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Research Article

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Development of High Temperature Thermocouples Calibration at NIS-Egypt

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Abstract Thermocouples of various kinds are the most widely temperature sensor used in the Egyptian industries for many heating processes, particularly those carried out at high temperatures. Therefore, National Institute for Standards (NIS-Egypt) has been interested in developing the calibration capabilities of high temperature thermocouples.
A complete set of ITS-90 fixed points and metal-carbon eutectic fixed points are used for improved calibrations of thermocouples and first results of their application at NIS-Egypt are presented.

Keywords Temperature, Thermocouples, NIS-Egypt

1. Introduction

The International Temperature Scale of 1990 (ITS-90) is intended to be a practical internationally agreed best approximation to the thermodynamic temperature scale. It extends from 0.65 K up to the highest temperature practically measurable using the Planck radiation law [1]. Temperatures above 1235 K can also disseminated by using thermocouples, which are used for the approximation of the ITS-90 in a large temperature range up to about 2450 K.
Where, thermocouples are increasingly used in the Egyptian industry and research, therefore NIS-Egypt was increased the calibration capabilities of high temperature thermocouples.
Herein, we describe the current progress in the NIS-Egypt facilities for high temperature thermocouple calibration.

2. Equipment, Standards and Facilities

2.1. Noble Metal Thermocouples

Thermocouples type Pt-10%Rh/Pt (Type S), Pt-13%Rh/Pt (Type R) and Pt/Pd were prepared using NIS-Egypt capabilities from Pt, Pt-10%Rh, 13%Rh/Pt and Pd wires [2], with 0.5mm in diameter and 200cm length. All wires were purchased from Johnson Matthey as reference grade, where the platinum wire of 99.999% purity and palladium wire of 99.997% purity. The Pt, Pt-10%Rh, 13%Rh/Pt and Pd wires were first annealed electrically at 1300^{o}C for approximately 10 hours, cooled rapidly to room temperature and then annealed for one hour at about 450^{o}C to reduce the lattice vacancies that may be quenched into the wires during cooling from the high temperature anneal. The annealed wires were assembled by threading the thermoelements into the bores of a twin bore high purity alumina tube with overall diameter 4.5mm and length 70cm. Before use all alumina tubes were baked at 1200 ^{o}C.
The reference junction of the thermocouple was maintained at 0 ◦C in a Dewar filled with distilled water and crushed ice (ice bath). The reference junction was inserted into closed end glass tube and was immersed 20 cm in the ice bath.

2.2. Fixed Points for Thermocouple Calibration

The freezing points of Al, Ag and Cu and the eutectic fixed-points of Co-C and Pd-C were used in this study .For the freezing points measurements, sealed cells containing high purity (99.9999%) metals Al, Ag and Cu and substantial ingot consisting of about 0.16 kg, of the alloy (minimum purity 99.95%) for the eutectic fixed-points of Co-C and Pd-C were used.

2.3. Fixed Point Furnaces

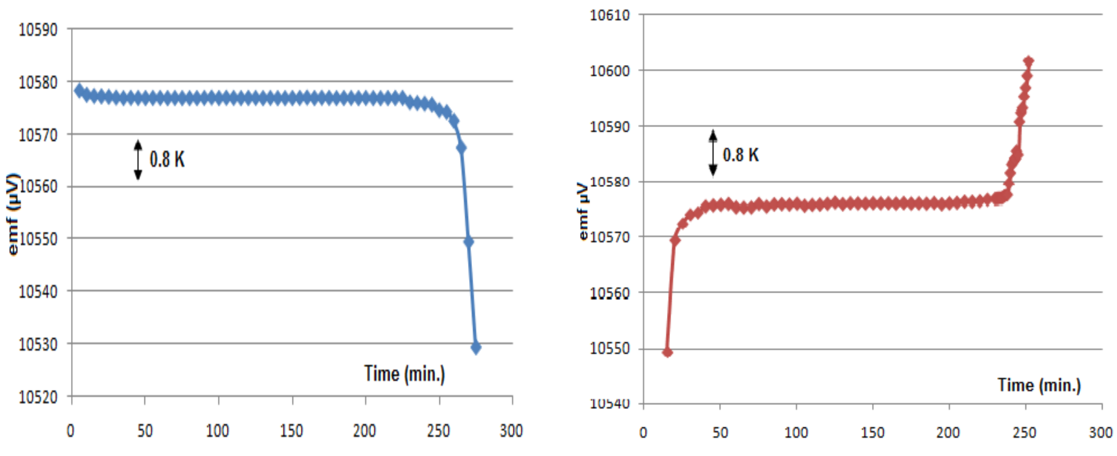
The furnace used for Al and Ag freezing points cells was made by carbolite-UK its maximum temperature 1000 ◦C and utilized double walled sealed sodium heat pipe, The temperature stability of Al and Ag freezing point furnaces was ±0.03 ◦C and ±0.3 ◦C respectively. The furnace used for Cu freezing point was three zone furnace made by ISOTECH – UK, its max temperature1200 ◦C. The furnace used for eutectic fixed-points of Co-C and Pd-C cells was three heater zones (Elite TMV16/75/610).

