# Final Report of COOMET.T-K5: Realizations of the ITS-90 at 1084.62 °C (COOMET Project 387/UA-a/07)

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CONTENT

## 1 Introduction

At the meeting of Technical Committee, TC 1.10 of COOMET in 2007 it was decided to undertake regional comparison between VNIIM (Russian Federation) and NSC IM (Ukraine) for realization of the ITS-90 at the freezing point of copper in the field of non-contact thermometry. The COOMET theme No. 387/UA-a/07, registration in the KCDB "COOMET.T-K5". The comparison supports the following CMC items: 2.5.1, 2.5.2, 2.5.3 and 2.5.4 at the fixed-point of copper.

The purpose of the regional comparison is dissemination of the metrological equivalence to the standard of the national metrology institute NSC IM (Ukraine), which did not participate in the CCT key comparisons. The degree of equivalence of the measurement standards of the NSC IM is determined relatively to the reference value of the CCT key comparisons through the measurement results received in the linking national metrology institute, which takes part in both comparisons.

VNIIM participated in the key comparisons CCT-K5. In the COOMET comparison VNIIM is the linking institute in terms of dissemination of the metrological equivalence to the NSC IM standards (Ukraine).

The protocol of the comparison covers two separate comparisons: a comparison of fixed-point cells (Stage 1), and a comparison of lamp calibrations (Stage 2) at 1084.62 $^{o}$C. The former is not directly comparable or linkable to the CCT-K5, whereas the latter is comparable and linkable. It will be the results of the latter comparison that are to be taken as the RMO-K5 results for linkage into the K5 data tables of the KCDB. The former, the cell comparison, results can still be used under the auspices of the MRA, as additional evidence for CMC support, but are more of a special regional supplementary comparison that happens at the same time as the KC.

## 2 Organization of the comparison

The principles and the scheme of comparisons are presented in this Section. The details and procedures are given in the Technical Protocol (Appendix I of this Report).

### 2.1 Participants of the comparison

Two NMIs took part in the COOMET comparison:

- D.I. Mendeleyev Institute for Metrology (VNIIM), Moskovsky prospect, 19, Saint-Petersburg, the Russian Federation

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E-mail: M.S.Matveyev@vniim.ru

- National Scientific Centre "Institute of Metrology" (NSC IM), Mironositskaya St., 42, Kharkov, Ukraine

Phone: +038 057 704 98 00, fax +038 057 704 34 47

E-mail: Rymma.Sergiyenko@metrology.kharkov.ua

## 2.2 Scheme and comparison schedule

Participants of comparisons approved the Technical Protocol of comparisons, the forms of presentation of the measurement results, the lists of the parameters for the equipment applied. An expertise of the Technical protocol has been carried out in CCT.

Comparisons were performed in two stages. At the first stage of the comparison graphite crucible, containing pure metal and blackbody cavity, was used as a transfer standard. This transfer standard is a part of the national measurement standard of NSC IM (Ukraine). Characteristics of NSCIM's crucible are presented in Appendix A and B of the Technical Protocol.

The graphite crucible was transported from NSC IM to VNIIM where it was compared with the corresponding crucible (fixed-point blackbody) of VNIIM on the basis of national measurement standard of the Russian Federation with the use of equipment of this national standard. Scheme of the first stage of comparison is presented in Fig.1.

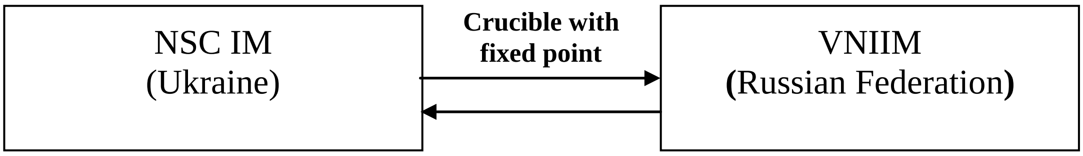



Fig. 1 – Scheme of the first stage of comparison

At the second stage (the scheme is shown in Fig. 2), vacuum tungsten ribbon lamp СИ (SI) 10-300 No. 5, which is provided by VNIIM, was used as a transfer standard. Description and characteristics of the lamp SI 10-300 No. 5 are given in Appendix C; rules for transportation – in Appendix D; description of operation with transfer standard – in Appendix E of the Technical Protocol.

On the basis of the second stage, the degree of equivalence of copper fixed-point realization by the NSC IM standard was determined relatively to the reference value of the CCT-K5 key comparisons.

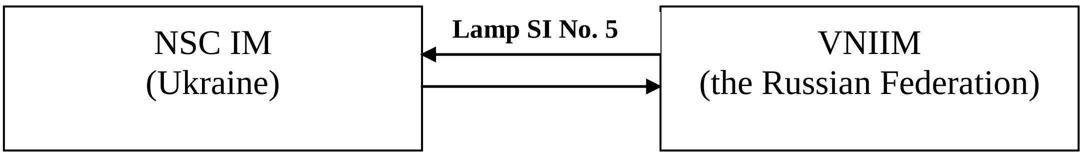



Fig. 2 – Scheme of the second stage of comparison

Vacuum tungsten ribbon lamp SI 10-300 No. 5 was calibrated at VNIIM before the comparison; its stability was controlled at VNIIM after performance of measurements at NSC IM.

Time schedule of the comparison

Measurements: August 2014 – October 2014
Data processing and compilation of report "A": October 2014 – November 2014
Preparation of report "B": November 2014 – December 2014

## 3 Results of the first stage of the comparison

## (Comparison of the copper fixed-point cells)

The result of the first stage of the comparison is the deviation of the temperature value reproduced by the fixed-point blackbody (crucible) of NSC IM under the VNIIM laboratory conditions from the value reproduced by the VNIIM fixed-point blackbody.

The measurements were taken using the standard photoelectric spectral comparator (SPC) and the equipment of the national measurement standard of temperature of the Russian Federation GET 34-2007. Information about crucibles and VNIIM equipment is given in Appendix II according to the Technical Protocol. Because of different dimensions of the crucibles (Appendix IV), the VNIIM furnace was redesigned.

Six plateaus of freezing for the NSC IM crucible and five plateaus of freezing for the VNIIM crucible were realized. The parameters of plateaus and the shape of plateau curves are given in Appendix III and Appendix IV, respectively. Drift of the standard photoelectric spectral comparator has being monitored before and after the measurements using the reference temperature lamp. The temperature difference has been calculated taking into account the SPC drift.

The results of the comparisons of fixed-point blackbodies, the average temperature difference and the uncertainty are presented in Table 1 and Fig. 3.

Table 1 – The comparison results of the copper fixed-point cells

| **Number of freezing plateau** | **SPC output signal in temperature equivalent, °C** | | **NSC IM – VNIIM** | | |
|---|---|---|---|---|---|
| | **VNIIM** | **NSC IM** | **Δ*T*, K** | **$u_A$, K** | **$u_\Delta$, K ($k$=1)** |
| 1 | 1084.63 | 1084.56 | -0.07 | | |
| 2 | 1084.64 | 1084.54 | -0.10 | | |
| 3 | 1084.66 | 1084.61 | -0.05 | 0.011 | 0.065 |
| 4 | 1084.68 | 1084.63 | -0.05 | | |
| 5 | 1084.65 | 1084.55 | -0.10 | | |
| **Average** | **1084.652** | **1084.578** | **-0.074** | | |

The estimation of the combined uncertainty $u_\Delta$ of the average difference between the crucibles is calculated by the formula:

$$u_\Delta = \sqrt{u_A^2 + u_B^2} ,$$

where $u_A$ – standard uncertainty of the temperature difference estimated by A type,

$u_B^2 = \sum_i u_{Bi}^2$ , $u_B$ – standard uncertainty of the result estimated by B type.

In the uncertainty budget, estimated by B type, such uncertainty components are included:

- components associated with the crucibles of VNIIM and NSC IM (impurities, emissivity, plateaus identification, heat transfer processes);

- components associated with characteristics of the VNIIM standard photoelectric spectral comparator , the NSC IM standard pyrometer (scattering and polarization of the radiation, size-of-source effect, others) and tungsten strip lamps characteristics.

The values of uncertainty components are given by VNIIM and NSC IM in corresponding items of the uncertainty budget in Appendix VIII.

The combined uncertainty $u_\Delta$ estimated by this components is equal to 0.064 K (for VNIIM, k=1) and 0.127 K (for NSC IM, k=1).

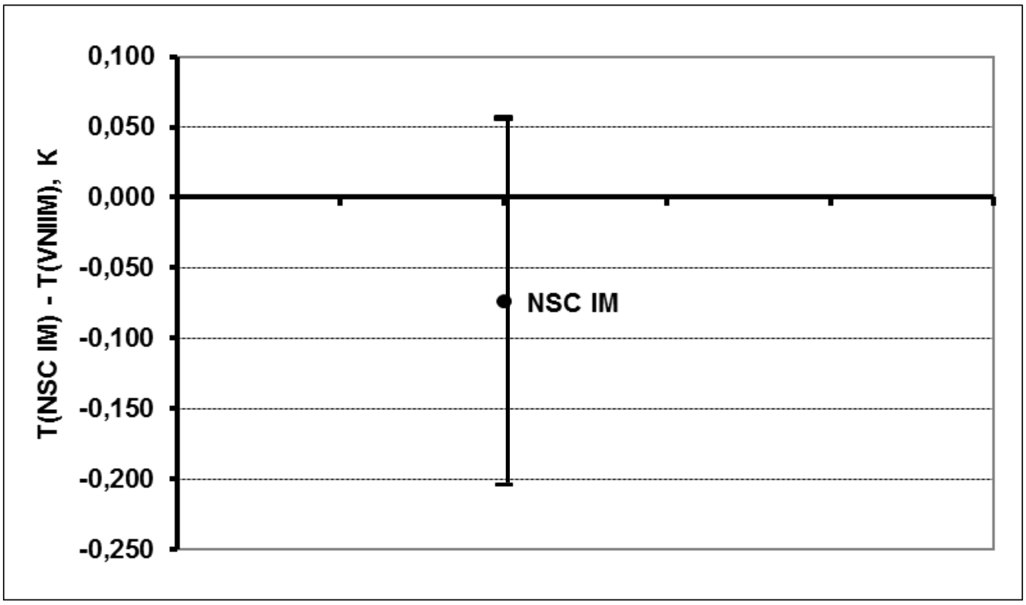



Fig. 3 – The comparison result for the NSC IM crucible, expanded uncertainty (*k* =2)

## 4 Results of the second stage of the comparison. The bilateral equivalence of the NSC IM standard relatively to the VNIIM standard

### 4.1 Comparison of lamp calibrations at 1084.62 °C

At the second stage, the comparison was carried out by means of a transfer standard – vacuum tungsten ribbon lamp SI 10-300 No. 5.

Information about furnaces, equipment, conditions of measurements, data processing for the measurements of the voltage instability at the lamp base and data processing for the measurements of the instability of the lamp base temperature are given in Appendix VI according to the Technical Protocol.

The information about measurements results and data processing is given in Appendix VII.

The information about the uncertainty budgets of the lamp calibration at the freezing point of copper is presented in Appendix VIII.

