REPORT to the CCT on Key Comparison «COOMET.T-K3.2»

(COOMET theme No. 494/RU/10)

Final Report

Realizations of the ITS-90 from 0.01ºC to 419.572ºC 2010 – 2012

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CONTENT

1. Introduction

The signing by the majority of COOMET countries of the Arrangement on Mutual recognition of national measurement standards and of calibration and measurement certificates issued by national metrology institutes had made it necessary to carry out regional comparisons within the COOMET Region. The results of these comparisons and their links with the results obtained in key comparisons form the basis for confirmation and support of metrological characteristics claimed by national metrology institutes.

The decision on undertaking the regional COOMET comparison was taken on Technical Committee TC1-10 meeting in 14.10.2009 in Kharkov. It was also proposed to organize a supplementary comparison of national measurement standards in the field of contact thermometry of the fixed points: melting of gallium, freezing of indium, tin and zinc. It is supposed that in this comparison a long stem standard platinum resistance thermometer should be used as a transfer standard.

Registration of “COOMET.T.-K-3” in the KCDB, the positions to be supported: 1.1.1, 1.3.1, 1.3.2, 2.2.2, 2.3.1.

The purpose of this supplementary regional comparison is to disseminate the metrological equivalence to the measurement standards of those national metrology institutes (NMIs) that did not participate in the key comparison organized by BIPM. The degree of equivalence of the NMIs standards is determined with respect to the CCT-K3 key comparison results through the measurement results obtained in the linking NMI having participated in both comparisons. In this comparison the linking NMI was VNIIM (“D.I. Mendeleyev Institute of Metrology”, St. Petersburg).

2. Organization of the comparison

In this Section the main provisions and the comparison scheme are described. The details and procedures are described in the Technical Protocol (see Appendix A).

2.1 Participating Laboratories

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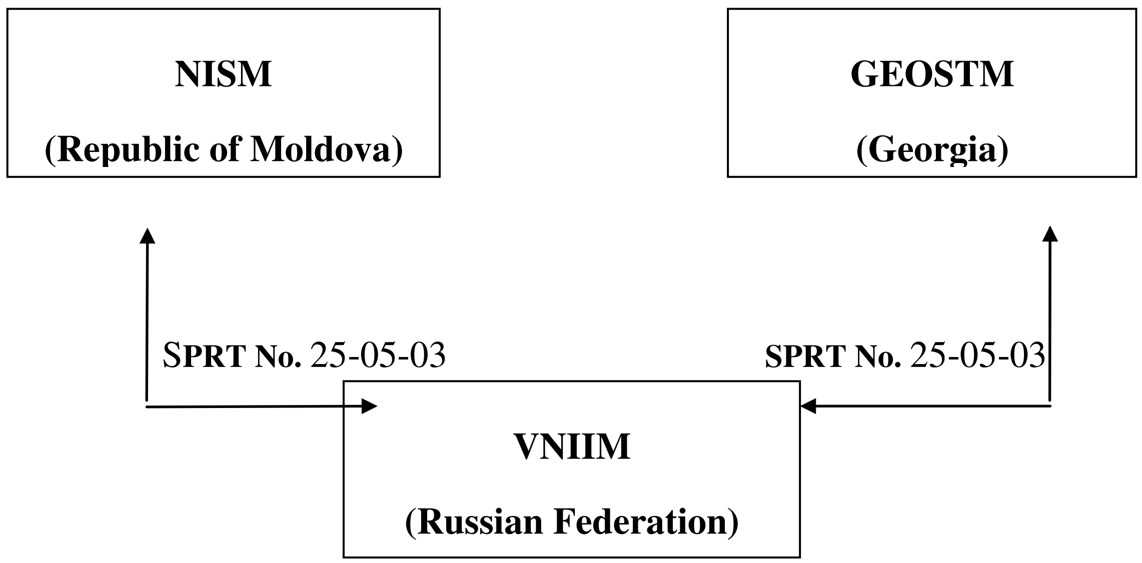
NISM – National Institute of Standardization and Metrology, Republic of Moldova, str.Coca, 28, 2064, Kishenev, Republic of Moldova,
e-mail moldovastandart@standart.mldnet.com

GEOSTM – Georgian National Agency for tandards, Technical Regulations and Metrology, Chargalsaya Str., 67, Tbilisi, Georgia.

The supplementary regional comparison was piloted and coordinated by VNIIM (Russian Federation).

2.2 The comparison scheme and schedule

As a transfer standard for this supplementary comparison the standard platinum resistance thermometer SPRT No.25-05-03, manufactured by VNIIM, was used. Before the comparison it was calibrated at VNIIM and its stability was controlled at VNIIM after the measurements at NMIs.



VNIIM-GEOSTM	2010
VNIIM-NISM	2011
VNIIM	2012

3 The bilateral equivalence of the GEOSTM and NISM standards relatively to the VNIIM standards

3.1. The measurement results of GEOSTM, NISM, VNIIM

The measurement results W_i, their uncertainties $U(W_i)(k=2)$, submitted by NMIs, differences $\Delta W_i = W_{NMI} - W_{VNIIM}$, and corresponding differences

$\Delta T_i = T_{NMI} - T_{VNIIM}$ are presented in Tables 1-4.

All the information about the uncertainty budgets are presented in Appendix C.

Table 1.Gallium fixed point

NMI	W_{Ga}	$U(W_{Ga})$ mK	ΔW_{Ga} (NMI-VNIIM)	T_{NMI}-T_{VNIIM} mK
VNIIM	1.118 100 71	0.18	0	0
NISM	1.118 100 15	0.41	0.000 000 56	-0.14

Table 2.Indium fixed point

NMI	W_{In}	$U(W_{In})$ mK	ΔW_{In} (NMI-VNIIM)	T_{NMI}-T_{VNIIM} mK
VNIIM	1.609 591 17	0.74	0	0
NISM	1.609 588 88	1.16	- 0.000 002 29	- 0.60

Table 3.Tin fixed point

NMI	W_{Sn}	$U(W_{Sn})$ mK	ΔW_{Sn} (NMI-VNIIM)	T_{NMI}-T_{VNIIM} mK
VNIIM	1.892 485 12	0.78	0	0
GEOSTM	1.892 472 95	2.61	0.000 012 17	- 3.29
NISM	1.892 481 54	0.81	0.000 003 58	- 0.97

Table 4.Zinc fixed point

NMI	W_{Zn}	U(W_{Zn}) mK	ΔW_{Zn} (NMI-VNIIM)	T_{NMI}-T_{VNIIM} mK
VNIIM	2.568 351 55	1.08	0	0
NISM	2.568 345 75	1.48	- 0.000 005 80	- 1.66

3.2. The measurement **r**esults taking into account instability of the transfer thermometer

In addition to the uncertainties reported by laboratories the uncertainties for possible changes in the transfer thermometer SPRT No.25-05-03 over the course of the comparison has to be taken into account for temperature difference. The uncertainty due to instability of the transfer standard u_{prt} (κ=1) was computed under the assumption that the thermometer resistance drift distribution in time was rectangular and asymmetrical:

$$u_{prt} = \left[(W_{VNIIM})_{end} - (W_{VNIIM})_{begin} \right] \times \frac{\partial T}{\partial W} \times \frac{1}{\sqrt{3}}$$

Table 5. Evaluation of the transfer standard thermometer instability for the VNIIM-GEOSTM period

Fixed point	$W_{VNIIM-GEOSTM}$	$W_{GEOSTM-VNIIM}$	Δ W	ΔT mK	u_{prt} mK (κ=1)
Sn	1.892 482 19	1.892 479 71	0.00000248	0.67	0.39

Table 6. Evaluation of the transfer standard thermometer instability for the VNIIM- NISM period

Fixed point	$W_{VNIIM-NISM}$	$W_{NISM-VNIIM}$	Δ W	ΔT mK	u_{prt} mK (κ=1)
Ga	1.118 101 64	1.118 100 81	0.00000083	0.21	0.12
In	1.609 591 84	1.609 590 17	0.00000167	0.44	0.25
Sn	1.892 483 39	1.892 480 92	0.00000247	0.67	0.38
Zn	2.568 352 10	2.568 349 13	0.00000297	0.85	0.49

For a direct comparison between two laboratories using a single transfer thermometer the uncertainty can be calculated

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

Table 7. The bilateral equivalence results taking into account SPRT instability

Fixed point	ΔT(**INSM -VNIIM**) **mK**	u(ΔT) (k=1), mK	ΔT(**GEOSTM -VNIIM**) **mK**	u(ΔT) (k=1), mK
Ga	**-0.14**	**0.25**	**-**	**-**
In	**-0.60**	**0.73**	**-**	**-**
Sn	**-0.97**	**0.68**	**-3.29**	**1.41**
Zn	**-1.66**	**1.04**	**-**	**-**

4 Linkage the COOMET comparison results of GEOSTM and NISM with the CCT-K3 results

In this COOMET comparison VNIIM was the linking institute for the determination of the degree of equivalence of the obtained results relative to CCT-K3 results.

As the results of CCT-K3 have been presented as differences [T_{NMI} - ARV (K3)], the degree of equivalence "***d***" of the NMI results can be calculated on the corresponding relationship: $d_i = (T_{NMI} - T_{VNIIM}) + \left[T_{VNIIM} - ARV(K3)\right]$,

the standard uncertainty u(d)

$$u^2(d) = u^2(T_{NMI} - T_{VNIIM}) + u^2\left[T_{VNIIM} - ARV(K3)\right]$$

The results of CCT-K3 used in the calculations are given in Table 8.

Table 8. Results of CCT-K3

NMI	**Fixed point**	**T_{NMI} – ARV(K3), mK**	**U[T_{NMI}-ARV(K3)] (к=2), mK**
VNIIM	Ga	0.05	0.25
	In	0.54	1.11
	Sn	0.59	0.99
	Zn	0.52	1.85

4.1 Gallium fixed point

VNIIM: result of CCT-K3 T_{VNIIM} - ARV (K3) = 0.05 mK,
result of COOMET $T_{NISM} - T_{VNIIM}$ = - 0.14 mK.

Equivalence of the NISM result to relatively ARV(K3) d_{Ga} = [T_{NISM} – ARV(K3)]

d_{Ga} = - 0.09 mK,

the standard uncertainty **u(d_{Ga}) = 0.28 mK**.

