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MODEL FOR UNCERTAINTY ESTIMATION IN COMPARISON CALIBRATION OF THERMOCOUPLES

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Abstract - The objective of this paper is to present the methodology for estimation of measurement uncertainties in comparison calibration of thermocouples used at Laboratory for Process Measurements (LPM). The methodology is applied for comparison calibration of rare-metal and industrial base-metal thermocouples within temperature range from –20°C to 660°C with LPM standard/working standard platinum resistance thermometers and from 600°C to 1050°C with LPM standard/working standard thermocouples.

Keywords: thermocouples, comparison calibration, uncertainty

1. INTRODUCTION

Rare metal thermocouples play no direct role in the realization of current temperature scale ITS-90 any more, like they used to in IPTS-68, since they have been replaced by HTSPRT-s (High Temperature Standard Platinum Resistance Thermometers). However, most calibration laboratories that do not calibrate directly on the ITS-90, use STC-s (Standard Thermocouples) as their calibration standards above aluminium melting point, because of their robustness, easiness of use and lower price. These are also the reasons why in industrial laboratories and manufacturing plants both rare and base metal thermocouples are the only choice for contact thermometry at higher temperatures and a very frequent choice for all other contact thermometry ranges. Since thermocouples are almost exclusively calibrated by comparison method, estimation of uncertainties in calibration procedures is an important step in establishing traceability of those temperature sensors.

2. MODELLING METHODOLOGY

The comparison method consists of measuring the emf (electromotive force) of the tested thermometer in an isothermal medium, whose temperature is determined by a calibrated (traceable to national standards) reference thermometer.
In order to evaluate uncertainties in comparison calibration of thermocouples appropriate measurement model should be established.

Temperature t_x of the hot junction of the thermocouple to be calibrated is calculated using following relations:

- temperature t_x measured with SPRT

$$t_x = t_S(R_{iS} + \delta R_{c1} + \delta R_{c2} + \delta R_{c3}) + \delta t_D + \delta t_F$$
$$t_x \cong t_S(R_{iS}) + C_S \cdot \delta R_{c1} + C_S \cdot \delta R_{c2} + C_S \cdot \delta R_{c3} + \delta t_D + \delta t_F \qquad (1)$$

- temperature t_x measured with STC

$$t_x = t_S(V_{iS} + \delta V_{c1} + \delta V_{c2} + \delta V_{c3} + \delta V_R - \frac{\delta t_{0S}}{C_{S0}}) + \delta t_D + \delta t_F$$
$$t_x \cong t_S(V_{iS}) + C_S \cdot \delta V_{c1} + C_S \cdot \delta V_{c2} + C_S \cdot \delta V_{c3} + C_S \cdot \delta V_R - \frac{C_S}{C_{S0}} \cdot \delta t_{0S} + \delta t_D + \delta t_F$$

(2)

where:

$R_{iS} \backslash V_{iS}$	- resistance of SPRT read on measuring bridge \ emf of the STC read on multimeter
$t_S(R_{iS} \backslash V_{iS})$	- temperature of the SPRT\STC obtained from the calibration relation for the used reference thermometer
$\delta R_{c1} \backslash \delta V_{c1}$	- correction obtained from the calibration of the bridge \ multimeter
$\delta R_{c2} \backslash \delta V_{c2}$	- correction linked to the drift of bridge \ multimeter
$\delta R_{c3} \backslash \delta V_{c3}$	- correction linked to the resolution of the bridge \ multimeter
δt_D	- correction linked to the drift of the SPRT \ STC
δt_F	- correction linked to the non-uniformity of the temperature profile in the equalising block and temperature stability of the thermal source
δV_R	-correction linked to the influence of the ambient parameters and connections
δt_{0S}	- correction due to deviation of the ice/water bath temperature from 0°C
C_S	- sensitivity of the standard thermometer at calibration temperature (°C/Ω \ °C/mV)
C_{S0}	- sensitivity of the STC at 0°C

It should be noted that the measuring bridge referred to in this paper directly reads the resistance.

