GULFMET. T-K9 draft protocol

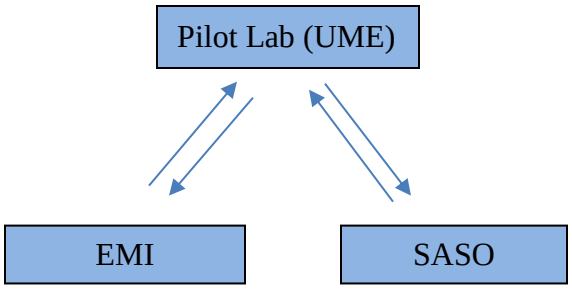
Regional key comparison

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

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 Hg (234.3156 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.

Method:
Since the number of the participants is very small (three including the pilot), the comparison will have a star structure with two arms. See the sketch below:



Pilot Lab: UME (Turkey)
Participants: EMI (United Arab Emirates), SASO (Saudi Arabia)

Coordinator:
Kalemci, Murat (UME)

The name, postal and e-mail addresses of the participants are given in appendix D

Projected Timeline:

Protocol Agreement:	November, 2016
Transfer Standards Sent to the pilot:	May, 2017
Transfer Standards Returned to NMIs:	July, 2017
Transfer Standards Re-Measured by NMIs:	September, 2017
Draft A Report Completed:	December, 2017

Participants will supply the following:

- 2 ITS-90 calibrated SPRTs

    - NMI participant will select their own SPRTs based on their own criteria for suitability and will convey the selection criteria to the Pilot Laboratory.
    - Two SPRTs must be calibrated by NMI participant before measurements are made by the Pilot Laboratory.
    - One SPRT will be carried to the Pilot Laboratory for calibration.

(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

- Prior to calibration an annealing procedure for both thermometers has to be performed as follows:
    - Determine the R_{TPW} value of the thermometer before annealing.
    - Carefully insert the SPRT into a furnace at 480 °C.
    - Anneal the SPRT for two hours at 480 °C.
    - Carefully remove the SPRT from the furnace directly to the room environment.
    - Re-determine the value of $R_{TPW.}$
    - If the resistance at TPW increases after annealing, consider using another thermometer for the comparison.
    - If the resistance at TPW of the SPRT is decreasing by an equivalent of 0.5 mK or greater, proceed to a second annealing procedure.
    - If the resistance decrease is less than an equivalent of 0.5 mK, proceed with the calibration.
- 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 equilibria 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
  - Please identify which components of the uncertainty budget are associated with random effects in $\bar{W}(FP)$ and which are associated with systematic effects in $\bar{W}(FP)$ within this comparison.

(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).
For information about uncertainties in the calibration of SPRTs, you could refer to the document "Uncertainties in the Realisation of the SPRT Subranges of the ITS-90", prepared by CCT-WG3, which can be downloaded from the following internet link:
http://www.bipm.org/cc/CCT/Allowed/24/D19_rev_WG3_Doc_rev_10July2009.pdf

- 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 pilot 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 pilot /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 number is assigned to the NMI SPRT by arrival date. L1 will be the thermometer first arrived to the pilot and L2 will be the second. In case the second thermometer is needed from some lab, the letter S will be added to the initial code (i.e. L1S).

Role of the coordinator / pilot:

- To prepare a protocol
- To send the protocol to all the participants for agreement
- To register the comparison and to keep contact with CCT and GULFMET at the different steps of the comparison.
- To be informed of the state of progress of the comparison and communicate with the participants in case of delays or any other issues.
- To liaise with NMIs participants regarding receipt and return of the SPRTs.
- To check their initial value of R (TPW), before and after annealing, against the final value reported by the participant. In the case of significant discrepancy, to consult with the participant before proceeding with the calibration.
- To receive all the results of measurements from the labs.

