

The Role of LATU as National Metrology Institute of Uruguay and its Responsibilities

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Abstract. Laboratorio Tecnológico del Uruguay (LATU) is the National Metrology Institute of Uruguay and has the obligation to maintain the national standards stated by National Law 15298. At present LATU is acting as a secondary laboratory as well as a primary laboratory. LATU was ISO 17025:2005 DKD (Deutscher Kalibrierdienst) accredited from 2001 up to 2007. By that time LATU decided to support its Capabilities of Measurement and Calibration (CMCs) at CIPM-MRA (Mutual Recognition Arrangement between national metrology institutes (NMIs)) by peer assessment. A Peer Review has been done in 2008 in order to get the QSTF (Sistema Interamericano de Metrología, Quality System Task Force) approval.

“New “CMCs for Industrial Thermometers have been approved by the JCRB on September 2010. CMCs claimed for Standard Resistance Platinum Thermometers (SPRTs) calibration at fixed points have not been approved yet because there were some requirements of traceability of employed cells that were not fulfilled but will be solved properly.

The declared CMCs have been chosen by LATU in order to cover the increasing calibration services required by the industry and the secondary calibration laboratories. To demonstrate its technical competence an support its declared “CMCs” LATU has also participated at bilateral and regional comparisons.

In recent years LATU, the National Accreditation Body (OUA), the Standards Institute, the National Institute of Quality and Compliance Bodies have become Members of a new Institution to strengthen the Quality Infrastructure of the country (SUNAMEC). As part of this new activities, LATU is giving training courses to the secondary laboratories performing calibrations in temperature that want to get accredited by the National Accreditation Body and to act as Technical Evaluators or Auditors when required by OUA.

It is expected, that in the future and in the frame of new accredited and recognized temperature calibration laboratories, LATU could strengthen its activities in maintaining its own national standards, developing new calibration services and performing comparisons as pilot laboratory for Uruguay and also regionally. The role of secondary laboratory could be diminished and therefore the activities as a reference laboratory in investigation would be benefited. This paper describes all the activities carried out at LATU in Temperature in the last years to reach the goals stated and the coming ones that have to be done to help developing main objectives as a country in this field.

Keywords: Calibration services, Comparisons, CMCs, Traceability.

INTRODUCTION

LATU (Laboratorio Tecnológico del Uruguay) is the National Metrology Institute of Uruguay. It was designated as the responsible of the maintenance of National Standards by National Law of Metrology N° 15298.

As part of being also a calibration laboratory it offers calibration services for different kind of thermometers.

The users of calibration services in thermometry are mostly the private industry, secondary calibration laboratories, public institutions and the other laboratories from LATU. . The requests for calibration services are constantly growing, both in amount as in quality and complexity and cause additional problems.

CALIBRATION SERVICES OFFERED

During the last nine years Uruguay grew at unprecedented economic rates. This was reflected in the number of calibrations performed in temperature, mostly for industry and secondary laboratories. The most important industry clients of LATU are:

- i) Food & beverage industry
- ii) Pharmaceutical industry
- iii) Metal mechanical industry
- iv) Wood and paper mills industry

Since Uruguay is a tiny country of 3 251 526 inhabitants (2012), all of those industrial branches need to export their products to other markets. Those markets are strongly regulated on product quality and safety. This implied a need for improved process control using calibrated instruments.

Number of calibrations requested to LATU

As an example, the number of calibration orders of 2003, 2009, 2010 and 2011 are shown in Table 1.

As it can be observed in the table the amount of calibrations performed in this laboratory has been multiplied by seven times during this period. The same growth has occurred in the global number of orders received at LATU.

TABLE 1. Number of Calibration Orders

Year	Calibration Orders
2003	232
2009	1422
2010	1194
2011	1494

Not only has the amount of work grown in numbers but also in diversity and difficulty. At the start the majority of calibrations were liquid in glass thermometers, but in recent years there has been an increase in radiation, non contact thermometry and contact thermometry different than liquid in glass thermometry, such as industrial platinum resistance thermometers (PRTs) and thermocouples with an extended temperature range. This was a result of the need to use better temperature instruments with lower uncertainty values. The export position of the country and LATU-users require LATU to obtain international recognition.

CMCs SELF DECLARATION AT CIPM MRA LEVEL

LATU was 17025:2005 ISO Deutscher Kalibrierdienst (DKD) accredited from 2001 to 2007 in mass, balances and temperature. In 2007 and since LATU is the NMI of Uruguay it was decided to drop the accreditation and declare in the CIPM MRA, choosing the path of peer assessment to support its quality system.. The CMCs were chosen to cover the calibration services frequently performed and that were supported by regional or bilateral comparisons with NMIs of lower CMC values declared.