The average value of the lamp current $\boldsymbol{I}_{\mathbf{Cu}}$ at the copper fixed-point, the evaluation of standard uncertainties $u(I_{\mathrm{NMI}})$ presented by comparison participants, the difference $(I_{\mathrm{NSCIM}} - I_{\mathrm{VNIIM}})$ in A and the corresponding temperature difference $(T_{\mathrm{NSC\ IM}} - T_{\mathrm{VNIIM}})$ in K, the uncertainty of this difference are presented in Table 2 and Fig. 4. The combined uncertainty $u(T_{\mathrm{NSC\ IM}} - T_{\mathrm{VNIIM}})$ has been calculated on the basis of the budgets of the lamp calibration at the freezing point of copper submitted by comparison participants (Appendix

VIII) and taking into account the uncertainty of measurement results due to the transfer standard instability ***u(L)*** (Section 4.2):

$$u^2(T_{NMI} - T_{VNIIM}) = u^2(T_{NMI}) + u^2(T_{VNIIM}) + u^2(L) \, .$$

Table 2 – The results of calibration of the transfer standard at the copper fixed-point

| **NMI** | $I_{Cu}$**, A** | $u(I_{Cu})$ **K (*k* =1)** | $I_{NSC\,IM} - I_{VNIIM}$ **A** | $T_{NSC\,IM} - T_{VNIIM}$ **K** | $u(T_{NSC\,IM} - T_{VNIIM})$ **K (*k* =1)** |
|---|---|---|---|---|---|
| **VNIIM** | 9.1899 | 0.088 | -0.0016 | -0.162 | 0.255 |
| **NSC IM** | 9.1883 | 0.235 | | | |

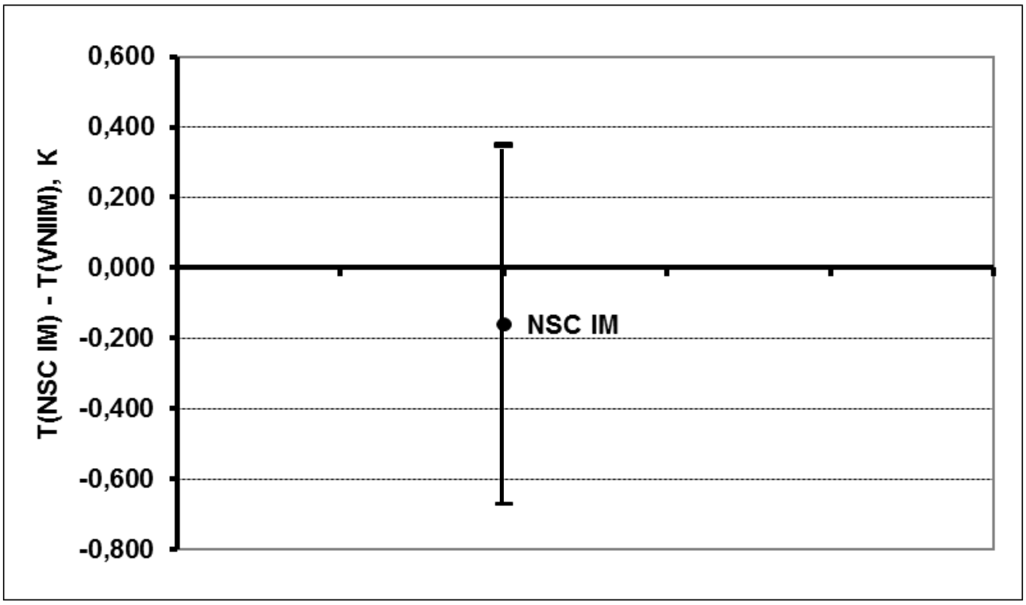



Fig. 4 – The comparison result for calibration of the temperature lamp – transfer standard at the copper fixed-point at NSC IM, expanded uncertainty (*k* =2)

### 4.2 Evaluation of the transfer standard instability

The uncertainty of the measurement results ***u(L)*** due to the transfer standard instability – vacuum tungsten ribbon lamp SI 10-300 No. 5, was calculated under the assumption that the distribution of the temperature drift of the lamp in time was rectangular and asymmetrical:

$$u(L) = \left[ (I_{VNIIM})_{end} - (I_{VNIIM})_{begin} \right] \times \frac{\partial T}{\partial I} \times \frac{1}{\sqrt{3}} \, .$$

The investigations carried out at VNIIM before and after the measurements made it possible to evaluate the standard uncertainty of the measurement results due to the transfer standard instability at the level of 0.045 K.

## 5 Comparison of the results of the first and second stages of the comparisons

The result of the first stage of comparisons is the difference of the temperature value reproduced by the fixed-point blackbody (crucible) of NSC IM under the VNIIM laboratory conditions from the value reproduced by the VNIIM fixed-point blackbody. The main reason for the difference in such a comparison is a different purity of metals in the crucibles. Thus, the difference obtained at the first stage of comparisons is interesting for evaluation of the correctness of the approach to estimation of the component associated with the purity of the metal.

The result of the second stage of comparisons is the difference of the temperature values of the copper fixed point realized by the national standards of NSC IM and VNIIM with the help of the transfer standard – vacuum tungsten ribbon lamp. In this case, there are many reasons for the differences: features of fixed-point blackbodies, characteristics of standard pyrometers, procedures for realization of phase-transitions at NMIs, techniques of calibration of the transfer standard at NMIs.

For illustration, the results of the first and second stages are shown in Table 3.

Table 3 – The results of the first and second stages of comparisons

| | **NSC IM – VNIIM** <br> **Δ*T*, K** |
|---|---|
| 1 stage | -0.074 |
| 2 stage | -0.162 |

As can be seen from Table 3, although the temperature difference obtained at the second stage, is more than twice the difference obtained at the first stage, both values are within the limits of the claimed uncertainties of the results. The results of the first stage of comparisons allowed estimating that the effect of impurities of metal of NSC IM crucible is slight.

## 6 Linkage of NSC IM result of the second stage of COOMET comparison with CCT-K5 results

The results of the COOMET comparisons were estimated in accordance with the document “Guidelines for data evaluation of COOMET key comparison”, 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 CCT-K5 comparisons.

The results of key comparisons K5 are presented as differences $[T_{\mathrm{NMI}} - KCRV(\mathrm{K5})]$ with the evaluations of their uncertainties for the fixed-points and fixed temperature values.

The degree of equivalence "***d***" of the NSC IM result at the copper fixed-point can be calculated on the corresponding relationship:

$$\boldsymbol{d}_{\mathrm{NSCIM}} = (T_{\mathrm{NSC\ IM}} - T_{\mathrm{VNIIM}}) + [T_{\mathrm{VNIIM}} - KCRV(\mathrm{K5})].$$

The standard uncertainty $u(d_{\text{NSC IM}})$ is evaluated by formula

$$u^2(d_{\text{NSC IM}}) = u^2(T_{\text{NSC IM}} - T_{\text{VNIIM}}) + u^2(T_{\text{VNIIM}} - KCRV).$$

The results of CCT-K5 obtained by VNIIM at the copper fixed-point are given in Table 4.

Table 4 – Results of CCT-K5 for VNIIM (Addendum to draft B of CCT-K5: from final results to KCDB values, April 2008)

| **Nominal temperature** | **($T_{\text{VNIIM}} - KCRV$) K** | **$U(T_{\text{VNIIM}} - KCRV)$ ($k$ =2), K** |
|---|---|---|
| 1084 | -0.103 | 0.408 |

**Copper fixed-point**

VNIIM: result of CCT-K5 $T_{\text{VNIIM}} - KCRV(\text{K5}) = -0.103$ K

result of COOMET $T_{\text{NSC IM}} - T_{\text{VNIIM}} = -0.162$ K

The equivalence of NSC IM result relatively to *KCRV*(K5):

$d_{\text{NSC IM}} = T_{\text{NSC IM}} - KCRV(\text{K5}) = -0.265$ K,

the standard uncertainty $u(d_{Cu}) = 0.327$ K.

## 7 Conclusion

The purpose of the comparison is the determination of the degree of equivalence of the NSC IM standard at the copper fixed-point relatively to the CCT-K5 results and the confirmation of the uncertainties, claimed by NSC IM for CMC. VNIIM was the linking NMI in this comparison.

Summary results of the comparison are presented in Table 5 and Fig. 5.

Table 5 – The degree of equivalence of the NSC IM standard and its expanded uncertainty

| **Fixed point** | **NMI** | **$d_{\text{NSC IM}} = T_{\text{NSC IM}} - KCRV(\text{K5})$, K** | **$U(d_{\text{NSC IM}})$ ($k$ =2) K** |
|---|---|---|---|
| *Cu* | NSC IM | -0.265 | 0.654 |

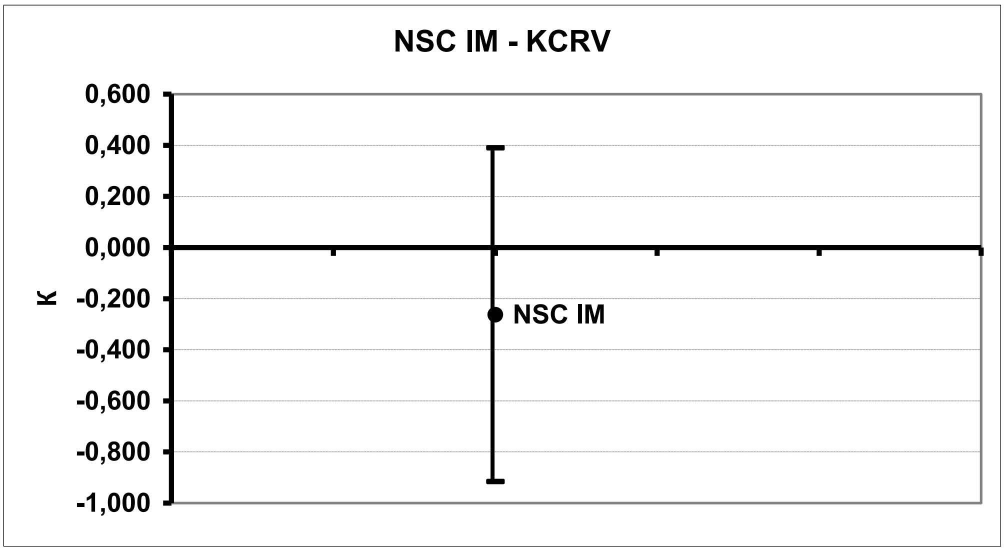



Fig. 5 – The difference [$T_{\mathrm{NSC\ IM}} - KCRV$(K5)] for the copper fixed-point, expanded uncertainty ($k$ =2)

According to the Document WG8 CCT "Inter-RMO CMC review committee 3-26-03", the values of the calibration uncertainty, claimed by participating laboratories, are acknowledged if the deviation value is less than its expanded uncertainty:

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

As can be seen, the result for $d_{\mathrm{NSC\ IM}}$ satisfies the above inequality.

# Appendix I

## TECHNICAL PROTOCOL (version 3)

## Comparison COOMET.T-K5
## Realizations of the ITS-90 at 1084.62 °C
## (COOMET Project 387/UA-a/07)



### Introduction

The decision about the necessity to perform regional comparisons of national measurement standards of the temperature unit in the field of non-contact thermometry was taken by the Technical Committee TC 1.10 "Thermometry and Thermal physics" of COOMET.

The linking institute for the dissemination of the metrological equivalence to the standards of the national metrology institutes is VNIIM (Russian Federation), which participated in the key comparisons CCT-K5.

The protocol covers two separate comparisons: a comparison of fixed point cells (Stage 1), and a comparison of lamp calibrations (Stage 2). The former is not directly comparable or linkable to the CCT-K5, whereas the latter is comparable and linkable. It will be the results of the latter comparison that are to be taken as the RMO-K5 results for linkage into the K5 data tables of the KCDB. The former, the cell comparison, results can still be used under the auspices of the MRA, as additional evidence for CMC support, but are more of a special regional supplementary comparison that happens at the same time as the KC.

The comparisons is restricted in the range of temperatures to "only" the copper point, and it is very clear that the Ukraine CMCs at higher or lower temperatures will not be supported by the use of this KC.

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

## 1 Organization of comparison

### 1.1 Participants and coordinator of comparison

Data about participants and coordinator of comparison are given in Table 1.

**Table 1**

| **Country,<br>National metrological institute** | **Address, contacts** |
|---|---|
| Ukraine<br>National Scientific Centre<br>«Institute of Metrology» (NSC IM)<br>**Coordinator of comparison**<br>Laboratory of thermometry and thermal measurements | Ukraine<br>61002, Kharkov,<br>Mironositskaya Str., 42<br><br>R.P.Sergiyenko<br>Rymma.Sergiyenko@metrology.kharkov.ua<br>Tel.: +038 057 704 98 00<br>Fax: +038 057 704 34 47 |
| Russian Federation<br>D.I. Mendeleev Institute for Metrology (VNIIM)<br>The Research Division on Thermodynamics | Russian Federation<br>190005, Saint-Petersburg,<br>Moskovsky prospect, 19<br>M.S.Matveyev<br>M.S.Matveyev@vniim.ru<br>Tel.: +7 812 323 96 37<br>Fax: +7 812 713 01 14 |

The linkage between participants and coordinator of all current issues should be carried out by these phones and e-mail addresses.

