FINAL REPORT OF BILATERAL COMPARISON SIM.M.P-S8
Hydraulic Pressure Comparison from 7 MPa to 70 MPa

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Abstract: This report shows the final results of the bilateral comparison SIM.M.P-S8 between the Custodian Laboratory of National Pressure Standards of Chile, LCPN-P (with the National Aeronautics Company - ENAER) and the National Institute of Technology, Standardization and Metrology, INTN of Paraguay, in the field of hydraulic pressure up to 70 MPa. The comparison was carried out between December 2011 and May 2012. Each laboratory used a pressure balance as a reference standard covering the range from 7 MPa to 70 MPa for the comparison. The transfer standard for the comparison was a digital manometer OMEGA, PCL-3000-4 with an accuracy of 0.05 % of full scale and a resolution of 0.01 MPa.
The pilot laboratory, LCPN-P, provided the transfer standard and made the analysis of the results, while the reference value was provided by INTN Paraguay.
An analysis of compatibility between the results of the present bilateral comparison and the supplementary comparison SIM.M.P-S7 was made, obtaining satisfactory results, which enables a link between both comparisons.

1.- INTRODUCTION

The comparisons between National Metrology Institutes (NMI), allow to ensure the equivalence of the measurements between the countries and the correct dissemination of the magnitude. This strengthens the robustness and confidence of measurement systems, both nationally and internationally.

A comparison in hydraulic gauge pressure was made between the NMIs of Paraguay and Chile (INTN and LCPN-P respectively), where a high accuracy digital manometer was calibrated by both NMIs. The calibration procedure used for this comparison is based on the DKD-R 6-1 and consists in 2 measurement cycles (4 series) of alternated ascendant and descendant series, each one of them with 10 measurement points corresponding to 10 %, 20 %, 30 %, 40 %, 50 %, 60 %, 70 %, 80 %, 90 % and 100 % of the measuring scope of the instrument, plus 0. In this way each pressure point is measured 4 times in total.

The results of this comparison can be linked to the SIM.M.P-S7 comparison, where the Mexico's NMI, CENAM, was the reference laboratory and advisor for the comparison, and INTN was a participant with satisfactory results. This link is possible since the SIM.M.P-S7 was carried out on the same period of time in which the comparison with the Chilean institute was made, the measurement range of both comparisons was equal (0 MPa to 70 MPa) and the reference standard instrument used by INTN in both comparison was the same.

The comparison was carried out within the regulatory framework of the SIM.

2.- OBJECTIVE

Make a comparison of the pressure (hydraulic relative) magnitude, in the measurement range of up to 70 MPa between the national metrological laboratories of the countries of Paraguay (INTN) and Chile (LCPN-P), in order to estimate the levels of agreement on measurements, including deviation and uncertainty.

3.- GENERAL DATA

3.1 Participant Laboratories

For this comparison, the two participating laboratories used high-accuracy pressure balances (dead weight balance) with single piston cylinder assemblies as reference standards. Table 1 presents the planned and the executed round, with the technical characteristics of the equipment used by each laboratory.

Table 1. *Participant Laboratories and its standard reference.*

Country	**Chile**	**Paraguay**
Institute	LCPN-P (ENAER)	INTN
Calibrated by	Angel Flores Torres angel.flores@enaer.cl	Natalia Vega Gamarra nvega@intn.gov.py
Planned measurement dates	1 Feb 2012 and 5 March 2012	21 Feb 2012
Date of calibration	29 Dec 2011 and May 2012	Feb 2012
Best measurement capability accredited	$4.0 \cdot 10^{-5} \cdot p_e$, p_e in bar	0.02 % of Reading
Fluid	Sebacate Oil	D22 Oil
Equipment used as a standard	Pressure Balance	Pressure Balance
Brand	Ruska	WIKA
Model	2485-983	CPB5000
Series No	J-352	50362
Identification	MPRh-1	LPR – PR – 02
Effective area Piston-cylinder (m^2)	7.10970E-06	5.000622E-06
Accuracy	0.0025 % of Reading	0.015 % of Reading
Unit	MPa	MPa
Local Gravity ms^{-2}	9.794 227	9.789 40
Scope	140 MPa	100 MPa
U, k = 2	$32\ Pa + 2.8 \cdot 10^{-5} \cdot p_e + 1.5 \cdot 10^{-13} \cdot p_e^2 / Pa$	0.01 % of Reading
Traceability	PTB	PTB

3.2 Transfer Standard Instrument

A digital manometer (electric pressure gauge) was used as the transfer standard instrument, which was facilitated by the LCPN-P. Its characteristics are presented in Table 2.

