# REPORT to the CCT on COOMET Comparison (COOMET theme N°. 417/UA-a/08) COOMET.T-K3.1

# Key Regional Comparison of the National Standards of Temperature in the Range from the Triple Point of Water to the Freezing Point of Zinc

Prepared by

A.I. Pokhodun

D.I. Mendeleyev Institute for Metrology (VNIIM)

19, Moskovskii pr., 190005, St.Petersburg, Russia

Prof. A.I. Pokhodun (coordinator)

fax: 107 812 713 01 14

phone: 107 812 315 52 07

e-mail: A.I.Pokhodun@vniim.ru

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#### 1 Introduction

Adoption of the international Arrangement on Mutual Recognition of the National Standards, Calibration and Measurement Certificates Issued by the National Metrology Institutes, which was signed by the majority of countries joining COOMET, was an incitement to perform regional comparisons within COOMET.

The purpose of regional key comparisons is dissemination of the metrological equivalence to the standards of the national metrology institutes, which do not participate in the BIPM key comparisons. The degree of equivalence of the measurement standards of the NMI's participating in the regional comparisons is determined relative to the reference value of the BIPM key comparisons through the measurement results received in the linking national metrology institutes, which take part in both comparisons.

The first COOMET comparison was "Comparison of the ITS-90 realizations in the range from 0.01 °C to 429.7485 °C (from the triple point of water to the freezing point of zinc", N°. 227/RU/01, 229/UA/01, registration in the KCDB "COOMET.T-K3". The comparisons supported the following CMC items: 1.1.1, 1.3.1, 1.3.2, 2.2.2, 2.3.1.

Four NMI's took part in the COOMET regional comparisons: VNIIM (Russian Federation), SMU (Slovakia), BelGIM (Republic of Belarus), NSC IM (Ukraine). The two of four participants: VNIIM (RF) and SMU (Slovakia) also participated in the key comparisons K3 and K4. They played the role of linking institutes in the COOMET comparison to disseminate the metrological equivalence to the measurement standards of the NSC IM (Ukraine) and BelGIM (Republic of Belarus).

The comparisons had two stages. At the first stage from August, 2005, to September, 2005, the cells of the main fixed points of the national metrology institutes were compared at VNIIM; at the second stage from March, 2006, to February, 2007, the national realizations of the main fixed points were compared using a SPRT as a transfer standard.

The NSC IM recalled its results of the second stage of the comparisons by an official letter of September, 2007. So, the final report of the comparisons в заключительном отчете по сличению (Theme No. 227/RU/01, 229/UA/01, registration in the KCDB "COOMET.T-K3") does not contain the comparison results of the second stage from the NSC IM. At the meeting of Technical Committee T-10 of COOMET it was decided to carry out key bilateral comparisons between VNIIM and the NSC IM for realization of the ITS-90 in the range from 0.01 °C to 429.7485 °C, COOMET Theme N°. 417/RU-08a/03.

### 2 Organization of the comparisons

The general principles and the scheme of comparisons are presented in this Section. The details and procedures for these comparisons are given in the Technical Protocol in Appendix 1.

#### 2.1 Participants of the comparisons

Two NMI's took part in the COOMET key comparisons:

VNIIM – D.I. Mendeleyev All-Russian Research Institute for Metrology, 19, Moskovskii pr., 190005, St.Petersburg, Russian Federation

Phone: 107 8123155207, fax: 1078127130114,

E-mail: A.I.Pokhodun@vniim.ru

NSC IM – National Scientific Center "Institute for Metrology", 42, Mironositskaya str., 61002, Kharkov, Ukraine

Phone: 8 10 380 57 04 97 57, fax: 8 10 380 57 00 34 47,

E-mail: vsl@metrologia.kharkov.ua

VNIIM (RF) also participated in the key comparisons K3 and K4. In the COOMET key comparisons it played the role of a linking institute to disseminate the metrological equivalence to the measurement standards of the NSC IM (Ukraine) in relation to the key comparisons K3.

VNIIM (RF) was appointed the pilot NMI of the key comparisons.

#### 2.2 General principles and scheme of the comparisons

The Technical Protocol of comparisons, the forms of presentation of the measurement results and their uncertainties at the fixed points, the lists of the parameters for the cells and equipment used were sent to the NSC IM. It accepted the Technical Protocol and Appendixes without any comments.

The VNIIM – NSC IM key bilateral comparisons included comparisons of the national realizations of the four ITS-90 main fixed points: the melting point of gallium and the freezing points of indium, tin and zinc.

The VNIIM standard platinum resistance thermometer N°. 25-02-03 was used a transfer standard. It was calibrated before comparisons at VNIIM, its stability being checked and VNIIM after the comparisons at the NSC IM.