LATU had published CMCs values at CIPM MRA that were not in accordance with the uncertainty values that were reached at that time. These new values had to be supported by comparisons and a corresponding traceability chain.

Before being Peer Reviewed by Sistema Interamericano de Metrología (SIM), LATU performed a series of Bilateral Comparisons in the

Frame of DKD- Surveillance visits to demonstrate its technical competence. Comparisons were performed with Physikalisch Technische Bundesantalt (PTB), PTB.

EVOLUTION OF LATU FIXED POINT REALIZATIONS

First Bilateral Comparison PTB-LATU

Parameters evaluated at all these bilateral comparisons were:

$$W_{FP} = \frac{R_{FP}}{R_{TPW}} \quad (1)$$

where:

R_{FP} : Resistance value of the calibration object (SPRT of PTB) at each fixed point (FP).

R_{TPW} : Resistance value of the calibration object (SPRT of PTB) at the triple point of water (TPW).

Since PTB gave the reference value it has been calculated in this case:

$$\Delta T_{LATU-PTB} \cong \frac{W_{LATU} - W_{PTB}}{\left(\frac{\partial W}{\partial T}\right)_{FP}} \quad (2)$$

where :

$\Delta W_{LATU-PTB}$: difference between W_{LATU} and W_{PTB} measured values at each fixed point divided by the first partial derivative of W with respect to temperature at each fixed point.

$\left(\frac{\partial W}{\partial T}\right)_{FP}$ = first derivative of the International

Temperature Scale of 1990 (ITS-90) polynomial reference function for W evaluated at each fixed point, K^{-1} .⁽¹⁾

Therefore :

$\Delta T_{LATU-PTB}$ is the difference of temperature between the measured values of PTB and LATU in mK.

The first calibration of a SPRT at the fixed points that were available at the first audit visit (TPW, Ga, Sn and Zn), gave the following results that are shown in Figure 1:

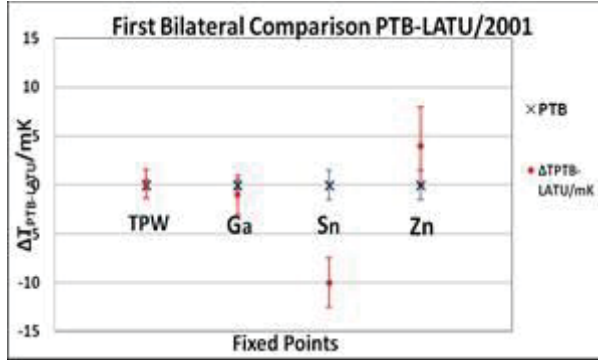


FIGURE 1. First Bilateral Comparison between PTB and LATU with PTB as Pilot Laboratory

The Table 2 shows the plotted data:

TABLE 2.

Fixed Point	$\Delta T_{PTB-LATU}/mK$	U_{LATU}/mK	U_{PTB}/mK	E_N
TPW	0.1	1.5	0.50	0.063
Ga	-1.0	2.0	0.50	0.49
Sn	-10.0	2.5	1.5	3.4
Zn	4.0	4.0	1.5	0.94

where :

$$E_N = \frac{|\Delta T_{PTB-LATU}|}{\sqrt{U_{PTB}^2 + U_{LATU}^2}} \quad (3)$$

It can be seen from Fig. 1 and Table.2 2 that realization at the Freezing Point of Sn was an outlier when evaluating the normalized error E_N using Equation(3).

Therefore, the Accreditation Scope accepted to be declared at DKD in September 2001 was:

TABLE 3. Calibration of SPRT at Fixed Points DKD accepted in September 2001

Fixed Point	Uncertainty Value/mK
TPW	1.5
Ga	3.0
Sn	15
Zn	6.0

When self declaring CMCs those were the uncertainty values chosen and these are the ones that remained until now at BIPM CIPM.

As part of the evaluation of those results obtained, PTB offered a thermometry expert assessment at

LATU in June 2002 and that activity was successful when performing further SPRT comparisons.

It was established that LATU had the capability to calibrate their own SPRTs using as a reference one SPRT that was calibrated every two years at PTB to maintain the traceability of the secondary laboratory calibration services. There have been done realizations of Zn freezing point and Sn freezing point and an evaluation of the immersion curve at the oven employed by that time for both fixed point cells.

After that LATU has bought a mercury apparatus for introducing the realization of the mercury triple point, this has been done due it has been detected the need to calibrate SPRTs and PRTs of LATU below TPW.

Second Bilateral Comparison PTB-LATU

In the frame of another DKD Surveillance visit another Comparison had been done. The results are given in Table 4:

TABLE 4. Second Bilateral Comparison PTB-LATU

Fixed Point	$\Delta T_{PTB-LATU}/mK$	U_{LATU}/mK	U_{PTB}/mK	E_N
Hg	-3.0	5.0	0.26	0.60
TPW	0.1	1.5	0.5	0.063
Ga	-0.3	1.5	0.25	0.20
Sn	2.1	5.0	0.85	0.41
Zn	-3.2	5.0	1.3	0.62

As part of the results obtained in this comparison, DKD accepted the new CMCs values. Figure 2 shows the plotted values.