Table 2. *Transfer standard characteristics*

Type of Instrument:	Digital calibrator
Measurement Range:	0 MPa to 70 MPa
Measuring unit:	MPa
Resolution:	0.01 MPa
Accuracy class:	±0.05 % of full scale
Transmission medium	Sebacate oil (LCPN-P), D22 oil (INTN)
Maker:	OMEGA
Model:	PCL-3000-4
Serial number:	A 25297

3.3 Comparison program

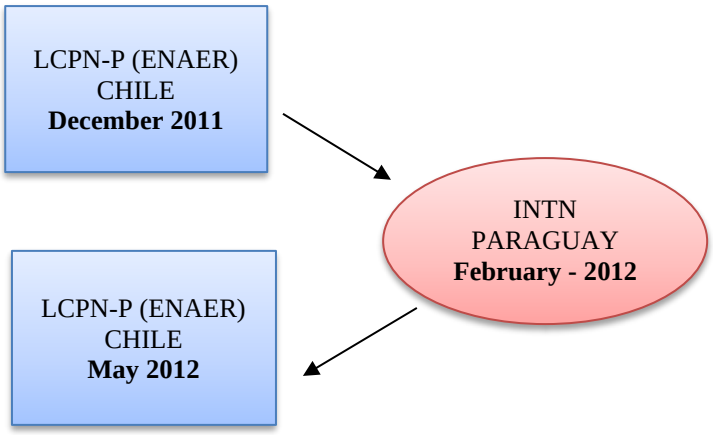

Figure 1. *Schematic of the comparison round.*

a) Preparation: LCPN-P made the initial measurements on December 29, 2011, establishing the general guidelines for the comparison.

b) LCPN-P made the corresponding measurements according to the scheduled dates. The pilot laboratory establishes the base documentation and activities to be carried out.

c) The transfer standard is personally taken by an LCPN-P's technician in February 2012 to INTN, where it is calibrated by INTN's technicians according to the procedure established by the pilot.

d) The instrument returns to LCPN-P and was calibrated in May 2012. This is the last measurement of the comparison.

e) The results obtained by each laboratory are sent to the pilot, who processes the data and presents its conclusions in a report.

f) The report is presented to the participants for corrections and observations. Once accepted by the participants, is sent to SIM for its final evaluation and publication.

4.- RESULTS

4.1 Behavior of the transfer standard

The pilot laboratory LCPN-P calibrated the transfer standard two times. The data obtained is presented in Table 3.

Table 3. *Error, E, and expanded uncertainty, measurements LCPN-P Chile*

P **MPa**	**Error M1**	***U*** **M1**	**Error M2**	***U*** **M2**
0	0.003	0.008	-0.002	0.012
7	0.003	0.007	-0.002	0.015
14	0.015	0.008	0.004	0.014
21	0.001	0.008	-0.006	0.015
28	0.002	0.008	-0.008	0.017
35	-0.011	0.012	-0.018	0.015
42	-0.019	0.009	-0.021	0.017
49	-0.016	0.009	-0.026	0.013
56	-0.021	0.009	-0.032	0.014
63	-0.018	0.009	-0.027	0.012
70	-0.023	0.010	-0.034	0.012

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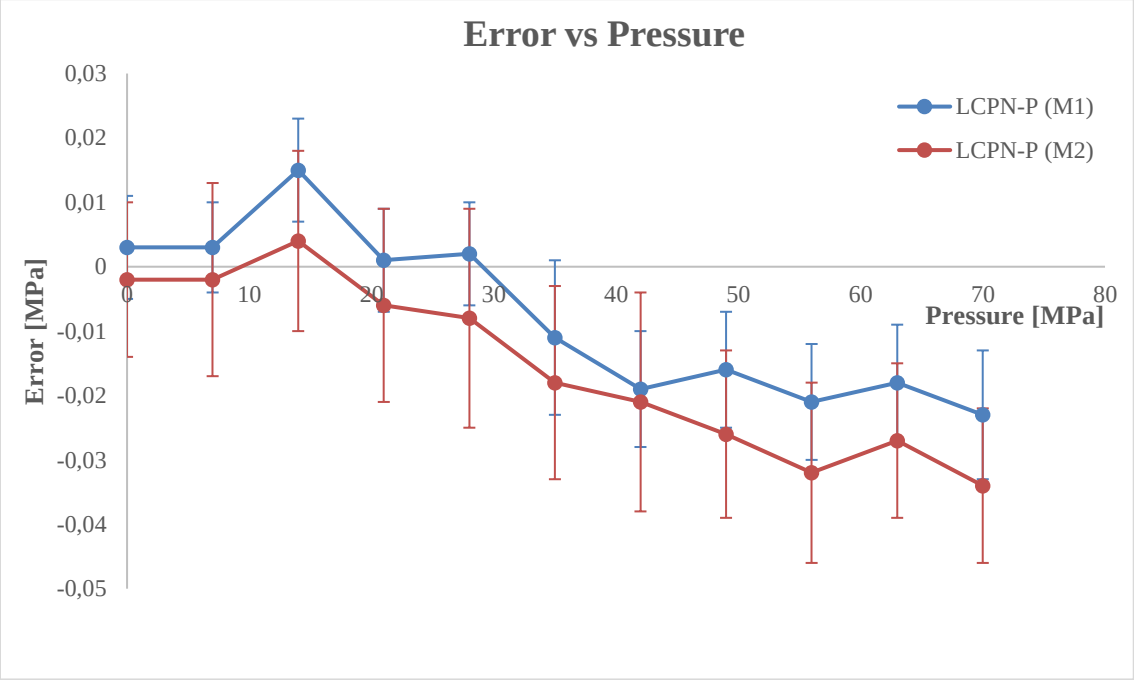
Table 3 shows the error *E* obtained at each measurement point and the measurement uncertainties associated with the calibration performed by the LCPN-P at the beginning and end of this comparison, the results are plotted in Graphic 1.



