Report No. 511B

Thermocouple Wire Proficiency Testing

- K TYPE -

September 2006

(Replacement for Report No. 511 – issued July 2006)

Acknowledgments

PTA gratefully wishes to acknowledge the technical assistance provided for this program by Dr Ferdouse Jahan, Scientist of Temperature Standard Group, National Measurement Institute, Australian Government.

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CONTENTS

1. Foreword

This report summarises the results of an interlaboratory comparison on Thermocouple Wire (K Type) calibration by laboratories accredited by the National Association of Testing Authorities, Australia (NATA).

The exercise was conducted during February ~ March 2006 by Proficiency Testing Australia (PTA). The main aim of the program was to assess laboratories' abilities to competently perform the prescribed calibration.

2. Program Features and Design

2.1 Each laboratory was randomly allocated a unique code number for the program to ensure confidentiality of results. Reference to each laboratory in this report is by code number only.

2.2 Laboratories were provided with the "Instructions to Participants" and "Results Sheet" (refer Appendix C & D). NATA accredited laboratories were requested to perform the tests according to their accredited methods.

2.3 Participants were provided with one 1.5 metres of K type thermocouple.

2.4 A total of eight NATA accredited laboratories received samples, and all of them submitted results.

2.4 Results (as reported by participants) with corresponding summary statistics are presented in Appendix A & B for each testing point for each laboratory.

3. Reporting by Participants

Laboratories were asked to report their test results on a proforma *Results Sheet* (refer Appendix D) and also to provide formal calibration reports for consideration of their NATA accredited status. The participating laboratories were asked to report uncertainties in 95% confidence levels.

4. Reference Values

The reference values for the K type thermocouple wire were carried out by National Measurement Institute (NMI), Australian Government.

5. Summary of Results

A summary of the results returned by the participating laboratories, compared to the reference values, appears in Appendix A & B in the tables and graphs. The

graphs plot the difference between each laboratory's result and the reference value (*Lab - Ref*). The general technical comments appears in Section 6.

In accordance with international practice, measurement performance has been assessed on the basis of an E_n ratio for each measurement. The E_n ratios are calculated using a standard statistical technique for comparing values and are derived from the following expression:

$$E_n = \frac{Lab\ Result - Ref\ Value}{\sqrt{(U_{Lab})^2 + (U_{Ref})^2}}$$

where U_{Lab} is the laboratory's reported uncertainty of measurement at a 95% confidence level, and U_{Ref} is the reference uncertainty of measurement at a 95% confidence level. For the results to be acceptable, values of E_n less than unity are required.

Values of $|E_n|>1$ require investigation and corrective action by both the laboratory concerned and its accreditation body.

6. PTA and Technical Adviser's Comments

This proficiency testing was performed up to a temperature of 1100°C. However, not all labs have accreditation up to 1100°C, so the results given by the participants are limited to their accreditation. The summary of the En number calculated at each 100°C interval for all of the participating labs are given in Table 1. The cells in Table 1 are bold font, if the En number is greater than 1.

At temperatures up to 600°C the majority of the labs had shown good performance. However at temperatures above 600°C, none of the labs performed well except lab code 3 & 4. The deviation of the lab's correction from the reference value is larger than the uncertainty quoted by the lab, which gives En number more than 1.

All 8 laboratories provided results at 0 °C, but En numbers were not calculated due to the absence of reference at 0 °C.

Lab code 1 provided results at 100 °C & 200 °C. Although En number is less than 1 at 200 °C, it is greater than 1 at 100 °C.

Lab code 3 performed well in general. This lab sent results up to 900℃. At three temperature points of 300℃, 400℃ and 900℃, En is greater than 1. The deviation from the reference value is not justified by the uncertainty value.

Lab code 4 also performed well. They provided results up to 1100℃. Their results are good up to 800℃, that is, En is less than 1, except at 400℃. Above 800℃, En is greater than 1.

For lab code 6, En is less than 1 at all temperature points up to 600℃, but above 600℃, En is always greater than 1.

Lab code 7 provided results only at 100 ℃ & 200 ℃. En number is less than unity at these temperatures.

Lab code 8 provided results up to 600℃ and only at two temperature points, 300℃ and 400℃, En number is greater than 1.

Lab code 5 and lab code 2 did not perform well. At most of the temperature points, En number is greater than 1, for these two labs.

In Figure 1, the lab corrections given by all labs are plotted as a function of temperature, including the reference lab correction. The figure shows that the dispersion of correction between the labs is within ± 0.5℃ at 100 and 200℃. Up to 600℃, the dispersion of the correction is ± 0.7℃. But above 600℃, the dispersion is large and also deviation from the reference value is large. The deviations from the reference values at each 100 ℃ temperature are plotted in Appendix B. It shows that the deviation is not covered by the estimated uncertainty, which gives En number greater than 1 for almost all the labs at temperatures higher than 600℃.

There are several reasons which could have led to this large deviation. One of these is the calculation of uncertainty. The dominant component of the uncertainty for Type K thermocouple calibration is its inhomogeneity, and it is estimated to be 0.1% for a new thermocouple[1]. Some labs used a smaller value than 0.1%, although there was no evidence of proper measurement of inhomogeneity.

