An SPRT Intercomparison at Hg, TPW, Ga, Sn and Zn ITS-90 Fixed Points between PTB and LATU with PTB as Pilot Laboratory

O, Robatto, Departamento de Metrología Científica, Laboratorio Tecnológico del Uruguay(LATU), Av, Italia 6201 C,P 11500, Montevideo, Uruguay
U, Noatsch and **S, Rudtsch**, Physikalisch-Technische Bundesanstalt (PTB), Braunschweig und Berlin, Abbestrasse 2-12,10587 Berlin, Deutschland

Kurzfassung

Laboratorio Tecnológico del Uruguay, LATU, ist das nationale Metrologieinstitut von Uruguay und Unterzeichner des CIPM Mutual Recognition Arrangement(CIPM MRA). Die Kalibrier- und Messmöglichkeiten (CMCs) wurden im Juni 2004 anerkannt und in die BIPM-Key Comparison Data Base (www.bipm.org/KCDB) aufgenommen.
LATU war zuvor von 2001 bis Juni 2007 im Deutschen Kalibrierdienst (DKD, DKD-K-25601) für die Größen Masse/Waagen und Temperatur akkreditiert. Es wurde entschieden dem CIPM MRA beizutreten und die Erfüllung der CIPM Kriterien und der Anforderungen aus der ISO/IEC 17025im Rahmen eines vor-Ort Gutachterbesuches nachzuweisen. Die Begutach-tung im LATU wurde in Januar 2008 durchgeführt, als Teil dieses "Peer Review Prozesses" wurde ein bilateraler Vegleich zwischen der PTB und dem LATU an den Fixpunkten der ITS 90 vom Hg-Punkt bis zum Zn-Punkt durchgeführt. Das Pilot Labor war die PTB.

Abstract

Laboratorio Tecnológico del Uruguay, LATU, is the National Metrology Institute of Uruguay, signatory of the CIPM Mutual Recognition Arrangement (CIPM MRA), has Calibration and Measurement Capabilities (CMCs) in temperature that were first published in the BIPM Key Comparison Data Base (www.bipm.org/KCDB) in June 2004,
LATU was a DKD Laboratory accredited in Mass, Balances and Temperature from 2001, until June 2007, when it decided to adopt the CIPM criteria for proving compliance with ISO/IEC 17025 through a peer evaluation process.
The LATU on site peer evaluation was carried out in January 2008, as part of the peer review process. As part of the evaluation, a bilateral intercomparison was performed between PTB and LATU at the ITS-90 fixed points covering the range from Hg TP to Zn FP, with PTB acting as the Pilot Laboratory.
In this paper the results are presented and discussed in order to support the declaration of new Calibration and Measurement Capabilities (CMCs),

Introduction

In the frame of the Mutual Recognition Arrangement of the CIPM, interlaboratory comparisons are the main tool for demonstrating equivalence of measurement capabilities among National Metrology Institutes. These comparisons are usually organized as multilateral key comparisons, either by CIPM or by Regional Metrology Organizations (RMOs).
This paper describes an intercomparison between LATU (member of SIM) and PTB (member of EURAMET) in the range between -38.8344 °C to 419.527 °C based on an SPRT as scale carrier with PTB acting as Pilot Laboratory, The SPRT was calibrated in the following sequence: PTB-LATU-PTB,
The main objective for carrying out this comparison was to demonstrate the capability of LATU to calibrate SPRTs in the temperature range cited above by using its own fixed point cells to support the Calibration and Measurement Capabilities declared at the Key Comparison Database, Appendix C of the CIPM MRA.

Facilities

Both laboratories used an ac resistance ratio bridge type ASL F18 with AC/DC standard resistors (Tinsley) for resistance measurements.
The nominal purity of the fixed point materials at PTB and LATU is specified to equal or better than 99.9999 % (6N).

Table1. Details about LATUs fixed point realizations used for measurement

Fixed point	Manufacturer of cell	Standard of LATU	Material	Furnace/Bath
Zn	Isotech	Sealed cell	Johnson Matthey	YSI 1 zone furnace
Sn	Isotech	Sealed cell	Johnson Matthey	ISOTECH 1 zone furnace
Ga	Isotech	Sealed cell	Johnson Matthey	ISOTECH calibrator
TPW	CENAM	Borosilicate glass		Dewar bath filled with ice
Hg	Isotech	Triple point		ISOTECH

Table2. Details about PTB´s fixed point realizations used for measurement

Fixed point	Manufacturer of cell	Reference cell of PTB(*)	Material	Furnace/Bath
Zn	Home made	Open cell No, XXV	Preussag Pure Metals GmbH	ISOTECH Model ITL 17703 3 zone furnace
Sn	Home made	Open cell No, X	Koch Chemicals Ltd,	ISOTECH Model 17701 3 zone furnace
Ga	Isotech	Sealed cell No 213	Johnson Matthey	ISOTECH calibrator
TPW	Hart Scientific	Quartz glass No, 1043	V-smow effect +10 μK	ISOTECH water triple point bath
Hg	Isotech	Triple point No, M006		YSI mercury triple point standard

