



## Traceability of temperature measurements in Estonia

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**Abstract.** This paper presents an overview of the establishment of the national measurement standard for temperature at METROSERT – the national metrology institute in Estonia. The temperature scale and traceability arrangements are realized in the temperature range from  $-40^{\circ}\text{C}$  to  $+300^{\circ}\text{C}$  by means of a group of calibrated standard platinum resistance thermometers and water triple point cells. The linkage of the realized water triple point temperature to the international key comparison value with the expanded uncertainty of  $187\text{ }\mu\text{K}$  (coverage factor  $k = 2$ ) is demonstrated. The established laboratory is capable of providing calibration services to customers by comparison calibration with the expanded uncertainty ( $k = 2$ ) values of  $5\text{ mK}$  in the range from  $-40^{\circ}\text{C}$  to  $+200^{\circ}\text{C}$  and  $12\text{ mK}$  in the range from  $+200^{\circ}\text{C}$  to  $+300^{\circ}\text{C}$ .

**Key words:** temperature scale, traceability, uncertainty.

### 1. INTRODUCTION

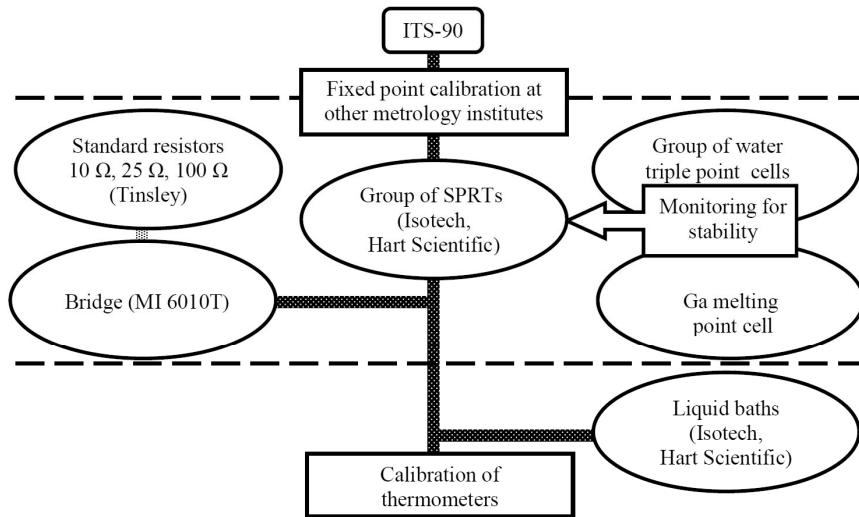
Social advantages and economic competitiveness in modern society are supported by the effective national measurement system which is a generally recognized instrument in providing reliable measurement results traceable to the units of SI [1]. The traceable measurement results are important in industry (e.g. fuel combustion technologies), consumer care (food production and inspection), emerging technologies (biotechnology and nanotechnology enterprises), etc. [2]. In general, most of the European countries have developed their national measurement systems with the history dating back to more than a hundred years. Nevertheless, new independent countries with evolving economy and needs for elaboration of the metrology infrastructure are still emerging. This is also the case for Estonia, where the development of the new national measurement system was initiated only in the early 1990s. The establishment of a new national measurement standard is a long-aimed process that must be supported by good planning for optimal use of resources. The development of the national standard for temperature in Estonia was

started with the survey initiated by the Ministry of Economic Affairs in 2002 [3]. The survey gathered the needs for the calibration services of the local leading industrial enterprises and scientific institutions. As a result of this survey, the initial task for the establishment of the national standard for temperature in the range from  $-40^{\circ}\text{C}$  to  $+300^{\circ}\text{C}$  with an approximate uncertainty level of  $10\text{ mK}$  was raised. The present paper describes on the example of METROSERT – the national laboratory for measurement standards in Estonia – how a system of standards for temperature can be established. The traceability arrangements, linkage of the realized water triple point temperature to the international key comparison value, stability issues of standard platinum resistance thermometers (SPRTs), dissemination of the temperature scale, and uncertainty budget are presented in detail.

