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# Pressure Standards Comparison between Germany and Mexico (Primary and Secondary Laboratories)

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**Abstract.** A pressure comparison was carried out among the primary laboratories of Mexico and Germany and secondary laboratories of Mexico and Germany, in order to estimate the level of agreement for the realization of the quantity and the uncertainty associated to its measurement. The comparison was carried out from 10 MPa up to 100 MPa, using an electronic comparison standard with $1 \cdot 10^{4}$ of full-scale accuracy. The results obtained and the deviations graphs that include the uncertainty for each participating laboratory are presented.

## 1. Introduction

The comparison was performed among laboratories using similar standards. The DKD (German Calibration Services) and SNC (Mexican National Calibration System) secondary laboratories have always had a keen interest in comparing their capabilities to realize the quantity of pressure, this was a welcome opportunity to perform a comparison with laboratories from other country. This comparison was carried out from 10 MPa up to 100 MPa (oil) and the national laboratories and at least 3 secondary laboratories from each country participated.

## 2. Scope of work

The ISO publication "International Vocabulary of Basic and General Terms of Metrology" (VIM) [1], and the International System of Units [2], SI, were used for the comparison and for the writing of this document. The recommendations established in the Guide to the Expression of Uncertainty in Measurement [3], were followed for the uncertainty evaluation.

### *2.1 Mexican participating secondary laboratories*

Table 1 presents the four participating laboratories from the Mexican National Calibration System (SNC). The Centro Nacional de Metrología, acted as the coordinator and pilot laboratory.

Table 1. Participating SNC laboratories.

| Accreditation ID. | Laboratory | Person in charge |
|---|---|---|
| P-34 | Industrias Técnicas Schob, S.A. de C.V. | Francisco González Hinojosa |
| P-36 | Caltechnix de México, SA de CV | Ana Lilia Hernández Cuevas |
| P-03 | LAPEM | David Jacobo Obregón |
| P-31 | MetAs | Víctor Aranda Contreras |

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### *2.2 German participating secondary laboratories*

There were three participating secondary laboratories which are listed in table 2. The table presents the laboratories from the German Calibration Service (DKD). The Physikalisch-Technische Bundesanstalt, PTB, acted as the coordinator and pilot laboratory.

Table 2. Participating DKD laboratories.

| Accreditation ID. | Laboratory | Person in charge |
|---|---|---|
| DKD-K-23501 | AKS-Messtechnik GmbH | Holger Guenther |
| DKD-K-05801 | DH-Budenberg (Desgranges et Huot) | Hendrik Schumacher |
| DKD-K-03401 | tecsis GmbH | H. J. Strube |

### *2.3 Comparison standard*

Table 3 shows the information of the comparison standard used, as it was provided by the manufacturer [4].

Table 3. Comparison standard data.

| | |
|---|---|
| Transducer Type: | Oscillating quartz crystal |
| Range: | 10 MPa to 100 MPa |
| Units: | kPa |
| Resolution: | 0,1 kPa |
| Accuracy Class: | 0,01% |
| Predicted stability | 0,009% in 1 year |
| Uncertainty: | 0,004% of full scale |
| Make: | DH Instruments |
| Model: | RPM3 A15000-L |
| Serial number or Identification: | 675 |

### *2.4 Comparison rounds*

The comparison was performed in two rounds, each one included initial and final measurements in the pilot laboratory (PTB or CENAM). The rounds were performed as shown in figure 1. A round was carried out in each country.

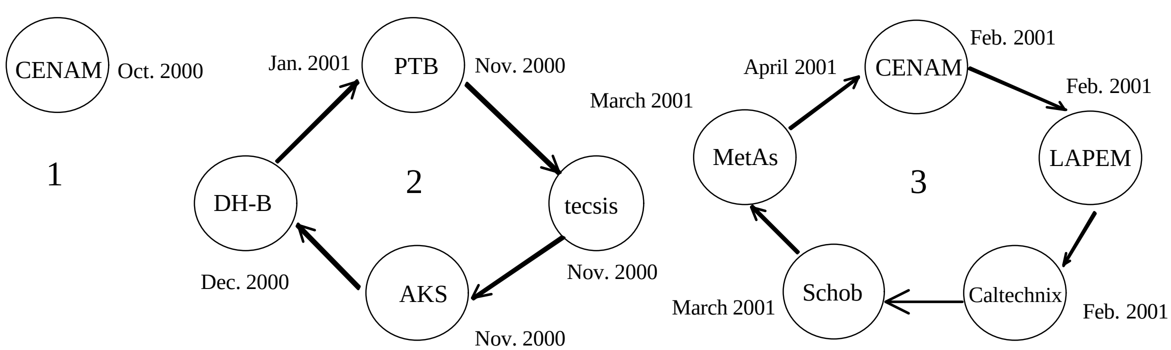


Figure 1. Comparison rounds.