On the basis of the comparison results the equivalence of realizations of the fixed points at the NSC IM was established, their link with the reference value of the K3 key comparisons was determined, as well as the correspondence of the CMC uncertainties claimed by the NSC IM to the criteria stated in the CCT WG-8 Review Protocol.

#### 2.3 Time schedule of the comparisons

2-nd quarter of 2008 – 4-th quarter of 2009

# 3 Measurement results of realizations of the main fixed points at VNIIM and the NSC IM

The temperatures of the fixed points at VNIIM and the NSC IM were measured by means of the transfer standard – PRT No. 25-02-03. The measurement results  $W_i$ ,  $U(W_i)(k=1)$  – uncertainties presented by the institutes,  $(W_{\text{NSCIM}}-W_{\text{VNIIM}})$  and their corresponding differences  $(T_{\text{NSCIM}}-T_{\text{VNIIM}})$  are shown in Tables 1-4 and Fig. 1.

**Table 1** - Measurement results of the melting point of gallium

NMI	$W_{\mathrm{Ga}}$	U(W <sub>Ga</sub> ) mK	$W_{ m NSCIM}$ $-W_{ m VNIIM}$	$T_{ m NSCIM}$ – $T_{ m VNIIM}$ mK
VNIIM	1.118 108 15	0.057	0	0
NSC IM	1.118 108 03	0.118	-0.000 000 12	-0.03

Table 2 - Measurement results of the freezing point of indium

NMI	$W_{ m In}$	U(W <sub>In</sub> ) mK	$W_{ m NSCIM}$ $-W_{ m VNIIM}$	$T_{ m NSCIM}$ – $T_{ m VNIIM}$ mK
VNIIM	1.609 681 26	0.172	0	0
NSC IM	1.609 676 96	0.520	-0.000 004 30	- 1.13

Table 3 - Measurement results of the freezing point of tin

NMI	$W_{\mathrm{Sn}}$	U(W <sub>Sn</sub> ) mK	$W_{ m NSCIM}$ – $W_{ m VNIIM}$	$T_{ m NSCIM}$ – $T_{ m VNIIM}$ mK
VNIIM	1.892 602 30	0.251	0	0
NSC IM	1.892 601 37	0.429	- 0.000 000 93	- 0.25

Table 4 - Measurement results of the freezing point of zinc

NMI	$W_{\mathrm{Zn}}$	U(W <sub>Zn</sub> ) mK	$W_{ m NSCIM}$ $-W_{ m VNIIM}$	$T_{ m NSCIM}$ – $T_{ m VNIIM}$ mK
VNIIM	2.568 547 13	0.296	0	0
NSC IM	2.568 545 94	0.472	- 0.000 001 19	- 0.34

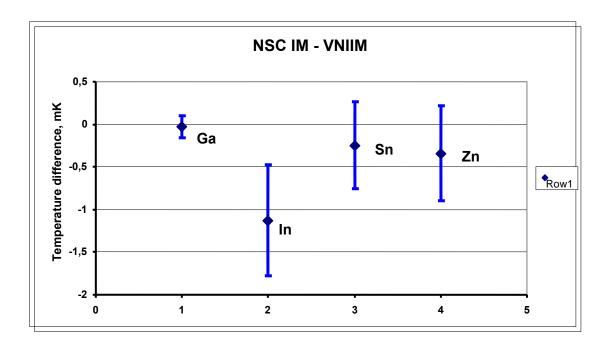


Figure1 - Comparison results of the realizations of the main reference points

# 3.1 Estimation of the uncertainty of the measurement results due to instability of the transfer standard – platinum resistance thermometer No. 25-02-03

The uncertainty of the measurement results due to instability of the transfer standard,  $u_{PRT}$  was calculated under the assumption of the resistance thermometer drift and the rectangular skewed distribution of resistance deviation in time:

$$u_{\text{PRT}} = [(W_{\text{VNIIM}})_{\text{final}} - (W_{\text{VNIIM}})_{\text{initial.}}] \times (\partial T/\partial W) \times 1/\sqrt{3}$$

**Table 5** - Estimation of the uncertainty of the measurement results due to instability of the transfer standard

Fixed point	(W <sub>VNIIM</sub> ) <sub>initial</sub>	$(W_{ m VNIIM})_{ m final}$	$(W_{ m VNIIM})_{ m final} - \ (W_{ m VNIIM})_{ m initial}$	ΔT mK	<i>u</i> <sub>PRT</sub> , mK (k=1)
Ga	1.11810817	1.11810786	0.00000031	0.08	0.04
In	1.60968136	1.60968041	0.00000095	0.25	0.14
Sn	1.89260245	1.89260126	0.00000119	0.32	0.18
Zn	2.56854718	2.56854577	0.00000141	0.40	0.23

#### 4 Results of the COOMET key comparisons

The results of the COOMET comparisons were estimated in accordance with the document "Guide on Estimation of the COOMET Key Comparison Data", COOMET, R/GM/14:2006. This document allows to link the results of the COOMET regional comparisons with those of the CIPM key comparisons, in this case with the results of the CCT comparisons K3.