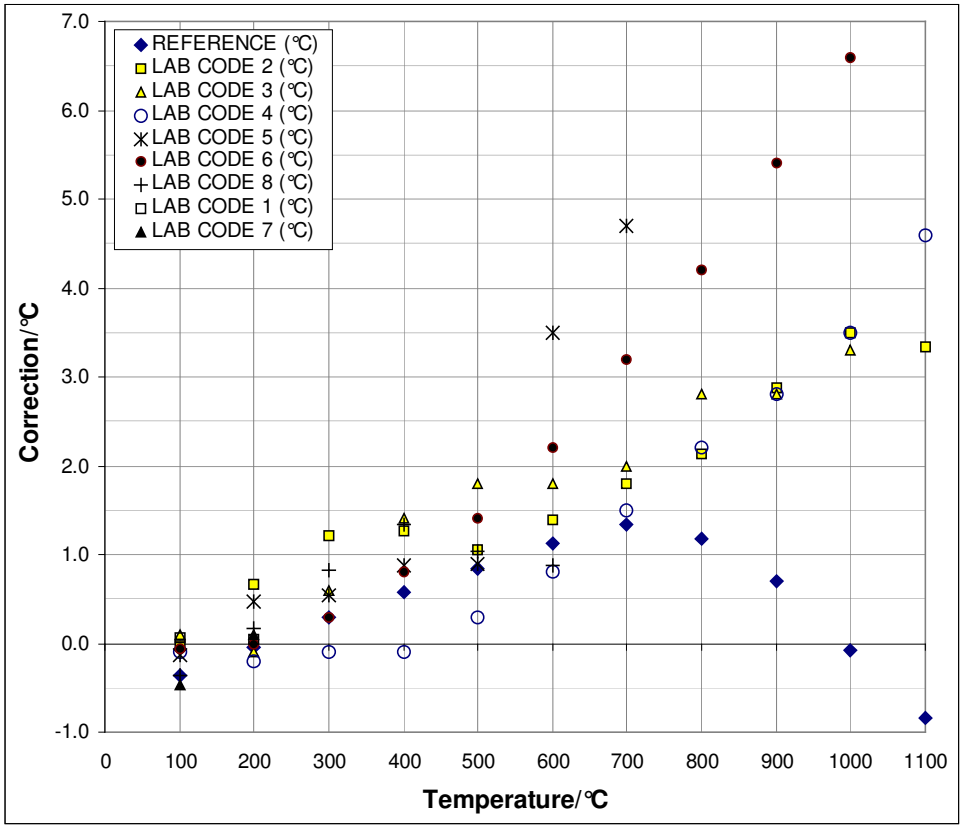
In some cases two different enclosures were used, one up to 600℃ and one above 600℃. The immersion in both enclosures may not be the same. Once the thermocouple was calibrated up to 600℃, the wire might be changed and then calibrating another enclosure with different immersion will give a different correction.The calculation of uncertainty did not incorporating this discrepancy.

One of the major problems of calibrating type K thermocouple is that, the type K thermocouple should always be used in an oxidising environment, and this may be the main cause of the large dispersion in the correction. Type K thermocouples are not recommended for use even in marginally reducing atmosphere, especially at high temperatures[2]. During calibration if the air is

stagnant inside the tube, there is not sufficient oxygen and the atmosphere reduces with time, which causes significant drift in calibration. The amount of drift of the thermocouple will depend with time and temperature and amount of oxygen available at the time. This explains the large variation in the correction between the labs at higher temperatures.

Laboratory should take care during calibration, so that there is enough air flow to supply oxygen.

Figure 1. Calibration results supplied by the participants plotted as a function of temperature, including the reference lab.



It is noted that the variation between laboratories is excessive for a number of test points. Overall however, the results achieved for this round were not good.

Table 1 : Caluacated En numbers at each 100 ^{0}C for all participating labs (Thermocouple Wire -Type K)

APPLIED TEMP. (ºC)	LAB CODE 1	LAB CODE 2	LAB CODE 3	LAB CODE 4	LAB CODE 5	LAB CODE 6	LAB CODE 7	LAB CODE 8
	En	En	En	En	En	En	En	En
0	-	-	-	-	-	-	-	-
100	**1.57**	**1.28**	0.81	0.62	**1.47**	0.90	-0.23	-0.04
200	0.28	**2.26**	0.64	-0.34	**2.47**	0.14	0.28	0.60
300	-	**2.42**	**1.07**	-0.83	0.38	0.01	-	**1.27**
400	-	**1.54**	**1.17**	**-1.33**	0.35	0.53	-	**1.03**
500	-	0.43	0.90	-0.55	0.06	0.71	-	0.23
600	-	0.46	0.43	-0.32	**1.86**	0.91	-	-0.26
700	-	0.69	0.71	0.16	**2.25**	**1.43**	-	-
800	-	**1.25**	0.78	0.95	**8.55**	**2.25**	-	-
900	-	**2.50**	**1.23**	**1.85**	**21.47**	**3.39**	-	-
1000	-	**3.68**	-	**3.00**	**30.39**	**4.66**	-	-
1100	-	**3.90**	-	**4.33**	**34.74**	**5.88**	-	-

$$E_n = \frac{\text{Lab - Ref}}{(U_{95}\text{Lab}^2 + U_{95}\text{Ref}^2)^{1/2}}$$

En: Values of E_n less that unity are reqired for the measurement to be acceptable.

7. References

[1]. Guide to Proficiency Testing Australia, 2006, Version 1. 1. (PTA website: www.proficiencytesting.com.au under “Documents”).

[2]. R E Bentley, “ Theory and Practice of Thermoelectric Thermometry” Vol.1, Springer-Verlag, Singapore, 1998.

[3]. T. P. Wang & C. D. Starr. ‘Oxidation resistance and stability of Nicrosil-Nisil in air and in reducing atmospheres. In J. F. Schooley, editor, Temperature: its Measurement and Control in Science and Industry, Vol. 5, 1147-48, 1982.