### *2.5 General guidelines and procedure*

The relevant aspects of the measurement protocol are summarized here, but were elaborated in deeper form in the documents *"General Guidelines for the:* Pressure Standards Comparison between Germany and Mexico (primary and secondary laboratories)" [5] and *"Measurement Instructions for the*: Calibration of an electronic (oscillating quartz resonator) pressure gauge up to 100 MPa" [6].

a) It was recommended that the comparison standard was connected to the power supply in the place where it was going to be calibrated and the default settings (normal operation settings) were checked and entered 24 hours before starting the measurement procedure, for both, warming up and stabilization.
b) The comparison standard was set to zero before the calibration began and after each loop. The first "zero" setting entered the local atmospheric pressure as the reference pressure, the following "zero" setting permitted to diminish the influence of the zero drift.
c) The measurements on the comparison standard were performed in three loops, each loop had two series (one ascending and one descending). In each series, ten points were measured, from 10 MPa up to 100 MPa (in 10 MPa increments). A total of 60 measurements were done for the complete comparison in each laboratory.
d) After finishing the corresponding readings, each participating laboratory sent to the pilot laboratory, the complete data file report of the measurements, including the associated uncertainty.
e) The technical staff of each participating laboratory performed the measurements and it was their sole responsibility to fulfil the requirements of the agreed regulating documents of this comparison.

## 3. Participating laboratories' standards

All participating laboratories used piston gages as their standard for this comparison.

## 4. Results

The results of the measurements made by the participating laboratories were entered into the data file provided for the comparison [7] and sent to the coordinating laboratory.
The uncertainties calculated by each laboratory were based mainly on three contributing elements: the standard used by the laboratory, repeatability and resolution of the comparison standard (instrument). Each laboratory applied all necessary corrections to the measured pressure and included the effect of influence quantities into the uncertainty evaluation.

Graph 1 shows the calibration results obtained at PTB and at CENAM in terms of the difference between the pressures indicated by the transfer standard and those measured by the laboratory standards, i.e. in terms of the error of the instrument. The values presented are the mean values of the two calibrations performed by PTB and the mean values of the three calibrations by CENAM. The general mean value of the 5 calibrations performed by the two primary laboratories is included to demonstrate the agreement between them (general average). The combined ISO standard uncertainty is used. The mean values at the calibration pressures and their combined uncertainties define the reference values for the comparison CENAM-PTB.