### 1.2 Transfer standard at the first stage of comparison

At the first stage of the comparison graphite crucible, containing pure metal and blackbody cavity, is used as a transfer standard. This transfer standard is a part of the national measurement standard of NSC IM (Ukraine) and is identified by the dimensions, weight and sketch. The graphite crucible is transported from NSC IM to VNIIM where it is compared with the corresponding crucible (fixed point blackbody) of VNIIM on the basis of national measurement standard of the Russian Federation with the use of equipment of this national standard.

Transfer standard should be transported from NSC IM to VNIIM and back in a special box. The coordinator – NSC IM will send the graphite crucible (selfmade in Ukraine) number 532 in its wooden box to participating laboratory of VNIIM. Since the crucible is extremely fragile, its transfer from laboratory to laboratory must be performed only by participants as a hand luggage and with great care.

To comply with customs regulations when crossing state borders the declaration – the ATA Carnet form – must be fulfilled carefully, indicating the recipient country and dates of import and export of the crucible. The issue of making the customs payment should be resolved between the participating laboratories.

It is the responsibility of each laboratory to arrange transport of the crucible No 532 after calibration back to the coordinator. It is the responsibility of each laboratory to obtain insurance for the crucible No 532.

For customs declaration:

The following artifact is circulated among the participants for calibration:

The graphite crucible No 532.

The purchase/manufacturing cost of artifact is uncertain. It 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 please contact the relevant person so that he/she can be present and help you unpack it.

Participating laboratories are responsible for transferring the crucible to coordinator, its return and customs registration for export from the Ukraine and import back in accordance with the ATA Carnet document.

Custom expenses on the Russian Federation territory are paid by the comparisons coordinator. Description of crucible has been used as the transfer standard of the comparison is given in Appendixes A and B.

An inspection procedure to be followed and e-mail sent to the pilot indicating either safe arrival or detailing the concerns after delivering of the transfer standard.

### 1.3 Transfer standard at the second stage of comparison

At the second stage of the comparison, temperature vacuum tungsten strip lamp is used as a transfer standard. This transfer standard is delivered by VNIIM and is identified by number placed on the top of lamp base.

VNIIM delivers a transfer standard with the following information:

- instruction as to the handling of the lamp, including an indication of the maximum lamp current, not to be surpassed. Current switching and setting (maximum A/min);
- temperature coefficients $dT_\lambda/dt_b$, where $T_\lambda$ and $t_b$ represent the spectral radiance temperature and the lamp base temperature, respectively;
- dependence of spectral radiance temperature on wavelength $dT_\lambda/d\lambda$;
- sensitivity of the lamp calibration curve $dI/dT_\lambda$ at the freezing point, where $I$ – the value of lamp current, $dT_\lambda$ – 1 °C;
- horizontal and vertical temperature distribution on the lamp strip.

Transfer standard should be transported from VNIIM to NSC IM and back in a special box, to be hand carried when transferring the lamp between the participating institutes.

Prior to operation the measurement of the room-temperature resistance of the lamp must be carried out. This measurement should be done with uncertainty less 0.01 %.

Two double-wire cables are used as current leads and voltage leads such that the room-temperature resistance $R_{amb}$ of the lamp elements, comprising tungsten strip and its internal support, can be measured (along with the temperature $T_{amb}$, measured with a calibrated resistance thermometer inserted in the lamp base) just before calibrating them. After the calibration has been completed $R_{amb}$ and $T_{amb}$ should be remeasured just before transferring the lamps to their next destination. All of this is to additionally monitor changes in the physical constitution of the lamp element, possibly induced during transport of the lamps.

An inspection procedure to be followed and e-mail sent to the pilot indicating either safe arrival or detailing the concerns after delivering of the transfer standard and informing about $T_{amb}$, $R_{amb}$ values.

The owner – VNIIM will send the lamp type СИ (SI) 10-300 (made by Moscow plant MELZ, Russia) No. 5 in its wooden box to participating laboratory of NSC IM. Since the lamp is extremely fragile, its transfer from laboratory to laboratory must be performed only by participants as a hand luggage and with great care.

To comply with customs regulations when crossing state borders the declaration – the ATA Carnet form – must be fulfilled carefully, indicating the recipient country and dates of import and export of the lamp. The issue of making the customs payment should be resolved between the participating laboratories.

It is the responsibility of each laboratory to arrange transport of the lamp No 5 after calibration back to the owner. It is the responsibility of each laboratory to obtain insurance for the lamp No 5.

For customs declaration:

The following artifact is circulated among the participants for calibration:

The transfer standard of the comparison – Tungsten ribbon lamp type СИ 10-300 (made by Moscow plant MELZ, Russia) No 5.

The purchase/manufacturing cost of artifact is uncertain. It 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 please contact the relevant person so that he/she can be present and help you unpack it.

Participating laboratories are responsible for transferring the lamp to coordinator, its return and customs registration for export from the Russia and import back in accordance with the ATA Carnet document.

Custom expenses on the Ukrainian territory are paid by the comparisons coordinator.

Description and characteristics of lamp has been used as the transfer standard of the comparison are given in Appendix C.

Rules for transportation of transfer standard is given in Appendix D.

Description of operation with transfer standard is given in Appendix E.

### 1.4 Comparison scheme and schedule

Scheme of the first stage of comparison is presented in Fig.1.

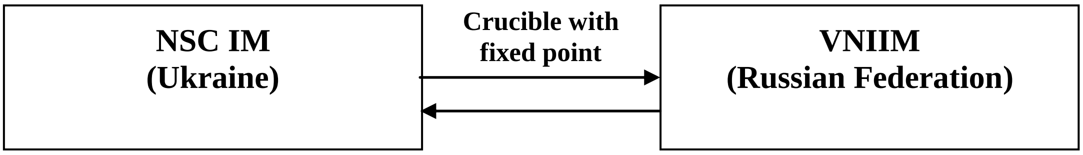



Fig. 1 – Scheme of the first stage of comparison

Scheme of the second stage of comparison is presented in Fig. 2.

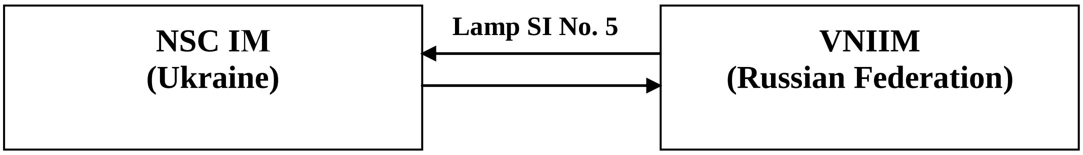



Fig. 2 – Scheme of the second stage of comparison

Time schedule:
Measurements: 1 August 2014 – 31 August 2014
Data processing and compilation of report "A": 1 September 2014 – 30 September 2014
Preparation of report "B": 1 October 2014 – 15 December 2014

## 2 Instructions on measurements

The purpose of the measurements at the first stage is to determine the deviation of the temperature value reproduced by the fixed-point blackbody (crucible) of NSC IM under the VNIIM laboratory conditions from the value reproduced by the VNIIM fixed-point blackbody. The measurements are performed using the standard pyrometer-comparator (SPC) that has a well-known effective wavelength in the visible range of spectrum.

The purpose of the measurements at the second stage is to establish the degree of equivalence of the NSC IM standard with respect to the results of the key comparison CCT-K5. At stage 2, the transfer standard – temperature lamp is calibrated through determining the value of current in circuit of this lamp during equalization of spectral brightness of corresponding area of the lamp strip with brightness of radiation of blackbody cavity with temperature of freezing point of pure metal. Equalization of brightness is realized with the help of the standard monochromatic pyrometer (or the standard spectral pyrometric setting), which is giving monochromatic radiation with known effective wavelength in the visible range of spectrum. Applied furnace with the model of blackbody and standard pyrometer should be included into primary standards of temperature unit in the field of non-contact thermometry of participating countries.

### 2.1 General requirements

<u>2.1.1 Conditions of measurements</u>

During the measurements it is necessary to control and record:

- ambient temperature
- relative humidity
- pressure

Indicated parameters should be in the limits of values, which are required in routine practice of participating laboratory. Actual values of parameters during the measurements should be indicated in protocol of measurements.

2.1.2 Reference conditions of measurements

So-called "reference" conditions of measurements apply to obtain comparable results of measurements, conducted in different laboratories. Reference conditions of measurements are:

- effective wavelength $\boldsymbol{\lambda}_{\mathbf{re}}$ = 656.3 nm;

- base temperature of transfer standard $\boldsymbol{t}_{\mathbf{re}}$ = 22.5 $^{o}$C (at stage 2).

**2.2 Stage 1**

2.2.1 Transfer standard

2.2.1.1 To avoid damage of the graphite crucible the last one should be kept according to rules for treatment of fragile devices that have been adopted in the participating laboratory.

2.2.1.2 Before the measurements, it is necessary to check that there are no defects on the surface of the graphite crucible.

2.2.1.3 Mounting the crucible into heating furnace should be done in accordance with the procedure adopted in the participating laboratory.

2.2.1.4 Rate of heating and cooling should be chosen on the basis of preliminary researches of furnace, in which the crucible of specified configuration and mass of the metal is placed.

2.2.1.5 At temperatures above 350 °C, it is necessary blowing of crucible using pure argon to prevent burning-out graphite.

2.2.1.6 During the measurements, it is necessary to record the total operating time of the graphite crucible.

2.2.2 Standard pyrometer-comparators

2.2.2.1 The optical system of the standard pyrometer-comparators (SPC) should provide the clear image of the aperture of the blackbody cavity.

2.2.2.2 The diameter of the field of view, the effective wavelength and half-width of spectral response band are specified by technical and metrological characteristics, indicated in the documentation for this SPC.

2.2.2.3 The diameter of the field of view should not exceed 1.0 mm.

2.2.2.4 The optical systems should provide the clear images of the lamp strip, the index and the cross, placed on the balloon of lamp.

2.2.2.5 The effective wavelengths should not differ from reference value, more than 10.0 nm, it means it should be in the limits ($\boldsymbol{\lambda}_{\mathbf{re}}$ ± 10.0) nm, $\boldsymbol{\lambda}_{\mathbf{re}}$ = 656.3 nm.

2.2.2.6 The half-width of spectral responsivity bands should not exceed 20.0 nm.

2.2.2.7 The use of SPC should be done according to Manual for this SPC.

2.2.2.8 The model of SPC of NSC IM is EOP serial No 2.

2.2.2.9 The model of SPC of VNIIM is SP-4K serial No 2.

**2.3 Stage 2**

2.3.1 Transfer standard

2.3.1.1 Description of operations with transfer standard is given in Appendix E.

Order of storage of transfer standard before and after conducting measurements should follow the rules of storage of standard lamps, which are included into primary standard.

2.3.1.2 Before starting the measurements, cleaning the window is necessary. The window of the lamp should be cleaned with a few drops of pure alcohol and immediately drying using lens cleaning paper. The contacts of lamp base should be also cleaned.

2.3.1.3 Order of installation and orientation of transfer standard:

- lamp is exploited in vertical position;
- plus of power source is given to the lamp base;
- direction of viewing should be athwart to the lamp strip;
- place of viewing is indicated by index;
- lamp is oriented in a such way that the optical axis of standard pyrometer goes parallel to the line, connecting the end of index with the center of the cross, placed on the balloon of lamp.

2.3.1.4 Before each cycle of measurements, the lamp should be adjusted once again. During the measurements, the adjustment should be controlled and should be corrected if necessary.

2.3.1.5 After switching on, the lamp current is increased with the velocity not more than 0.5 A/min.

2.3.1.6 After switching on, the burning time of lamp should be not less than 30 min. (for vacuum lamp not less than 40 min.) to achieve the temperature stabilization. A criterion of temperature stabilization is drift of voltage at the lamp base under the known value of current. The drift of voltage should not exceed 0.005 % during 10 minutes.

