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Calibration of Type S Pt/Pt–Rh Alloy Thermocouples and Uncertainty Estimation

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Thermocouples are the most widely used thermometric sensors for different applications due to wide temperature range and high temperature measuring capability. They are simple temperature sensors and can be made in very small sizes to convert heat into electricity. Additionally, they are inexpensive and one of the highly durable thermometer types. The purpose of this article is to perform the calibration of thermocouples with the comparison method and to calculate the expanded uncertainty by determining the uncertainty parameters affecting the measurement.

Keywords: Thermocouple; Seebeck effect; Temperature measurement; Thermometer sensor

1 Introduction

Thermocouples are the most widely used sensors for the measurement and control of high temperatures in scientific and industrial applications. All standardized and non-standardized thermocouples for such applications are made of metallic elements or alloys. The accuracy and precision of temperature measurement by thermocouples depend not only on the design and composition of the thermoelements, but also on how well they are protected from environmental conditions. Although numerous papers have been published on the applications of thermocouples and the basic and theoretical aspects of the metallic thermoelements in the literature. Therefore, a study on the thermal emf of metallic elements is of basic interest in thermocouple thermometry.

The thermoelectric principles are often described as if pairing of dissimilar materials is essential to the very occurrence of thermoelectric emf. To the contrary, the Seebeck effect that produces the thermoelectric emf is fundamentally associated with the transport of heat and electricity by individual electrically conducting materials under the influence of a temperature gradient.

If a conductor (such as a wire) is placed in a temperature gradient, the electrons at the hot end are more energetic than those at the cold end. In a simple picture they have a tendency to diffuse along the wire, so there are more electrons at the cold end than at the hot end. This is like gas in a pipe – there are fewer gas molecules at the hot end than the cold end, so there is a density gradient. However, electrons carry charge, so a density gradient also produces a voltage (it is strictly an 'electromotive force' or emf because the voltage is not just a potential difference but is actually generated in the wire, as in a battery)[1-3].

In order to detect or measure the emf it is necessary to make a complete circuit, so another wire must be connected at the hot end and lead back to the cold end. This second wire must be of a material with different thermoelectric properties, otherwise there would be no net emf at the cold end. Hence thermoelectric circuits and thermocouples are made with two dissimilar wires, *a* and *b* in Fig. 1, which are connected together at one end (t_1) and produce a net emf E at the other end t_2(Ref. 4).

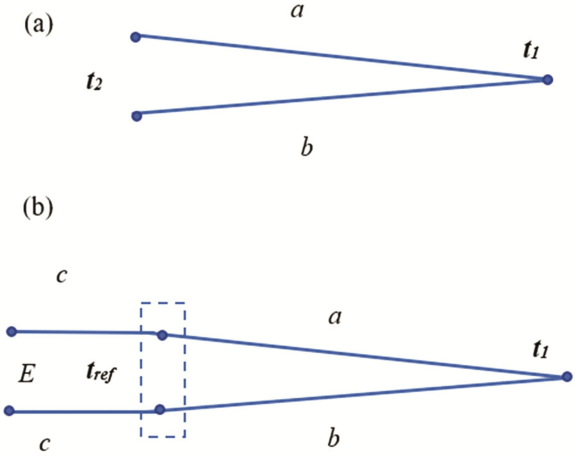

Fig. 1 — Thermocouple circuits to measure the temperature t_1: (a) connected directly to the voltmeter at temperature t_2, and (b) including reference junctions, at t_{ref}, which is often the ice point, 0 °C.

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The effect is called the Seebeck effect and the emf is known as the Seebeck emf, after Thomas Seebeck who discovered it in 1820[5-6]. The magnitude of the effect in each wire, or the relative magnitude in the pair of wires, is given by the Seebeck coefficient. The wires are selected to have widely different coefficients, so as to maximise the emf. Even so, a typical value for a thermocouple pair is only ~40 μV/°C, but with modern voltmeters it is possible to measure the emf with a resolution of 0.1 °C or less. Transition metal (nickel-based) alloys are mostly preferred for thermocouple thermometers since their physical and thermophysical properties are very similar to each other, but they can have very different Seebeck coefficients (even of opposite polarities). They can be used at temperatures up to about 1200 °C. For accurate work, or for measurements at temperatures up to 1600 °C, thermocouples of platinum and alloys of platinum and rhodium are used. Eight thermocouple combinations are standardised in IEC 60584 for industrial use[7]. They are designated by a Type letter, such as Type R which is platinum-13 % rhodium (positive conductor) versus pure platinum (negative conductor).