Electromotive force, V_x, generated by the thermocouple to be calibrated with the cold junction at 0°C is calculated using the relation:

$$V_x = V_{ix} + \delta V_{c1} + \delta V_{c2} + \delta V_{c3} + \delta V_R + \delta V_L + \delta V_H + \frac{t - t_x}{C_x} - \frac{\delta t_{0x}}{C_{x0}} \quad (3)$$

where :

V_{ix} - emf of the calibrated thermocouple read on multimeter

δV_{c1} - correction obtained from the calibration of the multimeter

δV_{c2} - correction linked to the drift of multimeter

δV_{c3} - correction linked to the resolution of the multimeter

δV_R -correction linked to the influence of the ambient parameters and connections

δV_L -correction linked to the compensation/extension cable

δV_H -correction due to inhomogeneity of the thermocouple wires

$\Delta t=t-t_x$ - deviation of the calibration point from the temperature of thermal source

t - calibration temperature

t_x - temperature of the thermal source measured with standard thermometer

δt_{0x} - correction due to deviation of the ice/water bath temperature from 0°C

C_x - sensitivity of the calibrated thermocouple at calibration temperature (°C/mV)

C_{x0} - sensitivity of the calibrated thermocouple at 0°C

In equations above $R_{iS}\backslash V_{iS}$ and V_{ix} are derived from at least 10 measurement cycles. To reduce the effects of the drift in the thermal source the following measurement sequence in each measurement cycle should be done:

ref -> cal -> cal -> ref (ms1)

where abbreviations "ref" and "cal" stand for reference thermometer and calibrated thermometer.

Such measurement sequence gives the effect like all measurements are done at the same time, if temperature of thermal source linearly rises or falls.

Reference [1] suggest using two reference thermometers in comparison calibration to check each other and instantly reveal error if one of thermometers malfunctions. In that case measurement sequence looks like:

ref1 -> cal -> ref2 -> ref2 -> cal -> ref1 (ms2)

where ref1 is first and ref2 is second reference thermometer.

Also [1] state that when closest accuracy is required measurements should be made of both forward and reverse polarities. This results in reducing the effect of stray thermal emfs in measuring system which can arise at a point in the measuring circuit where there is a change of temperature at the juncture of dissimilar metals.

However, from each measurement sequence one mean value for reference thermometer and calibrated thermocouple can be calculated and 10 or more such values are then used for calculation of mean values of $t_S(R_{iS}\backslash V_{iS})$ and V_{ix}. Also, these values are used for calculation of type A standard uncertainties of mean values in comparison calibration of thermocouples using well-known relations:

$$u(\overline{t_S}) = \sqrt{\frac{1}{n \cdot (n-1)} \cdot \sum_{i=1}^{n} (t_S^{(i)} - \overline{t_S})^2} \quad (4)$$

and

$$u(\overline{V_x}) = \sqrt{\frac{1}{n \cdot (n-1)} \cdot \sum_{i=1}^{n} (V_x^{(i)} - \overline{V_x})^2} \quad (5)$$

where is:

n - number of measurement cycles

$\overline{t_S}$ - mean temperature of the thermal source obtained from n measurement cycles

$t_S^{(i)}$ - mean thermal source temperature derived from i-th measurement cycle

$\overline{V_x}$ - mean emf generated by tested thermocouple obtained from n measurement cycles

$V_x^{(i)}$ - mean emf generated by tested thermocouple derived from i-th measurement cycle

Maximum deviation of t_S , i.e.

$$stability = max(t_S^{(i)}) - min(t_S^{(i)}),\ i = 1\ldots n$$

could be used to verify stability of the thermal source during measurements and if it is in accordance with an acceptance criterion then the measurements are valid. Otherwise measurements should be repeated. Acceptance criterion for the stability of thermal source mainly depends on laboratory calibration capabilities or can be tied to with accuracy of tested thermocouple (lower accuracy usually requires lower acceptance criterion).

