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RIHO VENDT

Combined method for establishment and dissemination of the international temperature scale





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This study was carried out at the Central Office of Metrology (AS Metrosert) and the University of Tartu.

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LIST OF ORIGINAL PUBLICATIONS

This thesis is based on the following publications (full texts included at the end of the thesis), which are referred to in the text by their Roman numerals. The papers are reprinted with the kind permission from the publishers.

- I R. Vendt, V. Vabson, T. Kübarsepp, M. Noorma, Traceability of temperature measurements in Estonia, P. Est. Acad. Sci., 62 (2) (2013) 116–121. DOI 10.3176/proc.2013.2.05.
- II R. Vendt, V. Vabson, T. Kübarsepp, M. Noorma, Traceability of the water triple point realization in Estonia, in: Proceedings of the 1. Regional Metrology Organizations Symposium RMO 2008; 20. International Metrology Symposium, (D., Ilić, M., Boršić, M., Jurčević, eds.), IMEKO & Metrology Consulting, Zagreb, (2008) 196–198.
- III V. Vabson, T. Kübarsepp, R. Vendt, M. Noorma, Traceability of mass measurements in Estonia, Measurement, 43 (2010) 1127–1133.
 DOI 10.1016/j.measurement.2010.05.002.
- IV V. Vabson, R. Vendt, T. Kübarsepp, M. Noorma, Method for revealing biases in mass measurements, Meas. Sci. Technol., 24 (2013) 025004.
 DOI 10.1088/0957-0233/24/2/025004.
- V R. Vendt, M. Juurma, P. Jaanson, V. Vabson, T. Kübarsepp, M. Noorma, Effects of Environmental Conditions on the Performance of Thermal Imagers, Int. J. Thermophys., 32 (1–2) (2010) 248–257.
 DOI 10.1007/s10765-010-0896-4.

AUTHOR'S CONTRIBUTION

Establishment and development of the national temperature scale in Estonia has been the main responsibility of the author. The achievements of the author's work in research and development activities are summarised in the listed publications.

- **I, II** Development of the measurement models and methods; design of the experiments; conducting all measurements; analysis of the measurement results; full text of the article.
- III Establishment of the metrological traceability for temperature measurements by designing the measurement procedures and calibration of the instruments.
- IV Design of the temperature measurement procedures. Establishment of the metrological traceability and uncertainty analysis of temperature measurements.
- V Design of the experiment and measurement setup; analysis of the measurement results; full text of the article.

ABBREVIATIONS

BIPM Bureau International des Poids et Measures

CCT Comité Consultatif de Thermométrie

CCT-K7 Key comparison of the water triple point cells CIPM Comité International des Poids et Mesures

CIPM MRA Mutual Recognition Agreement of national measurement

standards and of calibration and measurement certificates

issued by the National Metrology Institutes

CGPM Conférence Générale des Poids et Mesures CMC Calibration and Measurement Capability

EURAMET e.v., the European Association of National

Metrology Institutes

ITS-90 International Temperature Scale of 1990

KCRV Key Comparison Reference Value

LNE-INM/CNAM Laboratorie national de métrologie et d'essais,

Conservatoire national des arts et métiers –

National Metrology Institute of France

NMI National Metrology Institute

NSTE National Standard for Temperature of Estonia

MoEAC Ministry of Economic Affairs and Communications

PRT Platinum Resistance Thermometer

SI The International System of Units (Le Système

international d'unités)

SPRT Standard Platinum Resistance Thermometer

TPW Triple Point of Water

I. INTRODUCTION

I.I. Background

In modern society there is a need for reliable measurement results in almost every field of human activity [1]. Sustainable competitiveness and innovation in economy need a reliable and globally recognized metrology infrastructure to deal with national and international regulations, trade and industry policies, scientific and technical issues [2,3]. Discrepancies in measurement, testing and product certification have been identified as the major technical barriers to trade [4]. The basis of the world measurement system for comparable measurements is established by the Metre Convention [5]. Establishment of the national measurement infrastructure is the responsibility of the government of each individual country [1,2,6-8]. The Mutual Recognition Agreement of national measurement standards and calibration and measurement certificates issued by the National Metrology Institutes (CIPM MRA) provides the framework for National Metrology Institutes (NMI) and Designated Institutes to be recognized as the part of the world measurement system [9]. The tasks of a NMI are: maintaining the measurement standards for the units of SI, the International System of Units [10,11]: dissemination of the metrological traceability to the national users by means of calibration [11,12]; preserving the national expertise and advising the government and other domestic parties; performing the international comparisons to demonstrate the equivalence of national standards [9,13]; representing national interests in international organizations [2,8]. NMI-s ensure that the measurement standards in the country are uniform and credible on the international stage. Pool of expertise, links with other NMI-s, and familiarity with new measurement techniques are the features of NMI-s, that introduce new technologies to a country and help to keep up with international trends [11.13].

Most of the national measurement standards in different countries have been developed in a process of continuous scientific research over more than a hundred years [14–20]. Nevertheless, new independent states with evolving economy and urgent need for an operational metrology infrastructure have been emerging [21–23]. For example, the development of national standards was started in Estonia only in 1990-s [24–26]. Establishment of a new national measurement standard is a strategic process and must be well-planned for optimal use of limited resources [17,19,27]. In small countries it is important to make sure, that the infrastructures are optimal for the work load and unnecessary duplication of resources is avoided. It is necessary to estimate the technical level (i.e. the measurement range and capability) of the new standards in order to satisfy the present needs of economy, and support innovation for the future. A single calibration provided by national standards may establish the traceability for tens of calibration laboratories, and thousands of end users [11]. Also, the methods of gaining the credibility and equivalence with international standards have to

be described [28]. Usually the physical definitions of the units according to the definitions of SI are realized by NMI-s [10,13,29,30].

Temperature has been identified as one of the most influential quantity for all fields of measurement [13,26,31–39]. It is a common practice that a national standard for temperature represents a direct realization of the International Temperature Scale of 1990 (ITS-90) by prescribed procedures [40–47]. In 2002, the Ministry of the Economic Affairs and Communications of Estonia (MoEAC) initiated a survey that analyzed calibration services needed by local leading industry enterprises, science institutions, and other national standards. According to the results of the survey, the objective for development of the national standard for temperature establishing the temperature scale with metrological traceability to the ITS-90 in range from -40 °C to +300 °C and approximate expanded uncertainty of 10 mK (at approximate confidence level of 95%, coverage factor k = 2) was raised [48.49]. The aim to establish the temperature scale within the stated range and uncertainty limits corresponds to the secondary level of the metrological traceability chain in dissemination of the ITS-90 [12,42]. The task is unique, as new customized methods for independent and sustainable maintenance of the scale at secondary level had to be developed.

One of the main tasks of a NMI – dissemination of the temperature scale from national standards to other levels of the traceability chain in science, industry, healthcare etc. – is performed by comparison calibrations [13,42]. The list of consumers obtaining traceability from national standards for temperature also includes the other national standards, e.g. mass, length, and voltage, which need reliable and accurate temperature measurements [13,26].

By further development of special methods, the temperature scale established with contact thermometers can also be transferred to radiation thermometers and thermal imagers [41,50,51]. Calibration and use of radiation temperature measurement instruments involve different uncertainty contributions originating from the radiation source, the instrument itself, ambient conditions and the measurement procedures [52-54]. Several studies have been carried out by different metrology institutes in order to assess the uncertainty contributions from different effects [50,55–65]. Although thermal imagers have traditionally been considered only as visualizing or indicative devices, their use for novel applications, e.g., in non-destructive testing, thermal audit of buildings, medicine, and handling of food is enabled by careful studies of error sources [66-70]. For example, in Nordic countries thermal imagers are often used under conditions, which differ significantly from conditions during calibration in the laboratory environment. Nevertheless, the effect of ambient temperature on the output of thermal imagers is rarely taken into account [61]. Performance of thermal imagers can be improved by development of new methods in studying the effects of variable ambient conditions on measurement results.

1.2. Objectives and progress in this work

In this thesis, a method for establishment of the temperature scale by combining primary and secondary techniques of the ITS-90 in the temperature range from -40 °C to +300 °C with approximate uncertainty level of 10 mK (k=2) has been developed [I]. The studies were carried out at the national laboratory for temperature standards (Metrosert), where the author of the thesis has given major contribution to the establishment and development of the national temperature scale in Estonia.

The key element of the method is monitoring the stability of temperature standards for sustainable maintenance of the measurement capability by realization of the triple point of water (TPW) [I, II]. The realization of the TPW temperature has been linked to the international key comparison reference value (KCRV) of CCT-K7 and found to be in good agreement with the reference value as an average deviation of 43 μ K with expanded uncertainty of 190 μ K (k = 2) for the three cells at Metrosert [71, I].

Metrological traceability to the ITS-90 is disseminated from the established national temperature standard to the customers by specially developed calibration procedures [I]. Top-level application of the standard is providing metrological traceability for temperature measurements in evaluation of the different uncertainty sources in mass measurements [III, IV]. The expanded uncertainty estimation of 40 mK (k=2) presented in [III] for air temperature measurements in high accuracy mass comparator chamber is ensured by the methods and calibrations offered only by the national standard [I]. The measurement of the temperature differences for revealing biases in mass comparisons within ± 30 mK with the expanded uncertainty of 2 mK (k=2) is also achieved by development, and application of the special methods based on the availability of the national standards for temperature [I, IV].

