EURAMET.T-K9 final protocol

Regional key comparison

ITS-90 SPRT Calibration from the Ar TP to the Zn FP

1 Objective

This comparison is designed to compare the realization of the ITS-90 through the calibration of SPRTs. The range of temperature covered in this comparison is from the triple point of Ar (83.8058 K) to the freezing point of Zn (692.677 K). The transfer standards used will be long-stem SPRTs.

This protocol matches closely the corresponding CCT-K9 key comparison protocol.

2 Topology of the comparison

The comparison is organized in five separate collapsed stars, each star having a CCT-K9 participant as pilot linking the measurements of 4 to 6 other participants (see Figure 1). LNE-CNAM was chosen as coordinator of the comparison and charged with the preparation of the protocol, the collection of the measurement reports from all participants and the preparation of the final report.

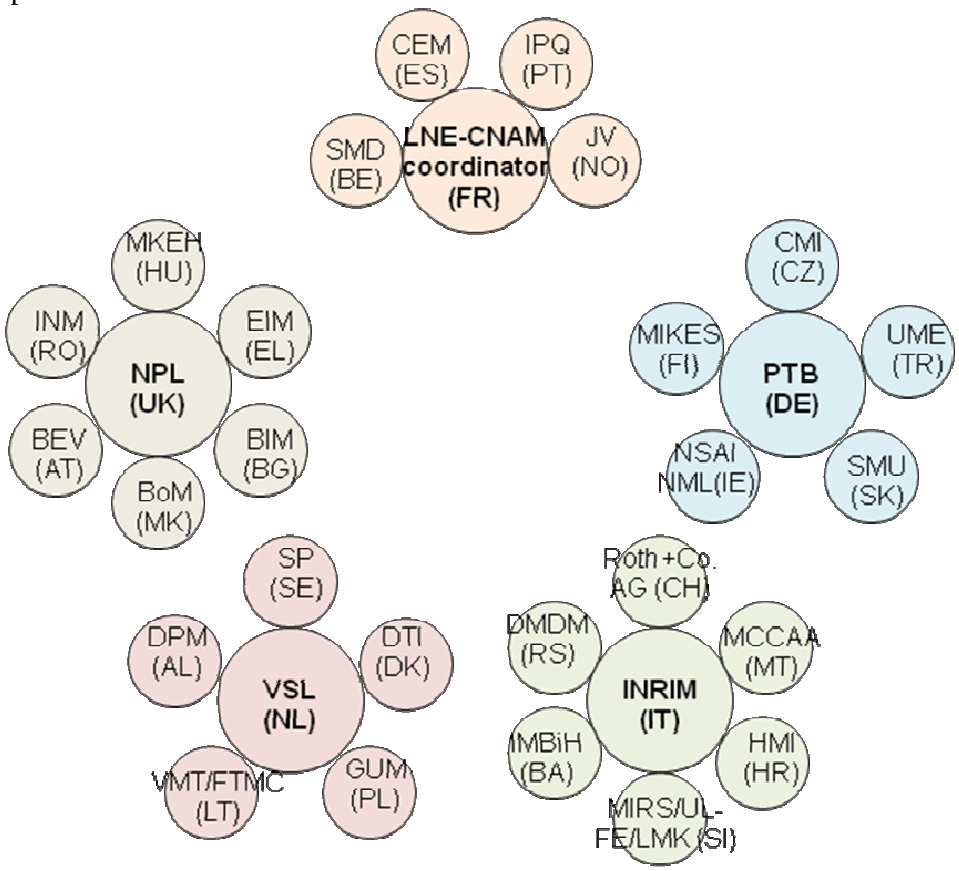

Figure 1: Pictorial view of the comparison topology with pilots and other participants.

3. Participants
The pilots of the 5 stars are:

- Star 1: LNE-Cnam (France)
- Star 2: PTB (Germany)
- Star 3: INRIM (Italy)
- Star 4: VSL (Netherlands)
- Star 5: NPL (United Kingdom)

The names, postal and e-mail addresses of the pilots are given in appendix D.
The other participants in each star are listed in appendix E.
The names, postal and e-mail addresses of the participants are given in appendix F.

