INTERNATIONAL COMPARISON OF PLATINUM RESISTANCE THERMOMETERS BETWEEN CHILE AND ECUADOR

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Abstract. An International Bilateral Comparison of Platinum Resistance Thermometers was performed between the National Metrology Institutes of Chile (LCPNT) and the Ecuadorian Standardization Institute of Ecuador (INEN), both represented by their National Laboratories of Temperature. This comparison was carried out in the range from -39 °C up to 232 °C. The final results obtained by each laboratory showed to be equivalent ($E_N < 1$) in the measured temperature range.

Keywords: **Temperature, comparison, LCPNT, Chile, Ecuador, INEN, SIM.**

INTRODUCTION

This comparison was developed under the technical and quality cooperation project signed between the government of Ecuador and the European Union, executed between 2007 and 2008, in which the national temperature laboratory of INEN participated.

Two steps were agreed and the whole process was completed between 2007 and 2008. The pilot laboratory was the National Temperature Laboratory of Chile (LCPNT), which is accredited by the German Accreditation Body DAkkS (earlier DKD) since 2001.

COMPARISON

General guidelines
A measurement protocol was given to each participant. It stated the following relevant technical aspects:

The first step was performed to compare the calibration of two Industrial Platinum Resistance Thermometer IPRT of 100 Ohm, covering the temperature range from -39 °C up to 232 °C. These artifacts are calibrated using the reference thermometers of each participant.

The working instructions of each participant comply with the calibration by comparison "Techniques for approximating the International Temperature Scale of 1990"[1] used the ITS-90 deviation equations and the calculation procedure cross of least squares. The deviation equations used for the range *(-40 ºC to +30 ºC):* was $W(T_{90}) - Wr(T_{90}) = A\,(W(T_{90}) - 1) + B\,(W(T_{90}) - 1)^2$ and for the range *(0 ºC to +232 ºC):* was $W(T_{90}) - Wr(T_{90}) = A\,(W(T_{90}) - 1) + B\,(W(T_{90}) - 1)^2$.

Calibrations of each participant were performed by direct comparison in stirred liquid baths. Results were analyzed using the difference in temperature obtained for each artifact when using the calibration coefficients determined for the participants and the Normalized Error (E_N). Only one measurement at -39 °C showed an $E_N > 1$ and corrective actions were agreed. A final report for this first step was emitted and signed by both participants.

The second step consisted in a comparison of an Standard Platinum Resistance Thermometer of 25.5 Ohm, covering the temperature range from -39 °C up to 232 °C. The same first step protocol to complete the measurements and analyze the results was followed. All temperatures show an $E_N < 1$. A final report for this second step was emitted and signed by both participants.

Uncertainties were evaluated according to the ISO *Guide to the Expression of Uncertainty in Measurement* [2].

Protocol for Bilateral Comparison for the SPRT Calibration by Comparison Over the Range from -39 °C to 232 °C

Participant information is as follows:

LCPNT – Chile

TABLE 1. Measurement Sequence

Measurement Sequence	Description	Time Period
1	LCPNT complete measurements of SPRT (pre-submission to INEN)	2007, November
2	LCPNT deliver SPRT to INEN	2008, June
3	INEN complete measurements	2008, July
4	LCPNT complete measurements of SPRT (post-submission to INEN)	2008, September
5	Draft A Report submitted to participants	2008, September

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The instructions and procedures given below must be followed by the participants. The laboratories agree to follow the general instructions and technical protocol written in this document, the MRA Appendix F document "Guidelines for CIPM Key Comparisons", and the JCRB document "A Note on Supplementary Comparison" by T.J. Quinn.

The MRA Appendix F and JCRB documents are found at:

1) www.bipm.fr\pdf\guidelines.pdf,
2) and,
www.bipm.org/CC/documents/JCRB/Supplementary_Compns.doc, respectively.

This comparison is designed as a bilateral comparison. The LCPNT supply one SPRT 25 Ohm. Comparison will be performed by a calibration by comparison covering the range of temperature from *-39 °C to 232 °C (points; -39 °C, -20 °C, 0 °C, 15 °C, 30 °C, 80 °C, 157 °C, 200 °C, 232 °C).* Participants will use their own procedures to perform their calibrations. Results of the comparison will be calculated by the LCPNT personnel.

