FINAL REPORT

Supplementary comparison COOMET.T-S1

Comparison of type S thermocouples at the freezing points of zinc, aluminium and copper 2014 - 2015

(COOMET project No. 487/RU/10)

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1. Introduction

Type S thermocouples are widely used for temperature measurement in a range 300-1100°C in many areas of science and industry. Besides type S thermocouples are used as the secondary standards by National Metrology Institutes (NMI) to disseminate the ITS-90 temperature scale to measuring instruments in their countries. Calibration of type S thermocouples by NMIs is performed using three fixed reference points of ITS-90: zinc point, aluminum point and copper point.
Regional comparison of type S thermocouples was initiated by COOMET TC1.1–10 (the technical committee of COOMET "Thermometry and thermal physics").
The purpose of the comparison is to evaluate the equivalence of the type S thermocouples calibration in fixed points by NMIs.
Three NMI take part in COOMET regional comparison: D.I. Mendeleev Institute for Metrology «VNIIM» (Russian Federation), National Scientific Centre «Institute of Metrology» (NSC IM, Ukraine), Republic State Enterprise «Kazakhstan Institute of Metrology» (KazInMetr, Republic of Kazakhstan).
VNIIM (Russia) was chosen as the coordinator-pilot of the regional comparison.
The Technical Protocol of the comparison, prepared by VNIIM, has been distributed between the participants after its consideration in Working Group 7 of Consultative committee on thermometry in May, 2014.

2. Summary of comparison

A star type comparison was used. The participants: KazInMetr and NSC IM constructed the type S thermocouples and calibrated them in three fixed points: zinc, aluminum and copper points, using methods of ITS-90 fixed point realizations. The inhomogeneity of thermocouples was measured in oil bath at temperature 200 °C.
Then the thermocouples have been sent to VNIIM together with the results of the calibration in three fixed points, with the values of the inhomogeneity and the uncertainty evaluations of the results. VNIIM calibrated the thermocouples of the participants in the same fixed points using methods of ITS-90 fixed point realizations and VNIIM's instruments and investigated their inhomogeneity.
After all measurements are finished, NMI thermocouples are returned to the participating laboratories.
Participating laboratories repeated the calibration of thermocouples in zinc, aluminum and copper points to determine the stability of its results. Results of thermocouples recalibration have been transferred to VNIIM.

The coordinator evaluated the degrees of the equivalence of calibration of the type S thermocouples in fixed points by NMIs and their uncertainties.

3. Measurement schedule

May – 15 June 2014:	VNIIM, NSC IM, KazInMetr – design and calibration the type S thermocouples for the comparison,
June 2014:	Sending the thermocouples by NSC IM, KazInMetr to pilot -VNIIM,
July 2014:	Sending the thermocouple calibration results, the estimation of the uncertainty by NSC IM, KazInMetr to pilot
July - August 2014:	Calibration the thermocouples by pilot,
September 2014:	Sending the thermocouples by pilot to NSC IM, KazInMetr
September - November 2014:	Repeated calibration the thermocouples by NSC IM, KazInMetr Sending the repeated calibration results by NSC IM, KazInMetr to pilot
November 2014:	Preparation of Draft A by pilot Draft A circulated to participants

4. Preparation of the thermocouples

Thermocouples submitted for comparison:
TC 1 - KazInMetr,
TC 085 - NSC IM,
Minimum length of the wires of the thermocouples, submitted by NMI for comparisons, was not less 1200 mm. The length of the ceramic tubes of the thermocouples was not less 460 mm at maximum diameter 6 mm. The interface between the ceramic tube and protection covering of exposed ends of the thermocouple was being fixated well.
The sequence of actions and annealing of the thermocouples by NMI was the next.
After the preparation the thermocouples were annealing at a temperature (1100±20) °C for 1 hour in the vertical annealing furnace with immersion depth 50 cm from the furnace top. After removing thermocouples from the furnace they cooled at room temperature.

5. **Measurement results by the Participants**

The realizations of all fixed points were carried out according to the methods of Supplementary Information [1] and the documents of CCT WG1 [2].
Duration of the freezing plateaus was not less than 6 hours. The changes of temperature during plateau did not exceed 1 мК.
Control of the freezing plateaus realizations was carried out using the SPRT or a standard thermocouple. The freezing plateaus are shown in Appendix D.
The thermocouples were inserted into the fixed point cells after start of the freezing plateau, indicated by the control thermometer for zinc and aluminum points and by the high temperature platinum resistance thermometer or the standard type S thermocouple for copper point.
The calibrations of the thermocouples were repeated at least 3 times in each reference fixed point. NMIs have presented to pilot the measurement results on 3 plateaus and the information necessary for calculation the thermocouple emf E_x in the fixed points according to the equation (1):

$$E_x = E_i + \delta V + \delta t_0 \cdot C_0 + \delta V_c + \delta t \cdot C_t \qquad (1),$$

where:
E_i– average emf for three plateaus (μV),
δV– correction due to the voltmeter calibration (μV),
δt_0– correction to the cold junction temperature of thecalibrated thermocouple (°C),
δV_c– correction due to the extension cables using (μV),
δt – correction to value of the reference fixed point temperature based on the results of the fixed point cell calibration of participating laboratory (°C) or on the certificate data of the cell,
C_0 – sensitivity coefficient for the thermocouple at 0 °C (μV/°C),
C_t– sensitivity coefficient for the thermocouple at the fixed
point temperature (μV/°C).

5.1. Measurement results of TC 085 thermocouple by NSC IM

Measured emf values E_i and standard uncertainty $u(E_i)$ of TC 085 at freezing points before sending to VNIIM are shown in table 1.
Table 1. Values E_i and $u(E_i)$ of TC 085

Measurement No	Emf , μV		
	Zn	Al	Cu
1st plateau	3442.83	5856.32	10577.04
2nd plateau	3443.16	5856.45	10576.53
3rd plateau	3443.4	5857.30	10577.15
E_i	3443.13	5856.69	10576.90
$u(E_i)$	0.15	0.25	0.33

Supplementary information of NSC IM:

Temperature of the cold junction of thermocouple TC 085 was 0,05 ºC , δV=0, δV_c=0, δt=0.

The values E_x of TC 085 in according to equation (1) are shown in table 2 .

Table 2. Values E_x of TC 085 at the fixed points

	Zn	Al	Cu
	μV	μV	μV
E_i	3443.13	5856.69	10576.90
δV	-	-	-
$\Delta t_0 \cdot C_0$	0.05·5.4	0.05·5.4	0.05·5.4
δV_c	-	-	-
$\delta t \cdot C_t$	0	0	0
E_x	3443.40	5856.96	10577.17

5.2 Measurement results of TC 1 thermocouple by KazInMetr

Measured emf values E_i and standard uncertainty $u(E_i)$ of TC 1 at freezing points before sending to VNIIM are shown in table 3.

Table 3. Values E_i of TC 1 and $u(E_i)$

Measurement No	**Emf , μV**		
	Zn	Al	Cu
1st plateau	3443.55	5862.68	10588.20
2nd plateau	3443.78	5860.99	10589.15
3rd plateau	3444.25	5861.77	10589.91
E_i	**3443.86**	**5861.81**	**10589.09**
$u(E_i)$	**0.20**	**0.49**	**0.51**

Supplementary information of KazInMetr:

Temperature of the cold junction of the thermocouple TC 1 was 0,05 ºC, δV=0, δV_c=0, δt(Cu) =0.
Corrections on fixed point temperature were accepted according to the certificates on cells:
δt(Zn)= - 0,0003 ºC, δt(Al)= - 0,0008 ºC.

The values E_x of TC 1 in according to equation (1) are shown in table 4.

Table 4. Values E_x of TC 1 at the fixed points

	Zn	Al	Cu
	μV	μV	μV
E_i	3443.86	5861.81	10589.09
δV	-	-	-
$\Delta t_0 \cdot C_0$	0.05·5.4	0.05·5.4	0.05·5.4
δV_c	-	-	-
$\delta t \cdot C_t$	$3.10^{-4}.9.6$	$8.10^{-4}.10.4$	0
E_x	**3444.13**	**5862.09**	**10589.36**

5.3. Measurement results of thermocouples TC 085 and TC 1 by VNIIM

5.3.1. Measurement results of TC 085

Measured emf values E_i and standard uncertainty $u(E_i)$ of TC 085 at freezing points are shown in table 5.

Table 5. Values E_i and $u(E_i)$ of TC 085

Measurement No	**Emf, μV**		
	Zn	Al	Cu
1st plateau	3444.21	5857.74	10578.63
2nd plateau	3443.73	5857.32	10579.46
3rd plateau	3443.93	5857.95	10577.91
E_i	**3443.96**	**5857.67**	**10578.67**
$u(E_i)$	**0.14**	**0.18**	**0.45**

Measured emf values E_i of TC 1 and standard uncertainty $u(E_i)$ at freezing points are shown in table 6.

Table 6. Values E_x and $u(E_x)$ of TC 1

Measurement No	**Emf, μV**		
	Zn	Al	Cu
1st plateau	3443.44	5861.52	10588.14
2nd plateau	3443.73	5861.01	10587.70
3rd plateau	3444.01	5861.22	10587.17
Ei	**3443.73**	**5861.25**	**10587.67**
$u(E_i)$	**0.16**	**0.26**	**0.28**

Supplementary information of VNIIM:

Temperature of the cold junction of thermocouples TC 085 and TC 1 was 0 ºC,
$\delta V=0$, $\delta V_c=0$, $\delta t(Zn)=0$, $\delta t(Al)=0$, $\delta t(Cu)=0$.

Therefore $E_i=E_x$.

5.4. Inhomogeneity and its contribution to the uncertainty of the comparison

Inhomogeneity of the thermocouple leads to the change of measurement result and the additional uncertainty owing to the temperature gradient changing along its length.
According to the Protocol, the participants were asked to submit the gradients of temperature in furnaces which have been used for calibration for an estimation of inhomogeneity influence on results of calibration. The participants have presented the temperature changes on height of furnaces.
The temperature profiles of furnaces are shown on fig.1-3.

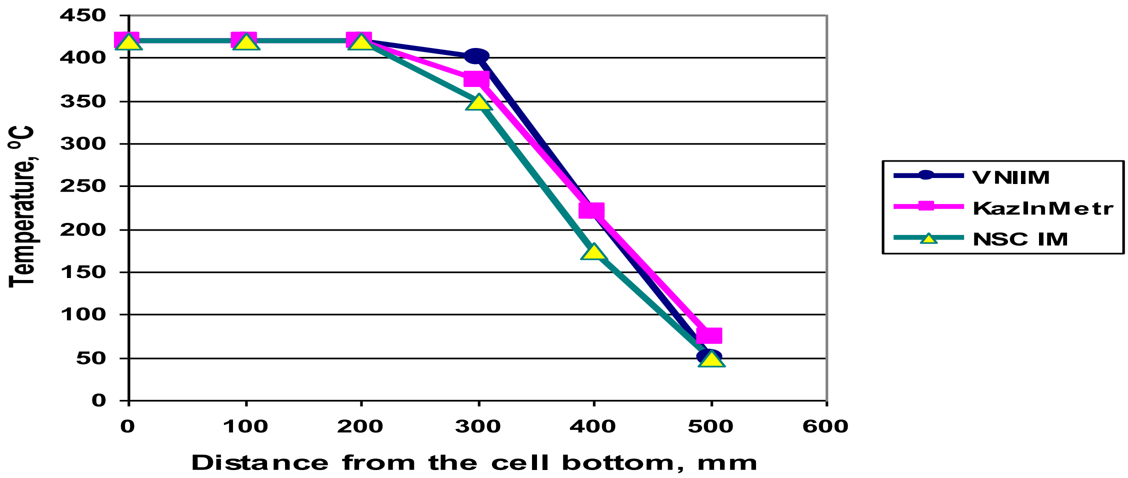

Fig.1. Temperature profiles of the Zn fixed point furnaces.