2.4. Nanovoltmeter

Digital Nanovoltmeter (DVM) (Keithly voltmeter type-182) with internal resistance higher than 10^9 Ω was used to measure emf, its resolution corresponds to temperature resolution of 1 mK and 48 nV accuracy.

2.5. Using ITS-90 Fixed Points for Thermocouple Calibration up to 1100 ^{o}C

For realization of the freezing points of Aluminum, Silver and Copper, the induced freezing technique was used. All fixed point cells were placed inside the heat pipe furnace whose temperature was kept about 5°C above the freezing point for at least one hour to ensure that the silver was molten throughout [3-4]. The temperature was monitored with a type K (type N for copper furnace) thermocouple placed in the thermometer well of the cell. During the freezing plateau the Pt-10%Rh/Pt (Type S), Pt-13%Rh/Pt (Type R) or Pt/Pd thermocouples were inserted gradually in steps into the cell well for measurements. About 15 minutes were required after the insertion of the thermocouple to be certain that it had come to an equilibrium temperature. When the Aluminum, Silver or Copper was completely frozen the thermocouple was gradually removed in steps from the cell to avoid quenching of the thermocouple by cooling rapidly from the Aluminum, Silver or Copper cell. Melting and Freezing plateaus of the Copper cell using type S thermocouple are shown in the fig. (1).



(a) (b)

Figure 1: Melting and Freezing plateaus of the Copper cell using type S thermocouple

The following tables (1, 2, and 3) represent the freezing point values on ITS-90, (E) measured emf of thermocouple in microvolt (µV),, the sensitivity coefficient, (E_{ref}) represent the corresponding reference value of the fixed point by thermocouple, ΔE is a difference between reference value and measured value ($ΔE=E_{ref} – E$) and (ΔT) is a corresponding difference in ^{o}C.

Table 1: Thermo emf of type S thermocouple in Al, Ag and Cu freezing points (F.P.)

Ingot (fixed point)	F.P. on ITS-90 °C	Measured emf E μV	Standard Deviation μV	Standard Deviation mK	dE/dt μV/°C	Reference Values in ITS-90 E_{ref} μV	ΔE $= E_{ref} - E$ μV	Corresponding Temperature Δ T °C
Al	660.323	5860.16	0.06	6.0	10.396	5860.13	-0.03	-0.003
Ag	970.78	9151.57	0.08	7.0	11.418	9148.38	-3.19	-0.279
Cu	1084.62	10575.90	0.07	6.0	11.798	10574.8	-1.1	-0.093

Table 2: Thermo emf of type R thermocouple in Al, Ag and Cu freezing point (F.P.)

Ingot (fixed point)	F.P. on ITS-90 °C	Measured emf E μV	Standard Deviation μV	Standard Deviation mK	dE/dt μV/°C	Reference Values in ITS-90 E_{ref} μV	ΔE $= E_{ref} - E$ μV	Corresponding Temperature Δ T °C
Al	660.323	6270.40	0.11	8.0	11.641	6277.09	6.69	0.5747
Ag	970.78	10003.39	0.13	9.0	13.065	10003.43	0.04	0.0031
Cu	1084.62	11631.90	0.15	11.1	13.545	11640.43	8.53	0.6298

Table 3: Thermo emf of type Pt/Pd thermocouple in Al, Ag and Cu freezing point (F.P.)