Transfer artifacts

The LCPNT will supply one SPRT for the comparison. The measurement sequence of the SPRT is as follows:

1) The SPRT is calibrated by the LCPNT by

2) comparison prior to sending the artifacts to INEN.
3) The SPRT is calibrated by the INEN by comparison. Calibration certificate is sent to LCPNT.
4) The SPRTs is returned to the LCPNT and is calibrated a second time to close the loop.
5) Results after each measurement set (steps 1, 2, and 3) are reported (Table 1). Measurement Sequence)

Technical Instructions

LCPNT measurements (Pre-submission to INEN)

The 25.5 Ω SPRT chosen by the LCPNT must meet the ITS-90 purity requirements and may have either a glass or metal sheath.

The triple point of water (TPW) was measured after every annealing cycle.

After annealing and stabilizing the SPRT, the measurement sequence to be followed is the TPW, *232 °C, 200 °C, 157 °C, TPW, 80 °C, 30 °C, 15 °C, 0 °C (Ice Point optional), TPW, -20 °C, -39 °C* and TPW. All results must be reported for 1 mA.

INEN measurements

Upon receipt of the SPRT, INEN will inspect the artifacts for damage. If there is damage, the LCPNT and the INEN will discuss and agree on how to proceed.

If no damage has been sustained, the SPRT will be measured at the TPW upon receipt. The "as received" [R(273.16 K) Ω, 0 mA] values will be reported to the LCPNT prior to annealing the SPRT.

Prior to the comparison measurements, the SPRT will be stabilized by annealing at a temperature of 425 °C. The stability criteria value for an SPRT is calculated from the difference between the R(273.16 K) Ω, 0 mA values measured before and after an anneal, the results are described in the tables 6 and 8.

After annealing and stabilizing the SPRT, the measurement sequence to be followed is the TPW, *232 °C, 200 °C, 157 °C,* TPW, *80 °C, 30 °C, 15 °C, 0 °C*

(or Ice Point), TPW, *-20 °C, -39 °C* and TPW. All results must be reported for 1 mA.

On completion of the set of measurements, INEN return the artifacts to LCPNT. Transport may be in the form of hand carrying or shipping. Hand carrying is the preferred method.

LCPNT measurements (Post-submission to INEN)

In order to close the measurement loop, the LCPNT will repeat steps outlined above in the LCPNT measurements (Pre-submission to INEN) section of this protocol.

Reporting of Data

The participating laboratories must submit the following:

1. Record the artifact Rt(1 mA) results, reference t90 (defined by the participating laboratory) and ITS-90 deviation function coefficients.

2. A calibration certificate will be submitted.

Reporting of Uncertainties

The individual uncertainty components should be listed along with the total combined uncertainty assigned to each of the calibration points. All expanded uncertainties should be expressed as k=2. In an effort to harmonize the calibration point uncertainty budgets used by the participants.
The uncertainties declared in the calibration certificates by the participants are the reported like Calibration and Measurements Capabilities.

The calibration point measurement reported in each calibration report and its associated uncertainties for the SPRT transfer artifact will be combined to generate only one difference and associated uncertainty for calibration point.

If the SPRT transfer artifact fails or is found to be unstable during the comparison (e.g. SPRT changes at the TPW during the measurements by more than the laboratory's allowable limit), the participating laboratories will discuss and agree upon a course of action. If the SPRT is stable at LCPNT but unstable (e.g. SPRT changes at the TPW during the measurements by more than the laboratory's allowable limit) for the second set of data, then only the first set of LCPNT data will be used.

The two outcome results to be reported are:

1. bilateral differences with associated uncertainties at each measured calibration point among the participants,

2. the Normalized Error (EN). Will be used the uncertainties (k=2) reported in the calibration certificates of the participants.

Artifacts
The LCPNT provided the artifacts to be compared in both steps. Artifacts features are shown in table 1.1.

Standards and Equipment
Features about standards and equipment are shown in table 2. Complementary equipment is shown in table 3.

TABLE 1.1. Comparison artifacts feature.