### 2. ESTABLISHMENT OF THE TEMPERATURE SCALE

The realization of the International Temperature Scale of 1990 (ITS-90) at METROSERT (Fig. 1) is based on a group of calibrated SPRTs (Table 1). Standard thermo-

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**Fig. 1.** Traceability chart for establishment of the temperature scale at METROsert.

**Table 1.** The SPRTs used for the establishment of the temperature scale at METROsert

Manufacturer	Type	Calibration at fixed points	Calibration uncertainty (mK) at fixed points ( $k = 2$ )
Isotech	670, Pt25, quartz sheath	Hg, H <sub>2</sub> O, Ga, Sn, Zn, Al	1–10
Isotech	670, Pt25, quartz sheath	Hg, H <sub>2</sub> O, Ga, Sn, Zn, Al	1–10
Isotech	670, Pt25, quartz sheath	Hg, H <sub>2</sub> O, Ga, Sn, Zn, Al	1–10
Hart Scientific	5699, Pt25, inconel sheath	Hg, H <sub>2</sub> O, Ga, Sn, Zn, Al	1–10
Hart Scientific	5699, Pt25, inconel sheath	Hg, H <sub>2</sub> O, Ga, Sn, Zn, Al	1–10
Hart Scientific	5682, Pt100, inconel sheath	Hg, H <sub>2</sub> O, Ga, Sn, Zn	1–5
Hart Scientific	5682, Pt100, inconel sheath	Hg, H <sub>2</sub> O, Ga, Sn, Zn	1–5
Hart Scientific	5682, Pt100, inconel sheath	Hg, H <sub>2</sub> O, Ga, Sn, Zn	1–5
Hart Scientific	5682, Pt100, inconel sheath	Hg, H <sub>2</sub> O, Ga, Sn, Zn	1–5

meters are regularly calibrated for the temperature range from  $-40^{\circ}\text{C}$  to  $+300^{\circ}\text{C}$  at the national metrology institutes that have published their calibration and measurement capabilities in the key comparison database of the International Bureau of Weights and Measures (BIPM KCDB) [4]. The calibration service is currently obtained from the National Metrology Institute of Finland on the basis of a long-term agreement, also signed as the EURAMET project No. 1001. The stability of SPRTs is periodically checked with the group of water triple point cells. A gallium melting point cell is mainly used for additional stability and consistency tests. The resistance measurements are performed by using an automated thermometry bridge Measurements International type MI6010T and standard resistors (Table 2). The properties of the bridge have been studied thoroughly in the range of the resistance ratios from 0.4 to 4.0. The deviations of the measured ratio values from the reference values were found to remain within  $\pm 0.1 \times 10^{-6}$  [5]. Taking into account additional uncertainty sources in calibration, and the use of the bridge (e.g. linearity of the bridge, use of

multichannel switch and environmental conditions), the total contribution to the uncertainty budget arising from the use of the thermometry bridge with the multiplexer is estimated to be less than 0.7 mK. The standard resistors are immersed into the temperature controlled oil bath Lauda Ecoline E200 in order to reduce the temperature effects on high-accuracy resistance ratios needed in temperature measurements. The temperature in the oil bath is set to the reference temperature at which the resistors have been calibrated, i.e.  $23^{\circ}\text{C}$  with stability of  $\pm 0.02^{\circ}\text{C}$ .

**Table 2.** The standard resistors used in accurate temperature measurements at METROsert

Type	Nominal resistance, $\Omega$	Calibration uncertainty ( $k = 2$ )
Tinsley 5685A	10	$0.4 \times 10^{-6}$
Tinsley 5685A	25	$0.4 \times 10^{-6}$
Tinsley 5685A	100	$0.4 \times 10^{-6}$