Graphic 1. *Error E, and expanded uncertainty, U (k=2, 95%). For each measurement, M1 and M2, made by the LCPN-P of ENAER.*

From the calibration results obtained by the LCPN-P, it was found that the device drifted between the measurements made in December 2011 and May 2012. The drift will be considered as a contribution to the uncertainty of the pilot laboratory as shown in equation 1.

$$U_{LCPN-P} = 2 * \sqrt{\left(\frac{U}{k}\right)^2 + \left(\frac{d}{\sqrt{3}}\right)^2} \qquad (1)$$

The values for the device drift found are shown in table 4

Table 4. *Drift found between the measurements made by LCPN-P Chile.*

P **MPa**	**Drift MPa**
0	-0.005
7	-0.005
14	-0.011
21	-0.007
28	-0.010
35	-0.007
42	-0.002
49	-0.010
56	-0.011
63	-0.009
70	-0.011

During the period of the comparison, the transfer standard showed good short-term stability, this can be seen in Table 3, where the mentioned calibrations were carried out in different dates with small variations of temperature and atmospheric pressure, maintaining the trend and curve of error, the small differences observed between Measurement 1 and Measurement 2 shows the drift of the transfer standard, which is presented as a decrease of the reading in the instrument with respect to the time. The maximum calculated drift is 0.011 MPa, which represents a maximum error of 0.016 % of full scale, this value is 3.2 times better than the declared accuracy for the instrument.

Due to the drift found, it was decided to analyze the measurements made by the LCPN-P separately and not as the mean of the measurements, in this way the normalized errors will be calculated for the data set M1, corresponding to measurement 1 performed at beginning of the round, and for data set M2 corresponding to the second measurement performed at the end of the comparison

4.2 Measurement data

Table 5 shows the measurement error *E* associated with the calibration performed by participating laboratories INTN and LCPN-P.

Table 5. *Error, E, obtained at each point.*

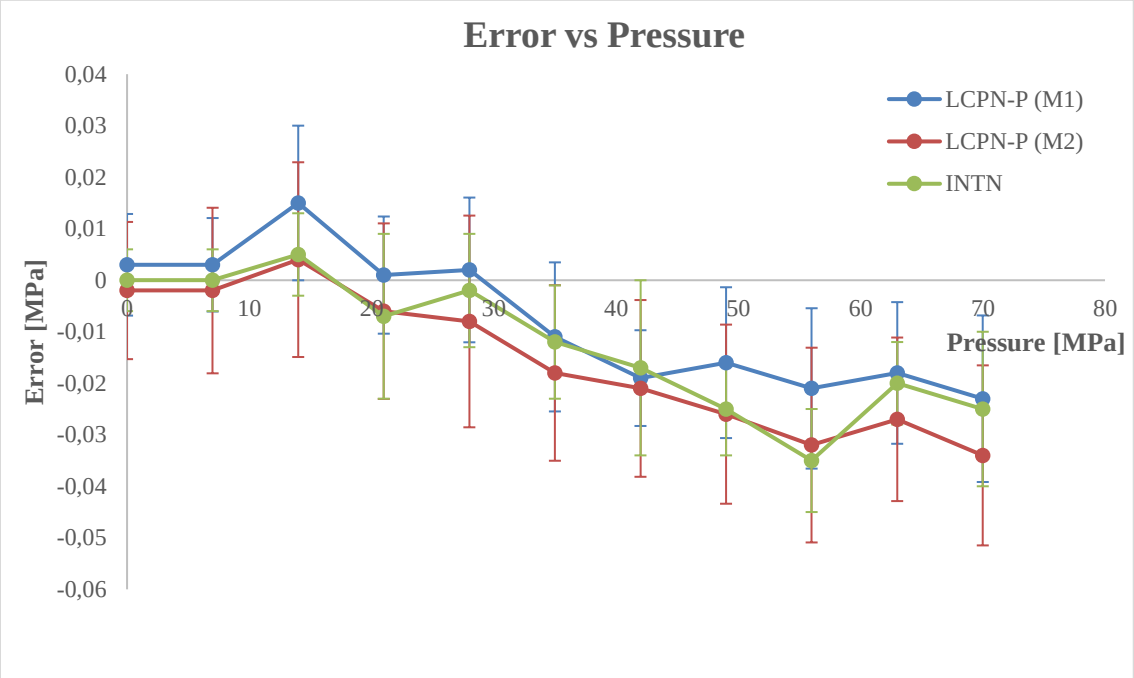
P **MPa**	**Error LCPN-P (M1)**	**Error LCPN-P (M2)**	**Error INTN**
0	0.003	-0.002	0
7	0.003	-0.002	0
14	0.015	0.004	0.005
21	0.001	-0.006	-0.007
28	0.002	-0.008	-0.002
35	-0.011	-0.018	-0.012
42	-0.019	-0.021	-0.017
49	-0.016	-0.026	-0.025
56	-0.021	-0.032	-0.035
63	-0.018	-0.027	-0.020
70	-0.023	-0.034	-0.025