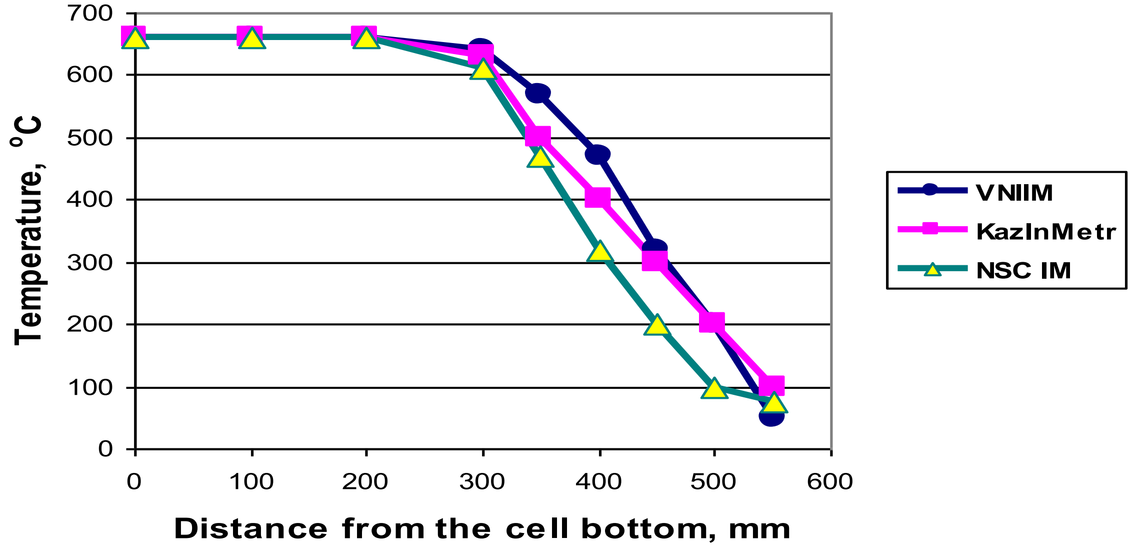

Fig.2. Temperature profiles of the Al fixed point furnaces

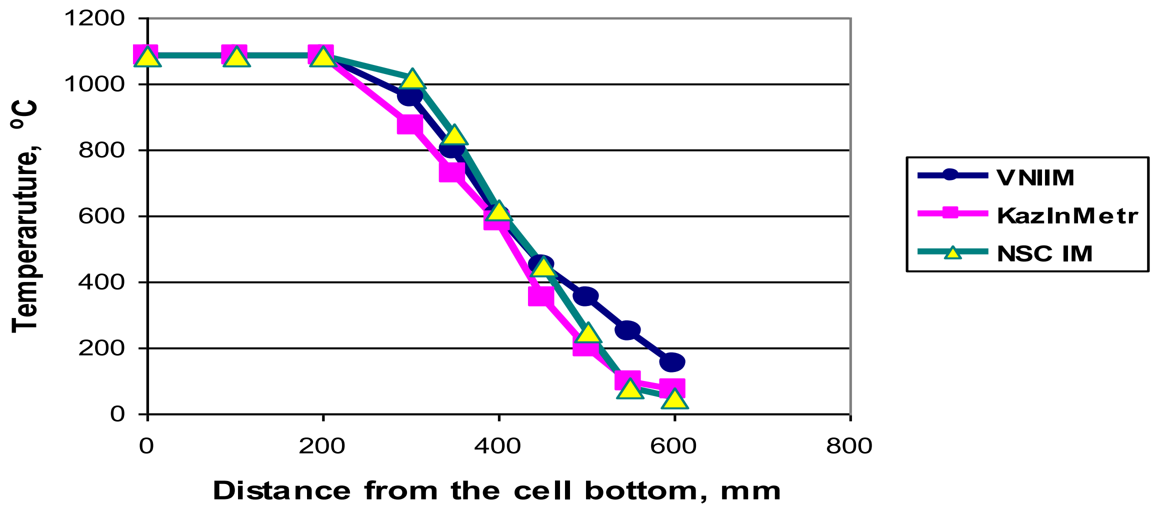

Fig.3. Temperature profiles of the Cu fixed point furnaces.

Apparently on fig. 1-3, the main influence on the measurement result due to the inhomogeneity of the thermocouple is likely to occur in the section of the thermocouple 300 - 500 mm from junction. It is necessary to notice that temperature gradients in furnaces of participants differ slightly since the height of the cells and the size of the heaters are almost identical.

According to the Protocol the participants investigated and estimated the inhomogeneity of thermocouples at their immersing in the thermostat with a homogeneous temperature field at 200 ºC.

The inhomogeneity estimation U_{inh} (in %) has been performed according to [3].

$$U_{inh}=\pm 0,5(E_{max} - E_{min})/(E_{ref}) \cdot 100 \quad (2)$$

The standard uncertainty due to inhomogeneity $u_{inh} = U_{inh}/\sqrt{3}$.

The measurement results of TC 085 inhomogeneity by NSC IM are shown on fig.4.

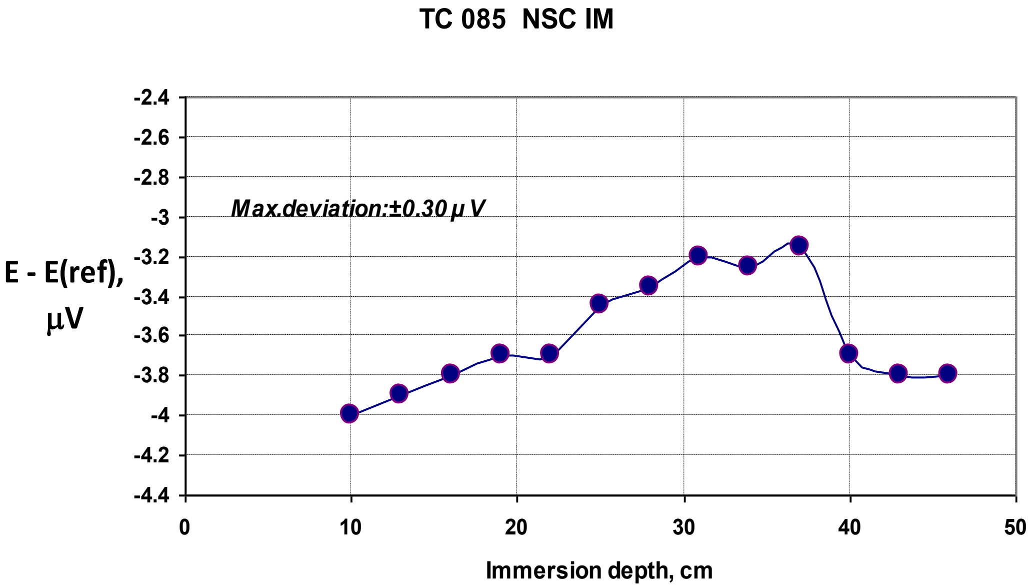

Fig.4. Emf deviations at immersion of hot junction in the oil thermostat

The inhomogeneity of TC 085 was calculated from maximum variations of its emf at immersion from 16 cm to 46 cm of hot junction in the oil thermostat.

u_{inh}= 0.012 %.

The measurement results of TC 1 inhomogeneity by KazInMetr are shown on fig.5.

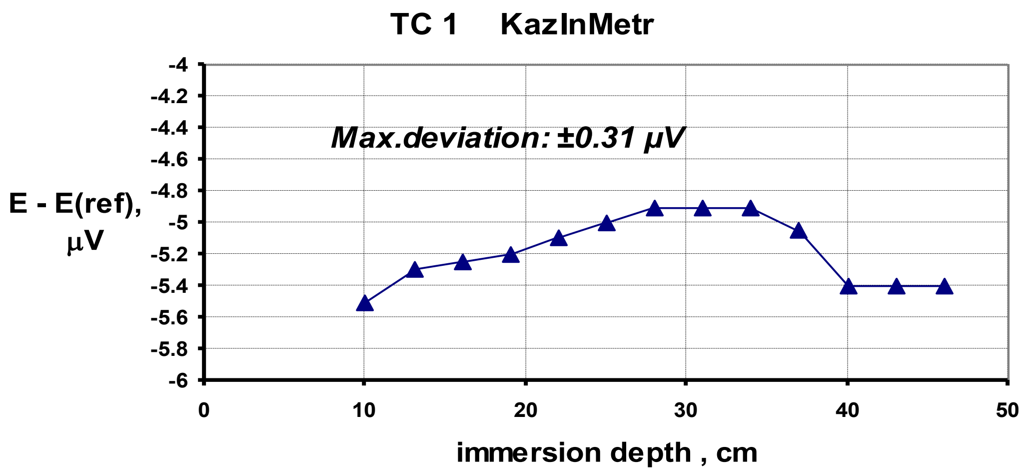

Fig.5. Emf deviations at immersion of hot junction in the oil thermostat

The inhomogeneity of TC 1 was calculated from maximum variations of its emf at immersion from 19 cm to 46 cm.

u_{inh}= 0.0125 %

The inhomogeneity of the transferred thermocouple TC 085 and TC 1 has been investigated in VNIIM at 200 °C in the oil thermostat. The results of the investigation are shown on fig.6 and 7.
The values of the standard uncertainty due to inhomogeneity u_{inh} were calculated to be 0.011 % for TC 085 and 0.013 % for TC 1.

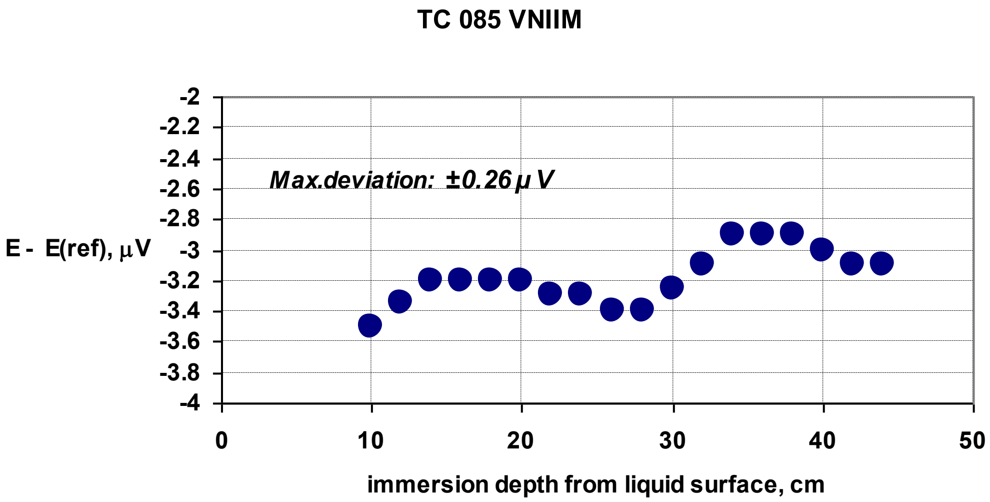

Fig.6. Emf deviations at immersion of hot junction in the oil thermostat, obtained in VNIIM

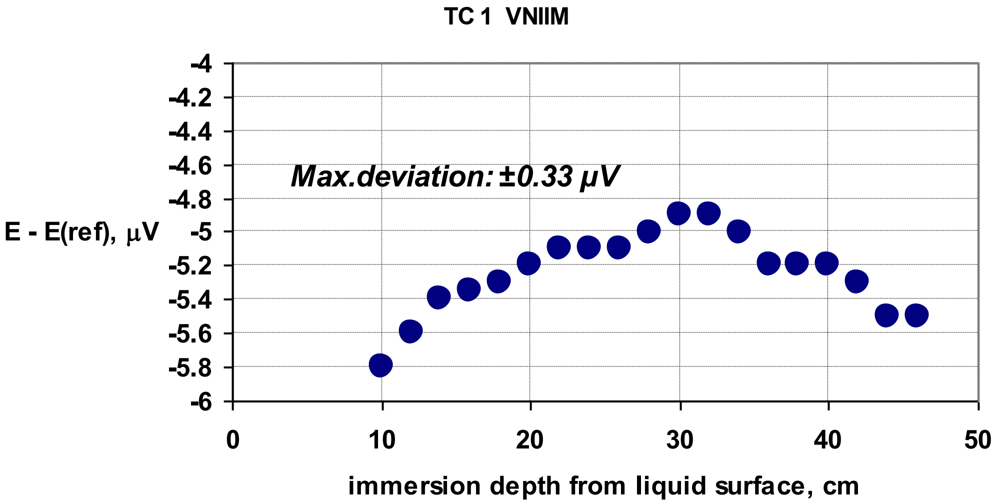

Fig.7. Emf deviations at immersion of hot junction in the oil thermostat, obtained in VNIIM

The most exact estimations of the influence of thermocouple inhomogeneity on the calibration results could be received by combination of results of inhomogeneity research of thermocouples and corresponding temperature gradients in furnaces. But because of insufficient reliability of the definition of the inhomogeneity in the thermostat and complexity of its combination with the temperature gradients it has been decided to consider the influence of the inhomogeneity by the introduction of the uncertainty component u_{inh} in the budget.
The uncertainties due to inhomogeneity of the thermocouples u(inh) in fixed points have been calculated taking into account a linear dependence of relative estimation of inhomogeneity from the emf [3].