APPENDIX A

Summary of Reported Results

SUMMARY OF REPORTED RESULTS

THERMOCOUPLE WIRE - Type K

Applied Temp (ºC)	LAB CODE 1 (℃)		LAB - REF (℃)	En	LAB CODE 2 (℃)		LAB - REF (℃)	En	LAB CODE 3 (℃)		LAB - REF (℃)	En
	CORRECTION	U_{LAB}			CORRECTION	U_{LAB}			CORRECTION	U_{LAB}		
0	-0.01	0.2	N/A	N/A	0.09	0.23	N/A	N/A	0.1	0.2	N/A	N/A
100	0.07	0.25	0.43	**1.57**	-0.02	0.24	0.34	**1.28**	-0.1	0.3	0.26	0.81
200	0.05	0.3	0.10	0.28	0.67	0.26	0.72	**2.26**	0.6	1.0	0.65	0.64
300	-	-	-	-	1.22	0.29	0.93	**2.42**	1.4	1.0	1.11	**1.07**
400	-	-	-	-	1.26	0.32	0.69	**1.54**	1.8	1.0	1.23	**1.17**
500	-	-	-	-	1.06	0.35	0.22	0.43	1.8	1.0	0.96	0.90
600	-	-	-	-	1.39	0.39	0.27	0.46	2.0	2.0	0.88	0.43
700	-	-	-	-	1.80	0.43	0.47	0.69	2.8	2.0	1.47	0.71
800	-	-	-	-	2.14	0.48	0.97	**1.25**	2.8	2.0	1.63	0.78
900	-	-	-	-	2.87	0.52	2.17	**2.50**	3.3	2.0	2.60	**1.23**
1000	-	-	-	-	3.49	0.57	3.57	**3.68**	-	-	-	-
1100	-	-	-	-	3.34	0.62	4.18	**3.90**	-	-	-	-

E_n: Values of **E_n** less that unity are reqired for the measurement to be acceptable.

Results: Reported correction results

Lab-Ref: Difference of lab & reference

$$E_n = \frac{\text{Lab - Ref}}{(U_{95}Lab^2 + U_{95}Ref^2)^{1/2}}$$

NMI: National Measurement Institute Australian Government

U_{95}: Uncertainty at 95% confidence level

Ref: Reference correction values from **NMI**

Table 2

SUMMARY OF REPORTED RESULTS

THERMOCOUPLE WIRE -Type K

Applied Temp (ºC)	LAB CODE 4 (℃)		LAB - REF (℃)	En	LAB CODE 5 (℃)		LAB - REF (℃)	En	LAB CODE 6 (℃)		LAB - REF (℃)	En
	CORRECTION	U_{LAB}			CORRECTION	U_{LAB}			CORRECTION	U_{LAB}		
0	-0.1	0.4	N/A	N/A	0.12	0.1	N/A	N/A	0.10	0.1	N/A	N/A
100	-0.1	0.4	0.26	0.62	-0.14	0.1	0.22	**1.47**	-0.07	0.3	0.29	0.90
200	-0.2	0.4	-0.15	-0.34	0.47	0.1	0.52	**2.47**	0.00	0.3	0.05	0.14
300	-0.1	0.4	-0.39	-0.83	0.54	0.6	0.25	0.38	0.30	0.6	0.01	0.01
400	-0.1	0.4	-0.67	**-1.33**	0.87	0.8	0.30	0.35	0.80	0.3	0.23	0.53
500	0.3	0.9	-0.54	-0.55	0.90	1.0	0.06	0.06	1.4	0.7	0.56	0.71
600	0.8	0.9	-0.32	-0.32	3.50	1.2	2.38	**1.86**	2.2	1.1	1.08	0.91
700	1.5	0.9	0.17	0.16	4.70	1.4	3.37	**2.25**	3.2	1.2	1.87	**1.43**
800	2.2	0.9	1.03	0.95	15.81	1.6	14.64	**8.55**	4.2	1.2	3.03	**2.25**
900	2.8	0.9	2.10	**1.85**	40.13	1.7	39.43	**21.47**	5.4	1.2	4.70	**3.39**
1000	3.5	0.9	3.58	**3.00**	62.39	1.9	62.47	**30.39**	6.6	1.2	6.68	**4.66**
1100	4.6	0.9	5.44	**4.33**	78.20	2.1	79.04	**34.74**	7.9	1.2	8.74	**5.88**

E_n: Values of **E_n** less that unity are reqired for the measurement to be acceptable.

Results: Reported correction results

Lab-Ref: Difference of lab & reference

$$E_n = \frac{\text{Lab - Ref}}{(U_{95}Lab^2 + U_{95}Ref^2)^{1/2}}$$

NMI: National Measurement Institute Australian Government

U_{95}: Uncertainty at 95% confidence level

Ref: Reference correction values from **NMI**

Table 3

SUMMARY OF REPORTED RESULTS

THERMOCOUPLE WIRE - Type K

Applied Temp (ºC)	Lab Code 7 (℃)		Lab - Ref (℃)	En	Lab Code 8 (℃)		Lab - Ref (℃)	En	Lab Code (℃)		Lab - Ref (℃)	En
	Results	U_{Lab}			Results	U_{Lab}			Results	U_{Lab}		
0	0.00	0.5	N/A	N/A	0.028	0.19	N/A	N/A				
100	-0.48	0.5	-0.12	-0.23	-0.37	0.28	-0.01	-0.04				
200	0.10	0.5	0.15	0.28	0.16	0.30	0.21	0.60				
300	N/A	N/A	N/A	N/A	0.83	0.34	0.54	**1.27**				
400	N/A	N/A	N/A	N/A	1.33	0.67	0.76	**1.03**				
500	N/A	N/A	N/A	N/A	1.03	0.75	0.19	0.23				
600	N/A	N/A	N/A	N/A	0.88	0.8	-0.24	-0.26				
700	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A				
800	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A				
900	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A				
1000	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A				
1100	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A				

E_n: Values of **E_n** less that unity are reqired for the measurement to be acceptable.