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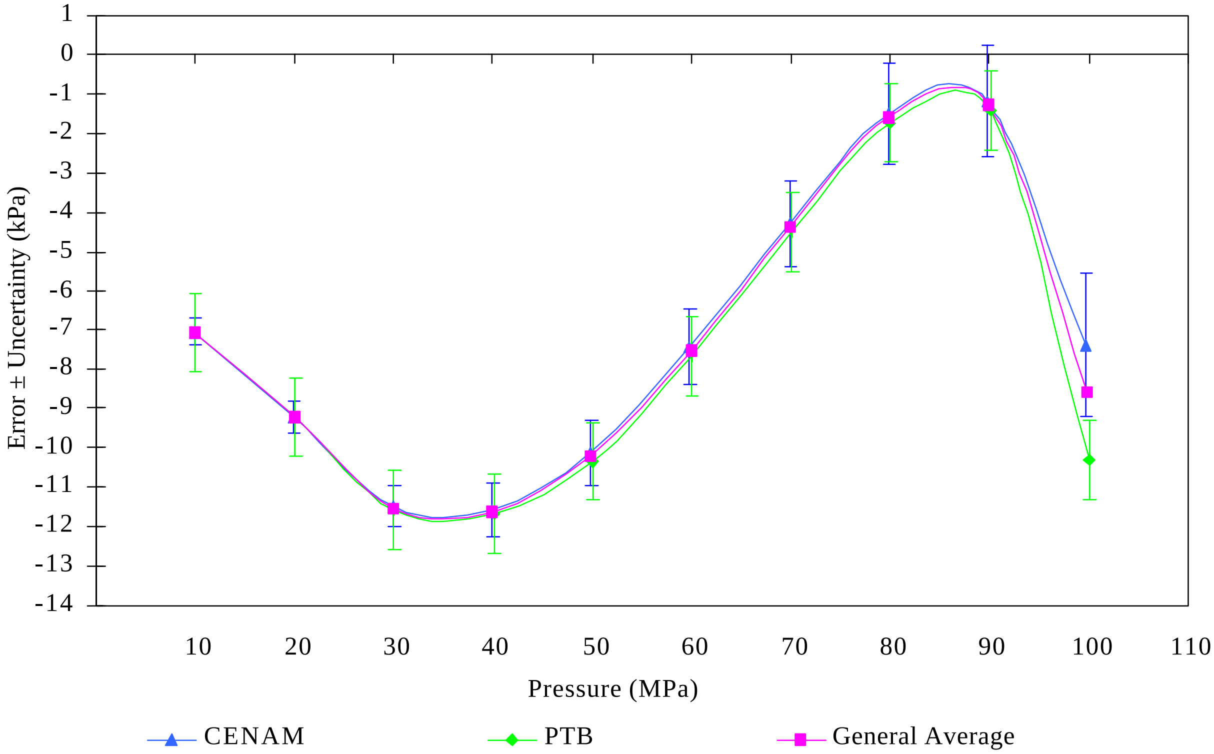
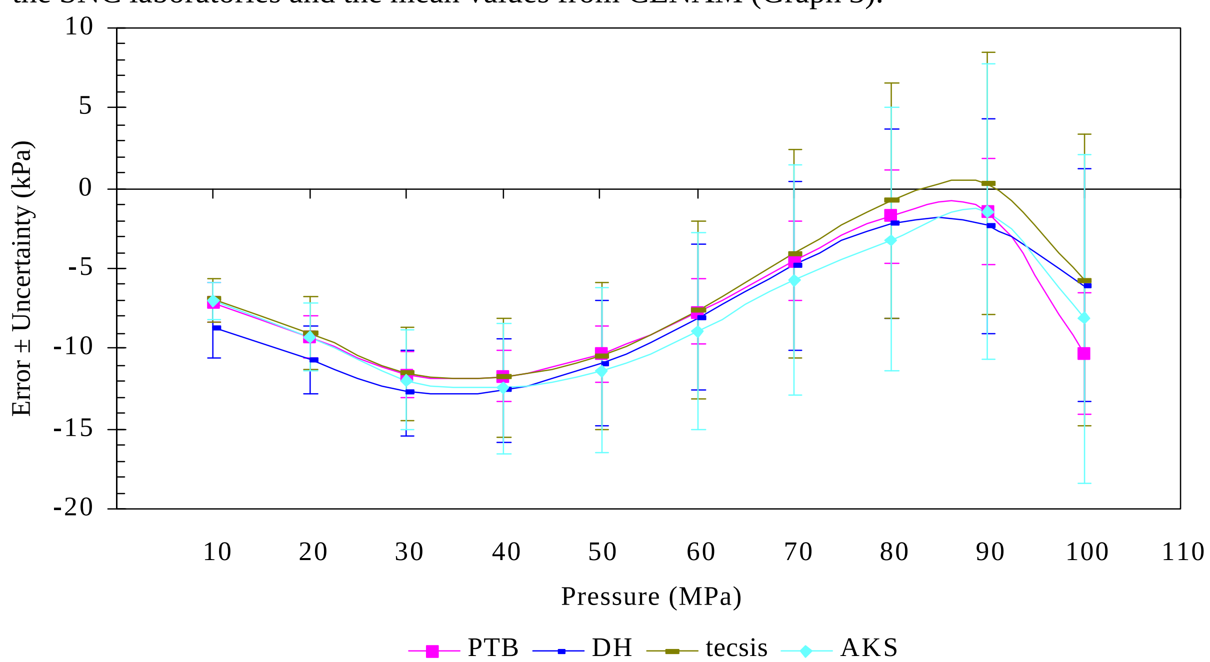



Graph 1. Results of measurements made by PTB and CENAM. The least squares best-fit lines have been superimposed over each laboratory's measurement results.

The following two graphs present the results of the two comparison rounds as described in 2.4.: in Germany by the DKD laboratories and the mean values from the PTB (Graph 2) and in Mexico by the SNC laboratories and the mean values from CENAM (Graph 3).




Graph 2. Results from the laboratories in the first comparison round. PTB's results define the reference values for this round.

