PROFICIENCY TEST REPORT

Type K Thermocouple Wire
100 °C to 1100 °C

Tested for the
IAAC Calibration Laboratories Accreditation Program

May 2014

08 May 2014

NIST
NVLAP
Attn.: Barbara Belzer
100 Bureau Dr., MS 2140
Gaithersburg, MD 20899

Subject: IAAC Proficiency Test for Type K Thermocouples

Dear Mrs. Belzer:

A second round of a type K thermocouple proficiency test was performed for four laboratories accredited by IAAC (designated by identification codes A, F, G, and H) over the temperature range of 100 °C to 1100 °C using a calibrated lot of bare type K thermocouple wire as the transfer standards.

Description of the Test

Type K thermocouples are one of the most commonly used temperature sensors in industry. The skills and facilities necessary for testing of type K thermocouples are also applicable to the testing of other base metal thermocouples, and, to a lesser extent, testing of platinum-rhodium alloy thermocouples.

A medium size wire gauge was acquired. The 16 gauge wire that was distributed provides a moderate level of difficulty in handling. Medium size wire diameters are relatively more difficult for the testing laboratory, so the proficiency test is more stringent compared to a test of fine diameter wire (e.g., 28 gauge). Second, experience at NIST with small diameter wire has shown that very large thermoelectric drifts occur at temperatures above 800 °C, and such drift would compromise the reliability of the proficiency test.

The testing of type K thermocouple wire above approximately 200 °C is considered destructive. Therefore, each participating laboratory was sent new, untested wire from the lot of material characterized by NIST. Two type K thermocouple wire sets were shipped to the participants in a coil of radius similar to the coil of the originating lot to prevent significant mechanical strain.

NIST Characterization of the Transfer Standard

The NIST Temperature and Humidity Group acquired 60 m of type K, uninsulated, 1.63 mm diameter (16 gauge) thermocouple wire that was cut into 1.1 m lengths. Each cut was numbered consecutively from one end of the wire.

To evaluate the thermoelectric inhomogeneity and the average emf versus temperature response of the wire, selected cuts from the lot were calibrated by two separate methods. For test temperatures of 500 °C and below, four thermocouples were tested by comparison to an ITS-90 calibrated Standard Platinum Resistance Thermometer (SPRT) in stirred liquid baths – an oil bath for 100 °C and 200 °C, and a salt bath for 400 °C and 500 °C. Four samples from the lot were tested in a horizontal tube furnace by comparison with a calibrated type S thermocouple, over the temperature range 100 °C to 1100 °C. An additional two samples were tested in the stirred liquid baths at 400 °C and 500 °C, and four others were tested in the tube furnace at 100 °C, 500 °C and 1000 °C for repeatability and drift information. Test methods are described in NIST Special Publication 250-35 and NISTIR 5340.

As shown in Table 1, for each of the sets of thermocouples calibrated by the two methods, the standard deviation of the emf readings at each temperature was calculated. There were no statistically significant differences between the standard deviations measured in each apparatus, so the results were pooled to obtain the second column from the left of Table 1. Values in this column give the Type A uncertainties of the NIST measurements. This uncertainty includes both test repeatability and thermoelectric inhomogeneity of the tested wire lot. The lot uniformity was remarkable, especially at temperatures above 500 °C. No trends were observed in the emf of one end of the lot versus the other end, and no outliers were seen. Thus, the emf versus temperature response of any one cut can be assumed equal to the average response of the tested cuts.

Table 1. Thermocouple inhomogeneity and repeatability. *s*: standard deviation; df: degrees of freedom.

