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Estimation of the Extrapolation Error in the Calibration of Type S Thermocouples

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Abstract. Measurement results from the calibration performed at NIST of ten new type S thermocouples have been analyzed to estimate the extrapolation error. Thermocouples have been calibrated at the fixed points of Zn, Al, Ag and Au and calibration curves were calculated using different numbers of FPs. It was found for these thermocouples that the absolute value of the extrapolation error, evaluated by measurement at the Au freezing-point temperature, is at most 0.10 °C and 0.27 °C when the fixed-points of Zn, Al and Ag, or the fixed-points of Zn and Al, are respectively used to calculate the calibration curve. It is also shown that absolute value of the extrapolation error, evaluated by measurement at the Ag freezing-point temperature is at most 0.25 °C when the fixed-points of Zn and Al, are used to calculate the calibration curve. This study is oriented to help those labs that lack a direct mechanism to achieve a high temperature calibration. It supports, up to 1064 ° C, the application of a similar procedure to that used by Burns and Scroger in NIST SP-250-35 for calibrating a new type S thermocouple. The uncertainty amounts a few tenths of a degree Celsius.

Keywords: extrapolation, calibration, type S thermocouples

INTRODUCTION

Platinum (Pt) based thermocouples were used to define the International Temperature Scale of 1968 in the range from [630 °C to 1064 °C]. It is well known that they are not stable, due to the oxidation, evaporation and migration of rhodium [1]. Currently they are used as secondary standards. Although it is well known that Pt-Pd thermocouples perform better than Pt-PtRh thermocouples [2-13], there are still many national laboratories that have not begun to use those types of thermocouples. It may also happen, due to limited resources, that they do neither have an appropriated high temperature furnace nor adequate fixed points to calibrate them in. Additionally type S thermocouple is still regarded in industry as a reference thermometer. The extrapolation of the calibration of type S thermocouples may be then an adequate solution for this case. We have then focused on the analysis of the error resulting from the application of this method.

EXPERIMENTAL DETAILS

The extrapolation procedure to calibrate type S thermocouples up to 1064 °C was applied to a set of ten thermocouples. These thermocouples were assembled at NIST from spools of wire purchased by NIST. The wire obtained was high purity 24 gauge platinum and platinum/ 10% rhodium alloy wire. The typical length of a thermocouple was 100 cm. Initially each thermocouple was exposed to an electrical anneal consisted of 45 min at 1450 °C and 30 min at 750 °C, as described in [14]. The 1450 °C anneal serves to relieve mechanical strain from packing and handling, as well as to remove the rhodium oxide layer. The 750 °C anneal is designed to stabilize the lattice vacancies and produce a uniform rhodium oxide layer. The final furnace anneal is described in [14].

Six thermocouples identified as sc-98-03, sc-98-04, sc-98-05, sc-98-06 sc-98-07 and sc-98-08 were made from the same spool of wire, produced by Sigmund Cohn. Other four thermocouples, identified as jm-88-19, jm-88-20, jm-88-21and jm-88-22 were made from another identical spool of wire, produced by Johnson Matthey. All thermocouples have 0.5 mm diameter. They were calibrated at NIST, at the fixed points of Zn, Al, Ag and Au with an expanded uncertainty lower than 0.1 °C [15].

PROCEDURE DESCRIPTION

The calibration consisted of a two-step procedure. In the first step, the differences between the measured electromotive forces and the corresponding NIST 175 reference values were fitted by a second order polynomial with null constant term. In the second step a calibration curve was constructed as the fitted curve plus the NIST 175 reference curve [16].

Several fixed points calibration curves were obtained by successively using the freezing temperature of gold, t_{Au}, silver, t_{Ag}, and aluminum, t_{Al},

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as the highest temperature of calibration. For example, the EMF´s measured at the Au, Ag, Al and Zn fixed points were utilized for the calculation of the calibration curve up to t_{Au}. Similarly, another calibration curve was determined from the same Ag, Al and Zn fixed-points measurements. In this case the extrapolation error was evaluated at t_{Au}. Finally a last fixed point calibration curve was calculated by only using the Zn and Al fixed point measurements. This curve was extrapolated to the Ag and Au points.