Manufacturer	Model	Series	Range of Measurement [*°C*]	R(0 °C) [Ω]	Nominal Alpha Coefficient [Ω/Ω/*°C*]	Dimension / Min. Immersion [*cm*]	Date of Measurement
Isotech	T100-NTS587	K0812A	-40 a 232	100 Ω	0,00392	0,635 x 46 / 30	2007
ASL	T100-450-3	B595354	-40 a 232	100 Ω	0,00392	0,635 x 45 / 30	2007
Hart Scientific	5628	61084	-40 a 232	25 Ω	0,00392	0,7 x 51 / 30	2008

TABLE 2. Standards and equipment

Laboratory	Model	Manufacturer	Model	Serial Number	Measure /Calibration Range	Calibration Method	Traceability
LCPNT	SPRT 25 Ω	Rosemount	162 CG	4592	TP-Hg … FP-Ga TP-water ... SP-Zn	Fixed point	CENAM
	SPRT 25 Ω	Rosemount	162 CG	4593	TP-Hg … FP-Ga TP-water ... SP-Zn	Fixed point	CENAM
	Resistor 100 Ω	Tinsley	5685 A	274560	100 Ohm	Comparison	CENAM
	Bridge	ASL	F18	4135-001-163	0 …1,2999999	--	--
	Triple point of water	NPL	Type 32	1064	0,01 °C	Comparison	NPL
INEN	SPRT 25 Ω	Hart Scientific	5681	1503	TP-Hg … FP-Ga TP-water ... SP-Zn	Fixed point	CENAM
	SPRT 25 Ω	Hart Scientific	5681	1514	TP-Hg … FP-Ga TP-water ... SP-Zn	Fixed point	CENAM
	Bridge	Fluke- Hart Scientific	1590	A63325	1Ω…10KΩ	Comparison	Fluke- Hart Scientific
	Triple point of water	Hart Scientific	Type B	D-G 1096	0,01 °C	Comparison	Hart Scientific

TABLE 3. Complementary equipment

Laboratory	Description	Manufacturer	Model	Medium
LCPNT	Stirred liquid bath	Hart Scientific	7037	Alcohol / distilled water
	Stirred liquid bath	Hart Scientific	6022	Silicon oil
INEN	Stirred liquid bath	Hart Scientific	7312	Alcohol / distilled water
	Stirred liquid bath	Isotech	915	Alcohol
	Stirred liquid bath	Hart Scientific	6022	Silicon oil

Comparison round.

The comparison sequence for both steps was performed as follows:

- LCPNT performs the initial measure,
- LCPNT transports the artifacts by hand to INEN,
- INEN performs its measure,
- LCPNT transports the artifacts by hand (return),
- LCPNT checks the artifact to complete its measure.

RESULTS

Each participant documents their results in a traceable calibration certificate. To evaluate the temperature difference Δt(INEN - LCPNT) between each participant, the Rt_{90} [Ohm] reported in the calibration certificate by the INEN is used to fix a temperature point to be compared. The temperature t_{90} [°C] calculated by each participant for each artifact was obtained using the coefficients informed in the individual calibration certificate. The expanded uncertainties (k=2) U [mK] used to evaluate de normalized error E_N correspond to the uncertainties informed by each participant in the individual calibration certificate for each artifact.

The normalized error E_N is defined by the following equation;

$$E_N = \frac{\Delta t(INEN - LCPNT)}{\sqrt{((U/INEN)^2 + (U/LCPNT)^2)}}$$

At the first step, results showed to be equivalent for both IPRTs Isotech (see table 4) and ASL (see table 5) in the measured temperature range. Only at the nominal temperature equal to -39 °C, the $E_N > 1$ was obtained for both artifacts. Investigation showed that the stirred liquid bath used by the INEN had a mechanical problem in the system of agitation, and the rheostat was repaired.

Corrective actions were agreed for the second step. IPRTs instability measured at the triple point of water $R_{0.01}$ [Ohm] for both artifact at the complete comparison round at the first step showed to be < 4 mK (see table 6).

In the case of the second step, results showed to be equivalent for the Hart Scientific SPRT (see table 7) in the measured temperature range. All measured temperatures showed $E_N < 1$.

Corrective actions agreed in the first step showed to be appropriate. SPRTs instability measured at the triple point of water $R_{0.01}$ [Ohm] at the complete comparison round at the second step showed to be < 2.5 mK (see table 8).