### 3. REALIZATION OF THE TRIPLE POINT OF WATER

Three different water triple point cells (Table 3) are used at METROsert to represent the temperature of the triple point of water (TPW) according to the ITS-90 as the national reference. The realization of the TPW is obtained by following the standard procedures [6]. In 2004, Metrosert participated in the EUROMET project No. 714 in order to confirm the equivalence of the realization of the TPW. The project was an extension of the previous EUROMET projects Nos 278 and 549 with the aim to assess the uncertainties associated to the practical realization of the TPW in various European laboratories [7–9]. The EUROMET project No. 714 involved five national metrology institutes: LNE-INM/CNAM (France, the coordinator of the project), METROsert (Estonia), MSA (Malta), LNMC (Latvia), and NSC (Ukraine). Each of these laboratories had to determine and report the temperature difference ( $T_x - T_{679}$ ) between the local cell “X” and the circulating cell LNE-679 with the associated uncertainty  $U(T_x - T_{679})$  according to the prescribed procedures [9]. The results of METROsert are presented in Table 4. In 2008, the pilot of the project (LNE-INM/CNAM) published a link allowing positioning the temperatures realized by the cells that participated in the EUROMET project No. 714 with unknown isotopic composition of water with respect to the SI definition of kelvin in accordance with the International Committee for Weights and Measures (CIPM) Recommendation 2 (CI-2005) [10,11]. The deviation in temperature between the realizations of the TPW with the cells listed

**Table 5.** The estimated deviations of the TPW realization values  $T_x$  at METROsert from the reference value of the international key comparison CCT-K7

Cell No. X	$(T_x - T_{\text{CCT-K7}})$ , $\mu\text{K}$	$U(T_x - T_{\text{CCT-K7}})$ , $\mu\text{K}$
MS 2027	-31	186
MS 320	-56	184
MS 284	-42	187

in Table 3 and the reference of LNE-INM/CNAM was found to be approximately  $-100 \mu\text{K}$  with the expanded uncertainty of  $180 \mu\text{K}$  (coverage factor  $k = 2$ ) [12]. As one of the cells from LNE-INM/CNAM (cell LNE-6) had participated in the international key comparison CCT-K7, it is possible to link the TPW realization values at METROsert to the key comparison reference value. The temperature difference between the cell LNE-6 and the reference value of CCT-K7 ( $T_{\text{LNE-6}} - T_{\text{CCT-K7}}$ ) =  $(-79 \pm 93) \mu\text{K}$  has been published in the final report of CCT-K7 [13]. The temperature differences between the cell LNE-6 and the transfer cell LNE-673 ( $T_{673} - T_{\text{LNE-6}}$ ) =  $(-36 \pm 60) \mu\text{K}$ , and the cells LNE-679 and LNE-673 ( $T_{673} - T_{679}$ ) =  $(28 \pm 20) \mu\text{K}$  have been published by LNE-INM/CNAM [11]. The temperature differences between the cells  $T_x$  at METROsert from the key comparison reference value  $T_{\text{CCT-K7}}$  can be calculated as

$$(T_x - T_{\text{CCT-K7}}) = (T_x - T_{679}) - (T_{673} - T_{679}) + (T_{673} - T_{\text{LNE-6}}) + (T_{\text{LNE-6}} - T_{\text{CCT-K7}}). \quad (1)$$

The results are presented in Table 5.

### 4. STABILITY OF SPRTs

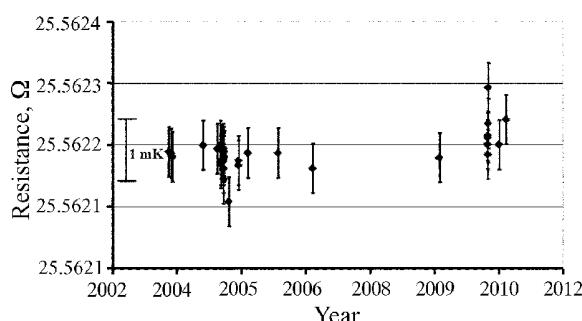
The properties of SPRTs are always affected by several factors even when handled with care [14]. Therefore, the stability of the SPRTs is periodically checked by measurements of the electrical resistance at the temperature of the TPW. As the temperature scale is imported to METROsert by calibration of the SPRTs at other laboratories, the resistance value at the TPW is checked before the SPRTs are shipped for calibration and after the SPRTs have returned from calibration. Comparison of these resistance values gives information about the shipping conditions and allows evaluation of the continuing validity of calibration results. A sample diagram of the stability monitoring of the SPRTs is depicted in Fig. 2. In general, the changes in the resistance values at the TPW are observed to remain within the limits of  $\pm 0.1 \text{ m}\Omega$  during the calibration period of 2 years (i.e.  $1 \text{ mK}$  in terms of temperature for  $25 \Omega$  SPRT).