2.3.1.7 To avoid hysteresis it is necessary to change current in lamp circuit from small values to bigger ones.

2.3.1.8 During the measurements, it is necessary to record the burning time of the lamp. Total burning time of the lamp under current, corresponded to freezing point of **me**tal ($\boldsymbol{I} \geq 0.95\boldsymbol{I}_{\mathbf{me}}$), should not exceed 2 hours.

2.3.1.9 During the measurements, it is necessary to stabilize the base temperature of the lamp, which should be controlled and registered.

2.3.1.10 To control the stability of characteristics of transfer standard, control of voltage at the lamp base should be conducted before, during and after the measurements.

2.3.1.11 To control the stability of characteristics of transfer standard (the room-temperature resistance) after back delivering to VNIIM, must be done in accordance with paragraph 1.3 in 2 weeks.

2.3.1.12 The results of measurements to control the stability of characteristics of transfer standard after back delivering to VNIIM, must be sent to the pilot laboratory by e-mail in period of 1 week after measurements. A probable drift component or lamp instability have to be incorporated into the final uncertainty of comparison.

2.3.2 Standard pyrometer (standard pyrometer-comparator)

The Standard pyrometer-comparators are used in both laboratories the same that described in paragraph 2.2.2

## 2.4 Measurements

2.4.1 Stage 1

In general, the measurement cycle contains the following procedures:

2.4.1.1 Realization of melting plateau for the NSC IM crucible: $T_m^{NSC}$.

2.4.1.2 Processing experimental data on melting plateau.

2.4.1.3 Realization of freezing plateau for the NSC IM crucible: $T_f^{NSC}$.

2.4.1.4 Processing experimental data on freezing plateau.

2.4.1.5 Calculation of difference $\Delta T_{m\text{-}f}^{NSC}$ for the NSC IM crucible:

$$\Delta T_{m\text{-}f}^{NSC} = (U_m^{NSC} - U_f^{NSC})/(dU/dT), \quad (1)$$

where $U_m^{NSC}$ and $U_f^{NSC}$, mV – the output signals of the standard pyrometer at melting and freezing point, respectively;

$dU/dT$, mV/1°C – sensitivity of the standard pyrometer at the fixed-point temperature.

2.4.1.6 Realization of melting plateau for the VNIIM crucible: $T_m^{VNIIM}$.

2.4.1.7 Processing experimental data on melting plateau.

2.4.1.8 Realization of freezing plateau for the VNIIM crucible: $T_f^{VNIIM}$.

2.4.1.9 Processing experimental data on freezing plateau.

2.4.1.10 Calculation of difference $\Delta T_{m\text{-}f}^{VNIIM}$ for the VNIIM crucible:

$$\Delta T_{m\text{-}f}^{VNIIM} = (U_m^{VNIIM} - U_f^{VNIIM})/(dU/dT), \quad (2)$$

where $U_m^{VNIIM}$ and $U_f^{VNIIM}$, mV – the output signals of the standard pyrometer at melting and freezing point, respectively;

$dU/dT$, mV/1°C – sensitivity of the standard pyrometer at the fixed-point.

2.4.1.11 Amount of measurement cycles, each of which includes points from 2.4.1.1 to 2.4.1.10, should be not less than 5.

2.4.2 Stage 2

In general, the measurement cycle contains the following procedures:

2.4.2.1 Realization of freezing plateau of pure **Me**tal: $\boldsymbol{T_{90}}$**(Me)**.

2.4.2.2 Processing experimental data on plateau. If necessary, input of corrections to $\boldsymbol{T_{90}}$**(Me)**: $\boldsymbol{T^*}$.

It is supposed that the corrections to $\boldsymbol{T_{90}}$**(Me)** due to such sources, as impurities, emissivity of the blackbody cavity, size-of-source effect and other, which are taken into account by participants of comparisons.

2.4.2.3 Calibration of the transfer standard for spectral **r**adiance **t**emperature $\boldsymbol{T_{rt}}$: $\boldsymbol{T^* \equiv T_{rt} = f(I, \lambda_{eff}, t_b)}$, where $\boldsymbol{\lambda_{eff}}$ – the effective wavelength of the applied standard pyrometer; $\boldsymbol{t_b}$ – the lamp base temperature; $\boldsymbol{I}$ – lamp current. The result is the average of at least 10 measurements of lamp current $\boldsymbol{I}$.

2.4.2.4 Input of correction $\Delta T_{\lambda eff}$ to the value of $\boldsymbol{T_{rt}}$, which is caused by the difference of effective wavelength $\boldsymbol{\lambda_{eff}}$ of applied standard pyrometer from the reference value $\boldsymbol{\lambda_{re}}$ = 656.3 nm.

For input of this correction, comparison participants use the data $\boldsymbol{dT_\lambda/d\lambda}$, which are presented in accompanying documents together with the lamp:

$$\Delta T_{\lambda eff} = (\lambda_{eff} - \lambda_{re}) \cdot dT_\lambda/d\lambda. \quad (3)$$

2.4.2.5 Input of correction $\Delta T_b$ to the value of $\boldsymbol{T_{rt}}$, which is caused by the difference of lamp base temperature $\boldsymbol{t_b}$ under measuring from the reference value $\boldsymbol{t_{re}}$ = 22.5 °C.

For input of this correction, comparison participants can use one of two variants:

a) using the data on temperature coefficient $dT_\lambda/dt_b$, which are presented in accompanying documents together with the lamp:

$$\Delta T_b = (\boldsymbol{t_b} - \boldsymbol{t_{re}}) \cdot dT_\lambda/dt_b. \quad (4)$$

b) determining the temperature coefficient of the transfer standard independently (in this case the uncertainty of this correction value is evaluated by participant).

Because of corrections according to 2.4.2.4 and 2.4.2.5, the corrected value $\boldsymbol{T_{rt}}^{©}$ should be:

$$\boldsymbol{T_{rt}}^{©} = \boldsymbol{T_{rt}} - \Delta T_{\lambda eff} - \Delta T_b. \quad (5)$$

2.4.2.6 Input of correction to the lamp current value $\boldsymbol{I}$ for transformation of $\boldsymbol{T}_{\mathbf{rt}}^{©}$ to $\boldsymbol{T}_{\mathbf{90}}$**(Me)**:

$$\boldsymbol{I}_{\mathbf{90}} = \boldsymbol{I} + [\boldsymbol{T}_{\mathbf{90}} - \boldsymbol{T}_{\mathbf{rt}}^{©}]\cdot[dI/dT_{\lambda}]. \qquad (6)$$

For input of this correction, comparison participants can use one of two variants:

a) using the data on sensitivity of the lamp calibration curve $dI/dT_{\lambda}$, which are presented in accompanying documents together with the lamp;

b) determining the sensitivity of the lamp calibration curve independently (in this case the uncertainty of this correction value is evaluated by participant).

2.4.2.7 Amount of measurement cycles, each of which includes points from 2.4.2.1 to 2.4.2.6, should be not less than 5.

## 3 Presentation of results

### 3.1 Stage 1

VNIIM shall submit information according to the form of Tables 2, 3 and 4.

NSC IM shall submit information concerning the characteristics of the graphite crucible with the fixed-point according to the form of Table 2.

The names of values in Tables 3 and 4 correspond to the ones, given in section 2.4.1.

### 3.2 Stage 2

Each participating laboratory shall submit information according to the form of Tables 5 and 6. The names of values in Tables 5 and 6 correspond to the ones, given in section 2.4.2.

Table 2

| **COOMET Project 387/UA-a/07, stage 1** | **National Metrology Institute:_______** |
|---|---|
| Parameter item | Value/Comments |
| **Radiator with the fixed-point: _____** | |
| Furnace: manufacturer / type | |
| Purity of the metal | |
| Effective emissivity of the cavity | |
| Dimensions of the graphite crucible | |
| Mass of the metal, g | |
| Diameter of the aperture of the blackbody cavity, mm | |
| Mounting the graphite crucible (if necessary) | |
| Total operating time of the graphite crucible | |
| **Standard pyrometer-comparator (SPC)** | |
| Diameter of field of view, mm | |
| Effective wavelength, nm | |
| Half-width of the spectral responsivity band, nm | |
| Focus distance of lens, mm | |
| Aperture ratio | |
| **Conditions of measurements** | |
| Ambient temperature: maximum, minimum and average from all measurement cycles, $^{o}$C | |
| Relative humidity: maximum, minimum and average from all measurement cycles, % | |
| Pressure: maximum, minimum and average from all measurement cycles, kPa | |

Table 3

| **COOMET Project 387/UA-a/07, stage 1** **Fixed point blackbody of NMI: _____** | | | | | | |
|---|---|---|---|---|---|---|
| Number of cycle: melting-freezing | Plateau duration, minutes [1] | Plateau slope, K/min | $U_m^{NMI}$, mV / $U_f^{NMI}$, mV | $dU/dT_{Cu}$, mV/1 °C | $\Delta T_{m-f}^{NMI}$, °C | SPC output signal in temperature equivalent, °C |
| 1-melting | | | | | | - |
| 1-freezing | | | | | | - |
| 2-melting | | | | | | - |
| 2-freezing | | | | | | |
| … | | | | | | - |
| 5-melting | | | | | | - |
| 5-freezing | | | | | | - |

[1] - participant can present the shape of plateaus of the fixed points

Table 4

| **COOMET Project 387/UA-a/07, stage 1** **Fixed point blackbody of NMI: _____** | | | |
|---|---|---|---|
| **Comparison of crucibles for realization of the freezing fixed-point of NSC IM and VNIIM** | | | |
| Number of freezing plateau | SPC output signal in temperature equivalent, °C | | $\Delta T = T_f^{NSC} - T_f^{VNIIM}$, °C |
| | $T_f^{NSC}$, °C | $T_{f.}^{VNIIM}$, °C | |
| 1 | | | |
| 2 | | | |
| 3 | | | |
| 4 | | | |
| 5 | | | |

Table 5

| **COOMET Project 387/UA-a/07, stage 2** | **National Metrology Institute:______** |
|---|---|
| **Parameter item** | **Value/Comments** |
| **Radiator with the fixed-point: _____** | |
| Type (1, 2 or 3 zones) | |
| Purity of the metal | |
| Mass of the metal, g | |
| Diameter of the aperture of the blackbody cavity, mm | |
| **Standard pyrometer (or standard spectropyrometric setting)** | |
| Diameter of field of view, mm | |
| Half-width of spectral responsivity band, nm | |
| Focus distance of lens, mm | |
| Aperture ratio | |
| **Temperature lamp – the transfer standard** | |
| Features of lamp orientation, if they differ from 2.3.1.3 | |
| Instability of voltage at lamp base during measurements, procedure of instability evaluation | |
| Instability of lamp base temperature during measurements, procedure of instability evaluation | |
| Total burning time of the lamp | |
| Operating time of lamp at fixed-point temperature ($\boldsymbol{I} \geq 0.95\boldsymbol{I}_{\mathbf{Me}}$) | |
| **Conditions of measurements** | |
| Ambient temperature: maximum, minimum and average from all measurement cycles, $^{o}$C | |
| Relative humidity: maximum, minimum and average from all measurement cycles, % | |
| Pressure: maximum, minimum and average from all measurement cycles, kPa | |

Table 6

| **COOMET Project 387/UA-a/07, stage 2** | | | | **National Metrology Institute:_________** | | | | |
|---|---|---|---|---|---|---|---|---|
| Number of freezing plateau | Plateau duration, minutes | $\boldsymbol{T^*}$, **K** [1] | $\boldsymbol{I}$, A [2] for $\boldsymbol{T^* \equiv T_{rt} = f(I, \lambda_{eff}, t_b)}$ | $\boldsymbol{\lambda_{eff}}$, nm | $\boldsymbol{\Delta T_{\lambda eff}}$, K [3] | Lamp base temperature, $\boldsymbol{t_b}$, °C [2] | $\boldsymbol{\Delta T_b}$, K [4] | $\boldsymbol{I_{90}}$, A for $\boldsymbol{T_{90} = f(I_{90}, \lambda_{re}, t_{re})}$ |
| 1 | | | | | | | | |
| 2 | | | | | | | | |
| 3 | | | | | | | | |
| 4 | | | | | | | | |
| 5 | | | | | | | | |
| | | | | | | | **Average** | |

[1] temperature value, calculated according to item 2.4.2.2

[2] measured value

[3] temperature value, calculated according to item 2.4.2.4

[4] temperature value, calculated according to item 2.4.2.5

## 4 Uncertainty Budget

The standard uncertainties of type A and type B, the combined standard uncertainty should be evaluated according to "**Guide to the Expression of Uncertainty in Measurement**" (GUM) and document of CCT-WG5 on radiation thermometry "**Uncertainty budgets for realization of scales by radiation thermometry**".

