

## **BILATERAL INTERCOMPARISON BETWEEN ACCREDITED LABORATORIES BY RBC (BRAZILIAN CALIBRATION NET)**

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**Abstract:** The purpose of this paper is to present the results of a bilateral interlaboratory comparison (ILC)[1] between the Mechanical Metrology Laboratory - IPT and Metrology Laboratory - Ecil. Both laboratories are accredited by Brazilian Calibration Net (RBC). In this program eight temperature artifacts have been calibrated in the range of -40°C to 1100°C. The artifacts are 02 thermocouples, 02 platinum resistance thermometers and 04 liquid in glass thermometers.

**Keywords:** intercomparison, calibration, temperature, metrology.

### **1. INTRODUCTION**

According to some standards and publications like ISO/IEC 17025 [2], EA-2/10 and DOC-CGCRE-005 [3], a laboratory has to control the quality of its results, in order to assure measurements and calibrations undertaken. There are some ways to ensure quality of measurements :

- certified reference materials (CRM)
- replicate test/calibration with different methods [4]
- retesting of retained items
- participation in interlaboratory comparisons or proficiency tests

It is important to control each measurement service provided by the laboratory, applying one of the methods listed above or another one that was appropriately validated and approved by a laboratory accreditation body. It has also to be considered the type and volume of work done by the laboratory. Following this rules, a calibration laboratory can provide evidence of its competence in measurement to its clients.

### **2. PURPOSE**

With the purpose to meet the standards requirements, exchange knowledge between participants, as well as to demonstrate competence in calibrations / measurements, the LMM-IPT and LM-Ecil decided to establish and implement a bilateral interlaboratory comparison program. These laboratories, accredited by Brazilian Calibration Net - RBC, have similar best uncertainty. In this paper, the laboratories had been identified by *Lab1* and *Lab 2*.

Of common agreement, eight artifacts were selected to be calibrated by the laboratories.

Thus, four liquid in glass thermometers-LGT, total immersion, 0.1°C resolution, were available by LMM-IPT. These thermometers have the following temperature range: -20°C to 20°C; 0°C to 80°C; 0°C to 200°C and 200°C to 250°C. Also, LM-Ecil put the following artifacts available for this program: two thermocouples-TC (a type R – ceramic insulation - and a type T- mineral insulation, in the temperature range of 0°C to 1100°C and 0°C to 350°C, respectively) and two platinum resistance thermometers-PRT ( type Pt 100 Ohms, metallic sheath, in the temperature range of 0°C to 420°C and 0°C to -40°C ).

The type of artifact and the temperature range were defined in order to cover great part of the services offered by the laboratories, according to its accreditation scope by RBC. The artifacts selected by the laboratories have several calibrations, performed by the laboratory itself or by another recognized laboratory.

The program started in August / 2005 and finished in April / 2006.

### **3. METHODS**

The first predetermined condition was that each laboratory must perform the artifact calibration according to their current calibration procedures. Also, the calibration results and calculations were developed according to each laboratory methodology, approved by the accreditation body.

The electrical standards (measuring instruments)[5] and temperature standards ( SPRT, PRT, Noble Thermocouples) used in the calibrations, are traceable to INMETRO (NMI)[6], other accredited laboratories by RBC or recognized organizations by RBC.

The interlaboratory comparison program established that an initial calibration (before the artifact has gone to the other laboratory) and final calibration (in the return of the artifact to the first laboratory) have to be performed by each one of the artifact's laboratory. This procedure had the objective to check any drift ( short term stability ) of the artifact, during this program. Great care were taken with handling and transporting the artifacts. The artifacts were packed appropriately and the transportation were done by the laboratory technicians.

A report was elaborated for each artifact calibrated and the results were analysed for both laboratories. The analysis of the results was done applying the calculation method of normalized error ( $En$ ). This method is widely employed in interlaboratory comparison programs and the purpose is to verify the laboratory quality measurement, related to the uncertainty [3].  $En$  values less than the unity indicate that the result is acceptable. In this case the error is covered by the measuring uncertainty declared.

$$E_N = \frac{|V_{lab1} - V_{lab2}|}{\sqrt{(U_{lab1})^2 + (U_{lab2})^2}} \quad (1)$$

$V_{lab1}$  – laboratory 1 value;

$V_{lab2}$  – laboratory 2 value;

$U_{lab1}$  – laboratory 1 uncertainty;

$U_{lab2}$  – laboratory 2 uncertainty .

