

# **COOMET Recommendation**

# Calibration of platinum resistance thermometers by comparison method

**COOMET R/T/33:2018** 

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#### 1. Scope

This procedure is for calibration of platinum resistance thermometers in the temperature range from (-80) °C to 650 °C.

#### 2. Abbreviations

EUT - equipment under test

SPRT - standard platinum resistance thermometer

PRT - platinum resistance thermometer

#### 3. Standards and equipment

# 3.1 Working standards

- 1. PRT Pt<sub>10</sub>
- 2. PRT Pt<sub>25</sub>
- 3. PRT Pt<sub>100</sub>
- 4. Temperature converter (thermometer readout, DC/AC bridge)
- 5. Multimeter (ohmmeter)

#### 3.2 Additional equipment used during calibration

- 1. water bath (thermostat)
- 2. oil bath
- 3. low temperature bath
- 4. block calibrator

# 3.3 Additional non-calibrateable equipment

1. Dewar vessel

#### 4. Valid documents

- 1. Guide to the Realization of ITS-90, BIPM (CCT), 2016;
- 2. Guideline DKD-R 5-1 «Calibration of Resistance Thermometers», 2003;
- 3. Guideline DKD-R 5-6 «Bestimmung von Thermometerkennlinien», 2008;
- 4. CMC review protocol for calibration of industrial thermometers, 6.05.2010;
- 5. IEC 60751: 2008;
- 6. GOST 6651-2009;
- 7. JCGM 100 «Evaluation of measurement data Guide to the expression of uncertainty in measurement», 2008;
- 8. operation manual, specifications for EUT.

# 5. Safety instructions

Safety instructions according to working instructions for EUT and for standards and equipment using for calibration should be taken into account.

#### 6. Ambient conditions in calibration

Normalized conditions:

- ambient temperature range:  $(23 \pm 5)^{\circ}$ C /  $(20 \pm 5)^{\circ}$ C
- ambient relative humidity:  $\leq 80 \%$ .

#### 7. Calibration steps

# 7.1 Visual inspection

Compliance of EUT with the following requirements must be stated:

1. Resistance thermometer and connecting leads should not have any visible damaged part, corrosion on metal parts. EUT should be clean.

2. Type, accuracy, temperature range, immersion depth of EUT should be determined.

#### 7.2 Preparation of auxiliary equipment

Additional equipment is prepared according to correspondent working instructions.

# 7.3 Preparation and installation of standards and EUT

SPRT and EUT are cleaned carefully using alcohol and tissue paper before installation into baths (auxiliary equipment). SPRT should be immersed as it was under its calibration. SPRT and EUT should not undergo to mechanical and temperature load. Clamps and holders should be used to ensure that the handle and cable are not immersed. Cooling or heating of the SPRT and EUT must be run gradually. **Do not screw clamps very strong. This can damage sheath of the thermometers.** Temperature transducer is prepared according to working instruction for it. SPRT and EUT should be installed as close as possible, but should not touch each other. EUT is

SPRT and EUT should be installed as close as possible, but should not touch each other. EUT is immersed at the marked depth (if the marked depth of EUT is absent, EUT should be immersed as deep as possible in order to exclude error associated with stem effect). Sensitive elements of SPRT and EUT should be immersed at the same level, if it is possible. **SPRT and EUT are installed in block calibrator's wells of appropriate size not tightly to not be stuck in them**. EUT is connected to the precision ohmmeter.

#### 7.4 Determination EUT resistance's dependence on temperature

- 1. Callendar-van Dusen equations (CVD-coefficients) are used for platinum resistance thermometer. According to the equations optimal number of calibration points is chosen by the following way: two points for negative range, four points for positive range and zero point 0 °C. Number of calibration points should be not less then number of coefficients of interpolation function. Calibration points should divide calibration range by equal intervals. Additionally calibration points to be included should be discussed with customer.
  - It is allowed to calculate coefficients for deviation function  $\Delta W$ . In this case, calibration points are chosen in accordance with correspondent sub-range of ITS-90. Number of calibration points should be not less then number of coefficients of deviation function.
- 2. Calibration is carried out from low temperatures to higher ones.
- 3. Measurement in zero point is carried out at the beginning and at the end of the calibration. Zero point is realized in Dewar vessel.
- 4. Resistance of EUT and reference temperature indicated by the SPRT is measured in each calibration points. Temperature is measured with accuracy of 0,001 °C, resistance is measured with accuracy of 0,0001  $\Omega$ . All measurements are performed after steady-state conditions have been reached. Steady-state conditions are considered to be reached when systematic variations of temperature are no longer observed.
  - 10 readings of SPRT and EUT correspondently are carried out. Readings are recorded in minimum and equal time periods.
- 5. Temperature value measured with EUT is calculated using measured resistance values of EUT and standard CVD- coefficients according to GOST 6651-2009 (IEC 60751:2008). Temperature is calculated with accuracy of 3 digits like measured standard temperature. Furthermore, temperature deviation between temperature measured with SPRT and temperature measured with EUT is calculated in each calibration point. The deviation should not exceed limits noted in technical specification for EUT or in IEC 60751:2008. Then customer can use standard tables and coefficients noted in technical specification for EUT or in IEC 60751:2008. If the deviation exceeds the limits, then EUT is assigned to lower accuracy class. For customer request individual CVD-coefficients or coefficients for deviation function of ITS-90 are calculated for EUT. In both cases, Individual CVD-coefficients A, B, C and R<sub>0</sub>, and coefficients for deviation function of ITS-90 are

calculated by less squares method. Temperature according individual CVD-coefficients is calculated by iteration method, realized with appropriate software. And temperature according deviation function are calculated using reverse function  $T(W_r)$ . Interpolation functions  $W_r$ , reverse functions  $T(W_r)$  and deviation functions  $\Delta W$  are given in Guide to the Realization of ITS-90, BIPM (CCT), 2016.