Establishment of the temperature scale by the methods described in [I] has offered support to innovation and enabled development of new calibration services for radiation thermometers and thermal imagers [72–74]. A method, technical setup, and uncertainty budget for evaluation of the performance of thermal imagers under variable ambient temperature have been developed [V]. The method and setup have been used for characterization of the relative changes in properties of thermal imagers under changing ambient temperature range from -13 °C to +23 °C and the target temperatures from -15 °C to +120 °C with the expanded uncertainty of 0.5 °C (k = 2) [V].

2. ESTABLISHMENT OF THE TEMPERATURE SCALE [Publications I and II]

2.1. Temperature units and scales

Establishment of the thermodynamic temperature scale is still recognized as proposed by Sir William Thomson (Lord Kelvin) in the middle of the 19th century [75–78]. He showed, that for a theoretical Carnot-cycle heat engine the ratio of the amount of heat Q_1 taken in at the higher temperature T_1 to the amount of heat Q_2 given out at the lower temperature T_2 is

$$\frac{Q_1}{Q_2} = \frac{T_1}{T_2} \tag{1}$$

and the T_1 and T_2 are the thermodynamic temperatures. Equation (1) is valid for a reversible Carnot-cycle and is independent from the working substance and the work W done [76,77,79]. The thermodynamic temperature scale is defined by the equation (1), the second law of thermodynamics setting the absolute zero, and assigning numerical value to the arbitrary selected fixed point [76,77]. This approach was officially adopted by CGPM in 1954 with assigning the numerical value of 273,16 K to the arbitrary selected fixed point – triple point of water [77,80]. The base unit of SI, the unit of thermodynamic temperature kelvin (symbol K) is now defined as the fraction 1/273,16 of the thermodynamic temperature of the triple point of water [10,81,82]. The thermodynamic temperature T can also be expressed in terms of its difference from the temperature of melting ice

$$t/^{\circ}C = T/K - 273,15.$$
 (2)

The temperature t defined by the equation (2) is the Celsius temperature with the measurement unit degree Celsius (symbol $^{\circ}$ C) equal in magnitude to the kelvin [40].

The reversible Carnot-cycle is fictitious; however, there exist thermometers with basic relation between the measurand and thermodynamic temperature T that can be written explicitly without having to introduce unknown temperature-dependent constants. These thermometers, generally called as primary thermometers (e.g. constant volume gas thermometer, acoustic thermometer, noise thermometer, radiation thermometer etc.), are used to determine thermodynamic temperatures [29,83,84]. In practice, the accuracy, repeatability, and ease of use of the thermodynamic thermometers do not meet present requirements of science and industry [13]. Therefore, practical temperature scales are designed and agreed internationally, although they are only best available approximations

of the physical thermodynamic temperature scale and strictly not consistent with the SI [40,85]. The uncertainty of reproduction of practical temperature scales is generally much smaller than the uncertainty of a direct realization and measurement of thermodynamic temperature [13,29,30].

The present international temperature scale ITS-90 was adopted in 1989 by the CIPM according to the Resolution 7 of the CGPM of 1987 and replaced the previous International Practical Temperature Scale of 1968 [40,86–88]. The ITS-90 defines international kelvin temperatures T_{90} and international celsius temperatures t_{90} related to each other as

$$t_{90}$$
/°C = T_{90} /K - 273,15. (3)

The unit of physical quantity T_{90} is the kelvin (symbol K), and the unit of physical quantity t_{90} is degree Celsius (symbol °C) as is the case for thermodynamic temperatures [40].

The definition ITS-90 is based on three elements: 1) defining fixed points, 2) interpolating thermometers, and 3) interpolating equations [40,41]. The fixed points are selected as highly reproducible melting, freezing, boiling, and triple points of pure substances with assigned temperature values that are the best available estimations of the thermodynamic temperature for each particular event. The interpolating thermometers of four different types (Figure 1) – helium vapor-pressure thermometer, helium- or hydrogen-gas thermometer, platinum resistance thermometer, and radiation thermometer – are calibrated at one or more fixed points using the defined interpolating equations [13,29,40,41].

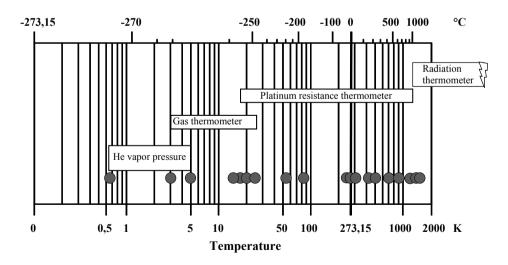


Figure 1. Temperature ranges of the ITS-90 defined by different interpolating thermometers. The dots indicate the temperature values realized by the fixed points of the temperature scale.

In conjunction with the definition of the scale, internationally agreed methods for realization of the ITS-90 allow individual competent laboratories to realize the scale independently [40–42,89,90]. In metrology, traceability for measurement results is established by hierarchical chain of measurements and comparisons [29,30]. The primary realization of the ITS-90 – usually done by NMI-s – cannot be checked by some higher authority, because none of this kind exists [40–47]. Instead, the equivalence of individual scale realizations at different NMI-s is checked with special comparison projects and related to the international key comparison reference value [91,92]. Further, the scale is disseminated to consumers (e.g. in industry, science, healthcare etc.) by comparison calibrations [42].

2.2. Concept and technical solution

The temperature scale is established with objective to cover the needs of research and industry in Estonia, as mapped by the survey of the MoEAC [48]. The aimed expanded uncertainty level of 10 mK (k=2) for calibration of temperature measurement instruments in the temperature range from $-40 \,^{\circ}\text{C}$ to $+300 \,^{\circ}\text{C}$ is generally larger than usually obtained by primary realizations of the ITS-90, but very difficult to achieve by secondary calibrations only [41,93,94]. Therefore, a method for establishment of the temperature scale by combining primary and secondary techniques of the ITS-90 is developed [I, II].

The principal concept and technical solution for establishment of the national standard for temperature in Estonia (NSTE) consists of three main elements (Figure 2):

- 1) import of the ITS-90:
- 2) maintenance and stability monitoring of the standard thermometers; and
- 3) dissemination of the scale [I, II].

The concept for establishment of the temperature scale by combined method is based on a group of standard platinum resistance thermometers (SPRT-s) conforming to the definition and requirements of the ITS-90 [40, I]. The detailed list of the SPRT-s and their properties are presented in Publication [I]. The SPRT-s are calibrated at the fixed points in the temperature range from the triple point of mercury up to the freezing point of aluminum by NMI-s that have established the primary realization of the ITS-90, and published their calibration and measurement capabilities (CMCs) in the BIPM key comparison database containing Appendix C of the CIPM MRA [94]. Selection of the particular fixed points (Figure 2) and interpolating equations is also determined by the definition of the ITS-90. The calibrated SPRT-s represent the realization of the temperature scale for the given range [40, I].

The set of measurement standards (Figure 2) involves cells for reproduction of the TPW and melting point of gallium. Although the configuration of

instruments allows direct realization of the ITS-90 by definition in the temperature range from the TPW to the melting point of gallium, the main purpose of the fixed point cells at Metrosert is to provide reliable and convenient means for monitoring of the properties of the SPRT-s [I].

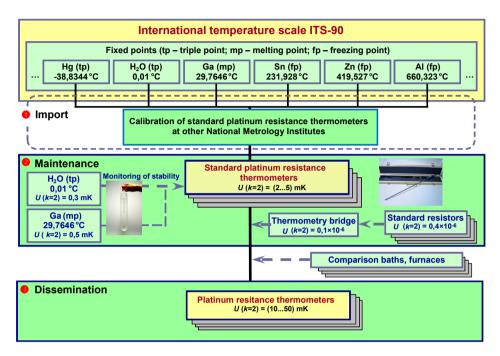


Figure 2. The concept and technical solution for establishment of the national standard for temperature in Estonia. The activities 2 – Maintenance, and 3 – Dissemination are performed at Metrosert.

The SPRT-s are affected by several factors even when handled with care [13,93,95]. For that reason, the stability monitoring of the SPRT-s is the key element for sustainable and reliable maintenance of the temperature scale, since a change of electric resistance at the temperature of the TPW will expose almost all signs of faulty behavior or misuse [13,93, I]. The properties of the resistance measurement bridge and standard resistors used for this purpose are described by Vendt et. al. [96, I]. As the SPRT-s are calibrated at other NMI-s, the effects of transport and handling are evaluated additionally by comparing the resistance values for each thermometer at the TPW before shipping the SPRT-s out from the laboratory, and again when they are returned back. A sample diagram of the stability monitoring of the SPRT-s is depicted on Figure 3.

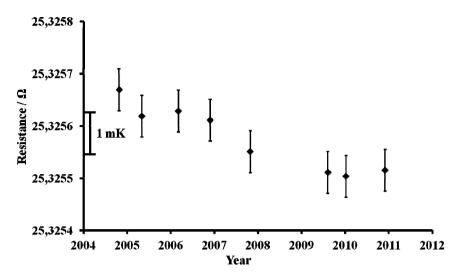


Figure 3. The measured resistance of the SPRT Isotech 970 no. 052 at TPW as an example of the stability monitored over the 6 years of use. The indicated points represent the last resistance values measured at the end of stability check procedure. The vertical bars indicate the expanded uncertainty (k = 2). The accepted drift per year is converted to the respective units of temperature (1 mK) and indicated by the separate bar.