In measurement model other sources of error such as radiation, electromagnetic fields, vibrations, etc have low influence on measurement so they can be ignored.

Also in this measuring model correction linked to the stray heat flow along the standard and reference probe is neglected because there is the assumption that both thermometers are immersed deep enough in thermal source. But, this should be taken into account in case when one of thermometers shows permanent change in indication during slowly increasing of its immersion depth. In this case correction is derived through appropriate extrapolation.

The correction linked to the use of standardised table for determining the temperature corresponding to the generated emf is taken to be zero with negligible uncertainty.

All the model input quantities are taken as non-correlated. Some of those input quantities are easy to understand like $\delta R_{c1}\backslash \delta V_{c1}$ which are taken from last calibration report for specific device. This correction can be present if instrument has some constant value offset in reading, which cannot be zeroed, and that value is determined by calibration with appropriate uncertainty. Also, $\delta R_{c2}\backslash \delta V_{c2}$ presents drift in reading of specific device and trend of such drift can be figured out from last few calibrations again with some uncertainty. Value of $\delta R_{c3}\backslash \delta V_{c3}$ is a matter of instrument design and can be read from manufacturer specification. It is fixed value, for specific range, and in measurement model that value is used as uncertainty.

Considering corrections and uncertainties that come from standard thermometer there is uncertainty in thermometer calibration which can be read from its calibration report (usually expressed with confidence level (CL) of 95%).

Correction linked to the drift of the standard thermometer can be estimated from last few calibrations as in the case with multimeter.

Correction linked to the quality of the thermal source, δt_F, usually is taken as zero but the maximum values of temperature non-uniformity inside equaliser block combined with temperature stability is taken as uncertainty of thermal source with rectangular distribution.

Impurities in water, such as dissolved salts, cause deviation of ice/water bath temperature from 0°C and this deviation, δt_{0S}, should be corrected with uncertainty linked to the determination of bath temperature. Propagation of this correction and associated uncertainty from 0°C to calibration temperature depends on the ratio of the thermocouple sensitivity coefficients as it is outlined in (2). It should be noted that term associated with δt_{0S} in relations (2) and (3) comes with negative sign, because if temperature of ice/water bath is for example lower than 0°C emf generated by thermocouple will be greater than if cold junction of thermocouple was at 0°C. Correction of temperature of ice/water bath, in case when it's lower than 0°C, would be positive, and negative sign in front of δt_{0S} term corrects generated emf in right way.

The measuring circuit should be checked for any residual emfs by measuring voltage when it is short-circuited at the thermocouple connection terminals and maximum absolute value of this reading is taken as voltage correction, δV_R, due to the influence of the ambient parameters and connections. Associated uncertainty should be taken with rectangular distribution.

Correction linked to the compensation/extension cable, δV_L, should be considered only in case when thermocouple to be calibrated is delivered without its own reference junction. Therefore, a suitable compensation/extension cable for this type of thermocouple has to be chosen. One end has to be connected to copper wire, insulated, inserted into a light close-fitting sheath, and immersed in ice/water bath while the other end has to be connected to the thermocouple to be tested.

The uncertainties of calibration associated with the use of compensation/extension leads can be estimated through the following experimental method:

1. compensation/extension cables are short-circuited at one end which actually form a thermocouple;
2. (+) end of tested thermocouple is connected at one of multimeter terminals;
3. (+) end of compensation/extension cable is connected at another terminal of multimeter;
4. (-) end of tested thermocouple and (-) end of compensation/extension cable are short-circuited (this actually forms one of basic thermoelectric circuits for measuring temperature difference);
5. measuring junction of tested thermocouple and "measuring junction" of thermocouple made of compensation/extension cable are brought to same temperatures of 0°C and then of 40°C;
6. any mismatch between wires of tested thermocouple and compensation/extension cable is manifesting in non-zero reading on multimeter, and maximum absolute value of this readings is taken as voltage correction due to compensation/extension cable with rectangular distribution.