Results: Reported correction results

Lab-Ref: Difference of lab & reference

$$E_n = \frac{\text{Lab - Ref}}{(U_{95}Lab^2 + U_{95}Ref^2)^{1/2}}$$

NMI: National Measurement Institute Australian Governmer

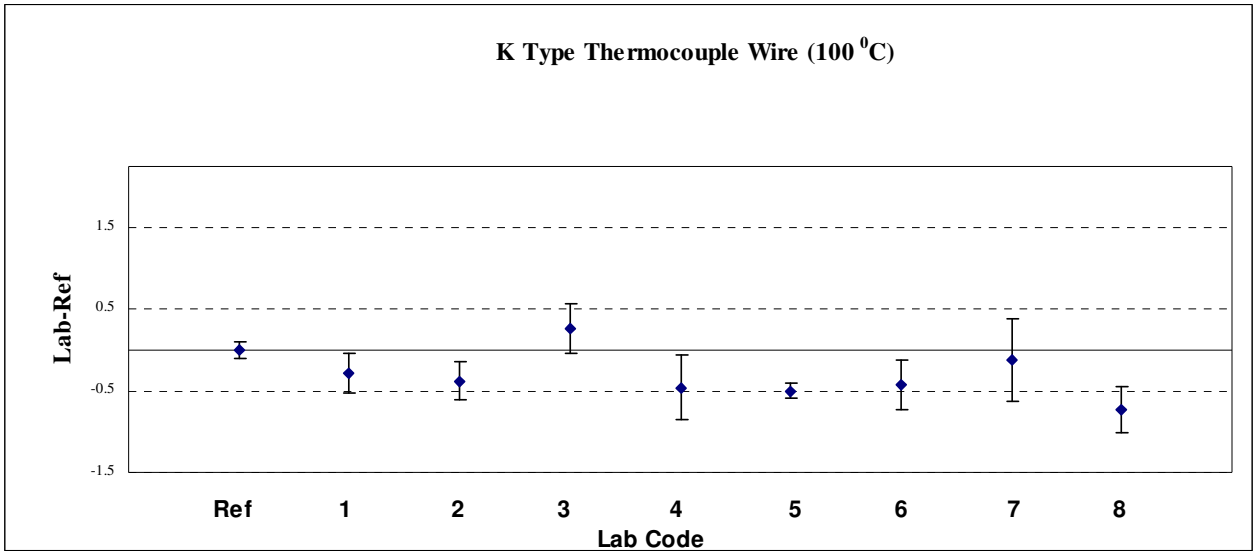
U_{95}: Uncertainty at 95% confidence level

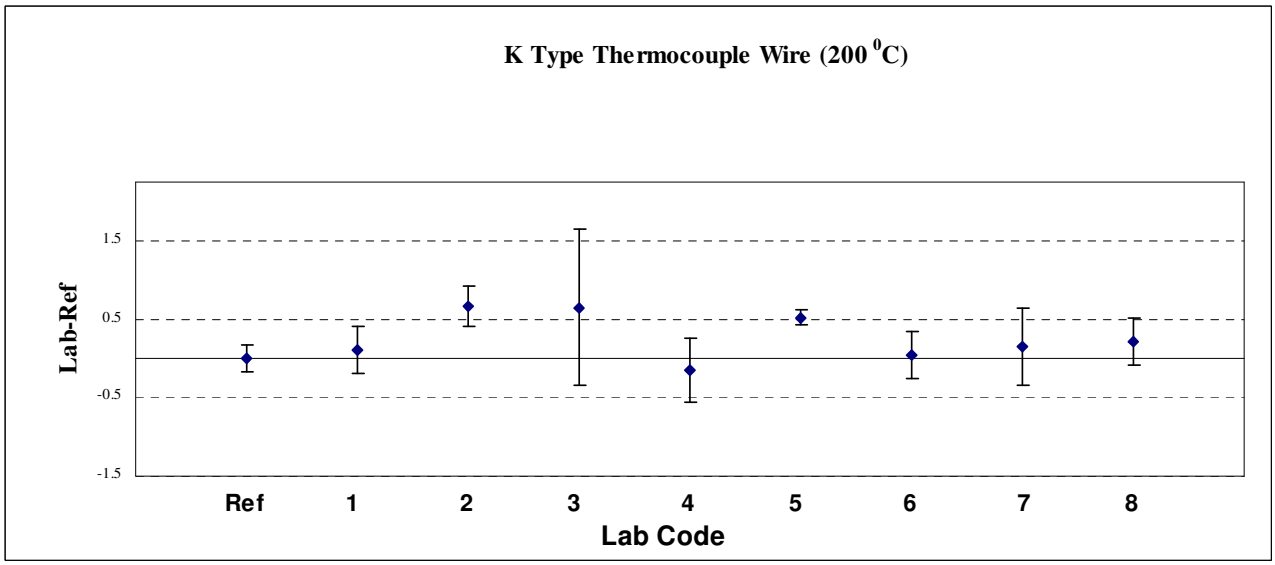
Ref: Reference correction values from **NMI**

Table 4

APPENDIX B

Plots for Difference of Reported Results & Reference Values at each 100 °C with the Reported Uncertainty


K Type Thermocouple Wire (100 ^{0}C)
Lab-Ref
1.5
0.5
-0.5
-1.5
Ref
1
2
3
4
5
6
7
8
Lab Code