2 Experiment

It is important to remember that the emf is generated along the wires where there is a temperature gradient, and not at the junctions, which are only needed to connect the wires together and make the circuit. Because it is a distributed sensor, not a point sensor, the emf generated depends on the temperatures at both ends of the wires. It is necessary to measure the reference junction temperature, or fix it, for example by using an ice point. The ice point is the melting temperature of air-saturated ice at atmospheric pressure 101, 325 Pa. In an ice point apparatus a Dewar (vacuum-insulated) flask contains a mixture of crushed ice and water. The ice and water should be made from distilled or de-ionised water so that the temperature is close to 0 °C. Thermometers are calibrated at the ice point by inserting them sufficiently into the ice/water mixture that they are not affected by heat conduction from the outsıde

The thermocouple *ab* of Fig. 1(a) is simply connected to the instrument (voltmeter) used to measure the emf, but the instrument must include 'cold junction compensation': it must measure the temperature of the input terminals, t_2, and take it into account in calculating the temperature t_1. In 1b the thermocouple wires *a* and *b* are connected to copper wires *c* in an ice point. The reference temperature is then fixed at 0 °C, and the measurement of t_1 can be more accurate. The emfs generated in the two copper wires in leading from the ice point to the instrument should be identical and cancel out because they are of opposite polarity in the circuit.

Thermocouples using alloys of platinum and rhodium (Types R, S and B) are used as standards for temperature measurement up to 1600 °C but various nickel-based alloy thermocouples (Types J, K, T, E and N) are commonly used at temperatures up to 1200 °C, because they are more sensitive and cheaper. The most common thermomouples and their temperature ranges of use are listed in Table 1.

This study covers the preparation of the experimental set-up, the realization of the measurements and the evaluation of the results.

This experiment is designed to show the calibration of a Type S (Pt10%Rh-Pt) thermocouple at different temperatures (200, 400,600,800 °C) in a heated metal 'dry block calibrator' or furnace.

2.1 Apparatus

One Type S test thermocouple, one calibrated platinum resistance thermometer (Pt-100) reference thermometer, one calibrated Platinum-Rhodium thermocouple (Type S) (Hart scientific), a heated metal block 'Dry Block calibrator' (Hart), tube Furnace (Carbolite TZF), Type S thermocouple meter (temperature indicator), Superthermometer (Hart), Multimeter (Hart scientific), Ice point apparatus.

2.2 Method

The test thermocouple consists of insulated wires of Chromel and Alumel, which are soldered together at one end to form the 'measuring junction'. The other ends are connected to two insulated copper wires to form the 'reference junctions' which are kept at the ice point, 0 °C. The copper wires lead to a sensitive

Table 1 — Type of Thermocouples and their ranges of use

Thermocouple Type	Temperature Range	Materials of Construction
B	0 – 1820 °C	Platinum – 30 % Rhodium / Platinum – 6 % Rhodium
E	-270 – 1000 °C	Chromel / Constantan
J	-210 – 1200 °C	Iron / Constantan
K	-270 – 1372 °C	Chromel /Alumel
N	-270 – 1300 °C	Nicrosil / Nisil
R	-50 – 1768 °C	Platinum – 13 % Rhodium / Platinum
S	-50 – 1768 °C	Platinum – 10 % Rhodium / Platinum

voltmeter, which registers the total EMF, E, generated in the circuit.

Figure 2 shows schematically a thermocouple inserted in a furnace, with the reference junctions in ice point, and connected to a temperature indicator. Most of the EMF signal is generated along the thermocouple wires as they pass through the temperature gradient in the furnace, and not at the junction.

2.3 Hazard

In this study, measurements were carried out at high temperature. For this reason, some precautions were taken against the risk of burning. During the calibration, staff were used lab coats, goggles, and high temperature resistant gloves.

2.4 Procedure

In this calibration process, a Type S thermocouple will be calibrated first in an ice point, then in the Dry Block calibrator at 200 °C, and 400 °C, using the PRT reference thermometer, and then in the Dry Block or Carbolite furnace at 600 °C and at 800 °C, using the calibrated Type S thermocouple. Immersion tests will be made at each point. Finally the calibration will be repeated at 200 °C and at the ice point, to check that the thermocouple calibration has not changed. The temperatures will be obtained from the calibrations of the PRT reference thermometer and Type S thermocouple.