TABLE 4. Results for Isotech IPRT, s/n K0812A

Rt_{90} [Ω]	Nominal Value [°C]	t_{90} / INEN [°C]	$U^{(1)}$ / INEN [mK]	t_{90} / LCPNT [°C]	$U^{(1)}$ / LCPNT [mK]	Δt(INEN - LCPNT) [mK]	E_N
189.051 86	232	231.505	30	231.493	15	11.8	0.35
177.080 36	200	199.393	30	199.382	15	10.6	0.32
160.950 66	157	156.639	30	156.622	15	17.5	0.52
131.611 89	80	80.275	20	80.269	12	6.1	0.26
111.973 75	30	30.151	20	30.147	12	3.6	0.15
106.143 11	15	15.417	20	15.413	12	3.8	0.16
100.014 02	0	0.001	10	-0.004	10	5.0	0.35
92.078 92	-20	-19.850	30	-19.856	15	6.2	0.19
84.457 88	-39	-38.768	30	-38.809	15	40.5	1.21

(1) All uncertainties in table 4 are expressed as expanded uncertainties (k=2).

TABLE 5. Results for ASL IPRT, s/n B535954

Rt_{90} [Ω]	Nominal Value [°C]	t_{90} / INEN [°C]	$U^{(1)}$ / INEN [mK]	t_{90} / LCPNT [°C]	$U^{(1)}$ / LCPNT [mK]	Δt(INEN - LCPNT) [mK]	E_N
189.147 43	232	231.506	30	231.501	15	4.8	0.14
177.167 37	200	199.395	30	199.391	15	3.9	0.12
160.023 38	157	156.631	30	156.624	15	6.8	0.20
131.661 90	80	80.276	20	80.270	12	5.8	0.25
112.00845	30	30.151	20	30.148	12	2.9	0.13
106.173 23	15	15.417	20	15.413	12	3.6	0.15
100.039 59	0	0.001	10	-0.004	10	4.1	0.29
92.097 74	-20	-19.850	30	-19.857	15	7.2	0.21
84.471 65	-39	-38.768	30	-38.808	15	40.5	1.21

(1) All uncertainties in table 5 are expressed as expanded uncertainties (k=2).

TABLE 6. IPRTs instability at the first step

IPRT	Laboratory	R(0.01 °C) [Ω]	ΔT(max – min) [mK]*
Isotech	LCPNT (initial)	100.019 60	3.8
	INEN	100.018 07	
	LCPNT (final)	100.018 60	
ASL	LCPNT (initial)	100.044 80	2.0
	INEN	100.043 99	
	LCPNT (final)	100.044 20	

* The ΔT(max – min) absolute value is calculated with de difference between the R(0.01 °C) maximum – R(0.01 °C) minimum measured for each artifact by each participant.

TABLE 7. Results for Hart Scientific SPRT, s/n 61084

Rt_{90} [Ω]	Nominal Value [°C]	t_{90} / INEN [°C]	$U^{(1)}$ / INEN [mK]	t_{90} / LCPNT [°C]	$U^{(1)}$ / LCPNT [mK]	Δt(INEN - LCPNT) [mK]	E_N
47.143 986	232	232.111	25	232.109	15	2,3	0.08
44.167 191	200	200.066	25	200.064	15	1,7	0.06
40.126 042	157	157.070	25	157.068	15	1,2	0.04
32.789 211	80	80.445	15	80.444	12	1,0	0.05
27.868 946	30	30.052	15	30.051	12	1,6	0.08
26.393 262	15	15.089	15	15.087	12	1,9	0.10
24.899 335	0	0,010	4	0.008	9	2,3	0.24
22.902 602	-20	-20.037	20	-20.038	15	1,4	0.06
20.980 237	-39	-39.220	20	-39.220	15	0,5	0.02

(1) All uncertainties in table 7 are expressed as expanded uncertainties (k=2).

TABLE 8. SPRTs instability at the second step

SPRT	Laboratory	R(0.01 °C) [Ω]	ΔT(max – min) [mK]*
Hart Scientific	LCPNT (initial)	24.899 58	2.4
	INEN	24.899 34	
	LCPNT (final)	24.899 46	

* The ΔT(max – min) absolute value is calculated with de difference between the R(0.01 °C) maximum – R(0.01 °C) minimum measured for each artifact by each participant.

CONCLUSION

Concluded the bilateral comparison, final results in the second step (see table 7) showed an improvement respect the results in the first step (see table 5 and 6). Improvement proposed by the LCPNT-CESMEC to INEN in its measurement system and its working instructions showed to be effective.

Calibration performed by comparison in the range *-40 °C to 232 °C* by the participant showed to be equivalent in accordance with the claimed CMCs.

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REFERENCES

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