The calibration points were previously defined, as shown in table 1.

**Table 1. Artifacts and calibration points.**

Artifact	Range (°C)	Calibration points (°C)
LGT-1	-20 to 20	-20; 0; 20
LGT-2	-0.5 to 0.5; 50 to 80	0; 50; 65; 80
LGT-3	200 to 250	200; 225; 250
LGT-4	-0.5 to 0.5; 150 to 200	0; 150; 175; 200
TC-R	0 to 1100	200; 400; 600; 800; 1000; 1100
TC-T	0 to 350	50; 150; 250; 350
PRT-1	0 to 420	0; 100; 200; 300; 400
PRT-2	-40 to 0	0; -10; -20; -30; -40

#### 4. RESULTS AND DISCUSSION

The calibration results ( correction, error, uncertainty ) and the normalized error,  $En$ , are presented in the tables below. Normalized error  $En$  was calculated according to equation 1. Temperature values in this paper follows International Temperature Scale-1990 (ITS-90). For thermocouples and platinum resistance thermometers the column *Error*, presented in the following tables, are related to the reference standards tables.

Table 2 shows the results for the artifact liquid in glass thermometer, LGT-1.

**Table 2. LGT-1 artifact results.**

Temperature (°C)	Lab 1		Lab 2	En	
	Correction / Uncertainty (°C) (1)*	(2)*	Correction / Uncertainty (°C)	(1)**	(2)**
-20	(-0,03 ±0,05)	(-0,04 ±0,04)	(-0,05 ±0,05)	0,28	0,16
0	(0,05 ±0,04)	(0,03 ±0,04)	(0,05 ±0,05)	0,00	0,31
20	(0,02 ±0,05)	(0,03 ±0,04)	(0,03 ±0,05)	0,14	0,00

\* (1) , (2) : initial and final calibration by the laboratory.

\*\* (1) , (2) : initial and final En calculation.

Table 3 shows the results for the artifact liquid in glass thermometer, LGT-2.

**Table 3. LGT-2 artifact results.**

Temperature (°C)	Lab 1		Lab 2	En	
	Correction / Uncertainty (°C) (1)*	(2)*	Correction / Uncertainty (°C)	(1)**	(2)**
0	(0,00 ±0,04)	(0,00 ±0,04)	(0,02 ±0,05)	0,31	0,31
50	(-0,05 ±0,05)	(-0,03 ±0,04)	(-0,03 ±0,05)	0,28	0,00
65	(-0,06 ±0,05)	(-0,06 ±0,04)	(-0,03 ±0,05)	0,42	0,47
80	(-0,04 ±0,05)	(-0,06 ±0,04)	(0,06 ±0,05)	1,41	1,87

\* (1) , (2) : initial and final calibration by the laboratory.

\*\* (1) , (2) : initial and final En calculation.

Table 4 shows the results for the artifact liquid in glass thermometer, LGT-3.

**Table 4. LGT-3 artifact results.**

Temperature (°C)	Lab 1		Lab 2	En	
	Correction / Uncertainty (°C) (1)*	(2)*	Correction / Uncertainty (°C)	(1)**	(2)**
200	(0,13 ±0,06)	(0,24 ±0,06)	(0,15 ±0,07)	0,22	0,98
225	(-0,15 ±0,06)	(-0,16 ±0,06)	(-0,12 ±0,07)	0,33	0,43
250	(-0,17 ±0,06)	(-0,13 ±0,06)	(-0,14 ±0,10)	0,26	0,09

\* (1) , (2) : initial and final calibration by the laboratory.

\*\* (1) , (2) : initial and final En calculation.

Table 5 shows the results for the artifact liquid in glass thermometer, LGT-4.

**Table 5. LGT-4 artifact results.**

Temperature (°C)	Lab 1		Lab 2	En	
	Correction / Uncertainty (°C) (1)*	(2)*	Correction / Uncertainty (°C)	(1)**	(2)**
0	(-0,01 ±0,06)	(-0,04 ±0,06)	(0,04 ±0,08)	0,50	0,80
150	(-0,51 ±0,07)	(-0,52 ±0,06)	(-0,46 ±0,09)	0,44	0,55
175	(-0,23 ±0,07)	(-0,19 ±0,06)	(-0,18 ±0,09)	0,44	0,09
200	(-0,19 ±0,07)	(-0,14 ±0,06)	(0,12 ±0,09)	0,61	0,18

\* (1) , (2) : initial and final calibration by the laboratory.