The results of the key comparisons K3 in the protocol of WG8 "Inter-RMO CMC review committee 3-26-03" are shown as differences [ $T_{NMI}$  – ARV (K3)] with the evaluations of their uncertainties for each reference point and NMI.

# 4.1 Evaluations of the weighted mean values $(T_{\rm wm}-T_{\rm VNIIM})$ and their uncertainties for the fixed points

The results of the comparison of the COOMET fixed points are deviations of the NMI's measurement results from the weighted mean value of the deviations. The weighted mean values of the deviations,  $\Delta T_{\rm wm} = (T_{\rm wm} - T_{\rm VNIIM})$ , for each fixed point and its standard uncertainty,  $u_{\rm wm}(\Delta T_{\rm wm})$  are calculated by the formulas:

$$\Delta T_{wm} = (T_{wm} - T_{VNIIM}) = \frac{\sum_{1}^{n} \frac{(T_{NMI} - T_{VNIIM})}{U^{2}(T_{NMI} - T_{VNIIM})}}{\sum_{1}^{n} \frac{1}{U^{2}(T_{NMI} - T_{VNIIM})}}, \qquad u_{wm}^{2} = \frac{1}{\sum_{1}^{n} \frac{1}{U^{2}(T_{NMI} - T_{VNIIM})}}$$

where  $(T_{\text{NMI}} - T_{\text{VNIIM}})$  are the differences shown in Tables 1–14,

$$U^{2}(T_{\text{NMI}} - T_{\text{VNIIM}}) = [U^{2}(W_{i}) + u^{2}_{\text{PRT}} + u^{2}(\text{VNNIM component A})];$$

the values of  $U(W_i)$  are shown in Tables 1–14, those for  $u_{PRT}$  in Table 15, and  $u_{PRT}$  in Table 16, and  $u_{PRT}$  in Table 17, and  $u_{PRT}$  in Table 18, and  $u_{PRT}$  in Table 18, and  $u_{PRT}$  in Table 19, and  $u_{PRT}$  in Table

 $u_{\rm wm}(\Delta T_{\rm wm})$  is the standard uncertainty of the weighted mean value  $\Delta T_{\rm wm}$ .

The calculation results of  $\Delta T_{\rm wm}$  and  $u_{\rm wm}$  of the fixed points are presented in Tables 6–9.

# 4.2 Deviations of the NMI results from the weighted mean values $(T_{\rm NMI}-T_{\rm wm})$ and their uncertainty evaluations

The measurement result deviations of the participants from the weighted mean value were calculated, after determination of  $\Delta T_{\rm wm}$  and  $u_{\rm wm}$  i.e.

$$(T_{\text{NMI}} - T_{\text{wm}}) = (T_{\text{NMI}} - T_{\text{VNIIM}}) - (T_{\text{wm}} - T_{\text{VNIIM}}).$$

The uncertainty of the measurement result deviations of the participants from the weighted mean value  $U^2(T_{\rm NMI}-T_{\rm wm})$  was calculated by the formula:

$$U^{2}(T_{\text{NMI}} - T_{\text{wm}}) = [U^{2}(T_{\text{NMI}} - T_{\text{VNIIM}}) + u^{2}_{\text{wm}}],$$

The calculation results of  $(T_{\text{NMI}} - T_{\text{wm}})$  and  $U(T_{\text{NMI}} - T_{\text{wm}})$  for all fixed points are given in Tables 6–9.

Table 6 - Fixed melting point of gallium

$$\Delta T_{\text{wm}} = -0.007 \text{ mK}, u_{\text{wm}} = 0.06 \text{ mK}$$

NMI	$T_{ m NMI} - T_{ m wm} \ { m mK}$	$T_{ m NMI} - T_{ m wm} \  m mK$	$U(T_{ m NMI} - T_{ m wm})$ mK ( $\kappa$ =1)
VNIIM	0	0.007	0.09
NSC IM	-0.03	-0.023	0.15

**Table 7 -** Fixed freezing point of indium

$$\Delta T_{\text{wm}} = -0.117 \text{ mK}, u_{\text{wm}} = 0.21 \text{ mK}$$

NMI	$T_{ m NMI} - T_{ m wm} \  m mK$	$T_{ m NMI} - T_{ m wm} \  m mK$	$U(T_{\text{NMI}} - T_{\text{wm}})$ mK (k=1)
VNIIM	0	0.117	0.30
NSC IM	-1.13	-1.013	0.69