The use of thermocouples as temperature sensors is based on the Seebeck´s effect i.e. on the fact that a difference of potentials occurs at the ends of any electrically conductive material made wire if those ends are at different temperatures:

$$de = s(t)\, dt \tag{6}$$

where is:

de - thermovoltage
$s(t)$ - Seebeck´s coefficient
dt - difference between temperatures at wire ends

In order to stress the dependence of thermovoltage on temperature gradients along thermocouple wire, relation (6) can be arranged as relation:

$$de = s(t,x) dx \tag{7}$$

in which another important fact is emphasized, the fact that Seebeck´s coefficient $s(t,x)$ depends also on a position x along the wire that is not generally homogeneous in its whole length. The reasons that cause inhomogeneity of wire are various: some of them are changes in metallurgical structure due to mechanical deformations, changes in metallurgical composition due to diffusion of other materials (diffusion alloying), corrosion of wire as well as the changes in wire diameter. Some of those inhomogeneities can be diminished by appropriate thermal treatment/annealing while some of them are irreversible.

Heat treatment/annealing of thermocouple should be seen as a kind of "adjustment" and, in case of recalibrations, such heat treatment should only be carried out with the formal agreement of the client.

For the best results, a thermocouple to be calibrated should be annealed at maximum immersion depth and at the highest temperature of intended use.

From the relation (7) two important conclusions can be deduced:

1. at parts of thermocouple wire that are in isothermal conditions i.e. without temperature gradients there are no thermovoltage generation (de = 0).
2. if the wire is ideal (homogeneous) i.e. $s(t,x) = s(t)$, the thermovoltage depends only on temperatures at the ends of the wire (proof by the integration of equation (7)).

The outcome of the first conclusion is that some thermocouple can produce good results in comparison calibration because inhomogeneity zones are in the isothermal conditions and therefore have no influence on sensor's thermovoltage. But during use the immersion depth of thermocouple can be such to cause the inhomogeneities to be found in the maximal temperature gradient causing intolerable errors in temperature reading.

Those are the reasons for carrying out the test of inhomogeneity.

There are various approaches to this problem and one of them is presented at the following figure:

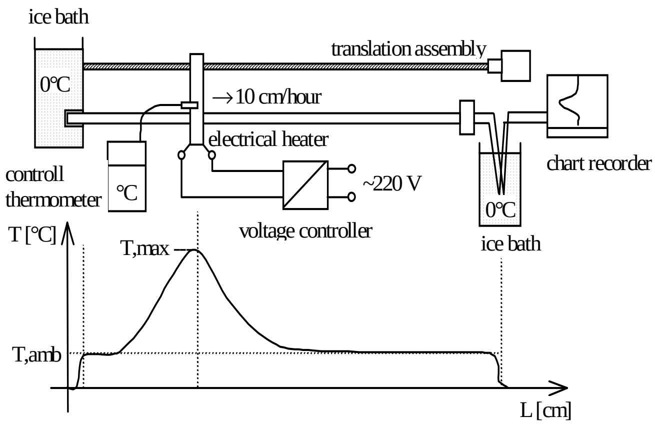

Fig.1. Measuring line for testing inhomogeneity of thermocouple wire

Both ends of thermocouple (measuring and reference junction) are kept at the same temperature of 0°C. In this case, according to conclusion 2, generated thermovoltage should be 0 µV if the wires are ideally homogeneous.

Then, using a translation assembly, the ring-shaped heater is moved along the thermocouple, with translation speed rate that is slow enough to allow thermal balance between heater and underlying part of tested thermocouple.

In BNM-LNE, France, instead of electrical heater they use lamp for heating tested thermocouple while some experts in thermometry state that heating of thermometer can be simple achieved with hot air blower.

The temperature of the heater is adjusted by a temperature controller and checked by a control thermometer.

Thermovoltage is continuously measured and recorded by the chart recorder.

The diagram in Fig.1. presents possible quality distribution of temperature along tested thermocouple at one time.