#### 7.5 Determination of stability of EUT at 0 °C

Stability of EUT is determined as difference between temperature values measured by EUT at 0 °C before and after resistance measurements of EUT in all temperature points. Obtained value gives additional contribution in calibration uncertainty of EUT.

#### 7.6 Determination of uncertainty contribution associated with self-heating of EUT

For determination of uncertainty contribution associated with self-heating of EUT, resistance of EUT is measured at working current I and then at  $I\sqrt{2}$ . Standard uncertainty is calculated as difference between resistance of EUT at working current I and  $I\sqrt{2}$  divided by  $2\sqrt{3}$ . Self-heating of EUT is determined at 0 °C.

# 7.7 Determination of uncertainty contribution associated with hysteresis of EUT

For determination of uncertainty contribution associated with hysteresis of EUT, resistance of EUT is measured at the same temperature point (about at midpoint of calibration range) in heating and then in cooling mode. Standard uncertainty is calculated as difference between resistance of EUT measured in two modes divided by  $\sqrt{3}$ .

Hysteresis is measured on customer request. In other case, into calibration certificate for resistance thermometers the following sentence should be added:

"The resistance thermometer was calibrated from low temperatures to high temperatures. If the thermometer will be used in reverse temperature sequence, it should be noted that measurement uncertainty may be larger because of hysteresis."

#### 8. Measurement uncertainty in calibration of resistance thermometers

1) For deviation  $\Delta t$  between temperature measured with SPRT  $t_{std}$  and temperature measured with EUT  $t_{ind}$  using **standard CVD-coefficients** the following mathematic model is obtained:

$$\Delta t = t_{ind} - t_{std} + \delta t_{std} + \delta t_{stdA} + \delta t_{ind} + \delta t_{indA} + \delta t_{inhom} + \delta t_{instab} + \delta t_{stab} + \delta t_{sheat}$$

where

 $\delta t_{std}$ : uncertainty contribution of type B is determined from data of calibration certificates of SPRT ( $\delta t_{cal,SPRT}$ ) and thermometer readout ( $\delta t_{cal,std}$ ):

 $\delta t_{std} = \delta t_{cal\ SPRT} + \delta t_{cal.\ std}$ .

Standard uncertainty associated with SPRT:

$$u(\delta t_{cal\ SPRT}) = \delta t_{cal\ SPRT}/2.$$

 $\delta t_{cal\,SPRT}$  - expanded uncertainty of SPRT.

Standard uncertainty associated with thermometer readout:

$$u(\delta t_{cal std}) = \delta t_{cal std} / \sqrt{3}$$

 $\delta t_{calstd}$  – accuracy of thermometer readout using internal standard resistor. Or

$$u(\delta t_{cal std}) = \delta t_{cal std}/2$$
,

when  $\delta t_{calstd}$  – expanded uncertainty of thermometer readout from its calibration certificate.

 $\delta t_{stdA}$ : uncertainty contribution of type A for SPRT:

$$u\left(\delta t_{stdA}\right) = \sqrt{\frac{\sum_{i=1}^{n} (t_{std\ i} - \overline{t_{std}})^{2}}{n\left(n-1\right)}},$$

where,

n – number of measurements;

i – number of single measurement.

 $\delta t_{indA}$ : uncertainty contribution of type A for EUT:

$$u\left(\delta t_{ind}\right) = \sqrt{\frac{\sum_{i=1}^{n} (t_{indi} - \overline{t_{ind}})^{2}}{n\left(n-1\right)}},$$

where.

n – number of measurements;

i – number of single measurement.

 $\Delta t_{ind}$ : uncertainty contribution of type B for EUT ohmmeter. Standard uncertainty is calculated by the following way:

$$u(\delta t_{cal}) = \delta t_{cal} / 2.$$

 $\delta t_{cal}$  - expanded uncertainty from calibration certificate of EUT ohmmeter. Or

$$u(\delta t_{cal}) = \delta t_{cal} / \sqrt{3}$$

if  $\delta t_{cal}$  – accuracy for EUT ohmmeter from its technical specifications.

 $\delta t_{inhom}$ : uncertainty contribution of type B associated with temperature spatial inhomogeneity of liquid bath or furnace. Standard uncertainty:

$$u(\delta t_{inhom}) = \delta t_{inhom} / \sqrt{3}$$
.

 $\delta t_{instab}$ : uncertainty contribution of type B associated with temperature instability of liquid bath or furnace. Standard uncertainty:

$$u(\delta t_{instab}) = \delta t_{instab} / \sqrt{3}$$
.