Table 8 - Fixed freezing point of tin

$$\Delta T_{\text{wm}} = -0.065 \text{ mK}, u_{\text{wm}} = 0.26 \text{ mK}$$

NMI	$T_{ m NMI} - T_{ m wm} \  m mK$	$T_{ m NMI} - T_{ m wm} \  m mK$	$U(T_{\text{NMI}} - T_{\text{wm}})$ mK (k=1)
VNIIM	0	0.065	0.41
NSC IM	- 0.25	- 0.185	0.58

Table 9 - Fixed freezing point of zinc

$$\Delta T_{\text{wm}} = -0.105 \text{ mK}, u_{\text{wm}} = 0.31 \text{ mK}$$

NMI	$T_{ m NMI} - T_{ m wm} \  m mK$	$T_{ m NMI} - T_{ m wm} \  m mK$	$U(T_{ m NMI} - T_{ m wm})$ $ m MK (k=1)$
VNIIM	0	0.105	0.37
NSC IM	- 0.34	- 0.235	0.64

## 5 Calculation of the additive corrections and the degrees of equivalence of the NSC IM fixed points relative to the key comparisons K3

VNIIM participated in the COOMET key regional comparisons as a linking institute for calculation of the additive corrections and the degrees of equivalence of the NSC IM fixed points relative to the key comparisons K3.

According to the document "Guide on Estimation of the COOMET Key Comparison Data", COOMET, R/GM/14:2006 the transformed result of the NSC IM measurement is equal to the sum of the result obtained in the COOMET comparisons and the additive correction.

In the case with one linking institute the additive correction  $\Delta$  and the corresponding uncertainty  $u(\Delta)$  are calculated by the formulas:

$$\Delta = [(T_{\text{VNIIM}} - \text{ARV}(\text{K3})] - [(T_{\text{VNIIM}} - T_{\text{wm}})],$$
  
$$u^2(\Delta) = 2S^2,$$

where *S* is the r.m.s. deviation of the measurement results in the linking NMI.

Since the results of the key comparisons K3 are presented as the differences [ $T_{\text{VNIIM}}$  – ARV(K3)], the degree of equivalence "d" of the NSC IM result is estimated by the following relation:

$$d_{\rm i} = (T_{\rm NMI} - T_{\rm wm}) + \Delta_{\rm i}$$

and presents the difference  $[T_{NSCIM} - ARV(K3)]$  with the standard uncertainty "d":

$$u^{2}(d) = U^{2}[(T_{\text{NMI}} - T_{\text{wm}})] + u^{2}(\Delta).$$

The VNIIM results obtained in the key comparisons K3 and used in the calculations are given in Table 10.

**Table 10 -** VNIIM results in the comparisons K3

	Fixed points	T <sub>NMI</sub> – ARV, mK	U(ARV) (k=2) mK	$U(T_{ m NMI} - { m ARV})$ $({ m k=2})$ ${ m mK}$
	Ga	0.05	0.08	0.25
	In	0.54	0.59	1.11
VNIIM	Sn	0.59	0.37	0.99
	Zn	0.52	0.32	1.85

# 5.1 Additive correction and degree of equivalence for the fixed melting point of gallium

VNIIM: K3 result 
$$T_{\text{VNIIM}} - \text{ARV(K3)} = 0.05 \text{ mK},$$
 in COOMET  $T_{\text{VNIIM}} - T_{\text{wm}} = 0.007 \text{ mK}.$ 

As a result the additive correction to the NSC IM result is

$$\Delta(Ga) = 0.043$$
 mK, its uncertainty is  $u(\Delta_{Ga})(k=1) = 0.06$  mK.

**NSC IM**: result in COOMET 
$$T_{\text{NSCIM}} - T_{\text{wm}} = -0.023 \text{ mK}$$

The degree of equivalence is  $d_{Ga} = (T_{NSCIM} - T_{wm}) + \Delta(Ga)$ ;

$$d_{Ga} = [T_{NSCIM} - ARV(K3)] = 0.02 \text{ mK};$$

the uncertainty is  $u(d_{Ga})(k=1) = 0.16$  mK.

# 5.2 Additive correction and degree of equivalence for the fixed freezing point of indium

VNIIM: K3 result

$$T_{\text{VNIIM}} - \text{ARV}(\text{K3}) = 0.54 \text{ mK},$$

in COOMET

$$T_{\text{VNIIM}} - T_{\text{wm}} = 0.117 \text{ mK}.$$

As a result the additive correction to the NSC IM result is

$$\Delta(In) = 0.423$$
 mK, its uncertainty is  $u(\Delta_{In})(k=1) = 0.16$  mK.

**NSC IM**: result in COOMET

$$T_{\rm NSCIM} - T_{\rm wm} = -1.013 \text{ mK}$$

The degree of equivalence  $d_{\text{In}} = (T_{\text{NSCIM}} - T_{\text{wm}}) + \Delta(\text{In})$ ;

$$d_{\text{In}} = [T_{\text{NSCIM}} - \text{ARV(K3)}] = 0.590 \text{ mK};$$

the uncertainty is  $u(d_{In})(k=1) = 0.71$  mK.

