

Draft B

**SIM-AFRIMETS Hydraulic Gauge Pressure
Comparison, Range from 10 MPa to 500 MPa**

**SIM/AFRIMETS (500 MPa)
CCM.P-K13.1**

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I. ABSTRACT

This report describes the results of a comparison in the range of 500 MPa of hydraulic gauge pressure. The aim of this comparison is to verify the equivalence statements and to provide a link to NMISA for CCM.P-K13. The participants were National Metrology Institute of South Africa (NMISA) from Intra-Africa Metrology System (AFRIMETS) and Centro Nacional de Metrología (CENAM) from Inter-American Metrology System (SIM). The transfer standard (TS) was a complete system including pressure balance base, the piston cylinder assembly and to provide better comparability its set of masses. The Centro Nacional de Metrología (CENAM), Mexico was the pilot laboratory and provided the reference values in this supplementary comparison, SC. The participants completed their measurements and reported the pressure-dependent effective areas of the transfer standard at specified pressures with their associated uncertainties.

II. INTRODUCTION

This comparison aimed to obtain the equivalence statements and link to NMISA for the KC CCM.P-K13, in the range from 10 MPa to 500 MPa, in hydraulic gauge pressure. This comparison will provide the means to the laboratory to support their uncertainty statements given in its CMCs table. The participants were National Metrology Institute of South Africa (NMISA) from Intra-Africa Metrology System (AFRIMETS) and Centro Nacional de Metrología (CENAM) from Inter-American Metrology System (SIM). Centro Nacional de Metrología (CENAM), Mexico, was the pilot laboratory and provided the reference values in this comparison. The technical protocol specified the procedures followed for the comparison and was prepared in accordance with the guidelines for CIPM supplementary comparisons.

III. COMPARISON PURPOSE

This comparison, CCM.P-K13.1, is to confirm the measurement and calibration capabilities (CMCs) for hydraulic gauge pressure in the range from 10 MPa to 500 MPa of NMISA; also, it allows setting the level of agreement and providing a link to the CIPM key comparison CCM.P-K13 to NMISA in the range from 100 MPa to 500 MPa.

IV. PARTICIPATING LABORATORIES

Table 1 shows the two participating laboratories with their delivery addresses, as well as the names of the contact persons.

Table 1. Participating laboratories.

Participating laboratory name and TS delivery address	Contact person
Centro Nacional de Metrología, CENAM (pilot laboratory) Pressure and Vacuum Group km 4.5 Carretera a los Cues 76241 El Marques Queretaro. Mexico	Comparison coordinator: Dr. Jorge C. Torres-Guzman / Mr. Francisco Flores-Martinez Phone: +52 442 211 0500 ext. 3741 Fax: +52 442 211 0578 E-mail: jorge.torres@cenam.mx
National Metrology Institute of South Africa, NMISA Pressure Laboratory Building No.5 CSIR Campus Meiring Naude Drive Brummeria, Pretoria South Africa Postal Code: 0040	Mr. Thulani Khoza Phone: +2712 841 2399 E-mail: tkhoza@nmisa.org

V. TRANSFER STANDARD AND PARTICIPATING LABORATORIES STANDARDS USED

The transfer standard is a piston-cylinder assembly of 2 mm² nominal effective area with serial number 2279, which was sent with a pressure balance equipped with a mass carrier and mass set. The manufacturer of all the equipment is Fluke Calibration, USA.

The mass pieces of the system included in the TS, to generate the nominal pressures, are given in table 2. The masses of the mass set pieces are given in Annex 1.

Table 2. Mass pieces to be used on the carrying bell of the TS to generate the nominal pressures.

Nominal pressure, MPa	Nominal mass pieces, kg
10	(1)
100	(9 + 10)
200	(9 + 10(1 to 3))
250	(9 + 10(1 to 4))
350	(9 + 10(1 to 6))
500	(9 + 10(1 to 8))

The standards used by the participating laboratories for the calibration of the transfer standard of the comparison are shown in table 3.

Table 3. Participating laboratories standards used for the comparison.

Country	Mexico	South Africa
NMI	CENAM	NMISA
Contact	Jorge Torres Guzman jtorres@cenam.mx	Thulani Khoza tkhoza@nmisa.org
Fluid	di(2-ethylhexyl) sebacate (DHS)	di(2-ethylhexyl) sebacate (DHS)
Standard used	Pressure Balance	Pressure Balance
Maker	DH Budenberg	FLUKE
Model	5316	PG7302
Serial N°	Base: 6367 Piston: 4923	Base: 1116; Piston: 2237
Range	0.4 MPa to 500 MPa	5 MPa to 500 MPa
Accuracy Class	0.005 %R	0.005 %R
Cylinder Material	Tungsten carbide	Tungsten carbide
Piston Material	Steel	Tungsten carbide
Effective area (A_0) at zero pressure in m^2	1.961 360 E-6	1.960 23 E-6
Relative Expanded Uncertainty of A_0 , in E-6	41	56
Elastic deformation coefficient b , in Pa^{-1}	7.89 E-13	7.29 E-13
Expanded Uncertainty of b , in Pa^{-1}	2.7 E-14	7.29 E-14
Traceability	CENAM	NMISA

VI. MEASUREMENTS PROCEDURE

The measuring method was cross float between the TS and the laboratory standard. The transfer standard and the piston-cylinder assembly were mounted in accordance with the instructions given in the user's manual pressure balance, FLUKE model PG7302. The comparison procedure was approved by the participant NMIs and included in a document named: SIM-AFRIMETS comparison for 500 MPa range of hydraulic gauge pressure - technical protocol (500 MPa).

The most important information is:

- The reference temperature of the comparison was 20 °C.
- The time between a comparison target pressure level change and the acquisition of data, for the cross-floating equilibrium of the laboratory standard and the TS, was no less than 10 minutes.
- The direction of the piston rotation was clockwise. The rotation, at the equilibrium between the reference standard and TS, was approximately 32 rpm.
- The time between a pressure level change and the acquisition of the data corresponding to the equilibrium of the laboratory standard and TS should be not shorter than 5 minutes.
- The measurements included five cycles, each with nominal pressures in the following order (10, 100, 200, 250, 350, 500, 500, 350, 250, 200, 100, 10) MPa. In total 60 measurements.
- The generated pressures should not deviate from these nominal values by more than 0.1 MPa.

g) To wait at least 15 minutes between one reading and the next consecutive one at 500 MPa and between two measurement cycles.

VII. COMPARISON ROUND

Table 4 presents the work program for the participating laboratories comparison round. During the comparison, no major delay or anomaly happened to the TS or in any laboratory site.

Table 4. Participating laboratories comparison round.

Date	Date	Institute
2021-01-19	2021-01-26	Initial measurements at NMISA
2021-02-26	2021-03-26	Transportation of the TS / sending of results
2021-03-31	2021-05-04	Measurements at CENAM (3 calibrations)
2021-06-17	2021-07-30	Transportation of the TS
2021-10-08	2021-11-12	Final measurements at NMISA
2021-11-12	2021-12-20	Sending of results
2022-01-10	2022-04-26	Draft report of the comparison, CENAM