In establishment of the NSTE the change of the electrical resistance at the TPW is converted to the respective units of temperature and the drift of not more than 1 mK per year is accepted (Figure 3) [I]. In case larger drifts are noticed, the SPRT is separated from the group maintaining the scale and directed to further tests and recalibration. By monitoring the stability of each individual thermometer and comparing the calibration curves of the different thermometers the temperature scale can be maintained with ensured confidence [I].

2.3. Realization of the triple point of water

Three different water triple point cells are used at Metrosert to represent the temperature of the TPW according to the definition of the ITS-90 as the national reference in Estonia [I]. Standard procedures for realization of the TPW are followed [40,41,89]. The equivalence of the realization of the TPW at Metrosert with other realizations at different NMI-s has been verified in a dedicated EURAMET comparison project no. 714 [97,98, I]. In this project, the temperature difference between the local realization of the TPW and the circulating transfer cell was measured according to the prescribed procedures [97]. The results were gathered by the piloting institute of the project, LNE-INM/CNAM, and published with a link allowing the participants of the comparison to

position the local TPW realization temperature with respect to the definition of kelvin in accordance with the CIPM Recommendation 2 (CI-2005) [98,99].

Utilizing the offered link, and taking the realization of the TPW at LNE-INM/CNAM as reference, the deviations of the TPW realization temperatures at Metrosert from the definitive value were found to be approximately $-100 \, \mu K$ with the expanded uncertainty of $180 \, \mu K$ (k=2) for all three cells [I, II]. The deviations from the reference value are small in relation to the measurement uncertainty, but still noticeably similar. The origin of this systematic behavior is not entirely known. The effect could be caused by the properties of the water and the construction of the cells as the artifacts were produced by the same manufacturer in years 1999 and 2003. Detailed investigation of this hypothesis is limited as the information on the composition of the water used in the cells at Metrosert is not available.

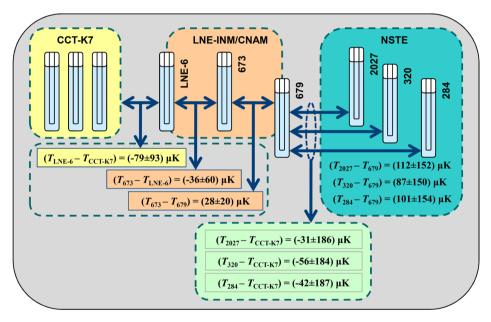


Figure 4. Establishment of the link between the local realization of the TPW at Metrosert and the key comparison reference value $T_{\text{CCT-K7}}$ [71,97,98,100, I, II].

A national metrology institute can be linked to the KCRV through a bilateral or a regional key comparison [101–105, I]. As the French institute LNE-INM/CNAM has also participated in a dedicated comparison project CCT-K7, the realization of the TPW at Metrosert can be also linked to the international key comparison reference value $T_{\rm CCT-K7}$ (Figure 4) [71, I]. The temperature values $T_{\rm X}$ realized locally by the TPW cells at Metrosert are in good agreement with $T_{\rm CCT-K7}$ as it can be seen from the deviations $(T_{\rm X}-T_{\rm CCT-K7})$ presented on Figure 5 [I].

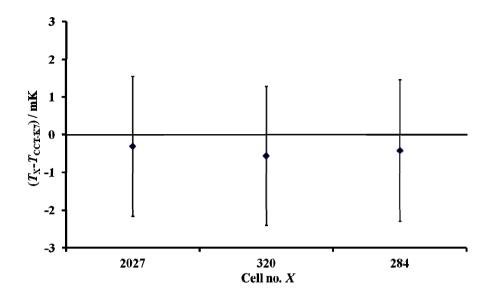


Figure 5. TPW realization values T_x at Metrosert with respect to the international key comparison reference value $T_{\text{CCT-K7}}$. Vertical bars represent the expanded uncertainty (k=2).

2.4. Dissemination of the scale

The temperature scale is disseminated to the temperature measurement instruments by comparison calibrations. A calibration routine has been designed in order to achieve the aimed expanded uncertainty level of 10 mK (k = 2) for thermometers having properties of SPRT-s [I]. The guidelines of the ITS-90 for calibration of SPRT-s are followed with exception, that instead of the measurements in the fixed point cells, the thermostatic liquid baths are fine-tuned to the approximate fixed point temperatures [40, I]. For that purpose the liquid baths with good stability characteristics (Hart Scientific 7341, Hart Scientific 7012, and Isotech 915) have been selected [25, I]. Since the cells of TPW and melting point of gallium are available, they can be also used, when the smallest uncertainties below 10 mK are pursued.

The stability and uniformity of the calibration environment in the baths is improved further by using cylindrical copper equalization blocks for symmetrical accommodation of the reference SPRT-s and thermometers under calibration. The use and design of the equalization blocks form a well controlled and reproducible calibration space within the large bath volume [25, I]. The reference temperature in the blocks is measured and adjusted by using two calibrated SPRT-s. The use of two reference thermometers avoids unexpected measurement errors in case one of the references has been drifted or failed. Comparison of the measurement data from two reference thermometers is also

used for continuous monitoring and evaluation of the stability and uniformity of the temperature in the equalization block. The stability of the reference SPRT-s and the thermometers under calibration is checked at the start, end, and middle of each calibration routine.

In addition to the measurements at approximate fixed point temperature values, supplementary measurements at temperatures between the defining points of the ITS-90 are performed (additional points). For example, the calibration points for the temperature range from 0,01 °C to +300 °C are selected according to the sub-range of ITS-90 from the TPW up to the freezing point of Sn as follows: 0,01 °C (triple point of water); 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 calibration coefficients for PRT-s under calibration are calculated from the measurements at approximate fixed point temperatures by using the interpolating equations of ITS-90. The additional calibration points are used for evaluation of the calibration curve and uncertainties at intermediate temperatures [I].

2.5. Evaluation of measurement uncertainty

Measurements always involve a list of factors having effect on the measurement result. It is accepted, that even if taking into account all suspected error sources and applying appropriate corrections, there is still a number of unknown influences left. Therefore, it is agreed, that a measurement result of a physical quantity must be accompanied with some kind of quantitative indication – measurement uncertainty – describing the quality of the measurements performed. Without such kind of indication, it is not possible to compare measurement results meaningfully either among themselves or with a given reference. The uncertainty evaluation in the following examples is based on the "Guide to the expression of uncertainty in measurement" [49]. A general measurement model for calibration of resistance thermometers is described by equation

$$t = t_e' + \delta t \,, \tag{4}$$

where t is the temperature of the thermometer under calibration; $t'_{\rm e}$ is the temperature measured by the reference standards; and

$$\delta t = \sum_{i} \delta t_{i} , \qquad (5)$$

is the correction due to several contributing effects in a measurement process, where i = 1, 2, 3, ... is the index denoting a particular effect. Each effect in

equation (5) has a corresponding uncertainty contribution u_i . Combined uncertainty u_c is calculated from the individual contributions u_i as

$$u_{\rm c}^2 = \sum_i u_i^2 \ . \tag{6}$$

The expanded uncertainty U is calculates as

$$U = k \times u_{\rm c} \,, \tag{7}$$

where k = 2 is the coverage factor for the approximate confidence level of 95% when assuming normal distribution of measurement results.

2.6. Uncertainties at the triple point of water

The resistance at the temperature of TPW is an important parameter describing the calibration characteristics and the quality of a thermometer [13,40,41]. All measurement errors and uncertainty contributions that occur during the measurements at TPW will propagate to the entire calibration range [106,107]. Monitoring the stability of reference thermometers as well as the thermometers under calibration is also one of the cornerstones of the combined method. The uncertainty budget for calibration of platinum resistance thermometers at the TPW is presented in Table 1.

Table 1. Uncertainty budget for the calibration of platinum resistance thermometers at the TPW [I].

Contributing effect x_i	Probability distribution	Standard uncertainty $u_i(y)$ / mK
Fixed point temperature (TPW) 0,01 °C	Normal	0,1
Calibration and use of the standard resistors	Rectangular	0,1
Calibration and use of the temperature measurement bridge	Rectangular	0,1
Uncertainty of type-A	Normal	0,1
Combined uncertainty u_c		0,2
Expanded uncertainty U (at approximate confidence level of 95 %, coverage factor $k = 2$)		0,4

The *fixed point temperature* takes into account the bias in realization of the fixed point temperature as linked to the international KCRV (Ch. 2.3) [97,98,100, **I**, **II**].

The *uncertainty of type-A* is calculated according to the guidelines in the "Guide to the expression of uncertainty in measurement" on a basis of three repeated measurement series consisting of 100 readings each [49,100].

2.7. Uncertainties for calibration by comparison

The example of the uncertainty budget for calibration of resistance thermometers by comparison in a liquid bath at a single calibration point within the range from -40 °C to +300 °C is presented in Table 2. The calibration is performed at the approximate temperature of a selected fixed point as described in Ch 2.4. It is assumed that the thermometer under calibration is featuring similar properties as SPRT-s: good stability, no hysteresis, and purity of the metal as described in ITS-90 [40, I].