### 4.1 Stage 1

Initial data for calculation of standard uncertainty of type A is a number of measurement results

$$\Delta T = T_f^{NSC} - T_f^{VNIIM}, \qquad (7)$$

which consists of 5 values as a minimum according to 2.4.1.11 and Table 4.

The sources of uncertainty components of type B are given by equipment and procedures, used during the measurement. At the first stage, these sources can be divided into following groups:

- furnace with the model of blackbody: impurities, emissivity, temperature gradients in the cavity and plateau identification;

- standard pyrometer (standard pyrometer-comparator): size-of-source effect; uncertainty of knowledge of effective wavelength, scattering, polarization (spectral parameters); instability of standard pyrometer, measurement of output signal (electrical parameters); and others.

Evaluation of uncertainty components is given by participants in corresponding items of uncertainty budget according to the form of Table 7.

### 4.2 Stage 2

Initial data for calculation of standard uncertainty of type A is a number of measurement results $\boldsymbol{I_{90}}$, which consists of 2 x 5 values as a minimum (including measurements before and after lamp transport) according to 2.4.2.7 and Table 6. Transformation of standard uncertainty, expressed in current units, into standard uncertainty in temperature units is made through the use of sensitivity of the lamp calibration curve $dI/dT_\lambda$ (2.4.2.6).

At the stage 2, the components, associated with temperature lamp, are added to the above-mentioned uncertainty components:

- measurement of lamp current; measurement of lamp base temperature; dependence $dT_\lambda/d\lambda$ and difference of effective wavelength $\lambda_{eff}$ of applied standard pyrometer from reference value $\lambda_{re}$; temperature coefficient of lamp $dT_\lambda/dt_b$ and difference of base temperature $t_b$ from reference value $t_{re}$; procedure of transformation of values in current units into values in temperature units and others.

Evaluation of uncertainty components is given by participants in corresponding items of uncertainty budget according to the form of Table 7.

Table 7

| **Uncertainty budget** | |
|---|---|
| **COOMET Project 387/UA-a/07** | **NMI: _______** |
| Component of uncertainty | Standard uncertainty, мK |
| Type A | |
| Repeatability of the $I_{90}$ values (including measurements before and after lamp transport) | |
| Type B | |
| **Fixed-point blackbody radiator** | |
| Impurities | |
| Emissivity | |
| Plateau identification | |
| The temperature difference in the walls of the cavity, heat transfer processes | |
| …… | |
| **Standard pyrometer** | |
| Spectral parameters | |
| Size-of-source effect | |
| Drift (instability) | |
| Resolution | |
| …… | |
| ……. | |
| **Temperature lamp** | |
| Current measurement | |
| Base temperature | |
| Positioning (adjustment) | |
| …….. | |
| ……. | |
| **Converting results to the reference conditions** | |
| Interpolation and integration | |
| Converting to the reference base temperature | |
| Converting to the reference effective wavelength | |
| …… | |
| …… | |
| | |
| The combined uncertainty, mK ($k$ = 1) | |
| The expanded uncertainty, mK ($k$ = 2) | |

### 4.3 Comparison results

In this COOMET comparison VNIIM is the linking institute for determination of the equivalence degree of the obtained results relative to CCT-K5 results.
The results of CCT-K5 have been presented as differences [$T_{NMI}$ - *KCRV(K5)*].
The degree of equivalence "*d*" of the NMI results for each fixed reference point can be calculated on the corresponding relationship:

$$d_i = (T_{NMI} - T_{VNIIM}) + \left[T_{VNIIM} - KCRV(K5)\right].$$

The standard uncertainty *u(d)* can be calculated with the corresponding relationship:

$$u^2(d) = u^2(T_{NMI} - T_{VNIIM}) + u^2\left[T_{VNIIM} - KCRV(K5)\right],$$

where $u(T_{\mathrm{NMI}} - T_{\mathrm{VNIIM}})$ can be calculated on the corresponding relationship:

$$u^2(T_{NMI} - T_{VNIIM}) = u^2(T_{NMI}) + u^2(T_{VNIIM}) + u^2(L),$$

where *u(L)* – standard uncertainty of lamp instability during VNIIM-NMI-VNIIM,

$$u(L) = \left[(I_{VNIIM})_{end} - (I_{VNIIM})_{begin}\right] \times \frac{\partial T}{\partial I} \times \frac{1}{\sqrt{3}}$$

The values of the calibration uncertainty, claimed by participating laboratories, are acknowledged if the deviation value is less than its expanded uncertainty:

$$/d/ < 2u(d)$$

(Document WG8 CCT "Inter-RMO CMC review committee 3-26-03").

# Appendix A

## Characteristics of crucible has been used as the transfer standard of the comparison (NSCIM's crucible)

| | | | |
|---|---|---|---|
| Make by: | NSCIM | Size: | Diameter 41.5 mm, Length 168.4 mm |
| Type: | No name | Weight: | 610 g |
| Serial Number: | 532 | Packaging: | Special wooden box with foam pad |

Fig. 1 Design of artifact:

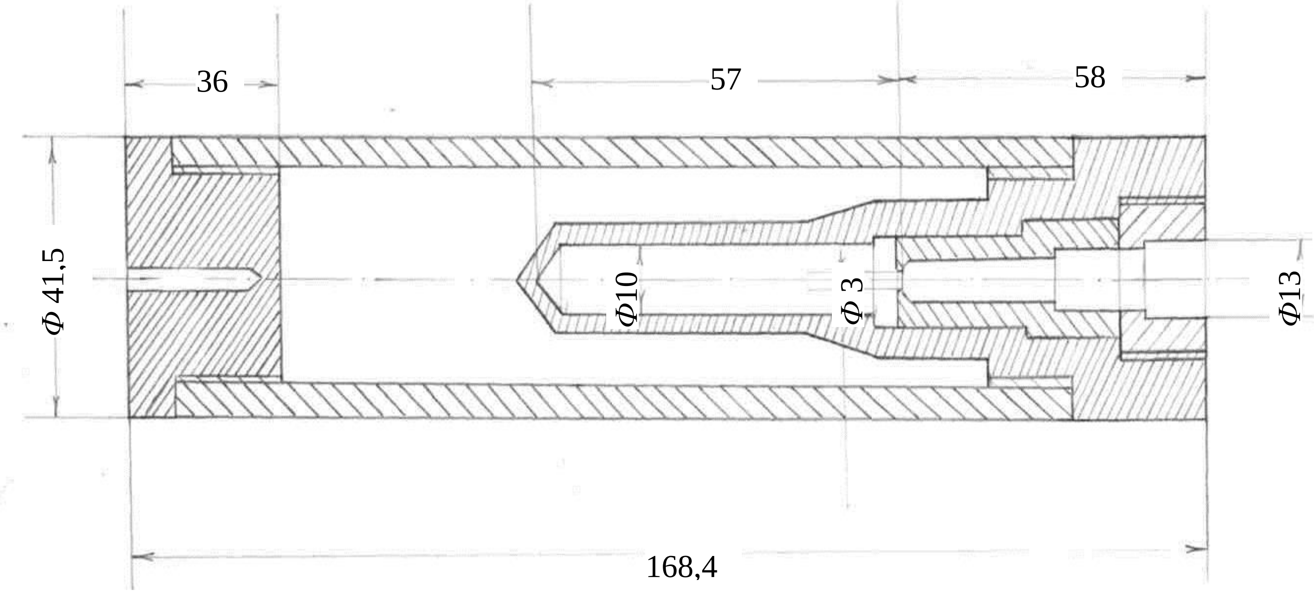


Fig. 2 Photos of artifact:

a) Crucible


b) Crucible in packed box

# Appendix B

## The transfer standard of the comparison – Graphite crucible containing pure copper

### Complete list of the content of the package

| | |
|---|---|
| Type: | Noname |
| Serial Number: | 532 |
| Make by: | National Scientific Centre «Institute of Metrology» (NSC IM) |
| Size: | Diameter 41.5 mm, Length 168 mm |
| Mass: | 610 g (Netto), |
| Owner: | National Scientific Centre «Institute of Metrology» (NSC IM) |
| Address: | 42, Mironositskaya Str. Kharkov, 61002, Ukraine |
| Packaging: | Special box with foam pad |
| Size: | 90 x 200 x 320 mm |
| Mass: | 1.1 kg (Crucible with packaging) |

### Handbook

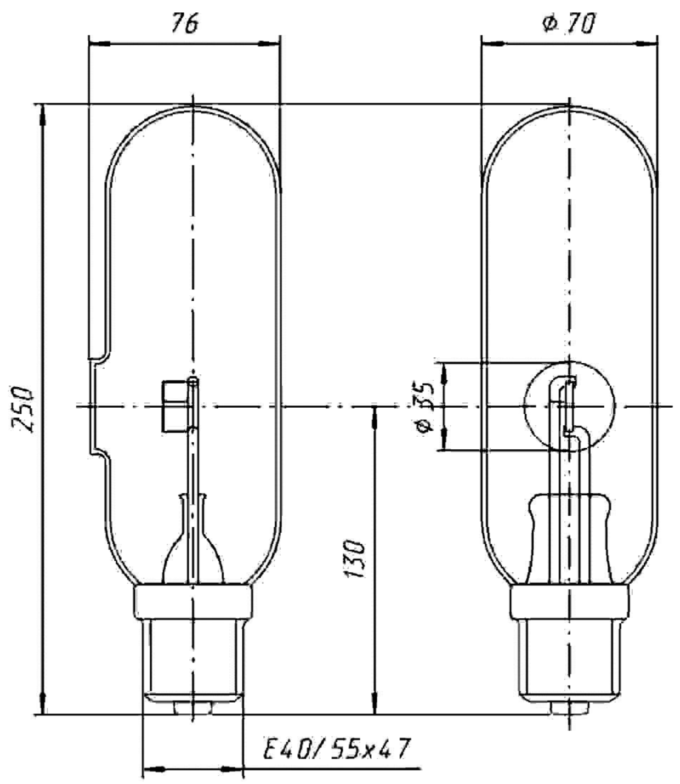
1. Transportation of crucible must be only by courier.
2. Should provide protection from bumps and a crucible sharp shock.
3. Should provide protection from heat a crucible above 50 °C.
4. It is forbidden to transport crucible by luggage.
5. When air transport crucible should be at the courier in the cabin.

# Appendix C

## Characteristics of lamp has been used as the transfer standard of the comparison (VNIIM's lamp)

| | |
|---|---|
| Make by: | Plant "МЭЛЗ" |
| Type: | СИ 10 - 300 |
| Serial Number: | 5 |
| Size: | Diameter 76 mm, Length 250 mm |
| Mass: | 180 g |
| Packaging: | Special box with foam pad |

Fig. 1 Design of artifact:




The Tungsten Ribbon Lamp of VNIIM

Ribbon width - 2.8 mm, length - 20+2x12 mm, thickness - 0.06 mm

Fig. 2 Photos of artifact:

a) Lamp

b) Lamp in packing box

# Appendix D

## The transfer standard of the comparison – Tungsten ribbon lamp

### Complete list of the content of the package

| | |
|---|---|
| Type: | СИ 10 - 300 |
| Serial Number: | 5 |
| Make by: | Plant "МЭЛЗ", Moscow, Russia (Formed USSR) |
| Size: | Diameter 76 mm, Length 250 mm |
| Mass: | 180 g (Netto) |
| Owner: | D.I. Mendeleyev Institute for Metrology (VNIIM) |
| Address: | 19 Moskovsky prospect , Saint-Petersburg, 190005, Russia |
| Packaging: | Special box with foam pad |
| Size: | 150 x 150 x 450 mm |
| Mass: | 2.8 kg (Mass with package) |

### Handbook

1. Transportation of lamp must be only by courier.
2. Should provide protection from bumps and a lamp sharp shock.
3. Should provide protection from heat a lamp above 50 °C.
4. It is forbidden to transport lamp by luggage.
5. When air transport lamp should be at the courier in the cabin.