VIII. RESULTS

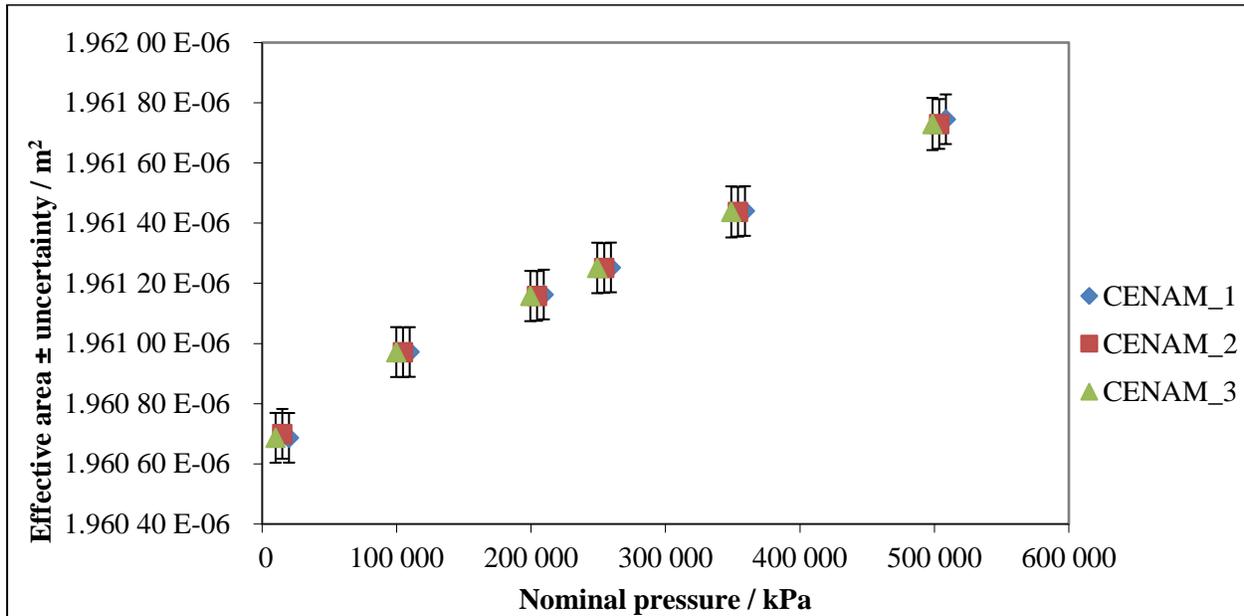
The pilot laboratory checked, during the period of six weeks, the TS for drift. The three calibrations made by CENAM to the TS are shown in table 5 and graph 1.

In table 6, it can be seeing that the TS has very small drift, over a period of 27 days. Also, the worst reproducibility calculated for the TS on the 3 calibrations made by CENAM was $2.6 \cdot E-12 \text{ m}^2$ (at 500 MPa). The combined uncertainty of the reproducibility with the area uncertainty was the uncertainty used by CENAM for this comparison.

As shown in graph 2, CENAM used the mean result of the 3 calibrations performed to the TS as its data for the comparison; the uncertainty includes the worst reproducibility as mentioned above.

Table 5. Calibrations made by CENAM to the TS.

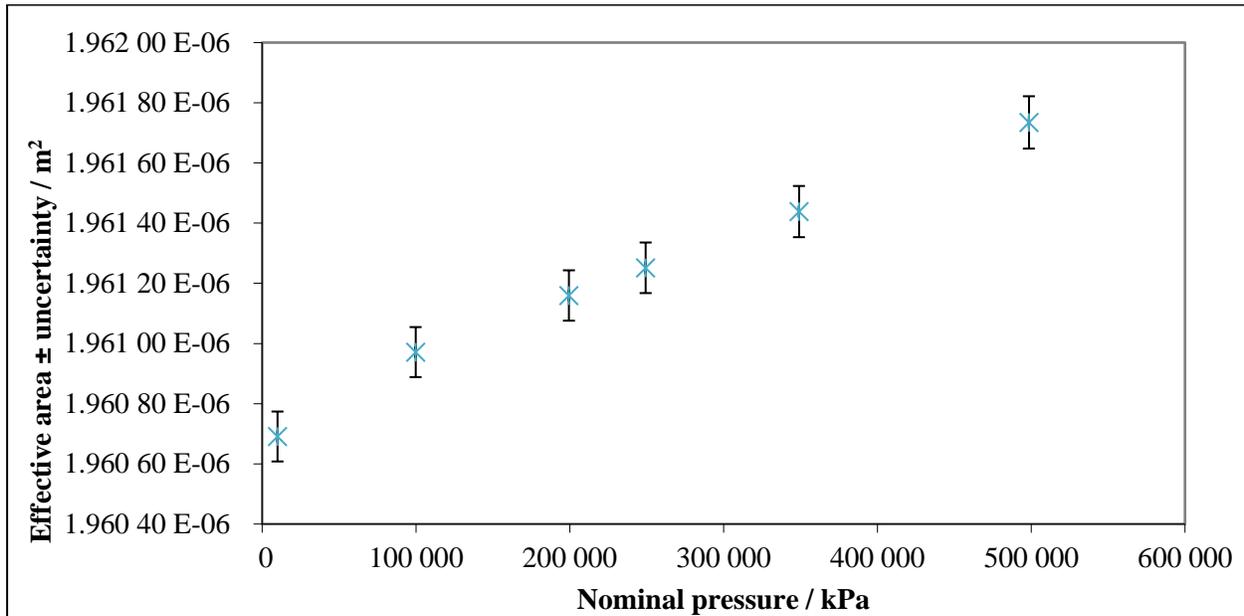
Nominal Pressure MPa	CENAM_1		CENAM_2		CENAM_3	
	2021/03/31		2021/04/13		2021/04/27	
	Area TS m^2	Uncertainty Area m^2	Area TS m^2	Uncertainty Area m^2	Area TS m^2	Uncertainty Area m^2
10	1.960 686 E-06	8.3 E-11	1.960 700 E-06	8.3 E-11	1.960 687 E-06	8.3 E-11
100	1.960 972 E-06	8.3 E-11	1.960 971 E-06	8.3 E-11	1.960 971 E-06	8.3 E-11
200	1.961 163 E-06	8.3 E-11	1.961 158 E-06	8.3 E-11	1.961 158 E-06	8.3 E-11
250	1.961 253 E-06	8.4 E-11	1.961 251 E-06	8.4 E-11	1.961 251 E-06	8.4 E-11
350	1.961 440 E-06	8.5 E-11	1.961 438 E-06	8.5 E-11	1.961 437 E-06	8.5 E-11
500	1.961 745 E-06	8.7 E-11	1.961 729 E-06	8.7 E-11	1.961 729 E-06	8.7 E-11



Graph 1. TS effective area and uncertainty as obtained by CENAM. Three calibrations of the TS.

Table 6. Drift (for 27 days) and reproducibility of the calibrations made by CENAM to the TS.

Nominal Pressure	Drift Over 27 days	Reproducibility 3 calibrations
MPa	m ²	m ²
10	2.2 E-14	2.2 E-12
100	-2.1 E-13	1.0 E-13
200	-5.0 E-12	7.9 E-13
250	-1.7 E-12	3.0 E-13
350	-3.0 E-12	4.6 E-13
500	-1.6 E-11	2.6 E-12

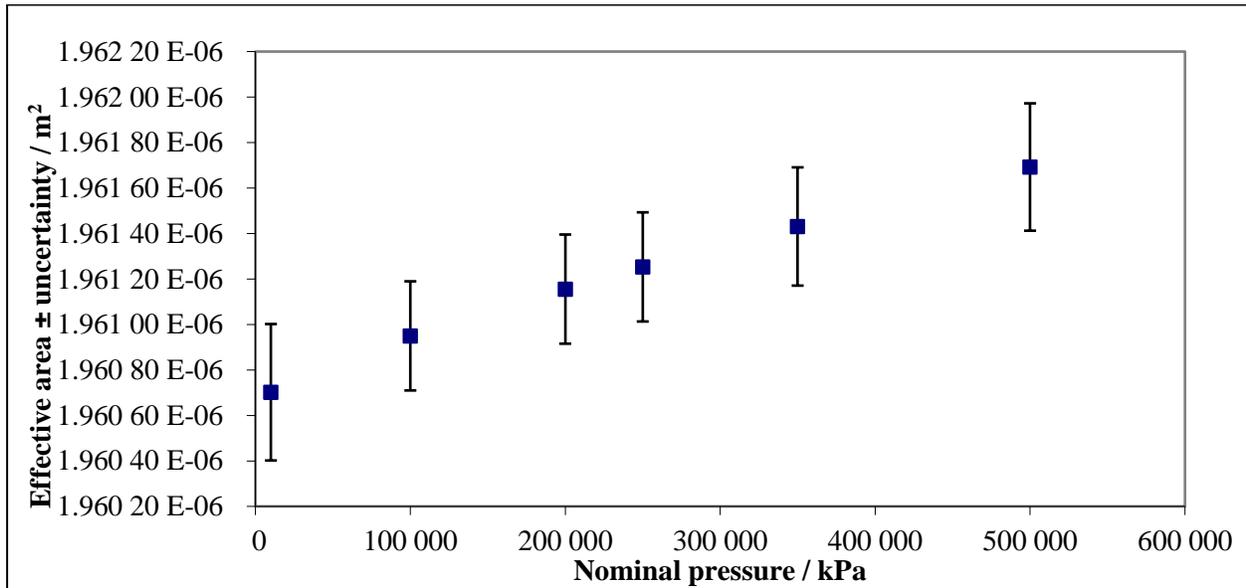


Graph 2. Mean TS effective area and combined uncertainty used by CENAM for this comparison.