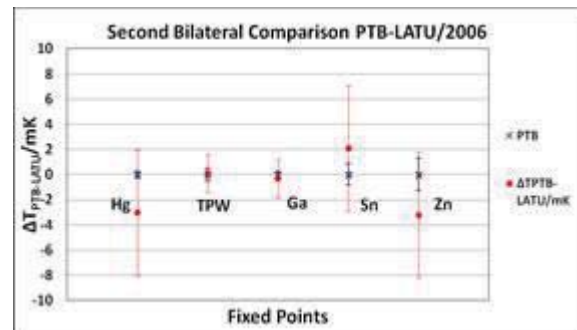


FIGURE 2. Second Bilateral Comparison between PTB and LATU with PTB as Pilot Laboratory.

Third Bilateral Comparison PTB-LATU

In June 2007, LATU decided not to continue with DKD Accreditation and to declare CMCs in the frame of CIPM-MRA and asked for a ISO 17025:2005 Peer Review in Temperature, Mass and Density.

LATU also requested for the withdrawal of CMCs from the BIPM Key Comparison Data Base (KCDB) for CMCs.

The Peer Review occurred in January 2008, with PTB again as Technical Peer Reviewer and another bilateral Comparison was performed between PTB and LATU. Table 5 and Figure 3 present the results.

LATU wanted to claim reduced CMCs values than the ones accepted by DKD at last audit visit but the Peer Reviewer recommended to maintain the same ones because only two of the fixed point cells had direct traceability to the International System of Units (SI) through an NMI with better CMCs values than LATU.

Table 5 presents the Comparison results.

TABLE 5. Third Bilateral Comparison PTB-LATU

Fixed Point	$\Delta T_{PTB-LATU/mK}$	$U_{LATU/mK}$	$U_{PTB/mK}$	E_N
Hg	-1.9	5.0	0.26	0.38
TPW	0.2	1.5	0.50	0.13
Ga	-0.1	1.5	0.25	0.066
Sn	-0.8	5.0	0.85	0.16
Zn	1.4	5.0	1.3	0.27

Figure 3. shows the plotted data.

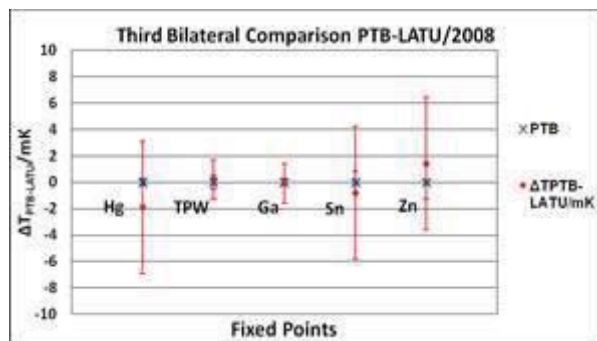


FIGURE 3. Third Bilateral Comparison between PTB and LATU with PTB as Pilot Laboratory.

LATU agreed with those terms and the results of this comparison have been presented at Temperatur 2009 Symposium, Berlin, Germany, ^{(2),(3)}.

In the Peer Review Report it was established that the last two comparisons performed with PTB could be used to support the technical competence for the calibration of SPRTs using the fixed point cells of LATU. But it was pointed out that the only two of the fixed point cells employed had direct traceability to SI Units through an NMI. Nevertheless, all CMCs were SIM approved but when they went to international Review the service categories Long stem SPRT calibration by Fixed Point Cells were not approved because of the non direct traceability of the cells. The other criteria for revision were fulfilled, but not complying with the latter one was determinant.

In order to solve this problem, it was proposed to perform an indirect comparison of the LATU cells with National Institute of Standards and Technology (NIST) using two SPRTs of NIST with the purpose to trace them to SI through NIST. Measurements have been ended at LATU but comparison hasn't been ended yet.

Declaring CMCs for calibration of long stem SPRTs is important because:

- i. Intrinsic maintenance of LATU standard SPRTs.
- ii. Offering the calibration service to secondary calibration laboratories of Uruguay with international recognition. This is a claim of the National Accreditation Body of Uruguay (OUA).
- iii. Participation in comparisons in order to demonstrate and maintain technical competence.