	Repeatability		Tube furnace		Stirred baths + SPRT		Pooled	
Temperature °C	*s*	df	*s* µV	df	*s* µV	df	*s* µV	df
100	0.88	7	1.58	3	0.17	3	1.1	7
200			1.71	3	0.89	3	1.8	7
400	1.24	1	1.45	3	1.62	3	2.7	7
500	2.70	8	3.20	3	2.09	3	2.7	7
600			3.35	3			2.4	6
800			2.44	3			1.8	6
1000	2.29	3	4.92	3			1.9	6
1100			4.25	3			2.1	6

Because the calibration using stirred baths and an SPRT as a reference thermometer has significantly lower Type B uncertainties than that of the calibration in the tube furnace, the average emf for the lot was obtained by averaging the emf values obtained in the stirred baths for temperatures of 500 °C and below, and averaging the emf values obtained in the tube furnace for higher temperatures. Figure 1 shows the measured deviation *D* of each thermocouple, as expressed in units of equivalent temperature, from the type K reference function.

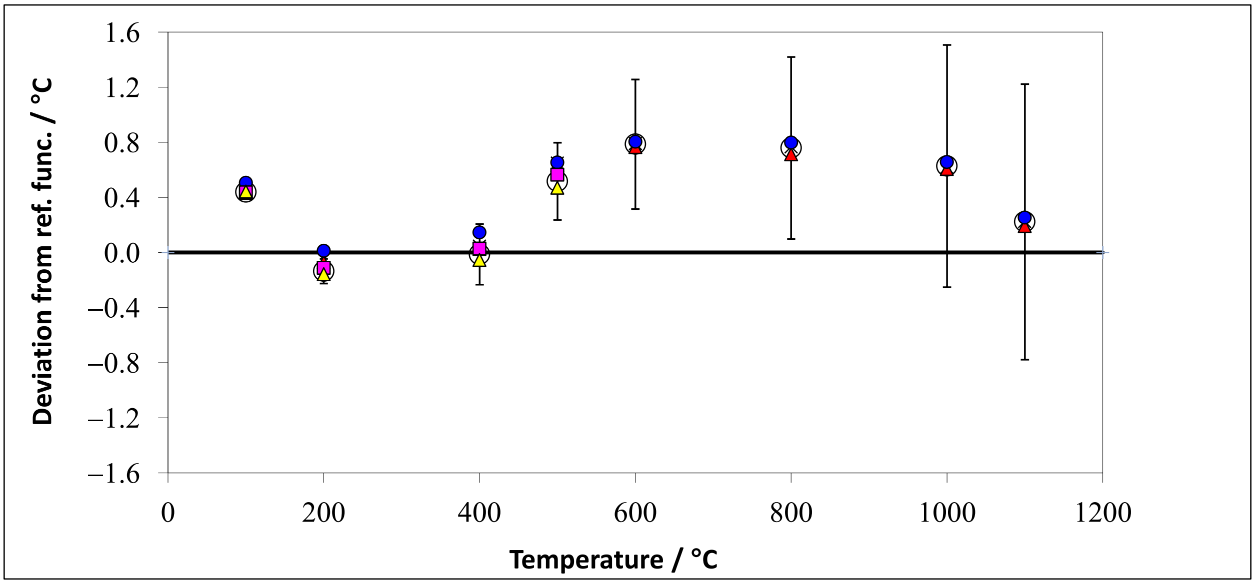

Fig.1. Deviation of thermocouple readings from the reference function for type K thermocouples. The uncertainty bars are *U*(NIST).

Measurement Uncertainties

The uncertainty budget is shown in Table 2. Components listed under “NIST Lot Calibration” are the uncertainties for determination of the average emf-temperature response of the lot. There is an additional uncertainty, listed under the “Comparison” column, in approximating the lot by the average of two test thermocouples sent to each participant. The combined expanded uncertainty (*k*=2), *U*(NIST), equals the uncertainty of the emf-temperature response of the average of the two samples sent to each laboratory.

In Table 2, the Type A uncertainties were evaluated by the formula $u_A = s/\sqrt{n}$, where s is the standard deviation from Table 1, n is equal either to the number of thermocouples used in the determination of the emf average (4), or to the number of thermocouples tested by each laboratory (2).