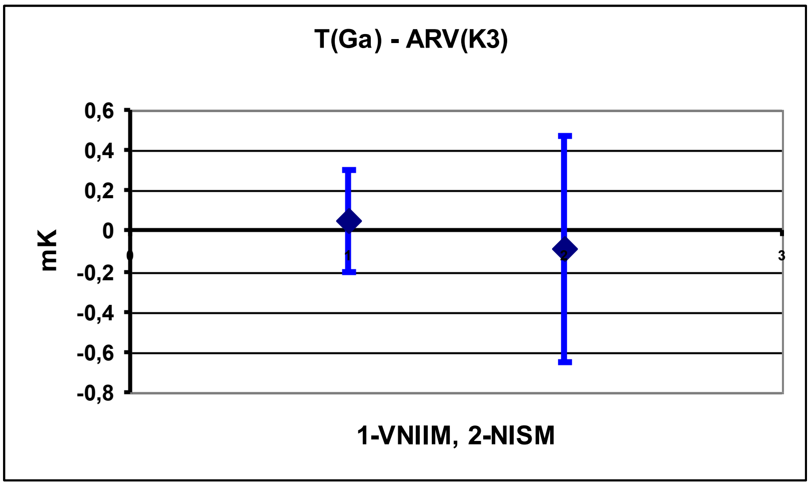


Fig.1 The differences [T_{NMI} – ARV(K3)] for Ga fixed point, expanded uncertainties (k=2)

4.2 Indium fixed point

VNIIM: result of CCT-K3 T_{VNIIM} - ARV (K3) = 0.54 mK,
result of COOMET $T_{NISM} – T_{VNIIM}$ = - 0.60 mK.

Equivalence of the NISM result to relatively ARV(K3)

$\mathbf{d_{In}}$ = [T_{NISM} – ARV(K3)] **d_{In} = 0.06 mK,**

the standard uncertainty **$u(d_{In})$ = 0.92 mK**.

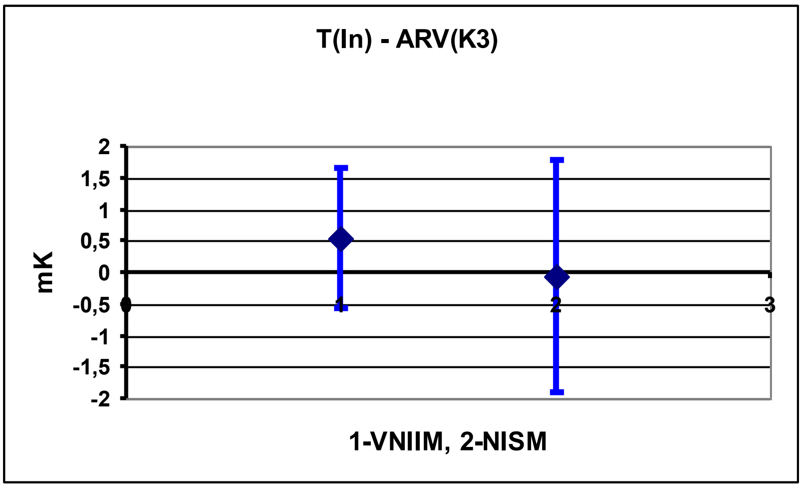


Fig.2. The differences [T_{NMI} – ARV(K3)] for In fixed point, expanded uncertainties (k=2)

4.3 Tin fixed point

VNIIM: result of CCT-K3 T_{VNIIM} - ARV (K3) = 0.59 mK,
result of COOMET $T_{NISM} – T_{VNIIM}$ = - 0.97 mK,
$T_{GEOSTEM} – T_{VNIIM}$= -3.29 mK.

Equivalence of the NISM result to relatively ARV(K3)
$\mathbf{d_{Sn}} = [\ T_{NISM} - ARV(K3)]$ $\mathbf{d_{Sn} = -0.38\ mK,}$
the standard uncertainty **$u(d_{Sn}) = 0.84$ mK**.

Equivalence of the GEOSTM result to relatively ARV(K3)
$\mathbf{d_{Sn}} = [\ T_{GEOSTM} - ARV(K3)]$ $\mathbf{d_{Sn} = -2.70\ mK,}$
the standard uncertainty **$u(d_{Sn}) = 1.50$ mK**.

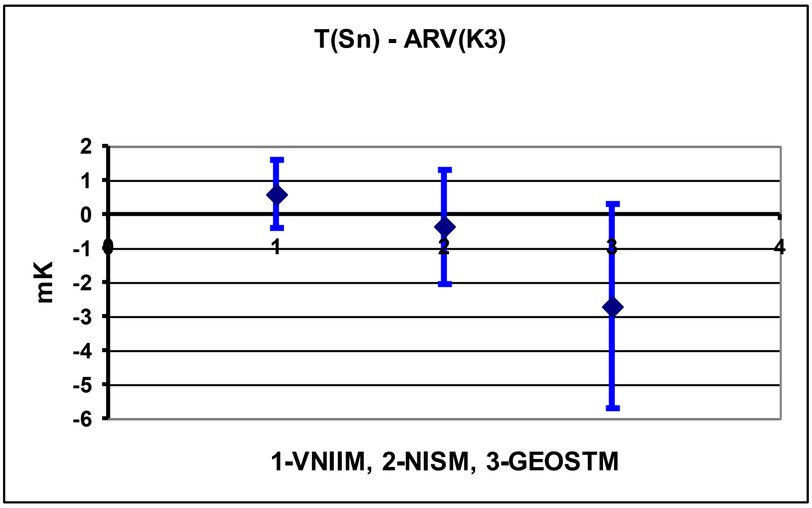


Fig.3 The differences $[T_{NMI} - ARV(K3)]$ for Sn fixed point, expanded uncertainties (k=2).

4.4 Zinc fixed point

VNIIM: result of CCT-K3 T_{VNIIM} - ARV (K3) = 0.52 mK,
result of COOMET $T_{NISM} - T_{VNIIM}$ = -1.66 mK.

Equivalence of the NISM result to relatively ARV(K3)
$d_{Zn} = [\ T_{NISM} - ARV(K3)]$ $\mathbf{d_{Zn} = -1.14\ mK,}$
the standard uncertainty **$u(d_{Zn}) = 1.32$ mK**.

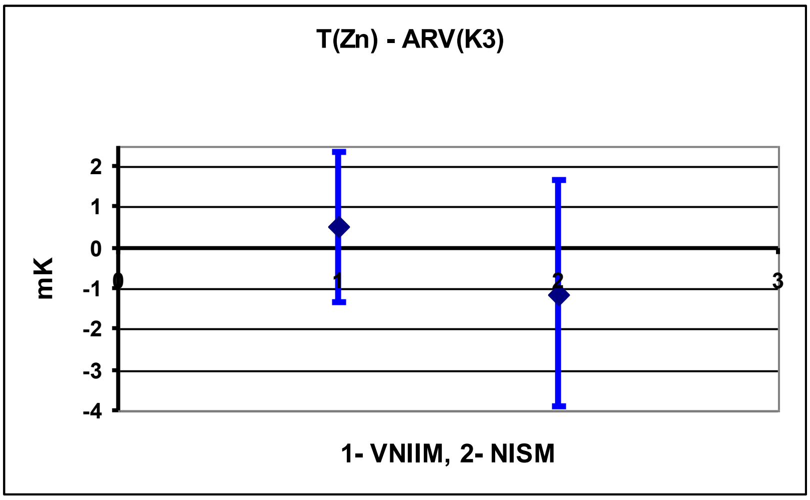


Fig.4 The differences [T_{NMI} – ARV(K3)] for Zn fixed point and expanded uncertainties (k=2).

5 Conclusion

The purpose of the comparison is the determination of the degree of equivalence of fixed points of the two national metrology institutes: NISM and GEOSTM relatively to the CCT-K3 results and the confirmation of the uncertainties, claimed by them for CMC. VNIIM was the linking NMI in this comparison.

Summary results of the comparison are presented in Table 9.

Table 9. The degrees of equivalence and their expanded uncertainties

Fixed point	NMI	ΔT [T_{NMI} – ARV(K3)], mK	U(ΔT) (k=2), mK
Ga	NISM	-0.09	0.57
In	NISM	-0.06	1.84
Sn	NISM	-0.38	1.68
Zn	NISM	-1.14	2.78
Sn	GEOSTM	-2.70	3.00

The received results allow to confirm the uncertainties declared by GEOSTM and NISM for the appropriate CMC lines.

Appendix A

Technical Protocol

Supplementary regional comparison of the national standards of temperature in the range from the triple point of water to the freezing temperature of zinc

COOMET theme No.494/RU/10

Introduction

The decision on undertaking the regional COOMET comparison was taken by Technical Committee TC1-10 in 14.10.2009 in Kharkov. It was also proposed to organize a supplementary comparison of national measurement standards in the field of contact thermometry in fixed points of melting of gallium, freezing of indium, tin and zinc. It is supposed that in this comparison a long stem standard platinum resistance thermometer should be used as a transfer standard.

The purpose of this supplementary regional comparison is to disseminate the metrological equivalence to the measurement standards of those national metrology institutes (NMIs) that did not participate in the key comparison organized by the BIPM. The degree of equivalence of the NMIs standards is determined with respect to the K3 key comparison results through the measurement results obtained in the linking NMI having participated in both comparisons. In this comparison the linking NMI was VNIIM ("D.I. Mendeleyev Institute of Metrology", St. Petersburg).

Registration of "COOMET.T.-K-3" in the KCDB, the positions to be supported: 1.1.1, 1.3.1, 1.3.2, 2.2.2, 2.3.1.

1 Participating Laboratories

See Page 3 of the draft B

2 The comparison scheme and schedule

See Page 4 of the draft B

3 Procedures

All the participants of the comparisons shall act according to the instruction given below. Each laboratory shall apply the accepted practice for realization of the ITS-90 during the comparisons.

The instruction is written in compliance with Appendix 1 of the CCT Report on key comparisons CCT-K3. The comparisons strictly follow the protocols given in the CIPM Guidance for key comparisons and in Appendix F to the document "On Mutual Recognition…" [ ].

3. 1 **Actions of VNIIM as a coordinating laboratory**

The coordinating laboratory VNIIM calibrates the SPRT No25-05-03.

The coordinating laboratory VNIIM transfers the calibrated SPRT to GEOSTM for its calibrations at the fixed points in accordance with the practice accepted at the laboratory. After the end of calibration GEOSTM returns the SPRT to VNIIM for checking the SPRT stability.

Then VNIIN transfers the SPRT to INSM for its calibrations at the fixed points in accordance with the practice accepted at the laboratory. After the end of calibration INSM returns the SPRT to VNIIM for checking the SPRT stability.

The coordinator collects reports on calibration of the SPRT from the participating laboratories and carries out the analysis of the results.

3.2 **Actions of the participating laboratories**

3.2.1 After receiving the calibrated SPRT No25-05-03 from the coordinator, GEOSTM and INSM perform the following procedures:

3.2.2 Measurement of the SPRT resistance at the triple point of water.

3.2.3 Annealing of the SPRT in a certain sequence:

a) the SPRT is inserted in the furnace at the temperature 500 ^{o}C;

b) the temperature value in the furnace is raised to 600 ^{o}C;

c) the SPRT is kept in the furnace during two hours;

d) the temperature value in the furnace is reduced to 450 ^{o}C during 2,5 hours;

e) the SPRT is quickly removed from the furnace to the air and cooled to the room temperature.