The *standard thermometers* (e.g. Isotech 670, Hart Scientific 5699) are regularly calibrated for the temperature range from –40 °C to +300 °C at the National Metrology Institutes (NMI) that have published their Calibration and Measurement Capabilities (CMC) in the key comparison database of the International Bureau of Weights and Measures (BIPM KCDB) [9]. The instability of the standard thermometer is estimated by the regular checks at the triple point of water. In calibration, the latest resistance value *R*(0,01 °C) for SPRT-s is used [I]. The uncertainty contribution from variation of the self-heating of the reference SPRT-s is taken into account as the reference thermometers are calibrated in fixed points, but used in equalization blocks immersed into the liquid bath. The self-heating characteristics for reference thermometers at the fixed points are obtained from their calibration data. The variation of the self-heating in liquid baths is evaluated from additional measurements [108].

The uncertainty contribution due to the use and calibration of the *standard resistors* is evaluated from the periodical calibration and comparison data. The standard resistors of type Tinsley 5685A 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 °C with stability of \pm 0,02 °C. The standard resistors are calibrated with relative expanded uncertainty (k = 2) of 0.4×10^{-6} and the average drift of the resistance value for the time period of 60 months is monitored to remain below $10 \, \mu\Omega$. Converted to the units of temperature, the overall standard uncertainty (k = 1) from the standard resistors is 0,1 mK. [25,96, I].

Table 2. Uncertainty budget for the calibration of platinum resistance thermometers by comparison [I].

Contributing effect x_i	Probability	Standard uncertainty $u_i(y)$ / mK		
	distribution	(-40+200) °C	(+200+300) °C	
Standards				
Calibration of the standard thermometer	normal	1,0	1,0	
Instability of the standard thermometer	rectangular	0,4	0,4	
Variation of the self-heating in fixed point cell and liquid bath	rectangular	0,6	1,0	
Calibration and use of the standard resistors	rectangular	0,1	0,1	
Calibration and use of the temperature measurement bridge	rectangular	0,7	0,7	
Measurement environment				
Temperature difference between the standard and the thermometer under calibration	rectangular	1,0	4,0	
Changes in temperature difference between the standard and thermometer under calibration	rectangular	1,5	4,0	
Thermometer under calibration				
Calibration and use of the standard resistors	rectangular	0,1	0,1	
Calibration and use of the temperature measurement bridge	rectangular	0,7	0,7	
Measurement of the resistance value at 0,01°C	normal	1,0	1,0	
Combined uncertainty u_c		2,6	6,0	
Expanded uncertainty U (at approximate confidence level of 95 %, coverage factor $k = 2$)		5	12	

The resistance measurements are performed by using an automated *temperature* measurement bridge. The properties of the bridge – Measurements International type MI6010T – 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 remaining within $\pm 0,1\times 10^{-6}$. Taking into account the additional uncertainty sources in calibration and the use of the bridge (e.g. linearity of the bridge, use of multichannel switch and environmental con-

ditions) 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 (k = 1). Measurements of the TPW temperature are performed without the multiplexer at resistance ratios very close to 1, that enables to estimate the contribution of standard uncertainty (k = 1) from the measurement bridge under these conditions to remain below 0,1 mK [25, I].

The thermal uniformity and stability of the *measurement environment* (liquid baths Hart Scientific 7341, Hart Scientific 7012, and Isotech 915) were evaluated in a special study [25, I]. The reference SPRT-s and the thermometers under calibration are inserted symmetrically into the cylindrical equalization block made of copper. The equalization block provides a well studied and reproducible calibration space with improved temperature stability and uniformity within the large volume of the liquid bath. The temperature difference between the reference SPRT-s and the thermometer under calibration as well as the stability of this difference are evaluated during each calibration by exploitation of the symmetrical immersion pattern of the thermometers and the use of two references SPRT-s.

The *thermometer under calibration* is connected to the same combination of the measurement bridge and standard resistors as the standard thermometer by using a multiplexer.

Due to the design of ITS-90 calibration functions, the uncertainty contribution from the *measurements of the resistance value* at 0,01 °C (TPW) propagates to the entire calibration range of the thermometer and therefore the largest estimated value for the whole range is added to the budget. The uncertainty includes any drift detected in the properties of the thermometer under calibration during the calibration procedure by repeated measurements of the resistance value at the TPW before the start and after the end of the routine.

Variation of the self-heating effect for the thermometer under calibration depends on the conditions of its further use. It is considered that the thermometer in the given example will be used under similar conditions as calibrated and the estimated variation of the self-heating effect is not more than few parts of a millikelvin [109].

The combined uncertainty is calculated according to the equation (6) and expanded uncertainty according to equation (7).

3. EFFECT OF TEMPERATURE ON OTHER PHYSICAL QUANTITIES [Publications III and IV]

Temperature is a special physical quantity among the others, because it has an effect on almost every kind of measurement [13,26,31–38]. The methods described in Section 2 are important as they support establishment of all officially approved national measurement scales in Estonia. The contribution from the national temperature scale to the other national scales – length, mass, voltage, and electrical resistance – is described in following chapters.

3.1. Length

It is well-known that all materials are subject to thermal expansion and the change of linear dimensions with the temperature change of Δt is

$$\Delta L = \alpha \Delta t L \,, \tag{8}$$

where L is the original length of an object and α is the coefficient of thermal expansion. A common practice for establishment a secondary length measurement scale is by using interferometrically calibrated gauge blocks. The gauge blocks are usually manufactured from hardened steel, but blocks made of tungsten or ceramics are also widely used. The temperature effect on the length of a standard gauge block can be evaluated with the example of a 100 mm steel gauge block the having thermal expansion coefficient $\alpha = 11.5 \times 10^{-6} \,\mathrm{K}^{-1}$. A change of the material temperature by 10 mK results in a change of 11.5 nm. A temperature measurement error of air temperature about 10 mK in interferometric calibration of gauge blocks would result in an error of 1 nm in the measured length. In practice, temperature nonuniformities in gauge blocks about 5 mK for interferometric and 30 mK for comparison are usually assumed [36,38]. Such kind of tight limits for temperature conditions require reliable temperature measurements. Normally, calibrated thermistors or platinum resistance thermometers with expanded calibration uncertainty 10 mK or less are used [36,38]. This is also the case for the national standards for length in Estonia. The accredited measurement capability (expanded uncertainty, k = 2) for calibration of gauge blocks at national standards laboratory in Estonia is $[0.054^{2} + (0.91 \times L)^{2}]^{\frac{1}{12}} \mu m$ in the range from 0.5 mm to 100 mm and $[0.28^{2} + (1.2 \times L)^{2}]^{\frac{1}{2}}$ µm in the range from 100 mm to 500 mm [74]. Here L denotes the length of the gauge block in meters.

Table 3. Uncertainty budget for the calibration of a L = 100 mm gauge block by comparison at national standards for length in Estonia [110].

Contributing effect x_i	Standard uncertainty $u(x_i)$	Probability distribution	Sensitivity coefficient c_i	Standard uncertainty $u_i(y)$
Calibration of the standard gauge	0,025 μm	normal	1	0,025 μm
Long term stability of the standard gauge	0,03 μm	rectangular	0,577	0,017 μm
Calibration of the comparator	0,01 μm	normal	1	0,01 μm
Repeatability	$0,007~\mu m$	normal	1	0,007 μm
Resolution of the comparator	0,01 μm	rectangular	0,289	0,003 μm
Temperature difference of the gauge blocks	30 mK	rectangular	$1,1\cdot10^{-2}\times L$	0,033 μm
Deviation from the reference temperature (20 °C)	0,1 K	rectangular	$4\cdot10^{-4}\times L$	0,004 μm
Shape uniformity of the gauge block	0,01 μm	rectangular	1	0,01 μm
Reproducibility	$0,01 \mu m$	normal	1	0,01 μm
Combined uncertainty u_c				0,05 μm
Expanded uncertainty U (at approximate confidence level of 95 %, coverage factor $k = 2$)				0,1 μm

The temperature measurements needed for reliable establishment and maintenance of the length scale at the national standards laboratory in Estonia (Table 3) is supported by the calibrations of different temperature sensors, e.q. miniature thermistors, thermocouples and fastresponse 100Ω platinum resistance thermometers. The sensors are usually calibrated by comparison at 0,01 °C, 20 °C, 23 °C, and 29,76 °C with expanded uncertainty (k = 2) of (20...60) mK by following the methods as described in Publication [I].