From the selected fixed point of highest temperature (FPHT) up to 1100 °C, two possibilities were considered for extending the calibration. The first one consisted in extrapolating the above mentioned differences by means of a straight line (linear extrapolation). This line takes the same value and derivative at the FPHT than the curve obtained by the two-steps procedure. The second possibility consisted in the extrapolation of those differences by means of the second order polynomial mentioned above (parabolic extrapolation).

In all cases the extrapolation error was evaluated as the difference between the EMF value obtained from the incomplete fixed point calibration curve, and the EMF value measured for the fixed point of higher temperature, that was not considered in the calibration curve.

RESULTS

Results are given for linear extrapolations performed successively from the Ag and the Al freezing point temperatures. The extrapolation error, evaluated as the difference between the extrapolated calibration curve and the measurement at the corresponding fixed point is presented. Then the differences resulting from the linear and parabolic extrapolations, up to 1100 °C, are presented. Finally the uncertainty evaluation is described.

Case 1 - Extrapolation from the Ag point

The extrapolation error is evaluated at t_{Au}, as:

$$e_{ext}^{Ag}(t_{Au}) = EMF_{ext}(t_{Au}) - EMF_{med}(t_{Au}) \qquad (1)$$

Where e_{ext}^{Ag} (t_{Au}) is the error at t_{Au} resulting from the Ag point extrapolation, $EMF_{ext}(t_{Au})$ is the extrapolated EMF and $EMF_{med}(t_{Au})$ is the EMF measured at the Au fixed point.

Table 1 show errors committed at the Au point, calculated by means of equation (1). The extrapolation range is about 100 °C. The mean errors for SC and JM thermocouples are 0.04 °C and 0.07 °C respectively and the standard deviation of the errors is 0.03 °C. The maximum error is equal to 0.1 °C for SC 98-4 thermocouple.

TABLE 1. Error, E, at t_{Au} corresponding to the linear extrapolation of the curve calculated by means of FPMs at t_{Zn}, t_{Al} and t_{Ag}.
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TC	E at t_{Au} (°C)	Mean E (°C)
JM 88-19	0.03	
JM 88-20	0.06	
JM 88-21	0.04	
JM 88-22	0.02	0.04
SC 98-3	0.06	
SC 98-4	0.10	
SC 98-5	0.05	
SC 98-6	0.02	
SC 98-7	0.08	
SC 98-8	0.09	0.07

Case 2 - Extrapolation from the Al point

The extrapolation error is evaluated at t_{Au}, as:

$$e_{ext}^{Al}(t_{Au}) = EMF_{ext}(t_{Au}) - EMF_{med}(t_{Au}) \qquad (2)$$

Where e_{ext}^{Al} (t_{Au}) is the error at t_{Au} resulting from the Al point extrapolation, $EMF_{ext}(t_{Au})$ is the extrapolated EMF and $EMF_{med}(t_{Au})$ is the EMF measured at the Au fixed point.

Similarly the extrapolation error evaluated at t_{Ag} is:

$$e_{ext}^{Al}(t_{Ag}) = EMF_{ext}(t_{Ag}) - EMF_{med}(t_{Ag}) \qquad (3)$$

Where e_{ext}^{Al} (t_{Ag}) is the error at t_{Au} resulting from the Al point extrapolation, $EMF_{ext}(t_{Ag})$ is the extrapolated EMF and $EMF_{med}(t_{Ag})$ is the EMF measured at the Ag fixed point.

Table 2 shows the errors committed at the Au and the Ag points, using equations (2) and (3). The extrapolation range is about 300 °C when the extrapolation is done from t_{Al} up to t_{Ag} and about 400 °C when the extrapolation is done from t_{Al} up to t_{Au}.