- To carry out the calibration of the SPRTs using the same equipment as was used in EURAMET.T-K9. It is necessary that one of the SPRTs used by the Pilot in EURAMET.T-K9 is also included in the calibration experiments.
- To prepare tables of results and uncertainties for the calibration of the Participants SPRTs in accordance with Appendices A, B and C, and to calculate the differences $\Delta T_{(Lj-P)}$ and uncertainties $u_{(Lj-P)}$ (see next paragraph)
- To write the draft A and draft B (using the entries of the labs)
- To exchange with the participants concerning draft A and draft B.

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

For the GULFMET.T-K9 comparison, for each fixed point, and each NMI/DI, the fixed-point realization temperature differences from the CCT KCRV and the associated uncertainty will be calculated using the deviation of each lab from the pilot lab, the deviation of the pilot lab to EURAMET KCRV and the deviation of EURAMET KCRV to CCT KCRV, following equations:

For each laboratory *Lj* in the comparison

$$\Delta T_{(Lj-KCRV)} = \Delta T_{(Lj-P)} + \Delta T_{(P-EURAMET.KCRV)} + \Delta T_{(EURAMET.KCRV-CCT.KCRV)}$$

$$u^2_{(Lj-KCRV)} = u^2_{(Lj-P)} + u^2_{(P-EURAMET.KCRV)} + u^2_{(EURAMET.KCRV-CCT.KCRV)}$$

The four values, $\Delta T_{P-EURAMET.KCRV}$, $\Delta T_{EURAMET.KCRV-CCT.KCRV}$, $u_{P-EURAMET.KCRV}$ and $u_{EURAMET.KCRV-CCT.KCRV}$ will be available from the EURAMET-K9 report and:

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

C_{Lj} 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 $C_{Lj} = 0$ and a standard uncertainty, $u_{C_{Lj}}$, of

$$u_{C_{Lj}} = \frac{abs[\overline{W}(FP)_{Ljbefore} - \overline{W}(FP)_{Ljafter}]}{(dW_r / dT).\sqrt{12}}$$

Appendix A: Measurement Reporting Worksheet

Participating NMI/DI

Before sending SPRTs to Pilot Laboratory

Initial R(TPW), after annealing

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

(*)*n, Number of equilibria realized*

Final *R*(TPW)

On return to participating NMI

On Receipt *R*(TPW), after annealing

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

(*)*n, Number of equilibria realized*

Final *R*(TPW)

Fixed-Point Cell Information

	Type /manufacturer	L (cm) (*)
Zinc		
Tin		
Indium		
Gallium		
Mercury		

(*)*Maximum thermometer Immersion depth into the substance, cm. Report the distance between the surface of the metal and the bottom of the thermometer well of the cell.*

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), are requested, in a table.

Appendix B: Example of an SPRT measurement

To obtain the value of W(FP) at the ITS-90 fixed point, corrections must be applied to the measured values, Wmeas(FP), as follows.

W(FP) = Wmeas(FP) + Cp + Ci,

where

Cp is the gas pressure correction multiplied by (dT/dp)(dW/dT),
Ci is the immersion depth correction multiplied by (dT/dh)(dW/dT)

Before sending SPRTs to Pilot Laboratory

	pressure		**immersion**	
	correction mK	$u_{correction}$ mK	correction mK	$u_{correction}$ mK
W(Zn)				
W(Sn)				
W(In)				
W(Ga)				
W(Hg)				

After sending SPRT to Pilot Laboratory

	pressure		**immersion**	
	correction mK	$u_{correction}$ mK	correction mK	$u_{correction}$ mK
W(Zn)				
W(Sn)				
W(In)				
W(Ga)				
W(Hg)				

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

Participating NMI

	Hg		Ga		In		Sn		Zn		
	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											
Moisture											
SPRT Pt Oxidation											
Gas pressure											
Slope of Plateau											
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 pilot and participants.

Turkey	UME	Kalemci, Murat murat.kalemci@tubitak.gov.tr	Uytun, Ali ali.uytun@tubitak.gov.tr
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