In table 6 are the expanded uncertainty *U* of measurement associated with the calibration performed by the participating laboratories INTN and LCPN-P.

Table 6. *Uncertainty U, obtained at each point. For the uncertainties of the LCPN-P an additional contribution by the drift of the transfer standard is considered.*

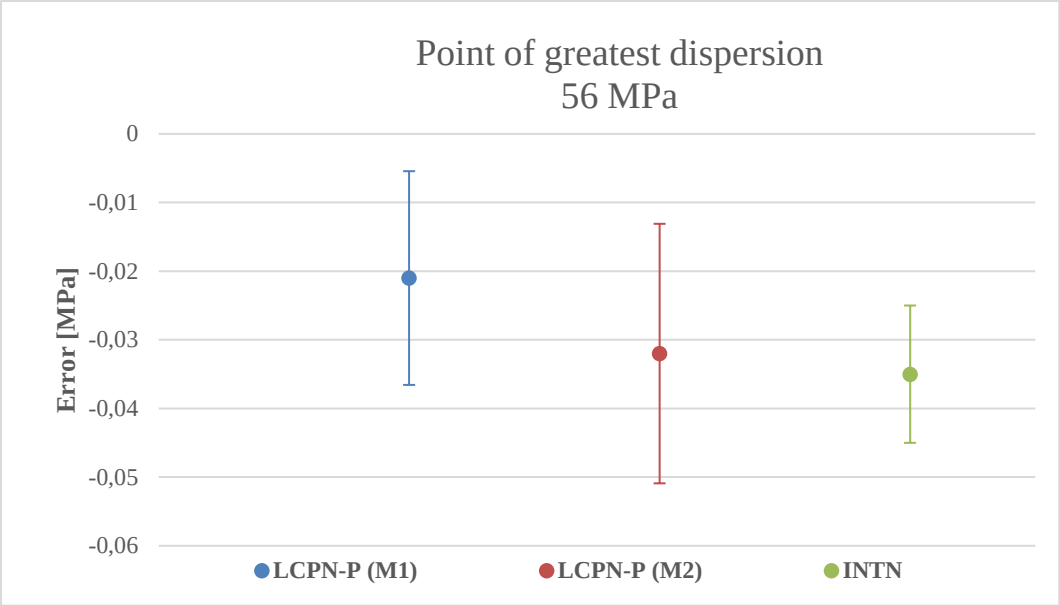
P **MPa**	***U*** **LCPN-P (M1)**	***U*** **LCPN-P (M2)**	***U*** **INTN**
0	0.010	0.013	0.006
7	0.009	0.016	0.006
14	0.015	0.019	0.008
21	0.011	0.017	0.016
28	0.014	0.021	0.011
35	0.014	0.017	0.011
42	0.009	0.017	0.017
49	0.015	0.017	0.009
56	0.016	0.019	0.010
63	0.014	0.016	0.008
70	0.016	0.017	0.015

The results of the measurements made by both laboratories and their respective uncertainties are plotted in Graphic 2



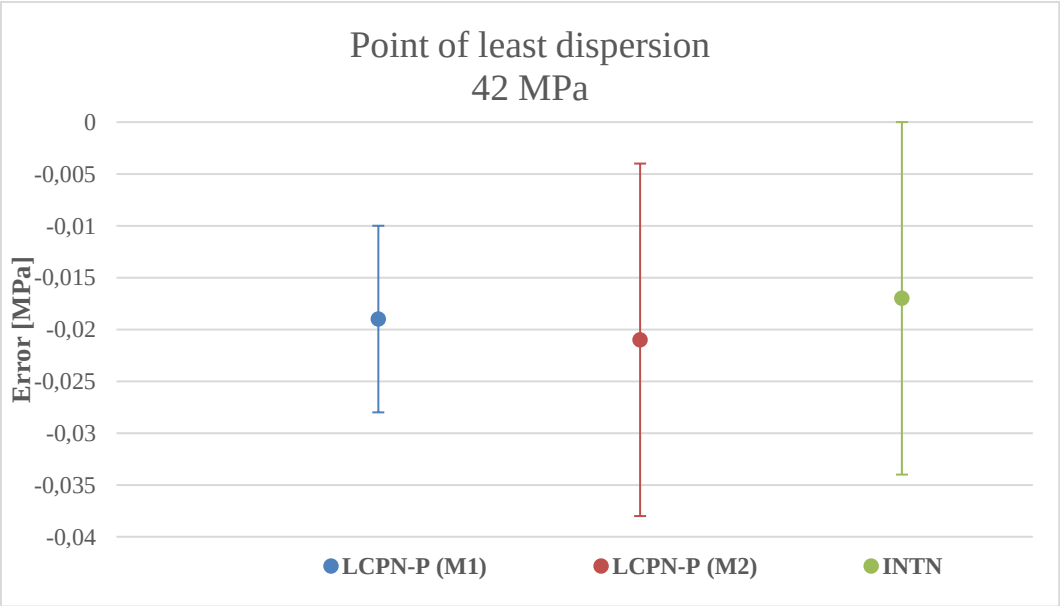
Graphic 2. *Error E, and expanded uncertainty, U (k = 2, 95 %). By each of the participating laboratories.*