 $\delta t_{stab}$ : uncertainty contribution of type B associated with stability of EUT at 0 °C. Standard uncertainty:

$$u(\delta t_{stab}) = \delta t_{stab} / 2\sqrt{3}$$
.

 $\delta t_{sheat}$ : uncertainty contribution of type B associated with self-heating of EUT. Standard uncertainty:

$$\mathbf{u}(\delta t_{sheat}) = (t(I) - t(I\sqrt{2}))/2\sqrt{3}.$$

Combined standard uncertainty for deviation  $\Delta t$  between temperature measured with SPRT  $t_{std}$  and temperature measured with EUT  $t_{ind}$  using standard CVD-coefficients:

$$\mathbf{u_{c}}(\Delta t) = \sqrt{u^{2}(\delta t_{cal\ SPRT}) + u^{2}(\delta t_{cal\ std}) + u^{2}(\delta t_{std\ A}) + u^{2}(\delta t_{ind}) + u^{2}(\delta t_{indA}) + u^{2}(\delta t_{inhom}) + u^{2}(\delta t_{instab}) + u^{2}(\delta t_{stab}) + u^{2}(\delta t_{sheat})}$$

And expanded uncertainty:

$$U(\Delta t) = k \cdot u_c(\Delta t),$$

k – coverage factor, equal 2.

2) For deviation  $\Delta t$  between temperature measured with SPRT  $t_{std}$  and temperature measured with EUT  $t_{ind}$  using **individual CVD-coefficients**.

In this case, uncertainty of temperature under determination of individual function R(t) is calculated according to uncertainty propagation (JCGM 100, DKD-R-5-6) by the following way:

$$u^2\big(R(t_i)\big) = \left(\frac{\partial R}{\partial A}u(A)\right)^2 + \left(\frac{\partial R}{\partial B}u(B)\right)^2 + \left(\frac{\partial R}{\partial R_0}u(R_0)\right)^2 + \\ 2\cdot \left(\frac{\partial R}{\partial A}\cdot\frac{\partial R}{\partial B}\cdot u_{AB} + \frac{\partial R}{\partial A}\cdot\frac{\partial R}{\partial R_0}\cdot u_{AR_0} + \frac{\partial R}{\partial B}\cdot\frac{\partial R}{\partial R_0}\cdot u_{BR_0}\right), \qquad \text{для } \boldsymbol{t} \geq \boldsymbol{0} \circ C$$

$$U(R(t_i)) = 2 \cdot u(R(t_i)) \quad U(t_i) = \frac{U(R(t_i))}{\left(\frac{\partial R}{\partial t_i}\right)}, \quad i = 1 \dots 5.$$

Uncertainty for individual coefficients is evaluated by the following way (this example for positive range):

$$u^{2}(B) = \left(\frac{\partial B}{\partial R_{1}}u(R_{1})\right)^{2} + \left(\frac{\partial B}{\partial R_{2}}u(R_{2})\right)^{2} + \left(\frac{\partial B}{\partial R_{3}}u(R_{3})\right)^{2} + \left(\frac{\partial B}{\partial R_{4}}u(R_{4})\right)^{2} + \left(\frac{\partial B}{\partial R_{5}}u(R_{5})\right)^{2} + \left(\frac{\partial B}{\partial t_{1}}u(t_{1})\right)^{2} + \left(\frac{\partial B}{\partial t_{2}}u(t_{2})\right)^{2} + \left(\frac{\partial B}{\partial t_{3}}u(t_{3})\right)^{2} + \left(\frac{\partial B}{\partial t_{4}}u(t_{4})\right)^{2} + \left(\frac{\partial B}{\partial t_{5}}u(t_{5})\right)^{2}.$$

$$\begin{split} u^2(A) &= \left(\frac{\partial A}{\partial R_1} u(R_1)\right)^2 + \left(\frac{\partial A}{\partial R_2} u(R_2)\right)^2 + \left(\frac{\partial A}{\partial R_3} u(R_3)\right)^2 + \left(\frac{\partial A}{\partial R_4} u(R_4)\right)^2 + \\ &+ \left(\frac{\partial A}{\partial R_5} u(R_5)\right)^2 + \left(\frac{\partial A}{\partial t_1} u(t_1)\right)^2 + \left(\frac{\partial A}{\partial t_2} u(t_2)\right)^2 + \left(\frac{\partial A}{\partial t_3} u(t_3)\right)^2 + \left(\frac{\partial A}{\partial t_4} u(t_4)\right)^2 + \left(\frac{\partial A}{\partial t_5} u(t_5)\right)^2. \end{split}$$

$$\begin{split} u^2(R_0) &= \left(\frac{\partial R_0}{\partial R_1} u(R_1)\right)^2 + \left(\frac{\partial R_0}{\partial R_2} u(R_2)\right)^2 + \left(\frac{\partial R_0}{\partial R_3} u(R_3)\right)^2 + \left(\frac{\partial R_0}{\partial R_4} u(R_4)\right)^2 + \\ &+ \left(\frac{\partial R_0}{\partial R_5} u(R_5)\right)^2 + \left(\frac{\partial R_0}{\partial t_1} u(t_1)\right)^2 + \left(\frac{\partial R_0}{\partial t_2} u(t_2)\right)^2 + \left(\frac{\partial R_0}{\partial t_3} u(t_3)\right)^2 + \left(\frac{\partial R_0}{\partial t_4} u(t_4)\right)^2 + \left(\frac{\partial R_0}{\partial t_5} u(t_5)\right)^2. \end{split}$$