3.2.4 Measurement of the SPRT resistance at the triple point of water.

If the SPRT resistance at the triple point of water after annealing is changed by less than 0,5 mK in the temperature equivalent, it is possible to start its calibration at the fixed points.

If the SPRT resistance at the triple point of water after annealing is changed by 0,5 mK or more in the temperature equivalent, annealing should be repeated.

If the SPRT resistance at the triple point of water after second annealing is changed by 0,5 mK or more in the temperature equivalent, annealing should be repeated.

The SPRT is calibrated in the following order: TPW, Zn, TPW, Sn, TPW, In, TPW, Ga, TPW.

The practice accepted in the laboratory is used for each fixed point. On the basis of measurement results for each fixed point the relative resistance value of the SPRT is calculated: $W = R_t/R_{TPW}$, where R_{TPW} is the SPRT resistance at the triple point of water obtained directly after measuring R_t. The values of R_t and R_{TPW} shall be corrected for self-heating with measuring current, for hydrostatic pressure and pressure in the cell.

The calibration cycle shown above shall be repeated not less three times.

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

4 Submission of the results

GEOSTM and INSM shall send the following information to the coordinator:

a) Parameters of the cells of the fixed points, furnaces, measuring instruments used by the participating laboratories in the comparisons according to the Appendix B;

b) Measurement results $W = R_t/R_{TPW}$, where R_t is the resistance of the SPRT at each fixed point; R_{TPW} is the SPRT resistance at the triple point of water obtained after measuring R_t. The values of R_t and R_{TPW} shall be corrected for self-heating of measuring current, for hydrostatic pressure and pressure in the cell according to form A of the Protocol;

c) Examples of the experimental freezing curves for the fixed points of In, Sn, Zn and the melting curves for the fixed point of Ga;

d) Experimental curves of dependence of fixed point temperature on depth of immersion of the SPRT applied;

e) Uncertainty budget for each fixed point calculated in accordance with Form B of the Protocol.

Form A

Measurement results						
COOMET						
Laboratory:						
W(t) = R(t)/R(TPW), where R(TPW) is the resistance at the TPW after measuring R(t)						
Correction for pressure in the cell						
Point to be measured:						
Thermometer number:						
Point to be measured:	R measured at 1mA	Overheat	Hydrostatics	Pressure	Corrected R	W
	ohm	ohm	ohm	ohm	ohm	

Average W						

Form B

Uncertainty components	NMI
1. Reproducibility of the W_t values	
2. Component due to metal purity	
3. Component due to correction for hydrostatic pressure	
4. Component due to correction for self-heating	
5. Component due to deviation from thermal equilibrium	
6. Component due to pressure in the cell	
7. Component due to uncertainty of TPW	
8. Component due to nonlinearity of the bridge	
9. Component due to temperature of the reference resistor	
10. Component due to stability of the reference resistor	
Components due to measurement in the TPW	
11. Component due to purity and isotopic composition of water	
12. Component due to correction for hydrostatic pressure	
13. Component due to correction for overheating	
14. Component due to deviation from thermal equilibrium	
15. Component due to a.c. or d.c.	
16. Component due to nonlinearity of the bridge	
Total uncertainty	
Expanded uncertainty	

Explanation and proposals for estimation of the uncertainty components

1. Repeatability of the W_t values. The type A uncertainty component –standard uncertainty of the measured “n” values of W. The number of the degrees of freedom is (n–1).

2. The uncertainty component due to the influence of metal purity on the fixed point temperature value (Type B).

At present, the CCT Working Group 1 has prepared the document on the recommended methods for estimation of this component. The first method called “*Sum of individual estimates* ” (SIE) is more preferable. This method assumes calculation of the correction for the influence of impurities, which should be applied to the measurement result. The correction is calculated by the following formula:

$$T_{\text{liq}} - T_{\text{pure}} = \Sigma_i c_{11,I} \times \partial T/\partial c_{1,i} = \Sigma_i c_{11,I} \times m_{1,i},$$

where T_{liq} is the "liquidus" temperature of the substance used;
T_{pure} is the temperature of the phase transition of pure substance;
$c_{11,i}$ is the concentration (mole fraction) for the i-impurity;
$\partial T/\partial c_{1,i} = m_{1,i}$ is the initial inclination of the liquidus line in the binary phase diagram for the i-impurity.
In this case the uncertainty component due to the influence of purity of metal is the uncertainty of the applied correction. It can be calculated by the following formula:

$$u^2_{\text{sie}} = \Sigma_i[u^2(c_{11,i}) \times m^2_{1,I} + u^2(m_{1,i}) \times c^2_{11,i}],$$

where u is the uncertainty of the parameters used for calculation of the correction.

As is seen from the above ratios, for application of the SIE method it is necessary to know the concentration of all impurities from the certificate to the metal used, the uncertainty of its determination, the inclinations of the "liquidus" curves of the binary phase diagrams for each impurity and their uncertainty.

The second method for estimation of the uncertainty due to the influence of impurities called "*Overall maximum estimate* " (OME) can be applied in those cased where the data on concentration of individual impurities are absent, or the inclinations of the "liquidus" curves are not known. It is assumed in this case that no impurity forms solid solutions, and the following inequality is correct:

$$T_{\text{liq}} - T_{\text{pure}} < \Delta T_{\text{оме,мах}} = c_{11}/A,$$

where $c11$ is the sum of concentrations (mole fraction) of all impurities;
A is the cryoscopic constant.
If we accept that the "liquidus" temperature is within the range $T_{\text{liq}} - T_{\text{pure}}$, then the uncertainty component can be estimated as follows:

$$u^2_{\text{ome}} = (c_{11}/A)^2/3.$$

The third method for estimation of this uncertainty component presents a combination of the SIE and OME methods. It is allowed to apply the SIE method for impurities with the prevailing influence and the OME method for the other impurities.

3. Uncertainty of the correction for hydrostatic pressure (Type B)
The correction for hydrostatic pressure, which is applied to the measurement result, is equal to the product of the immersion depth of the sensing element middle of the thermometer relative to the metal surface in the cell "h" and the hydrostatic pressure ratio dT/dh for the given metal presented in the ITS-90. The uncertainty of the given correction is determined as follows:

$$u^2_{\text{h}} = (dT/dh)^2 \times u^2(h),$$

where $u^2(h)$ is the uncertainty of the estimation of the immersion depth determination h.
If $u(h)$ is the maximum uncertainty estimation of the immersion depth determination h, the standard deviation of the given uncertainty component is $u^2_{\mathrm{h}}/3$.

4. Uncertainty component due to pressure in the cell
For open cells this component is determined by the uncertainty of gas pressure measurement above the metal and its maintenance in correspondence with the ITS-90 equal to 103 Pa, and is estimated as follows:

$$u^2_{\mathrm{p}} = (dT/dp)^2 \times u^2(p),$$

where dT/dp is the ratio presented in the ITS-90, and $u(p)$ is the uncertainty of measurement and maintenance of pressure in the period of phase transition.
If u(p) is the maximum uncertainty, the standard deviation of the given uncertainty component is $u^2_{\mathrm{p}}/3$
For closed cells it is necessary to know which pressure value is in the cell in the period of phase transition. This pressure value is determined by pressure and temperature at the moment of sealing the cell. If it differs from 103 Pa, it must be specified in the certificate to the cell for calculation of the corresponding correction for pressure.
The uncertainty component is estimated on the basis of the pressure measurement uncertainty at the moment of sealing the cell.

5. The uncertainty component of the correction for self-heating with measuring current
In the existing practice the correction for self-heating is calculated on the basis of assumption of linear dependence of self-heating on measuring current power generated in the thermometer. This assumption leads to calculation of the correction as follows:
$\Delta R = 2R(1) - R(\sqrt{2})$,
where $R(1)$ is the thermometer resistance with the measuring current 1 mA, and $R(\sqrt{2})$ is its resistance with the current $\sqrt{2}$ mA.
The uncertainty of the correction depends on validity of the assumption of the linear dependence of self-heating on power for the given thermometer type, on uncertainty of measuring current values, and on resolution of a bridge used in measurements. The simplest way to estimate the uncertainty of the correction is estimation of the standard deviation of the obtained series of values ΔRi.

6. The uncertainty component due to deviation of thermal equilibrium between the thermometer and the interface from ideal equilibrium.
This uncertainty component appears due to heat dissipation along the thermometer, due to thermal resistance of the walls and gaps between the

thermometer and the metal interface. The presence of this uncertainty component is confirmed by deviation of the temperature dependence on the immersion depth of the thermometer from the expected hydrostatic dependence, variations of the thermometer indications when the heat exchange conditions between the thermometer and the crucible of the furnace elements are changed. The uncertainty component can be received from the results of the experimental studies carried out in the laboratory.

The uncertainty due to immersion $u(\Delta T_{thm})$ preferably can be estimated from the residual departures the experimental immersion curve from the expected hydrostatic-pressure line using the formula:

$$u^2(\Delta T_{thm}) = \frac{1}{N-1}\sum_i \left[T(h_i) - T(h_o) - \frac{dT}{dh}(h_i - h_o) \right]^2,$$

where $[T(h_i) - T(h_0)]$ – change of temperature corresponding to the experimental curve at a displacement from h_0 to h_i, dT/dh –coefficient from ITS-90 (CCT/08-19/rev.)

7. The uncertainty component due to non-linearity of the bridge

The estimation of the uncertainty component can be obtained from the parameter of the bridge used, and from the comparison of its indications with other bridges.

8. The uncertainty component due to direct or alternating measuring current

The estimation of the uncertainty component can be obtained by comparison of the results of measurement with d.c. and a.c. bridges.

9. The uncertainty component due to variation of reference resistance temperature

The estimation of the uncertainty component can be calculated on the basis of estimation of measured temperature instability of the reference resistor (temperature instability of the thermostat for the reference resistor), its temperature coefficient and uncertainty of the measurement results of the reference resistor temperature:

$$u^2_{sr} = u^2_t + u^2_n,$$

где u^2_t is the standard deviation of the temperature measurements of the reference resistor;

u^2_n is the standard deviation of the reference resistor due to temperature instability.

$$u^2_n = u^2_{\delta T} \times N^2 \times (\partial T/\partial R)^2,$$

where $u_{\delta T}$ is the standard deviation of the temperature during measurement of the reference resistor; N is the temperature coefficient of the reference resistor; $(\partial T/\partial R)$ is the thermometer sensitivity.

10. The uncertainty component due to temporary variation of resistance

This component is insignificant if measurements are taken during a short period of time.