6. Uncertainty analysis of measurement results

The uncertainty analysis of the emf E_x was presented by evaluation of 6 uncertainty components in each fixed point:
$u(E_x)$ - standard uncertainty, type A,
$u(\delta t)$ - uncertainty of the fixed point temperature according to the primary standard or cell certificate,
$u(\delta t_0)$ - uncertainty of the temperature of reference junction ,
$u(\delta V)$ - uncertainty due to voltmeter calibration,
$u(\delta Vc)$ - uncertainty of correction due to the using extension cables,
u (inh) - uncertainty due to inhomogeneity of the thermocouple,
and by calculating $u(E_x)$ - the combined standard uncertainty,
$U(E_x)$ - expanded uncertainty.

Uncertainty budgets of VNIIM, NSC IM and KazInMetr for TC 1 and NC 085 in fixed points are given in Appendix C.

7 Comparison results

7.1 Comparison results of NSC IM and VNIIM for TC 085

The degree of equivalence $D_{TC\ 085}$ and its uncertainty u_D :

$$D_{TC\ 085} = E_x(\text{NSC IM}) – E_x(\text{VNIIM}) = (E_{\text{NSC IM}} - E_{ref}) – (E_{\text{VNIIM}} - E_{ref}),$$

$$u_D^2 = u_{\text{NSC IM}}(E_{x\text{NSC IM}})^2 + u_{\text{VNIIM}}(E_{x\text{VNIIM}})^2 + (\text{drift}/2\sqrt{3})^2$$

$D_{TC\ 085}$ and u_D are shown in table 8.

The differences between initial and final calibrations of TC 085 submitted by NSC IM are shown in table 7.

Table 7. Differences between initial and final calibrations of TC 085 by NSC IM

Fixed point	initial value, μV $E_{NSC\ IM}$	final value, μV $E_{NSC\ IM}$	drift μV,
Zn	3443.40	3443.74	0.34
Al	5856.96	5857.41	0.45
Cu	10577.17	10578.05	0.88

Table 8. Values of Degree of equivalence $D_{NSC\ IM}$ and u_D (k=1) of TC 085

NMI	Zn, μV		Al, μV		Cu, μV	
	E_x(NMI) - E_{ref}	u(E_x)	E_x(NMI) - E_{ref}	u(E_x)	E_x(NMI) - E_{ref}	u(E_x)
NSC IM	-3.49	0.46	-3.17	0.76	2,37	1,32
VNIIM	-2.93	0.41	-2.45	0.67	3,87	1,25
$D_{TC\ 085}$	- 0.56		- 0.71		- 1.5	
u_D	0.62		1.02		1.83	

Comparison results of TC 085 are shown on Fig.8.

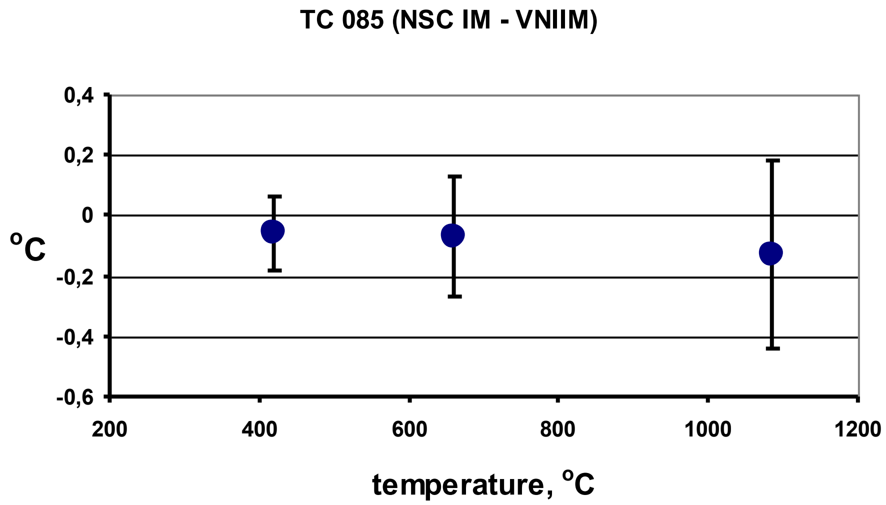

Fig.8. Degree of equivalence of TC 085 calibration. The error bars denote the combined uncertainty U_D (k=2).

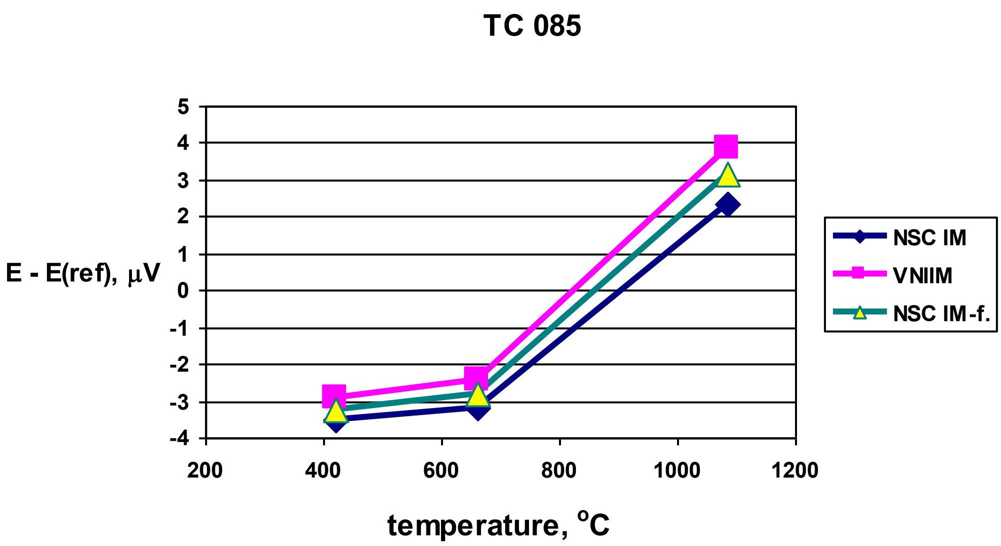

Fig.9. Deviations of E_x of TC 085 from reference function at fixed points

7.2 Comparison results of KazInMetr and VNIIM for TC 1

The degree of equivalence $D_{TC\ 1}$ and its uncertainty u_D:

$D_{TC\ 1} = E_x(\text{KazInMetr}) - E_x(\text{VNIIM}) = (E_{KazInMetr} - E_{ref}) - (E_{VNIIM} - E_{ref})$,

$u_D^2 = u_{KazInMetr}\ (E_{\ xKazInMetr})^2 + u_{VNIIM}\ (E_{\ xVNIIM})^2 + (drift/2\sqrt{3})^2$

$D_{TC\ 1}$ and u_D are shown in table 10.

The differences between initial and final calibrations of TC 1 submitted by KazInMetr are shown in table 9.

Table 9. Differences between initial and final calibrations of TC 1 by KazInMetr

Fixed point	initial value, **µV** $E_{NSC\ IM}$	final value, **µV** $E_{NSC\ IM}$	drift **µV**,
Zn	3444.10	3443.94	0.16
Al	5862.09	5862.57	0.46
Cu	10589.36	10588.95	0.41

Table 10. Values of the degree of equivalence $D_{TC\ 1}$ and u_D (k=1) for TC 1

NMI	Zn, μV		Al, μV		Cu, μV	
	E_x(NMI) - E_{ref}	u(E_x)	E_x(NMI) - E_{ref}	u(E_x)	E_x(NMI) - E_{ref}	u(E_x)
KazInMetr	-2,76	0,52	1,96	0,91	14,56	1,45
VNIIM	-3,16	0,48	1,40	0,80	12,87	1,40
$D_{TC\ 1}$	0,40		0,84		1,7	
u_D	0,71		1,22		2,02	

Comparison results of TC 1 are shown on Fig,10

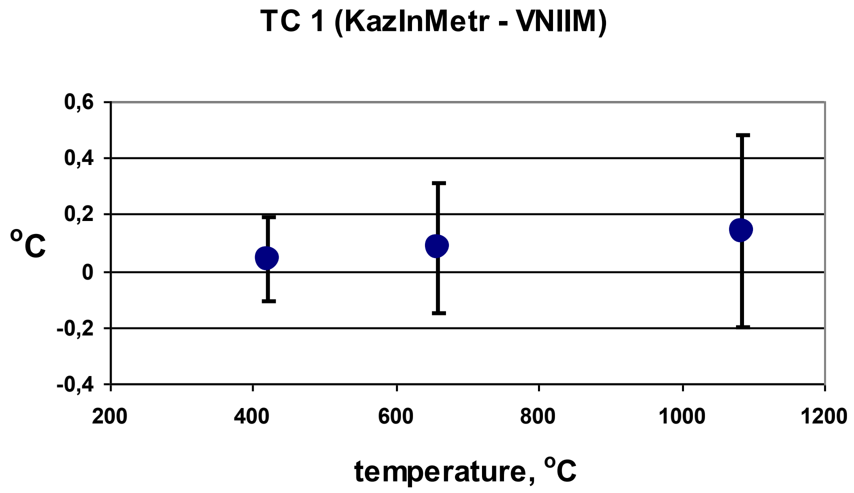

Fig.10. Degree of equivalence of TC 1 calibration. The error bar denotes the combined uncertainty U_D (k=2).

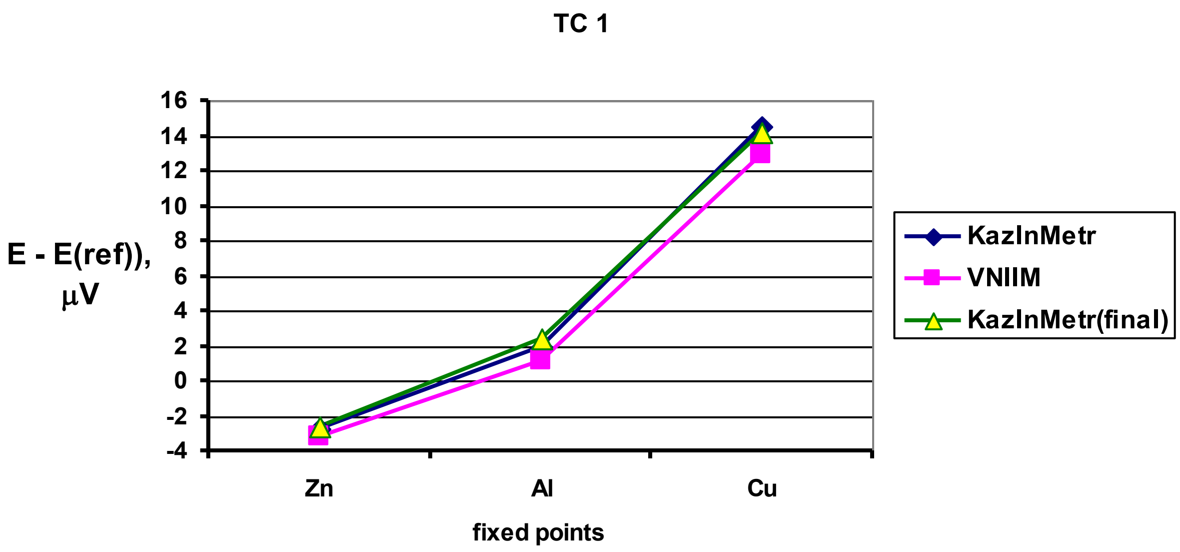

Fig.11. Deviations of emfs of TC 1 from reference function at fixed points

8. Conclusion

The purpose of the comparison was to evaluate the equivalence of the type S thermocouples calibration in fixed points by NMIs to confirm corresponding lines of international website for NMI's Calibration and Measurement Capabilities (CMC).