Finally, chart records obtained through this procedure are analyzed and decision is made out following two possibilities:

1. inhomogeneity of wire of the tested thermocouple is so high (higher than acceptable tolerances) that this sensor has to be rejected and replaced with new one;

2. inhomogeneity exists but it is within acceptable tolerances, and in such case e_{max} (maximum deviation of generated thermovoltage during this test) is handled as measurement uncertainty of the tested thermocouple due to inhomogeneity of the wires, type B with rectangular distribution.

Now, when all influence factors are defined, uncertainty budget in comparison calibration of thermocouples can be fulfilled as it is shown in a numerical example below.

3. NUMERICAL EXAMPLE

The following example deals with the calibration of a type S thermocouple by comparison with reference standard type S thermocouple at calibration temperature of 1000°C.

Reference standard thermometer is calibrated by comparison by an accredited calibration laboratory.

The comparison medium is thermostatically controlled tubular furnace with a thermal equaliser block.

The reference junctions of the thermocouples are kept at 0°C using an ice/water bath.

For measuring emfs generated by both thermocouples an 7½ digit multimeter, regularly calibrated by an laboratory accredited for electricity, is used.

Series of n = 10 measurement cycles are done following measurement sequence (ms1) and the results derived from those measurements are:

- mean temperature of thermal source measured with standard thermocouple:

$\overline{t_S}$ = 1003,1320°C with associated uncertainty of $u(\overline{t_S})$ = 0,0037°C

- mean emf generated by tested thermocouple:

V_x = 9631,7 µV with associated uncertainty of $u(\overline{V_x})$ =0,04 µV

Voltage sensitivity coefficient of the reference thermometer is taken from its calibration certificate and for 1000°C it is:

$$\frac{dE_S}{dt} = 11{,}6\frac{\mu V}{°C} \Rightarrow C_S = \frac{dt}{dE_S} = 86{,}0\frac{mK}{\mu V}$$

while for 0°C it is:

$$\frac{dE_{S0}}{dt} = 5{,}4\frac{\mu V}{°C} \Rightarrow C_{S0} = \frac{dt}{dE_{S0}} = 185{,}2\frac{mK}{\mu V}$$

Voltage sensitivity coefficient of the calibrated thermocouple is taken from standardised reference table for S type thermocouples and for 1000°C it is:

$$\frac{dE_x}{dt} = 11{,}6\frac{\mu V}{°C} \Rightarrow C_x = \frac{dt}{dE_x} = 86{,}0\frac{mK}{\mu V}$$

while for 0°C it is:

$$\frac{dE_{x0}}{dt} = 5{,}4\frac{\mu V}{°C} \Rightarrow C_{x0} = \frac{dt}{dE_{x0}} = 185{,}2\frac{mK}{\mu V}$$

Various corrections and associated uncertainties present in this example of comparison calibration are as follows:

$\delta t_S(V_{iS})$: the expanded uncertainty (k=2) of reference thermocouple is given in its calibration certificate and it is 0,7°C corresponding to a standard uncertainty of 0,35°C

δV_{c1} : the calibration certificate provides the multimeter calibration correction and its value is zero with expanded uncertainty for used range (200 mV) of 1,56 µV corresponding to a standard uncertainty of 0.78 µV

δV_{c2} : the calibration history of multimeter reveals no significant drift with regard to standard uncertainty so this correction is zero with a standard uncertainty estimated to be 0,8 µV

δV_{c3} : correction due to resolution of multimeter is taken to be zero with standard uncertainty for 7½ digital multimeter and used range of 0,01 µV.

δt_D : the calibration history of standard thermocouple reveals no significant drift between two calibrations, so this correction is zero with a standard uncertainty estimated to be close to 0,40°C

δt_F : the correction linked to the characterised comparison medium is taken as zero with a standard uncertainty close to 0,26°C

δV_R : the correction linked to the influence of the ambient parameters and connections is taken to be zero within limit of 1µV corresponding to a standard uncertainty of close to 0,7 µV

δt_{0S} : the correction linked to the ice/water bath is taken as 0,022°C with standard uncertainty of the reference junction temperature of the thermocouples of 0,002 °C

δV_L : the correction liked to the use of compensation cable is taken to be zero with standard uncertainty of 0 µV

δV_H : the correction linked to the influence of the inhomogeneity of the thermocouple wires is taken to be zero with standard uncertainty of 0,75 µV.