\*\* (1) , (2) : initial and final En calculation.

Table 6 shows the results for the artifact thermocouple TC-R. The calibration method performed by Lab 1 was measuring junctions welded [7].

**Table 6. TC-R artifact results.**

Temperature (°C)	Lab 2		Lab 1	En	
	Error / Uncertainty (°C) (1)*	(2)*	Error / Uncertainty (°C)	(1)**	(2)**
200	(-0,5 ± 0,3)	(-0,4 ± 0,3)	(-0,3 ± 0,9)	0,22	0,11
400	(-0,7 ± 0,3)	(-0,6 ± 0,3)	(-0,6 ± 0,9)	0,13	0,03
600	(-0,8 ± 0,9)	(-0,7 ± 0,9)	(-0,5 ± 0,9)	0,23	0,15
800	(0,1 ± 0,9)	(-0,6 ± 0,9)	(-0,4 ± 0,9)	0,37	0,18
1000	(0,3 ± 0,9)	(-0,4 ± 0,9)	(-0,1 ± 0,9)	0,31	0,24
1100	(0,6 ± 0,9)	(-0,2 ± 0,9)	(0,0 ± 0,9)	0,45	0,18

\* (1) , (2) : initial and final calibration by the laboratory.  
 \*\* (1) , (2) : initial and final En calculation.

Table 7 shows the results for the artifact thermocouple TC-R. The calibration method performed by Lab 1 was measuring junctions separated (each one immersed in a different hole in the furnace) [7].

**Table 7. TC-R artifact results.**

Temperature (°C)	Lab 2		Lab 1	En	
	Error / Uncertainty (°C) (1)*	(2)*	Error / Uncertainty (°C)	(1)**	(2)**
200	(-0,5 ± 0,3)	(-0,4 ± 0,3)	(-0,2 ± 0,9)	0,29	0,18
400	(-0,7 ± 0,3)	(-0,6 ± 0,3)	(-0,6 ± 0,9)	0,14	0,03
600	(-0,8 ± 0,9)	(-0,7 ± 0,9)	(-0,8 ± 0,9)	0,03	0,11
800	(0,1 ± 0,9)	(-0,6 ± 0,9)	(-0,7 ± 0,9)	0,61	0,06
1000	(0,3 ± 0,9)	(-0,4 ± 0,9)	(-0,4 ± 0,9)	0,56	0,01
1100	(0,6 ± 0,9)	(-0,2 ± 0,9)	(-0,3 ± 0,9)	0,73	0,10

\* (1) , (2) : initial and final calibration by the laboratory.  
 \*\* (1) , (2) : initial and final En calculation.

Table 8 shows the results for the artifact thermocouple TC-T.

**Table 8. TC-T artifact results.**

Temperature (°C)	Lab 2		Lab 1	En	
	Error / Uncertainty (°C) (1)*	(2)*	Error / Uncertainty (°C)	(1)**	(2)**
50	(0,1 ± 0,2)	(0,1 ± 0,2)	(0,1 ± 0,5)	0,00	0,00
150	(-0,1 ± 0,3)	(0,2 ± 0,3)	(0,1 ± 0,5)	0,34	0,17
250	(0,1 ± 0,3)	(-0,1 ± 0,3)	(-0,3 ± 0,5)	0,69	0,34
350	(-0,2 ± 0,4)	(-0,7 ± 0,4)	(-0,8 ± 0,5)	0,94	0,16

\* (1) , (2) : initial and final calibration by the laboratory.  
 \*\* (1) , (2) : initial and final En calculation.

Table 9 shows the results for the artifact platinum resistance thermometer PRT-1.

**Table 9. PRT-1 artifact results.**

Temperature (°C)	Lab 2		Lab 1	En	
	Error / Uncertainty (°C) (1)*	(2)*	Error / Uncertainty (°C)	(1)**	(2)**
0	(0,05 ± 0,01)	(0,04 ± 0,01)	(0,03 ± 0,02)	0,89	0,45
100	(0,08 ± 0,03)	(0,07 ± 0,03)	(0,08 ± 0,04)	0,00	0,20
200	(0,11 ± 0,03)	(0,10 ± 0,03)	(0,08 ± 0,05)	0,51	0,34
300	(0,13 ± 0,06)	(0,13 ± 0,06)	(0,12 ± 0,05)	0,13	0,13
400	(0,13 ± 0,06)	(0,17 ± 0,06)	(0,19 ± 0,05)	0,77	0,26

\* (1) , (2) : initial and final calibration by the laboratory.  
 \*\* (1) , (2) : initial and final En calculation.