And covariations associated with individual coefficients:

$$u_{AB} = \frac{\partial A}{\partial R_1} \cdot \frac{\partial B}{\partial R_2} u^2(R_1) + \frac{\partial A}{\partial R_2} \cdot \frac{\partial B}{\partial R_2} u^2(R_2) + \frac{\partial A}{\partial R_3} \cdot \frac{\partial B}{\partial R_3} u^2(R_3) +$$

$$\begin{split} &+\frac{\partial A}{\partial R_4}\cdot\frac{\partial B}{\partial R_4}u^2(R_4)+\frac{\partial A}{\partial R_5}\cdot\frac{\partial B}{\partial R_5}u^2(R_5)+\frac{\partial A}{\partial t_1}\cdot\frac{\partial B}{\partial t_1}u^2(t_1)+\frac{\partial A}{\partial t_2}\cdot\frac{\partial B}{\partial t_2}u^2(t_2)+\\ &+\frac{\partial A}{\partial t_3}\cdot\frac{\partial B}{\partial t_3}u^2(t_3)+\frac{\partial A}{\partial t_4}\cdot\frac{\partial B}{\partial t_4}u^2(t_4)+\frac{\partial A}{\partial t_5}\cdot\frac{\partial B}{\partial t_5}u^2(t_5).\\ &u_{AR_0}=\frac{\partial A}{\partial R_1}\cdot\frac{\partial R_0}{\partial R_1}u^2(R_1)+\frac{\partial A}{\partial R_2}\cdot\frac{\partial R_0}{\partial R_2}u^2(R_2)+\frac{\partial A}{\partial R_3}\cdot\frac{\partial R_0}{\partial R_3}u^2(R_3)+\\ &+\frac{\partial A}{\partial R_4}\cdot\frac{\partial R_0}{\partial R_4}u^2(R_4)+\frac{\partial A}{\partial R_5}\cdot\frac{\partial R_0}{\partial R_5}u^2(R_5)+\frac{\partial A}{\partial t_1}\cdot\frac{\partial R_0}{\partial t_1}u^2(t_1)+\frac{\partial A}{\partial t_2}\cdot\frac{\partial R_0}{\partial t_2}u^2(t_2)+\\ &+\frac{\partial A}{\partial t_3}\cdot\frac{\partial R_0}{\partial t_3}u^2(t_3)+\frac{\partial A}{\partial t_4}\cdot\frac{\partial R_0}{\partial t_4}u^2(t_4)+\frac{\partial A}{\partial t_5}\cdot\frac{\partial R_0}{\partial t_5}u^2(t_5).\\ &u_{BR_0}=\frac{\partial B}{\partial R_1}\cdot\frac{\partial R_0}{\partial R_1}u^2(R_1)+\frac{\partial B}{\partial R_2}\cdot\frac{\partial R_0}{\partial R_2}u^2(R_2)+\frac{\partial B}{\partial R_3}\cdot\frac{\partial R_0}{\partial R_3}u^2(R_3)+\\ &+\frac{\partial B}{\partial R_4}\cdot\frac{\partial R_0}{\partial R_4}u^2(R_4)+\frac{\partial B}{\partial R_5}\cdot\frac{\partial R_0}{\partial R_5}u^2(R_5)+\frac{\partial B}{\partial t_1}\cdot\frac{\partial R_0}{\partial t_1}u^2(t_1)+\frac{\partial B}{\partial t_2}\cdot\frac{\partial R_0}{\partial t_2}u^2(t_2)+\\ &+\frac{\partial B}{\partial t_3}\cdot\frac{\partial R_0}{\partial t_3}u^2(t_3)+\frac{\partial B}{\partial t_4}\cdot\frac{\partial R_0}{\partial t_4}u^2(t_4)+\frac{\partial B}{\partial t_5}\cdot\frac{\partial R_0}{\partial t_5}u^2(t_5). \end{split}$$

For negative range of temperatures, coefficient C is additionally calculated and included to the above formulas.

For deviation function, the same calculations are valid. In general form, formulas are written as follows:

$$W_r = W - \Delta W$$

 $W_r$  - reference function,

W - relative resistance,

 $\Delta W$  - deviation function.