Graphic 3 shows the measurement point 56 MPa where the highest dispersion and therefore the least agreement between the results of the participating laboratories was found, according to the data in tables 5 and 6.



Graphic 3. *Error of the participating laboratories, E, with expanded uncertainty, U, of each laboratory at the measurement point with greater dispersion.*

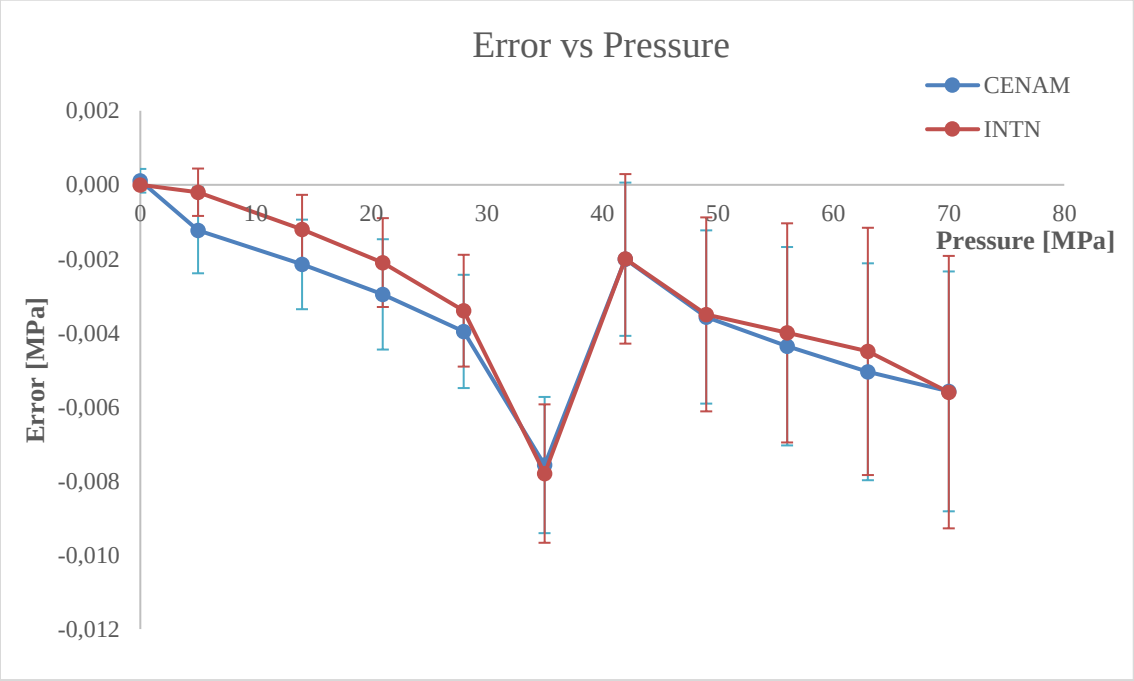
Graphic 4 shows the measuring point 42 MPa, where the least dispersion and therefore the best agreement between the results of the participating laboratories was presented, according to the data in tables 5 and 6.



Graphic 4. *Error of the participating laboratories, E, with expanded uncertainty, U, at the measurement point with less dispersion.*

5. DISCUSSION OF RESULTS

In the same period of time in which this comparison was carried out, the INTN was participating in the supplementary comparison SIM.M.P-S7, where the reference laboratory and advisor for the comparison was CENAM, Mexico. The P-S7 comparison was in hydraulic pressure, in the range 0 MPa to 70 MPa, and INTN participated using the same reference standard used for the comparison presented in this report. In graphic 5 are displayed the results obtained by INTN and CENAM



Graphic 5. *Error E, and expanded uncertainty, U (k = 2, 95 %) of CENAM and INTN in SIM.M.P-S7*

To evaluate the performance of the participating laboratories in P-S7, the normalized error criteria, E_n, was used for each pressure point measured using the equation 2:

$$E_n = \frac{E_{lab} - E_{ref}}{\sqrt{U_{lab}^2 + U_{ref}^2}} \qquad (2)$$

Where:
E_{lab}: is the average error of a participating laboratory
E_{ref}: is the average error as determined by the reference
U_{lab}: is the expanded uncertainty of a participating laboratory
U_{ref}: is the expanded uncertainty of the reference

The normalized error criterion is: $|E_n| \leq 1.0$ for satisfactory performance and $|E_n| > 1.0$ for unsatisfactory performance.