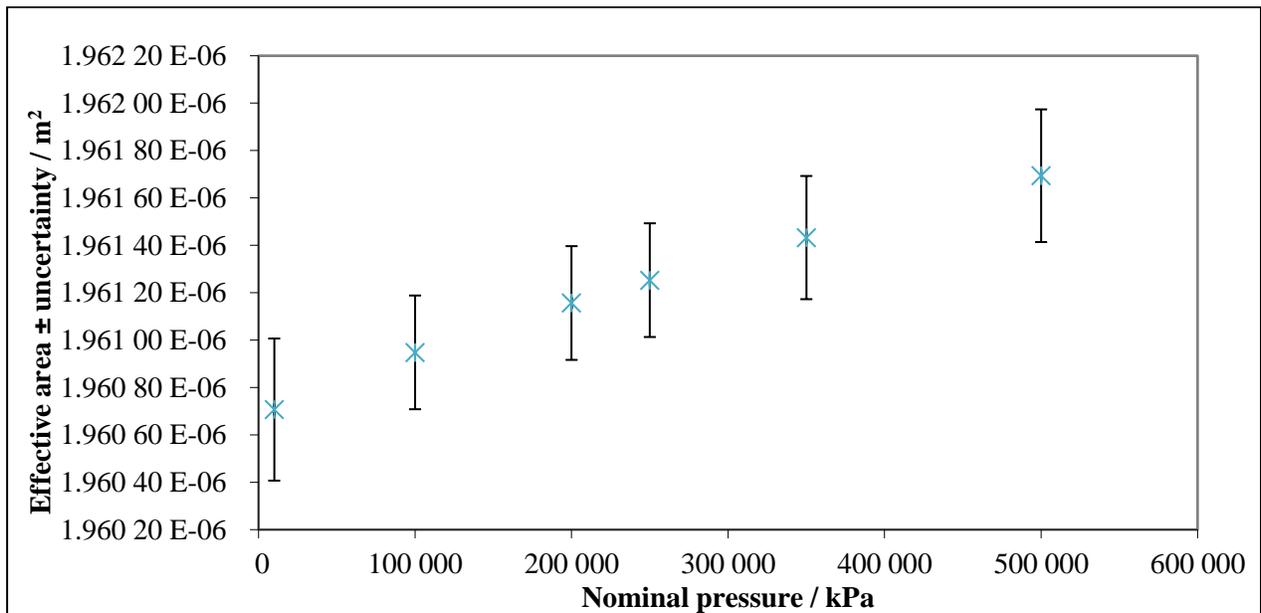
Table 7 and graphs 3 to 4 show the 2 calibrations of the TS made by NMISA. The TS showed no drift, as well as no major dispersion. As shown in graph 5, NMISA used for the comparison the mean of its 2 calibrations performed.

Table 7. Calibrations made by NMISA to the TS.

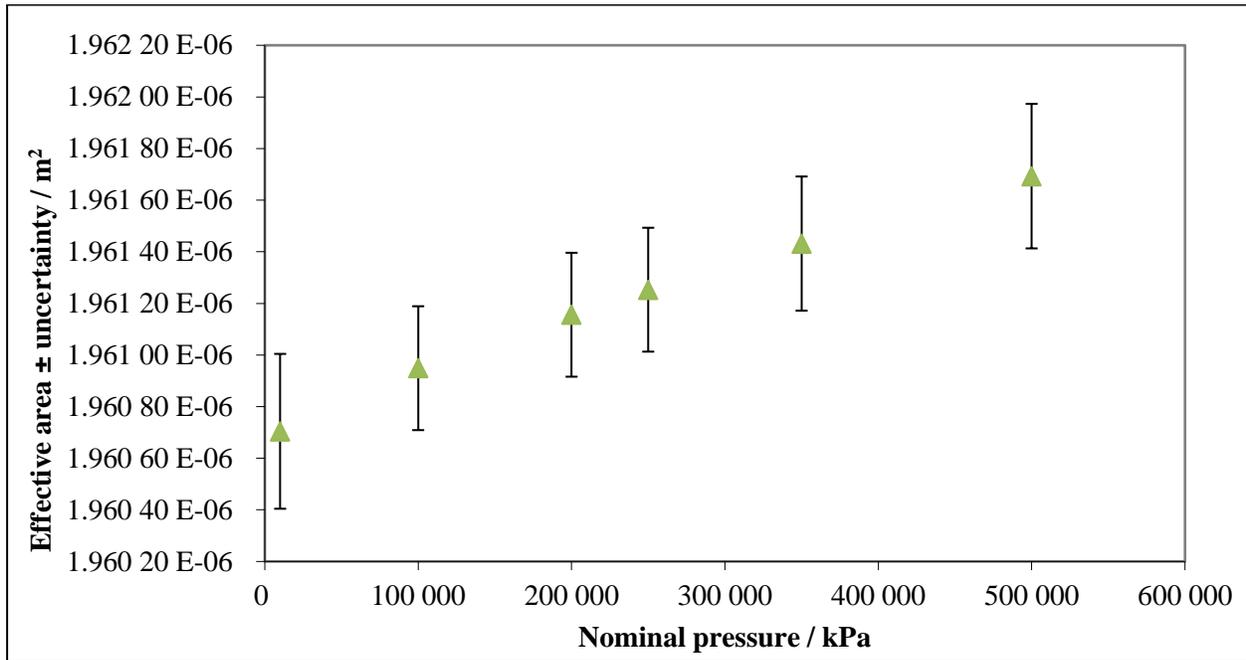
Nominal Pressure MPa	NMISA_1 Initial calibration 19/01/2021		NMISA_2 Final calibration 08/10/2021		Long-term drift 262 days m ²
	Area TS m ²	Uncertainty Area m ²	Area TS m ²	Uncertainty Area m ²	
	10	1.960 702 E-06	3.0 E-10	1.960 707 E-06	
100	1.960 950 E-06	2.4 E-10	1.960 948 E-06	2.4 E-10	-1.5 E-12
200	1.961 155 E-06	2.4 E-10	1.961 157 E-06	2.4 E-10	1.0 E-12
250	1.961 253 E-06	2.4 E-10	1.961 253 E-06	2.4 E-10	-4.4 E-14
350	1.961 431 E-06	2.6 E-10	1.961 433 E-06	2.6 E-10	1.8 E-12
500	1.961 692 E-06	2.8 E-10	1.961 694 E-06	2.8 E-10	1.5 E-12



Graph 3. TS effective area and uncertainty as obtained by NMISA. Initial calibration.