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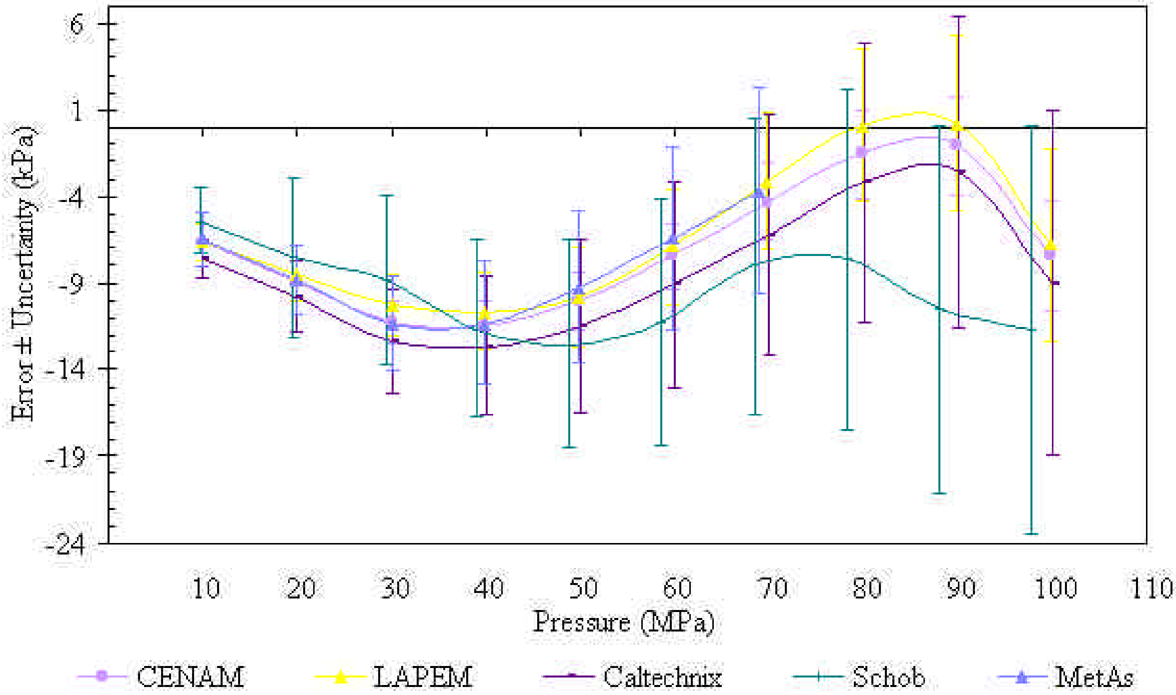