The dominant components of *U*(NIST), in addition to the thermocouple inhomogeneity, are furnace uniformity and test thermocouple drift. Furnace uniformity for the tube furnace was assessed by comparing measurements of the same lot of thermocouples taken in both the tube furnace and the stirred-liquid baths. The average difference was linear in temperature from 100 °C to 500 °C. This difference was taken as the standard uncertainty for the tube furnace uniformity, and extrapolated up to 1100 °C. Thermocouple drift was evaluated by independent measurements on a separate lot of type K wire. Several cuts of wire were calibrated with a total time of heating of either approximately 2 h or 7 h. The average difference of the emf for the two calibration methods was taken as the standard uncertainty of the emf due to thermoelectric changes (drift) in the wire during the calibration.

Table 2. Measurement uncertainty for emf-temperature response of the lot of type K thermocouple wire.

	Type A		Type B						U(NIST)
Temperature	Inh. & Rep.	Ref. Junct.	Ref. therm.	Furnace / Bath Unif.	EMF meas.	TC drift	inhomogeneity comparison	Combined
K	K	K	K	K	K	K	K	k=2
100	0.01	0.001	0.001	0.001	0.003	0.00	0.02	0.04
200	0.02	0.001	0.001	0.001	0.003	0.01	0.02	0.06
400	0.02	0.001	0.001	0.003	0.003	0.09	0.03	0.19
500	0.03	0.001	0.001	0.003	0.004	0.13	0.05	0.28
600	0.04	0.001	0.067	0.157	0.004	0.17	0.06	0.50
800	0.03	0.001	0.067	0.210	0.005	0.25	0.04	0.68
1000	0.05	0.001	0.067	0.262	0.006	0.35	0.07	0.90
1100	0.06	0.001	0.076	0.288	0.007	0.40	0.08	1.02

Evaluation of Degree of Equivalence with NIST

Table 3 gives the comparison results, the expanded uncertainty of each participating laboratory, *U*(Lab X) (*k*=2), the uncertainty of the NIST-determined emf-temperature response, *U*(NIST), the combined uncertainty U_c (*k*=2), and the degree of equivalence values, E_n, for each temperature of the proficiency test. The participating laboratory results from the two supplied type K thermocouple wire sets were averaged. In several cases, the laboratory uncertainty on the calibration certificate did not agree with the uncertainty supplied in the proficiency test survey. The E_n values are given for both sets of uncertainty values. The E_n results are computed for the average D_A(Lab X) of both tested thermocouples for each laboratory. E_n is calculated as:

$$E_n = \frac{D_A(\text{Lab X}) - D(\text{NIST})}{\sqrt{U(\text{NIST})^2 + U(\text{Lab X})^2}} \quad .$$

An $|E_\text{n}| < 1$ signifies compliance for the participating company.

Table 3. Proficiency test results for the four participating laboratories. An $|E_\text{n}| < 1$ signifies compliance for the participating company. E_n values in bold red text with a pink background are for those values larger 1.

	Laboratory E_n			
t, °C	**A**	**F**	**G**	**H**
100	0.1	0.2	0.4	0.3
200	0.1	0.3	0.0	0.3
400	0.4	0.0	0.1	0.0
500	0.5	0.1	0.2	**1.3**
600	0.2			**1.3**
800	0.0			0.3
1000	0.2			0.2
1100	0.7			0.1

Figures 2-5 individually shows the IAAC proficiency test results for each participating laboratory with respect to NIST.