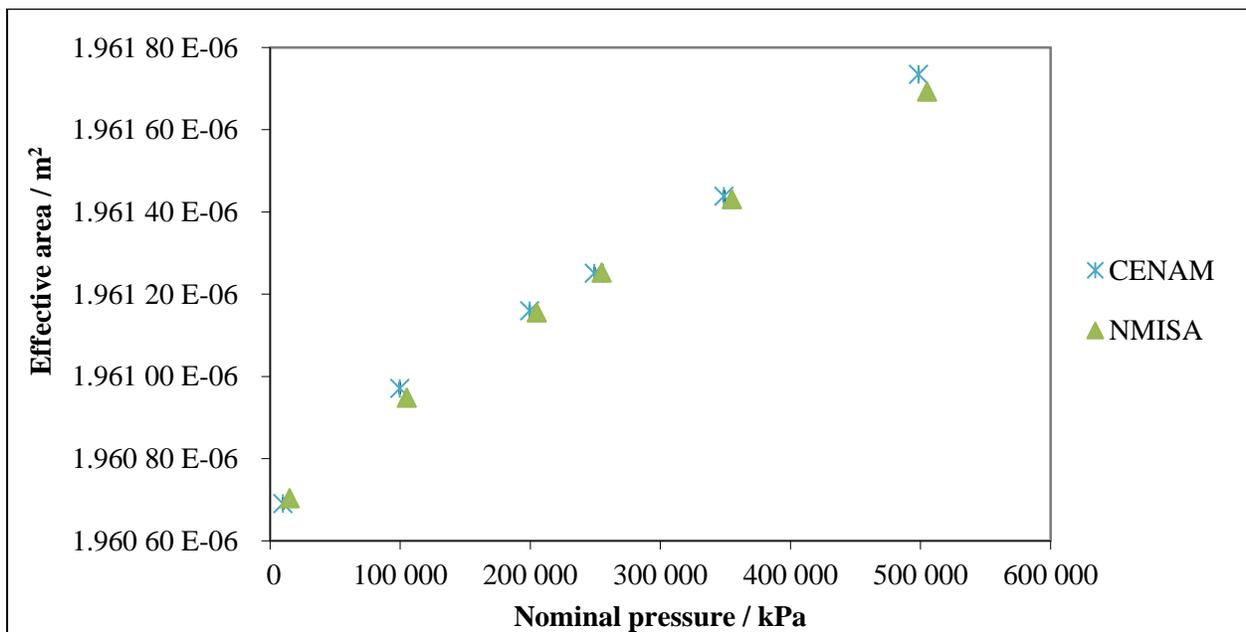


Graph 4. TS effective area and uncertainty as obtained by NMISA. Final calibration.



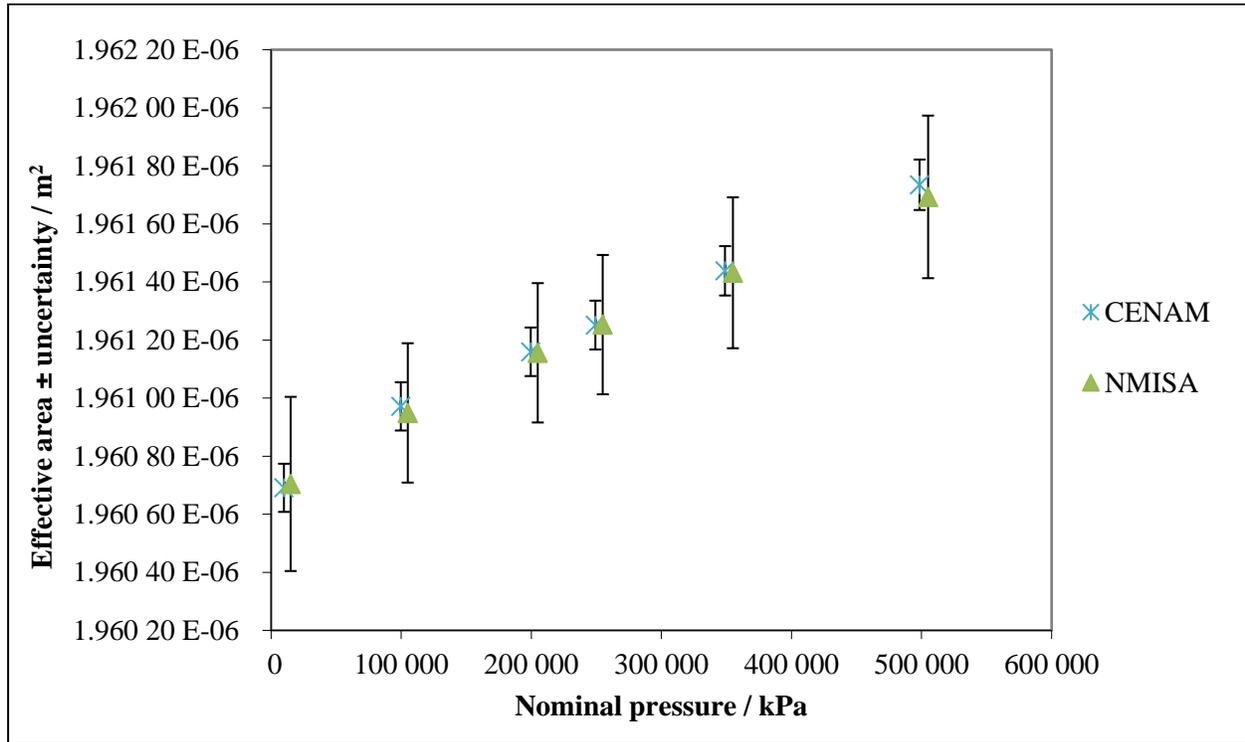
Graph 5. Mean TS effective area and uncertainty reported by NMISA.

Graph 6 shows the results of the 2 laboratories for the TS effective area as obtained by each NMI.



Graph 6. TS effective area as obtained by each NMI.

Graph 7 presents the effective area of the TS and its corresponding expanded uncertainty for each NMI. The values obtained by each participating laboratory are included in table 8, effective area and its corresponding uncertainty.

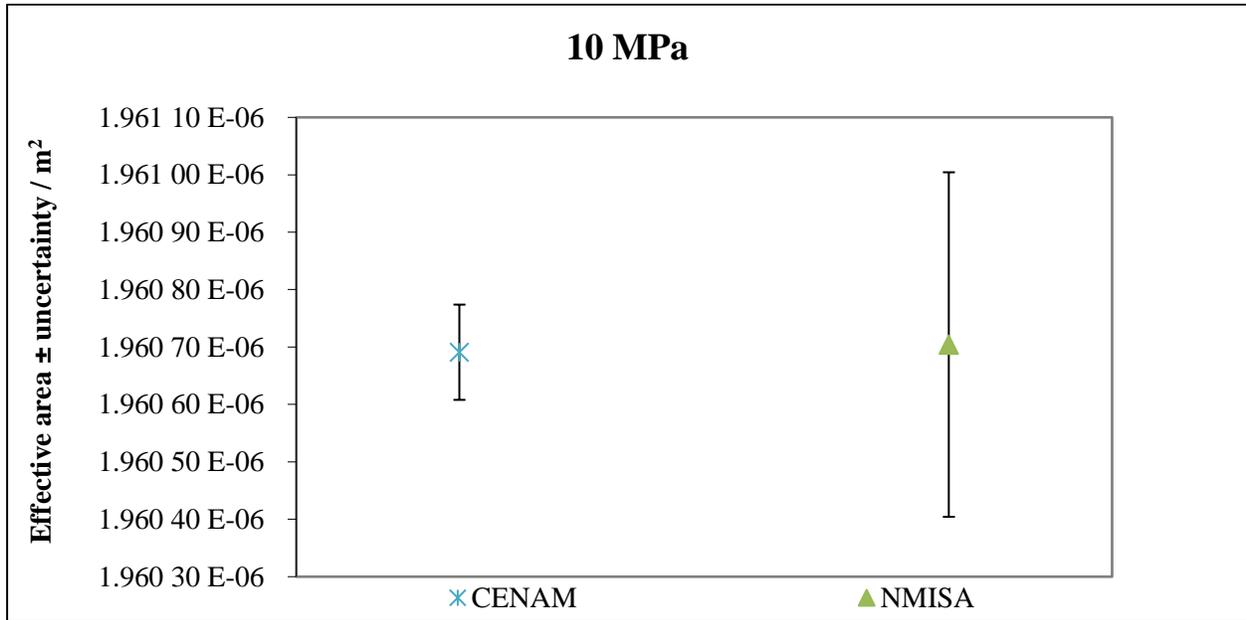


Graph 7. TS effective area and its uncertainty as obtained by each NMI.