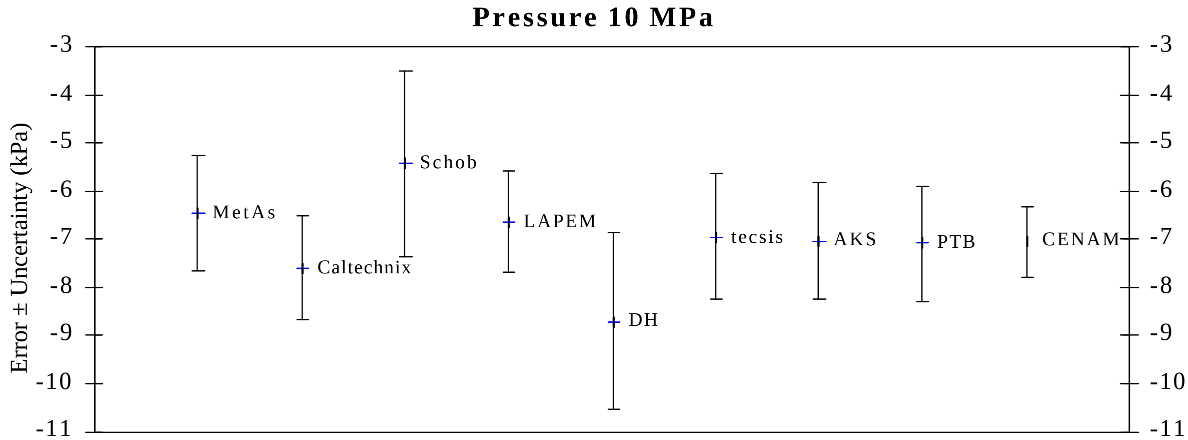
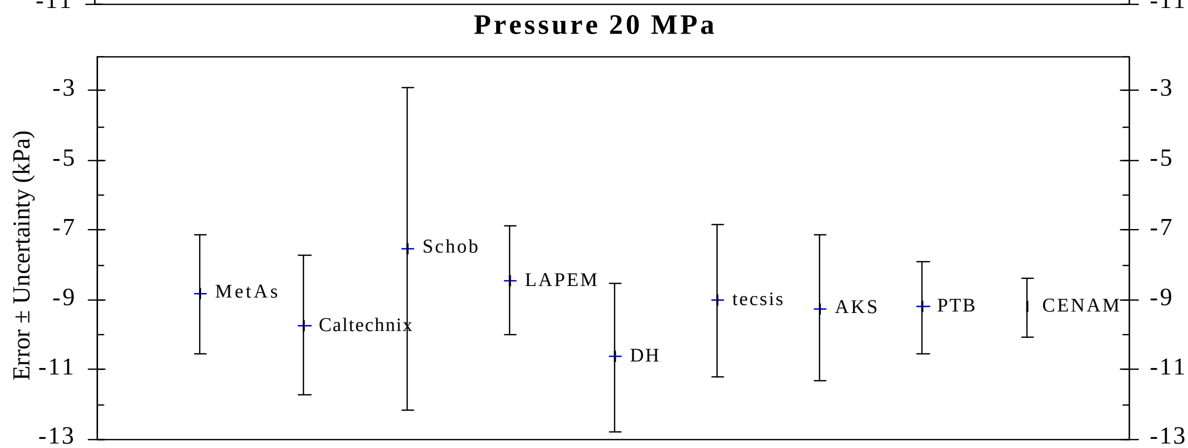
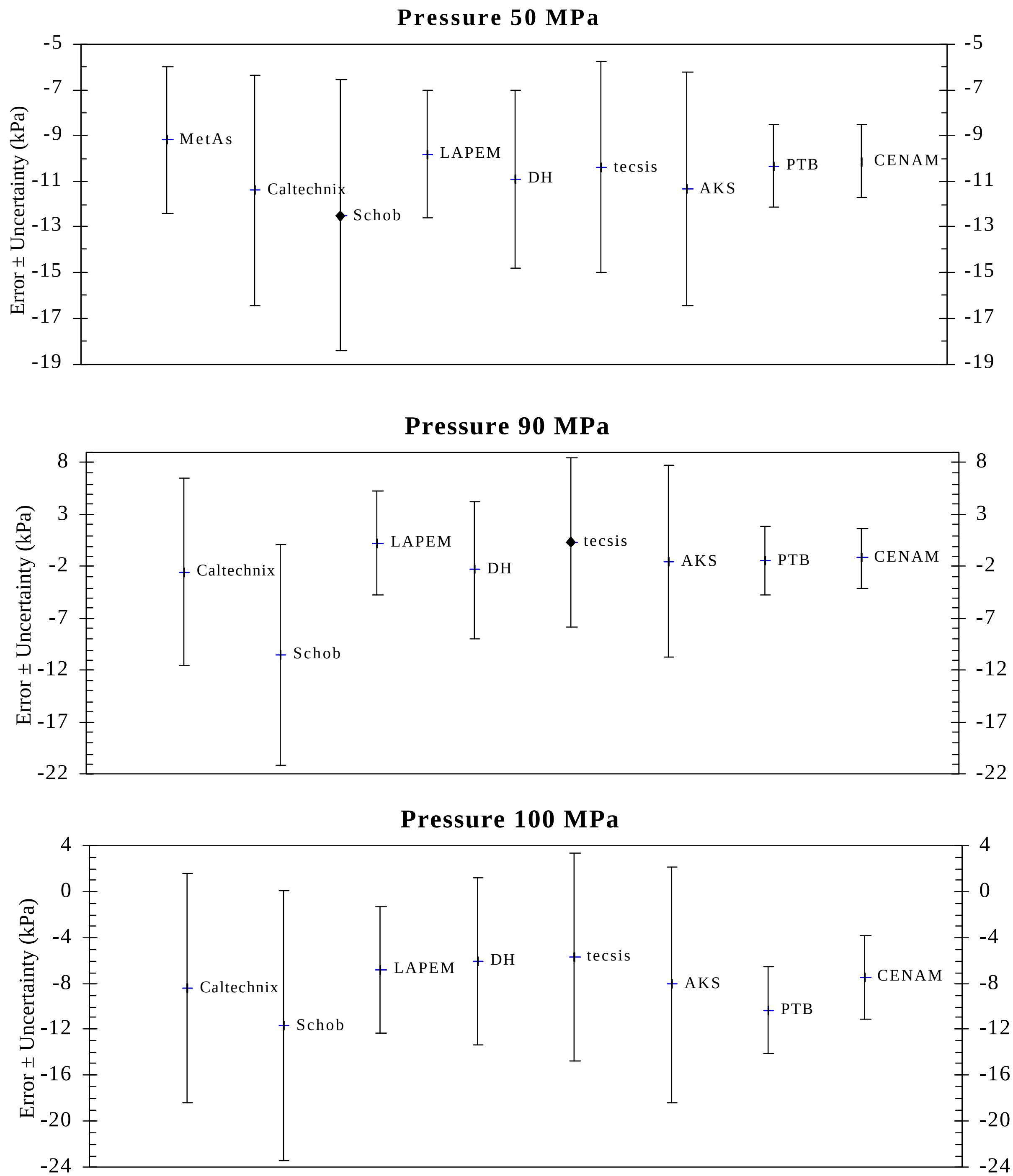