Ingot (fixed point)	F.P. on ITS-90 °C	Measured emf E μV	Standard Deviation μV	Standard Deviation mK	dE/dt μV/°C	Reference Values in ITS-90 E_{ref} μV	ΔE $= E_{ref} - E$ μV	Corresponding Temperature Δ T °C
Al	660.323	5778.40	0.10	7.2	13.9	5782.39	3.99	0.2871
Ag	970.78	10808.90	0.10	5.2	19.2	10813.08	4.18	0.2177
Cu	1084.62	13272.50	0.10	4.8	20.9	13277.66	5.16	0.2469

2.6. Using New Eutectic Fixed Points for Thermocouples Calibration up to 1500 °C

The calibration is carried out by inserting the thermocouple into a substantial ingot of Co-C and Pd-C. This is encased in a very pure graphite crucible. Prior to use the ingot assembly is placed in a hollow graphite shield (mounted on a disk of graphite felt and not touching the shield at the sides) in the most uniform region of a three zone furnace. This arrangement is mounted on an alumina brick in a recrystallised alumina tube, which is sealed to permit a continuously flowing argon gas atmosphere with a controlled pressure.

The emf output of this thermocouple is recorded at least every 10 s by a computer logging program. To start the melt, the furnace is warmed to 1332 °C (Co-C) or 1500 °C (Pd-C), with a ramp rate of 5 °C/min. The melting takes place at approximately 1324.0 °C (Co-C), or 1491.5 °C (Pd-C), with a melting range of around 0.2 °C. On completion of melting, the temperature will rise to a plateau corresponding to the furnace set-point. The melting occurs over a range of approximately 0.2 °C. The convention is that the fixed point emf is given by the point of inflection of the melting curve. It is therefore necessary to fit a 3rd order polynomial to the melting curve, and evaluate the emf where the second derivative of the polynomial is equal to zero.

The following tables (4, 5 and 6) represent the melting point values on ITS-90, (E) measured emf of thermocouple, standard deviation in microvolt, standard deviation in mille Kelvin, sensitivity coefficient, (E_{ref}) the corresponding reference value of the fixed point by thermocouple, ΔE (difference between reference value and measured value ($\Delta E = E_{ref} - E$), (ΔT) difference in °C.

Table 4: Thermo emf of type S thermocouple in Co-C & Pd-C eutectic fixed points (melting point M.P.)

Ingot (fixed point)	M.P. on ITS-90 °C	Measured emf E μV	Standard Deviation μV	Standard Deviation mK	dE/dt μV/°C	Reference Values in ITS-90 E_{ref} μV	ΔE = E_{ref} - E μV	Corresponding Temperature Δ T °C
CoC	1324.29	13453.41	0.35	29	12.135	13453.74	0.33	0.0272
PdC	1491.50	15484.95	0.56	46	12.049	15479.3	-5.65	-0.469

Table 5: Thermo emf of type R thermocouple in Co-C & Pd-C eutectic fixed points (melting point M.P.)

Ingot (fixed point)	M.P. on ITS-90 °C	Measured emf E μV	Standard Deviation μV	Standard Deviation mK	dE/dt μV/°C	Reference Values in ITS-90 E_{ref} μV	ΔE = E_{ref} - E μV	Corresponding Temperature Δ T °C
CoC	1324.29	14963.91	0.22	15.6	14.102	14971.0	7.09	0.5028
PdC	1491.50	17340.8	0.33	23.4	14.074	17331.07	-9.73	-0.691

Table 6: Thermo emf of type Pt/Pd thermocouple in Co-C & Pd-C eutectic fixed points

Ingot (fixed point)	M.P. on ITS-90 °C	Measured emf E μV	Standard Deviation μV	Standard Deviation mK	dE/dt μV/°C	Reference Values in ITS-90 E_{ref} μV	ΔE = E_{ref} - E μV	Corresponding Temperature Δ T °C
CoC	1324.29	18605.11	0.1	4.1	23.6	18628.44	23.33	0.9886
PdC	1491.50	22690.23	0.1	4	25.3	22716.95	26.72	1.0561