4. Projected Timeline

Protocol Agreement	February, 2015
Transfer Standards Sent to the pilots	June, 2015
Transfer Standards Returned to NMIs	December, 2015
Transfer Standards Re-Measured by NMIs	June, 2016
Draft A Report Completed	December, 2016

5. Duties of the participants
Each participant will provide the following:

- 2 ITS-90 calibrated SPRTs
  - The participant will select his own SPRTs based on his own criteria for suitability and will convey the selection criteria to the Pilot Laboratory in his star,
  - The participant will calibrate the two selected SPRTs,
    After calibrating the two selected SPRTs, the participant will deliver one of the two calibrated SPRTs to the pilot of his star.

(here is the slight difference with the CCT-K9 protocol. A second thermometer is prepared and immediately kept at disposal in case of trouble with the transfer standard, particularly in case of carrier problems)

  - Each participant will repossess their thermometer and will make a re-calibration.
  - SPRTs are to be measured at every available fixed-point over the range of the comparison including the In FP and Ga MP

- 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 systematic effects 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 that includes 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) that includes 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 to the pilots and to the coordinator.

If you have questions about any aspect of the protocol or if you are not sure how to report something that is requested, please contact the coordinator prior to submitting your report. After reviewing all submitted reports, the pilots and/or coordinator will contact you if there is anything that is unclear or if any additional information is needed to complete the analysis of the data.

Note:
The thermometer numbers are assigned to NMI/DI SPRTs by loop and arrival date.

Example:
Third thermometer received in loop 4: L43 (each pilot is in charge to identify the laboratory associated with the thermometer L43, and to give to the laboratory the same name that the name of the thermometer)
The pilot in loop 4 is named P4. So the thermometer L43 is belonging to the participant 3 in loop 4.
It leads to a given number of Pi (i=1 to 5) and Lij (i=1 to 5), (j=1 to x depending of the number of participants in a loop)

In case the second thermometer is needed, the letter S will be added to the code. Example L43S will be the second thermometer sent by the participant 3 to the pilot of the loop 4.

6. Duties of the coordinator:

- To identify the participants in each loop
- To prepare the protocol (with the help of the pilots)
- To send the protocol to all the participants for agreement
- To register the comparison and to keep contact with CCT and EURAMET at the different stages of the comparison
- To be informed of the state of progress of the comparison in the different stars / to communicate with the pilots (and participants) in case of delays or any other issues.
- To receive all the results of measurements, but the pilots will exploit the measurements made in their loop.
- To write the draft A and draft B, using the entries of the pilots and with the help of the pilots.
- To exchange with all the participants concerning draft A and draft B.

7. Duties of the pilots:

- To liaise with NMI/DI participants in their star regarding receipt and return of the SPRTs
- To check the initial value of R (TPW) of the SPRT delivered by each participant, before and after annealing, against the final value reported 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 SPRTs using the same equipment as was used in CCT-K9, or equipment which is known to be compatible with that equipment. It is recommended that one of the SPRTs used by the Pilot in CCT-K9 is also included in the calibration experiments
- To prepare tables of results and uncertainties for the Pilot's calibration of the Participants' SPRTs in accordance with Appendices A, B and C, and to calculate the differences $\Delta T(Lij - Pi)$ and uncertainties $u(Lij - Pi)$ (see below)
- To send this information to the Coordinator

8. Other instructions

- **Advice on handling the SPRTs:** The SPRTs 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 viceversa 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 pilot at the time they deliver the SPRT to the pilot. In case the results of the recalibration (measurements performed after the return of the SPRT from the pilot lab) significantly deviates from the initial measurements, the participant must notify the pilot lab and deliver the recalibration measurements not later than the end of December 2015.

- **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. Method of Analysis and link to the CCT-K9 KCRV value.

For the EURAMET.T-K9 comparison, for each fixed points, and each NMI/DI, the fixed-point realization temperature differences from the KCRV and the associated uncertainty will be calculated using the following equations:

For each laboratory *Lij* in star i

$$\Delta T_{(Lij-KCRV)} = \Delta T_{(Lij-Pi)} + \Delta T_{(Pi-KCRV)}$$

$$u^2_{(Lij-KCRV)} = u^2_{(Lij-Pi)} + u^2_{(Pi-KCRV)}$$

The two values, $\Delta T_{Pi-KCRV}$ and $u_{Pi-KCRV}$ will be available from the CCT-K9 report.

$$\Delta T_{(Lij-Pi)} = \frac{[\overline{W}(FP)_{Lij} - \overline{W}(FP)_{Pi}]}{dW_r / dT} + C_{Lij}$$