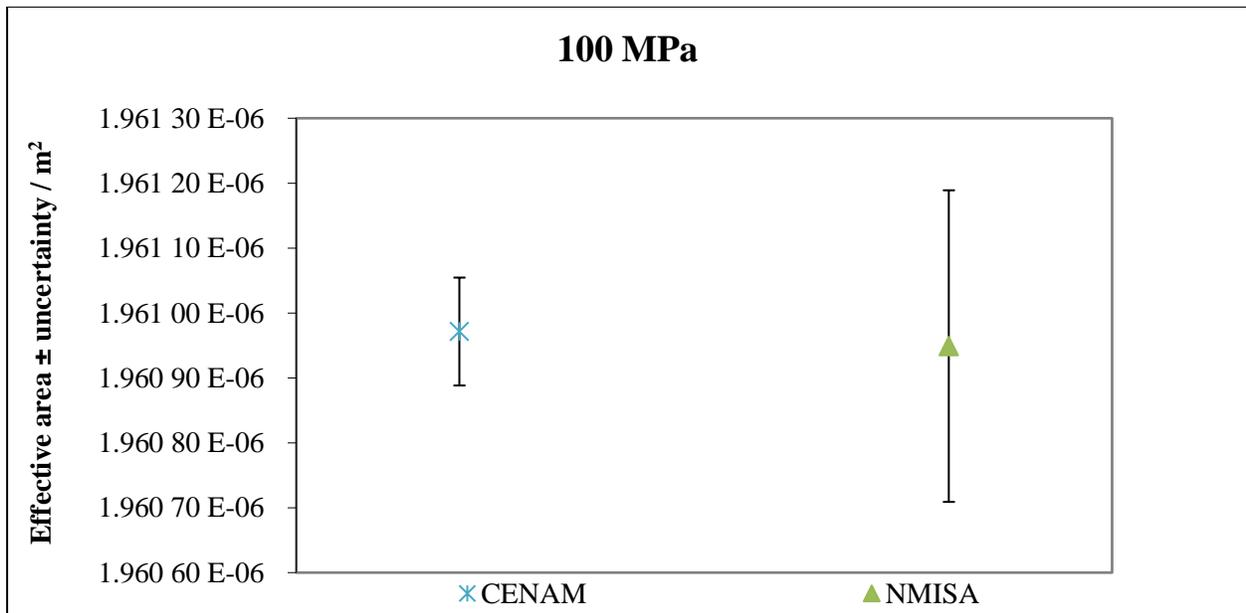
Table 8. TS effective area and its uncertainty as obtained by each NMI, m².

Nominal Pressure	CENAM		NMISA	
	Area	<i>U</i>	Area	<i>U</i>
MPa	m ²	m ²	m ²	m ²
10	1.960 691 E-06	8.3 E-11	1.960 70 E-06	3.0 E-10
100	1.960 971 E-06	8.3 E-11	1.960 95 E-06	2.4 E-10
200	1.961 159 E-06	8.3 E-11	1.961 16 E-06	2.4 E-10
250	1.961 252 E-06	8.4 E-11	1.961 25 E-06	2.4 E-10
350	1.961 438 E-06	8.5 E-11	1.961 43 E-06	2.6 E-10
500	1.961 735 E-06	8.7 E-11	1.961 69 E-06	2.8 E-10

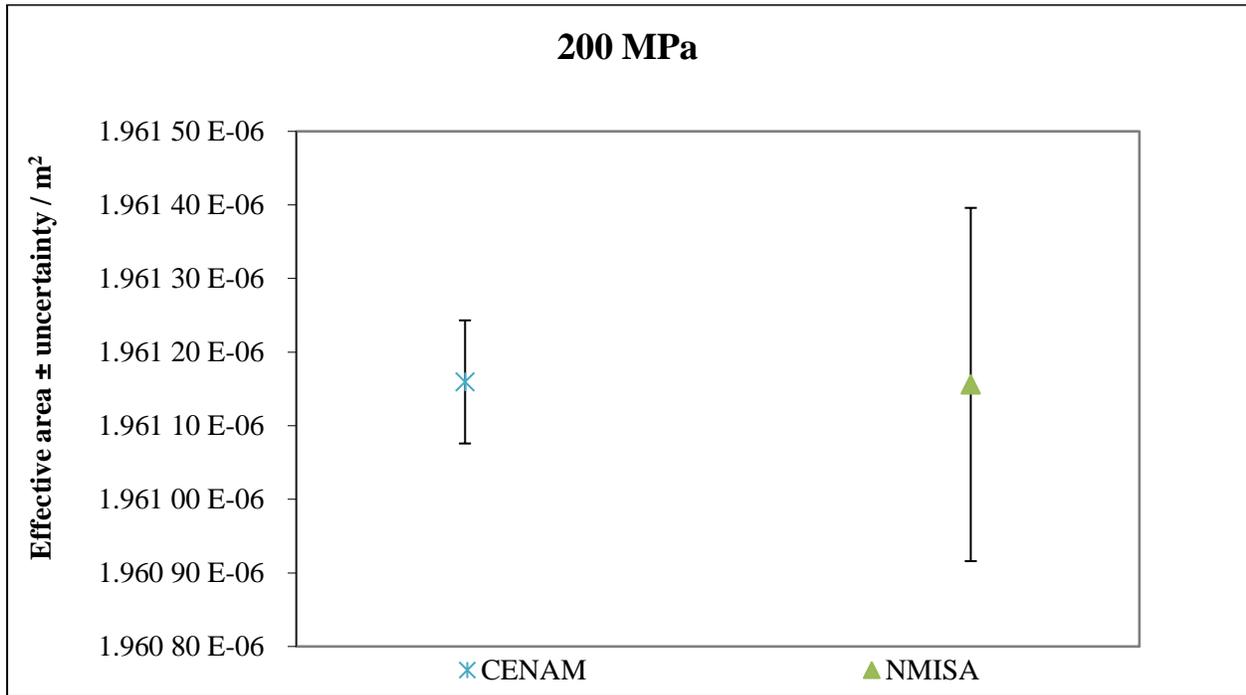
Graphs 8 to 13, show the effective area and its corresponding expanded uncertainty as calculated by each NMI for each applied pressure.



