INTERLABORATORY COMPARISON OF A DIGITAL THERMOMETER WITH GOLD PLATINUM THERMOCOUPLE

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Abstract: This paper presents the most relevant results of an interlaboratory comparison of a digital thermometer, which involved three calibration laboratories accredited by RBC, the Brazilian Calibration Network. The artifact under evaluation in this comparison is a digital thermometer with resolution of 0.01 °C and a gold-platinum thermocouple. The calibrations were performed by comparison with temperature standards, in furnaces and baths, in the following temperatures: 0 °C, 230 °C, 420 °C, 660 °C, and 960 °C. The results were evaluated using three different approaches: normalized error, whiskers chart and Youden diagrams.

Key-words: Au/Pt thermocouple, digital thermometer, interlaboratory comparison.

1. INTRODUCTION

According to some technical standards and publications in metrology [1,2], a laboratory shall assure the quality of calibration and measurement results, in order to evidence the laboratory technical competence and measurement capability. This requirement may be achieved by taking part in interlaboratory programs, since the laboratory best measurement capability can be evaluated. This work involved three secondary calibration laboratories, accredited by the Brazilian Calibration Network (RBC), which decided to undertake an interlaboratory comparison program using a digital thermometer as the artifact.

The laboratories have similar best measurement capability for calibration of this kind of instrument in their accreditation scope. The definition of the type and quality of the artifact used in the comparison was based on the growing application of digital thermometers in laboratorial, industrial and scientific areas. The choice was also based on the higher accuracy, reproducibility, lead-wire homogeneity and stability of the Au/Pt thermocouple, when compared with S- or R-type thermocouples.

Another relevant feature is related to the stability of the indicator and thermocouple. Regarding the round robin realization, the laboratories should observe good metrology practices, paying special attention to material handling and transportation, and specific rules, e.g., the laboratory shall undertake the calibration following its own procedure.

All laboratories performed two calibrations of the artifact (two rounds were performed), in order to detect any possible drift. The reports issued by the laboratories showed the calibration results including their best measurement capability for each temperature calibration point. The results were evaluated using three different methods: normalized error (En), whiskers chart and Youden diagrams. The normalized error was calculated between each participant and also considering the artifact calibrations performed by the Brazilian NMI - Diter/Inmetro, in January 2008 and June 2011.

2. MEASUREMENTS

2.1 Artifact

The higher performance of Au/Pt thermocouples in terms of accuracy, stability and lead-wire homogeneity, however, impacted the preliminary consideration, and thus the participants have decided to perform a comparison of Au/Pt thermocouple connected to a digital thermometer [3,4,5].

The Au/Pt thermocouple has the following technical features: gold and platinum wires with 99.999% purity assembled on ceramic capillary, protection sheath of quartz with diameter of 7 mm and length of 560 mm, measurement junction with thermoelements of gold and platinum soldered directly, reference junction with stainless steel sheath. The digital indicator connected to the thermocouple features a resolution of 0.01 °C and an operating range from 0 °C to 1000 °C, and it displays the transducer reading directly in temperature unit. The digital indicator has been factory configured to operate with Au/Pt thermocouples.

2.2 Procedures

The interlaboratory program protocol stated that each laboratory should execute the artifact calibration as per the respective laboratory calibration procedure. In addition, the calibration results and any computation should observe the laboratory methodology previously assessed and approved by the accreditation body.

The artifact calibration method employed within the interlaboratorial program was based on the comparison with standard temperature sensors (S- or R-type thermocouples, SPRT, PRT) within the calibration range specified. The comparisons of the artifact with the laboratory's measuring standards were carried out on bench furnaces, portable furnaces, oil bath, salt bath, as well as fluidized bed. It is worth mentioning that no heat treatment for Au/Pt thermocouple stabilization (i.e., annealing) was performed when running the interlaboratory comparison.

All temperature transducers and measurement equipment (voltmeter, bridges) used for the comparisons were traceable to the International System of Units (SI). It is important to notice that during the calibration process, the thermocouple was immersed at different depths (from 140 mm to 300 mm) in the furnaces or baths, according to the equipment used by each laboratory.