K Type Thermocouple Wire (200 ^{0}C)
Lab-Ref
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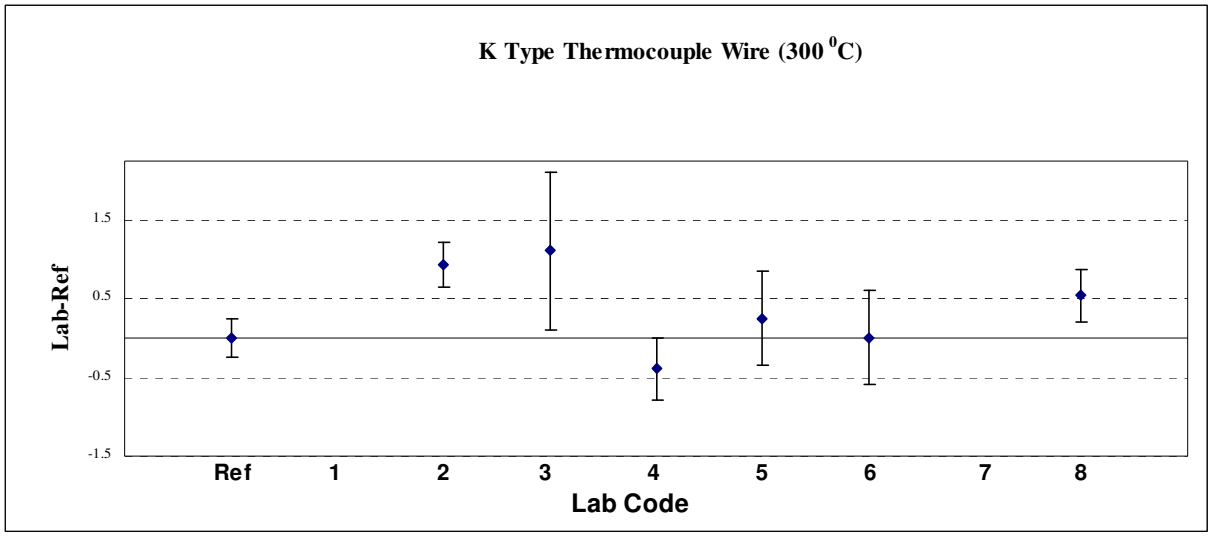
0.5
-0.5
-1.5
Ref
1
2
3
4
5
6
7
8
Lab Code


K Type Thermocouple Wire (300 ^{0}C)
Lab-Ref
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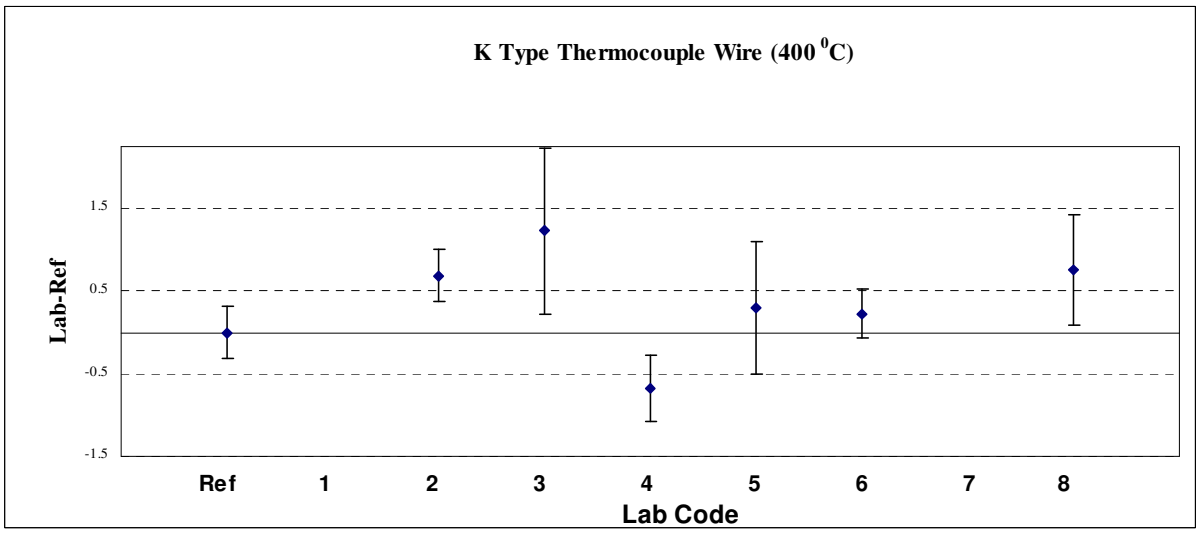
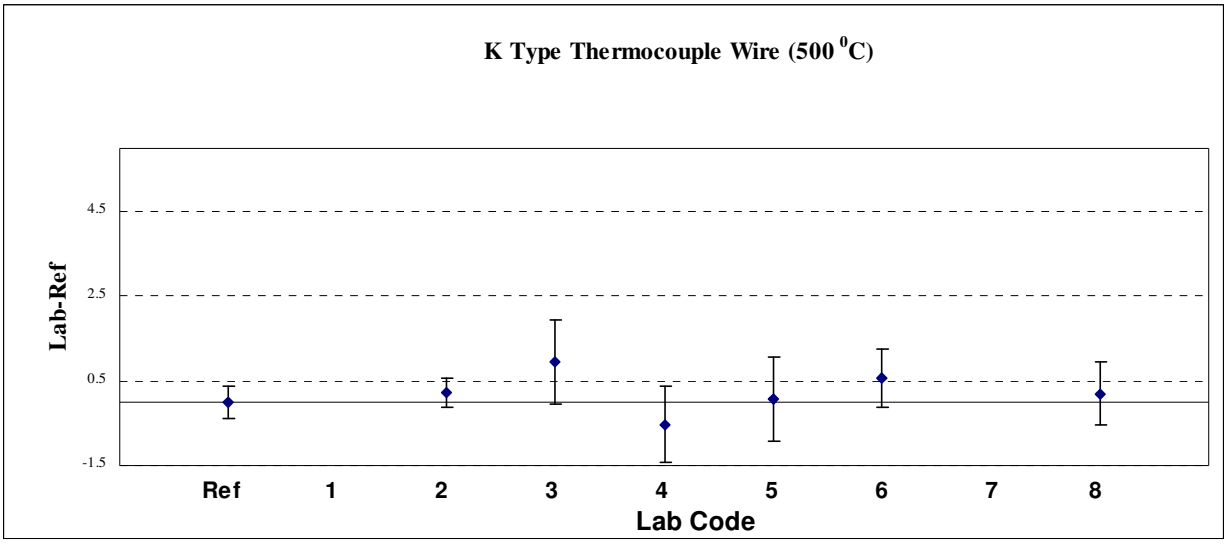
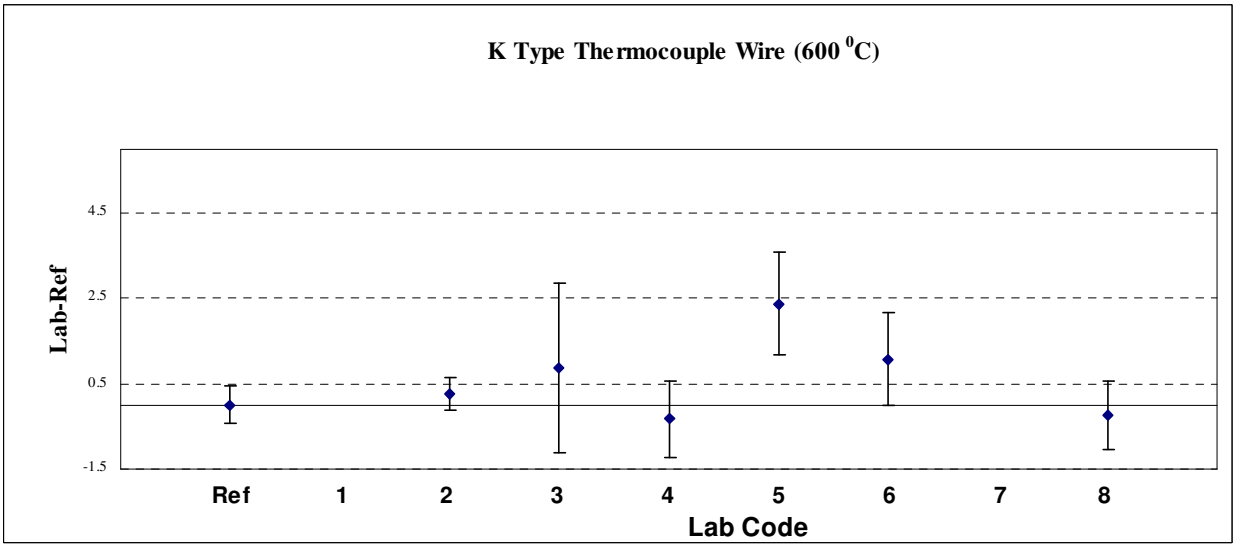
0.5
-0.5
-1.5
Ref
1
2
3
4
5
6
7
8
Lab Code