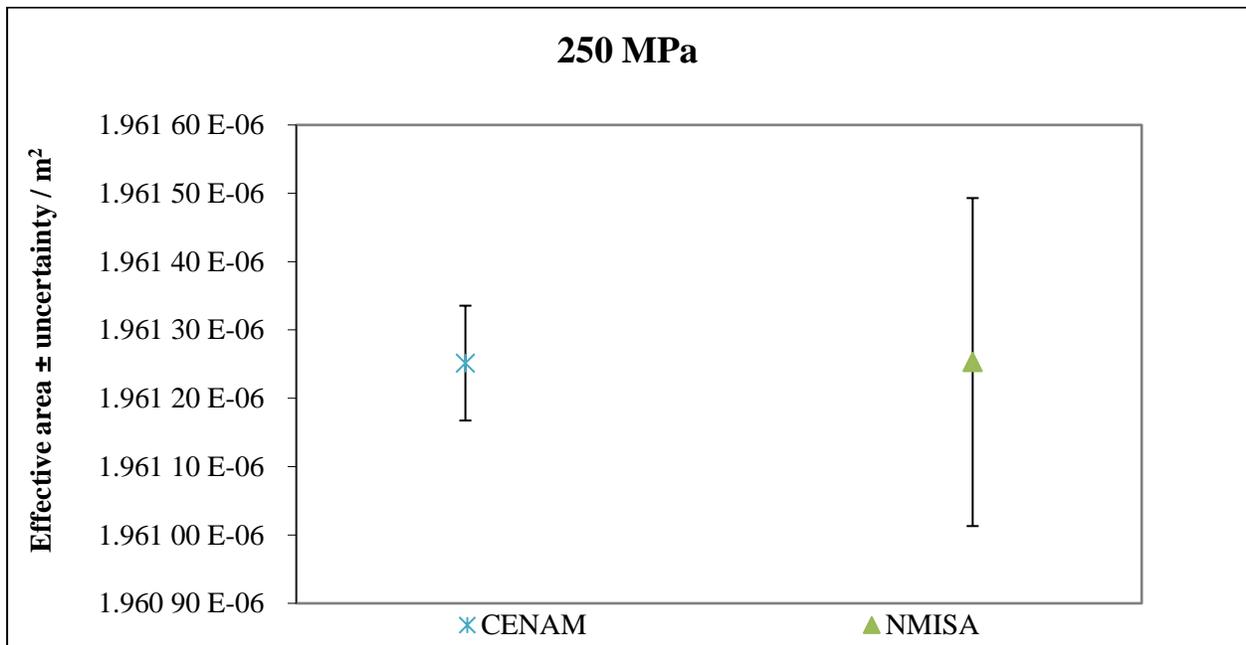
Graph 8. TS effective area and its expanded uncertainty as obtained by each NMI, for 10 MPa.



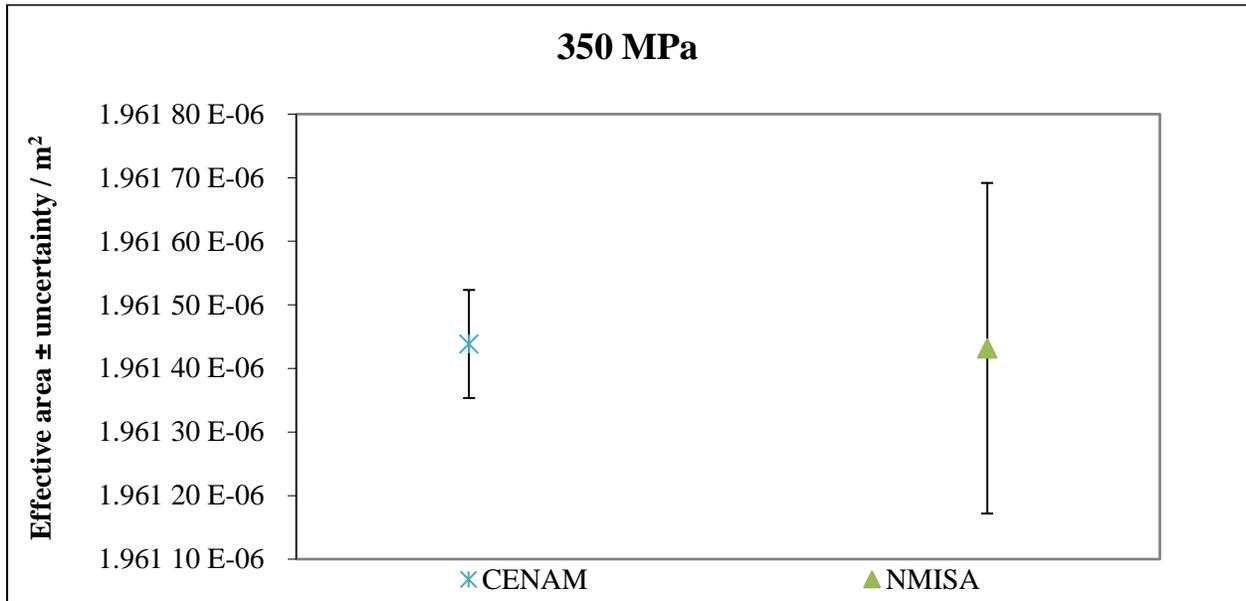
Graph 9. TS effective area and its expanded uncertainty as obtained by each NMI, for 100 MPa.



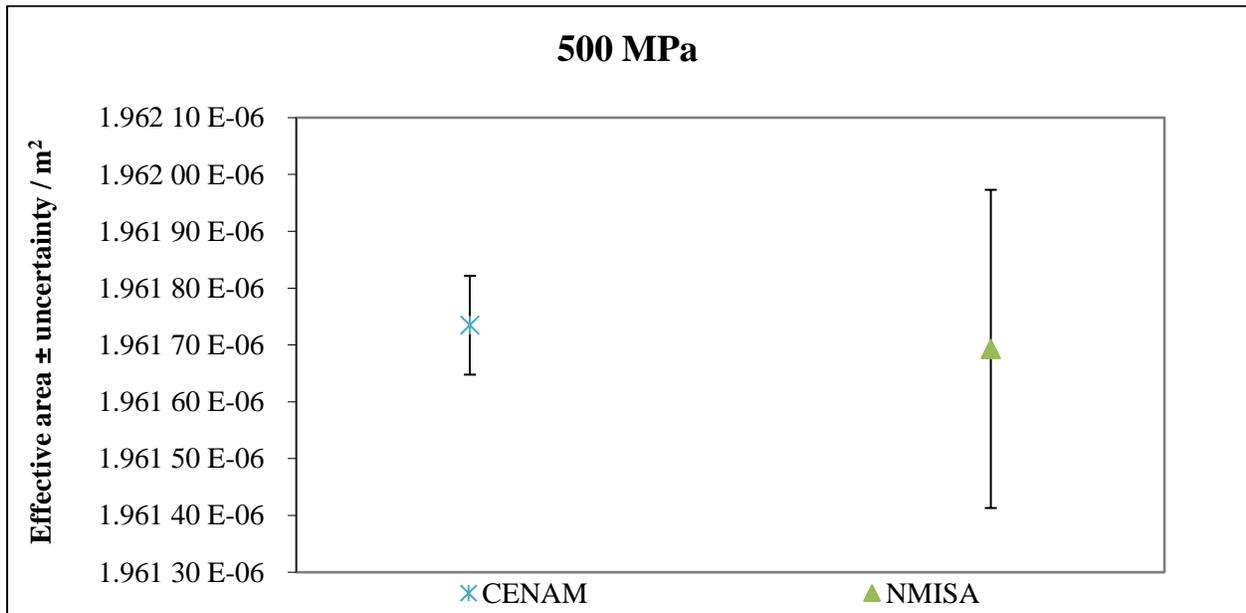
Graph 10. TS effective area and its expanded uncertainty as obtained by each NMI, for 200 MPa.



Graph 11. TS effective area and its expanded uncertainty as obtained by each NMI, for 250 MPa.



Graph 12. TS effective area and its expanded uncertainty as obtained by each NMI, for 350 MPa.