# Appendix E

## Features of the transfer standard – vacuum tungsten ribbon lamp СИ 10-300 No. 5 (stage 2 of the comparison)

| | |
|---|---|
| Name | - Temperature vacuum tungsten ribbon lamp |
| Type of lamp | - СИ 10-300 |
| Serial number | - 5 |
| Date of manufacture | - 1971 |
| Manufacturer | - Lamp Factory "МЭЛЗ", Moscow |
| State | - the USSR (now - the Russian Federation) |
| City | - Moscow |
| Belongs | - D.I. Mendeleev Institute for Metrology (VNIIM) |
| Operating time | - since 1990 |
| The highest operating temperature | - 1300 $^{o}$C |

The researches of the lamp were carried out from 2008 to 2010 on the state primary measurement standard of the temperature unit of the Russian Federation with the aim to use it as a transfer standard.

The researches were carried out under the following conditions:

| | | |
|---|---|---|
| ambient temperature | – | (22 ± 2) °C |
| relative humidity | – | (55 ± 10) % |
| pressure | – | (105 ± 15) kPa |

The researches were conducted at the effective wavelength 656.3 nm centering of the target area 0.7 mm x 0.7 mm at the level of the index.

Conditions of use:
The stabilized current must be of unambiguous polarity – plus of power source is given to the thread of the lamp base.

The rate of change of the lamp current in the circuit should not exceed:

| | | |
|---|---|---|
| in the range of 0 to 6 A | – | 10 mA/s |
| in the range of 6 to 9 A | – | 6 mA/s |
| over 9 A | – | 3 mA/s |

When switching off, the current change rate should not exceed these values.

The measurements should be performed in the direction of increasing current only to avoid the hysteresis effect.

Before the measurements, the lamp window should be cleaned with few drops of pure alcohol and finally with distilled water, and immediately dried with the help of the lens cleaning paper.

The lamp must be installed vertically with the base down. It is important not to touch the glass by hands and use a clean cloth or gloves.

Positioning: The lamp should be oriented in such way that the optical axis of the standard pyrometer goes parallel to the line, connecting the end of index with the center of the cross, placed on the rear wall of the lamp bulb.

The base of the lamp should be thermostated during the measurements.

The current, the voltage, the lamp base temperature and the ambient temperature should be recorded during the measurements.

It is important to check the resistance of the lamp periodically. Changing the resistance of more than 0.01 % with respect to the resistance of the previous calibration indicates the structural changes in the lamp that needs attention and possibly the extraordinary verification.

The characteristics of the lamp in the range of radiance temperature from 1050 °C to 1100 °C

| | |
|---|---|
| Time necessary to reach thermal equilibrium: | |
| after switching on to 1100 °C | 50 minutes |
| after changing the current not more than 0.5 A | 10 minutes |
| The voltage instability at the lamp base (at current instability ± 0.5 mA) | < 50 mV |
| The temperature instability of the lamp ribbon (at current instability ± 0.5 mA) | < 40 mK |
| At the copper fixed-point temperature: | |
| slope of the lamp calibration characteristic $dI/dT_\lambda$ | 9.9 мA/1 K |
| base temperature coefficient $dT_\lambda/dt_b$ | 0.08 °C/1 °C |
| temperature drift | < 2 mK |

Temperature inhomogeneity along and across the lamp ribbon:
(within the area ± 0.75 mm relative to the position determined by the index and the center of the cross, placed on the rear wall of the lamp bulb)

| | |
|---|---|
| horizontally: | |
| left | -0.3 K |
| right | + 0.3 K |
| vertically: | |
| upwards | + 0.3 K |
| downwards | + 1.6 K |

## Appendix II

**Information presented by VNIIM according to the Table 2 of the Technical Protocol**

| **COOMET Project 387/UA-a/07, stage 1** | **National Metrology Institute: VNIIM** |
|---|---|
| Parameter item | Value/Comments |
| **Radiator with the fixed-point: Copper** | |
| Furnace: manufacturer / type | BB3500 YY No 300963/a-1 |
| Purity of the metal | 99.992 % |
| Effective emissivity of the cavity | 0.99993 |
| Dimensions of the graphite crucible | See Appendix V |
| Mass of the metal | 160 g |
| Diameter of the aperture of the blackbody cavity | 2.0 mm |
| Installation of the graphite crucible (if necessary) | - |
| Total operating time of the graphite crucible | 32 hours |
| **Standard photoelectric spectral comparator** (No 300963/a-3) | |
| Diameter of the field of view | 0.7 x 0.7 mm (square) |
| Effective wavelength | 656.3 nm |
| Half-width of the spectral responsivity band | 5.0 nm |
| Focus distance of lens | 600 mm |
| Aperture ratio | 1:15 |
| **Conditions of measurements** | |
| Ambient temperature: maximum, minimum and average from all the measurement cycles, °C | $T_{max}$= 22.9 °C; $T_{min}$=21.1 °C; $T_{av}$= 21.8 °C |
| Relative humidity: maximum, minimum and average from all the measurement cycles, % | $\psi_{max}$= 71 %; $\psi_{min}$= 56 %; $\psi_{av}$= 64 % |
| Pressure: maximum, minimum and average from all the measurement cycles, kPa | $P_{max}$ =101.9 кПа; $P_{min}$ = 99.6 кПа; $P_{av}$ = 100.7 кПа |

**Information presented by NSC IM according to the Table 2 of the Technical Protocol**

| **COOMET Project 387/UA-a/07, stage 1** | **National Metrology Institute: NSC IM** |
|---|---|
| Parameter item | Value/Comments |
| **Crucible with the fixed-point: Copper** | |
| Furnace: manufacturer / type | 3-heating coil; self-made, No 001a |
| Purity of the metal | 99.996 % |
| Effective emissivity of the cavity | 0.99981 |
| Dimensions of the graphite crucible | See Appendix A of the Technical Protocol |
| Mass of the metal | 510 g |
| Diameter of the aperture of the blackbody cavity | 3.0 mm |
| Installation of the graphite crucible into VNIIM furnace (if necessary) | An additional diaphragm with aperture diameter of 2.5 mm was installed for alignment |
| Total operating time of the graphite crucible | 34 hours |
| **Standard pyrometer** (Self-made, No 001b) | |
| Diameter of the field of view | - |
| Effective wavelength | - |
| Half-width of the spectral responsivity band | - |
| Focus distance of lens | - |
| Aperture ratio | - |
| **Conditions of measurements** | |
| Ambient temperature: maximum, minimum and average from all the measurement cycles, $^{o}$C | - |
| Relative humidity: maximum, minimum and average from all the measurement cycles, % | - |
| Pressure: maximum, minimum and average from all the measurement cycles, kPa | - |

# Appendix III

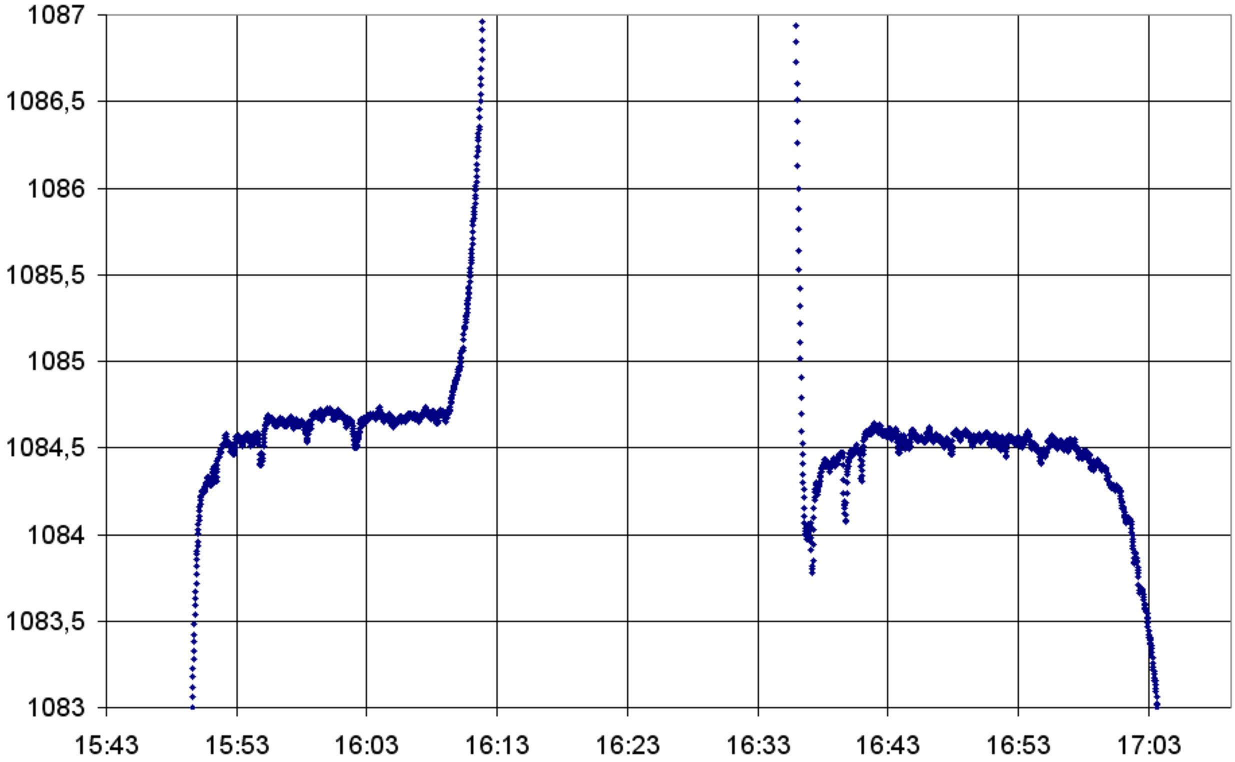
**Information presented by VNIIM according to the Table 3 of the Technical Protocol**

| **COOMET Project 387/UA-a/07, stage 1** | | | | **Fixed-point blackbody of NSC IM** | | |
|---|---|---|---|---|---|---|
| **Graphite crucible with the copper fixed-point of NSC IM** | | | | | | |
| Number of the cycle: melting-freezing | Plateau duration, minutes [1] | Plateau slope, K/min | $U_m^{NSC}$, mV<br>$U_f^{NSC}$, mV | $dU/dT_{Cu}$, мV/1 °C | $\Delta T_{m\text{-}f}^{NSC}$, °C | SPC output signal in the temperature equivalent, °C |
| 1-melting | 3 | 0.040 | 32.318 | 0.3860 | 0.06 | - |
| 1-freezing | 10 | 0.016 | 32.295 | | | 1084.56 |
| 2- melting | 4 | 0.024 | 32.300 | | 0.03 | - |
| 2- freezing | 11 | 0.012 | 32.287 | | | 1084.54 |
| 3- melting | 12 | 0.020 | 32.103 | 0.3856 | 0.10 | - |
| 3- freezing | 24 | 0.004 | 32.064 | | | 1084.61 |
| 4- melting | 17 | 0.009 | 32.077 | | 0.10 | - |
| 4- freezing | 21 | 0.005 | 32.041 | | | 1084.55 |
| 5- melting | 6 | 0.016 | 32.245 | 0.3858 | 0.06 | - |
| 5- freezing | 9 | 0.010 | 32.222 | | | 1084.63 |
| 6- melting | 14 | 0.010 | 32.213 | | 0.07 | - |
| 6- freezing | 13 | 0.015 | 32.187 | | | 1084.54 |