The mean errors at t_{Ag} for the SC and JM thermocouples are respectively 0.01 °C and -0.17 °C . The standard deviation of the errors is 0.10 °C. The largest difference is -0.25 °C and occurred for the thermocouple SC-98-5.

The mean errors at t_{Au} for SC and JM thermocouples are respectively 0.06 °C and -0.16 °C. The standard deviation of the errors is 0.12 °C. The largest difference -0.27 °C occurred for the same

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thermocouple SC-98-5, as in the previous extrapolation to t_{Ag}.

TABLE 2. Error, E, at t_{Au} and t_{Ag} corresponding to the extrapolation of the curve calculated by means of FPMs at t_{Zn} and t_{Al}

TC	E at t_{Au}, °C	Mean E (°C)	E at t_{Ag} (°C)	Mean E (°C)
JM 88-19	0.04		0.00	
JM 88-20	0.09		0.03	
JM 88-21	0.06		0.01	
JM 88-22	0.04	0.06	0.01	0.01
SC 98-3	-0.22		-0.21	
SC 98-4	-0.08		-0.12	
SC 98-5	-0.27		-0.25	
SC 98-6	-0.12		-0.09	
SC 98-7	-0.14		-0.15	
SC 98-8	-0.14	-0.16	-0.16	-0.16

Linear vs. parabolic extrapolation

Temperature differences resulting from the application of the linear and parabolic extrapolation are shown in Figures 1 and 2.

In Fig. 1 can be seen that, for the extrapolation from the Ag point up to t_{Au}, the difference is less than 0.010 °C and 0.002 °C for thermocouples SC and JM respectively. From the foregoing values, it follows that for both set of thermocouples the difference between the linear and parabolic extrapolations is negligible, one magnitude order smaller than the measurement uncertainty at the Au point.

As shown in Fig. 2, the difference, when extrapolating from the Al point to t_{Au}, is less than 0.13 °C and 0.03 °C for thermocouples SC and JM respectively. For the SC thermocouples the difference is of the same order as the measurement uncertainty at the Au point.

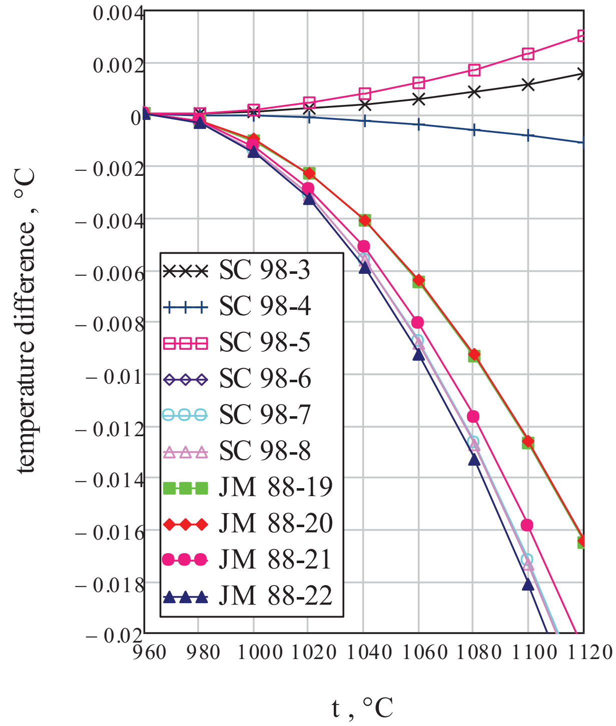

FIGURE 1 Temperature differences = (parabolic – linear) extrapolations, for a Zn, Al and Ag FPs calibration.