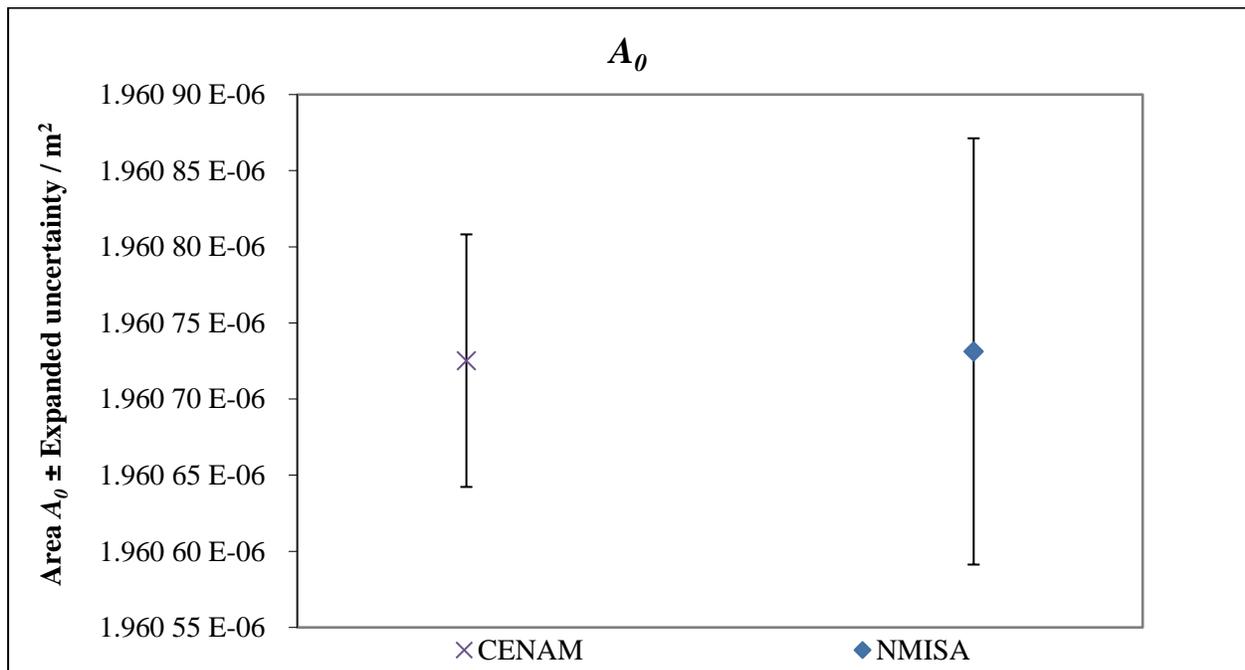


Graph 13. TS effective area and its expanded uncertainty as obtained by each NMI, for 500 MPa.

Table 9 and graph 14 present the results for A_0 and its corresponding expanded uncertainty for each participating NMI. To compare results CENAM made the calculations by means of the lineal regression method.

Table 9. TS A_0 and b its corresponding expanded uncertainty as obtained by each NMI.

	A_0 / m^2	U_{A_0} / m^2	$b / 1/\text{Pa}$	$U_b / 1/\text{Pa}$
CENAM	1.960 725 E-06	9.6 E-11	1.046 E-12	3.8 E-14
NMISA	1.960 73 E-06	1.4 E-10	1.011 E-12	1.01 E-13



Graph 14. TS A_0 and its corresponding expanded uncertainty as obtained by each NMI.

IX. EVALUATION OF RESULTS

For evaluation of the NMISA performance and the equivalence of the NMISA and CENAM results, the normalized error equation (E_n) criterium was applied to their TS *effective area* results. The purpose of this SC is to produce equivalence statements and a link of NMISA to CCM.P-K13.

IX.1 Direct results compatibility for this Comparison, CCM.P-K13.1.

The normalized error equation (E_n) criterium can be applied to the TS effective area results according to the following equation (1).

$$E_n = \frac{\bar{a} - \bar{A}}{\sqrt{U_{NMISA}^2 + U_{CENAM}^2}} \quad (1)$$

Where:

E_n : Normalized error with respect to CENAM reference values.

\bar{a} : *Effective area* of the TS as obtained by NMISA.

\bar{A} : *Effective area* of the TS as obtained by CENAM, TS reference *effective area*.

U_{NMISA}^2 : Expanded uncertainty assigned to the TS *effective area* as obtained by NMISA.

U_{CENAM}^2 : Expanded uncertainty assigned to the TS *effective area* as obtained by CENAM, TS *effective area* reference uncertainty.

The normalized error equation results have as criteria the following:

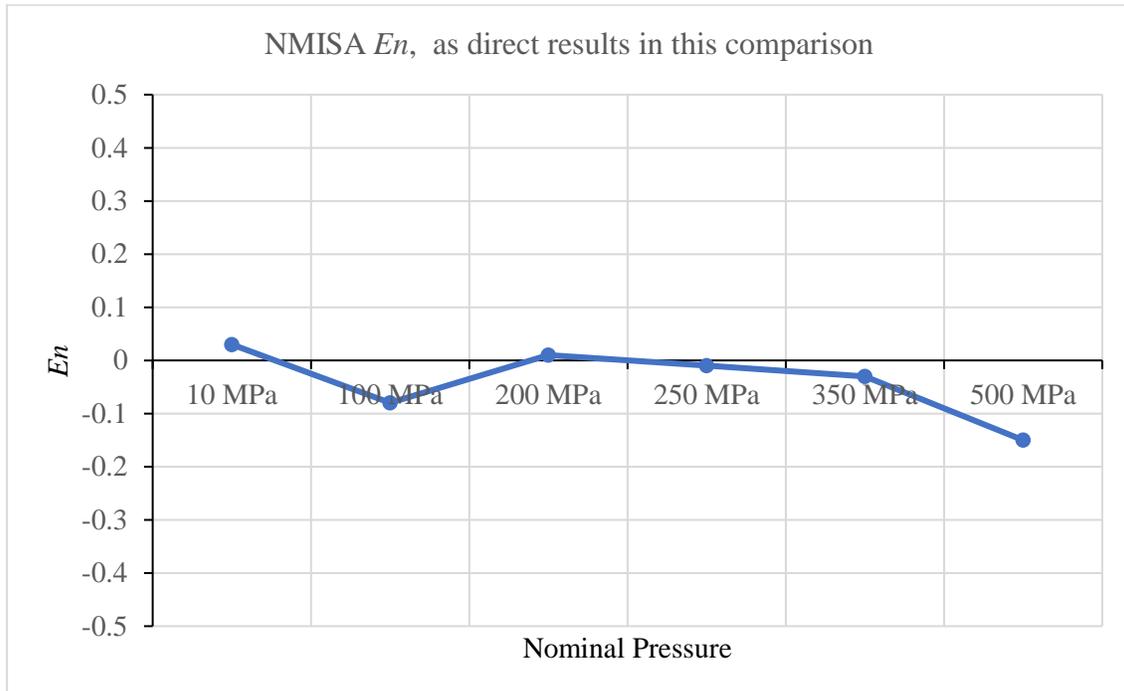
$|E_n| \leq 1.0$ Satisfactory result.

$|E_n| > 1.0$ Non-satisfactory result.

Table 10 and graph 15 present the results for this direct comparison by means of equation 1.

Table 10. Normalized error equation values of NMISA with respect to the reference laboratory (CENAM) for effective area. Direct data as obtained by each laboratory for this comparison, CCM.P-K13.1.

Nominal Pressure	10 MPa	100 MPa	200 MPa	250 MPa	350 MPa	500 MPa
<i>En</i>	0.03	-0.08	0.01	-0.01	-0.03	-0.15



Graph 15. NMISA normalized error equation results with respect to CENAM's reference values.

This direct observation between the 2 laboratories is only with the purpose of comparing results obtained by each laboratory and analyze the compatibility of results in a direct form for the results obtained in this exercise.

The best approach, since the purpose of this SC is to produce equivalence statements and a link of NMISA to CCM.P-K13, is the one presented in the next subsection.

IX.2 Linkage of this Comparison CCM.P-K13.1 with CCM.P-K13.

For the evaluation of the NMISA performance, the normalized error equation ($E_{n\text{ NMISA}}$) criterium was applied to their TS effective area results, according to the following equation (2).

The purpose of this SC is to produce equivalence statements and a link of NMISA to the key comparison CCM.P-K13.

To produce a link of NMISA to the KC reference value of CCM.P-K13, it is necessary to consider the results of CENAM at the CCM.P-K13, and the reference values for the CCM.P-K13.