Table 10 shows the results for the artifact platinum resistance thermometer PRT-2.

**Table 10. PRT-2 artifact results.**

Temperature (°C)	Lab 2		Lab 1	En	
	Error / Uncertainty (°C) (1)*	(2)*	Error / Uncertainty (°C)	(1)**	(2)**
-40	(-0,02 ± 0,04)	(-0,03 ± 0,04)	(-0,04 ± 0,04)	0,35	0,18
-30	(-0,01 ± 0,04)	(-0,02 ± 0,04)	(-0,03 ± 0,04)	0,35	0,18
-20	(0,01 ± 0,04)	(0,00 ± 0,04)	(-0,01 ± 0,04)	0,35	0,18
-10	(0,02 ± 0,04)	(0,01 ± 0,04)	(0,00 ± 0,04)	0,35	0,18
0	(0,03 ± 0,01)	(0,03 ± 0,01)	(0,01 ± 0,02)	0,89	0,89

\* (1) , (2) : initial and final calibration by the laboratory.  
 \*\* (1) , (2) : initial and final En calculation.

Analyzing the final data shown in tables 2 to 10, we observe that the results of the measurements are compatible for the majority of the calibrations. Only in the LGT-2 artifact calibration, at the point of 80°C, we found incompatible result. It was established that the laboratories have to carry out a critical analysis, in order to find out possible causes of the problem, in that artifact calibration. This investigation involves to search problems such as: error in the calculations, incorrect measuring procedure, standards problems, calibration apparatus, influence factors, ambient conditions, transcription of data. However, if we will not find any problem in this initial inquiry, both laboratories will perform a new calibration of the LGT-2 artifact. The new results will be compared following the same procedure described in this paper. It is important to emphasize that for TC-R artifact, two calibrations were performed by Lab.1, using different methods (welded and separated measuring junctions). As shown in tables 6 and 7, we can evidence the compatibility of the results for both calibrations.

## 5. CONCLUSION

This bilateral interlaboratory comparison program was one excellent opportunity to evaluate the quality of measurements and calibrations, performed by the laboratories participants.

We consider that this interlaboratory program were very successful as shown in the result tables. Eight artifacts were calibrated (at least 3 times) and the total calibration points performed were 102. The normalized error (*En*) calculated for seven artifacts showed total calibration results compatibility (*En* less than 1). However, for LGT-2 artifact we verify that the *En* calculated at 80°C showed results not compatible. The *En* result in this point was above 1. The laboratories started investigation to find the possible problem. Unfortunately, we did not have enough time to present, in this article, the investigation conclusion. Certainly, we will have the opportunity to present final investigation in the Congress lectures.

This program also provided opportunity to establish an exchange of experiences between the laboratories, improving the knowledge of the technicians and quality of services.

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## REFERENCES

- [1] ABNT NBR ISO/IEC 17025:2005 – “General Requirements for the Competence of Testing and Calibration Laboratories”. ABNT,2005.
- [2] EA-2/10 – “EA Policy for Participation in National and International Proficiency Testing Activities-rev.00”, EA European co-operation for Accreditation, 6p, August 2001.
- [3] DOC-CGCRE-005 – “Orientações para a organização de comparações interlaboratoriais pelas comissões técnicas da DICLA-rev.00”, INMETRO, 11p, setembro 2002.
- [4] M. A. P. Castanho, M. F. F. Pereira and W. Link, “Validação de Calibração de Termopares Pelo Método da Ponte (Pontos do Ouro e do Paládio)”, Proceedings of the 3<sup>th</sup> Brazilian Congress of Metrology-SBM, Recife, Brazil, September 2003.
- [5] VIM–Vocabulário Internacional de Termos Fundamentais e Gerais de Metrologia – INMETRO – Instituto Nacional de Metrologia, Normalização e Qualidade Industrial.
- [6] ILAC-G2:1994 – “Traceability of Measurements”, ILAC, 53p, 1996.
- [7] Norma técnica *NBR 13522:1995 – Termopar – Calibração por comparação com termopar de referência*. ABNT, 1995. 13p.