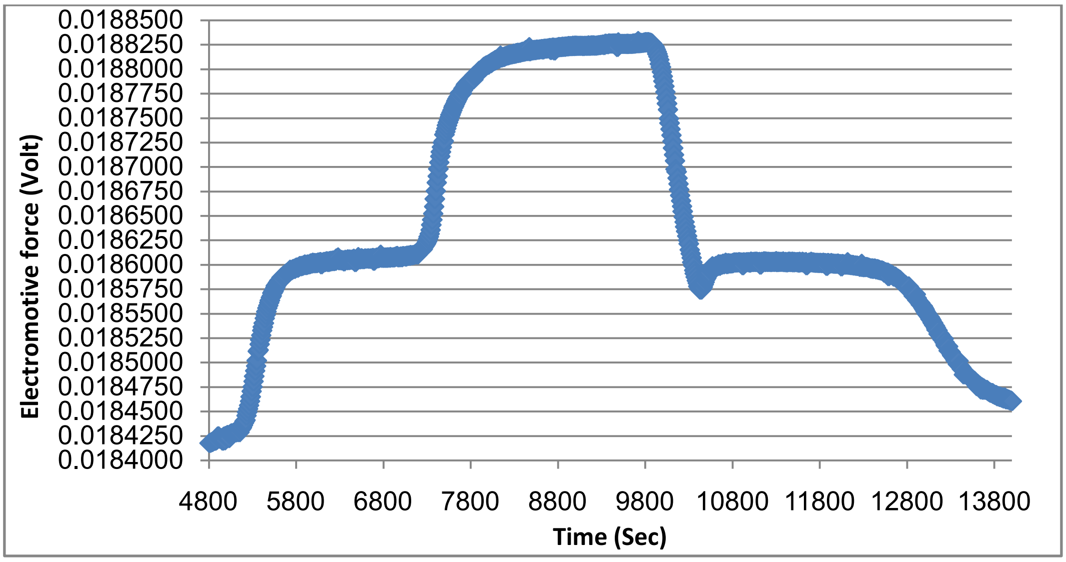


Figure 2: Melting and freezing points of Pt/Pd thermocouple in Co-C eutectic fixed point

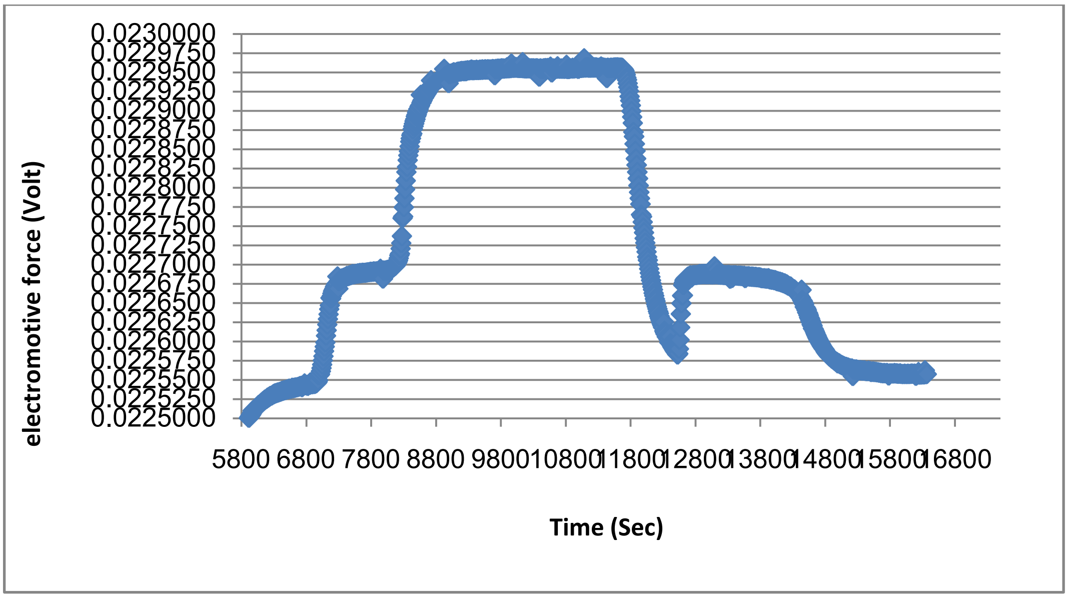


Figure 3: Melting and freezing points of Pt/Pd thermocouple in Pd-C eutectic point

3. Results and Uncertainty Evaluation

Uncertainties of measurement shall be calculated in accordance with EA publication EA-4/02 'Expression of the Uncertainty of Measurement in Calibration' [5]. The uncertainty budget of all thermocouples at fixed points incorporating the various contributory factors is shown in tables 7, 8 and 9. Uncertainties for the items labeled repeatability are typically evaluated as Type A uncertainties, using statistical methods. The other items are primarily evaluated using Type B methods. The major contribution to the uncertainty is attributed by inhomogeneity of thermocouple has been estimated from the immersion profile of the fixed points observed during the measurement. The uncertainty due to purity of ingots was evaluated using its certificate. The thermocouple drift was estimated from the earlier fixed point measurements. Other uncertainty sources, related electrical measuring system, are included in the total uncertainty budget as digital voltmeter and reference junction. The combined uncertainty U_C expressed in the form of 95% confidence level.