The suggested format of tables that could be used for calculation of uncertainty budgets is as follows.

TABLE 1 Uncertainty budget for temperature of thermal source

Quantity	Symbol	Estimation	Standard uncertainty	Distribution	Sensitivity coefficient	Contribution
Mean temp. of standard TC	$\overline{t_S}$	1003,1320 °C	0,0037 °C	normal	1	3,7 mK
Calibration of STC	$\delta t_S(V_{iS})$	0 °C	0,35 °C	normal	1	350,0 mK
STC drift	δt_D	0 °C	0,4041 °C	normal	1	404,1 mK
Multimeter calibration	δV_{c1}	0 µV	0,78 µV	normal	86,0 mK/µV	66,9 mK
Multimeter drift	δV_{c2}	0 µV	0,78 µV	normal	86,0 mK/µV	66,9 mK
Multimeter resolution	δV_{c3}	0 µV	0,01 µV	rectangular	86,0 mK/µV	0,5 mK
Comparison medium	δt_F	0 °C	0,2598 °C	rectangular	1	259,8 mK
Influence factors	δV_R	0 µV	0,69 µV	rectangular	86,0 mK/µV	59,6 mK
Ice/water bath	δt_{0S}	0,022 °C	0,002 °C	Rectangular	0.47	1,1 mK
Temp. of the hot junction of the tested TC	t_x	1003,1218 °C				0,605 °C

TABLE 2. Uncertainty budget for emf of the calibrated thermometer

Quantity	Symbol	Estimation	Standard uncertainty	Distribution	Sensitivity coefficient	Contribution
Mean emf of tested TC	$\overline{V_x}$	9631,7 µV	0,04 µV	normal	1	0,04 µV
Multimeter calibration	δV_{c1}	0 µV	0,77 µV	normal	1	0,77 µV
Multimeter drift	δV_{c2}	0 µV	0,77 µV	normal	1	0,77 µV
Multimeter resolution	δV_{c3}	0 µV	0,01 µV	rectangular	1	0,006 µV
Influence factors	δV_R	0 µV	0,69 µV	rectangular	1	0,69 µV
Compensation cable	δV_L	0 µV	0,0 µV	rectangular	1	0,0 µV
Inhomogeneity	δV_H	0 µV	0,75 µV	rectangular	1	0,75 µV
Ice/water bath	δt_{0S}	0,022°C	0,002 °C	normal	5,4 µV/°C	0,01 µV
Dev. cal. temp from temp of thermal source	Δt	0,00	0,605 °C	normal	11,6 µV/°C	6,97 µV
Emf of cal. TC at cal. temp.	V_x	9631,6 µV				7,128 µV

Finally, the reported expanded uncertainty of comparison calibration is stated as the standard uncertainty of measurement multiplied by the coverage factor k = 2, which for a normal distribution corresponds to a coverage probability of approximately 95%. Reported result for calibrated thermocouple could be in following format:
calibrated thermocouple at temperature of 1000°C ±1,4°C generate, with its cold junction at the temperature of 0°C, electromotive force of 9631,6 μV ±14,5 μV (14,5 μV correspond to 1,25°C, C_x is used)

5. CONCLUSION

The dissemination of traceability to ITS-90 through comparison calibration of thermocouples is important for many laboratory and industrial measurements as well as for subsequent calibrations in many laboratories. This paper describes estimation of measurement uncertainties in comparison calibration of thermocouples used at Laboratory for Process Measurements. The procedure takes into account many influencing factors, and special emphasis is devoted to inhomogeneity of tested thermocouples, with description of method and apparatus used for inhomogeneity assessment. The scope of the method is presented with sample calibration results and uncertainty budgets presented in tabular form.

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