The values of the degree of equivalence D and its uncertainty u_D (k=2) of the calibration results for thermocouples TC 1and TC 085 are shown in table 11. Expanded uncertainties U_{NMI}, declared by the participants, are shown in table 12.

Table 11. Values of the degree of equivalence D and its uncertainty U_D (k=2) of the thermocouples TC 1 and TC 085 calibrations, expressed in ºC

Fixed point	KazInMetr - VNIIM		NSC IM - VNIIM	
	$D_{TC\ 1}$	**U_D**	**$D_{TC\ 085}$**	**U_D**
	ºC	ºC	ºC	ºC
Zn	0.04	0.15	- 0.06	0.12
Al	0.08	0.23	- 0.07	0.20
Cu	0.14	0.34	- 0.13	0.31

Table 12. Expanded uncertainties U_{NMI} of the calibration results, expressed in ºC

Fixed point	VNIIM	KazInMetr	NSC IM
	ºC	ºC	ºC
Zn	0.09	0.11	0.09
Al	0.14	0.18	0.15
Cu	0.23	0.25	0.22

9. References

1. H. Preston-Thomas, P. Blombergen, T.J. Quinn, *Supplementary Information for International Temperature Scale of 1990*, (BIPM, 1990)

2. B.W. Mangum, P. Blombergen, M. Chattle, B. Felmuth, P. Marcarino, A.I. Pokhodun, *Working Documents of the 20th Meeting of the Consultative Committee for Thermometry*, BIPM Document CCT/2000-13

3. F.Jahan, M.J.Ballico "A study of the temperature dependence of inhomogeneity in platinum based thermocouples" Temperature: Its Measurement and Control in science and industry, vol.7, 469-474, 2002.

Appendix A

TECHNICAL PROTOCOL

Supplementary comparison COOMET.T-S1

Comparison of type S thermocouples at the freezing points of zinc, aluminium and copper 2014 - 2015

COOMET Project 487/RU/10

March 21, 2014

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Introduction

Type S thermocouples are widely used by National Metrology Institutes (NMI) to transfer the ITS-90 to temperature measuring instruments at a range of temperatures from 300 to 1100 °C. Calibration of type S thermocouples in NMIs is performed using three fixed reference points of ITS-90: zinc point, aluminum point and copper point. Regional comparison of type S thermocouples was initiated by COOMET TC1.1–10 (the technical committee of COOMET "Thermometry and thermal physics"). The purpose of the comparison is to evaluate the equivalence of the type S thermocouples calibration in fixed points by NMIs to confirm corresponding lines of international website for NMI's Calibration and Measurement Capabilities (CMC).

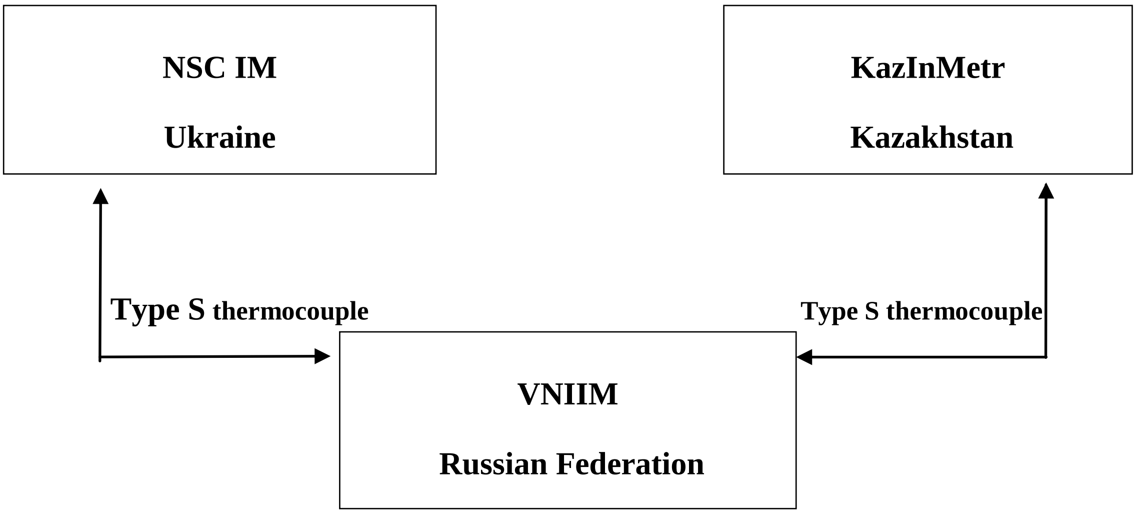
Three NMI take part in COOMET regional comparison: D.I. Mendeleev Institute for Metrology «VNIIM» (Russian Federation), National Scientific Centre «Institute of Metrology» (Ukraine), Republic State Enterprise «Kazakhstan Institute of Metrology» (Republic of Kazakhstan).

VNIIM (Russia) was chosen as the coordinator-pilot of the regional comparison since its uncertainty evaluations of the type S thermocouples in zinc, aluminum and copper fixed reference points were recognized on interregional scrutiny and presented at the international CMC site VNIIM (k=2): Zn - 0,4 K, Al - 0,4 K, Cu - 0,4K.

Comparison participants must operate in accordance with the instructions below. Each laboratory should use its own methods of ITS-90 fixed point realizations and calibration procedure for thermocouples.

1. Scheme of comparison

The type S thermocouples, calibrated in zinc, aluminum and copper fixed reference points of the participating laboratories, are used as a transfer standard in the comparison.



Comparison include four stages

Stage 1

Each participating laboratory prepares the type S thermocouple. The laboratory calibrates the thermocouple in zinc, aluminum and copper points and measures of its inhomogeneity in oil bath at temperature 250 °C, then transfers the thermocouple to VNIIM together with the results of the calibration in three fixed points and the uncertainty evaluations of the results.

Stage 2

VNIIM calibrates thermocouples of the participating laboratories in zinc, aluminum and copper points using VNIIM’s instruments for fixed point realization.

After all measurements are finished, transfer thermocouples are returned to the participating laboratories.

Stage 3

Participating laboratories repeat the measurement the inhomogeneity of the thermocouples and their calibration in zinc, aluminum and copper points to determine the stability of its results. Results of thermocouples recalibration are transferred to VNIIM.

Stage 4

VNIIM prepares the comparison results: estimation of the equivalence of the transferred thermocouples calibration in all of the fixed points with uncertainty evaluation.

2. Participating laboratories

Name of Laboratory	Contact person	Adress
VNIIM - Russian Federation	Ivanova A.G. a.g.ivanova@vniim.ru	Moskovsky pr., 19, 190005, St. Petersburg
NSC IM - Ukraine	Ivanova E.P. ikp@metrology.kharkov.ua	Mironosickaya str., 42, 61002, Kharkov
KazInMetr - Republic of Kazakhstan	Dysebaeva K.K. kuralay_12@mail.ru	Left bank, 35thStreet, building11, 010000, Astana,

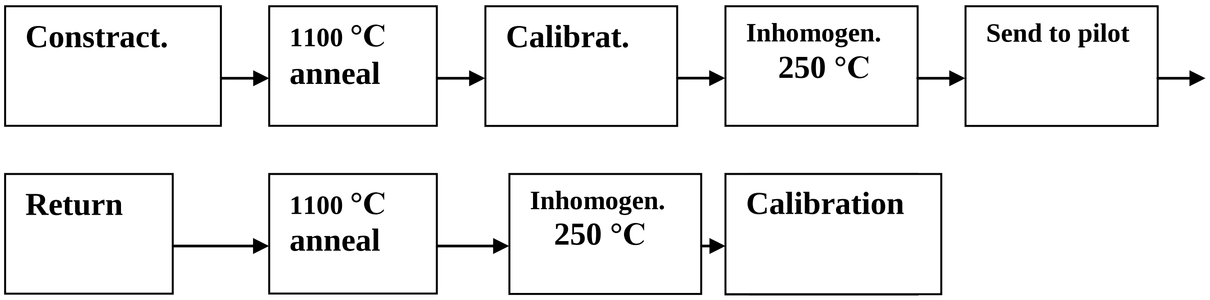
3. Comparisons schedule

May – 15 June 2014: VNIIM, NSC IM, KazInMetr – design and calibration the type S thermocouples for the comparison,
June 2014: Sending the thermocouples by NSC IM, KazInMetr to pilot -VNIIM,
July 2014: Sending the thermocouple calibration results and the estimation of the uncertainty by NSC IM, KazInMetr to pilot
July - August 2014: Calibration the thermocouples by pilot,
September 2014: Sending the thermocouples by pilot to NSC IM, KazInMetr
15 September - November 2014: Repeated calibration the thermocouples by NSC IM, KazInMetr
December 2014: Sending the repeated calibration results by NSC IM, KazInMetr to pilot
December - January 2015: Draft report
January 2015: Draft A circulated to participants

4. Preparation of the thermocouples

Thermocouples, submitted by NMI for comparisons, must have minimum length of the thermocouple wires 1200 mm. The length of the ceramic tube of the thermocouple must be at least 460 mm, with maximum diameter 6 mm. The

interface between the ceramic tube and protection covering of exposed ends of the thermocouple must be fixated well.
Ceramic tube should have a clean surface, thermocouple wires must move freely in the ceramic tube.
Thermocouples, submitted for comparisons, must have clean thermocouple wires and ceramic covering.
The sequence of actions and annealing are given below



Transportation of the transfer standard

The following artifact is circulated among the participants for calibration:

A type S thermocouple, number ……. .

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

Delivery of the thermocouples must be carried out by participants as a hand luggage.

Participating laboratory is responsible for transferring the thermocouple to the coordinator, its return and customs registration for import to the Russian Federation and export in accordance with the «ATACornet» document.

Custom expenses on the territory of the Russian Federation are paid by the comparisons coordinator.

5. Comparison procedures

5.1 Comparison participants' actions

Each laboratory calibrates the type S thermocouple in zinc, aluminum and copper points in accordance with the procedure used in its usual practice.

During ice point measurement a quartz (glass) tube was applied to the thermocouple to protect it from water. At other fixed points the thermocouple was inserted directly into the quartz thermometer well of each fixed point.

Laboratories must measure the inhomogeneity of the transferred thermocouple.

For this purpose it is necessary to measure the maximum thermocouple readings change in oil thermostat at a temperature of 250°C with its junction immersion depth change from 100 mm to 500 mm. At homogeneity scanning the thermocouple should be immersed in a quartz (glass) tube. The inhomogeneity estimation in relative units (percent) U_{inh} is accepted equal to:

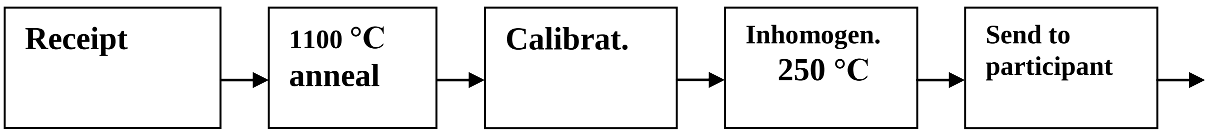
$$U_{inh}=\pm 0{,}5(E_{max} - E_{min})/(E_{ref}) \cdot 100$$

Participating laboratory transfers to VNIIM the thermocouple calibration results and the estimation of the uncertainty taking into account the result of measurement of inhomogeneity, carried out in accordance with the Annex A of this Protocol.

After returning the thermocouples from VNIIM, laboratories must repeat the their calibration and the measurement of the inhomogeneity. The results of the repeated measurements must be transmitted to VNIIM.