K Type Thermocouple Wire (400 ^{0}C)
Lab-Ref
1.5
0.5
-0.5
-1.5
Ref
1
2
3
4
5
6
7
8
Lab Code


K Type Thermocouple Wire (500 °C)
Lab-Ref
4.5
2.5
0.5
-1.5
Ref
1
2
3
4
5
6
7
8
Lab Code


K Type Thermocouple Wire (600 °C)
Lab-Ref
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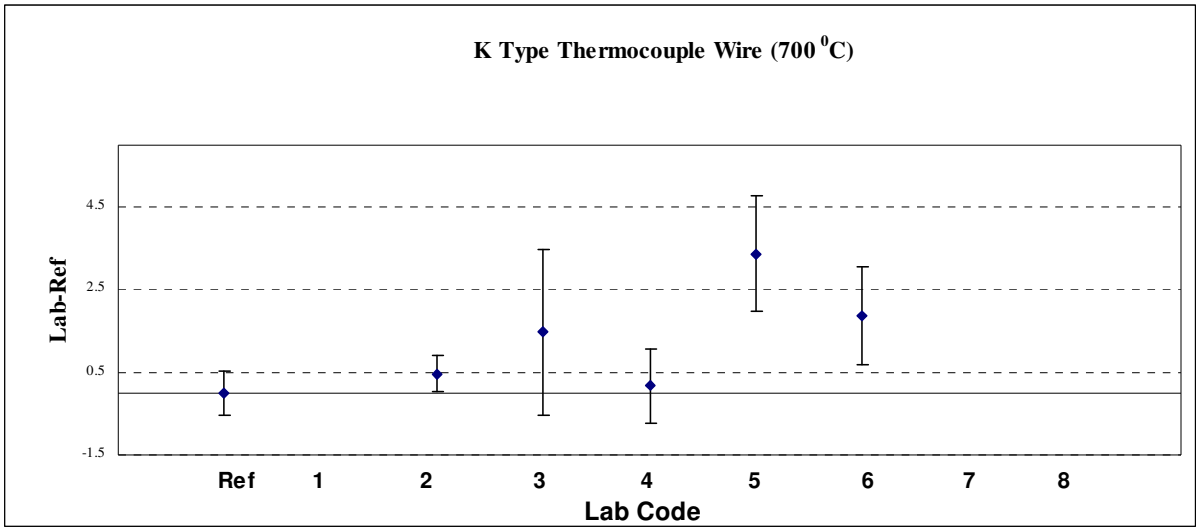
2.5
0.5
-1.5
Ref
1
2
3
4
5
6
7
8
Lab Code


