpose is the subject of the bilateral comparison and furthermore to check that the SPRT is suitable according the ITS-90 and meets the condition *W*(-38,8344 °C) ≤ 0,844 235

(*)n, Number of equilibria realized

Final *R*(TPW) 25,53786054

TPW Uncertainty: 0,35 mK(*k*=2)

Measurements performed by SMU

On Receipt R(TPW) 25,53798948

	$\overline{W}(FP)$	$u[\overline{W}(FP)], mK$	n(*)	Comments (if any)
Gallium	1,118 115 369	0,22	1	This W values purpose is to check that the SPRT is suitable according the ITS-90 and meets the condition W(29,7646 °C) ≥ 1,118 070
Mercury	0,84416083	0,50	4	This W values purpose is the subject of the bilateral comparison and furthermore to check that the SPRT is suitable according the ITS-90 and meets the condition W(-38,8344 °C) ≤ 0,844 235

(*)n, Number of equilibria realized

Final R(TPW) 25,53798608

TPW Uncertainty: 0,15 mK (k=2)

Fixed-Point Cell Information

	Type /manufacturer/serial number	L (cm) (*)	Open or sealed cell	Traceability
Gallium (BEV)	Modell 5943, Fluke/Hart Scientific, Serial No. Ga-43139, MM-Nr. MM003319	16,8	Sealed	EUROMET. T-K3
Mercury (BEV)	Type ITL-M-17924, Isotech, Serial No. 010294 mi, MM-Nr. MM003324	16	Sealed	EUROMET. T-K3
Gallium (SMU)	Open fixed point cell cell/SMU/Ga-97B, SMU, Ga-97B	26	Open	CCT-K3
Mercury (SMU)	Type ITL-M-17724, Isotech, Serial No. Hg 216	15	Sealed	CCT-K3

(*)Maximum thermometer Immersion depth into the substance measured to the thermal centre of the SPRT sensing element, cm

Measurement System

Resistance Ratio Bridge Model (BEV)	Fluke Super-Thermometer 1594A, S No B27129 (MM002765)
Reference Resistor Model (BEV)	MM003246,S No. 5685A26975, 25Ω
Resistor Enclosure Stability (BEV), mK	1K (*Resistor is enclosure by a precise constant temperature bath, withset-point accuracy of ±1 °C and typical short and long term temperature stability at 25 °C of ± 0,001 °C.*)
Resistance Ratio Bridge Model (SMU)	ASL F18, S No. 756-5034
Reference Resistor Model (SMU)	Tinsley 25Ω
Resistor Enclosure Stability (SMU), mK	17 mK (*active compensation for temp. drift applied*)
SPRT transfer standard (SMU)	Fluke SPRT 5681, s/n: 1875

***R*(TPW) values during the calibration process:**

All the R(TPW) values and the moment when they are measured according to the measurements at the other fixed points (gallium, mercury) are requested, in a table.

Fixed point	R	R (TPW)
Ga*	BEV - 28,55424473 Ω*	BEV - 25,53787083 Ω*
	SMU - 28,554 455 44 Ω*	SMU - 25,538 015 66 Ω*
Hg	BEV -21,558 542 66 Ω	BEV - 25,537 860 53 Ω
	SMU - 21,558 486 04 Ω	SMU -25,53798948 Ω
	SMU -21,558 498 90 Ω	SMU -25,537 999 48 Ω
	SMU -21,558 480 08 Ω	SMU -25,538 000 21 Ω
	SMU -21,558 473 23 Ω	SMU -25,537 968 58 Ω

***Measured values are only used for the platinum purity check according to the ITS-90**

SPRT stability:

The selected SPRTs stability was evaluated based on the measurements in the TPW. The$R$(TPW) values used to evaluate this parameter were taken from the measurements prior and after the measurement in the Ga and Hg points were made. Based on this evaluation the stability of the SPRT was better than 0,11 mK.

Appendix B: Corrections applied to the measured values

The ratio value $W(FP_i)$ at the fixed point FP_i is given by the ratio of the SPRT resistance $R(FP_i)$ at thefixed point FP_i to the SPRT resistance R(TPW) at the triple point of water. The SPRT resistance measured at the fixed point FP_i(and the SPRT resistance R(TPW) measured at the triple point of water) must be corrected for the self-heating effect, the hydrostatic head effect and the residual gas pressure effect (see respectively section 2.1, 2.2 and 8.4 of CCT/08-19-rev document on “Uncertainties in the Realization of the SPRT Subranges of the ITS-90”).

With:
$R_{meas}(FP_i)$ the resistance measured at fixed point FP_i
ΔR_{sh} the self-heating correction to the resistance
ΔR_{hydr} the hydrostatic head correction to the resistance
ΔR_p the residual gas pressure correction to the resistance
$R(FP_i)$ is computed as:

$$R(FP_i) = R_{meas}(FP_i) + \Delta R_{sh} + \Delta R_{hydr} + \Delta R_p$$

The applied corrections can be expressed in terms of temperature by simply dividing each one of them by the SPRT sensitivity coefficient $(dR/dT)_{FPi}$ at the temperature of the fixed point FP_i:

$$\Delta T_{sh} = \Delta R_{sh}/(dR/dT)_{FPi}$$

$\Delta T_{hydr} = \Delta R_{hydr}/(dR/dT)_{FPi}$
$\Delta T_{p} = \Delta R_{p}/(dR/dT)_{FPi}$