3.2. Voltage and resistance

Realization of the conventional practical unit for voltage is based on the Josephson effect [111–113]. The maintenance and dissemination of the volt is usually carried out by using solid-state references – Zener diodes [34,114,115]. The output of a solid-state voltage U standard is dependent on time t, temperature T, humidity H, and pressure P

$$U(t,T,H,P) = f(t-t_0,T_{ref},H_{ref},P_{ref}) + \alpha(T-T_{ref}) + h(H-H_{ref}) + \beta(P-P_{ref}) + \varepsilon(t), (9)$$

where $f(t - t_0, T_{ref}, H_{ref}, P_{ref})$ is a function describing the long term drift of the standards output under reference environmental conditions T_{ref} , H_{ref} , and P_{ref} ; α is the temperature coefficient with the temperature correction from the reference temperature T_{ref} ; h is the humidity coefficient with the humidity correction from the reference humidity H_{ref} ; β is the pressure coefficient with the pressure correction from the reference pressure P_{ref} ; and $\varepsilon(t)$ is the intrinsic noise of the voltage standard output [116]. There are several studies describing the sensitivity of solid-state voltage standards to temperature, but the effect may be dependent on the device construction and even for identical items. Therefore the effects of changing ambient conditions on the performance of individual standards must be evaluated [26, 33, and 34]. The national reproduction of the volt in Estonia is also based on solid-state Zener diodes and the properties of the reference standards are described by Pokatilov [26,114]. The values of the temperature coefficient in the range from $-0.013 \times 10^{-6} \text{ K}^{-1}$ to $0.017 \times 10^{-6} \text{ K}^{-1}$ with standard deviation of $0.025 \times 10^{-6} \,\mathrm{K}^{-1}$ for four Zener diodes were measured at the national standards laboratory for electrical quantities [26]. The measurement capability (expanded uncertainty, k = 2) for measurement of voltage at national standards laboratory in Estonia is 1×10^{-6} at 10 V, and 3×10^{-6} at 1 V and 1,018 V [74].

Realization of the conventional practical unit for ohm is based on the quantum Hall effect [111–113]. The maintenance and dissemination of the resistance scale is normally done by using standard resistors [117–119]. The standard resistors, usually manufactured from the Manganin wire, exhibit the resistance – temperature characteristic

$$R(t) = R(25^{\circ}C)[1 + \alpha(t - 25) + \beta(t - 25)^{2}]$$
(10)

in the temperature range from 20 °C to 30 °C, where $R(25 \, ^{\circ}\text{C})$ is the resistance of the resistor at the temperature of 25 °C, and α and β are the characteristic constants. For good quality Manganin the nominal variation of resistance with temperature change over the range from 15 °C to 35 °C remain in the range from $3 \times 10^{-6} \, \text{K}^{-1}$ to $6 \times 10^{-6} \, \text{K}^{-1}$ [117]. In use, it is practical to maintain the standard resistors at reference temperature close to the ambient temperature in laboratory. At Metrosert, this is performed in a temperature controlled oil bath at 23 °C within $\pm 0.01 \, ^{\circ}\text{C}$ [118,119]. The measurement capability for measurement of electrical resistance at Metrosert for the national standards of Estonia is accredited in the range from 1 m Ω to 10 k Ω with relative expanded uncertainties (k = 2) from 5×10^{-6} to 1×10^{-6} respectively [74].

The temperature measurements that are needed for reliable reproduction and maintenance of the national voltage and resistance scales in Estonia are supported by the metrological traceablity from NSTE. For example, a set of

light and fastresponse 100Ω platinum resistance thermometers was calibrated by comparison at $0.01 \,^{\circ}\text{C}$, $20 \,^{\circ}\text{C}$, $23 \,^{\circ}\text{C}$, and $29.76 \,^{\circ}\text{C}$ with expanded uncertainty (k=2) of $20 \,^{\circ}\text{mK}$ by following the methods as described in Publication [I].

3.3. Mass

Mass measurement with balances is influenced by the conditions of ambient air. For example, air buoyancy reduces the weight force on the balance pan, and the buoyancy effect is evaluated by calculation of air density from several parameters such as air pressure, temperature, dew point temperature and content of CO₂ [31,120,121]. The correction for air buoyancy can be taken into account as long as the air conditions are stable and in equilibrium with the balance body and the weight. The equilibrium is disturbed, when the conditions of the weighing system are not homogeneous, especially in case the weight has a different temperature from the surrounding air [31,32]. It has also been estimated that in comparison of 1 kg standard weights a temperature difference of 1 mK between standards and the surrounding air may cause an apparent change of mass approximately by 1 µg [32]. The measurement capability of the national standard for mass at Metrosert for 1 kg weight less than 0,1 mg (k = 2) requires measurement of temperature effects with expanded uncertainty less than 40 mK (k = 2). The temperature measurements that are needed for reliable mass measuremets at the national standards laboratory in Estonia are supported by the metrological traceability from NSTE [III, IV].

For evaluation of the temperature effects in mass measurements, four thermocouple sensors featuring absence of internal heating, small contact gradients, small mass and area, were connected to a high precision climate monitoring instrument Meteolabor AG Klimet A30, and calibrated for the temperature range from 18 °C to 22 °C in steps of 1 °C by comparison with two reference SPRT-s in a thermostatic water bath [IV]. Traceability of this calibration in reference to the ITS-90 is established by the methods described in Publication [I]. The uncertainty budget for the calibration of the thermocouple sensors in conjunction with the climate monitoring instrument Meteolabor AG Klimet A30 is presented in Table 4. The uncertainty contributions for the measurement standards and measurement environment are evaluated as described in Ch. 2.5. The uncertainty from the limited resolution of the thermometer under calibration is evaluated according to the standard guidelines [49].

Table 4. Uncertainty budget for the calibration of the thermocouple sensors in conjunction with the climate monitoring instrument Meteolabor AG Klimet A30 in the temperature range from 18 °C to 22 °C.

Contributing effect x_i	Probability distribution	Standard uncertainty $u_i(y)$ / mK
Standards		
Calibration of the standard thermometer	normal	1,0
Instability of the standard thermometer	rectangular	0,4
Variation of the self-heating in fixed point cell and liquid bath	rectangular	0,6
Calibration and use of the temperature measurement bridge	rectangular	1,0
Measurement environment		
Temperature difference between the standard and the thermometer under calibration	rectangular	1,0
Changes in temperature difference between the standard and thermometer under calibration	rectangular	1,5
Thermometer under calibration		
Resolution of the thermometer	rectangular	0,3
Repeatability (uncertainty of type-A)	normal	0,1
Reproducibility over the calibrated range (uncertainty of type-A)	normal	2,0
Combined uncertainty u_c		3,1
Expanded uncertainty U (at approximate confidence level of 95 % coverage factor $k = 2$)		6
of 95 %, coverage factor $k = 2$)		

The *repeatability* and *reproducibility* are evaluated as standard uncertainties according to the guidelines [49] from the three measurement series consisting of 90 reading each.

A sample of the calibration results for the temperature sensors at 18 °C is presented on Figure 6. With correcting the average systematic temperature difference about 2,5 mK between the calibrated sensor and the SPRT-s, agreement with the ITS-90 with expanded uncertainty (k = 2) better than 10 mK is achieved [IV]. Typical average differences between calibrated sensors are measured below 0,5 mK with the standard uncertainty (evaluation of type-A) 0,1 mK. Thus, the expanded uncertainty for measurement of temperature differences of weights less than 2 mK can be achieved [IV].

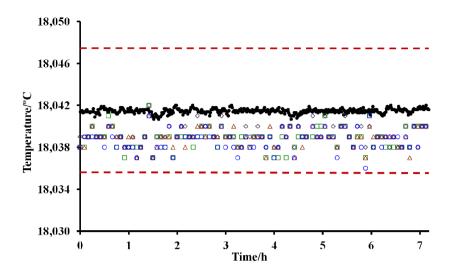


Figure 6. A sample of the calibration results for the thermocouple sensors at 18 °C. The reference temperature $\bullet - T_{\rm ref}$ was measured by two SPRT-s. The temperatures indicated by four thermocouple sensors under calibration are $\Box - T_1$, $\circ - T_2$, $\Delta - T_3$, $\Diamond - T_4$. Dashed lines indicate the expanded uncertainty (k = 2) of the calibration in relation to the reference value $T_{\rm ref}$.

4. NON-CONTACT TEMPERATURE MEASUREMENTS [Publication V]

4.1. Introduction

Nowadays low temperature direct reading radiation thermometers and thermal imagers are widely used for different application in various fields of use. Radiation thermometers measure temperature indirectly by registration of the response signal to the radiant flux of radiation incident on the detector in a specific spectral band. This kind of non-contact temperature measurement below the freezing point of silver (961,78 °C) are based on principles of radiometry in the infrared spectral wavelength range from 8 µm to 14 µm [54].

Thermal imagers have also become easily available as useful measurement instruments in wide area of different and novel applications, e.g. in nondestructive testing, thermal audit of buildings, medicine, and handling of food [66–70]. Traditionally thermal imagers were used in military applications for night-vision and observation of surroundings in difficult atmospheric conditions. In these applications, a relative temperature distribution of the view was observed. Although thermal imagers were originally considered only as visualizing or indicative devices, they can be related to the temperature scale established with contact thermometers and used as measurement instruments either for relative or absolute temperature measurements. Thermal imagers operate on the same principles as radiation thermometers i.e. registration and analysis the response signal of the detector to the radiant flux of the incident radiation. Detectors of thermal imagers can be described as matrices consisting of miniature radiation thermometers representing the pixels of the camera. Therefore the methods developed for calibration of radiation thermometers can also be applied on thermal imagers.

In order to ensure the quality of the measurement results, the instruments must be regularly calibrated [50,51,122]. Calibration and use of radiation temperature measurement instruments involve different uncertainty contributions originating from the radiation source, the instrument itself, ambient conditions and the measurement procedures [52–54]. Several studies have been carried out by different metrology institutes in order to assess the uncertainty contributions from different effects [50,55–65]. Several calibration methods and setups for thermal imagers have been developed and described by several authors [41,51,53,61,62,65,122–126].