The calibration procedure will follow is detailed below[8,9]:

1. Prepare an ice point and insert the thermocouple reference junctions into it, insulated from each other, inside a clean and dry glass tube.
2. Connect the Type S thermocouple to the indicator and check that it responds correctly to temperature (*i.e*., that the polarity is correct).
3. Measure the temperature output of the thermocouple and the reference thermometer at the ice point using the indicator and the Superthermometer. When the temperature is stable, take at least 10 sets of measurements at intervals of about 10 seconds
4. Insert the thermocouple and PRT into closely fitting holes in the dry block and set the temperature to 200 °C. When the temperature is stable, measure the outputs of the thermometer and thermocouple. Take at least 10 sets of measurements at intervals of about 10 seconds.
5. Perform immersion tests by keeping the reference thermometer constant and raising the test thermocouple by 1 cm, and then 2 cm. Take at least 10 measurements at intervals of about 10 seconds at each position.
6. Set the dry block to 400 °C and make measurements as at 200 °C,
7. Make the reference junctions for the Type S platinum-rhodium thermocouple by twisting them with copper wires.
8. Put the insulated reference junctions inside a glass tube and insert it into the melting ice. Connect the other ends of the copper wires to the multimeter.
9. Insert the S thermocouples into the tube furnace as in Fig. 3 and set the temperature to 600 °C. When the temperature is stable, measure the outputs of the both thermocouples. Take at least 10 sets of measurements at intervals of about 10 seconds.
10. Perform immersion tests in the tube furnace by keeping the Type S thermocouple fully inserted and removing the test thermocouple by 1 cm, and then 2 cm. Take at least 10 measurements at intervals of about 10 seconds at each temperature.
11. Repeat instruction 9 and 10 for the tube furnace at 800 °C

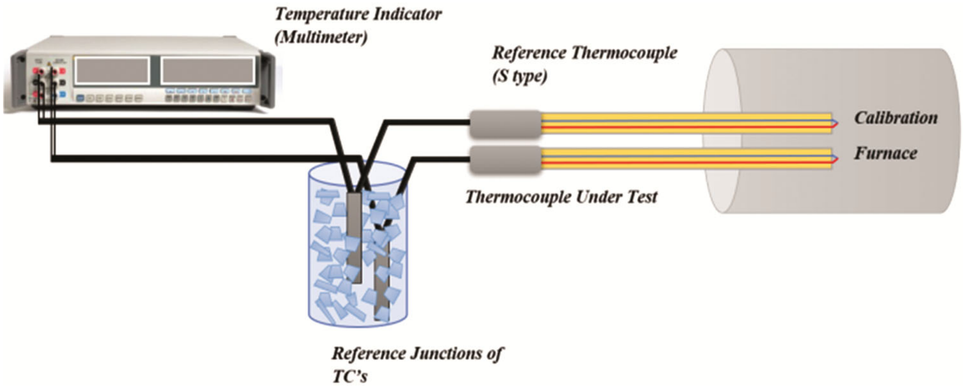

Fig. 2 — Schematic diagram a thermocouple with an ice-point reference junction.

12. Prepare an ice point (refresh if ice point has melted), Take at least 10 measurements at intervals of about 10 seconds at each temperature.
13. Calculate the uncertainty budgets.

3 Measurements and Results

For the evaluation of the results and the uncertainty estimation, firstly the average values and standard deviation of the measurements are calculated. The results of the measurements are given in Table 2, 3 and 4.

The ice-point measurements were carried out before and after measurements to check reproducibility of the measurement, and no significant changes were observed. Calibration points were 200, 400, 600 and 800 °C. The K type test thermocouple was inserted to a full immersion depth of 30 cm in the dry block and then it was raised 2 cm and 4 cm. Immersion depth value were investigated by taking the difference between full immersion value and raised 4 cm measurement value. For each calibration point, the stability and uniformity of the dry block, and other uncertainties, were evaluated.

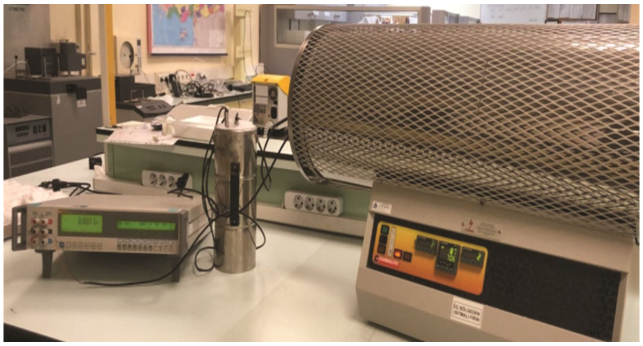

Fig. 3 — The experiental set-up.