$$u^{2}(W_{r}) = \left(\frac{\partial W_{r}}{\partial W}u(W)\right)^{2} + \left(\frac{\partial W_{r}}{\partial(\Delta W)}u(\Delta W)\right)^{2},$$

$$u^{2}(W_{i}) = \left(\frac{\partial W}{\partial R_{i}}u(R_{i})\right)^{2} + \left(\frac{\partial W}{\partial R_{TPW}}u(R_{TPW})\right)^{2},$$

$$u^{2}(\Delta W) = \sum_{j=1}^{m} \left(\frac{\partial \Delta W}{\partial c_{j}}u(c_{j})\right)^{2} + 2 \cdot \sum_{\substack{j=1\\at \ j \neq k}}^{m-1} \sum_{k=j+1}^{m} \frac{\partial \Delta W}{\partial c_{j}} \cdot \frac{\partial \Delta W}{\partial c_{k}} \cdot u_{c_{j}c_{k}},$$

$$u^{2}(c_{k}) = \sum_{i=1}^{n} \left(\frac{\partial c_{k}}{\partial W_{r}} \cdot \frac{\partial W_{r}}{\partial T_{i}}u(T_{i})\right)^{2} + \sum_{i=1}^{n} \left(\frac{\partial c_{k}}{\partial W_{i}}u(W_{i})\right)^{2},$$

$$u_{c_j c_k} = \sum_{\substack{j=1\\at\ j \neq k}}^{m-1} \sum_{k=j+1}^{m} \left[ \frac{\partial c_j}{\partial W_r} \cdot \frac{\partial c_k}{\partial W_r} \cdot \left( \frac{\partial W_r}{\partial T_i} u(T_i) \right)^2 + \frac{\partial c_j}{\partial W_i} \cdot \frac{\partial c_k}{\partial W_i} \cdot u^2(W_i) \right],$$

 $c_k$ ,  $c_j$  - coefficients for deviation function, calculated by approximation measured data ( $T_i$  and  $W_i$ ) using least square method.

i – number of calibration points,

j, k – number of coefficients for deviation function;

 $R_{TPW}$  – resistance at temperature of water triple point,  $R_{TPW} = 0.99996 \cdot R_0$ ;

 $R_0$  – resistance at temperature of ice melting 0 °C.

$$U(W_r) = 2 \cdot u(W_r)$$
  $U(t_i) = U(T_i) = \frac{U(W_r(T_i))}{\left(\frac{\partial W_r}{\partial T_i}\right)}$ ,  $i = 1 \dots n$ .

But for both cases, calculation of uncertainty may be simplified and assumed as: Uncertainty in any point within calibration range can be assumed to be equal to the sum of maximum uncertainty in the calibration range  $(u_c(\Delta t))_{max}$  and maximum deviation from individual function in calibration points  $\Delta_{max}$ :

$$U(t) = 2\sqrt{[(\mathbf{u}_{c}(\Delta t))_{\text{max}}]^{2} + [\Delta_{\text{max}}]^{2}}.$$

More complicative option is to give uncertainty propogation function in calibration certificate:

U(t) = f(t) for CVD interpolation equation and

U(t) = f(W) for deviation function.

#### **Uncertainty budget**

Contribution	Source	Distribution	Coefficient of sensitivity
1. SPRT	Calibration certificate	Normal	-1
2.Thermometer readout for SPRT	Calibration certificate/ specifications	Normal/ rectangular $(\sqrt{3})$	-1
3.Standard deviation for SPRT	Measurement data		-1
4.Temperature standard deviation for EUT	Measurement data		$1/\left(\frac{\partial R}{\partial t}\right) \text{ or } 1/\left(\frac{\partial W_r}{\partial T_i}\right)$
5.Thermometer readout (precision ommeter) for EUT	Calibration certificate/ specifications	Normal/ rectangular ( $\sqrt{3}$ )	$1/\left(\frac{\partial R}{\partial t}\right)$ or $1/\left(\frac{\partial W_r}{\partial T_i}\right)$
6.Stability of EUT at 0 °C	Measurement data	rectangular $(2\sqrt{3})$	$1/\left(\frac{\partial R}{\partial t}\right) \text{ or } 1/\left(\frac{\partial W_r}{\partial T_i}\right)$
7.Self-heating of EUT	Measurement data	rectangular $(2\sqrt{3})$	$1/\left(\frac{\partial R}{\partial t}\right) \text{ or } 1/\left(\frac{\partial W_r}{\partial T_i}\right)$

8.Instability of temperature in	Investigation	rectangular ( $\sqrt{3}$ )	-1
liquid bath or block calibrator			
9.Spatial inhomogeneity of liquid	Investigation	rectangular ( $\sqrt{3}$ )	-1
bath or block calibrator			
10.Maximum deviation from	Measurement		1
individual function	data		

#### Example of uncertainty budget in calibration of platinum resistance thermometer

**EUT:** 

Pt100, electrotherm, Germany Calibration range: 0 °C to 200 °C

Calibration points: (0/30/50/100/150/200) °C

Total length of the EUT: 31,5 cm

Immersion depth: 20 cm Measuring current: 0,5 mA Calibration point: 100 °C Standard and additional equipment

**SPRT:** 

Pt<sub>25</sub>, Fluke 5699

Range of measurement: (-200) °C to 660 °C

Immersion depth: 20 cm

Expanded uncertainty from calibration certificate:

0,0042 K

Thermometer readout:

Fluke 1575A

Accuracy of temperature measurement with internal

reference resistor: ±0,002 °C

Accuracy of resistance measurement: ±8 ppm

Oil bath:

1. Fluke 6331

Range: 40 °C to 300 °C

Inhomogeneity: 0,007 K (for 50 °C, 100 °C and

150 °C)

0,017 K (for 200 °C)

Instability: 0,0031 K (for 50 °C)

0,0061 K (for 100 °C and 150 °C)

0,0132 K (for 200 °C)

EUT and SPRT are set in the bath at the same depth and distance  $\approx 1$  cm.