The development of the system for the absolute calibration of radiation thermometers and thermal imagers in relation to the ITS-90 has been started at Metrosert by application of the established temperatures scale (Ch. 2). In the first stage of the study, a special method and setup for characterization of the relative changes in properties of thermal imagers under changing ambient

temperature range from -13 °C to +23 °C and the target temperatures from -15 °C to +120 °C has been developed [V].

4.2. Radiation thermometry

Radiometric temperature measurements are usually based on Planck's radiation law

$$L_b(\lambda, T) = \frac{2hc^2}{\lambda^5} \frac{1}{e^{\frac{hc}{\lambda k_B T}} - 1},$$
(11)

where $L_b(\lambda, T)$ is the spectral radiance of the blackbody at wavelength λ and temperature T; h is the Planck's constant; c is the speed of light; k_B is the Boltzmann constant [127]. The response signal from a radiation detector corresponding to the radiation of a standard blackbody can be described as

$$S(T) = \int_{0}^{\infty} R(\lambda) L_b(\lambda, T) d\lambda , \qquad (12)$$

where S(T) is the measurement signal corresponding to the radiation of the blackbody object at temperature T; $R(\lambda)$ is the absolute spectral responsivity of the thermometer including all optical, geometrical, and electronic factors; $L_b(\lambda, T)$ is the spectral radiance of the blackbody according to the Planck's law at wavelength λ and temperature T [54,55,62,64]. Equations (11) and (12) form a basis for temperature measurements according to the ITS-90 above the freezing point of silver and are also applicable for temperatures in the lower range [40,54]. Temperature is measured by forming the ration of equation (12) at the unknown temperature and the defined temperature of either the silver, gold, or copper fixed point, thus requiring knowledge of the relative spectral responsivity $s(\lambda)$ only [40,64]. Application of this method is precluded for practical calibration of radiation thermometers below the temperature freezing point of silver because of the difficulties associated with direct measurement of the relative spectral responsivity at the longer wavelengths [54,59,64]. Instead, the signal S(T) can be approximated by an algebraic interpolation equation in terms of several adjustable parameters. Interpolation equations coupled with measurements at multiple fixed points or comparisons with contact thermometers provide highly practical and efficient means for temperature measurement. This method allows calibration of radiation thermometers without knowing the spectral responsivity [54,59,64]. While different interpolation functions can be used, it has been suggested by several studies, that the Sakuma-Hattori equation (13) provides the best compromise between accuracy,

ease of use and number of parameters [54,59,128]. The Planck version of the Sakuma-Hattori equation is

$$S(T) = \frac{C}{e^{\frac{c_2}{AT + B}} - 1},\tag{13}$$

where A, B, and C are calibration constants for a particular imager, and c_2 is the second radiation constant. The parameters A, B, and C are determined from measurements of the response signal of the thermometer at given temperature. The temperature-signal pairs (T_i, S_i) are measured at several calibration points i = 1, 2, 3, ..., N, with $N \ge 3$. In case, N = 3, the equation (13) is solved so that the equation passes exactly through each of the calibration points. When N > 3, the parameters A, B, and C are calculated by using the method of the least-squares [54,59].

4.3. Characterization of radiation thermometers

For characterization and uncertainty evaluation of the radiation thermometers, the measurement signal $S_r(T)$ related to the target blackbody temperature under real measurement conditions, can be described as

$$S_r(T) = \varepsilon \tau S_{obj}(T) + (1 - \varepsilon)\tau S_{refl}(T_{refl}) + (1 - \tau)S_{env}(T_{env}) + S_{opt}(T_{opt}) - S_{det}(T_{det}) + \Delta S_{sse} + \Delta S, \quad (14)$$

where $S_{\text{obj}}(T)$ is the signal caused by the radiation emitted by the object; ε is the effective emissivity of the real target and τ is the effective transmissivity of the atmosphere. The term "effective" here and in following represents the superposition of different factors (e.g. dimensions of the objects, distance to the object, temperature non-uniformities of the object, imager and ambient environment etc.) as the real values of the quantities may depend on several different parameters and are difficult to describe [62]. $S_{refl}(T_{refl})$ is the signal caused by radiation from surrounding objects at the effective temperature T_{refl} reflected by the target. $S_{env}(T_{env})$ is the signal corresponding to the radiation emitted (or absorbed) from the environment between the object and imager at effective temperature T_{env} ; $S_{opt}(T_{opt})$ is the signal caused by the radiation emitted from the optical components of the imager at the effective temperature T_{opt} , including the radiation emitted by other components of the imager and reflected from the optics or directly incident to the detector; $S_{det}(T_{det})$ is the signal caused by the radiation from the detector itself at the temperature T_{det} . Under the real measurement conditions, additional error sources like the size-of-source effect ΔS_{SSE} [129] and any residual error ΔS (e.g. from limited digital resolution of electrical channel, temperature dependent variations of the gain and offset in the electrical channels) have to be taken into account. The measured signal $S_r(T)$ is

processed in circuits of a radiation thermometer and the corresponding measured temperature T_{meas} is given to output. Following the equation (14), calibration and use of radiation temperature measurement instruments involve different error sources originating from the properties of measurement objects, instruments and ambient conditions [52–54,61,130]. Several studies have been carried out by different metrology institutes in order to assess particular uncertainty contributions from different effects [57,58,60–65,131].

4.4. Ambient temperature effects

Radiation thermometers and thermal imagers are often used under ambient conditions significantly different from the conditions during calibration in the laboratory environment [133,134]. However, the effects of the ambient temperature on the output of the thermal imagers are rarely taken into account by users [61, V].

Temperatures of surrounding air as well as the body temperature of a radiation thermometer or a thermal imager have been identified as significant sources of uncertainty of the measurement results [65,130,132, V]. As described in equation (14), the net signal given by the detector of a radiation thermometer is the difference between the signal $S_{\rm obj}(T)$ at the source radiance temperature T and the signal $S_{\rm det}(T_{\rm det})$ originating from the detector itself at temperature $T_{\rm det}$. This requires that the temperature of the detector has to be stabilized and measured, but in the wide selection of different commercial radiation thermometers and thermal imagers available, there exist lot of instruments without internal temperature control or compensation [50,54].

Another combined effect from the ambient temperature on the properties of radiation thermometers and thermal imagers is generally caused by the change in background radiation, change of the detector responsivity due to changes in detector temperature, temperature dependence of the amplification in electric circuits, changes in optics etc. It is usually impossible to distinguish and characterize these effects independently, and therefore they are usually treated as a combined net effect [50,54].

4.5. Experimental setup for calibration of radiation thermometers and thermal imagers at variable ambient temperature

An experimental setup (Figure 7.) was designed and constructed for evaluation of the effect of ambient temperature on the performance of radiation thermometers and thermal imagers [V]. The setup can be used for calibration of radiation thermometers and thermal imagers at various ambient temperature conditions.

The setup features of a custom-made climatic chamber with 296 L of test volume designed to operate in the temperature range from $-20\,^{\circ}\text{C}$ to $+30\,^{\circ}\text{C}$. The temperature in the chamber is controlled by a commercial cooling system and an additional heating source with the working temperature range from $+20\,^{\circ}\text{C}$ to $+100\,^{\circ}\text{C}$ and stability of $\pm0,01\,^{\circ}\text{C}$. In order to improve the temperature homogeneity in the chamber, circulation of air in the chamber is generated by constant speed electric fans. Several calibrated temperature sensors (PRT-s) are installed to monitor the temperature distribution and stability in the chamber. The uniformity of the air temperature in the climatic chamber was measured to remain within $\pm1\,^{\circ}\text{C}$ with stability of $\pm0,5\,^{\circ}\text{C}$ at $-13\,^{\circ}\text{C}$, the most difficult set point to maintain. Temperatures of the chamber walls and the imager housing were also monitored in order to evaluate the possible effects from the internal and reflected radiation. The setup has no humidity control – the relative humidity in the chamber was measured to vary from 12 % at $+21\,^{\circ}\text{C}$ to $46\,^{\circ}\text{C}$ at $-13\,^{\circ}\text{C}$.

A commercially manufactured temperature-controlled flat-plate blackbody with a diameter of 152,4 mm is used as a radiation source. The emissivity of the source is $\varepsilon_{\text{target}}$ =0,95 as specified by the manufacturer. The uncertainty of the emissivity of the source is important for the absolute calibration of thermometers with traceability to the ITS-90. During the first stage of the development process only the relative temperature measurements are observed. However, the emissivity of the source has to be measured more carefully in the future.

The temperature of the flat radiator is recorded by a calibrated temperature sensor, positioned right behind the centre of the radiating surface of the blackbody. The port in the cover of the climate chamber is large enough to ensure a clear field of view for the imager. The metrological traceability of the temperature measurement results to the units of SI is obtained from contact temperature measurements with link to the NSTE [I].

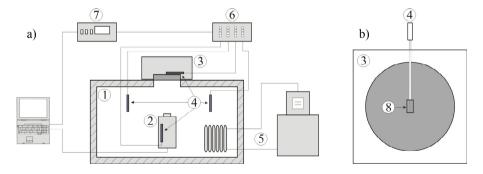


Figure 7. a) A schematic view of the measurement system and b) the configuration of the reference blackbody: 1 - climatic chamber; 2 - thermal imager; 3 - flat plate blackbody radiation source; 4 - platinum resistance thermometers; 5 - heating source with the circulator and fans for maintaining the environment temperature; 6 - thermometer selector switch; 7 - thermometer readout device connected to computer; 8 - area of pixels extracted for data analysis [V].