5.2 Coordinator’s actions – VNIIM

Calibration of the thermocouples will be performed by the coordinator, starting in copper fixed point, then in aluminum fixed point and in zinc fixed point. The fixed point temperature realization will be based on the procedure, adopted in VNIIM.
Before performing the calibration the thermocouples will be subjected to annealing for 1 hour at a temperature of (1100±20) °C, whereupon thermocouples will be removed from the furnace to cool to a room temperature.



At the calibration the thermocouples are immersed into cells with the metal after the start of the freezing plateau, indicated by the control SPRT-25 for zinc and aluminum points and by high temperature platinum resistance thermometer or standard type S thermocouple for copper point.

Minimum 10 readings of the calibrated thermocouple must be performed.

Thermocouple calibration is repeated at least 3 times in each reference fixed point.

Deviations of the results of the transferred thermocouples in each of the three calibration points in VNIIM from the results, submitted by participating laboratories, and their uncertainties are defined by coordinator.

The degree of equivalence: $D_{NMI} = X_{NMI} - X_{VNIIM}$, $X = E - E_{ref}$,
E – the result of calibration (mv),
its uncertainty:
$u_D^2 = u_{NMI}(E_{NMI})^2 + u_{VNIIM}(E_{VNIIM})^2 + (drift/2\sqrt{3})^2$,

u – combined standard uncertainty, presented by NMI,
drift – difference of the thermocouple readings between initial and final calibrations by NMI

6. Presentation of results

6.1 Calibration results on the form 1.
Appendix A - Explanation of the uncertainty calculation
6.2 Uncertainty budget on the form 2 and form 3
6.3 Measuring instruments, used for thermocouples calibration, on the form 4.

Form 1
Calibration results:

Calculation of the thermocouple emf in the metal freezing fixed point.

Measurement No.	Emf value, mV		
	1st plateau	2nd plateau	3rd plateau
1			
2			
.			
.			
.			
10			
E_i			

E_i –average value of the emf for three plateaus.

Annex A.

Emf value of the thermocouple, calibrated in the fixed point, is calculated by the equation:
$E_x = E_i + \delta V + \delta t_x \cdot C_0 + \delta V_c + \delta t \cdot C_t$(1),
where:
E_i– average mean of the emf for three plateaus (mV),
δV– instrumental correction (mV),
δt_x– correction factor to the value of the cold lead temperature of the calibrated thermocouple (°C) ,
δV_c– correction due to the use of extension cables (мВ),
δt– correction factor to the value of the reference fixed point temperature based on the results of the calibration of fixed point cell of participating laboratory (°C),
C_0 –sensitivity factor of the calibrated thermocouple at a temperature of 0 °C (mV/°C), determined by standard dependence for the thermocouple.
C_t– sensitivity of the calibrated thermocouple at a temperature

of the fixed point (mV/°C), determined by standard dependence for the thermocouple.

Form 2

Uncertainty budget for the thermocouple, calibrated in fixed point

Uncertainty component	Distribution,type	Standard uncertainty	sensitivity, mV/°C	Contribution to the combined uncertainty, mV
Standard uncertainty u (E_i)	normal, A	…..mV	1	
Uncertainty of the fixed point temperature according to the primary standard (Form 3) or cell certificate u(δt)	uniform,B	**…. (°C)**		
Uncertainty of the ice temperature u(δt_x)	uniform,B	**…. (°C)**		
Uncertainty of the correction to the measurement instrument readings u(δV)	uniform,B		1	
Uncertainty of the correction due to the use of extension cables u(δVc) [2]	uniform,B		1	
Uncertainty due to inhomogeneity of the thermocouple u (inhom) [1]	uniform,B		1	
Combined standard uncertainty u(E_x)				u(E_x)
Expanded uncertainty				U(E_x)
Temperature equivalent U(E_x)				°C

Uncertainty due to inhomogeneity of the thermocouple u(inhom) is calculated based on the linear dependence of relative estimation of the inhomogeneity on the temperature, obtained in oil thermostat [1].

Form 3

Uncertainty budget for the fixed point temperature

Uncertainty components W_t	
Repeatability of values, $W_t - u_1$	
Component due to metal purity, u_{sie}	
Component due to the hydrostatic pressure correction, u_h	

Component due to the self-heating correction, u_i	
Component due to the deviation from heat equilibrium, u_q	
Component due to the pressure in the cell, u_p	
Component due to the resistance bridge non-linearity, u_r	
Component due to the direct or alternating measuring current	
Component due to the reference resistance temperature, u_R	
Component due to the reference resistance stability, u_t	
Component due to the uncertainty of TPW, $u_{tpw} = W \bullet u(tpw)$	
Combined uncertainty	
Expanded uncertainty	

Component due to R_{tpw} measurement	
Component due to the water purity and isotope composition	
Component due to the hydrostatic pressure correction	
Component due to the self-heating correction	
Component due to the deviation from heat equilibrium	
Component due to the resistance bridge non-linearity	
Combined uncertainty, u(tpw)	

Form 4

Measuring instruments, used for thermocouples calibration

Instrument	Type	Manufacturer	Description	Characteristics	Immersion depth, mm
0°C fixed point (or room temperature)					
Zn fixed point					
Al fixed point					
Cu fixed point					
Voltmeter					
Control voltmeter					
Control thermocouple					
Control thermometer					

Appendix B

Table B1.Information on the measuring devices, used in this comparison of VNIIM

Devices		Manufacturer	Model (type)	Immersion depth	Parameters
DMM		Keithley	2002		
Ice point		VNIIM- made	Water+ ice		
Cu	cell	VNIIM- made	Closed	190 mm	
	furnace	VNIIM- made	Three zones		
Al	cell	VNIIM- made	Open	180 mm	
	furnace	VNIIM- made	Three zones		
Zn	cell	VNIIM- made	Open	180 mm	
	furnace	VNIIM- made	Three zones		
Control thermometer		VNIIM- made	SPRT 25		
Control thermocouple.		VNIIM- made	Type S		

Table B2.Information on the inhomogeneity test system of VNIIM

items	Description
Measuring DMM	Keithley 2002
Temperature enclosure	Oil Bath TR-1M-500, JSC RPI "Etalon", Omsk, Russia Maximum immersion depth of 500 mm
Scanning method	Manual scanning with 30 mm interval
Test temperature	200°C
Stability	Below 10 mK
Reference thermometer	SPRT 25

Table B3.Information on the measuring devices, used in this comparison of KazInMetr

Devices		Manufacturer	Model (type)	Immersion depth	Parameters
DMM		JSC RPI “Etalon”, Omsk, Russia	B2 -99		№ 037
Ice point		JSC RPI “Etalon”, Omsk, Russia	TN-3M	195 mm	
Cu	cell	Fluke 5909	Closed	195 mm	
	furnace		Na heat pipe		
Al	cell	Fluke 5907	Closed	195 mm	Al-07152
	furnace		Three zones		
Zn	cell	Fluke 5906	Closed	195 mm	Zn-06108
	furnace		Three zones		
Control thermometer		JSC RPI “Etalon”, Vladimir, Russia	SPRT 25		
Control thermocouple.		KazInMetr made	Type S		

Table B4. Information on the inhomogeneity test system of KazInMetr

items	Description
Measuring DMM	B2-99, JSC RPI “Etalon”, Omsk, Russia
Temperature enclosure	Oil Bath TR-1M-300, JSC RPI “Etalon”, Omsk, Russia Maximum immersion depth of 500 mm
Scanning method	Manual scanning with 30 mm interval
Test temperature	200°C
Stability	Below 10 mK
Reference thermometer	SPRT 25

Table B5.Information on the measuring devices, used in this comparison of NSC IM

Devices		Manufacturer	Model (type)	Immersion depth	Parameters
DMM		SPA ”Specialavtomatic”, Kiev, Ukraina	CA320-1		№ 12
Ice point		NSC IM - made	Water+ice		
Cu	cell	NSC IM - made	Open	190 mm	
	furnace	NSC IM - made	Three zones		
Al	cell	NSC IM - made	Open	190 mm	
	furnace	NSC IM - made	Three zones		
Zn	cell	VNIIM - made	Closed	180 mm	
	furnace	NSC IM - made	Three zones		
Control thermometer		VNIIM -made	SPRT 25		
Control thermocouple.		PJSC SPA «Thermoprylad» , Ukraina	Type S		

Table B6.Information on the inhomogeneity test system of NSC IM

items	Description
Measuring DMM	CA 320 – 1, SPA ”Specialavtomatic”, Kiev, Ukraina
Temperature enclosure	Oil Bath TR-200, JSC RPI “Etalon”, Omsk, Russia Maximum immersion depth of 500 mm
Scanning method	Manual scanning with 30 mm interval
Test temperature	200°C
Stability	Below 10 mK
Reference thermometer	SPRT 25

Appendix C

Uncertainty evaluation tables

Table C-1. Uncertainty budget of VNIIM for TC 1 at Zn point

Uncertainty component	Distribution type	Standard uncertainty	Sensitivity coefficient	Contribution to the combined uncertainty, μV
u (E_i)	normal, A	0.16 μV	1	0.16
(δt)	uniform,B	0.0005°C	9.6 μV/°C	0.005
u(δt_0)	uniform,B	0.005°C	5 μV/°C	0.025
u(δV)	uniform,B	0.02 μV	1	0.02
u(δVc)	uniform,B	-	1	-
u (inh)	uniform,B	0.013%	3443.73 μV	0.45
u(E_x)				0.48
U(E_x)				0.96
Temperature equivalent U(E_x)	0.10 °C			

Table C-2. Uncertainty budget of VNIIM for TC 1 at Al point

Uncertainty component	Distribution type	Standard uncertainty	Sensitivity coefficient	Contribution to the combined uncertainty, μV
u (E_i)	normal, A	0,26 μV	1	0,26
u(δt)	uniform,B	0,0009°C	10,4 μV/°C	0,009
u(δt_0)	uniform,B	0,005°C	5 μV/°C	0,025
u(δV)	uniform,B	0,05 μV	1	0,05
u(δVc)	uniform,B	-	1	-
u (inh)	uniform,B	0,013 %	5861,25 μV	0,76
u(E_x)				0,80
U(E_x)				1,60
Temperature equivalent U(E_x)	0,15 °C			

Table C-3. Uncertainty budget of VNIIM for TC 1 at Cu point

Uncertainty component	Distribution type	Standard uncertainty	Sensitivity coefficient	Contribution to the combined uncertainty, μV
u (E_i)	normal, A	0,28 μV	1	0,28
u(δt)	uniform,B	0,005 °C	11,8 μV/°C	0,06
u(δt_0)	uniform,B	0,005 °C	5 μV/°C	0,025
u(δV)	uniform,B	0,08 μV	1	0,08
u(δVc)	uniform,B	-	1	-
u (inh)	uniform,B	0,013 %	10587,67 μV	1,37
u(E_x)				1,40
U(E_x)				2,80
Temperature equivalent U(E_x)	0,24 °C			

Table C-4. Uncertainty budget of KazInMetr for TC 1 at Zn point

Uncertainty component	Distribution, type	Standard uncertainty	Sensitivity coefficient	Contribution to the combined uncertainty, μV
u (E_i)	normal, A	0,2 μV	1	0,2
u(δt)	uniform,B	0,00045 °C	9,6 μV/°C	0,004
u(δt_0)	uniform,B	0,01 °C	5 μV/°C	0,05
u(δV)	uniform,B	0,22 μV	1	0,22
u(δVc)	uniform,B	-	1	-
u (inh)	uniform,B	0,0125 %	3444,13 μV	0,43
u(E_x)				0,52
U(E_x)				1,04
Temperature equivalent U(E_x)	0,11°C			

Table C-5. Uncertainty budget of KazInMetr for TC 1 at Al point

Uncertainty component	Distribution, type	Standard uncertainty	Sensitivity coefficient	Contribution to the combined uncertainty, μV
u (E_i)	normal, A	0,49 μV	1	0,49
u(δt)	uniform,B	0,00065 °C	10,4 μV/°C	0,007
u(δt_0)	uniform,B	0,01 °C	5 μV/°C	0,05
u(δV)	uniform,B	0,25 μV	1	0,25
u(δVc)	uniform,B	-	1	-
u (inh)	uniform,B	0,0125 %	5862,09 μV	0,73
u(E_x)				0,91
U(E_x)				1,82
Temperature equivalent U(E_x)	0,18 °C			