Comparisons supporting Industrial CMCs

LATU has participated also in the following SIM Pilot Comparisons:

SIM.T-S1 /SIM supplementary comparison 3.9:
Type K thermocouple wire over the range 100 °C to 1100 °C

This comparison has been published and approved ^{(4),(5)}, LATU has participated from 100 °C to 1000 °C. The results obtained have been a support when declaring CMCs for the calibration of base metal thermocouples, Figure 4 shows the results obtained for LATU:

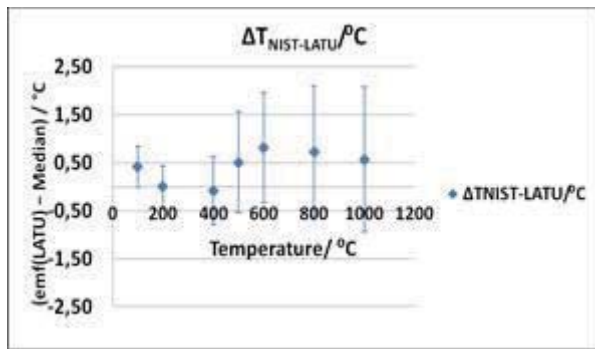


FIGURE 4. SIM.T-S1/SIM supplementary comparison, Results obtained at LATU.

The results of that comparison supported the self Declaration of CMCs calibration services of base metal thermocouples in the range from 0 °C to 1000 °C.

LATU has participated at SIM Type S Supplementary Comparison in the range from 100 °C to 1000 °C which is still active.

New Industrial CMCs were approved on November 22nd 2010, more at :

http://www.bipm.org/exalead_kcdb/exa_kcdb.jsp?c=+11942941663638602024&C=eJyLz2FizWOIL8tj8HZ2cYp3LChzUvJrHBmiM8vKMnMzytmMIOzglMTi5IzQAKJBQwGDPE5uSB2AZgsZAgtKk0vTaxkiM*ILHERzclhMDJgAACIPBzw&p=AppC&s=20

CMCs were declared taking into account the most relevant services in number and uncertainty values required for the secondary calibration laboratories of Uruguay. Other CMCs are of low level and the reason for this is that LATU is still acting as a secondary laboratory.

It is intention of LATU to leave those low level calibration to secondary accredited laboratories by the National Accreditation Body of Uruguay (OUA). At this time there are only two laboratories accredited by OUA.

RESEARCH WORK

Temperature Profiles of Fixed Point Cells

Peer Review in 2008 recommended performing activities of research especially in the characterization of fixed point cells.

TPW cells have been studied in relation with their stabilization time and with respect to hydrostatic pressure behavior. A paper⁽⁶⁾ has been published in which the results exhibited show that isolating better the cell shortened the stabilization time and adjusted

the immersion profiles more close to the ideal behavior of the hydrostatic pressure.

It was also studied the use of bushings.

Figure 5 shows the obtained results:

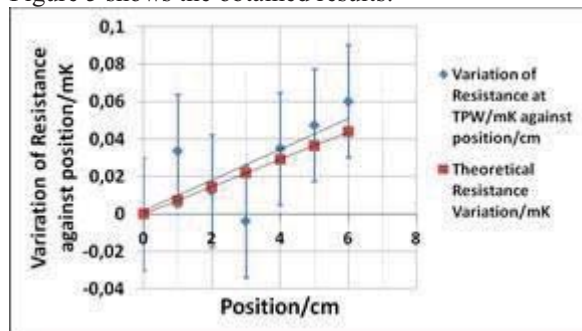


FIGURE 5. Variation of Resistance at TPW against position from bottom of cell expressed in mK.

The other fixed point cells have been studied with respect to hydrostatic pressure behavior to estimate uncertainty values for the realizations at the fixed points. have not been studied better profiles as it is needed in order to diminish the uncertainty values .

This is one of the investigation lines that has to be continued.

New Services

LATU is receiving every year more requirements from the clients in radiation thermometry and it has purchased a commercial blackbody source to calibrate radiation thermometers up to 500 °C. This equipment must be characterized before beginning with the new service. A new radiation thermometer which has to be calibrated at another NMI will be employed for that purpose.

CONCLUSIONS

LATU is carrying out all of the duties a NMI has to carry out and needs to delegate the tasks of the ordinary calibrations to secondary laboratories.

In the interim period of time between that there could not be more accredited laboratories doing low accuracy calibrations, LATU has to perform both the role of a secondary recognized laboratory and as a NMI. That is a reason for carrying out both activities.

Evolution of LATU has been satisfactory in carrying the activities of a NMI such as comparisons and has to continue in order to obtain national and international recognition.

It is important to get approval of the CMCs presented of long stem SPTs calibration at the fixed points of Zn, Sn ,Ga, TPW and Hg to

continue growing as a National Metrology Institute . It is important that secondary accredited laboratories can receive a service with international recognition without having to send their equipment to other countries with the additional disadvantage of the cost .

Obtaining traceability of LATU cells should be the last step in this stage.

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