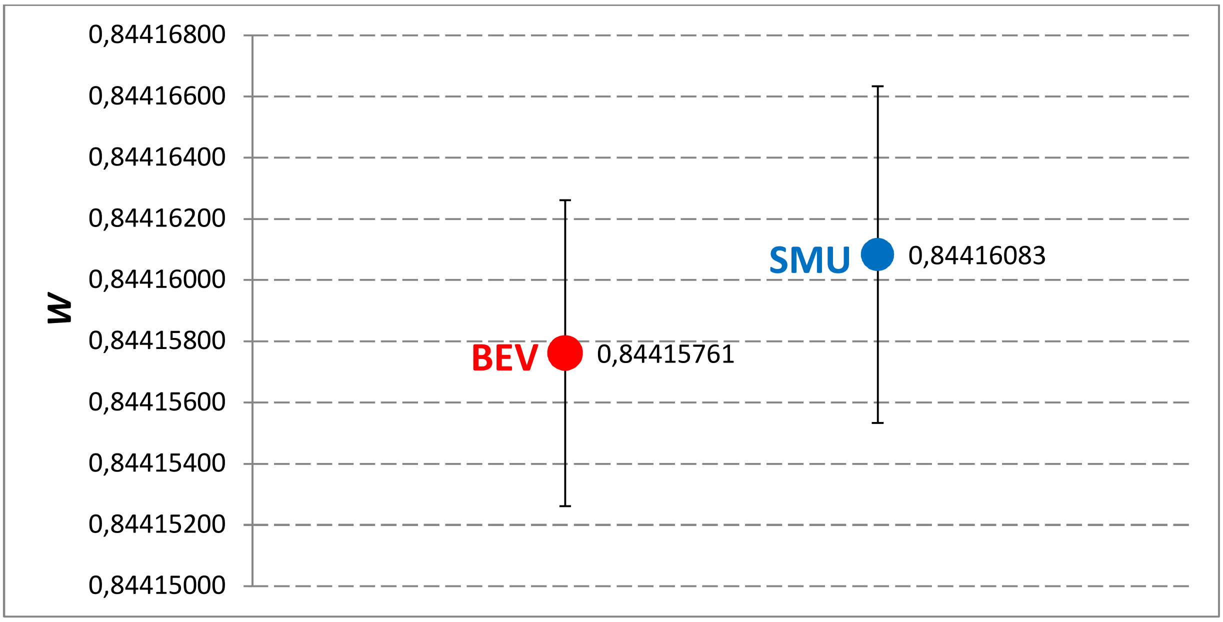
The applied corrections, expressed in terms of temperature, should be reported by the participants using the two tables below.

Corrections applied during measurements at BEV

	self-heating		Hydrostatic and heat-flux		pressure	
	correction mK	$u_{correction}$ mK	correction mK	$u_{correction}$ mK	correction mK	$u_{correction}$ mK
$\overline{R}$ (Hg)	0,350	0,200	1,244	0,100	0,017	0,010

Corrections applied during measurements at SMU

	self-heating		Hydrostatic and heat-flux		pressure	
	correction mK	$u_{correction}$ mK	correction mK	$u_{correction}$ mK	correction mK	$u_{correction}$ mK
$\overline{R}$ (Hg)	0,310	0,178	1,560	0,270	0,020	0,011



Appendix C: Suggested Uncertainty Budget for the Determination of the *W*-Value of an SPRT

Participating NMI | BEV

	Hg mK	df	Type A or B (*)
Phase Transition Realization Repeatability	0,024	59	A
Bridge (repeatability, non-linearity, AC quadrature)	0,016	29	B
Reference resistor stability	0,048	59	B
Chemical Impurities	0,026	59	B
Hydrostatic-head	0,041	24	B
Propagated TPW	0,120	19	B
SPRT self-heating	0,179	24	B
(522 perturbing)Heat Flux	0,027	19	B
Insulation leakage	0,005	49	B
SPRT Pt Oxydation	0,006	19	B
Gas pressure	0,010	59	B

Combined Standard Uncertainty	0,229
Effective degrees of freedom	51
Expanded Uncertainty (k=2 level)	**0,46**

(*) write A or B depending on the method used

Participating NMI | SMU

	Hg mK	df	Type A or B (*)
Phase Transition Realization Repeatability	0,021	11	A
Bridge (repeatability, non-linearity, AC quadrature)	0,012	19	B
Reference resistor stability	0,010	49	B
Chemical Impurities	0,038	34	B
Hydrostatic-head	0,061	19	B
Propagated TPW	0,052	24	B
SPRT self-heating	0,200	24	B
(522 perturbing)Heat Flux	0,121	24	B
Insulation leakage	0,005	49	B
SPRT Pt Oxydation	0,012	11	B
Gas pressure	0,011	14	B

Combined Standard Uncertainty	0,252
Effective degrees of freedom	52
Expanded Uncertainty (*k*=2 level)	**0,50**

(*) write A or B depending on the method used

Appendix D:

Name, postal and e-mail addresses of the participants:

Slovakia	SMU	Peter Pavlasek	pavlasek@smu.gov.sk
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Name, postal and e-mail addresses of the pilots

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Bundesamt für Eich- und Vermessungswesen Arltgasse 35 1160 Wien AUSTRIA			