2.2.1 Temperature calibration

For the purpose of this interlaboratory comparison, the artifact should be calibrated at the following temperature points: 0 °C, 230 °C, 420 °C, 660 °C, and 960 °C; with maximum temperature deviation from those points inferior to 3 °C. The laboratories should pay particular attention to the upper temperature limit of the Au/Pt thermocouple, in order to not cause damage to the instrument.

2.2.2 Uncertainties

The most significant uncertainty components and their estimates to the digital thermometer calibration are shown in Tables 1, 2 and 3. In the uncertainty budget the main factors come from the reference transducer and the furnace / bath gradient. The uncertainties stated on the calibration report of each laboratory, however, are fully consistent with the best measurement capability, within the scope of accreditation of the laboratory.

3 RESULTS AND DISCUSSION

In this intercomparison the artifact was calibrated eight (8) times. Table 4 shows the results of the first artifact calibration round carried out by each laboratory. In Table 5 the results of the second calibration round is presented. The results of the first and second calibration in the Brazilian NMI, performed in January 2008 and June 2011, are shown in Tables 6 and 7.

Table 1. Summary of the standard uncertainties of the digital thermometer calibration - Laboratory A

Uncertainty Component	Temperature (°C)				
	0	230	420	660	960
Standard sensor	0.003	0.002	0.003	0.35	0.35
Standard sensor drift	-	0.006	0.009	0.14	0.15
Standard Measuring Instrument	-	0.004	0.005	0.091	0.095
Standard Measuring Instrument drift	-	0.007	0.006	0.075	0.075
Repeatability of the measurements (standard)	-	0.001	0.003	0.005	0.007
Repeatability of the measurements (digital thermometer)	0.01	0.006	0.012	0.013	0.005
Ice bath	0.003	0.003	0.003	0.003	0.003
Furnace / bath gradient	0.003	0.01	0.011	0.27	0.32

Table 2. Summary of the standard uncertainties of the digital thermometer calibration - Laboratory B

Uncertainty Component	Temperature (°C)				
	0	230	420	660	960
Standard sensor	0.003	0.014	0.035	0.2	0.2
Standard sensor drift	-	0.002	0.021	0.07	0.1
Standard Measuring Instrument	-	0.002	0.002	0.004	0.004
Standard Measuring Instrument drift	-	0.0001	0.0001	0.017	0.016
Repeatability of the measurements (standard)	-	0.008	0.003	0.004	0.018
Repeatability of the measurements (digital thermometer)	0.01	0.014	0.0025	0.0025	0.015
Ice bath	0.003	0.003	0.003	0.003	0.003
Furnace / bath gradient	0	0.04	0.036	0.12	0.16

Table 3. Summary of the standard uncertainties of the digital thermometer calibration - Laboratory C

Uncertainty Component	Temperature (°C)				
	0	230	420	660	960
Standard sensor	-	0.016	0.016	0.15	0.15
Standard sensor drift	-	0.006	0.006	0.12	0.12
Standard Measuring Instrument	-	-	-	-	-
Standard Measuring Instrument drift	-	-	-	-	-
Repeatability of the measurements (standard)	-	0.004	0.013	0.01	0.03
Repeatability of the measurements (digital thermometer)	0.015	0.006	0.015	0.009	0.03
Ice bath	0.006	0.003	0.003	0.002	0.002
Furnace / bath gradient	-	0.062	0.073	0.14	0.19

Table 4. Calibration results of the artifact (first round) - Expanded Uncertainties (k=2)

	Laboratory A (29/01/2010)		Laboratory B (11/02/2010)		Laboratory C (26/02/2010)	
Temperature (ºC)	Correction (°C)	Uncertainty (°C)	Correction (°C)	Uncertainty (°C)	Correction (°C)	Uncertainty (°C)
0	0.02	0.10	-0.08	0.10	0.00	0.20
230	0.04	0.10	-0.04	0.20	0.06	0.15
420	-0.06	0.10	-0.17	0.50	-0.09	0.17
660	-0.48	1.06	0.86	0.90	0.06	0.60
960	-0.39	1.06	0.12	0.90	-0.23	0.75