Appendix B

Parameters of the cells of fixed points, furnaces, Instruments

Laboratory	NISM	VNIIM
Bridge, potentiometer	Resistance Thermometry Bridge	Bridge
Manufacturer		
Type	6010C	F 900
	No.1102102	serial No.009340/01
A.C. or D.C.	direct	A.C.
A.C. frequency		75 Hz
D.C. period	10 s	
Normal measurement current	1 mA	1 mA
Self-heating current	1.414 mA	1.414 mA
Resistance linearity		
Bridge (yes/no)		
Reference resistor	100 Ω	100 Ω
Manufacturer	Tinsley	ZIP, RF
Type	5685 A, No.13946/15	MC3020, No. 053
Temperature control of reference resistor (yes/no)	yes	yes
TPW cell		
Manufacturer	VNIIM, No.0/30	VNIIM, No.0/41
Outer diameter	50 mm	50 mm
Thermometer well diameter	11mm	11 mm
Immersion depth of the middle of the SPRT sensing element	260 mm	257 mm
Thermostat for maintaining the TPW	Thermostat Hart Scientific, model 7312	Thermostat Hart Scientific, model 7312
Zn fixed point cell	Модель 5906, No.06074	No.12
Manufacturer	Hart Scientific	VNIIM
Open or closed	closed	open,
Outer diameter(crucible container)	48 mm	52 mm
Thermometer well diameter	8 mm	12 mm
Metal purity	99,9999%	99.9999 %
Immersion depth of the middle of the SPRT sensing element	195 mm	175 mm
Furnace for the fixed point of Zn		
Manufacturer	Hart Scientific	VNIIM

Type (1, 2 or 3 zones)	3 zones	3 zones
Freezing plateau duration	9 hours	10 hours
Sn fixed point cell	Модель 5905, No.05071	No.88
Manufacturer	Hart Scientific	VNIIM
Open or closed	closed	open,
Outer diameter (crucible container)	48 mm	52 mm
Thermometer well diameter	8 mm	12 mm
Metal purity	99,9999%	99,9999%
Immersion depth of the middle of the SPRT sensing element	195 mm	190 mm
Furnace for the fixed point of Sn		
Manufacturer	Hart Scientific	VNIIM
Type (1, 2 or 3 zones)	3 zones	3 zones
Freezing plateau duration	9 hours	9 hours
In fixed point cell	Модель 5904, No.04034	No.1
Manufacturer	Hart Scientific	VNIIM
Open or closed	closed	open,
Outer diameter	48 mm	43 mm
Thermometer well diameter	8 mm	8 mm
Metal purity	99,9999%	99,9999%
Immersion depth of the middle of the SPRT sensing element	195 mm	182 mm
Furnace for the fixed point of In		
Manufacturer	Hart Scientific	VNIIM
Type (1, 2 or 3 zones)	3 zones	3 zones
Freezing plateau duration	12 hours	7 hours
Ga fixed point cell	Модель 5943, No.43087	No.1
Manufacturer	Hart Scientific	VNIIM
Open or closed	closed	open,
Outer diameter	38,1 mm	36 mm
Thermometer well diameter	8,2 mm	8 mm
Metal purity	99,99999%	99.99999 %
Immersion depth of the middle of the SPRT sensing element	168 mm	125 mm
Furnace for the fixed point of Ga	модель 7312	
Manufacturer	Termostat Hart Scientific	VNIIM
Type (1, 2 or 3 zones)		2 zones
Melting plateau duration	17 hours	17 hours

Parameters of the cells of fixed points, furnaces, measuring instruments applied by GEOSTM

Potentiometer	
Manufacturer	ZIP, RF
Model	P - 3003
Reference resistor	
Manufacturer	ZIP, RF
Model	P - 3030
Temperature control of reference resistor (yes/no)	yes
TPW cell	No.13
Manufacturer	VNIIM
Outer diameter	50 mm
Thermometer well diameter	12 mm
Immersion depth of the middle of the SPRT sensing element	250 mm
Thermostat for TPW	Ice thermostat
Sn fixed point cell	
Manufacturer	-
Open or closed	closed
Outer diameter	70 mm
Thermometer well diameter	9 mm
Metal purity	6N
Immersion depth of the middle of the SPRT sensing element	175
Furnace for the fixed point of Sn	
Manufacturer	-
Type (1, 2 or 3 zones)	3 zones
Melting plateau duration	
Freezing plateau duration	5 hours

Appendix C

Uncertainty budgets

Uncertainty budget for the melting point of Ga

Uncertainty components	NISM mK	VNIIM, mK
Repeatability of the W_t values	0.089	0.045
Component linked with purity	0.047	0.055
Component linked with the hydrostatic pressure correction	0.007	0.003
Component linked with self-heating correction	0.04	0.010
Component due to immersion effect	0.03	0.015
Component linked with pressure in the cell	0.003	0.0002
Component due to propagated of TPW	0.156	0.038
Component due to nonlinearity of the bridge	0.06	0.035
Component due to a.c. or d.c.	-	not measured
Component due to temperature of the reference resistor	0.01	0.0001
Component due to stability of the reference resistor	-	negligible
Components of measurements in the TPW		
Component linked with purity, isotopic composition of water	0.11	0.010
Component linked with the hydrostatic pressure correction	0.004	0.001
Component linked with self-heating correction	0.014	0.006
Component due to immersion effect	0.06	0.010
Component due to a.c. or d.c.	-	not measured
Component due to nonlinearity of the bridge	0.06	0.030
Total uncertainty (k=1)	**0.203**	**0.090**
Expanded uncertainty (k=2)	**0.406**	**0.180**

Uncertainty Budget for the Freezing Point of Indium

Uncertainty components	NISM, mK	VNIIM, mK
Repeatability of the W_t values	0.493	0.130
Component linked with purity	0.162	0.290
Component linked with the hydrostatic pressure correction	0.019	0.005
Component linked with self-heating correction	0.05	0.012
Component due to immersion effect	0.09	0.035
Component due to pressure in the cell	0.07	0.0006
Component due to propagated of TPW	0.22	0.084
Component due to nonlinearity of the bridge	0.067	0.120
Component due to a.c. or d.c.	-	not measured
Component due to temperature of the reference resistor	0.01	0.0001
Component due to stability of the reference resistor	-	negligible
Components due to measurement in the TPW		
Component linked with purity, isotopic composition of water	0.11	0.010
Component linked with the hydrostatic pressure correction	0.004	0.001
Component linked with self-heating correction	0.005	0.006
Component due to immersion effect	0.05	0.010
Component due to a.c. or d.c.	-	not measured
Component due to nonlinearity of the bridge	0.067	0.05
Total uncertainty (k=1)	**0.581**	**0.372**
Expanded uncertainty (k=2)	**1.162**	**0.744**

Uncertainty Budget for the Freezing Point of Tin

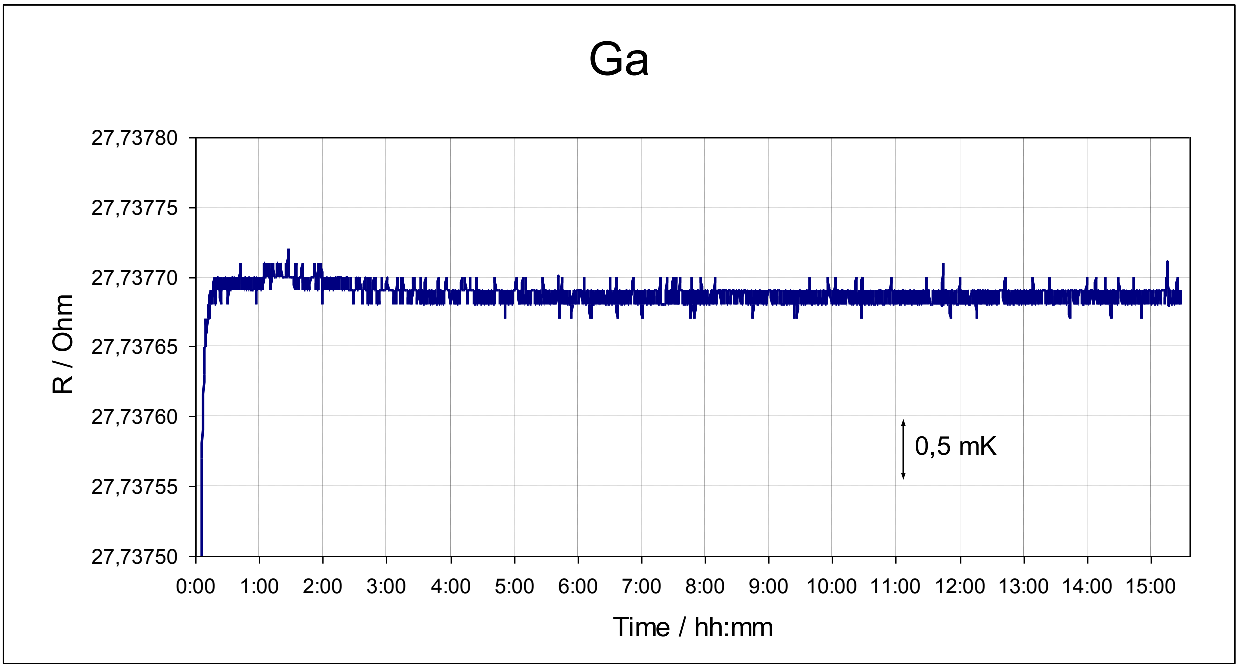
Uncertainty components	**NISM, mK**	**VNIIM mK**	**GEOSTM mK**
Repeatability of the W_t values	0.194	0.230	0.134
Component linked with purity	0.102	0.27	0.52
Component linked with the hydrostatic pressure correction	0.014	0.003	0.013
Component linked with self-heating correction	0.04	0.015	0.03
Component due to immersion effect	0.05	0.033	0.05
Component due to pressure in the cell	0.03	0.0006	0.3
Component due to propagated of TPW	0.327	0.099	1.084
Component due to nonlinearity of the bridge	0.06	0.120	0.572
Component due to a.c. or d.c.	-	not measured	-
Component due to temperature of the reference resistor	0.04	0.0001	0.045
Component due to stability of the reference resistor	negligible	negligible	0.005
Components due to measurement in the TPW			
Component linked with purity, isotopic composition of water	0.11	0.010	0.02
Component linked with the hydrostatic pressure correction	0.004	0.001	0.004
Component linked with self-heating correction	0.055	0.006	0.004
Component due to immersion effect	0.1	0.010	0.02
Component due to a.c. or d.c.	-	not measured	not measured
Component due to nonlinearity of the bridge	0.068	0.050	0.572
Total uncertainty (k=1)	**0.407**	**0.389**	**1.304**
Expanded uncertainty (k=2)	**0.814**	**0.778**	**2.608**