Table 7: Uncertainty budget of thermocouple type S in different fixed points

Expected components of Uncertainty	Fixed point ingots				
	Al	**Ag**	**Cu**	**Co C**	**Pd C**
			μV		
Statistical standard uncertainty	0.06	0.08	0.07	0.35	0.56
Determination of the calibration fixed point temperature	0.0052	0.0063	0.0071	2.6686	4.4585
Drifts of the means used to determine the calibration fixed point	0.1720	0.1106	0.1128	0.1187	1.0174
Voltmeter calibration	0.38	0.38	0.38	0.38	0.38
Voltmeter resolution	5.77E-06	5.77E-06	5.77E-06	5.77E-06	5.77E-06
Voltmeter drift	0.0577	0.0577	0.05773	0.05773	0.05773
Thermocouple homogeneity	0.2483	0.2483	0.2483	0.2483	0.2483
Reference junction	0.1443	0.1443	0.1443	0.1443	0.1443
Combined standard uncertainty U_C (μV)	0.5132	0.4989	0.4979	2.7365	4.6322
Corresponding Combined uncertainty U_C in ^oC	**0.0496**	**0.0437**	**0.0422**	**0.2256**	**0.3844**
Expanded uncertainty U, k=2 (^{o}C)	**0.0987**	**0.0874**	**0.0844**	**0.4512**	**0.7688**

Table 8: Uncertainty budget of thermocouple type R in different fixed points

Expected components of Uncertainty	Fixed point ingots (μV)				
	Al	**Ag**	**Cu**	**Co C**	**Pd C**
Statistical standard uncertainty	0.11	0.13	0.15	0.22	0.33
Determination of the calibration fixed point temperature	0.0058	0.0071885	0.0081	3.102	5.2059
Drifts of the means used to determine the calibration fixed point	0.1919	0.1266	0.1290	0.1380	1.1880
Voltmeter calibration	0.38	0.38	0.38	0.38	0.38
Voltmeter resolution	5.77E-06	5.77E-06	5.77E-06	5.77E-06	5.77E-06
Voltmeter drift	0.0577	0.0577	0.0577	0.0577	0.0577
Thermocouple homogeneity	0.2425	0.2425	0.2425	0.2425	0.2425
Reference junction	0.1443	0.1443	0.1443	0.1443	0.1443
Combined standard uncertainty U_C (μV)	0.5257	0.5102	0.5163	3.1491	5.3711
Corresponding Combined uncertainty U_C in °C	**0.0453**	**0.0390**	**0.0382**	**0.2233**	**0.3817**
Expanded uncertainty U, k=2 (°C)	**0.0906**	**0.0781**	**0.0765**	**0.4467**	**0.7635**

Table 9: Uncertainty budget of thermocouple type Pt/Pd in different fixed points

Expected components of Uncertainty	Fixed point ingots (μV)				
	Al	**Ag**	**Cu**	**Co C**	**Pd C**
Statistical standard uncertainty	0.1	0.1	0.1	0.1	0.1
Determination of the calibration fixed point temperature	0.0071	0.0106	0.0125	5.1920	9.3610
Drifts of the means used to determine the calibration fixed point	0.2332	0.1859	0.1998	0.2309	2.1361
Voltmeter calibration	0.38	0.38	0.38	0.38	0.38
Voltmeter resolution	5.77E-06	5.77E-06	5.77E-06	5.77E-06	5.77E-06
Voltmeter drift	0.0577	0.0577	0.0577	0.0577	0.0577
Thermocouple homogeneity	0.4619	0.4619	0.4619	0.4619	0.4619
Reference junction	0.15299	0.15299	0.15299	0.15299	0.15299
Combined standard uncertainty U_C (μV)	0.6700	0.6551	0.6592	5.2349	9.6222
Corresponding Combined uncertainty U_C in °C	**0.0475**	**0.0341**	**0.0315**	**0.222**	**0.3803**
Expanded uncertainty U, k=2 (°C)	**0.0950**	**0.0682**	**0.0631**	**0.4436**	**0.7606**

4. Conclusion

High temperature thermocouple calibration activities at NIS-Egypt have developed significantly, represented in the following;

- Assembly a set of noble metal thermocouples to be the lab reference standards,
- Bring updated Nanovoltmeters to measure thermocouple output with high accuracy,
- Using a complete set of ITS-90 fixed points and for thermocouple calibration and eutectic fixed points to cover the temperature range from 660 up to 1500°C,
- Significant development of measurement capabilities through improved uncertainty values to be about 0.1 °C up to 0.7 °C in the temperature range from 660 up to 1500°C.

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