2. Fluke 7381

Range: (-80) °C to 110 °C

Inhomogeneity: 0,0054 K (при 30 °C)

Instability: 0,003 K (при 30 °C).

3. Ice point is realized in Dewar vessel. Instability

and inhomogeneity are negligible.

# **1.** For deviation $\Delta t$ between temperature measured with SPRT $t_{std}$ and temperature measured with EUT $t_{ind}$ using **standard CVD-coefficients**:

Input	Evaluation	Divider	Standard	Sensitivity	Contribution	Contribution
quantity	of input		uncertainty	coefficient	$c_i^2 \cdot u_i^2$ , K <sup>2</sup>	$c_i^2 \cdot u_i^2, \%$
	quantity		$u_j$	$c_{\rm j}$		, ,
$\delta t_{cal\ SPRT}$	0,0042 K	2	0,0021 K	-1	441·10 <sup>-8</sup>	11,6
$\delta t_{cal. \ std}$	0,002 K	$\sqrt{3}$	0,0012 K	-1	$144 \cdot 10^{-8}$	3,8
$\delta t_{stdA}$	0,0003 K	1	0,0003 K	-1	9·10 <sup>-8</sup>	0,2

$\delta t_{indA}$	0,00012 Ω	1	$0,00012~\Omega$	$1/0,380=2,63 \text{ K/}\Omega$	$10 \cdot 10^{-8}$	0,3
$\delta t_{ind}$	$0,0008 \Omega$	$\sqrt{3}$	$0,00046~\Omega$	$1/0,380=2,63 \text{ K/}\Omega$	146,4·10 <sup>-8</sup>	3,9
$\delta t_{sheat}$	0,0003 Ω	$2\sqrt{3}$	$0,00009 \Omega$	$1/0,380=2,63 \text{ K/}\Omega$	5,6·10 <sup>-8</sup>	0,1
$\delta t_{stab}$	0,002 Ω	$2\sqrt{3}$	$0,00056~\Omega$	$1/0,391=2,56 \text{ K/}\Omega$	$205,5\cdot 10^{-8}$	5,4
$\delta t_{inhom}$	0,007 K	$\sqrt{3}$	0,004 K	-1	1600·10 <sup>-8</sup>	42,3
$\delta t_{instab}$	0,0061 K	$\sqrt{3}$	0,0035 K	-1	1225·10 <sup>-8</sup>	32,4
				$\sum c_j^2 \cdot u_j^2$	3786,5·10 <sup>-8</sup>	100,0
Combined standard uncertainty, K				$\mathbf{u}_{\mathrm{c}}(\Delta t)$	0,0062	
Expanded uncertainty (k=2), K				U(∆t)	0,013	

**2.** For deviation  $\Delta t$  between temperature measured with SPRT  $t_{std}$  and temperature measured with EUT  $t_{ind}$  using **individual CVD-coefficients or deviation function**  $\Delta \mathbf{W}$ .

Uncertainty in calibration point 100 °C and in any point within calibration range can be assumed to be equal to the sum of maximum uncertainty in the calibration range (0,026 K for 200 °C) and maximum deviation from individual function in calibration points (0,003 K):

$$U(t) = 2\sqrt{(0.026/2)^2 + 0.003^2} = 0.027 \text{ K}.$$

#### 9. Statement of calibration results

Calibration results are stated in the following tables:

Standard thermometer's readings, °C	Readings of the thermometer under test, $\Omega$	Temperatures of EUT according to <b>IEC 60751:2008</b> , °C	<i>∆t</i> , K	U( <i>∆t</i> ), K
				$U(\Delta t)=k \cdot u_c(\Delta t)$

And additional information, if individual CVD-coefficients are calculated:

$$R(t)=R_0\cdot(1+A\cdot t+B\cdot t^2+C\cdot(t-100^{\circ}C)\cdot t^3)$$
, for  $t>0^{\circ}C$  C=0

Individual coefficients: R<sub>0</sub>, A, B, C.

Or if the deviation function is calculated:

$$W_r = W - \Delta W$$
,  $\Delta W = f(W, c_i)$  and  $c_i$ .

Standard thermometer's readings, °C	Readings of the thermometer under test, $\Omega$	Temperatures of EUT according to individual CVD coefficients or calculated using deviation, °C	$\Delta t$ (not zero in case of approximation),	U( <i>∆t</i> )= <i>U</i> ( <i>t</i> ), K
				$U(t) = 2\sqrt{[(\mathbf{u}_{\mathrm{c}}(\Delta t))_{\mathrm{max}}]^2 + [\Delta_{\mathrm{max}}]^2}$ unu $U(t) = f(t)$ $U(t) = f(W)$

# Determination of instability and spatial inhomogeneity of liquid baths, block calibrators, climatic chambers and furnaces

For operation in one temperature range, the calibration needs to be carried out at least three temperatures for the range of use.