K Type Thermocouple Wire (700 0C)
Lab-Ref
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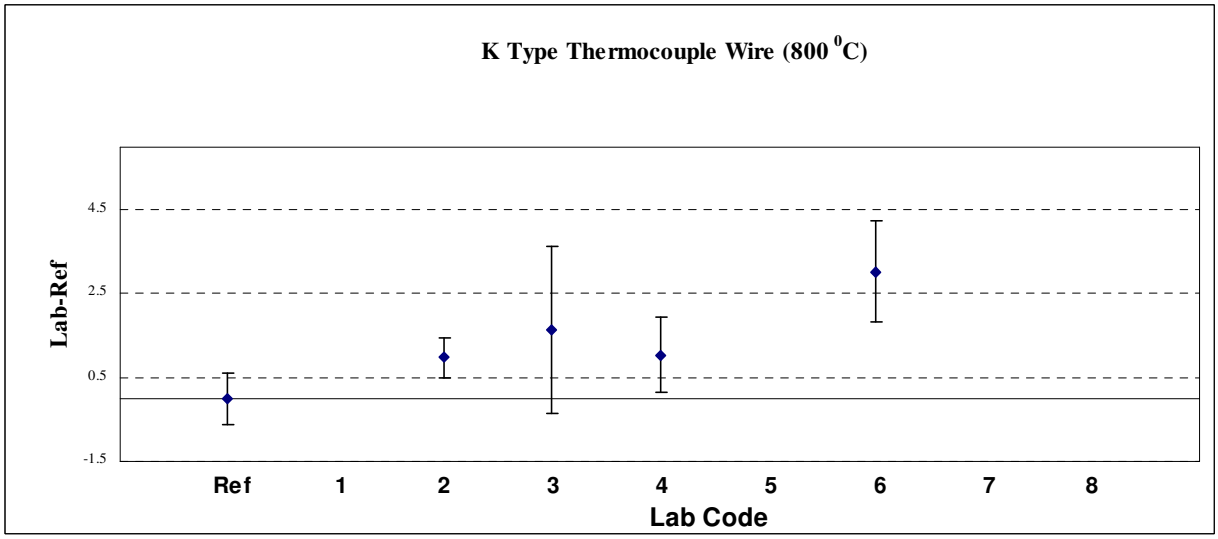
2.5
0.5
-1.5
Ref
1
2
3
4
5
6
7
8
Lab Code


K Type Thermocouple Wire (800 0C)
Lab-Ref
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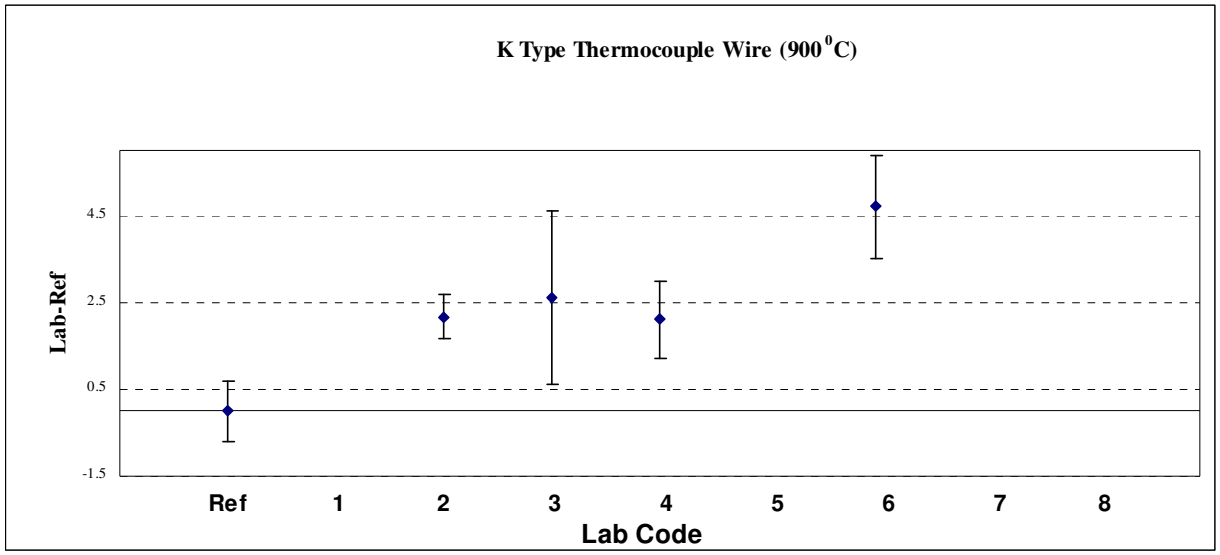
2.5
0.5
-1.5
Ref
1
2
3
4
5
6
7
8
Lab Code