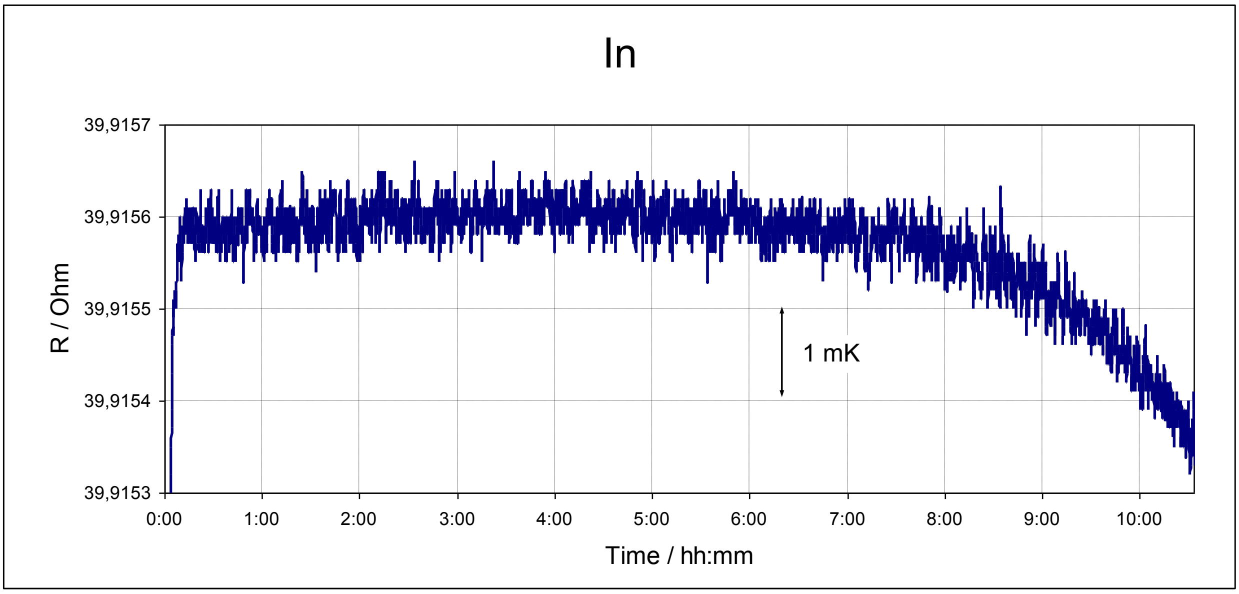
Uncertainty Budget for the Freezing Point of Zinc

Uncertainty components	**NISM, mK**	**VNIIM mK**
Repeatability of the W_t values	0.278	0.180
Component linked with purity	0.196	0.44
Component linked with the hydrostatic pressure correction	0.016	0.010
Component linked with self-heating correction	0.295	0.025
Component due to immersion effect	0.22	0.090
Component due to pressure in the cell	0.02	0.0012
Component due to propagated of TPW	0.530	0.184
Component due to nonlinearity of the bridge	0.07	0.150
Component due to a.c. or d.c.	-	not measured
Component due to temperature of the reference resistor	0.03	0.0001
Component due to stability of the reference resistor	-	negligible
Components due to measurement in the TPW		
Component linked with purity, isotopic composition of water	0.12	0.010
Component linked with the hydrostatic pressure correction	0.004	0.001
Component linked with self-heating correction	0.058	0.006
Component due to immersion effect	0.1	0.010
Component due to a.c. or d.c.	-	not measured
Component due to nonlinearity of the bridge	0.07	0.07
Total uncertainty (k=1)	**0.742**	**0.540**
Expanded uncertainty (k=2)	**1.484**	**1.080**

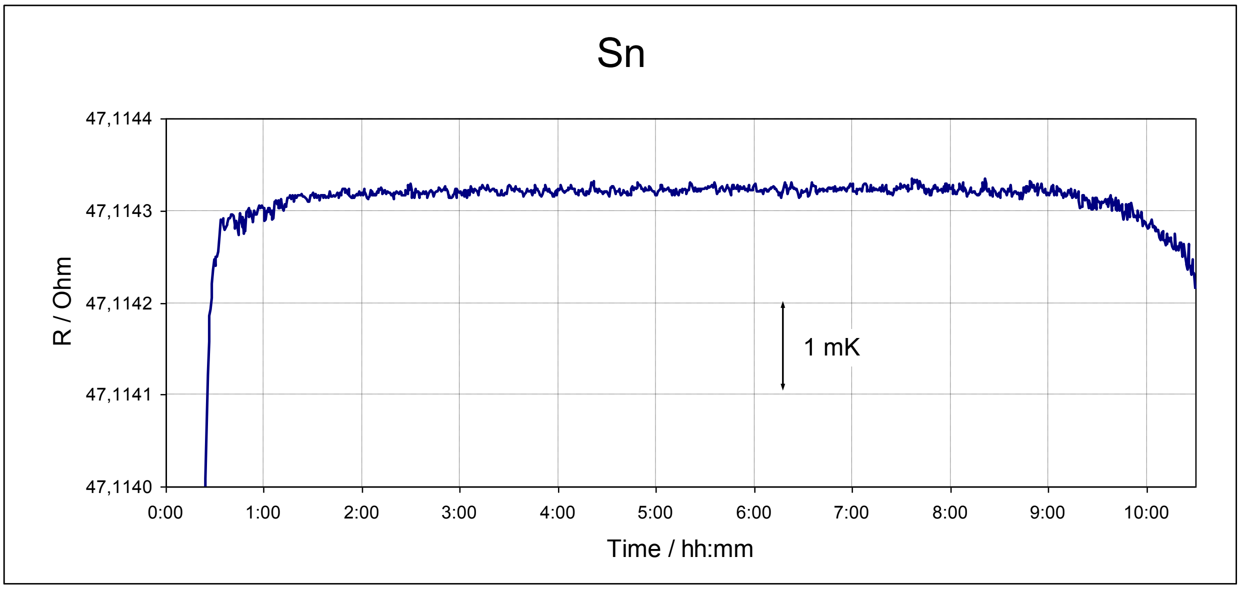
Appendix D. Immersion profiles and plateaus of the fixed points