The capability of the experimental setup to detect the effects of ambient temperature on measurement instruments has been investigated with two commercial imagers: a high-end thermal imager for scientific use with a Stirling cooled 320×240 pixels detector (Imager 1) and a mid-range imager for industrial applications with 120×120 pixels non-cooled detector (Imager 2) [135,136, V]. The temperatures measured with the imagers, T_{meas} , were compared to the reference temperatures, T_{ref} , measured by the reference thermometer. The resulting biases

$$\delta T = T_{meas} - T_{ref} \tag{15}$$

for the high-end imager (Imager 1), as depicted on Figure 8, remain within the uncertainty limits at all ambient temperatures, while the biases for mid-range imager (Imager 2) vary with change of the ambient temperature (Figure 9).

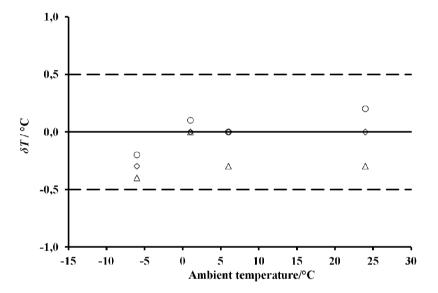


Figure 8. Measured bias δT at the target temperatures $-10 \,^{\circ}\text{C} - \Delta$, $0 \,^{\circ}\text{C} - \Delta$ and $+10 \,^{\circ}\text{C} - \Diamond$ as a function of the ambient temperature for Imager 1. Dashed lines indicate the estimated expanded uncertainty limits (k=2) [V].

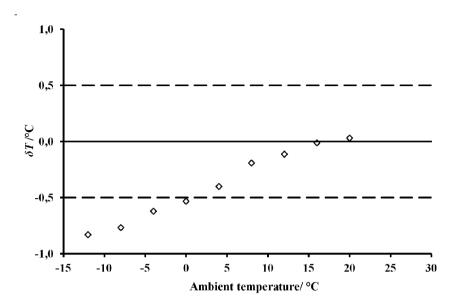


Figure 9. Change of the bias δT as a function of the ambient temperature for Imager 2 with the target temperature of +10 °C. Dashed lines indicate the estimated expanded uncertainty limits (k = 2) [V].

4.6. Uncertainty evaluation

The uncertainty evaluation (Table 5) is based on two assumptions: 1) the purpose of the measurement here is to study the capability of the test setup to detect the ambient temperature effects on the performance of thermal imagers; calibration of the radiation thermometers and thermal imagers in relation to the absolute temperature scale with this setup is not yet intended; 2) only the changes in measured bias are observed. Following these assumptions, some uncertainty contributions of systematic character are reduced by strong correlation. Evaluation of the uncertainty budget in Table 5 is based on the "Guide to the expression of uncertainty in measurement" [49].

The short term instability of the reference thermometer was estimated from the calibration data. Changes of the resistance value R (0,01 °C) were monitored during the calibration of the thermometer in the standard uncertainty limits of 0,005 °C.

Temperature instability of the target was monitored by the reference thermometer and was found to remain in standard uncertainty limits of 0,06 °C.

Temperature non-uniformity of the target. The uncertainty limits of 0,01 °C for the temperature non-uniformity over the target area were monitored with a calibrated thermal imager, and were found in agreement with the estimation according to the values published by Liebmann [58].

Table 5. Uncertainty budget for the change in the bias $\delta T(T)$ of the thermal imager at each ambient temperature in respect to the temperature at calibration [V].

Contributing effect x_i	Probability distribution	Standard uncertainty $u_i(y)/$ °C	
		Imager 1	Imager 2
Target stability			
Short term instability of the reference PRT	rectangular	0,005	
Temperature instability of the target	rectangular	0,06	
Temperature non-uniformity of the target	rectangular	0,01	
Change of the temperature gradient between the target surface and the reference PRT	rectangular	0,01	
Change of the target emissivity ε	rectangular	0,1	
Thermal imager			
Thermal sensitivity of the imager	rectangular	0,01	0,03
Type-A uncertainty of the selected pixel values	normal	0,1	0,1
Effect of ambient conditions			
Effective reflected temperature value T_{refl}	rectangular	0,1	0,1
Atmospheric absorption	rectangular	0,1	0,1
Method			
Repeatability and reproducibility	normal	0,1	0,1
Combined standard uncertainty (<i>k</i> =1)		0,23	0,23
Expanded uncertainty (<i>k</i> =2)		0,46	0,46

Changes of the gradient between the target surface and reference thermometer are due to the heat exchange between the target surface and ambient environment. At different ambient temperatures, the target temperature will stabilize at slightly different values. The uncertainty limits of 0,01 °C were estimated according to the research described by Liebmann [58].

Change of the target emissivity. Target emissivity $\varepsilon(\lambda,T)$ is a function of wavelength λ and target temperature T. Emissivity of the target can also be affected by ice crystals forming on the surface at freezing temperatures. These contributions were estimated to be within 0,1 °C according to the studies of Liebmann [58] and Horwitz [137]. The uncertainty from the long term stability of the target emissivity is estimated to have insignificant contribution to the uncertainty budget here, as only relative measurements in a short period of time are carried out in this study.

Thermal sensitivity of the thermal imagers. The uncertainty contribution due to the limited thermal sensitivity of the thermal imagers was estimated according to the manufacturers' specifications: 0,01 °C for Imager 1 and 0,03 °C for Imager 2.

Type-A uncertainty of the temperature values for the selected pixels. The uncertainty of type-A, resulting with a value of 0,1 °C, was calculated from the selected pixels (described in section 3.2) of each thermal image according to the guidelines [49].

Effective value of the reflected temperature. Both thermal imagers used in this experiment had an option for correction of the effective reflected temperature value. The effective reflected temperature values were measured for both thermal imagers at each ambient temperature. This was done by replacing the flat blackbody target with highly reflective target – a wrinkled sheet of aluminum foil and setting the imagers emissivity parameter $\varepsilon_i = 1$. By variation of the effective temperature input value in the software provided by manufacturers of the thermal imagers, the standard uncertainty limits of 0.1 °C for the correction of effective reflected temperature value were found. The measured effective value for reflected temperature was compared to the temperature of the walls of the climatic chamber, the body of the thermal imager and air temperature. The effective value for reflected temperature is a superposition of a complicated temperature distribution in the climatic chamber and therefore difficult to compare directly with the contact measurements. The average value calculated from contact measurements was found in agreement with the radiation measurement within estimated uncertainty limits.

Atmospheric absorption. By variation of the input values for an ambient temperature ± 10 °C and a relative humidity $\pm 20\%$ in the software provided by manufacturers of the thermal imagers, the standard uncertainty limits of 0,1 °C for the correction of atmospheric absorption were found. The uncertainty contributions due to atmospheric absorption are reduced by the unchanged target distance, absolute humidity and content of CO_2 as the changes in deviations of the imagers are observed.

Size-of source effect. The uncertainty contribution from the size-of-source effect in a particular case is considered insignificant as: 1) the changes in measured bias are observed, 2) the distance between the target and imager remained unchanged for each ambient temperature value and 3) only a small portion of pixels from the centre of the field-of-view was selected. The effect of the scattered radiation from the surroundings on the selected pixels was studied by covering the target partly with an aperture at an ambient temperature –13 °C and the target temperature set to +10 °C. The abrupt temperature change caused by the aperture was detected no further than the ten closest pixels from the aperture edge on the thermal image. As the pixels used for the analysis in this work were selected from the central part of the camera detector, the corresponding contribution to the uncertainty of the measurement result is considered below the detection level and therefore not added to the uncertainty budget in Table 5.

Repeatability and reproducibility. The type-A uncertainty 0,1 °C was calculated from the repeated measurements according to the guidelines [49].

5. CONCLUSIONS

In this study, the temperature scale in the range from -40 °C to +200 °C with expanded uncertainty of 5 mK and from +200 °C to +300 °C with expanded uncertainty of 12 mK (at approximate confidence level of 95%, coverage factor k=2) has been established and linked to the international temperature scale ITS-90. The targeted uncertainty levels – generally larger, than usually obtained by primary realisations and too small for the secondary methods – have been achieved at Metrosert, the national laboratories of Estonia, with combination of primary and secondary techniques of the ITS-90. The TPW realisations at Metrosert have been linked to the international reference value with expanded measurement uncertainty below 0,19 mK (at approximate confidence level of 95%, coverage factor k=2).

The methods for dissemination of the established temperature scale with the expanded uncertainty of 10 mK (at approximate confidence level of 95%, coverage factor k=2) have been developed to support the measurement capability of the Estonian national measurement standards for length, mass, voltage, and electrical resistance. Temperature measurements with the expanded measurement uncertainty from 10 mK to 40 mK (at approximate confidence level of 95%, coverage factor k=2) have been provided for studies in mass metrology. The established temperature scale and dissemination methods also support the measurement capability and reliability of industrial and scientific measurements carried out in Estonia by providing customers with about 50 temperature calibrations per year. The combined method as described on the example of the national standard laboratory in Estonia can be applied for establishment of the temperature scale in other laboratories striving to the comparable level of the measurement capability.