Graph 3. Results from the laboratories in the second comparison round. CENAM's results define the reference values for this round.

Graphs 4.1 to 4.5 present the errors and uncertainties of each laboratory at different pressures.






Graphs 4.1 to 4.5. Errors and expanded uncertainties of all participating laboratories at different calibration pressures.

## 5. Discussion

In order to compare (in a better way) the measurement results from the participating laboratories, the normalized error was calculated for the results of all laboratories using a modified equation of the one described in NORAMET´s document 8 [8] and SEA–2/03 [9].

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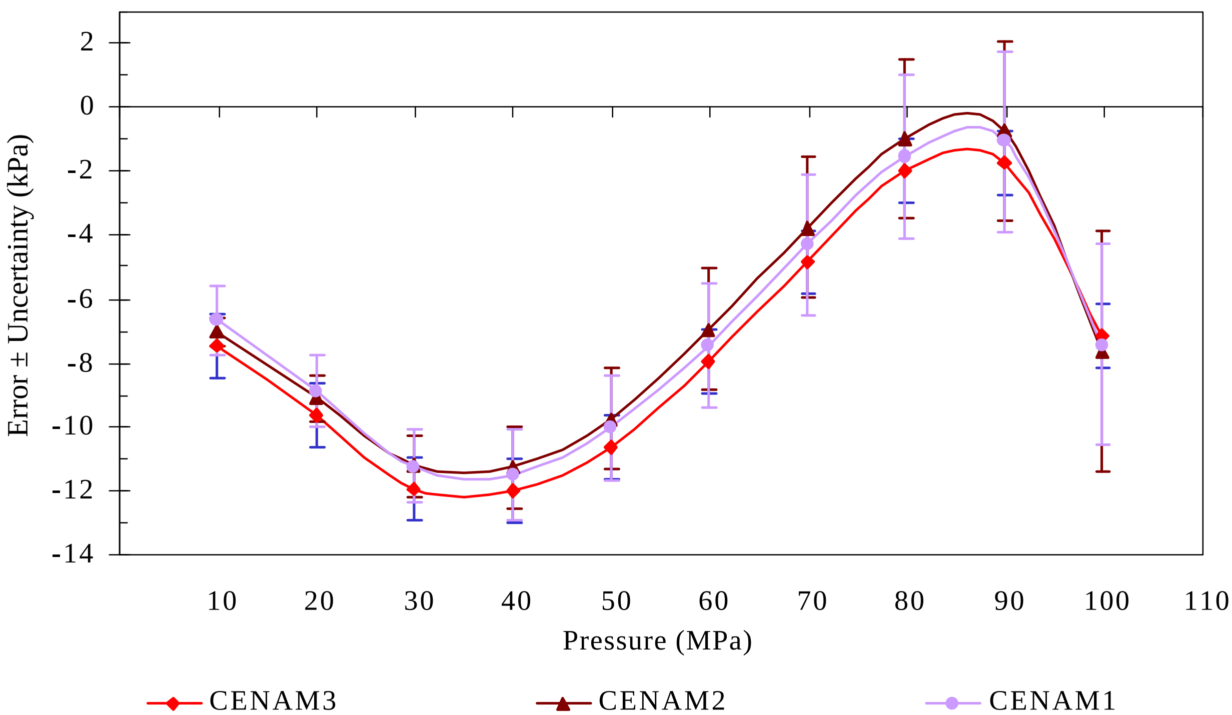
The equation used here (equation 1) takes into account the results from all laboratories and its aim is to compare all laboratories with a general average in one graph. As the real pressure values differ for each laboratory, the estimated error is considered instead of using a pressure lecture. Additionally, the reference values used in the equation are the average error and the combined uncertainty obtained from all the participating laboratories.

$$e_n = \frac{e_{lab} - e_{avg}}{\sqrt{U_{lab}^2 + U_{avg}^2}} \quad (1)$$

Where,
- $e_n$ - normalized error calculated at each calibration pressure
- $e_{lab}$ - laboratory's estimated error
- $e_{avg}$ - average of CENAM and PTB estimated error
- $U_{lab}$ - laboratory's expanded uncertainty
- $U_{avg}$ - CENAM and PTB average expanded uncertainty (see equation 2)

$$U_{avg} = \left[\left(U_{PTB}^2 + U_{CENAM}^2\right)/4 + U_{stability of\ transf.\ std.}^2\right]^{1/2} \quad (2)$$

CENAM made a stability study of the comparison standard. CENAM's 3 full calibrations made, were analyzed to assess the stability of the instrument and the different measurement results are presented as follows.