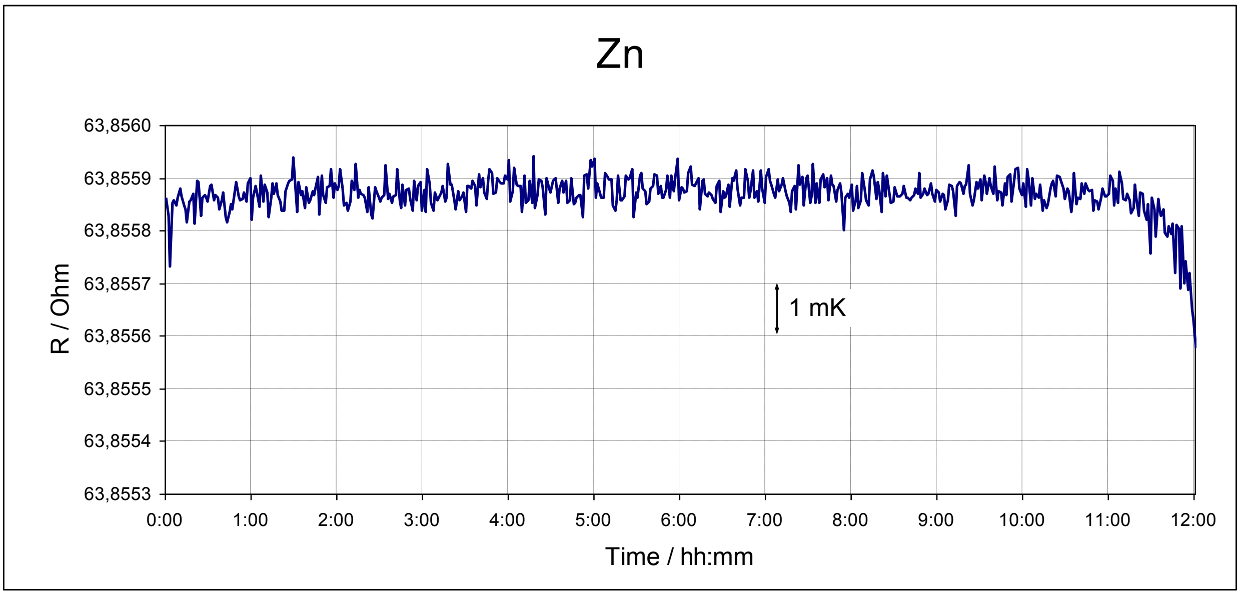
VNIIM



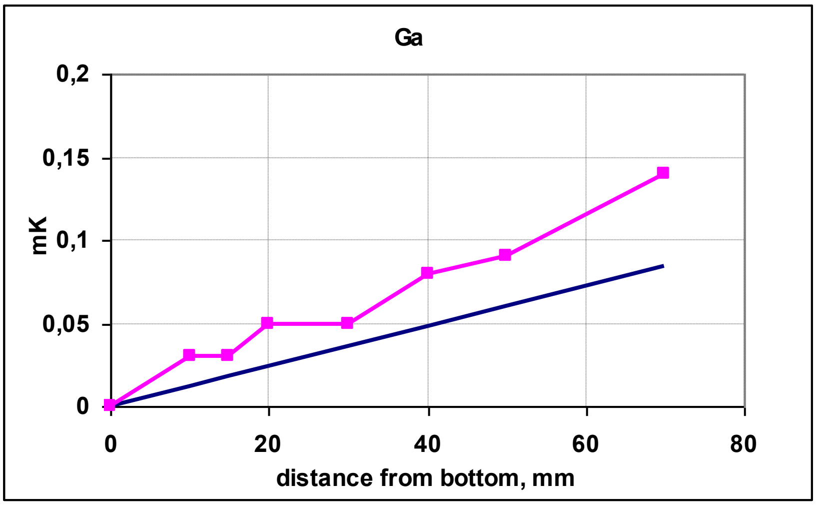


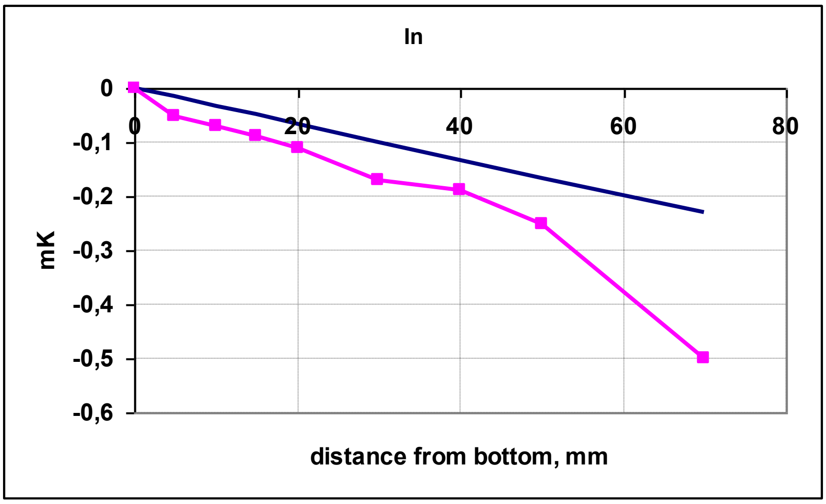
VNIIM

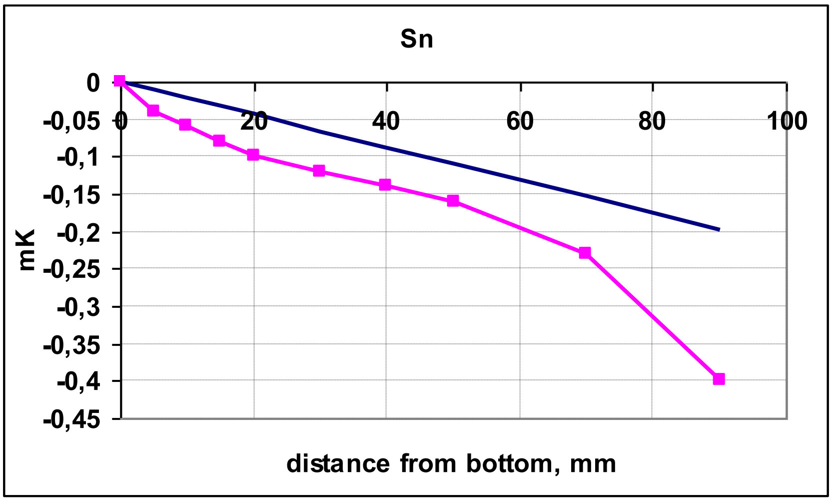

Sn
47,1144
47,1143
47,1142
47,1141
47,1140
R / Ohm
1 mK
0:00
1:00
2:00
3:00
4:00
5:00
6:00
7:00
8:00
9:00
10:00
Time / hh:mm

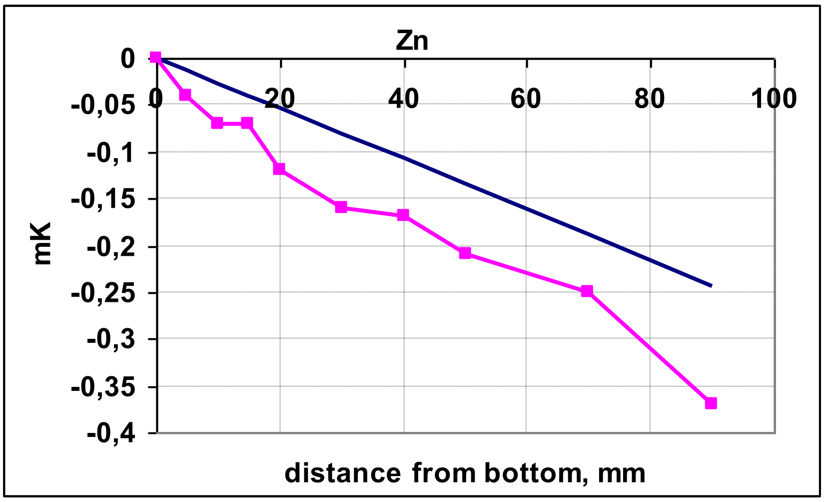

Zn
63,8560
63,8559
63,8558
63,8557
63,8556
63,8555
63,8554
63,8553
R / Ohm
1 mK
0:00
1:00
2:00
3:00
4:00
5:00
6:00
7:00
8:00
9:00
10:00
11:00
12:00
Time / hh:mm

VNIIM

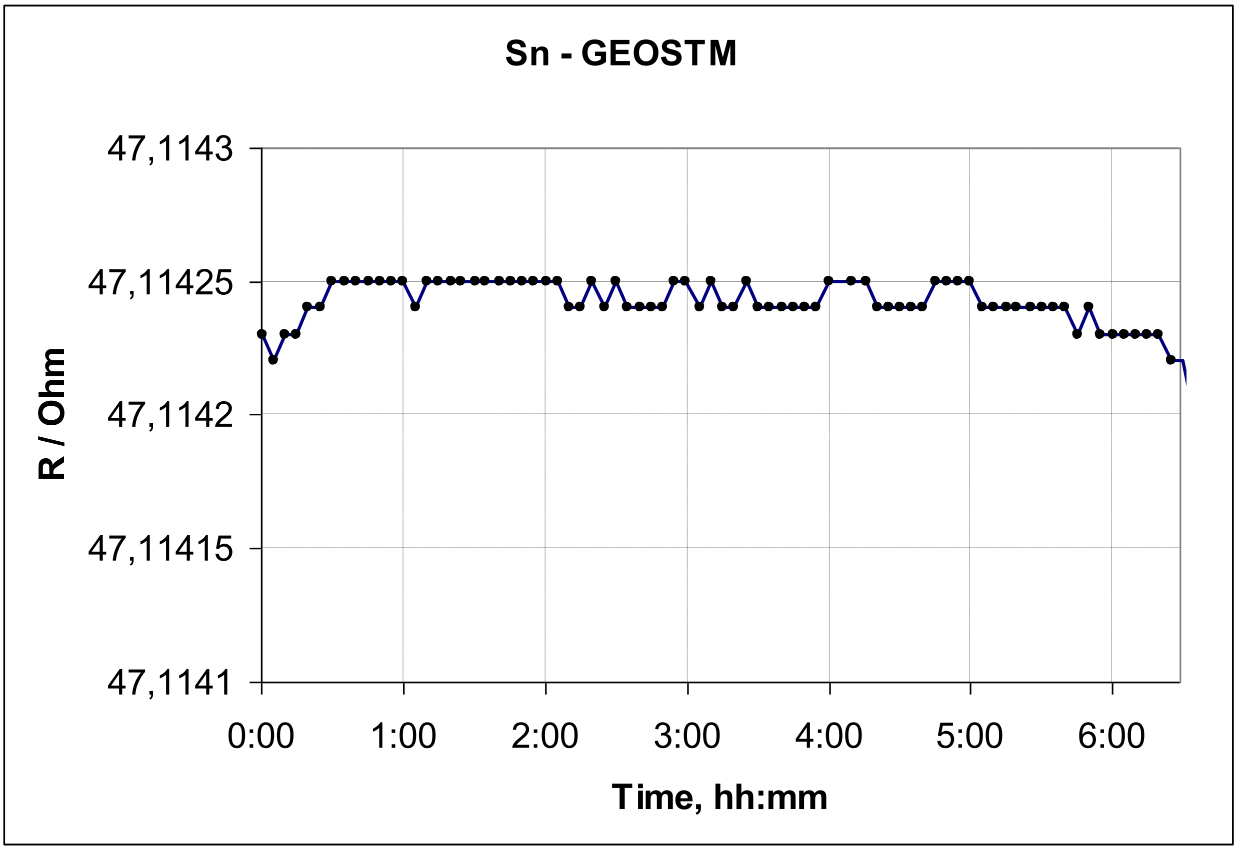

Ga
0,2
0,15
0,1
0,05
0
mK
0
20
40
60
80
distance from bottom, mm


In
0
-0,1
-0,2
-0,3
-0,4
-0,5
-0,6
mK
0
20
40
60
80
distance from bottom, mm

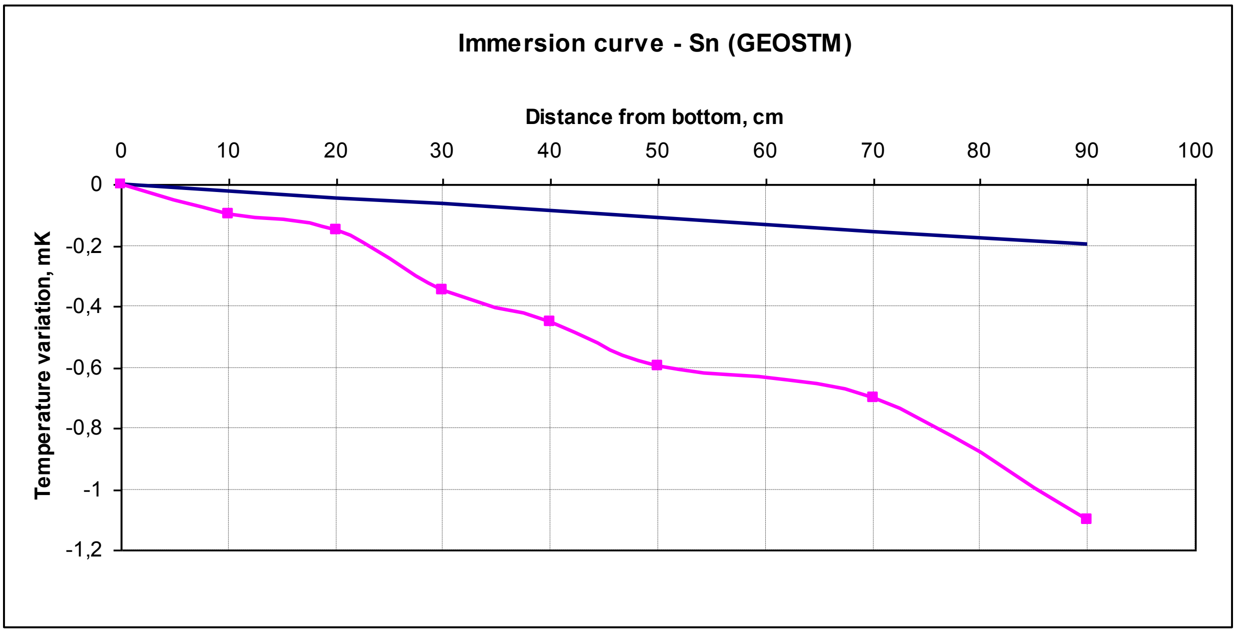

Sn
0
-0,05
-0,1
-0,15
-0,2
-0,25
-0,3
-0,35
-0,4
-0,45
mK
0
20
40
60
80
100
distance from bottom, mm


Zn
mK
0
-0,05
-0,1
-0,15
-0,2
-0,25
-0,3
-0,35
-0,4
0
20
40
60
80
100
distance from bottom, mm