Table C-6. Uncertainty budget of KazInMetr for TC 1 at Cu point

Uncertainty component	Distribution, type	Standard uncertainty	Sensitivity coefficient	Contribution to the combined uncertainty, μV
u (E_i)	normal, A	0,51 μV	1	0,51
u(δt)	uniform,B	0,005 °C	11,8 μV/°C	0,06
u(δt_0)	uniform,B	0,01 °C	5 μV/°C	0,05
u(δV)	uniform,B	0,3 μV	1	0,3
u(δVc)	uniform,B	-	1	-
u (inh)	uniform,B	0,0125 %	10598,36 μV	1,32
u(E_x)				1,45
U(E_x)				2,90
Temperature equivalent U(E_x)	0,25 °C			

Table C-7. Uncertainty budget of VNIIM for TC 085 at Zn fixed point

Uncertainty component	Distribution type	Standard uncertainty	Sensitivity coefficient	Contribution to the combined uncertainty, μV
u (E_i)	normal, A	0,14 μV	1	0,14
u(δt)	uniform,B	0,0005 °C	9,6 μV/°C	0,005
u($δt_0$)	uniform,B	0,005 °C	5 μV/°C	0,025
u(δV)	uniform,B	0,02 μV	1	0,0 2
u(δVc)	uniform,B	-	1	-
u (inh)	uniform,B	0,011 %	3444,96 μV	0,38
u(E_x)				0,41
U(E_x)				0,82
Temperature equivalent U(E_x)	0,08°C			

Table C-8. Uncertainty budget of VNIIM for TC 085 at Al fixed point

Uncertainty component	Distribution type	Standard uncertainty	Sensitivity coefficient	Contribution to the combined μV uncertainty,
u (E_i)	normal, A	0,18 μV	1	0,18
u(δt)	uniform,B	0,0015°C	10,4 μV/°C	0,016
u($δt_0$)	uniform,B	0,005 °C	5 μV/°C	0,025
u(δV)	uniform,B	0,05 μV	1	0,05
u(δVc)	uniform,B	-	1	-
u (inh)	uniform,B	0,011 %	5857,67 μV	0,64
u(E_x)				0,67
U(E_x)				1,34
Temperature equivalent U(E_x)	0,13 °C			

Table C-9. Uncertainty budget of VNIIM for TC 085 at Cu fixed point

Uncertainty component	Distribution type	Standard uncertainty	Sensitivity coefficient	Contribution to the combined uncertainty, μV
u (E_i)	normal, A	0,45 μV	1	0,45
u(δt)	uniform,B	0,005 °C	11,8 μV/°C	0,06
u(δt_0)	uniform,B	0,005 °C	5 μV/°C	0,025
u(δV)	uniform,B	0,08 μV	1	0,08
u(δVc)	uniform,B		1	
u (inh)	uniform,B	0,011 %	10578,67 μV	1,16
u(E_x)				1,25
U(E_x)				2,50
Temperature equivalent U(E_x)	0,21 °C			

Table C-10. Uncertainty budget of NSC IM for TC 085 at Zn fixed point

Uncertainty component	Distribution,type	Standard uncertainty	Sensitivity coefficient	Contribution to the combined uncertainty, μV
u (E_i)	normal, A	0,15 μV	1	0,15
u(δt)	uniform,B	0,0005 °C	9,6 μV/°C	0,005
u(δt_0)	uniform,B	0,01 °C	5 μV/°C	0,05
u(δV)	uniform,B	0,12 μV	1	0,12
u(δVc)	uniform,B	-	1	-
u (inh)	uniform,B	0,012 %	3443,40 μV	0,41
u(E_x)				0,46
U(E_x)				0,92
Temperature equivalent (E_x)	0,09°C			

Table C-11. Uncertainty budget for TC 085 at Al fixed point of NSC IM

	Distribution, type	Standard uncertainty	Sensitivity coefficient	Contribution to the combined uncertainty, μV
u (E_i)	normal, A	0,25 μV	1	0,25
u(δt)	uniform,B	0,0015 °C	10,4 μV/°C	0,016
u(δt_0)	uniform,B	0,01 °C	5 μV/°C	0,05
u(δV)	uniform,B	0,13 μV	1	0,13
u(δVc)	uniform,B	-	1	-
u (inh)	uniform,B	0,012 %	5856,96 μV	0,70
u(E_x)				0,76
U(E_x)				1,52
Temperature equivalent U(E_x)	0,15 °C			

Table C-12. Uncertainty budget for TC 085 at Cu fixed point of NSC IM

Uncertainty component	Distribution, type	Standard uncertainty	Sensitivity coefficient	Contribution to the combined uncertainty, μV
u (E_i)	normal, A	0,33 μV	1	0,33
u(δt)	uniform,B	0,010 °C	11,8 μV/°C	0,06
u(δt_0)	uniform,B	0,01 °C	5 μV/°C	0,05
u(δV)	uniform,B	0,14 μV	1	0,14
u(δVc	uniform,B	-	1	-
u (inh)	uniform,B	0,012 %	10577,17 μV	1,27
u(E_x)				1,32
U(E_x)				2,64
Temperature equivalent U(E_x)	0,22°C			

Uncertainty Budget for the Freezing Point of Zn and Al VNIIM

Uncertainty components	mK	
	Zn	**Al**
Repeatability of the W_t values	0,291	0,61
Component due to metal purity	0,310	0,53
Component due to correction for hydrostatic pressure	0,010	0,010
Component due to correction for self-heating	0,025	0,045
Component due to deviation from thermal equilibrium	0,160	0,280
Component due to pressure in the cell	0,0012	0,0012
Component due to propagated of TPW	0,039	0,039
Component due to a.c. or d.c.	-	-
Component due to nonlinearity of the bridge	0,003	0,013
Component due to temperature of the reference resistor	0,0001	0,0001
Component due to stability of the reference resistor	-	-
Components due to measurement in the TPW		
Component due to purity, isotopic composition of water	0,010	0,010
Component due to correction for hydrostatic pressure	0,001	0,001
Component due to correction for self-heating	0,006	0,006
Component due to deviation from thermal equilibrium	0,006	0,006
Component due to a.c. or d.c.	-	-
Component due to nonlinearity of the bridge	0,003	0,003
Total uncertainty (k=1)	**0,457**	**0,86**
Expanded uncertainty (k=2)	**0,914**	**1,72**

Uncertainty Budget for the Freezing Point of Cu VNIIM

Uncertainty components	Cu
	mK
Repeatability of the emf	2,0
Component due to metal purity	5,0
Component due to correction for hydrostatic pressure	0,0013
Component due to pressure in the cell	0,04
Total uncertainty (k=1)	5,4
Expanded uncertainty (k=2)	10,8

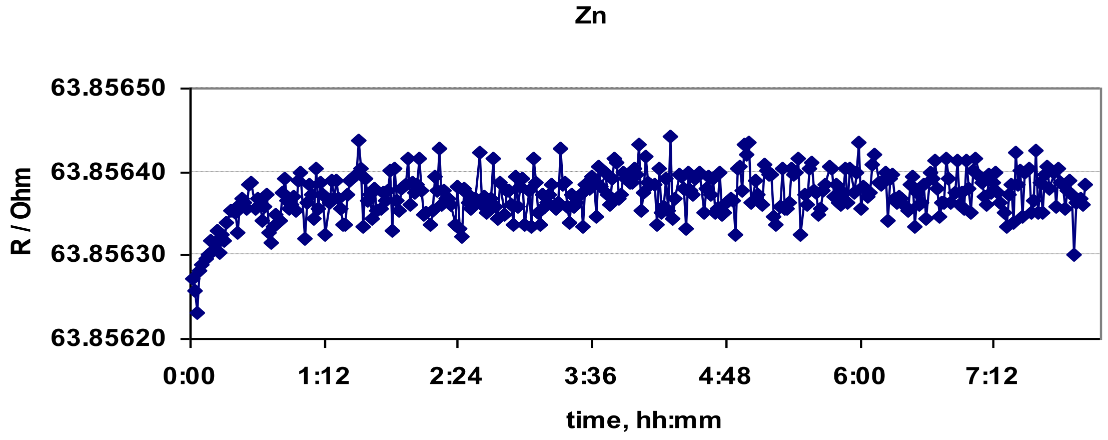
Uncertainty Budget for the Freezing Point of Zn, Al, Cu NSC IM

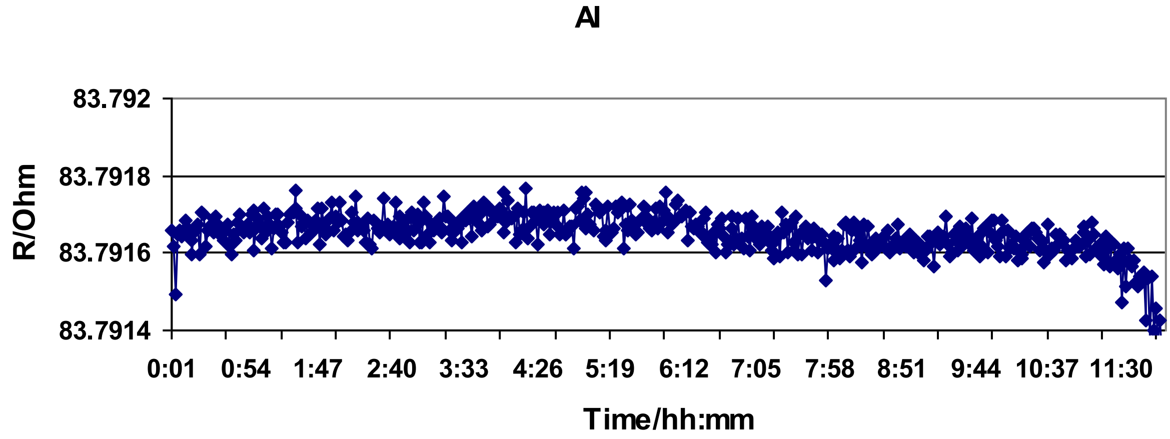
Uncertainty components	mK		
	Zn	**Al**	**Cu**
Repeatability of the W_t values	0,210	0,580	0,980
Component due to metal purity	0,400	0,570	0,660
Component due to correction for hydrostatic pressure	0,008	0,005	0,008
Component due to correction for self-heating	0,024	0,043	0,074
Component due to deviation from thermal equilibrium	0,070	0,120	0,311
Component due to pressure in the cell	0,005	0,008	0,004
Component due to propagated of TPW	0,256	0,256	0,256
Component due to a.c. or d.c.	-	-	-
Component due to nonlinearity of the bridge	0,074	0,088	0,097
Component due to temperature of the reference resistor	0,005	0,005	0,005
Component due to stability of the reference resistor	-	-	-
Components due to measurement in the TPW			
Component due to purity, isotopic composition of water	0,050	0,050	0,050
Component due to correction for hydrostatic pressure	0,002	0,002	0,002
Component due to correction for self-heating	0,042	0,042	0,042
Component due to deviation from thermal equilibrium	0,050	0,050	0,050
Component due to a.c. or d.c.	-	-	-
Component due to nonlinearity of the bridge	0,029	0,029	0,029
Total uncertainty (k=1)	0,530	0,870	1,257
Expanded uncertainty (k=2)	1,060	1,740	2,514