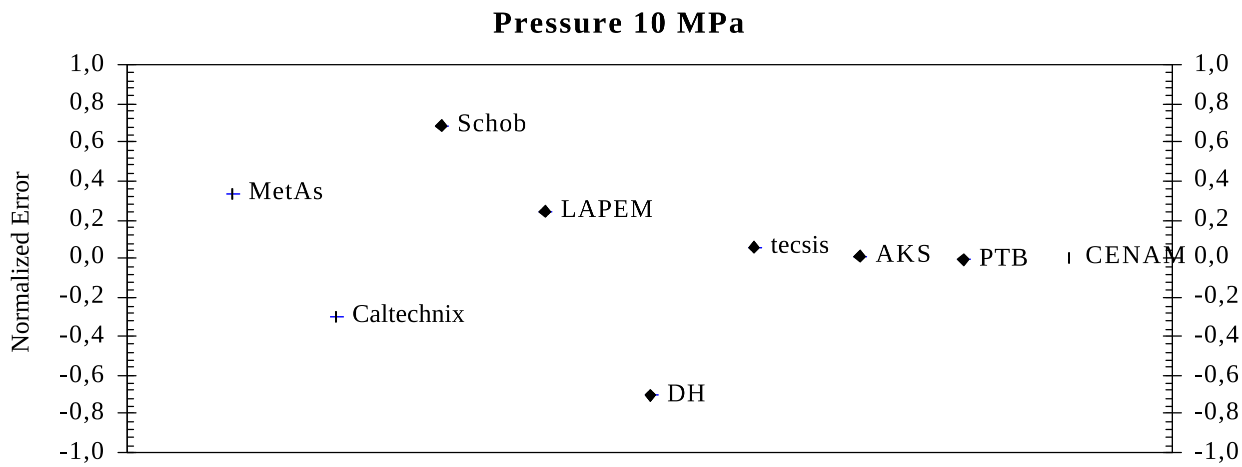


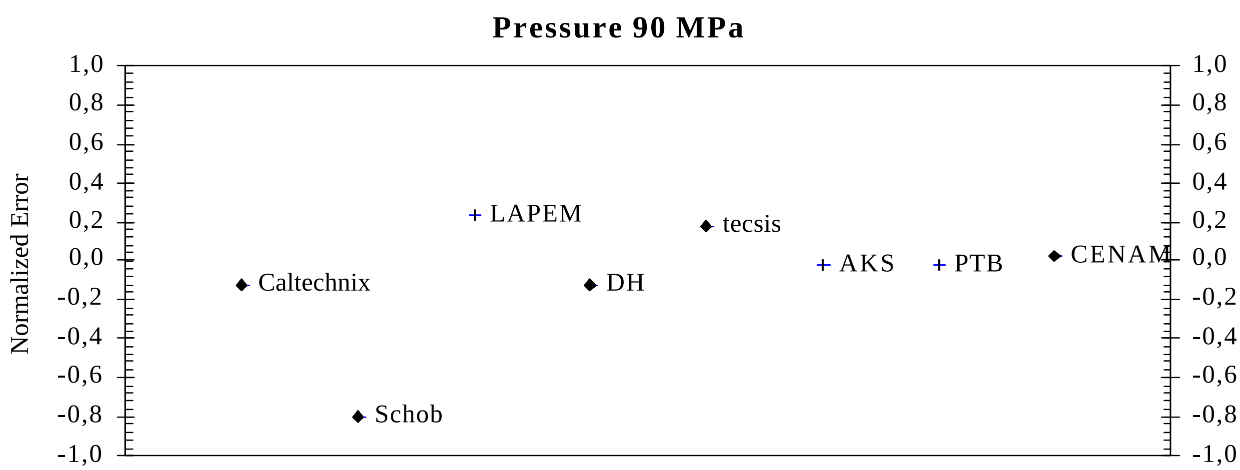

Graph 5. Results from the 3 calibrations of the comparison standard made at CENAM.

A very small time dependant variation on the instrument's response was detected, as it is shown in graph 5. This drift seems to be pressure dependent too. To compensate this effect, a no linear correction would have to be made; as the maximum drift during the complete period of the comparison is less than 30 $10^{-6}$ (relative to the applied pressure) the correction of this effect is unnecessary.

Considering the variability range for the lower pressure point, where 2a is ≤ 1 kPa, the estimated standard deviation is $S_{stability}$ = 0,5 kPa / $(3)^{1/2}$ = 0,29 kPa, from the mean for a pressure of 10 MPa which corresponds to 29 $10^{-6}$ in relative terms.