GEOSTM

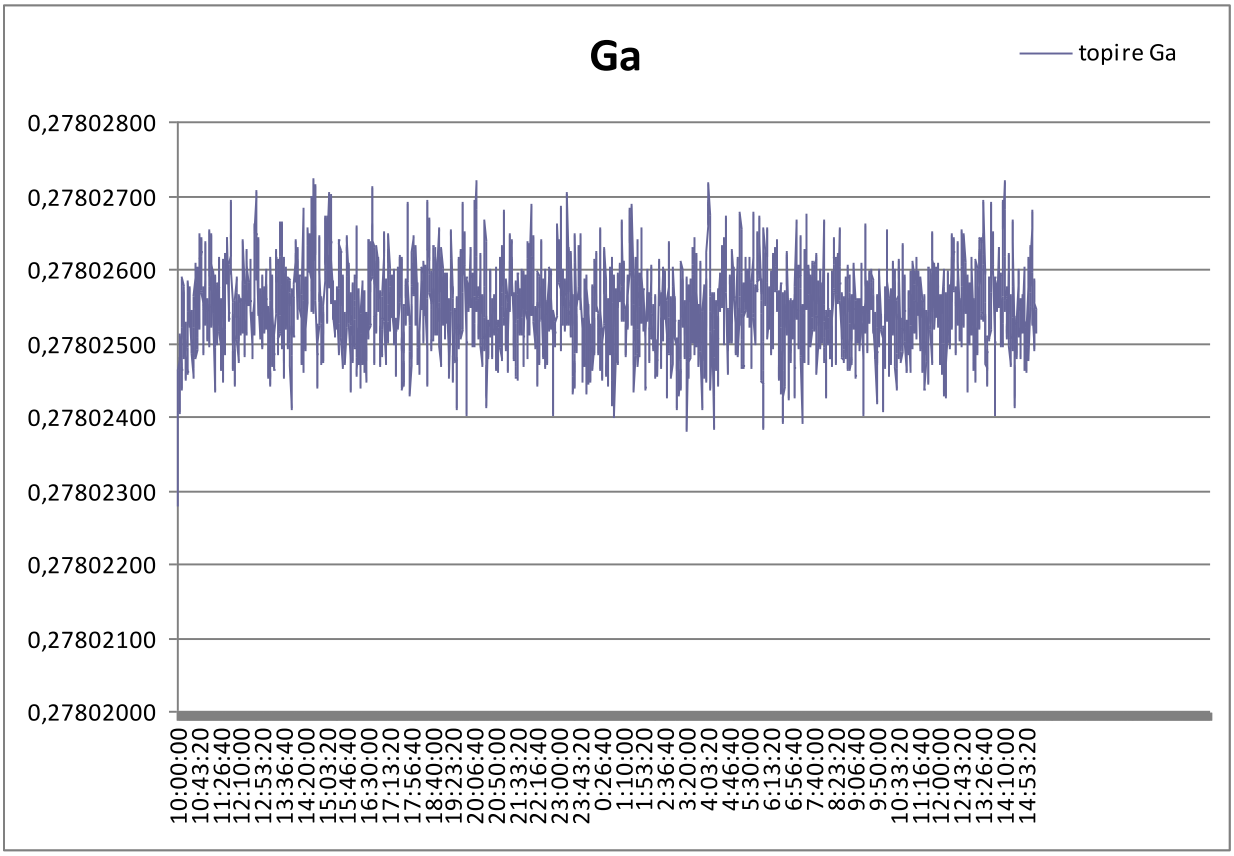


Sn - GEOSTM
47,1143
47,11425
47,1142
47,11415
47,1141
R / Ohm
0:00
1:00
2:00
3:00
4:00
5:00
6:00
Time, hh:mm

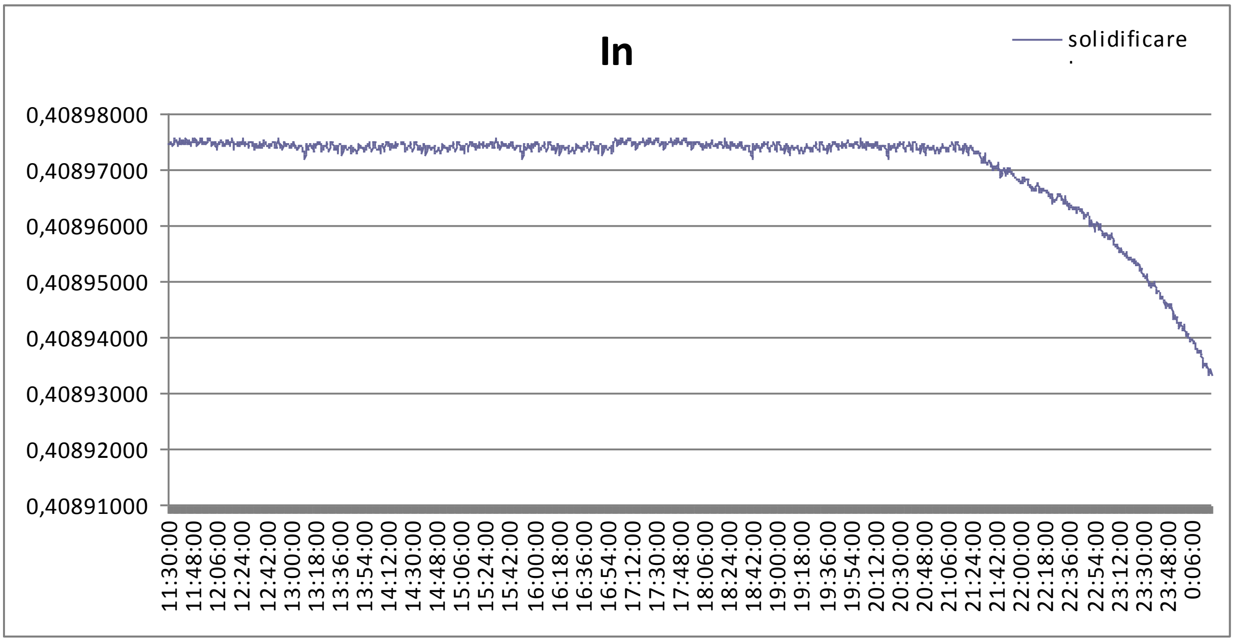


Immersion curve - Sn (GEOSTM)
Distance from bottom, cm
0
10
20
30
40
50
60
70
80
90
100
0
-0,2
-0,4
-0,6
-0,8
-1
-1,2
Temperature variation, mK