# 5.3 Additive correction and degree of equivalence for the fixed freezing point of tin

**VNIIM**: K3 result

$$T_{\text{VNIIM}} - \text{ARV(K3)} = 0.59 \text{ mK},$$

in COOMET

$$T_{\text{VNIIM}} - T_{\text{wm}} = 0.065 \text{ mK}.$$

As a result the additive correction to the NSC IM result is

$$\Delta(Sn) = 0.525 \text{ mK}$$
, its uncertainty is  $u(\Delta_{Sn})(k=1) = 0.31 \text{ mK}$ .

**NSC IM**: result in COOMET

$$T_{\rm NSCIM} - T_{\rm wm} = -0.185 \text{ mK}$$

The degree of equivalence  $d_{Sn} = (T_{NSCIM} - T_{wm}) + \Delta(Sn)$ ;

$$d_{\rm Sn} = [T_{\rm NSCIM} - ARV(K3)] = 0.340 \text{ mK};$$

the uncertainty is  $u(d_{Sn})(k=1) = 0.66$  mK.

# 5.4 Additive correction and degree of equivalence for the fixed freezing point of zinc

BHИИМ: K3 result 
$$T_{\text{VNIIM}} - \text{ARV}(\text{K3}) = 0.52 \text{ mK},$$
 in COOMET  $T_{\text{VNIIM}} - T_{\text{wm}} = 0.105 \text{ mK}.$ 

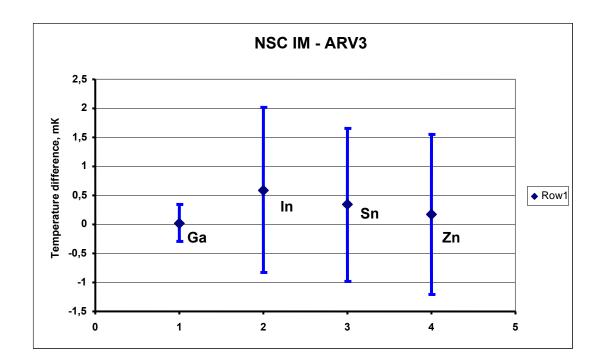
As a result the additive correction to the NSC IM result is

$$\Delta(Zn) = 0.415 \text{ mK}$$
, its uncertainty is  $u(\Delta_{Zn})(k=1) = 0.27 \text{ mK}$ .

**NSC IM**: result in COOMET 
$$T_{\text{NSCIM}} - T_{\text{wm}} = -0.235 \text{ mK}$$

The degree of equivalence  $d_{\mathbf{Z}\mathbf{n}} = (T_{\text{NSCIM}} - T_{\text{wm}}) + \Delta(\mathbf{Z}\mathbf{n});$ 

$$d_{\rm Zn} = [T_{\rm NSCIM} - {\rm ARV(K3)}] = 0.180 \text{ mK};$$
  
the uncertainty is  $u(d_{\rm Zn})({\rm k=1}) = 0.69 \text{ mK}.$ 



**Figure 2 -** Equivalence of the realizations of the NSC IM fixed points relative to the results of the key comparisons K3.

#### **6 Conclusions**

The uncertainties claimed by the NMI's for the CMC are considered to be confirmed by the results of the regional comparisons if the degree of equivalence "d" of the NMI result in the comparisons is less than the double value of its uncertainty ("Guide on Evaluation of the COOMET Key Comparison Data", COOMET.R/GM/14:2006), i.e. the following inequality is held:

$$|d| < 2u(d)$$
.

This inequality corresponds to the criteria of the recognition of the CMC uncertainties claimed by the NMI, stated in the CCT WG8 Review Protocol.

As evident from the results presented in 5.1-5.4, the inequality |d| < 2u(d) is held for all four fixed points. Hence, it is possible to draw a conclusion that the COOMET key comparisons have proved the CMC uncertainties claimed by the NSC IM for the melting point of gallium 0.236 mK(k=2), and the freezing points of indium 1.040 mK(k=2), tin 0.858 mK(k=2) and zinc 0.944 mK(k=2).

## **Appendix 1**

## **Technical Protocol**

# COOMET comparisons of the ITS-90 realizations in the range from 0 °C to 419.527 °C

#### 1 Introduction

The D.I. Mendeleyev Institute for Metrology (VNIIM) (RF), which has participated in the key comparisons K3, K4 and K7, and the National Scientific Center "Institute for Metrology" (Ukraine) take part in the COOMET key regional comparisons.

The participants of the comparisons shall act in accordance with the instruction given below. Each laboratory during the comparisons shall apply the adopted realization of the ITS-90.

The instruction is written in compliance with Appendix 1 to the Report on the CCT Key Comparisons K3 []. The comparisons carefully follow the protocols given in the Guidance for the CIPM key comparisons, in Appendix F to the document "Arrangement on Mutual Recognition of the National Standard, Calibration and Measurement Certificates Issued by the National Metrology Institutes".