Graph 6 shows the same error of the comparison for all laboratories (as graph 4) but uses the normalized error equation, only the most illustrative graphs are shown.

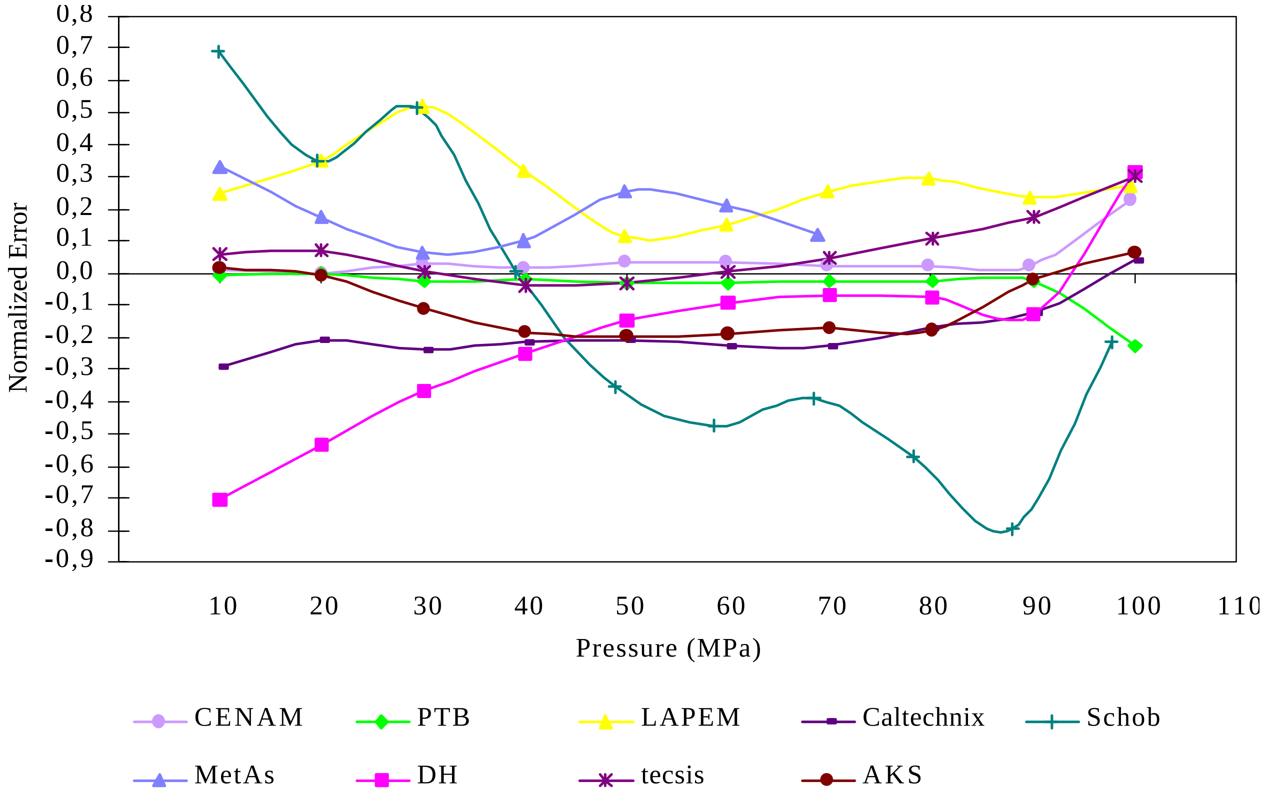



Graph 6. Comparison error, for a given pressure (10 MPa and 90 MPa), of all participating laboratories using the normalized error equation.

Graph 7 presents the normalized error equation graph results for all participating laboratories. It is important to notice that no laboratory obtains values greater than 0,8.

This graph provides a better view of the comparison results and of the equivalence of measurements.




Graph 7. Results of the comparison using the normalized error equation.

## 6. Conclusion

Two national and seven secondary laboratories (PTB, AKS, tecsis, DH, SCHOB, LAPEM, METAS, CALTECHNIX and CENAM) compared their pressure standards by means of an electronic transducer without performing preliminary measurements prior to the reported data.

The transducer is not a typical client's transducer received for calibration and its response was such as to challenge the measurement capability, even of experienced operators. In general, the results demonstrate agreement among the nine laboratories with negligible differences observed.

The normalized error equation employed has been proposed as means of assessing comparability among laboratories. As the comparison was among primary and secondary laboratories, to demonstrate equivalence of measurements the measurements of the secondary laboratories were referred to those of the primary ones by the modification presented in equation 1. The modified normalized error equation was very useful in particular for our case, as there were 7 laboratories which have bigger uncertainties.

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