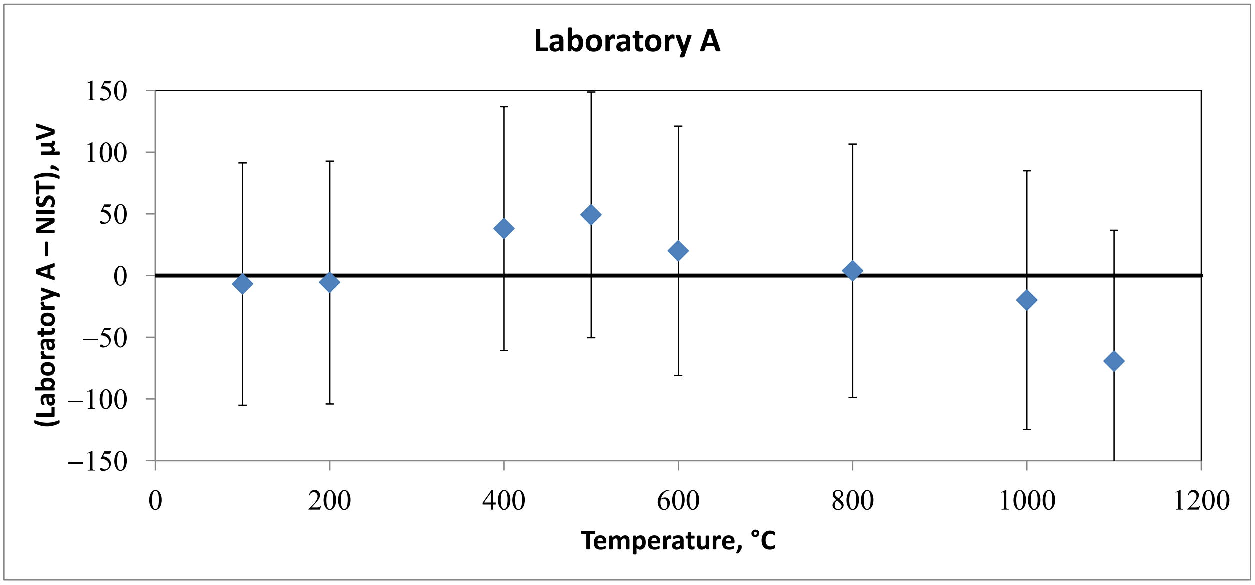

Fig. 2. Type K Thermocouple proficiency test results for IAAC participant laboratory A.

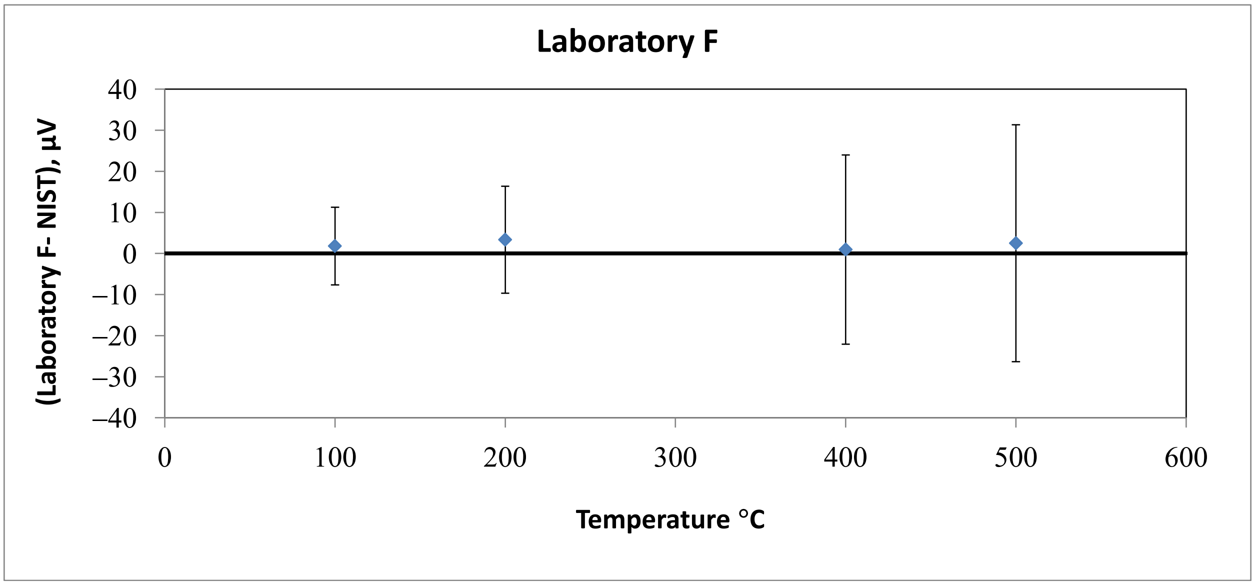

Fig. 3. Type K Thermocouple proficiency test results for IAAC participant laboratory F.