VNIIM (RF) being a participant of the key comparisons K3, K4 and K7 is a coordinator of the regional comparisons.

During the comparisons the equivalence of the fixed point realizations at the national institutes shall be estimated. A standard platinum resistance thermometer shall be used as a transfer standard. The equivalence of the standards and their link with the key comparison reference value shall be established on the basis of the results received. The standard platinum resistance thermometer SPRT N°. 25-02-03 shall be calibrated at VNIIM before the comparisons, its stability being checked at VNIIM afterwards. Thermometers are extremely fragile objects and must be only transported by courier with a great care.

The NSC IM calibrates the received thermometer at the fixed points and draws up a report on the basis of measurement results. This report is sent directly to VNIIM (during a month after the measurements). VNIIM calculates the deviations between the realizations of the fixed points at the NSC IM and VNIIM, estimates the equivalence of the realizations of the fixed points at the NSC IM in relation to the key comparisons K3 and the CMC uncertainties claimed by the NSC IM.

#### 2 Time schedule of the comparisons

It takes three weeks to transfer the thermometers between the laboratories with an intermediate stability check of the SPRT at VNIIM.

#### 3 Procedures

The participants of the comparisons shall act in accordance with the instruction given below. Each laboratory during the comparisons shall apply the adopted realization of the ITS-90.

The instruction is written in compliance with Appendix 1 to the Report on the CCT Key Comparisons K3 []. The comparisons carefully follow the protocols given in the Guidance for the CIPM key comparisons, in Appendix F to the document "Arrangement on Mutual Recognition of the National Standard, Calibration and Measurement Certificates Issued by the National Metrology Institutes".

The regional comparisons within COOMET assume comparison of the ITS-90 realizations in the range from the triple point of water (0.01°C) to the freezing point of zinc (419.527 °C) using standard resistance platinum thermometers.

#### 3.1 Actions of VNIIM as a coordinating laboratory

VNIIM prepares and calibrates the SPRT for comparisons.

VNIIM transfers the calibrated SPRT to the NSC IM for calibration at the fixed points in accordance with the practice accepted at the laboratory.

After completion of measurements at the NSC IM and returning the SPRT to VNIIM, it checks the SPRT stability.

After receiving a report on calibration of the SPRT from the NSC IM, VNIIM carries out the analysis of the comparison results.

#### 3.2 Actions of the NSC IM

After receiving the SPRT from VNIIM, the NSC IM performs the following procedures:

- 3.2.1 Measurement of the SPRT resistance at the triple point of water.
- 3.2.2 Annealing of the SPRT in a certain sequence:
- a) The SPRT is inserted into the furnace at the temperature 500 °C,
- b) The temperature in the furnace is raised to 600 °C,
- c) The SPRT is kept in the furnace during two hours,
- d) The temperature in the furnace is reduced to 450 °C during 2.5 hours,
- e) The SPRT is quickly removed from the furnace to the air and cools down to room temperature.
  - 3.2.3 Measurement of the SPRT resistance at the triple point of water.

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.

3.2.4 The SPRT is calibrated in the following order: TPW, Zn, TPW, Sn, TPW, In, TPW, Ga, TPW. The practice accepted in the NSC IM is used for each fixed point. The relative resistance is calculated for each fixed point: W = Rt/RTPW, where RTPW is the SPRT resistance at the triple point of water obtained directly after measuring Rt. The values of Rt and RTPW shall be corrected for overheating with measuring current, hydrostatic pressure and pressure in the cell.

The above calibration cycle shall be repeated minimum three times.

The values of W received in three measurement cycles and their average value for each fixed point are passed over to the VNIIM. On completion of measurements the SPRT is returned to the VNIIM for stability check.

#### 4 Submission of the results

The NSC IM shall send the following information to the coordinator:

- a) Parameters of the cells of the fixed points, furnaces, measuring instruments used in the comparisons.
- b) Measurement results W = Rt/RTPW, where Rt is the SPRT resistance at each fixed point; RTPW is the SPRT resistance at the triple point of water obtained after measuring Rt. The values of Rt and RTPW shall be corrected for overheating with measuring current, hydrostatic pressure and pressure in the cell according to Appendix A.
- c) Examples of the experimental freezing curves for the fixed points of In, Sn, Zn and the melting curves for the fixed point of Ga.
- d) Experimental curves of the dependence of fixed point temperature on depth of immersion of the SPRT used.
- e) Uncertainty budget for each fixed point corresponding to Table 2 calculated in accordance with Appendix B of the Protocol.