Table 5. Calibration results of the artifact (second round) - Expanded Uncertainties (k=2)

	Laboratory A (12/03/2010)		Laboratory B (31/03/2010)		Laboratory C (05/08/2010)	
Temperature (ºC)	Correction (°C)	Uncertainty (°C)	Correction (°C)	Uncertainty (°C)	Correction (°C)	Uncertainty (°C)
0	0.02	0.10	0.04	0.11	0.01	0.20
230	-0.03	0.10	-0.02	0.20	-0.04	0.15
420	-0.13	0.10	-0.17	0.50	-0.17	0.17
660	0.04	0.79	0.23	0.90	-0.21	0.60
960	-0.24	0.82	0.40	0.90	-0.28	0.75

Table 6. Calibration results of the artifact (NMI first calibration) - Expanded Uncertainties (k=2)

	NMI (22/01/2008)	
Temperature (ºC)	Correction (°C)	Uncertainty (°C)
0	0.05	0.14
230	0.04	0.076
420	-0.02	0.076
660	0.00	0.050
960	0.01	0.050

Table 7. Calibration results of the artifact (NMI second calibration) - Expanded Uncertainties (k=2)

	NMI (07/06/2011)	
Temperature (ºC)	Correction (°C)	Uncertainty (°C)
0	-0.1	0.19
230	-0.05	0.09
420	-0.07	0.07
660	-0.02	0.05
960	-0.01	0.05

The results of the interlaboratory comparison were evaluated using the normalized error (En-value) [6]. The En-value describes the difference between the result obtained by the laboratory and the reference value compared to the stated uncertainties. If En is less or equal to 1, there is good agreement between the two results, and if En is greater than 1, then the results are not equivalent.

Since for this interlaboratory program no reference laboratory has been previously defined, one has decided to directly compare the results from one participant to another. One has also compared each laboratory calibration with the results of the artifact calibrations at Brazilian NMI.

Table 8 and 9 show the normalized error obtained from the crossed comparison of laboratories A, B and C. Table 10 and 11 present the En-values of each laboratory against the first and second Brazilian NMI calibration.

Table 8. Normalized error - En for Labs A, B, C - first round

Temperature (ºC)	Lab A x B En	Lab A x C En	Lab B x C En
0	0.71	0.09	0.36
230	0.36	0.11	0.40
420	0.22	0.15	0.15
660	0.96	0.44	0.74
960	0.37	0.12	0.30

Table 9. Normalized error - En for Labs A, B, C - second round

Temperature (ºC)	Lab A x B En	Lab A x C En	Lab B x C En
0	0.13	0.04	0.13
230	0.04	0.06	0.08
420	0.08	0.20	0.00
660	0.16	0.25	0.41
960	0.53	0.04	0.58

Table 10. Normalized error - En for Labs A, B, C x NMI 1st calibration

Temperature (ºC)	A x NMI En	B x NMI En	C x NMI En
0	0.17	0.76	0.20
230	0.00	0.37	0.12
420	0.32	0.30	0.38
660	0.45	0.95	0.10
960	0.38	0.12	0.32

Table 11. Normalized error - En for Labs A, B, C x NMI 2nd calibration

Temperature (ºC)	A x NMI En	B x NMI En	C x NMI En
0	0.56	0.09	0.36
230	0.67	0.05	0.63
420	0.08	0.20	0.11
660	0.43	0.98	0.13
960	0.36	0.14	0.29

From the evaluation of the normalized errors one could observe an En-value agreement in all comparisons. It is worth mentioning that the Au/Pt thermocouple was submitted to different calibration conditions, either in terms of furnace type or immersion depth.

As the normalized error might give an erroneous picture of the situation, depending on the stated uncertainty, the results were also analyzed using other two different methods: Youden diagrams and whiskers chart.

The Youden diagrams [7] are a graphic tool that allows the evaluation of the measurement results of comparisons between laboratories when each laboratory has carried measurements with two or more similar devices. The horizontal axis of a Youden plot represents the results of the first round and the vertical axis represents the results of the second round, at a given temperature.