In table 7 are the normalized error for each pressure point measured by INTN in SIM.M P-S7, where the biggest obtained was 0,78 in the measured point 5 MPa and the smaller absolute value of the normalized error got was 0 in the pressure points 42 MPa and 70 MPa. It's important to notice that for the P-S7 comparison it was suggested, during the meeting in Lima, to cover the range starting from 10 % of the full span, so that the nominal values of the first measurement point should be very close to 7 MPa, according to the values that the pressure balance of each institution could reproduce. In accordance with this agreement, the minimum point reported by the reference laboratory, CENAM, was 5 MPa, while the INTN reported 7 MPa.

As all the normalized errors calculated were smaller than 1, the performance of INTN in the comparison with CENAM is considered satisfactory, indicating that its calibration results are compatible with the reference measurements.

Table 7. *Normalized error obtained by INTN in SIM.M.P-S7.*

$P_{reference}$ **MPa**	**Normalized Error**
0	-0.32
5	0.78
14	0.62
21	0.45
28	0.26
35	-0.09
42	0.00
49	0.02
56	0.09
63	0.12
70	0.00

To determine if the values of the measurements made by INTN in the bilateral comparison with LCPN-P, SIM.M.P-S8, and the reference values of the supplementary comparison are consistent, and therefore the two comparisons can be linked, the degrees of equivalence are calculated using the equation 3. The degree of equivalence between two measurement standards is expressed as the difference between their respective deviations from the comparison reference value and the uncertainty of this difference:

$$d_{lab} = E_{lab} - E_{ref} \qquad u(d_{lab}) = \sqrt{\left(\frac{U_{lab}}{2}\right)^2 + \left(\frac{U_{ref}}{2}\right)^2} \qquad (3)$$

Where

d_{lab}: is the degree of equivalence

$u(d_{lab})$: is the standard uncertainty of de degree of equivalence

Then, the measurements are considered compatibles if equation 4 is true

$$|d_{lab}| \leq 2 * u(d_{lab}) \quad (4)$$

In the two comparisons, where the INTN was a participant, different transfer standards were used, with some variations of its metrological characteristics as seen in table 8, where the differences in resolution and accuracy can be noted, having the one used in the present comparison 100 times the resolution of the one used for SIM.M.P.S-7 and with an accuracy up to 6 times bigger, therefore a previous treatment to the data is needed to study the results of the participants by means of the degrees of equivalence, in order to take out the influence of the transfer standard.

Table 8. *Characteristics of the transfer standard used in both comparisons*

	SIM.M.P.S-8	**SIM.M.P.S-7**
Type of Instrument:	Digital calibrator	Reference Pressure Monitor
Measurement Range:	0 MPa to 70 MPa	Vaccum to 280 MPa
Measuring unit:	MPa	MPa
Resolution:	0.01 MPa	0.000 1 MPa
Accuracy class:	0.05 % of full scale	0.008 % of reading
Transmission medium	Sebacate oil (LCPN-P), D22 oil (INTN)	Shell Tellus oil 22 (INM), oil (INEN, INDECOPI), Sebacate oil (IBMETRO, LATU), D22 oil (INTN)
Maker:	OMEGA	FLUKE DH Instruments
Model:	PCL-3000-4	RPM4
Serial number:	A 25297	119

To remove the influence of the transfer standard, the differences between the informed errors of each participating laboratory with its respective reference was calculated, i.e., for the comparison SIM.M.P-S7 the difference between the error informed by INTN and CENAM is calculated; while for SIM.M.P-S8 the differences between the error of LCPN-P at M1 (first measurements at the beginning of the round) and the error of INTN is calculated, and the difference between the error of LCPN-P at M2 (second and last measurement made at the end of the round) and the error of INTN is calculated. The difference between the results, and its associated uncertainty, is considered to calculate the degrees of equivalence between both comparisons.