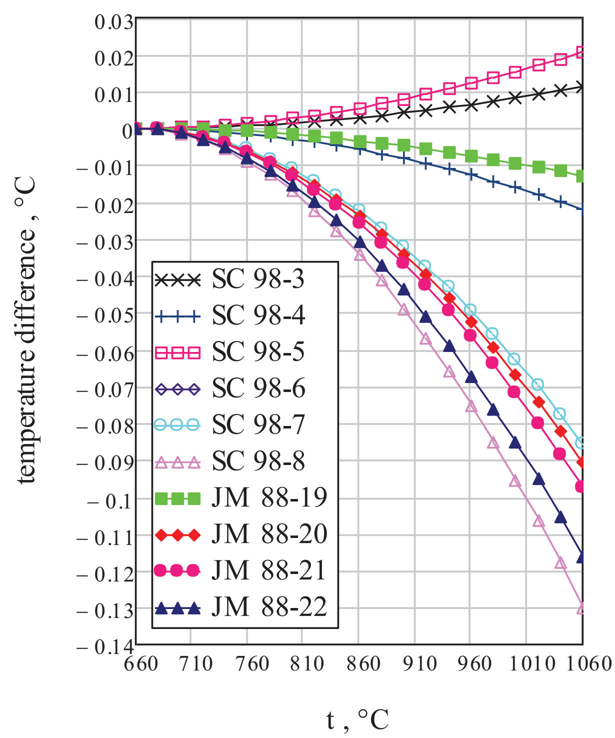

FIGURE 2 Temperature differences = (parabolic – linear) extrapolation, for a Zn and Al FPs calibration.

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UNCERTAINTY EVALUATION

The combined uncertainty (k=1), u, of the extrapolation error has been estimated for equations (1), (2) and (3) as:

$$u \approx \sqrt{u_{extrap}^2 + u_{fp}^2 + u_{\mathrm{mod}}^2}$$

Where u_{extrap} is the uncertainty assigned to the extrapolated fem. Its value was estimated as follows. First for every fixed-point temperature t_{fp}, a new extrapolated curve for the thermocouple´s emf was calculated by incrementing the emf at t_{fp} in its corresponding calibration uncertainty (u_{fp} = 0.05 °C, k = 1). At a second step, the differences between the emfs obtained in this way and the not incremented emfs were calculated. These differences were then added in quadrature to obtain u_{extrap} as a function of temperature. u_{fp} is the measurement uncertainty at the fixed point to which the extrapolation is performed. u_{mod} is the uncertainty assigned to the extrapolation mechanism. It was estimated as the difference between the linear and the parabolic extrapolated values.

The maximum absolute value of the extrapolation error, the corresponding combined uncertainty and its components are given in tables 3 and 4 for the cases corresponding to equations (1), (2) and (3).

TABLE 3. Uncertainty components (k=1)

t	FP used	u_{extrap} °C	u_{fp} °C	u_{mod} °C
t_{Au}	Zn, Al, Ag	0.07	0.05	0.006
t_{Au}	Zn, Al	0.18	0.05	0.081
t_{Ag}	Zn, Al	0.15	0.05	0.046

TABLE 4. Maximum error and corresponding combined uncertainty (k=1)

t	FP used	Error °C	u °C
t_{Au}	Zn, Al, Ag	0.10	0.08
t_{Au}	Zn, Al	0.27	0.21
t_{Ag}	Zn, Al	0.25	0.16

CONCLUSIONS

The results presented suggest that the calibration of the ten type S thermocouples in Zn, Al and Ag fixed points, could be extrapolated about 100 °C up to the Au point with an error whose maximum absolute value is about 0.10 °C ($u_{k=1}$ = 0.08 °C). When the calibration is performed only by using the Zn and Al points, it might be extrapolated about 300 °C up to the Ag or even about 400 °C to the Au point, with maximum absolute values of the extrapolation errors that are respectively 0.25 °C ($u_{k=1}$ = 0.16 °C) and 0.27 °C ($u_{k=1}$ = 0.21 °C).

The use of linear or parabolic extrapolation is irrelevant in terms of the error due to extrapolation from the Ag to the Au point. ($|\Delta t| < 0.01$ °C at t_{Au}) but it increases significantly up to 0.14 °C when the extrapolation is performed from the Al to the Au point.

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