## Appendix A

		N	<b>Ieasurement</b>	results		
COOMET						
<b>Laboratory:</b>						
W(t) = R(t)/Rtance at the T						
Correction fo	r pressure in	the cell				
Point to be measured:						
Thermometer number:						
Point to be measured:	R measured at 1mA	Overheat	Hydrostatics	Pressure	Corrected R	W
	ohm	ohm	ohm	ohm	ohm	
Average W						

## Appendix 2

# Parameters of the cells of fixed points, furnaces, measuring instruments applied by the participating laboratories

Laboratory	VNIIM
Bridge, potentiometer	
Manufacturer	
Type	Guideline 9975
	Guidennie 7773
A.C. or D.C.	D.C.
A.C. frequency	
D.C. period	4 s
•	
Normal measurement current	1 mA
Overheating current	1.414 mA
Resistance linearity	
Bridge (yes/no)	
Reference resistor	
Manufacturer	ZIP, RF
Type	MC3020
Temperature control of reference resistor (yes/no)	yes
TPW cell	
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	Thermostat with
	ice
Zn fixed point cell	
Manufacturer	VNIIM
Open or closed	open
Outer diameter	52 mm
Thermometer well diameter	14 mm
Metal purity	99.9999m%
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	2 201102
Freezing plateau duration	10 hours

Laboratory	VNIIM
Sn fixed point cell	
Manufacturer	VNIIM
Open or closed	open
Outer diameter	52 mm
Thermometer well diameter	14 mm
Metal purity	99.9999 %
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	
Manufacturer	VNIIM
Open or closed	open
Outer diameter	42 mm
Thermometer well diameter	10 mm
Metal purity	99.9999 %
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	
Manufacturer	VNIIM
Open or closed	open
Outer diameter	42 mm
Thermometer well diameter	10 mm
Metal purity	99.99999 %
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

Laboratory	NSC "Institute for Metrology"
Bridge, potentiometer	bridge
Manufacturer	NPP "Spetsavtomatika", Kiev, Ukraine
Type	CA300-1
Турс	C/1300-1
A.C. or D.C.	A.C.
A.C. frequency	125 Hz
D.C. period	
Normal measurement current	1 mA
Overheating current	1.414 mA
overheating earrent	1.11111111
Resistance linearity	
Bridge (yes/no)	yes
Reference resistor	
Manufacturer	ZIP, RF
Туре	MP3000
Temperature control of reference re-	yes
sistor (yes/no)	<i>y</i>
TPW cell	No. 9209
Manufacturer	VNIIFTRI, Russia
Outer diameter	65 mm
Thermometer well diameter	20 mm
Immersion depth of the middle of the	225 mm
SPRT sensing element relative to wa-	
ter level in the cell	
Thermostat for maintaining the TPW	NSC "Institute for Metrology", Ukraine
Zn fixed point cell	
Manufacturer	FGUP "D.I. Mendeleyev Institute for Metrology",
	Russia
Open or closed	closed
Outer diameter	54 mm
Thermometer well diameter	9 mm
Metal purity	99.9999 %
Immersion depth of the middle of the	175 mm
SPRT sensing element	
Furnace for the fixed point of Zn	
Manufacturer	NSC "Institute for Metrology", Ukraine
Type (1, 2 or 3 zones)	3 zones
Melting plateau duration	
Freezing plateau duration	10 hours

Laboratory	NSC "Institute for Metrology"
Sn fixed point cell	Ov.
Manufacturer	FGUP "D.I. Mendeleyev Institute for Metrology",
	Russia
Open or closed	closed
Outer diameter	54 mm
Thermometer well diameter	9 mm
Metal purity	99.9999 %
Immersion depth of the middle of the SPRT sensing element	175 mm
Furnace for the fixed point of Sn	
Manufacturer	NSC "Institute for Metrology", Ukraine
Type (1, 2 or 3 zones)	3 zones
Melting plateau duration	o Honey
Freezing plateau duration	9.5 hours
In fixed point cell	
Manufacturer	NSC "Institute for Metrology", Ukraine
Open or closed	open
Outer diameter	50 mm
Thermometer well diameter	9 mm
Metal purity	99.9999 %
Immersion depth of the middle of the SPRT sensing element	155 mm
Furnace for the fixed point of In	
Manufacturer	NSC "Institute for Metrology", Ukraine
Type (1, 2 or 3 zones)	1 zone (heater coil with varied pitch)
Melting plateau duration	
Freezing plateau duration	11 hours
Ga fixed point cell	
Manufacturer	NSC "Institute for Metrology", Ukraine
Open or closed	open
Outer diameter	50 mm
Thermometer well diameter	9 mm
Metal purity	99.9999 %
Immersion depth of the middle of the SPRT sensing element	155 mm
Furnace for the fixed point of Ga	
Manufacturer	NSC "Institute for Metrology", Ukraine
Type (1, 2 or 3 zones)	1 zone (heater coil with varied pitch)
Melting plateau duration	19 hours