In table 9 are the differences found, where:

d_{INTN} : is the difference between the error informed by INTN with respect to the error informed by CENAM in SIM.M.P-S7

$d_{LCPN-P\,(M1)}$: is the difference between the error informed by LCPN-P in its first measurement with respect to the error informed by INTN in SIM.M.P-S8

$d_{LCPN-P\,(M2)}$: is the difference between the error informed by LCPN-P in its second and last measurement of the round, with respect to the error informed by INTN in SIM.M.P-S8

$u(d_{INTN})$: is the standard uncertainty of the difference between the error informed by INTN with respect to the error informed by CENAM in SIM.M.P-S7

$u(d_{LCPN-P\,(M1)})$: is the standard uncertainty of the difference between the error informed by LCPN-P in its first measurement, M1, with respect to the error informed by INTN in SIM.M.P-S8

$u(d_{LCPN-P\,(M2)})$: is the standard uncertainty difference between the error informed by LCPN-P in its second and last measurement of the round, with respect to the error informed by INTN in SIM.M.P-S8

Table 9. *Differences calculated between participating laboratories and the respective reference standard in comparisons SIM.M.P-S7 and SIM.M.P-S8, and its associated expanded uncertainties.*

P MPa	d_{INTN} MPa	$2 * u(d_{INTN})$ MPa	$d_{LCPN-P\,(M1)}$ MPa	$2 * u(d_{LCPN-P\,(M1)})$ MPa	$d_{LCPN-P\,(M2)}$ MPa	$2 * u(d_{LCPN-P\,(M2)})$ MPa
0	-0.000 11	0.000 35	0.003	0.012	-0.002	0.015
7	0.001 03	0.001 32	0.003	0.011	-0.002	0.017
14	0.000 95	0.001 53	0.010	0.017	-0.001	0.021
21	0.000 86	0.001 91	0.008	0.020	0.001	0.023
28	0.000 56	0.002 15	0.004	0.018	-0.006	0.023
35	-0.000 23	0.002 62	0.001	0.018	-0.006	0.020
42	0.000 01	0.003 09	-0.002	0.019	-0.004	0.024
49	0.000 07	0.003 51	0.009	0.017	-0.001	0.020
56	0.000 36	0.003 99	0.014	0.019	0.003	0.021
63	0.000 55	0.004 44	0.002	0.016	-0.007	0.018
70	-0.000 02	0.004 90	0.002	0.022	-0.009	0.023

In tables 10.a and 10.b are shown the degrees of equivalence obtained for the measurements made in this bilateral comparison of INTN and LCPN-P, considering as reference values the differences between the errors informed by INTN and CENAM for the SIM.M.P-S7 comparison, and its respective uncertainties, where:

$$d_{P-S8\,(M1)} = d_{LCPN-P\,(M1)} + d_{INTN} \qquad u\left(d_{P-S8\,(M1)}\right) = \sqrt{u\left(d_{LCPN-P\,(M1)}\right)^2 + u(d_{INTN})^2}$$

$$d_{P-S8\,(M2)} = d_{LCPN-P\,(M2)} + d_{INTN} \qquad u\left(d_{P-S8\,(M2)}\right) = \sqrt{u\left(d_{LCPN-P\,(M2)}\right)^2 + u(d_{INTN})^2}$$

Table 10.a *Degrees of equivalence between the first measurement M1 made by the participating laboratory LCPN-P in SIM.M.P-S8 and the measurements reported by INTN and CENAM in SIM.M.P-S7.*

P **MPa**	$\|d_{P-S8\,(M1)}\|$ **MPa**	$2 * u(d_{P-S8\,(M1)})$ **MPa**	$\|d_{P-S8\,(M1)}\| \leq 2 * u(d_{P-S8\,(M1)})$
0	0.002 89	0.012	True
7	0.004 03	0.011	True
14	0.010 95	0.017	True
21	0.008 86	0.020	True
28	0.004 56	0.018	True
35	0.000 77	0.018	True
42	0.001 99	0.020	True
49	0.009 07	0.018	True
56	0.014 36	0.019	True
63	0.002 55	0.017	True
70	0.001 98	0.023	True

Table 10.b *Degrees of equivalence between the first measurement M2 made by the participating laboratory LCPN-P in SIM.M.P-S8 and the measurements reported by INTN and CENAM in SIM.M.P-S7.*

P **MPa**	$\|d_{P-S8\,(M2)}\|$ **MPa**	$2 * u(d_{P-S8\,(M2)})$ **MPa**	$\|d_{P-S8\,(M2)}\| \leq 2 * u(d_{P-S8\,(M2)})$
0	0.002 11	0.015	True
7	0.000 97	0.017	True
14	0.000 05	0.021	True
21	0.001 86	0.023	True
28	0.005 44	0.023	True
35	0.006 23	0.020	True
42	0.003 99	0.024	True
49	0.000 93	0.020	True
56	0.003 36	0.022	True
63	0.006 45	0.018	True
70	0.009 02	0.024	True

As all the degrees of equivalence calculated satisfy the criteria of equation 4, and considering that the working interval, the reference standard used by INTN and the period of time in which the measurements were performed was the same, the comparisons can be considered compatible.

5.1 Reference values

As the INTN performance in the supplementary comparison with CENAM was satisfactory, its informed values of error and uncertainty will be considered as reference for the bilateral comparison with LCPN-P.

As mentioned above, an additional component for the device drift is considered in the measurement uncertainty of LCPN-P, as the difference between the errors obtained by the laboratory in the calibrations at the beginning and the end of the round of the comparison, and with a rectangular probability distribution associated.