**Table 3.** The TPW cells used for monitoring the stability of the SPRTs at METROsert

ID	Type	Manu-facturer	Isotopic composition	Year of purchase
MS 2027	Jarret A-11	ISOTECH	Not available	1999
MS 320	ISOTECH B11	ISOTECH	Not available	2003
MS 284	ISOTECH E11	ISOTECH	Not available	2003

**Table 4.** The differences between the TPW realization values  $T_x$  measured at METROsert in the EUROMET comparison project No. 714 and the values  $T_{679}$  realized by the circulating cell LNE-679 with the expanded uncertainties ( $k = 2$ ) [9]

Cell No. X	$(T_x - T_{679})$ , $\mu\text{K}$	$U(T_x - T_{679})$ , $\mu\text{K}$
MS 2027	112	152
MS 320	87	150
MS 284	101	154



**Fig. 2.** The resistance of the SPRT Hart Scientific 5699 No. 0091 at the TPW as a typical sample diagram of the stability monitored. Multiple measurement results in years 2004 and 2009 depict performance of the thermometer due to annealing at 670°C. The vertical bars indicate expanded uncertainty ( $k = 2$ ).

## 5. DISSEMINATION OF THE SCALE

The temperature scale is disseminated to the customers by comparison calibration. The calibration points for the platinum resistance thermometers are selected as the approximate fixed point temperature values according to the defined sub-ranges of ITS-90 with additional temperature values between the fixed point temperatures. For example, the calibration points for the temperature range from 0.01°C to +300°C are selected according to the ITS-90 sub-range from the TPW up to the freezing point of Sn as follows: 0.01°C (TPW), 100°C (additional point), 156°C (freezing point of In), 200°C (additional point), 232°C (freezing point of Sn), 300°C (additional point). The selection of calibration points in this way justifies the use of standard evaluation procedures of ITS-90. The calibration by comparison is performed in the liquid baths (Table 6). The particular parameters of the baths (temperature range, stability and uniformity) have been selected with the aim to achieve the overall expanded calibration uncertainty 0.01°C ( $k = 2$ ) in the temperature range from -40°C to +300°C. The average number of 50 routine comparison calibra-

**Table 6.** The list of liquid baths used for comparison calibrations at METROsert

Liquid bath	Range of use, °C	Medium	Stability*, mK	Uniformity*, mK
Hart Scientific 7341	-40 to 0	Oil	3	3
Hart Scientific 7012	0 to +80	Water	2	2
Isotech 915	+80 to +300	Oil	3	3

\* In equalization block.

tions per year since the official recognition is an indication of a live and well-operational laboratory. The established temperature scale is also a basis for scientific research activities and non-contact temperature measurements [15].

## 6. UNCERTAINTIES

The uncertainty evaluation is based on the *Guide to the Expression of Uncertainty in Measurement* [16]. The uncertainty budget for calibration of platinum resistance thermometers (PRTs) at the TPW is presented in Table 7. The uncertainty estimation for the realization of the TPW temperature 0.01°C takes into account the bias caused by isotopic and other effects according to the results of the EUROMET project No. 714. The uncertainty budget for comparison calibration in a liquid bath at a single calibration temperature within the range from -40°C to +300°C is presented in Table 8. It is assumed that the PRTs under calibration are featuring similar properties as SPRTs: good stability, no hysteresis, and purity of the metal as described in ITS-90 [6].