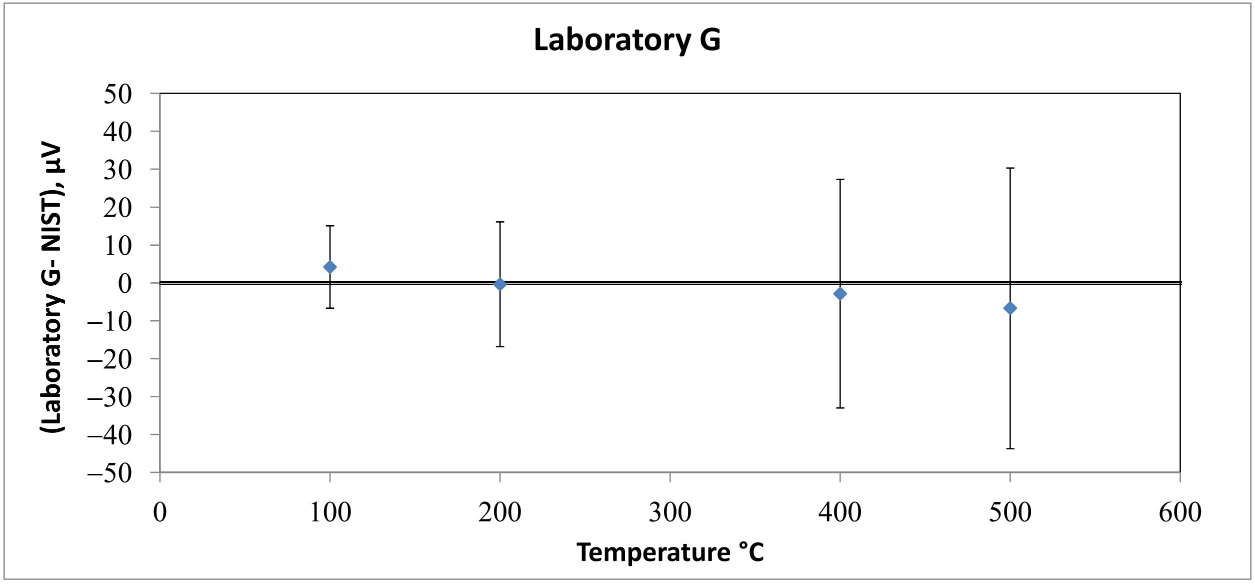

Fig. 4. Type K Thermocouple proficiency test results for IAAC participant laboratory G.