# Appendix 3

## **Uncertainty budgets**

## Uncertainty budget for the melting point of Ga

	mK	
Uncertainty components	VNIIM	NSC IM
1. Reproducibility of the $W_t$ values	0.044	0.029
2. Component due to metal purity	0.025	0.07
3. Component due to correction for hydrostatic pressure	0.003	0.003
4. Component due to correction for overheating	0.010	0.012
5. Component due to deviation from thermal equilibrium	0.020	0.02
6. Component due to pressure in the cell	0.0002	0.005
7. Component due to a.c. or d.c.	-	-
8. Component due to nonlinearity of the bridge	0.003	0.032
9. Component due to temperature of the reference resistor	0.0001	0.005
10. Component due to stability of the reference resistor	-	-
Components due to measurement in the TPW		
11. Component due to purity and isotopic composition of water	0.010	0.050
12. Component due to correction for hydrostatic pressure	0.001	0.002
13. Component due to correction for overheating	0.006	0.017
14. Component due to deviation from thermal equilibrium	0.006	0.05
15. Component due to a.c. or d.c.	-	-
16. Component due to nonlinearity of the bridge	0.003	0.029
Total uncertainty	0.057	0.118
Expanded uncertainty	0.114	0.236

## **Uncertainty Budget for the Freezing Point of Indium**

	mK	
<b>Uncertainty components</b>	VNIIM	NSC IM
1. Reproducibility of the $W_t$ values	0.117	0.099
2. Component due to metal purity	0.120	0.500
3. Component due to correction for hydrostatic pressure	0.005	0.01
4. Component due to correction for overheating	0.012	0.012
5. Component due to deviation from thermal equilibrium	0.030	0.060
6. Component due to pressure in the cell	0.0006	0.013
7. Component due to a.c. or d.c.	-	-
8. Component due to nonlinearity of the bridge	0.003	0.047
9. Component due to temperature of the reference resistor	0.0001	0.005
10. Component due to stability of the reference resistor	-	-
Components due to measurement in the TPW		
11. Component due to purity and isotopic composition of water	0.010	0.050
12. Component due to correction for hydrostatic pressure	0.001	0.002
13. Component due to correction for overheating	0.006	0.004
14. Component due to deviation from thermal equilibrium	0.006	0.050
15. Component due to a.c. or d.c.	-	-
16. Component due to nonlinearity of the bridge	0.003	0.029
Total uncertainty	0.172	0.520
Expanded uncertainty	0.344	1.040

## **Uncertainty Budget for the Freezing Point of Tin**

	mK	
Uncertainty components	VNIIM	NSC IM
1. Reproducibility of the $W_t$ values	0.218	0.096
2. Component due to metal purity	0.120	0.400
3. Component due to correction for hydrostatic pressure	0.003	0.006
4. Component due to correction for overheating	0.015	0.019
5. Component due to deviation from thermal equilibrium	0.030	0.060
6. Component due to pressure in the cell	0.0006	-
7. Component due to a.c. or d.c.	-	-
8. Component due to nonlinearity of the bridge	0.003	0.055
9. Component due to temperature of the reference resistor	0.0001	0.005
10. Component due to stability of the reference resistor	-	-
Components due to measurement in the TPW		
11. Component due to purity and isotopic composition of water	0.010	0.050
12. Component due to correction for hydrostatic pressure	0.001	0.002
13. Component due to correction for overheating	0.006	0.049
14. Component due to deviation from thermal equilibrium	0.006	0.050
15. Component due to a.c. or d.c.	-	-
16. Component due to nonlinearity of the bridge	0.003	0.029
Total uncertainty	0.251	0.429
Expanded uncertainty	0.502	0.858

## **Uncertainty Budget for the Freezing Point of Zinc**

Unacutainty company	mK	
Uncertainty components	VNIIM	NSC IM
1. Reproducibility of the $W_t$ values	0.191	0.210
2. Component due to metal purity	0.210	0.400
3. Component due to correction for hydrostatic pressure	0.010	0.008
4. Component due to correction for overheating	0.025	0.024
5. Component due to deviation from thermal equilibrium	0.080	0.070
6. Component due to pressure in the cell	0.0012	_
7. Component due to a.c. or d.c.	-	_
8. Component due to nonlinearity of the bridge	0.003	0.074
9. Component due to temperature of the reference resistor	0.0001	0.005
10. Component due to stability of the reference resistor	-	-
Components due to measurement in the TPW		
11. Component due to purity and isotopic composition of water	0.010	0.050
12. Component due to correction for hydrostatic pressure	0.001	0.002
13. Component due to correction for overheating	0.006	0.042
14. Component due to deviation from thermal equilibrium	0.006	0.050
15. Component due to a.c. or d.c.	-	_
16. Component due to nonlinearity of the bridge	0.003	0.029
Total uncertainty		
Expanded uncertainty	0.296	0.472