#### **Definitions**

#### Measuring location:

A measuring location is the spatial position in which a temperature sensor is arranged in the useful volume for calibration. A measuring location thus is a small volume which is defined by the dimensions of the sensor elements and their positioning accuracy (i. e. approx. 5 cm max. in each dimension). If the measurements are carried out in one location only, the calibration result is valid for this location only. Extrapolation to a larger volume is not admissible.

#### Useful volume:

The useful volume of a liquid bath is the partial volume of the liquid bath spanned by the measuring locations of the sensors used for calibration. According to the arrangement of the measuring locations, the useful volume can considerably differ from the total volume of the liquid bath. The calibration of the liquid bath is basically valid only for this useful volume. The minimum requirements for the position of the measuring location must be fulfilled.

# Reference measuring location:

The reference measuring location is the position in the useful volume for which the difference between bath's temperature and the indicated values is stated. In most cases, the geometrical centre of the useful volume is selected as reference location. Other definitions for the reference measuring location are also possible. The position of reference measuring location must be noted.

#### Standards and equipment

- 1. PRT
- 2. temperature converter
- 3. thermocouples, type S
- 4. millivoltmeter

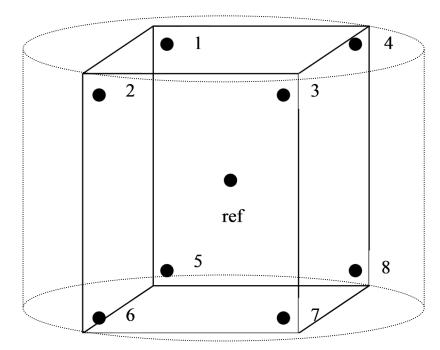
#### Valid documents

- 1. Guideline DKD-R 5-7 «Calibration of Climatic Chambers», 2004.
- 2. working instruction (operation manual, specifications) for bath to be investigated.

#### **Installation of temperature probes**

- 1) For the spatial number of measuring locations for the calibration of the useful volume, the following requirements are valid:
  - For useful volumes < 2000  $\ell$ , at least nine measuring locations are to be selected, i. e. the measuring locations form the corner points and the spatial centre of a cuboid spanning the useful volume (nine measuring locations in all). Other positions are also possible but it must be ensured that the useful volume is enclosed by the volume spanned by the measuring points. These requirements are valid for baths of rectangular construction and for round or oval cross-section (with inscribed rectangular in it).

The dimensions of the total liquid bath's volume and the selected position of the measuring points must be given in a sketch. The measuring locations must be numbered. An example of the sketch is shown in the figure below:



Spatial inhomogeneity is valid for the useful volume enclosed by the measuring points. Extrapolations of the measurement result beyond the volume spanned by the measuring locations are not admissible.

- 2) For the spatial number of measuring locations for the calibration of individual measuring locations, the following requirements are valid:
  - the calibration can be performed in individual measuring locations only. In this case, however, some uncertainty components will not be determined and not be taken into account. The result then will be valid only for these locations, not however for the complete liquid bath. As calibration item "measuring location(s) in the liquid bath" is to be stated. The contribution of the local spatial inhomogeneity in the measuring locations must be determined for each measuring location using two thermometers (reference and auxiliary) arranged with a spacing of approx. 2 cm to 5 cm (at least a spacing in accordance with the active sensor length has to be selected). One of these thermometers (reference) is arranged in the position defined for the statement of the calibration result (measuring location) and the other one (auxiliary) at the necessary distance.

In the calibration for more than one measuring location, the use of two thermometers in one measuring location can be dispensed with if the uncertainty contribution due to the local inhomogeneity and the positioning accuracy is adequately estimated from the difference of the thermometers for the individual measuring locations.

The dimensions of the total liquid bath's volume and the selected position of the measuring points must be given in a sketch.

## Determination of temperature spatial inhomogeneity $\delta t_{inhom}$

The spatial inhomogeneity is determined as the maximum deviation of mean value at a corner or at wall measuring location from mean value at the reference location (in most cases in the centre of the useful volume). It is to be determined for all calibration temperatures. The spatial inhomogeneity is investigated only in calibrations for a useful volume by method 1. For the calculation of the spatial inhomogeneity, at least 10 temperature values measured with the SPRT at the reference location and at least 10 temperature values measured with the SPRT (or PRT) at each measuring location are to be recorded at more or less constant time intervals. Then mean values at each measuring location and at reference location are calculated. And then the spatial inhomogeneity is determined.

In calibrations according to method 2, local inhomogeneity is determined to estimate the uncertainty contribution due to inhomogeneity (see sections 4 and 5). The local inhomogeneity is determined as the maximum deviation of mean temperature value measured with the reference SPRT from mean temperature value measured with the auxiliary SPRT (or PRT) at one measuring location. For the calculation of the local inhomogeneity, at least 10 temperature values measured with the reference SPRT and at least 10 temperature values measured with the auxiliary SPRT (or PRT) at one measuring location are to be recorded at more or less constant time intervals in the following sequence: indication of the reference SPRT, then indication of the auxiliary SPRT (or PRT), then indication of the auxiliary SPRT (or PRT) again, indication of the reference SPRT, then indication of the reference SPRT again, etc. Then the mean value of the indications of the reference SPRT and the mean value of the indications of the auxiliary SPRT (or PRT) are calculated. And then the local inhomogeneity is determined.