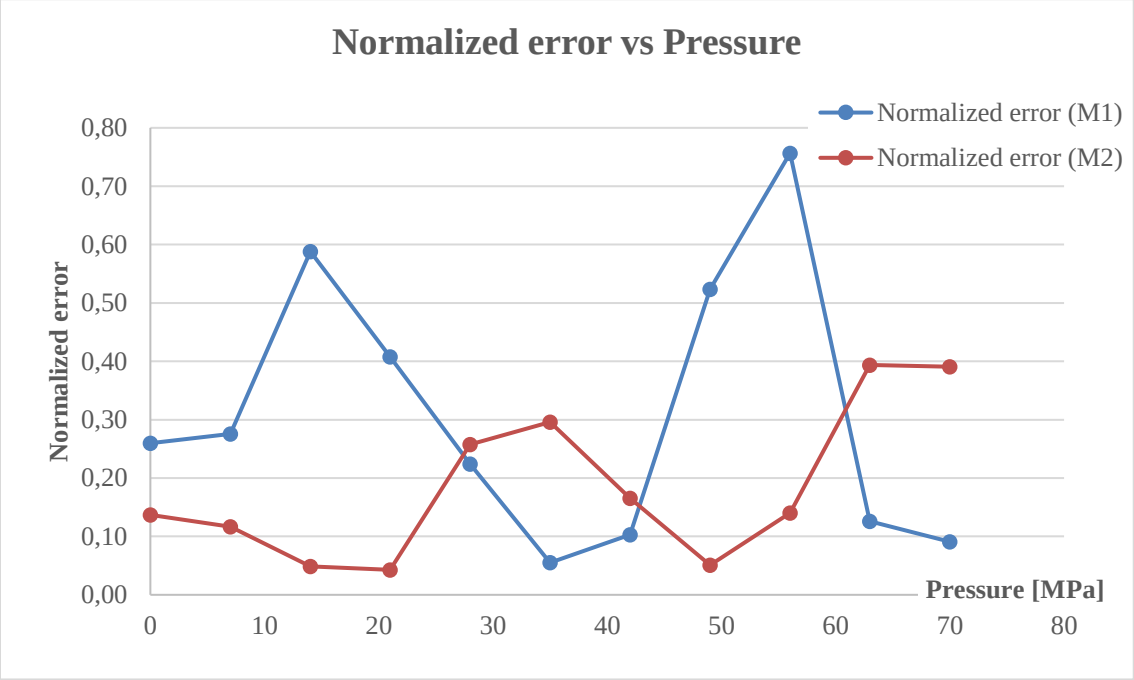
5.2 Performance of laboratories

The results obtained by the LCPN-P were analyzed using the normalized error criterion, E_n. The normalized error, E_n is applied for each measurement point examined by the laboratory, considering as reference the values reported by INTN.

The normalized errors, E_n, calculated for each measurement made by the LCPN-P in every measured point are shown in Table 11.

Table 11. *Normalized error, data of LCPN-P.*

Normalized error table (*En*)		
P **MPa**	**LCPN-P M1**	**LCPN-P M2**
0	0.26	0.14
7.0	0.28	0.12
14.0	0.59	0.05
21.0	0.41	0.04
28.0	0.22	0.26
35.0	0.06	0.30
42.0	0.10	0.17
49.0	0.52	0.05
56.0	0.76	0.14
63.0	0.13	0.39
70.0	0.09	0.39



Graphic 5. *Curves of the normalized error obtained for the LCPN-P measurements.*

6. CONCLUSIONS

The performance of the participating laboratories was satisfactory as all the normalized errors obtained were less than 1, indicating that the measurements between the reference, INTN, and the LCPN-P are compatible.

During the round of measurements a drift in the transfer standard was detected and its impact was considered as an additional contribution to the uncertainty of the LCPN-P, nevertheless the maximum drift found was 3 times less than the accuracy informed by the instrument's manufacturer and the device showed a good performance in repeatability and stability during all the measurements made, demonstrating the reliability of the instrument used as a transfer standard for the effects and purposes of this comparison.

From the results obtained in this comparison we can conclude that there is an agreement between the measurements made by each participating laboratory.

ACKNOWLEDGEMENTS.

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REFERENCES

[1] Deutscher Kalibrieerdienst DKD. Guideline DKD-R 6-1:2010 Calibration of Pressure Gauges.

[2] ISOIIEC 17025:2017 General requirements for the competence of testing and calibration laboratories

[3] ISO/IEC 17043:2010 Conformity assessment - General requirements for proficiency testing

[4] Statistical Interpretation of Key Comparison Reference Value and Degrees of Equivalence, R. N. Kacker, R. U. Datla, and A. C. Parr

[5] Method for confirmation of CMC, based on degree of equivalence of measurement standards, A.Chunovkina, N.Burmistrova

[6] Degrees of equivalence in a key comparison, Thang H. L., Nguyen D. D., Vietnam Metrology Institute