The temperature scale established by the contact thermometry can also be transferred to non-contact temperature measurements. As the number of different radiation thermometers and thermal imagers in various fields of use is increasing, there is also a need for reliable calibration methods ensuring metrological traceability to ITS-90. This study started the development of the calibration method and setup for calibration of radiation thermometers and thermal imagers under variable ambient temperature range from -13 °C to +23 °C and object temperatures from -15 °C to +120 °C with traceability to ITS-90. In this thesis, the first results of the study are described. The method and setup have been proven to detect bias in readings of a target temperature due to the change of ambient temperature with expanded uncertainty of 0.5 °C (at approximate confidence level of 95%, coverage factor k = 2). The development of the method will continue in further studies.

The achievements of the study were acknowledged first in 2005 with the approval of the national standard for temperature in Estonia (NSTE) by the Minister of Economic Affairs and Communications. In 2009 the national standards laboratory of Estonia (Metrosert) was accredited for calibration of

platinum resistance thermometers. In 2011, a survey was organized by the Ministry of economic affairs and communications in order to evaluate the impact of the present metrology infrastructure on industry and further needs for development. The survey concluded that the current measurement range and capability of the NSTE cover the present needs of research and industry in Estonia and form a good base for the further development of new measurement services.

SUMMARY

Reliability and international equivalence of measurement results are important issues in sustainable operation and development of the modern society. Discrepancies in measurement results have even been identified as one of the major barriers to trade and innovation. It is a common practice that each independent country has established a national measurement infrastructure. In most countries these infrastructures have been developed in a process of scientific research over more than hundred years. Nevertheless new independent countries with evolving economy and needs for a reliable metrology system are still emerging. This was also the case for Estonia, where the development of national standards and measurement scales started only in the 1990-s.

Establishment of a new national measurement standard must be designed carefully for optimal use of limited resources. In this study the establishment and dissemination of the national temperature scale in Estonia with the link to the international temperature scale ITS-90 in the temperature range from -40 °C to +200 °C with expanded uncertainty of 5 mK and from +200 °C to +300 °C with expanded uncertainty of 12 mK (at approximate confidence level of 95%, coverage factor k=2) has been described. The temperature range and uncertainty level values originate from the actual needs mapped by a special survey carried out by the Ministry of Economic Affairs and Communications in 2002. The stated uncertainty values are generally larger than usually obtained by primary realizations, but too small to be achieved by secondary methods only. Therefore the temperature scale is established by combining primary and secondary techniques of the ITS-90. The method includes monitoring of the stability of the measurement standards at the triple point of water that has been linked to the international key reference value. The combined method for establishment of the temperature scale is described on the example of Metrosert – the laboratory for national standards in Estonia, but it can be also applied in other laboratories aiming the similar measurement range and capability.

The second part of the study describes top level applications of the established temperature scale supporting the reliable measurement of other physical quantities e.g. mass, length, and voltage. For example, evaluation of temperature effects in accurate mass measurements needs temperature measurements methods with expanded calibration uncertainty of temperature measurement instruments about 20 mK (at approximate confidence level of 95%, coverage factor k = 2).

The temperature scale established on the basis of contact thermometry can be extended to non-contact temperature measurements. Development of technology pushes a wide selection of different radiation thermometers and thermal imagers towards traceable calibration to ITS-90. In this study the development of a method for calibration of radiation thermometers and thermal imagers under varying ambient conditions has been started. The sources of uncertainty are discussed on an example of the measurement model and one of

the uncertainty sources – the effect of environmental conditions on measurement results has been studied. A technical setup and method for calibration of radiation thermometers and thermal imagers under variable ambient temperature is described over the ambient temperature range from –13 °C to +23 °C and object temperatures from –15 °C to +120 °C. The method and setup have been proven to detect bias in the readings of a target temperature due to the change of an ambient temperature with expanded uncertainty of 0,5 °C (k=2). The development of the method for establishment of the traceability for non-contact temperature measurements in relation to the ITS-90 will continue in further studies.

In 2011, a survey conducted by the Ministry of Economic Affairs and Communication concluded, that the current measurement range and capability developed at the national laboratory for temperature meet the present needs of research and industry in Estonia.

SUMMARY IN ESTONIAN

Rahvusvahelise temperatuuriskaala esitamine ja edastamine kombineeritud meetodil

Kaasaegse ühiskonna konkurentsivõimeliseks ja jätkusuutlikuks toimimiseks on oluline tagada mõõtetulemuste usaldusväärsus ja rahvusvaheline ekvivalentsus. Usaldusväärsed mõõtmistulemused on vajalikud teadusuuringutes, tööstuses, kaubanduses, riiklikus järelevalves, meditsiiniteenuste osutamisel, keskkonnakaitses ja paljudel teistel erialadel. Lisaks riigisiseste mõõtetulemuste seostatuse kindlustamisele tuleb igal riigil täita ka rahvusvahelisi leppeid, mis soodustavad riikidevahelist kaupade ja teenuste vaba liikumist ning aitavad tugevdada riigi majanduslikku arengut ja konkurentsivõimet. On heaks tavaks, et iga iseseisev riik hoolitseb riikliku mõõtesüsteemi arendamise ja toimimise eest. Enamike riikide mõõtesüsteemide arendamine on kestnud juba rohkem kui sada aastat. Aeg-ajalt tekib uusi iseseisvaid riike, mille arenev majandus vajab suhteliselt kiireid lahendusi usaldusväärsete mõõtetulemuste tagamiseks. Riikliku mõõtesüsteemi ja riigietalonide vajadus Eestis tekkis eelmise sajandi üheksakümnendatel aastatel.

Riigietalonide arendamine on aeganõudev ja ressursimahukas protsess, mida tuleb piiratud vahendite optimaalseks kasutamises hoolikalt planeerida. Majandus- ja Kommunikatsiooniministeerium kaardistas 2002. aastal mõõteteenuste vajadused, mille tagamiseks oli tarvilik välja arendada etalonibaas Eestis. Vajaduste rahuldamiseks arendati välja riigietalonid rahvusvahelise temperatuuriskaala ITS-90 taasesitamiseks mõõtepiirkonnas ($-40 \dots +200$) °C laiendmõõtemääramatusega 5 mK (usaldusnivool ligikaudu 95%, kattetegur k=2) ja mõõtepiirkonnas ($+200 \dots +300$) °C laiendmõõtemääramatusega 12 mK (usaldusnivool ligikaudu 95%, kattetegur k=2). Nimetatud mõõtemääramatused on üldjuhul suuremad kui temperatuuriskaala esitamisel primaartasemel, kuid sellist taset on keeruline saavutada ainult sekundaarseid vahendeid kasutades. Seetõttu on temperatuuriskaala esitamiseks Eesti riigietalonilaboris (Metrosert) arendatud primaar- ja sekundaarmeetodeid kombineeriv meetod. Eesti temperatuuri riigietaloni näitel kirjeldatud meetod on kasutatav ka teistes laborites, mille eesmärgiks on tagada mõõteteenused sarnasel mõõtemääramatuse tasemel.

Uurimuse teises osas esitatakse näited väljaarendatud temperatuurimõõtmiste tähtsast rollist teiste füüsikaliste suuruste, näiteks massi, pikkuse ja alalispinge mõõteskaalade usaldusväärsel esitamisel. Temperatuuri mõju hindamiseks erinevate suuruste täppismõõtmistel peavad teatud juhtudel temperatuurimõõtevahendid olema kalibreeritud laiendmääramatusega alla 20 mK (usaldusnivool ligikaudu 95%, kattetegur k = 2), mis uurimuse käigus arendatud meetodite kasutamisega on saavutatav.

Uurimuse tulemusena väljaarendatud kontakttermomeetritel põhinevat temperatuuriskaalat on võimalik üle kanda ka puutevabadele temperatuurimõõtevahenditele. Tehnoloogia arenguga on puutevaba temperatuuri mõõtmist

võimaldavad kiirgustermomeetrid ja termokaamerad leidnud laialdast rakendamist erinevates kasutusvaldkondades. Usaldusväärsete, rahvusvahelise temperatuuriskaalaga ITS-90 seostatud mõõtetulemuste tagamiseks vajavad puutevabad mõõtevahendid sobivaid kalibreerimismeetodeid ja -vahendeid. Seetõttu alustati temperatuuri riigietalonilaboris katsesüsteemi ja -meetodi arendamist, mis võimaldab kalibreerida kiirgustermomeetreid ja termokaameraid arvestades muutuva keskkonnatemperatuuri mõju mõõtetulemustele. Arendustöö esimese osana on valminud katsesüsteem, mille abil saab uurida puutevabade temperatuurimõõtevahendite mõõtehälbe muutust sõltuvalt keskkonnatemperatuurist. Katseskeem on kasutatav vahemikus $(-13 \dots +23)$ °C ja mõõteobjekti temperatuuridel $(-15 \dots +120)$ °C mõõtemääramatusega 0,5 °C (usaldusnivool ligikaudu 95%, kattetegur k=2). Katsesüsteemi arendus mõõtetulemuste absoluutseks seostamiseks temperatuuriskaalaga ITS-90 jätkub.

Majandus- ja Kommunikatsiooniministeeriumi tellimusel toimus 2011 a. uuring, mille tulemused näitasid, et käesoleva uurimuse tulemusena temperatuuri riigietalonilaboris välja arendatud mõõteteenused katavad Eesti tööstuse ja teaduse hetkevajadused.

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