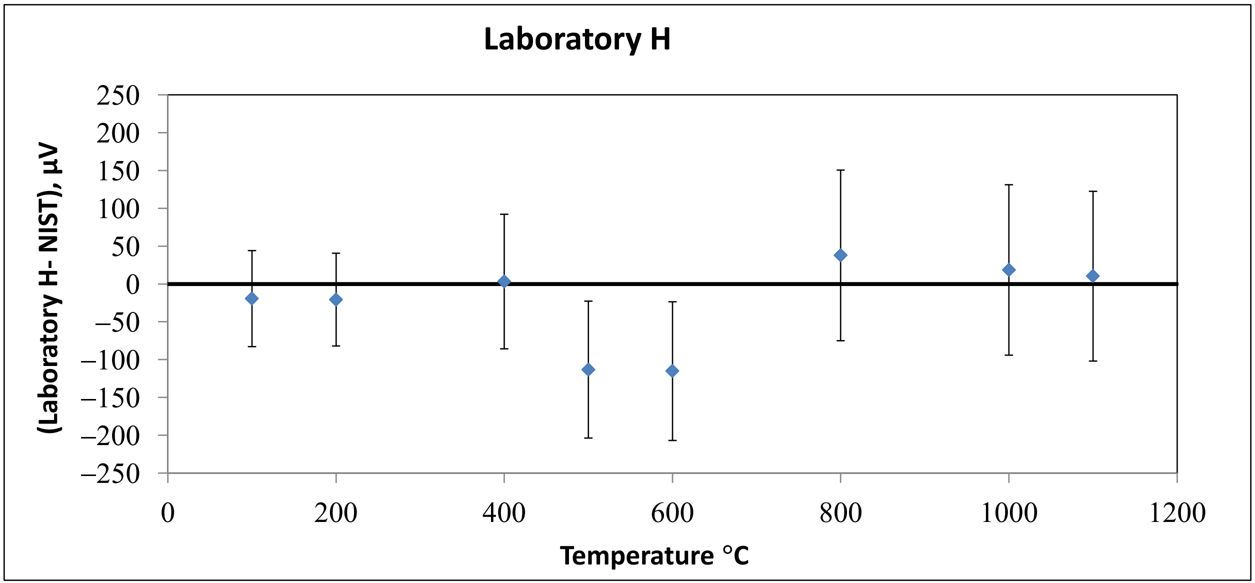

Fig. 5. Type K Thermocouple proficiency test results for IAAC participant laboratory H.

Discussion of Survey Responses

The act of calibrating a base metal thermocouple at temperatures greater than approximately 200 °C will itself alter the homogeneity and emf-temperature response of the thermocouple.

Calibration of a base metal thermocouple is typically done for one of two purposes:

a. to establish the emf-temperature response of the particular thermocouple being tested, which the customer will then use at possibly a different immersion as a temperature probe; or

b. to establish the emf-temperature response of a lot of wire or probes, fabricated from the same material as the test thermocouple.

In case a., drift and inhomogeneity induced in the thermocouple during the calibration will directly affect the emf-temperature response of the thermocouple when it is used by the customer. In case b., inhomogeneity of the lot and drift of the thermocouple during test are additional uncertainties in determining the emf-response of another lot sample at the time of first heating. In either case, these are uncertainties inherent in the thermocouple which the customer will incur in application of the calibration result. All of the participants are strongly encouraged to give thermocouple drift and uncertainty values. (Note that these components have been accounted for in U(NIST) so that inclusion of these components will not reduce the E_n values for the present proficiency test.)
Based on the surveys, the data and understood characteristics of base metal thermocouples, the following are general suggestions for base metal calibrations. Above 200 °C the thermoelectric properties of the thermocouple are being altered as you are heating it. Particularly between 300 and 500 °C the thermocouple experiences reversible affects that will alter the emf produced. It is better at all temperatures above to hold the thermocouple at temperature for as little time as possible.

The emf generated may be affected by varying immersion during the calibration. If you decrease the immersion distance, particularly at the higher temperatures you can alter the emf by a magnitude or more. If possible the immersion distance should not change throughout the calibration. It is advisable to switch baths or furnaces as little as possible and if required only at the lowest temperatures.

The more complete the uncertainty budgets were the better the participant did. If unit under test drift and inhomogeneity uncertainties were not included, we added an uncertainty value at each temperature calculated with the CCT WG-8 document equation (http://www.bipm.org/wg/CCT/CCT-WG8/Allowed/CMC_review_protocols/CMC_review_protocol_-_Industrial_thermometers_-_2010-05-061.pdf).

General Comments:
For general guidance on the construction of uncertainty budgets for thermocouple calibrations, the following two references are recommended:

1. *Theory and Practice of Thermoelectric Thermometry*, Vol. 3 of the *Handbook on Temperature Measurements*, by R. Bentley, Springer Verlag, 1999.
2. ASTM Standard Test Method for Calibration of Thermocouples by Comparison Techniques (E220-02), ASTM, West Conshohocken, PA, 2002.

Sincerely,

Gregory Strouse
Leader, Thermodynamic Metrology Group