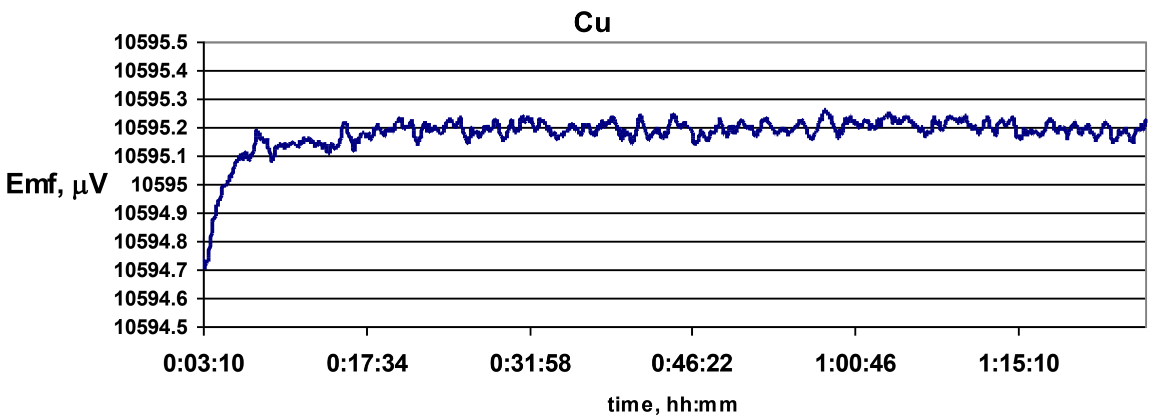
Appendix D

Freezing plateaus measured with control SPRT for Zn, Al and with control thermocouple for Cu and temperature profiles in cells are presented below

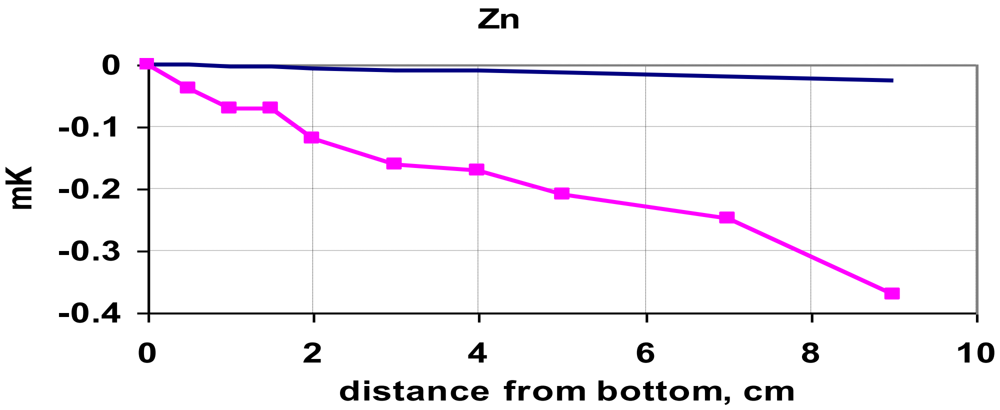
VNIIM

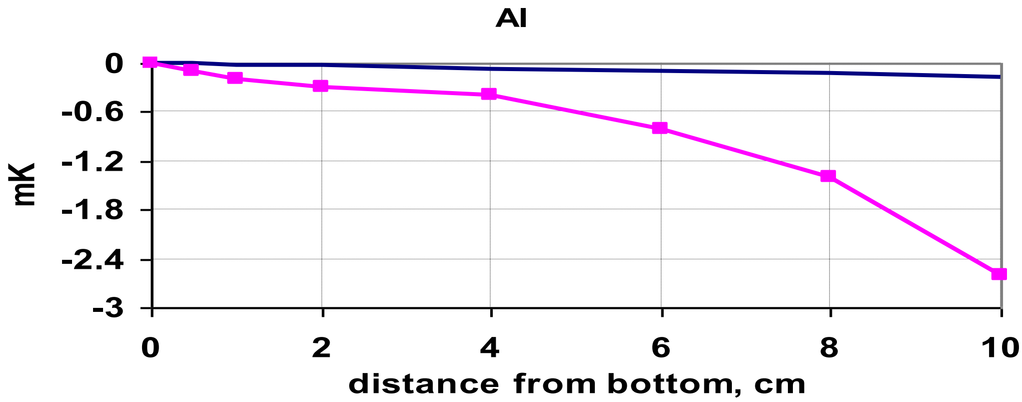


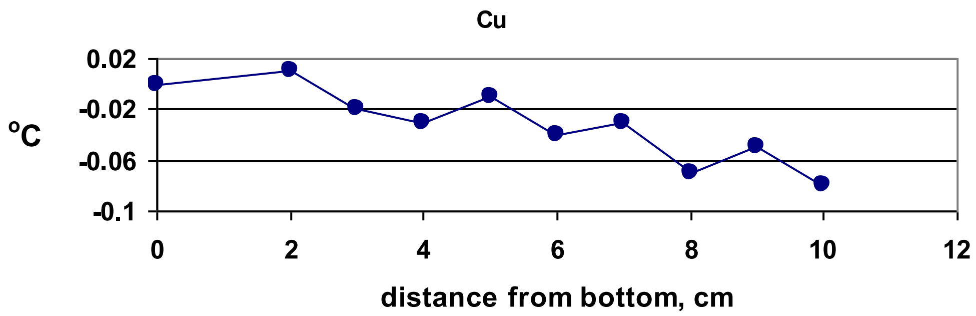




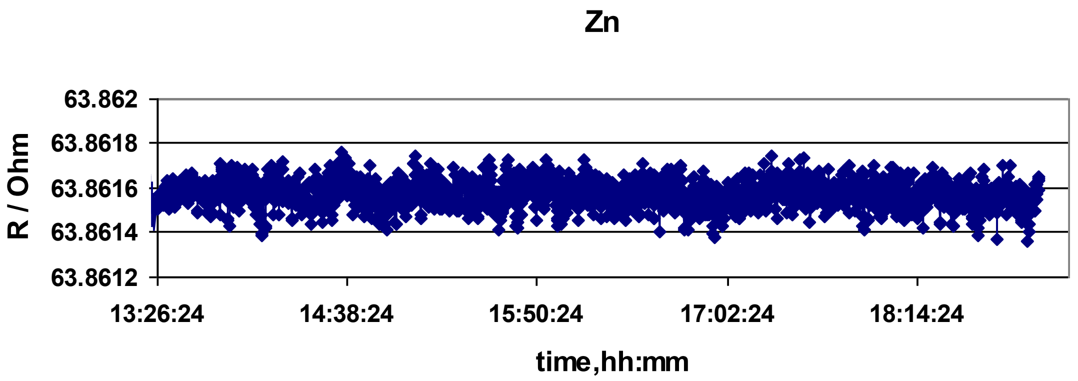
VNIIM


Zn
0
-0.1
-0.2
-0.3
-0.4
mK
0
2
4
6
8
10
distance from bottom, cm

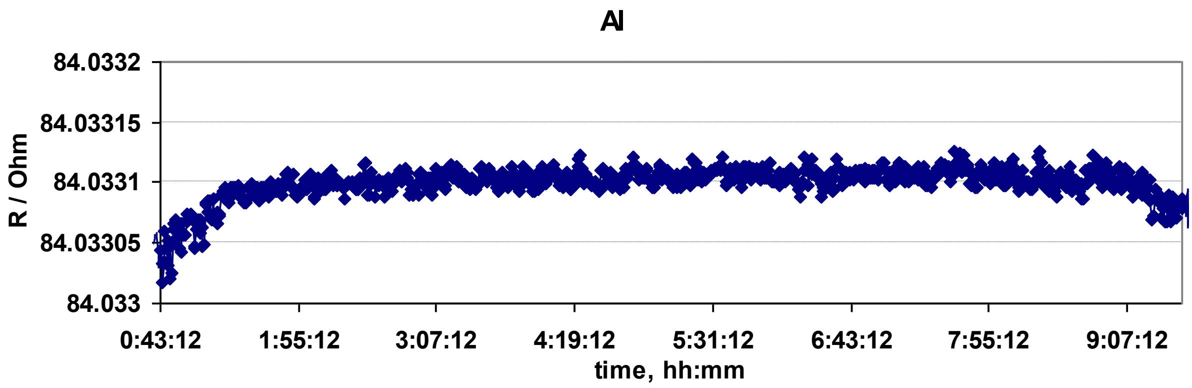

Al
0
-0.6
-1.2
-1.8
-2.4
-3
mK
0
2
4
6
8
10
distance from bottom, cm


Cu
0.02
-0.02
-0.06
-0.1
°C
0
2
4
6
8
10
12
distance from bottom, cm

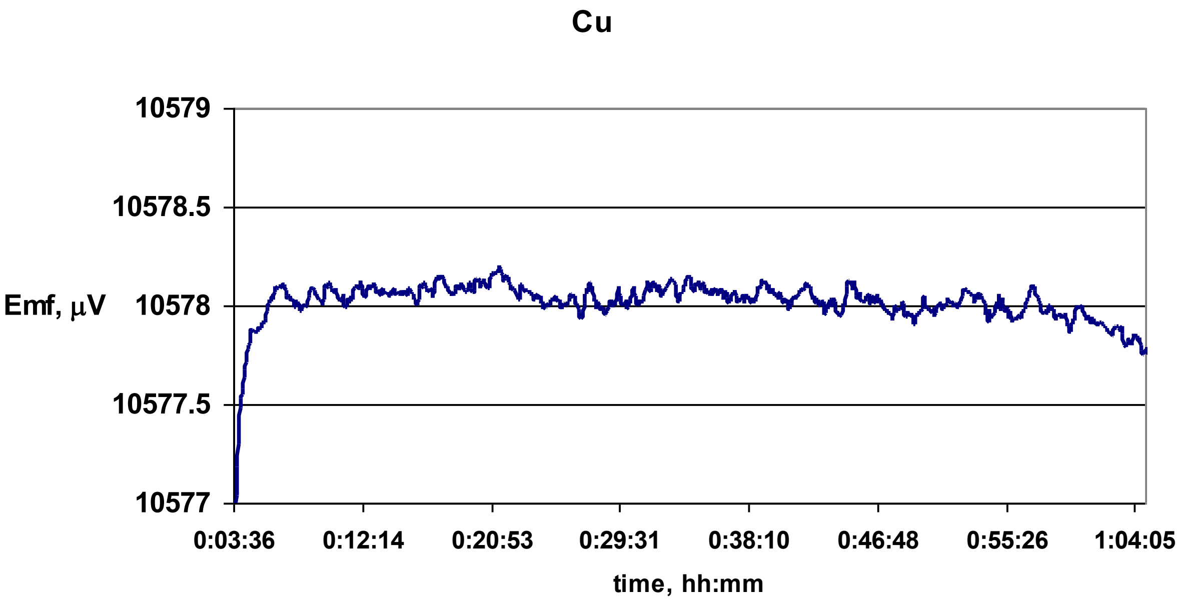
NSC IM



Zn
63.862
63.8618
63.8616
63.8614
63.8612
R / Ohm
13:26:24
14:38:24
15:50:24
17:02:24
18:14:24
time,hh:mm

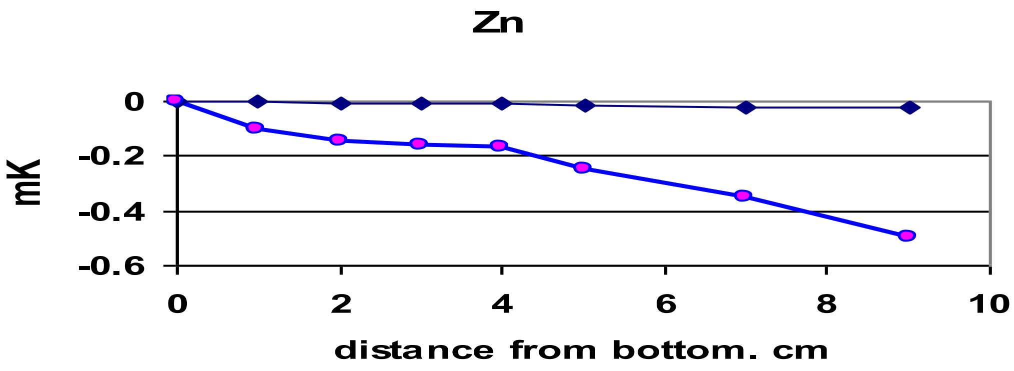


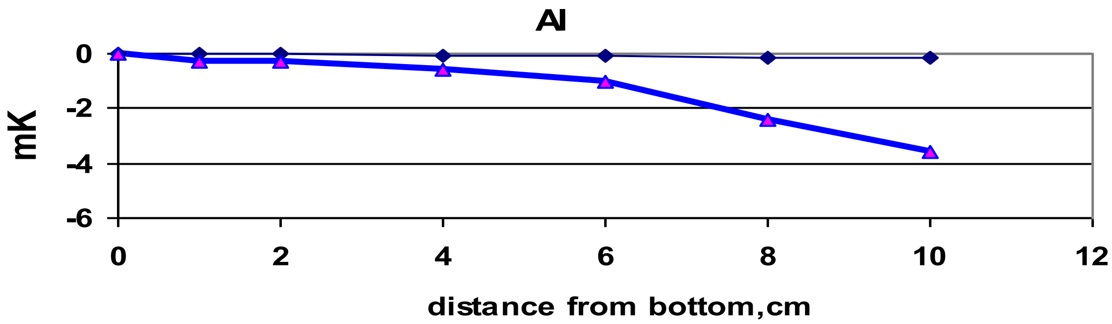
Al
84.0332
84.03315
84.0331
84.03305
84.033
R / Ohm
0:43:12
1:55:12
3:07:12
4:19:12
5:31:12
6:43:12
7:55:12
9:07:12
time, hh:mm