(*) Traceable to the national standard

Results

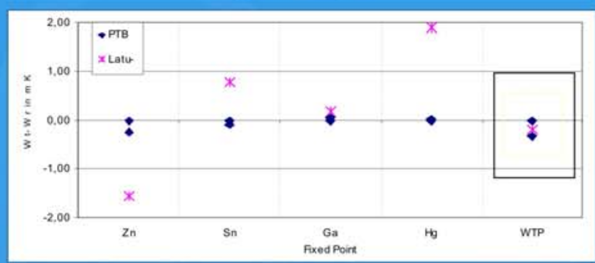
The comparison results for the PTB-Latu-PTB measurements obtained were the following ones:

Table 3: Comparison results $W_t = R_t / R_{TPW}$

Fixed Point	W_{T90}(PTB) 0 mA	W_{T90}(LATU) 0 mA	W_{T90}(PTB) 0 mA	T_{PTB}-T_{LATU} /mK
Dates	Jan 11 to Jan18, 2008	Jan 28 to Feb 2, 2008	Feb 7 to Feb 18, 2008	
Hg	0.84418626	0.84419396	0.84418634	-1.9 mK
Ga	1.11810344	1.11810407	1.11810367	-0.1 mK
Sn	1.89252229	1.89252512	1.89252197	-0.8 mK
Zn	2.56842888	2.56842346	2.56842799	1.4 mK

Data Results from Table 3, are plotted in Graph 1

Graph 1. Results of the comparison



Uncertainties

The uncertainty values from LATU are listed in Table 4, type A and type B contributions to the uncertainty budget for each fixed point are listed separately to give a better approach to the parameters considered in the estimation carried out.

Table 4. Measurement uncertainties of Latu (k=1) in mK

Fixed Point Material	Hg	H_2O	Ga	Sn	Zn
Purity	6N		6N	6N	6N
Immersion length / cm	17.9	26.7	25.7	18.3	18.4
Type B Uncertainty Contribution					
Isotopic Impurities	0.25	0.10	0.20	0.52	0.71
Hydrostatic Pressure Correction	0.03	0.03	0.01	0.02	0.02
Gas Pressure	0.01	0.15	0.01	0.70	1.70
Standard Resistor	0.50	0.50	0.50	1.40	1.40
Resistance Measuring Bridge	0.05	0.05	0.02	0.012	0.012
Propagation from WTP	0.17	0.00	0.22	0.38	0.51
Self heating	0.05	0.04	0.05	0.20	0.20
Heat flux immersion	0.02	0.01	0.01	1.0	1.0
Plateau behaviour	0.20	0.15	0.10	0.40	0.35
Total Type B	0.62	0.56	0.59	1.76	2.41
Type A Uncertainty Budget Contribution					
Total Type A Uncertainty (k = 1)	0.16	0.18	0.19	0.80	0.19
Total Expanded Uncertainty (k = 2)	1.99	1.17	1.25	3.87	4.84

Table 5. The new CMCs to be declared by LATU are:

Fixed Point	U_{LATU} (k=2) 2006 DKD-accepted
Hg	5.0 mK
TPW	1.5 mK
Ga	1.5 mK
Sn	5.0 mK
Zn	5.0 mK

These CMCs are the ones that were DKD accepted in the last DKD assessment in March 2006, and the Peer Reviewer agreed to maintain these capabilities and he recommended to characterize the fixed point cells that are employed in the calibration of SPRTs and to maintain the traceability chain of LATU to another NMI before reducing values of CMCs.

Table 6. The PTB CMCs published in the KCDB are:

Fixed Point	U_{PTB} (k=2)
Hg	0.26 mK
Ga	0.25 mK
Sn	0.85 mK
Zn	1.30 mK

Normalized error, E_n

Once the differences and associated uncertainties are determined there are some useful techniques for data analysis. There is the normalized error |En|, which is defined by equation

$$|E_n| = |(T_{LATU} - T_{PTB}) / \sqrt{(U_{LATU}^2 - U_{PTB}^2)}| \qquad (2)$$

It is expected an $|E_n|$ value lesser or equal to 1 if the measured difference is consistent with the declared uncertainties.
The normalized error En calculated from the column of table 3, 5 and 6 are listed in table 7.

Table 7. Normalized error $|E_n|$

Fixpunkt	$\|E_n\|$
Zn	0.27
Sn	0.16
Ga	0.07
Hg	0.38
TPW	0.14

All points fulfil with $|E_n| \leq 1$

Conclusion

The results obtained support LATUs published calibration measurement capabilities (CMCs) to be submitted for review and publication in the KCDB of the CIPM (MRA). However, and in agreement with the Peer Review Team, LATU has to characterize its fixed point cells to determine whether their behaviour complies with the ideal one respect to hydrostatic pressure effect and heat transfer to the environment .

There have been done some steps in this direction, by studying in detail one of the TPW cells successfully, by working on a better isolation from the environment and using bushings.

LATU has acquired one MSL Resistance Bridge Calibrator Unit (RBC) in order to calibrate its measuring bridge.

Literature

(1) H. Preston Thomas: The International Temperature Scale of 1990 (ITS-90), Metrologia,1991, 27, 3-10.
(2) Wöger, W. PTB-Mitteilungen 109, 24-27 (1999)

Kontakt: Ofelia Robatto, Laboratorio Tecnológico del Uruguay T: 00(598) 2 6013724/298 orobatto@latu.org.uy