The following equation 2 is to be used to make these considerations.

$$E_n \text{ NMISA RV} = \frac{(x_{\text{NMISA}} - x_{\text{CENAM}})_{\text{SIM.P-SXX}} - (x_{\text{CENAM}} - x_{\text{RV}})_{\text{CCM.P-K13}}}{\sqrt{U_{\text{NMISA SIM.P-SXX}}^2 + U_{\text{CENAM SIM.P-SXX}}^2 + U_{\text{CENAM CCM.P-K13}}^2 + U_{\text{RV CCM.P-K13}}^2}} \quad (2)$$

Where:

- $E_n \text{ NMISA RV}$: Normalized error of NMISA with respect to CCM.P-K13 reference values.
- $x_{\text{NMISA SIM.P-SXX}}$: Effective area of the TS as obtained by NMISA, in this CCM.P-K13.1.
- $x_{\text{CENAM SIM.P-SXX}}$: Effective area of the TS as obtained by CENAM, TS reference area in CCM.P-K13.1.
- $x_{\text{CENAM CCM.P-K13}}$: Effective area of the TS as obtained by CENAM, TS reference area in CCM.P-K13.
- $x_{\text{RV CCM.P-K13}}$: Effective area of the TS as reference area in CCM.P-K13.
- $U_{\text{NMISA SIM.P-SXX}}^2$: Expanded uncertainty assigned to the TS effective area as obtained by NMISA, in this CCM.P-K13.1.
- $U_{\text{CENAM SIM.P-SXX}}^2$: Expanded uncertainty assigned to the TS effective area as obtained by CENAM, TS effective area reference uncertainty in this CCM.P-K13.1.
- $U_{\text{CENAM CCM.P-K13}}^2$: Expanded uncertainty assigned to the TS effective area as obtained by CENAM, TS effective area reference uncertainty in CCM.P-K13.
- $U_{\text{RV CCM.P-K13}}^2$: Expanded uncertainty assigned to the TS effective area, TS effective area reference uncertainty in CCM.P-K13.

The normalized error equation results have the following criteria:

- $|E_n| \leq 1.0$ Satisfactory result.
- $|E_n| > 1.0$ Non-satisfactory result.

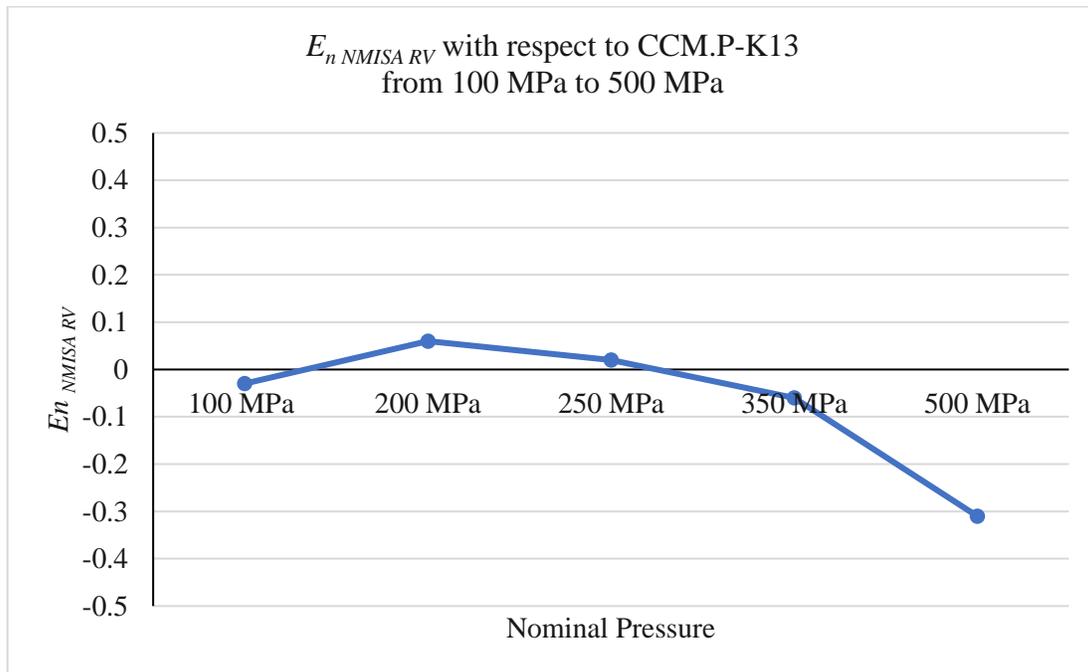
The information of the data used by CENAM in the CCM.P-K13 comparison as well as the reference values of the CCM.P-K13 are including in table 11. The information given in table 8 are also included in table 11 for ease reference.

Table 11. Data for TS effective area as obtained by CENAM in CCM.P-K13.1, in CCM.P-K13 and the reference value for de piston-cylinder in the CCM.P-K13, m².

Nominal Pressure (MPa)	CENAM / CCM.P-K13.1		CENAM / CCM.P-K13		Reference Values, CCM.P-K13	
	Area TS m ²	U Area m ²	Area TS m ²	U Area m ²	Area TS m ²	U Area m ²
100	1.960 971E-06	8.3E-11	1.961 250E-06	1.7E-10	1.961 261 E-06	4.7E-12
200	1.961 159E-06	8.3E-11	1.961 470E-06	1.7E-10	1.961 486 E-06	3.5E-12
250	1.961 252E-06	8.4E-11	1.961 580E-06	1.7E-10	1.961 589 E-06	5.5E-12
350	1.961 438E-06	8.5E-11	1.961 790E-06	1.7E-10	1.961 780 E-06	7.9E-12
500	1.961 735E-06	8.7E-11	1.962 100E-06	1.7E-10	1.962 041 E-06	1.1E-11

Table 12. Normalized error equation values of NMISA with respect to CCM.P-K13 reference values for effective area. Obtained by means of equation 2.

Nominal Pressure	100 MPa	200 MPa	250 MPa	350 MPa	500 MPa
$E_{n\ NMISA\ RV}$	-0.03	0.06	0.02	-0.06	-0.31



Graph 16. NMISA normalized error equation results with respect to CCM.P-K13 reference values.

X. CONCLUSIONS

The conclusions from the results of the comparison method, the normalized error equation method, as presented in section IX; as shown firstly, in table 10 and graph 15, and finally for the evaluation of results with link between this CCM.P-K13.1 and CCM.P-K13 presented in Table 12 and Graph 16, are:

- The normalized error equation values were very low, showing compatibility of results, but also due to the cautious values of the uncertainty used by NMISA. Which, although are not very big, could be lower.
- As conclusion it can be said that NMISA have very good compatibility of results with the reference values provided by CENAM, as it can be seen in Graphs from 8 to 16 and in tables 9, 10 and 12.
- Finally, NMISA has compatibility of results to the comparison CCM.P-K13, as shown in table 12 and graph 16.

ANNEX 1

Mass values, as calibrated at NMISA.

Mass set

The mass set consists of (all pieces being identified by serial number 2972):

Five mass pieces of 10 kg nominal mass, marked by numbers from 1 to 8.

One mass piece of 9 kg nominal mass,

One mass piece of 5 kg nominal mass,

Two mass pieces of 2 kg marked by numbers from 1 to 2,

One mass piece of 1 kg nominal mass.

The material density of the mass pieces is:

$$\rho_m = (7\,900 \pm 79) \text{ kg/m}^3$$

Table 13. The mass of the mass pieces, as calibrated at NMISA, are given in the next table.

IDENTIFICATION	MASS	U_{MASS} $k = 2$
N.S.: 2972	kg	kg
9 kg	9.000 04	1.0E-05
10 kg (1)	10.000 05	1.0E-05
10 kg (2)	10.000 08	1.0E-05
10 kg (3)	10.000 11	1.0E-05
10 kg (4)	10.000 08	1.0E-05
10 kg (5)	10.000 13	1.0E-05
10 kg (6)	10.000 11	1.0E-05
10 kg (7)	10.000 11	1.0E-05
10 kg (8)	10.000 10	1.0E-05
5 kg	5.000 011	5.0E-06
2 kg (1)	2.000 007	2.0E-06
2 kg (2)	1.999 998	2.0E-06
1 kg	1.000 000	1.0E-06