In calibrations according to method 2 for more than one measuring location, the local inhomogeneity is determined as the maximum deviation of mean temperature values measured with the SPRTs at measuring locations. For the calculation of the local inhomogeneity, at least 10 temperature values measured with the SPRTs at each measuring location, respectively, are to be recorded at more or less constant time intervals. Then the mean values of the indications of the SPRTs at each measuring location are calculated. And then the local inhomogeneity is determined.

All measurements are performed after steady-state conditions have been reached. Steady-state conditions are considered to be reached when systematic variations of temperature are no longer observed.

The spatial inhomogeneity is equivalent to the half-width of a rectangularly distributed contribution with the expected value 0.

In calibration by method 1:

$$|\delta t_{inhom}| = Max |t_{ref} - t_i|$$

where,  $t_{ref}$  – temperature mean value at reference measuring location measured with the SPRT;  $t_i$  – temperature mean value at the  $i^{th}$  measuring location measured with the SPRT or PRT;  $1 \le i \le n$ , where n is a number of measuring locations spanning the useful volume.

In calibration by method 2:

a) for single individual measuring location:

$$|\delta t_{inhom}| = |t_{ref} - t_{auxil}|$$

where,  $t_{ref}$  – temperature mean value measured with the reference SPRT at the measuring location;

 $t_{auxil}$  – temperature mean value measured with the auxiliary SPRT or PRT at the measuring location.

b) for more than one individual measuring location:

$$|\delta t_{inhom}| = |t_{std}| - t_{std}|$$

where,  $t_{std\,i}$  – temperature mean value measured with the SPRT at the  $i^{st}$  measuring location;  $t_{std\,j}$  – temperature mean value measured with the SPRT at the  $j^{st}$  measuring location;  $1 \le i \le n$  and  $1 \le j \le n$ , where n is a number of individual measuring locations.

For the associated standard uncertainty the following equation is obtained:

$$u(\delta t_{inhom}) = |\delta t_{inhom}|/\sqrt{3}$$
.

#### Determination of temperature temporal instability $\delta t_{instab}$

The temporal instability is determined from the recorded temporal variation of temperature over a period of time of at least 30 min after steady-state conditions have been reached. Steady-state conditions are considered to be reached when systematic variations of temperature are no longer measured.

For the measurement of the temporal instability, at least 30 temperature values measured with the SPRT are to be recorded in 30 min at more or less constant time intervals. The measurement needs to be performed at least for the reference measuring location of the useful volume (method 1) or for the each individual measuring location (method 2), respectively, and for each calibration temperature.

The maximum deviation over 30 min from the temporal mean value is formulated as the half-width of a rectangularly distributed contribution with the expected value 0.

$$|\delta t_{instab}| = Max |t_{ref} - t_i|$$

where,  $t_{ref}$  – temperature mean value measured with the SPRT for 30 minutes;  $t_i - i^{th}$  temperature value measured with the SPRT for 30 minutes;

For the associated standard uncertainty the following equation is obtained:

$$u(\delta t_{instab}) = |\delta t_{instab}| / \sqrt{3}$$
.

There is a one of possible variants for calculation of temperature instability and inhomogeneity in the bath (EUT). Instability can be also evaluated as standard deviation  $(2\sigma)$ , and inhomogeneity – not relatively of the center of useful volume, but as maximum temperature deviation between measuring locations.

In case of baths with equalizing block and furnaces instability and inhomogeneity (axial and radial) is investigated.

Measurement of radial inhomogeneity: 10 measurements of temperature are carried out in each used block's well at immersion for total depth of wells. Radial inhomogeneity is maximum deviation between the biggest and the least temperature mean values in block's wells.

Measurement of instability: 10 measurements of temperature during 10 minutes are carried out at the same conditions as radial inhomogeneity. Instability is maximum deviation from maximum deviations between single measurement results and mean temperature values for each block's well correspondently.

Determination of axial inhomogeneity: measurements are carried out under immersion of PRT (SPRT) or TC for total depth of the block's well and under gradual decreasing of immersion

depth for several centimeters (it is dependent on depth of the block's well). As minimum in 2 positions: under immersion for total depth and under immersion of sensor only (the minimum immersion). 10 temperature measurements are made in each position. Maximum deviation between the biggest and the least mean temperature values at correspondent immersions is assumed as axial inhomogeneity. Axial inhomogeneity is investigated in one block's well only. All measurements are carried out at 3 temperature points of working range of liquid bath, block calibrator or furnace: minimum, medium and maximum one.

Chosen measurement wells should be marked and numbered in a sketch.

#### **INFORMATION**

#### **COOMET Recommendation R/T/32:2018**

- 1. Coordinator: Center for standardization and metrology under the Ministry of economy of the Kyrgyz Republic (CSM under MoE KR)
- 2. COOMET project 633/KG/14
- 3. The Recommendation was approved at the  $28^{th}$  COOMET Committee Meeting (Sarajevo, Bosnia and Herzegovina, April 11-12, 2018)
- 4. The Recommendation was agreed and approved by members and chairperson at the Meeting of TC 1.10 COOMET "Thermometry and thermal physics" 8-9<sup>th</sup> of November, 2017, Sarajevo, Bosnia and Herzegovina.