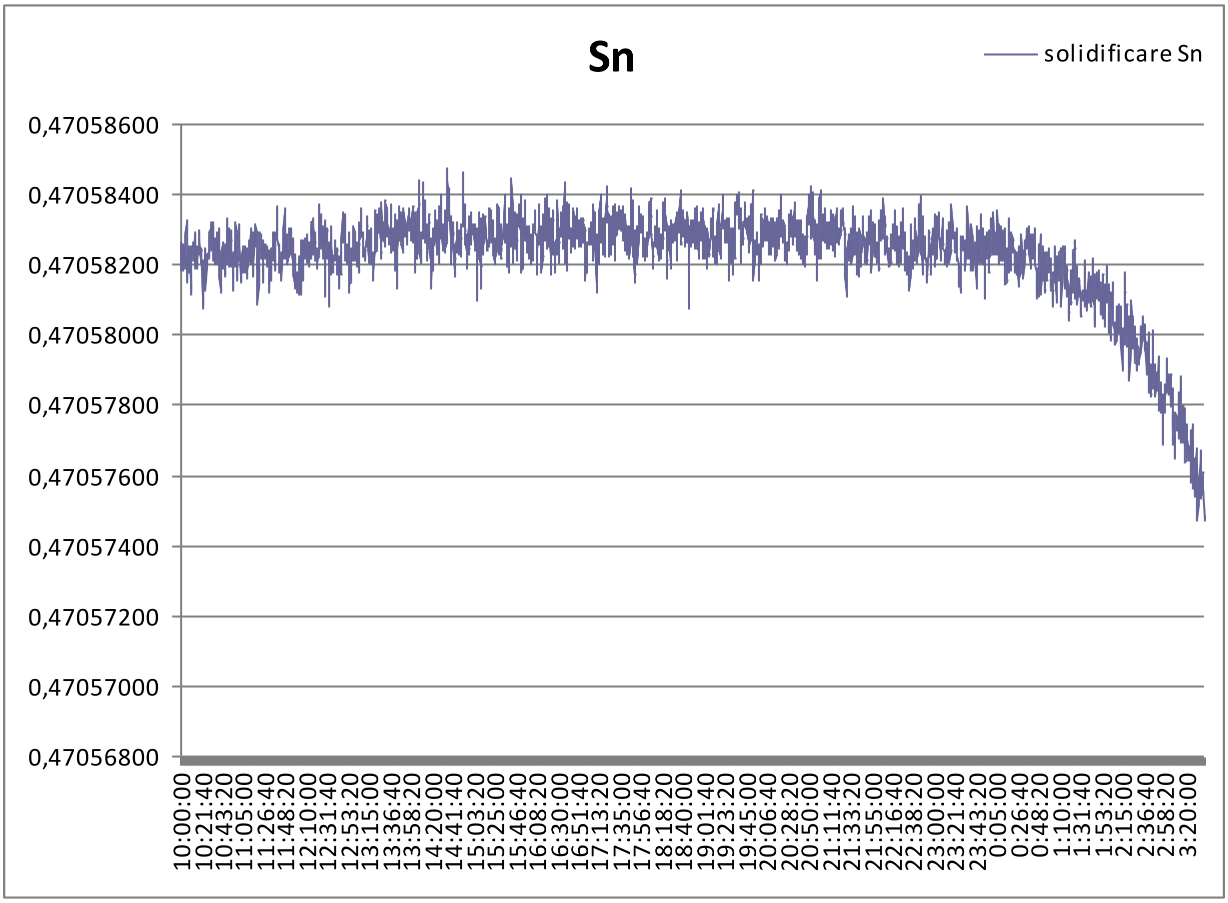
NISM

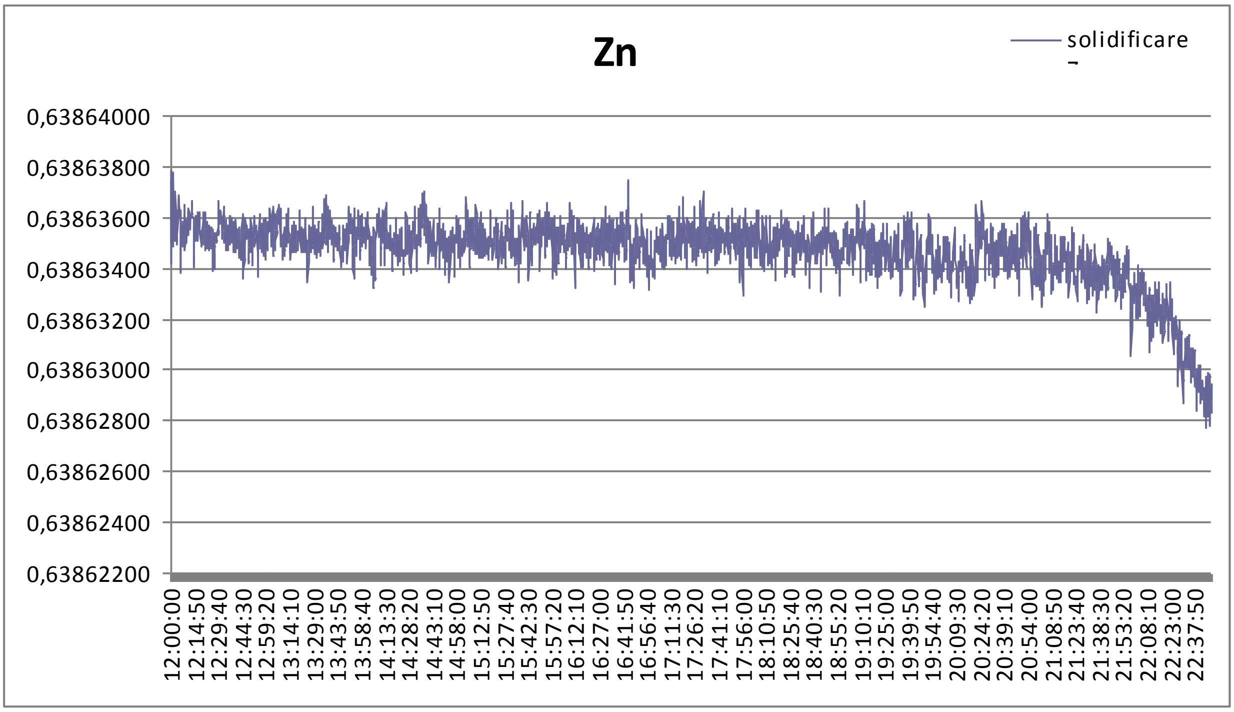


Ga
topire Ga
0,27802800
0,27802700
0,27802600
0,27802500
0,27802400
0,27802300
0,27802200
0,27802100
0,27802000
10:00:00
10:43:20
11:26:40
12:10:00
12:53:20
13:36:40
14:20:00
15:03:20
15:46:40
16:30:00
17:13:20
17:56:40
18:40:00
19:23:20
20:06:40
20:50:00
21:33:20
22:16:40
23:00:00
23:43:20
0:26:40
1:10:00
1:53:20
2:36:40
3:20:00
4:03:20
4:46:40
5:30:00
6:13:20
6:56:40
7:40:00
8:23:20
9:06:40
9:50:00
10:33:20
11:16:40
12:00:00
12:43:20
13:26:40
14:10:00
14:53:20

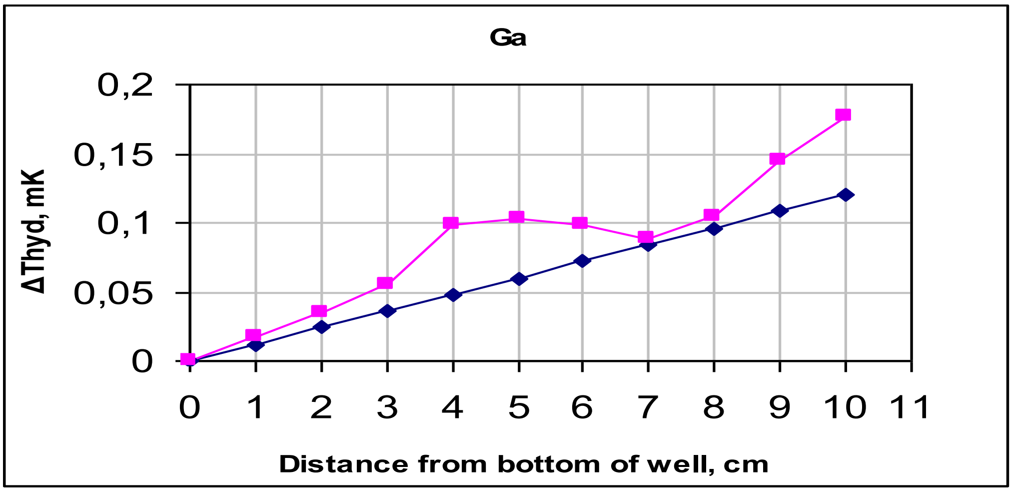


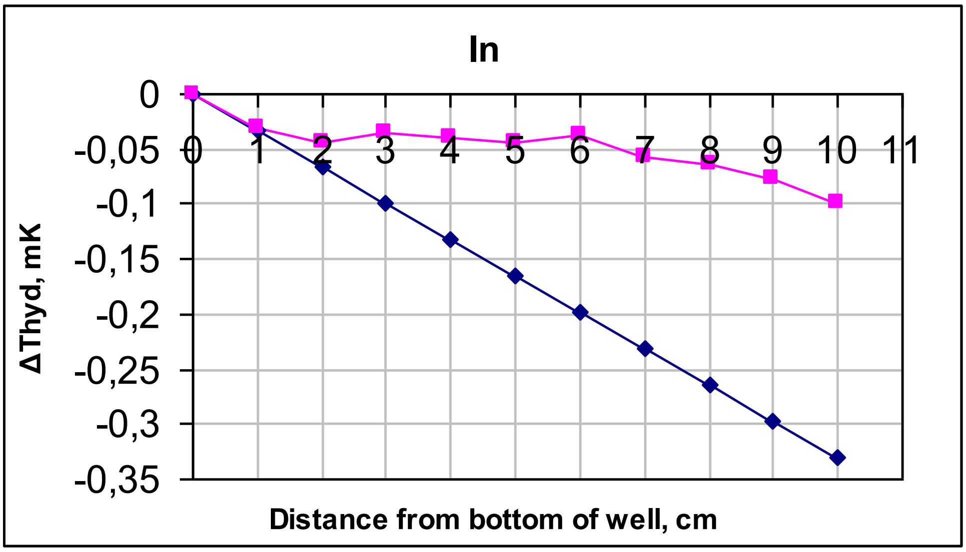
In
solidificare
0,40898000
0,40897000
0,40896000
0,40895000
0,40894000
0,40893000
0,40892000
0,40891000
11:30:00
11:48:00
12:06:00
12:24:00
12:42:00
13:00:00
13:18:00
13:36:00
13:54:00
14:12:00
14:30:00
14:48:00
15:06:00
15:24:00
15:42:00
16:00:00
16:18:00
16:36:00
16:54:00
17:12:00
17:30:00
17:48:00
18:06:00
18:24:00
18:42:00
19:00:00
19:18:00
19:36:00
19:54:00
20:12:00
20:30:00
20:48:00
21:06:00
21:24:00
21:42:00
22:00:00
22:18:00
22:36:00
22:54:00
23:12:00
23:30:00
23:48:00
0:06:00

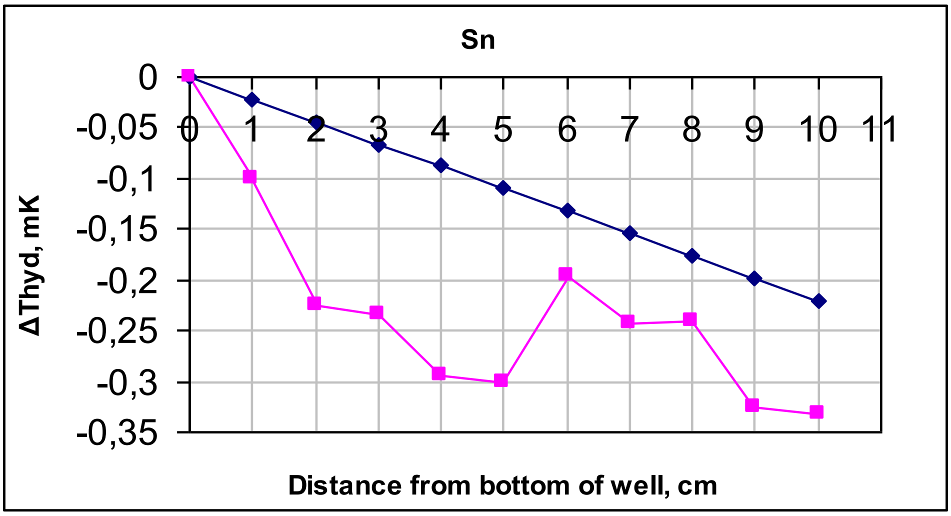

Sn
solidificare Sn
0,47058600
0,47058400
0,47058200
0,47058000
0,47057800
0,47057600
0,47057400
0,47057200
0,47057000
0,47056800


Zn
solidificare
0,63864000
0,63863800
0,63863600
0,63863400
0,63863200
0,63863000
0,63862800
0,63862600
0,63862400
0,63862200

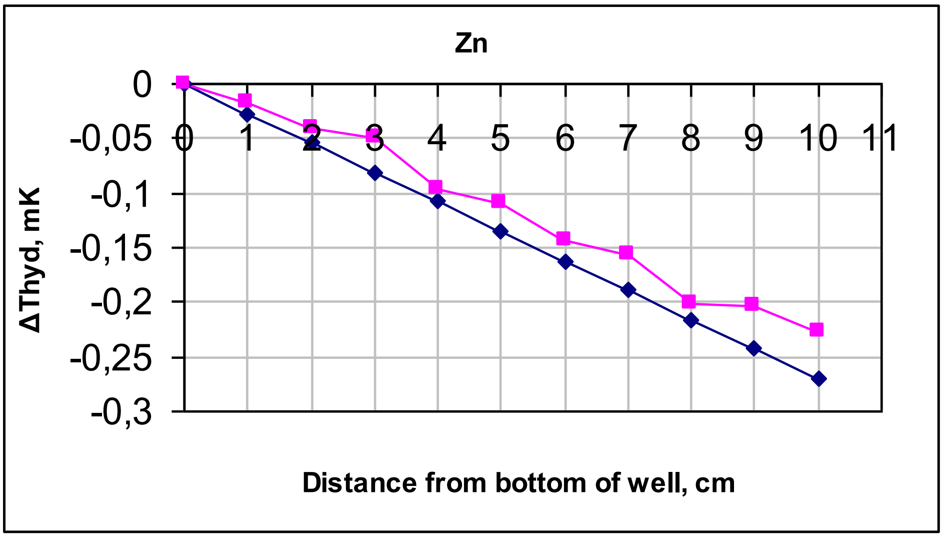
NISM


Ga
0,2
0,15
0,1
0,05
0
ΔThyd, mK
0 1 2 3 4 5 6 7 8 9 10 11
Distance from bottom of well, cm


In
0
-0,05
-0,1
-0,15
-0,2
-0,25
-0,3
-0,35
ΔThyd, mK
0 1 2 3 4 5 6 7 8 9 10 11
Distance from bottom of well, cm



Sn
ΔThyd, mK
0
-0,05
-0,1
-0,15
-0,2
-0,25
-0,3
-0,35
0 1 2 3 4 5 6 7 8 9 10 11
Distance from bottom of well, cm



Zn
ΔThyd, mK
0
-0,05
-0,1
-0,15
-0,2
-0,25
-0,3
0 1 2 3 4 5 6 7 8 9 10 11
Distance from bottom of well, cm