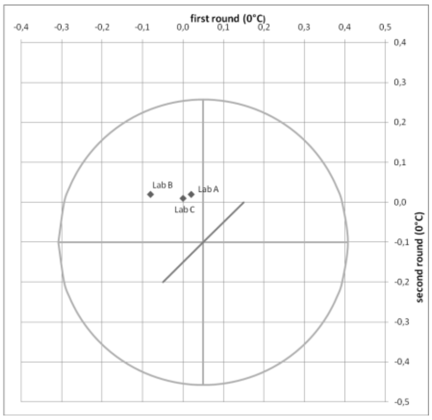

Fig. 1. Corrections at 0 °C in K

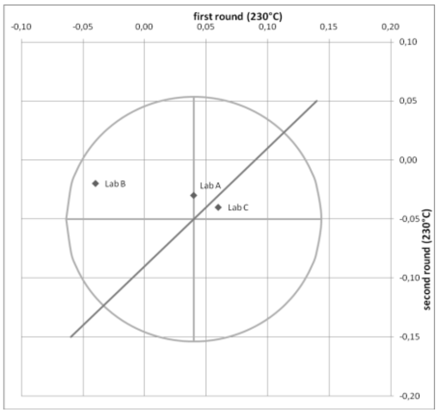

Fig. 2. Corrections at 230 °C in K

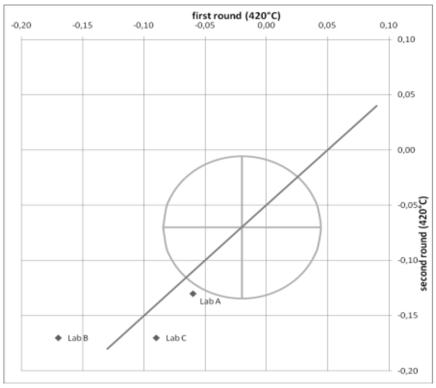

Fig. 3. Corrections at 420 °C in K

One considers a laboratory with a suitable quality system when the results reach similar deviation to the reference laboratory (Brazilian NMI) with the same artifact in two different calibrations. The distance between the experimental points (correction) and the straight line in the diagram represent the reproducibility and the quality system: the closer to the diagonal line, the better the reproducibility. Fig. 1-5 present the Youden diagrams for each temperature that the artifact was calibrated.

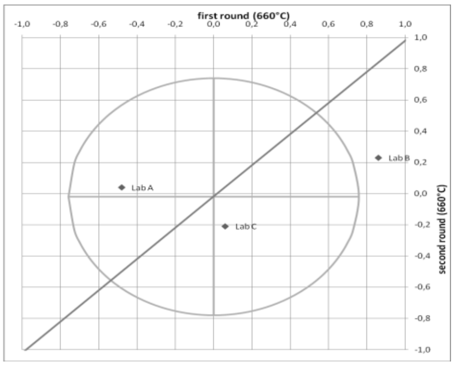

Fig. 4. Corrections at 660 °C in K

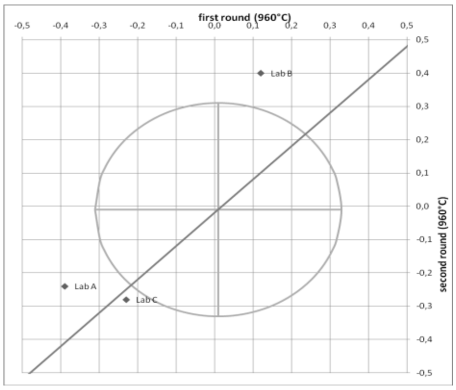

Fig. 5. Corrections at 960 °C in K

Figure 1 shows the results at 0 °C. One notices that all laboratories could reproduce the results within 0.1 K. Labs A and C exhibited higher reproducibility than Lab B, which displayed larger deviation from the NMI calibration (circle centre) for both rounds. Similar analysis can be made for the other calibration points, with particular interpretations. The Youden plot, however, does not take into account the stated uncertainty for each calibration point (and round).