## 7. CONCLUSIONS

The international temperature scale ITS-90 has been established on the secondary level at the national standard laboratory for temperature in Estonia, METROsert, in the temperature range from -40°C to +300°C. The traceability of the scale is based on the group of SPRTs calibrated regularly by the National Metrology Institute of Finland. The temperature values realized by the TPW cells at METROsert are in good agreement with the definition of kelvin and the reference value of the

**Table 7.** Uncertainty budget for the calibration of PRTs at the TPW 0.01°C

Contributing factor	Standard uncertainty $u_i(y)$ , mK
Fixed point temperature 0.01°C (including isotopic effects)	0.1
Calibration and use of the standard resistors	0.1
Calibration and use of the temperature measurement bridge	0.1
Uncertainty of type A	0.1
Combined uncertainty $u_c$	0.2
Expanded uncertainty $U$ (at confidence level of 95%, coverage factor $k = 2$ )	0.4

**Table 8.** Uncertainty budget for the calibration of PRTs by comparison

Contributing factor	Standard uncertainty $u_i(y)$ , mK	
	(−40 to +200) °C	(+200 to +300) °C
<b>Standards</b>		
Calibration of the standard thermometer	1.0	1.0
Instability of the standard thermometer*	0.4	0.4
Calibration and use of the standard resistors	0.1	0.1
Calibration and use of the temperature measurement bridge	0.7	0.7
<b>Measurement environment</b>		
Temperature difference between the standard and thermometer under calibration	1.0	4.0
Changes in temperature difference between the standard and thermometer under calibration	1.5	4.0
<b>Thermometer under calibration</b>		
Calibration and use of the standard resistors	0.1	0.1
Calibration and use of the temperature measurement bridge	0.7	0.7
Measurement of the resistance value at 0.01 °C	1.0	1.0
Combined uncertainty $u_c$	2.5	5.9
Expanded uncertainty $U$ (at confidence level of 95%, coverage factor $k = 2$ )	5	12

\* Instability of the standard thermometer is estimated by the regular checks at the TPW. In calibrations, the latest resistance value  $R(0.01\text{ }^\circ\text{C})$  for SPRTs is used.

international key comparison CCT-K7 within the estimated measurement uncertainties below 0.19 mK for all three cells. The temperature scale is disseminated to customers by comparison calibrations at selected temperatures with expanded uncertainty ( $k = 2$ ) values of

5 mK in the range from −40 °C to +200 °C and 12 mK in the range from +200 °C to +300 °C. The national standard laboratory for temperature has proved itself as a functional institution, providing customers routinely with about 50 comparison calibrations per year in Estonia.

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## Temperatuurimõõtmiste jälgitavus Eestis

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Täppismõõtmiste olulisus paljudes valdkondades (tööstus, teadus- ja arendustöö, tervisekaitse jne) seab kõrgendatud nõuded ka temperatuurimõõtmiste usaldusväärsusele. Täpsete temperatuurimõõtmiste kindlustamiseks Eestis on Eesti metrooloogia keskusasutus AS Metrosert välja arendanud temperatuuri riigietalonilabori, mis tagab temperatuurimõõtevahendite kalibreerimise mõõtepiirkonnas  $-40^{\circ}\text{C} \dots +200^{\circ}\text{C}$  laiendmõõtemääramatusega alla 5 mK (kattetegur  $k = 2$ ), ja piirkonnas  $+200^{\circ}\text{C} \dots +300^{\circ}\text{C}$  laiendmõõtemääramatusega alla 12 mK ( $k = 2$ ). Eesti temperatuuri riigietalon põhineb rahvusvahelise temperatuuriskaala ITS-90 kinnispunktides kalibreeritud etalontakistustermomeetritel, mille stabiilsust jälgitakse vee kolmikpunktis. Võrdluskatsed kinnitavad Eesti riigietalonilabori võimet realiseerida vee kolmikpunktide temperatuuri kooskõlas rahvusvahelise tugiväärtusega. Eestis realiseeritud vee kolmikpunktide temperatuuri erinevus rahvusvahelisest tugiväärtusest oli 56  $\mu\text{K}$  laiendmääramatusega 187  $\mu\text{K}$  ( $k = 2$ ).