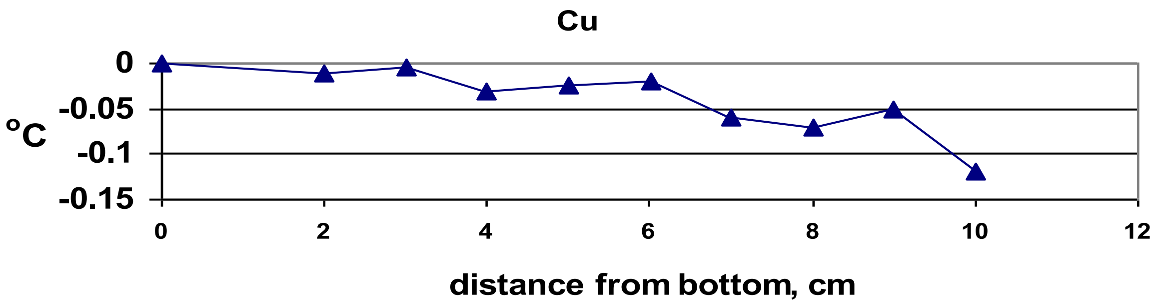


Cu
10579
10578.5
10578
10577.5
10577
Emf, µV
0:03:36
0:12:14
0:20:53
0:29:31
0:38:10
0:46:48
0:55:26
1:04:05
time, hh:mm

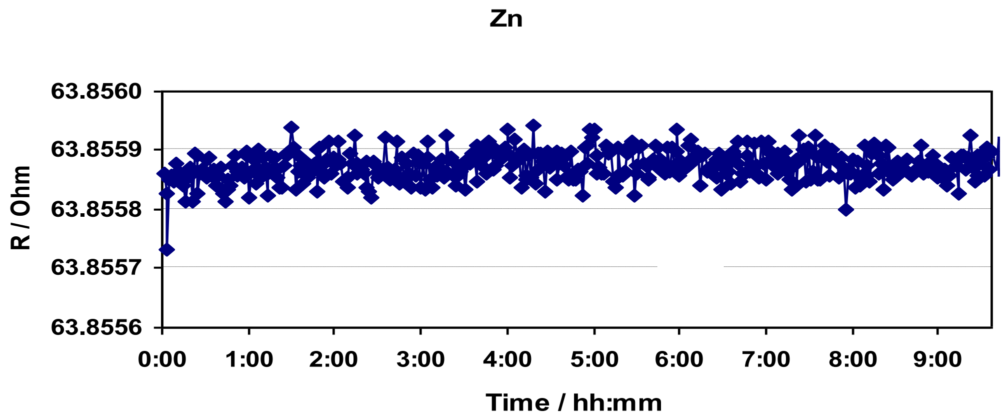
NSC IM


Zn
0
-0.2
-0.4
-0.6
mK
0
2
4
6
8
10
distance from bottom. cm

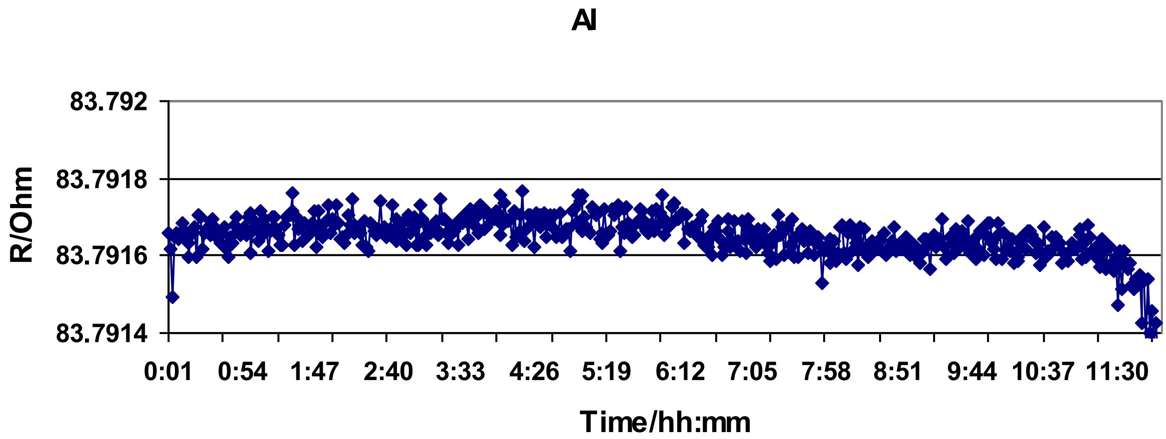

Al
0
-2
-4
-6
mK
0
2
4
6
8
10
12
distance from bottom,cm


Cu
0
-0.05
-0.1
-0.15
°C
0
2
4
6
8
10
12
distance from bottom, cm

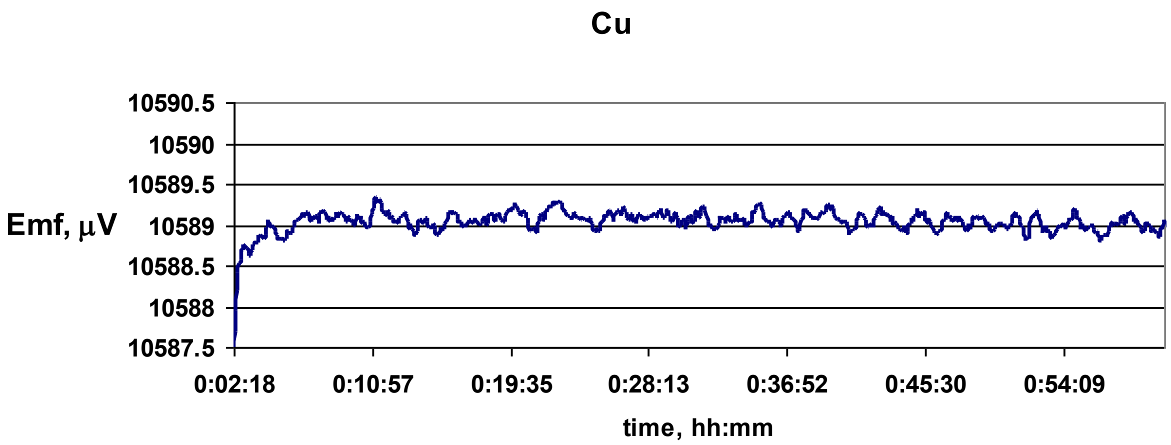
KazInMetr



Zn
63.8560
63.8559
63.8558
63.8557
63.8556
R / Ohm
0:00
1:00
2:00
3:00
4:00
5:00
6:00
7:00
8:00
9:00
Time / hh:mm

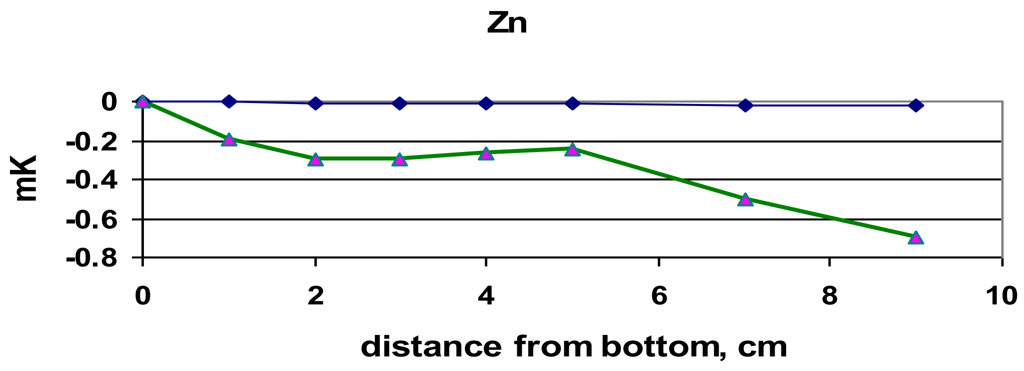


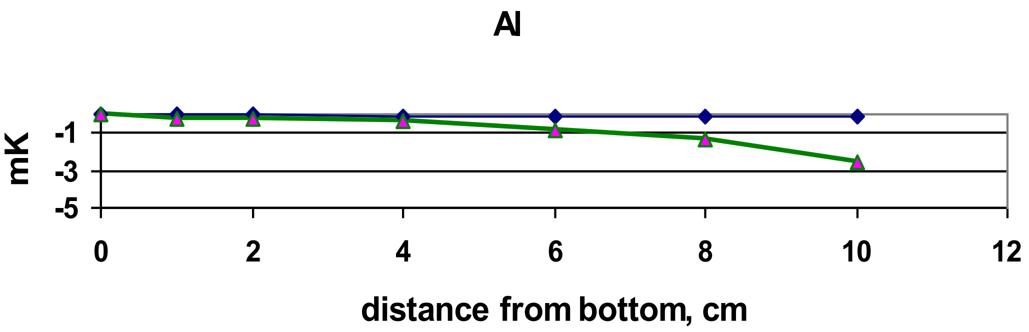
Al
83.792
83.7918
83.7916
83.7914
R/Ohm
0:01
0:54
1:47
2:40
3:33
4:26
5:19
6:12
7:05
7:58
8:51
9:44
10:37
11:30
Time/hh:mm

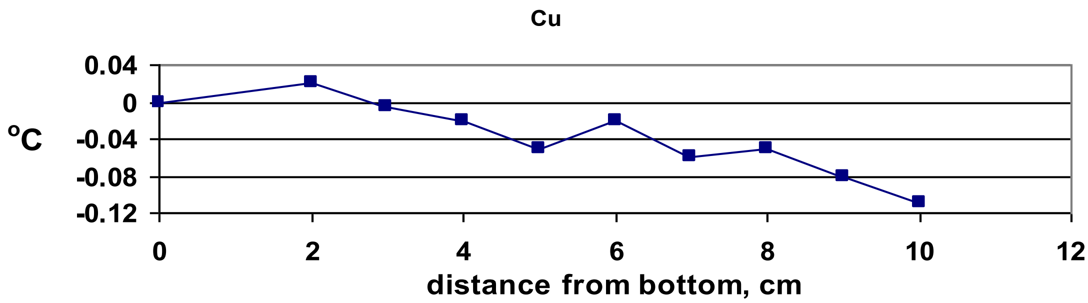


Cu
10590.5
10590
10589.5
10589
10588.5
10588
10587.5
Emf, µV
0:02:18
0:10:57
0:19:35
0:28:13
0:36:52
0:45:30
0:54:09
time, hh:mm

KazInMetr


Zn
0
-0.2
-0.4
-0.6
-0.8
mK
0
2
4
6
8
10
distance from bottom, cm


Al
-1
-3
-5
mK
0
2
4
6
8
10
12
distance from bottom, cm


Cu
0.04
0
-0.04
-0.08
-0.12
°C
0
2
4
6
8
10
12
distance from bottom, cm