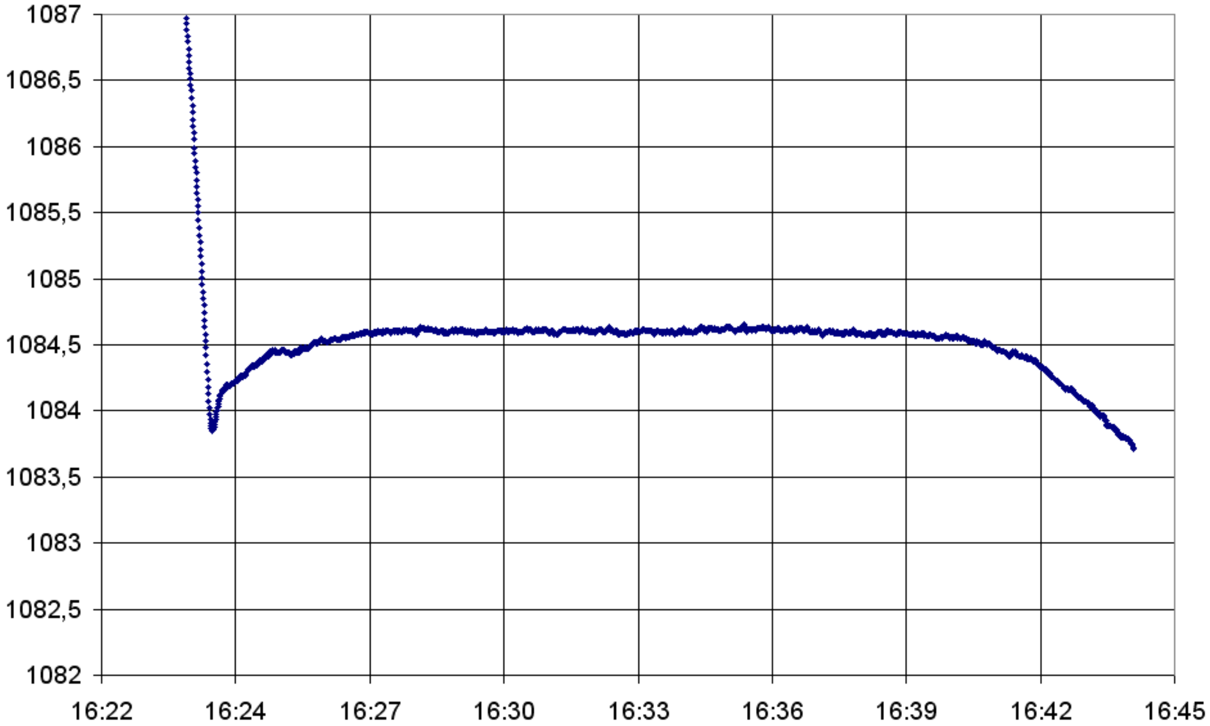
| **COOMET Project 387/UA-a/07, stage 1** | | | | **Fixed-point blackbody of VNIIM** | | |
|---|---|---|---|---|---|---|
| **Graphite crucible with the copper fixed-point of VNIIM** | | | | | | |
| Number of the cycle: melting-freezing | Plateau duration, minutes [1] | Plateau slope, K/min | $U_m^{VNIIM}$, mV<br>$U_f^{VNIIM}$, mV | $dU/dT_{Cu}$, mV/ 1 °C | $\Delta T_{m\text{-}f}^{VNIIM}$, °C | SPC output signal in the temperature equivalent, °C |
| 1-melting | 7 | 0.020 | 32.341 | 0.3860 | 0.070 | - |
| 1-freezing | 12 | 0.006 | 32.313 | | | 1084.63 |
| 2- melting | 18 | 0.007 | 32.319 | | 0.005 | - |
| 2- freezing | 32 | 0.003 | 32.318 | | | 1084.64 |
| 3- melting | 6 | 0.018 | 32.245 | 0.3858 | 0.050 | - |
| 3- freezing | 10 | 0.009 | 32.226 | | | 1084.66 |
| 4- melting | 12 | 0.008 | 32.248 | | 0.070 | - |
| 4- freezing | 14 | 0.004 | 32.223 | | | 1084.65 |
| 5- melting | 23 | 0.010 | 32.250 | | 0.040 | - |
| 5- freezing | 21 | 0.004 | 32.233 | | | 1084.68 |

## Appendix IV

### The shape of the freezing plateaus for the crucibles of the comparison participants




Plateau of melting and freezing of copper for NSC IM crucible

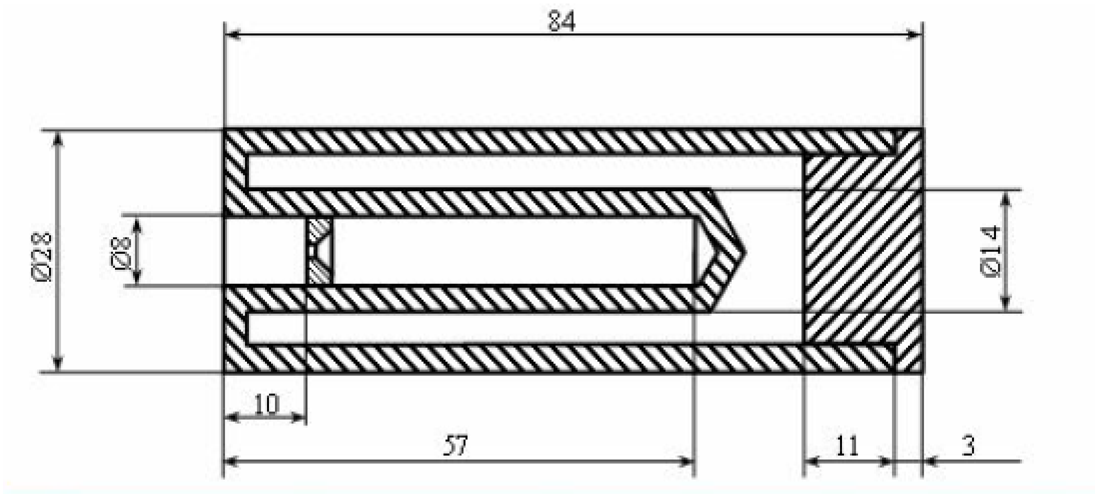



Plateau of freezing of copper for VNIIM crucible

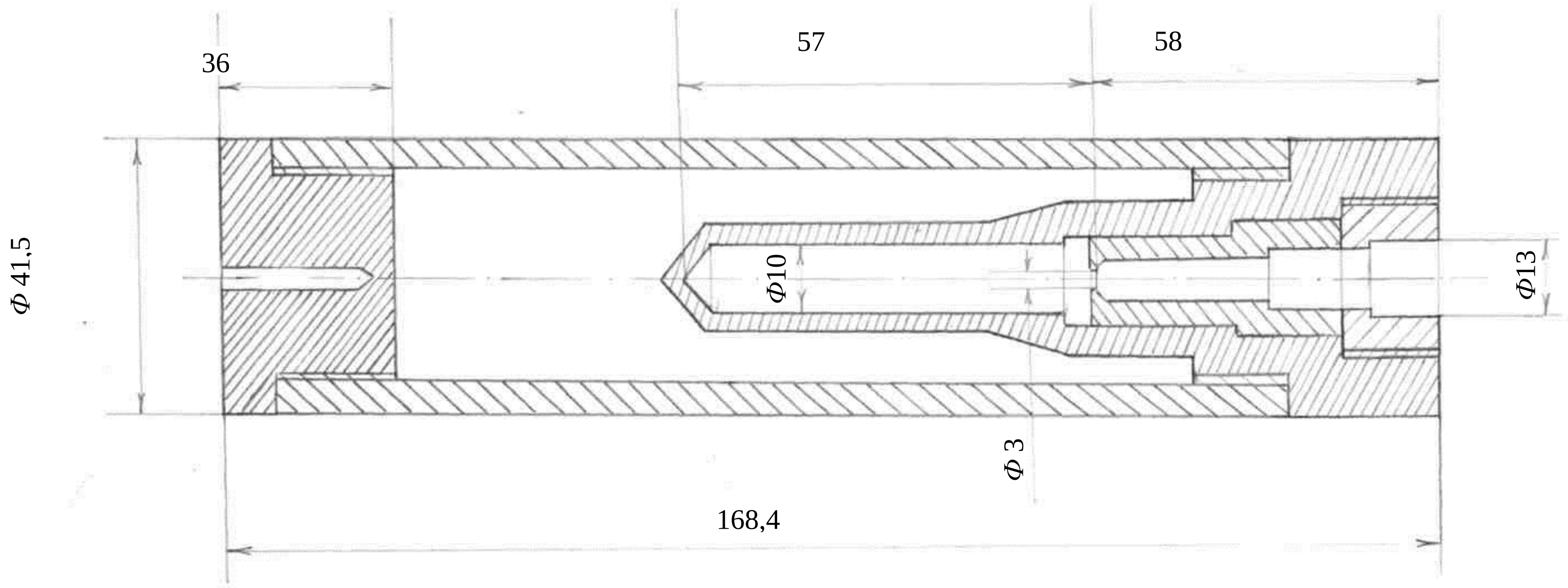
# Appendix V

## The drawing of the graphite crucibles of the comparison participants

### The design of the VNIIM crucible



**The design of the NSC IM crucible**



## Appendix VI

**Information presented by VNIIM according to the Table 5 of the Technical Protocol**

| **COOMET Project 387/UA-a/07, stage 2** | **National Metrology Institute: VNIIM** |
|---|---|
| Parameter item | Value/Comments |
| **Radiator with the fixed-point: Copper** | |
| Type (1, 2 or 3 zones) | 3 zones |
| Purity of the metal | 99.992 % |
| Mass of the metal, g | 205 г |
| Diameter of the aperture of the blackbody cavity, mm | 1.5 mm |
| **Standard photoelectric spectral comparator** | |
| Diameter of the field of view | 0.7 x 0.7 mm |
| Half-width of the spectral responsivity band | 5.0 nm |
| Focus distance of lens | 1800 mm |
| Aperture ratio | 1:15 |
| **Temperature lamp – transfer standard** | |
| Features of the lamp orientation, if they differ from 2.3.1.3 | |
| Instability of voltage at the lamp base during the measurements, the procedure of the instability evaluation | When measuring, thread of base and terminal clamp are connected to the multimeter Keithley2700. The form of the data processing is shown in Table 5.1, where: $\overline{U}$ - the average value of the voltage during the measurements. The evaluation of the voltage instability at the lamp base has been calculated by the formula $S_V = \sqrt{\frac{\sum_{i=1}^{n}(U_i - \overline{U})^2}{n(n-1)}}$. |
| Instability of the lamp base temperature during the measurements, the procedure of instability evaluation | The semiconductor resistance thermometer was mounted on the base with the use of the ring holder. The measurements were performed using the multimeter B7-39. The temperature value has been calculated according to the individual calibration curve of the resistance thermometer. The form of data processing is shown in Table 5.2, where: $\bar{t}$ - the average value of the base temperature. The evaluation of the temperature instability of the lamp base |

| | |
|---|---|
| | has been calculated by the formula: $S_b = \sqrt{\frac{\sum_{i=1}^{n}(t_i - \bar{t})^2}{n(n-1)}}$ . |
| Total burning time of the lamp | 12 hours |
| Operating time of the lamp at fixed-point temperature ($\boldsymbol{I} \geq 0.95\boldsymbol{I}_{Cu}$) | did not exceed 8 hours |
| **Conditions of Measurements** | |
| Ambient temperature: maximum, minimum and average from all the measurement cycles | $T_{min}$ =19.3 °C; $T_{max}$ =22.3 °C; $T_{av}$ = 20.6 °C |
| Relative humidity: maximum, minimum and average from all the measurement cycles | $\psi_{max}$= 56 %; $\psi_{min}$= 44 %; $\psi_{av}$= 48 % |
| Pressure: maximum, minimum and average from all the measurement cycles | $P_{min}$ = 98.8 kPa; $P_{мах}$ = 101.6 kPa; $P_{av}$ = 100.3 kPa |