C_{Lij} is a term used to account for uncertainty associated with the travel, handling, or stability of the SPRT and is taken to have a value of $C_{Lij} = 0$ and a standard uncertainty, $u_{C_{Lij}}$, of

$$u_{C_{Lij}} = \frac{abs\,[\overline{W}(FP)_{Lijbefore} - \overline{W}(FP)_{Lijafter}]}{(dW_r / dT).\sqrt{12}}$$

Appendix A: Measurement Reporting Worksheet

Participating NMI/DI

Before sending SPRTs to Pilot Laboratory

	$\overline{W}(FP)$	$u[\overline{W}(FP)], mK$	n (*)	Comments (if any)
Zinc				
Tin				
Indium				
Gallium				
Mercury				
Argon				

(*)*n, Number of equilibria realized*

Final *R*(TPW) | TPW Uncertainty:

On return to participating NMI

On Receipt *R*(TPW)

	$\overline{W}(FP)$	$u[\overline{W}(FP)], mK$	n (*)	Comments (if any)
Zinc				
Tin				
Indium				
Gallium				
Mercury				
Argon				

(*)*n, Number of equilibria realized*

Final *R*(TPW)

Fixed-Point Cell Information

	Type /manufacturer/serial number	L (cm) (*)	Open or sealed cell	Traceability
Zinc				
Tin				
Indium				
Gallium				
Mercury				
Argon				

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

Measurement System

Resistance Ratio Bridge Model	
Reference Resistor Model	
Resistor Enclosure Stability, mK	

R(WTP) values during the calibration process:

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

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 the fixed 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.

Before sending SPRTs to Pilot Laboratory

	self-heating		**hydrostatic**		**pressure**	
	correction mK	$u_{correction}$ mK	correction mK	$u_{correction}$ mK	correction mK	$u_{correction}$ mK
$\overline{R}$ (Zn)						
$\overline{R}$ (Sn)						
$\overline{R}$ (In)						
$\overline{R}$ (Ga)						
$\overline{R}$ (Hg)						
$\overline{R}$ (Ar)						

After sending SPRT to Pilot Laboratory

	self-heating		**hydrostatic**		**pressure**	
	correction mK	$u_{correction}$ mK	correction mK	$u_{correction}$ mK	correction mK	$u_{correction}$ mK
$\overline{R}$ (Zn)						
$\overline{R}$ (Sn)						
$\overline{R}$ (In)						
$\overline{R}$ (Ga)						
$\overline{R}$ (Hg)						
$\overline{R}$ (Ar)						

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

Participating NMI

	Ar		Hg		Ga		In		Sn		Zn		
	mK	df	mK	df	mK	df	mK	df	mK	df	mK	df	Type A or B (*)
Phase Transition Realization Repeatability													
Bridge (repeatability, non-linearity, AC quadrature)													
Reference resistor stability													
Chemical Impurities													
Hydrostatic-head													
Propagated TPW													
SPRT self-heating													
Heat Flux													
Insulation leakage													
SPRT Pt Oxydation													
Gas pressure													
Combined Standard Uncertainty													
Expanded Uncertainty (*k*=2 level, using effective df)													

(*) write A or B depending on the method used
df: degree of freedom

Appendix D:

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

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Name, postal and e-mail addresses of the pilots

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Appendix E:
NMi/DI participants in each loop.

Loop 1: LNE-Cnam (France)	Loop 2: PTB (Germany)	Loop 3: INRIM (Italy)	Loop 4: VSL (Netherlands)	Loop 5: NPL (United Kingdom)
	MIKES (Finland)	Roth+Co.AG (Switzerland)	SP (Sweden)	MKEH (Hungary)
SMD (Belgium)	CMI (Czech Republic)	MCCAA (Malta)	DTI (Denmark)	EIM (Greece)
CEM (Spain)	UME (Turkey)	HMI (Croatia)	GUM (Poland)	BIM (Bulgaria)
IPQ (Portugal)	SMU (Slovakia)	MIRS/UL-FE/LMK (Slovenia)	VMT/FTMC (Lithuania)	BoM (FYR Macedonia)
JV (Norway)	NSAI NML (Ireland)	IMBiH (Bosnia-Herzegovina)	DPM (Albania)	BEV (Austria)
		DMDM (Serbia)		INM (Romania)

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

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