Table 2 — Measurement result of S type thermocouple at different temperatures

Nominal Value (°C)	Reference Thermometer (°C)	S Type Test Thermocouple (°C)
0	0.006	0.01
200	199.630	199.61
400	398.921	398.91
600	598.453	598.41
800	799.102	798.98
0	0.002	0.01

Table 3 — Measurement result of S type thermocouple at different depths at 200 and 400 °C

Immersion Depth (cm)	S Type Test Thermocouple (°C)
2	199.61
4	199.58
2	398.91
4	398.84

An example of the measurements performed in accordance with the above procedure, the measurements made at 400 °C are given in the Table 4.

After the measurements are completed, the next step will be evaluation of the measurement uncertainty of thermocouple. An example of the application of guideline EA-4/02 to the calculation of uncertainty for type S thermocouple is given in Table 2 of [10]. In this process, each error source affecting the measurement caused by both the S type thermocouple and the reference thermometer is carefully considered in the uncertainty budget.

The data used in the calculations were obtained from the measurement results, the certificates of the devices or the user manuals. The probability distributions for the uncertainties were examined in two groups; Type A (normal, Gaussian distribution) or Type B (as upper limits of a rectangular distribution). The half-widths of rectangular distributions were divided by √3 to calculate the standard uncertainties. The standard uncertainties are calculated and these are then combined in quadrature, as the square root of the sum of the squares. The expanded uncertainty assigned corresponds to a coverage probability of approximately 95 %. All factors affecting the measurements are evaluated in the uncertainty budgets and, as an example, an uncertainty calculation at 400 °C is given in the Table 5.

The following uncertainty contributions affected the measurements; reference thermometer repeatability, reference thermometer calibration, indicator uncertainty, drift, resolution, furnace stability and uniformity, compenstaion cable, ice point uncertainty,

Table 4 — Output of the thermometers at 400 °C

	Reference Thermometer (°C)	K Type Test Thermocouple (°C)
1	398.926	398.91
2	398.921	398.91
3	398.924	398.90
4	398.920	398.91
5	398.923	398.91
6	398.919	398.91
7	398.920	398.92
8	398.919	398.91
9	398.920	398.90
10	398.919	398.90
Average	398.921	398.91
Standard Deviation	0.002	0.01

Table 5 — Uncertainty Budget for the calibration of the S type thermocouple at 400 °C with PRT reference thermometer

Estimated Value		400 °C		
Uncertainty Component	Type	Uncertainty °C	Statistical Distribution	Standard Uncertainty °C u_i
Reference Thermometer Repeatability	A	0.002	Normal	2.00E-03
Reference Thermometer Calibration	B	0.020	Normal	10.00E-03
Indicator Resolution	B	0.01	Rectangular	5.78E-03
Dry Block Uniformity	B	0.3	Rectangular	1.73E-01
Compensation Cable	A	0.002	Normal	2.00E-03
Inhomogeneity	B	0.012	Rectangular	6.94E-03
Ice Point Uncertainty	B	0.01	Normal	5.00E-03
Test Repeatability	A	0.01	Normal	10.00E-03
Test Resolution	B	0.01	Rectangular	5.78E-03
Immersion Effect	B	0.07	Rectangular	9.25E-03
Combined Standard Uncertainty, *u*				0.205
Expanded Uncertainty = *ku*				0.41 °C

Table 6 — Measurement uncertainty outputs

Nominal Value	S Type Test Thermocouple Uncertainty (±°C)
0	0.3
200	0.4
400	0.4
600	0.6
800	0.7

test thermocouple repeatability, resolution and uniformity are given in Table 5[11-14]. The expanded uncertainty was estimated for all temperatures in Table 6 (where the coverage factor k=2). It was easily observed that the reason for the increasing uncertainty is that the uniformity values of the test thermocouple increase as the temperature increases.

4 Conclusion

Precise and accurate measurement of high temperatures is important in industrial measurements. Along with the measurements, the measurement uncertainty should be estimated with the same precision. In this study, the process followed by the calibration laboratories during these measurements is explained in detail. It was investigated which parameters have an effect on the results in the uncertainty estimation in order to make the measurements more accurate and precise. As a conclusion we can see that uniformity of the furnace had significant effect on the uncertainty budget. The stability and homogeneity values of the temperature source used to reduce the measurement uncertainty should be taken into account.

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