Table 5.1 (VNIIM) – Form of the data processing for the measurements of the voltage instability at the lamp base

| **Number of the measurements** | **Interval between the measurements, seconds** | **Average $\bar{U}$ , B** | ***S*, B** | ***S*, %** |
|---|---|---|---|---|
| 182 | 1.5 | 0.85337 | 0.000008 | 0.001 |
| 178 | 1.5 | 0.85318 | 0.000006 | 0.001 |

Table 5.2 (VNIIM) – Form of the data processing for the measurements of the instability of the lamp base temperature

| **Number of the measurements** | **Interval between the measurements, seconds** | **Average $\bar{t}$ , °C** | ***S*, °C** |
|---|---|---|---|
| 133 | 1.5 | 22.38 | 0.001 |
| 129 | 1.5 | 22.61 | 0.002 |

**Information presented by NSC IM according to the Table 5 of the Technical Protocol**

| **COOMET Project 387/UA-a/07, stage 2** | **National Metrology Institute: NSC IM** |
|---|---|
| Parameter item | Value/Comments |
| **Radiator with the fixed-point: Copper** | |
| Type (1, 2 or 3 zones) | 3 zones |
| Purity of the metal | 99.996 % |
| Mass of the metal, g | 510 g |
| Diameter of the aperture of the blackbody cavity, mm | 3.0 mm |
| **Standard pyrometer** | |
| Diameter of the field of view | 1.0 mm |
| Half-width of the spectral responsivity band | 14.6 nm |
| Focus distance of lens | 254 мм |
| Aperture ratio | 1:3.2 |
| **Temperature lamp – the transfer standard** | |
| Features of the lamp orientation, if they differ from 2.3.1.3 | - |
| Instability of voltage at the lamp base during the measurements, the procedure of the instability evaluation | When measuring, thread of base and terminal clamp are connected to the voltmeter Keithley2182. The form of the data processing is shown in Table 5.3, where: $\overline{U}$ - the average value of the voltage during measurements. The evaluation of the voltage instability at the lamp base has been calculated by the formula $S_V = \sqrt{\frac{\sum_{i=1}^{n}\left(U_i - \overline{U}\right)^2}{n(n-1)}}$ . |
| Instability of the lamp base temperature during the measurements, the procedure of the instability evaluation | The measuring junction of the thermocouple of L type was mounted on the base with the use of the ring holder. The measurements were performed using the voltmeter Keithley2182. The temperature value has been calculated according to the individual calibration curve of the thermocouple. The form of the data processing is shown in Table 5.4, where: $\bar{t}$ - the average value of the base temperature. The evaluation of the temperature instability of the lamp |

| | |
|---|---|
| | base has been calculated by the formula: $S_b = \sqrt{\frac{\sum_{i=1}^{n}(t_i - \bar{t})^2}{n(n-1)}}$. |
| Total burning time of the lamp | did not exceed 8 hours |
| Operating time of the lamp at the fixed-point temperature ($\boldsymbol{I} \geq 0.95\boldsymbol{I}_{\mathbf{Cu}}$) | did not exceed 5.5 hours |
| **Conditions of Measurements** | |
| Ambient temperature: maximum, minimum and average from all the measurement cycles | $T_{min}$ =18.0 °C; $T_{max}$ =20.7 °C; $T_{av}$ = 18.5 °C |
| Relative humidity: maximum, minimum and average from all the measurement cycles | $\psi_{max}$= 33 %; $\psi_{min}$= 45 %; $\psi_{av}$= 41 % |
| Pressure: maximum, minimum and average from all the measurement cycles | $P_{min}$ = 98.4 kPa; $P_{мax}$ = 101.8 kPa; $P_{av}$ = 100.1 kPa |

Table 5.3 (NSC IM) – Form of the data processing for the measurements of the voltage instability at the lamp base

| **Number of the measurements** | **Interval between the measurements, seconds** | **Average $\bar{U}$, B** | ***S*, B** | ***S*, %** |
|---|---|---|---|---|
| 141 | 5.0 | 0.86532 | 0.000011 | 0.001 |
| 147 | 5.0 | 0.86535 | 0.000007 | 0.001 |

Table 5.4 (NSC IM) – Form of the data processing for the measurements of the instability of the lamp base temperature

| **Number of the measurements** | **Interval between the measurements, seconds** | **Average $\bar{t}$, °C** | ***S*, °C** |
|---|---|---|---|
| 100 | 1.0 | 19.98 | 0.002 |
| 102 | 1.0 | 20.11 | 0.002 |

## Appendix VII

### Information presented by VNIIM according to the Table 6 of the Technical Protocol

**COOMET Project 387/UA-a/07, stage 2** — **National Metrology Institute: VNIIM**

| Number of the freezing plateau | Plateau duration, minutes | $T^{*}$(Cu), K | Lamp SI 10-300 № 5 $I$, A, for $T^{*}(Cu) \equiv T_{rt} = f(I, \lambda_{eff}, t_b)$ | $\Delta T_{\lambda eff}$, K for $\lambda_{re}$=656,3 nm | Base temperature, $t_b$, °C | $\Delta T_b$, **K** for $t_{re}$ = 22.5 °C | Lamp SI 10-300 № 5 $I_{90}$, A, for $T_{90}(Cu) = f(I_{90}, \lambda_{re}, t_{re})$ |
|---|---|---|---|---|---|---|---|
| 1 | 14 | 1357.96 [5] | 9.1911 | 0 | 22.3 | – 0.01 | 9.1891 |
| 2 | 17 | 1357.96 [5] | 9.1932 | 0 | 22.9 | – 0.03 | 9.1910 |
| 3 | 10 | 1357.96 [5] | 9.1916 | 0 | 22.4 | – 0.01 | 9.1896 |
| 4 | 33 | 1357.96 [5] | 9.1920 | 0 | 22.6 | + 0.01 | 9.1902 |
| 5 | 15 | 1357.96 [5] | 9.1906 | 0 | 22.4 | – 0.01 | 9.1896 |
| | | | | | | **Average** | **9.1899** |

[5] Considering the effect of the source size (0,19 ± 0,02) K

**Information presented by NSC IM according to the Table 6 of the Technical Protocol**

| **COOMET Project 387/UA-a/07, stage 2** | | | | | | **National Metrology Institute: NSC IM** | | |
|---|---|---|---|---|---|---|---|---|
| **Number of the freezing plateau** | **Plateau duration, minutes** | $T^*$(Cu), K | **Lamp SI 10-300 № 5** $I$, **A, for** $T^*(Cu) \equiv T_{rt} = f(I, \lambda_{eff}, t_b)$ | $\lambda_{eff}$, **nm** | $\Delta T_{\lambda eff}$, **K for** $\lambda_{re}$=656,3 nm | **Base temperature,** $t_b$, °C | $\Delta T_b$, **K for** $t_{re}$ = 22.5 °C | **Lamp SI 10-300 № 5** $I_{90}$, **A, for** $T_{90}(Cu) = f(I_{90}, \lambda_{re}, t_{re})$ |
| 1 | 8.0 | 1357.77 | 9.1968 | 661.0 | -0.63 | 20.0 | -0.15 | 9.1891 |
| 2 | 14.5 | 1357.77 | 9.1894 | 661.0 | -0.63 | 20.1 | -0.14 | 9.1818 |
| 3 | 17.5 | 1357.77 | 9.1976 | 661.0 | -0.63 | 20.0 | -0.15 | 9.1899 |
| 4 | 21.5 | 1357.77 | 9.1941 | 661.0 | -0.63 | 22.0 | -0.03 | 9.1876 |
| 5 | 17.0 | 1357.77 | 9.1993 | 661.0 | -0.63 | 22.1 | -0.02 | 9.1929 |
| | | | | | | | **Average** | **9.1883** |

# Appendix VIII

## Information presented by VNIIM according to the Table 7 of the Technical Protocol

### The uncertainty budget for calibration of the copper fixed-point

| **COOMET Project 387/UA-a/07, stage 1** | **NMI: VNIIM** |
|---|---|
| Component of the uncertainty | Standard uncertainty, мК |
| Type A | |
| Repeatability of the $\boldsymbol{I}_{90}$ values | 11 |
| Type B | |
| **Fixed-point blackbody radiator** | |
| Impurities | 20 |
| Emissivity | 10 |
| Plateau identification | 30 |
| The temperature difference in the walls of the cavity, heat transfer processes | 10 |
| Positioning (adjustment) | 40 |
| **Standard photoelectric spectral comparator** | |
| Spectral parameters | 10 |
| Size-of-source effect | 20 |
| Drift (instability) | 20 |
| Resolution | 4 |
| The combined uncertainty, mK ($k$ = 1) | 64 |
| The expanded uncertainty, mK ($k$ = 2) | 128 |

**Information presented by NSC IM according to the Table 7 of the Technical Protocol**

**The uncertainty budget for calibration of the copper fixed-point**

| **COOMET Project 387/UA-a/07, stage 1 NMI: NSC IM** | |
|---|---|
| Component of the uncertainty | Standard uncertainty, мК |
| Type A | |
| Repeatability of the $\boldsymbol{I_{90}}$ values | 72 |
| Type B | |
| **Fixed-point blackbody radiator** | |
| Impurities | 30 |
| Emissivity | 20 |
| Plateau identification | 5 |
| The temperature difference in the walls of the cavity, heat transfer processes | 10 |
| Positioning (adjustment) | 50 |
| **Standard photoelectric spectral comparator** | |
| Spectral parameters | 55 |
| Size-of-source effect | 60 |
| Drift (instability) | 20 |
| Resolution | 5 |
| The combined uncertainty, mK ($k$ = 1) | 127 |
| The expanded uncertainty, mK ($k$ = 2) | 254 |

**Information presented by VNIIM according to the Table 7 of the Technical Protocol**

**The uncertainty budget for calibration of the lamp – the transfer standard at the copper fixed-point**

| **COOMET Project 387/UA-a/07, stage 2** | **NMI: VNIIM** |
|---|---|
| Component of the uncertainty | Standard uncertainty, мК |
| Type A | |
| Repeatability of the $\boldsymbol{I_{90}}$ values | 33 |
| Type B | |
| **Fixed-point blackbody radiator** | |
| Impurities | 20 |
| Emissivity | 10 |
| Plateau identification | 30 |
| The temperature difference in the walls of the cavity, heat transfer processes | 10 |
| **Standard photoelectric spectral comparator** | |
| Spectral parameters | 20 |
| Size-of-source effect | 20 |
| Drift (instability) | 20 |
| Resolution | 4 |
| **Temperature lamp** | |
| Current measurement | 30 |
| Base temperature | 40 |
| Positioning (adjustment) | 30 |
| **Converting results to the reference conditions** | |
| Interpolation and integration | 20 |
| Converting to the reference base temperature | 10 |
| The combined uncertainty, mK ($k$ = 1) | 88 |
| The expanded uncertainty, mK ($k$ = 2) | 176 |

**Information presented by NSC IM according to the Table 7 of the Technical Protocol**

**The uncertainty budget for calibration of the lamp – the transfer standard at the copper fixed-point**

| **COOMET Project 387/UA-a/07, stage 2** | **NMI: NSC IM** |
|---|---|
| **Component of the uncertainty** | **Standard uncertainty, мК** |
| Type A | |
| Repeatability of the $I_{90}$ values | 185 |
| Type B | |
| **Fixed-point blackbody radiator** | |
| Impurities | 30 |
| Emissivity | 20 |
| Plateau identification | 5 |
| The temperature difference in the walls of the cavity, heat transfer processes | 10 |
| **Standard pyrometer** | |
| Spectral parameters | 55 |
| Size-of-source effect | 60 |
| Drift (instability) | 20 |
| Resolution | 5 |
| **Temperature lamp** | |
| Current measurement | 75 |
| Base temperature | 16 |
| Positioning (adjustment) | 30 |
| **Converting results to the reference conditions** | |
| Interpolation and integration | 25 |
| Converting to the reference base temperature | 15 |
| Converting to the reference effective wavelength | 70 |
| The combined uncertainty, mK ($k$ = 1) | 235 |
| The expanded uncertainty, mK ($k$ = 2) | 470 |