The whiskers chart in Fig. 6 display the correction values determined by each calibration laboratory participant in both rounds, including Brazilian NMI. Since the Brazilian NMI employs fixed-point cells for all calibration points, the uncertainty interval remains quasi-constant over the entire range (see NMI1 and NMI2 in the graph). For the other three laboratories, which use liquid baths, temperature block calibrators, vertical and horizontal furnaces and fluidized bed bath to reach the target temperature, the uncertainties are considerably larger for the entire range, and in particular for the two higher temperature points.

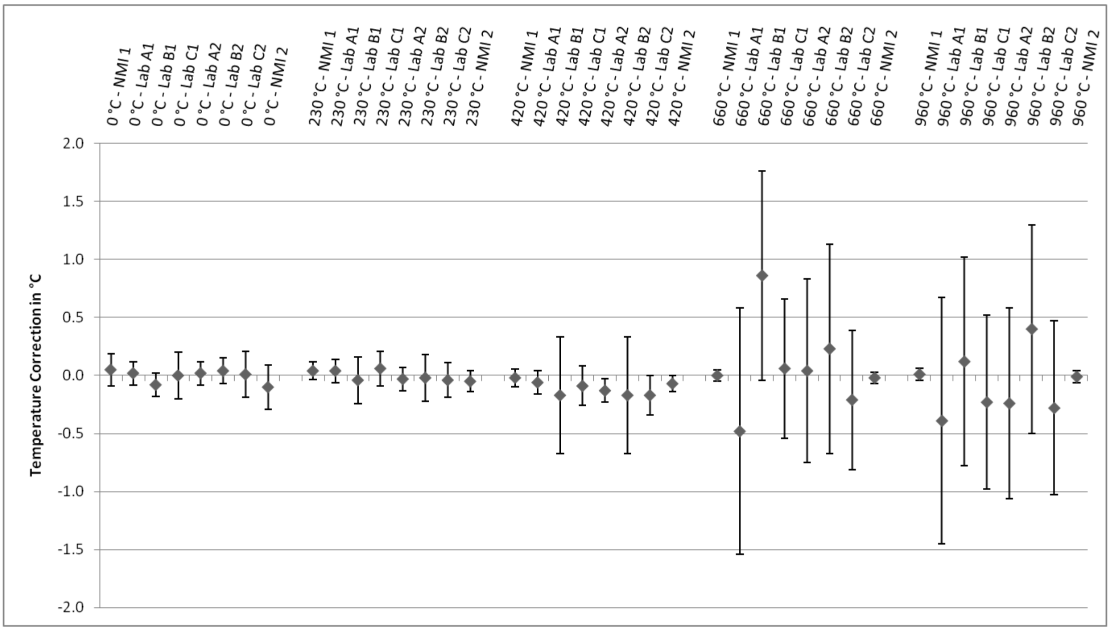

Fig. 6. Interlaboratory deviation chart displaying all calibration results for each temperature point of each laboratory

4 CONCLUSION

All participants of this interlaboratory program agreed that the chief objective of the study was fully accomplished. That means not only the activities execution but also the exchange of experiences provided by the interaction among professionals of laboratories accredited by the Brazilian Calibration Network and by the use of a Au/Pt thermocouple.

The study results agreed when evaluating the normalized error metric and were considered quite reasonable when considering the differences in laboratories' procedures. However, for higher temperatures, particularly 660 °C, the En-value approached the unitary value. For this reason it has been accepted by the participants to undertake a profound analysis regarding the furnaces used to calibrate the Au/Pt thermocouple.

Both calibrations performed by Laboratories agreed with each other, as shown in tables in this paper. It is important to emphasize that both NMI calibrations were compatible with each other. From this one could check the temporal stability of the artifact.

AKNOWLEDGMENTS

We would like to thank Dr. Renato Teixeira and his group (Diter/Inmetro-NMI), for the great contribution to this work, performing the artifact calibration.

We would also like to thank Presys, Ecil and IPT, particularly the technicians of the laboratories, whose support and participation were decisive for the development of this work.

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