K Type Thermocouple Wire (900 0C)
Lab-Ref
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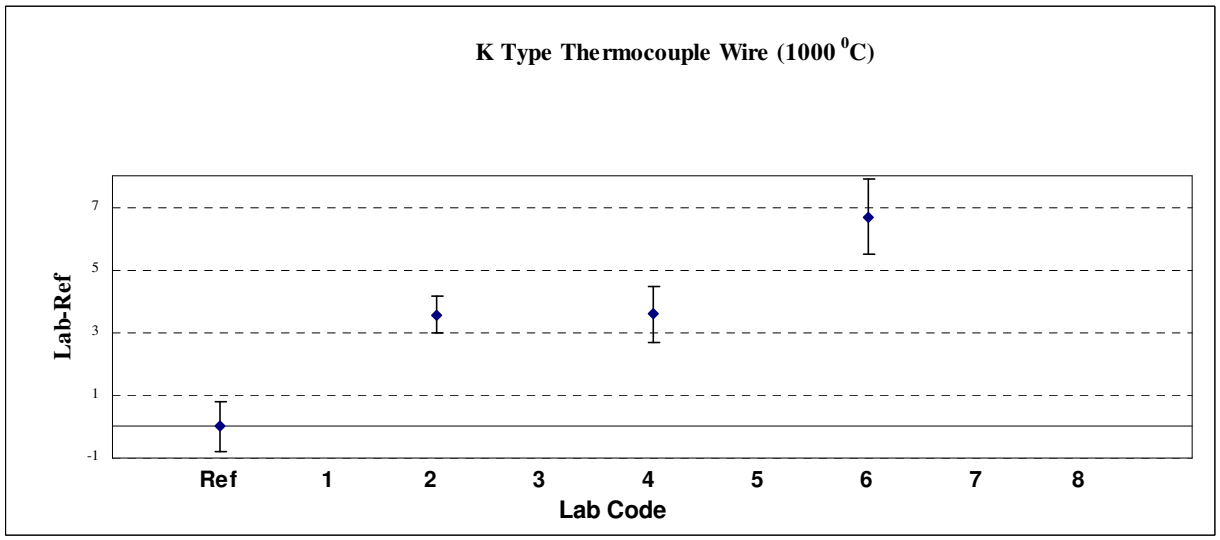
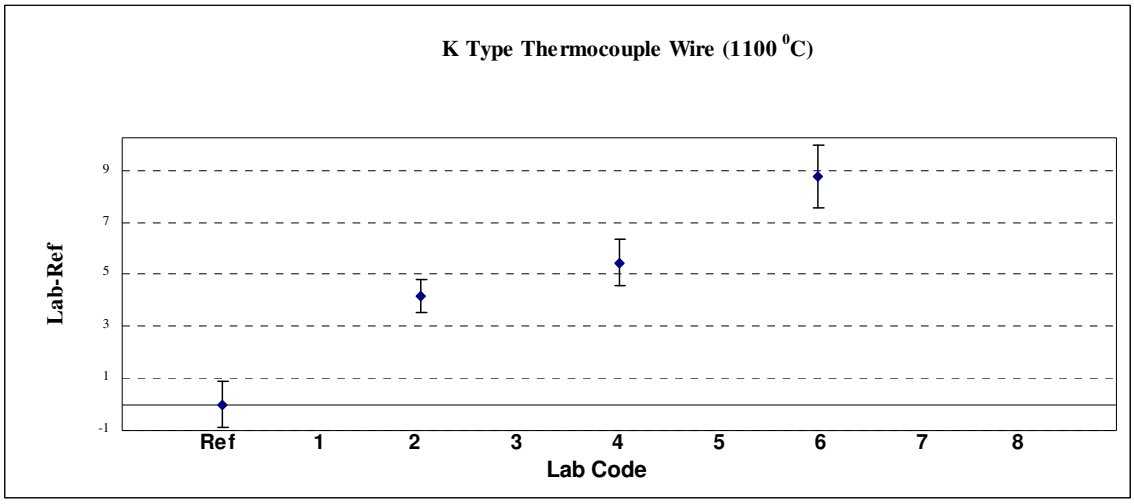
2.5
0.5
-1.5
Ref
1
2
3
4
5
6
7
8
Lab Code


K Type Thermocouple Wire (1000 0C)
Lab-Ref
7
5
3
1
-1
Ref
1
2
3
4
5
6
7
8
Lab Code


K Type Thermocouple Wire (1100 ^{0}C)
Lab-Ref
9
7
5
3
1
-1
Ref
1
2
3
4
5
6
7
8
Lab Code

APPENDIX C

Instruction to Participants

PTA PROFICIENCY TESTING PROGRAM

THERMOCOUPLE WIRE (K TYPE)

INSTRUCTIONS TO PARTICIPANTS

To ensure that results from this program can be analysed properly, participants are asked to adhere carefully to the following instructions.

1. Use test methods and equipment that will enable you to work to your proposed least uncertainties of measurement. Participants are welcome to report results for any other tests for which NATA accreditation is not held.

2. Make the thermocouple junction.

3. Do not anneal - calibrate in the “as received” condition.

4. Calibrate from 0°C to 1100°C (in 100°C steps).

5. Report formally with a proposed NATA endorsed report. Include in the report a table and graph (curve of best fit) for temperature vs $E - E_{ref}$.

6. Provide worksheets, NATA endorsed report and details of derivation of uncertainties of measurement.

7. Laboratories are also requested to calculate and report an estimate of measurement uncertainty (MU) for each reported measurement result. All estimates of measurement uncertainty must be given as a 95% confidence interval (coverage factor $k \approx 2$).

8. Testing may commence as soon as samples are received. All laboratories must return worksheets, NATA endorsed report and details of derivation of uncertainties of measurement no later than **10 February 2006** to:

Dr Michael Li
Proficiency Testing Australia
Silverwater NSW 2128

Telephone: (02) 9736 8397
Fax: (02) 9743 6664

APPENDIX D

Results Sheet

PROFICIENCY TESTING AUSTRALIA

THERMOCOUPLE WIRE PROFICIENCY TESTING PROGRAM

- K TYPE-

Lab Code

RESULTS SHEET

Laboratory Name:
Accreditation No:
Ambient Temperature:
Calibration Date:
Report No:
Report Date:

APPLIED TEMP. (ºC)	LAB (°C)	
	CORRECTION	U_{LAB}
0		
100		
200		
300		
400		
500		
600		
700		
800		
900		
1000		
1100		

Signed: Date: