SIM-AFRIMETS Comparison for 500 MPa Range of Hydraulic Gauge Pressure

Technical Protocol

Pilot laboratory:

Centro Nacional de Metrología (CENAM), Mexico

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1. Introduction

It was decided to carry out a supplementary comparison (SC) in the range of 500 MPa of hydraulic gauge pressure. This comparison is aimed to verify the equivalence statements and to provide a link to NMISA for CCM.P-K13 KCs. The participants are 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). It was agreed to send a pressure balance base and piston cylinder assembly as transfer standard (TS) to provide better stability. The Centro Nacional de Metrología (CENAM), México, has agreed to be a pilot laboratory in this SC.

This Technical Protocol specifies the procedures to be followed in the comparison and has been prepared in accordance with the Guidelines for CIPM Key Comparisons,

2. Participants

The list of the participating laboratories with their addresses being the delivery addresses for the transfer standard (TS) and the names of the contact persons in the participating laboratories with their phone and their e-mail addresses is given below.

- Centro Nacional de Metrología (CENAM) - pilot laboratory

  TS delivery address:

  CENAM
  Pressure Working Group
  Dr. Jorge C. Torres-Guzman
  Carretera a Los Cues km 4.5
  76246 El Marques Queretaro
  Mexico.

  Contact person (comparison co-ordinator):

  Dr. Jorge C. Torres-Guzman
  Phone: +52 442 110572
  E-mail: jorge.torres@cenam.mx

- National Metrology Institute of South Africa (NMISA)

  TS delivery address:

  NMISA Building No.5
  CSlR Campus
  Meiring Naude Drive
  Brummeria, Pretoria
  South Africa
  Postal Code: 0040
  Pressure Laboratory

  Contact person (Participant):

  Mr. Thulani Khoza
  Phone: +2712 841 2399
  E-mail: tkhoza@nmisa.org

3. Time table

The measurements with the transfer standard will be performed in accordance with the schedule given below

Measurement time	Measurement time	Institute
2021-01-04	2021-03-26	NMISA
2021-03-30	2021-04-16	Transportation of the TS / sending of results
2021-04-20	2021-05-14	CENAM
2021-05-24	2021-06-07	Transportation of the TS
2021-06-14	2021-08-16	NMISA
2021-09-30	2021-09-30	Sending of results
2021-10-02	2021-10-13	CENAM, Draft report of the comparison

Each participant is requested to keep strictly to this schedule.

Sending TS, the laboratory has to inform the other laboratory all delivery information before sending the TS.

The receipt should be confirmed to the sender.

In the case of any delay or circumstances which will lead to a delay, the participant shall inform the destination laboratory about the problem.

4. Transportation

Each laboratory is responsible for the transportation of TS to the other laboratory. The transport way should be chosen by each laboratory to deliver TS safely and in time. All TS parts should be carefully packed using original packing materials and containers. Completeness of all components is to be checked before departure and after arrival of TS.

Declaration Letters will be prepared which should relieve the temporal import of TS.
Care should be taken that the Declaration Letter is sent prior to sending the TS.
The indication that the instrument can be unpacked and handled only by qualified personnel should be clearly seen on the package. After arrival, TS shall be first inspected visually and deviations, if any, recorded and reported to the pilot laboratory.
Additional transportation rules regarding a mass set to be used with TS are specified in Section 6.

5. Costs

Each participant bears the costs for its own measurements, any customs charges, transport costs and the costs occurring from a lost or damage of TS within its country.

6. Transfer standard

The transfer standard is a piston-cylinder assembly of 2 mm^2 nominal effective area with serial number 2279. It is built in a pressure balance equipped with a mass carrier, all parts having been manufactured by Fluke Calibration, USA.

The TS stability will be checked by the pilot laboratory comparing the results of all calibrations (those obtained at the beginning of the comparison, at the intermediate test and at the final investigation).

All relevant technical data of the TS are given in Annex 1.

The list of TS components is given below:
1. Piston-cylinder assembly serial no. 2279 in carrying case with a special mounting key.
2. Carrying bell, serial no. 1201
3. Pressure balance model PG-7302, serial no. 1177
4. Pressure balance control unit.
5. 2 glass bottles with sebacate.
6. Oil run-off cup.
7. Temperature probe serial no. 1102 (built in pressure balance).
8. Power supply, AC input (100-240) V, (50-60) Hz.
9. Power supply cord.

The transfer standard is sent in 6 boxes. They are made of plastic hard case, has a sizes 1 x [46x63x51] cm3, 1 x [43x42x33] cm3 and 4 x [53x53x26] cm3 and the weight (with contents) of about 150 kg (inclusive of mass set). It contains the dead weight base, the carrying bell, the piston-cylinder assembly in a plastic hard case as well as all other parts of the TS.

The transfer standard and all accompanying parts are the property of the NMISA. The total cost of TS including the pressure balance, the masses and the piston-cylinder unit is R1 400 000.00 (ZAR).

7. Arrival and departure checks

When TS arrives at the laboratory, it should be checked by the laboratory personnel for damage and completeness of its components. The arrival check report prepared in Annex 2 should be completed.

When sending the TS, it should be checked again and the departure check report given in Annex 3 should be filled in and sent to the pilot laboratory and to the destination laboratory by e-mail.

Completely screw in the four adjustable feet of the pressure balance before putting into the hard case box.

The piston-cylinder shall be removed from the pressure balance and placed in its carrying case prior to shipment.

Follow the instruction in the User's Manual when preparing the pressure balance for the work or for the shipping.

8. Measurement procedures

The transfer standard and the piston-cylinder assembly mounted should be handled in accordance with the instructions given in the User's Manual Reference Pressure Balance, PG-7000 Series, Model PG-7302.

The results of a metrological characterisation of TS are presented in Annex 1. They should help to verify that the TS operates normally. In the case of any anomaly or significant deviation from the results of the pilot laboratory it should be contacted.

Gloves should be worn when handling the piston, the carrying bell and the masses of TS.

Before mounting the assembly the piston and cylinder should be cleaned according to the usual practice in the laboratory.

The cylinder should be installed in the mounting post and secured.

The TS pressure balance is operated with di(2-ethylhexyl) sebacate (DHS) as a pressure transmitting medium whose properties are reported in Annex 1. No other liquid may be used in the TS pressure balance. The laboratory receiving TS should check that TS is filled with a clean DHS, and, if it is contaminated, report this in Arrival check protocol.

To check the tightness of TS, the piston fall rate shall be measured preferably at pressures of (500, 250 and 10) MPa. Wait a minimum of 10 minutes after generating the pressure in the TS measurement system prior to starting the piston fall rate measurements in order to stabilise the TS temperature. During these measurements both the low and the high pressure shut off valves should be closed because even a minimal leak in the variable volume screw press can significantly disturb the results. The target fall rates are given in Annex 1. The measured fall rates shall be reported to the pilot laboratory in the laboratory report.

The temperature of TS is measured with a platinum resistance thermometer (see Annex 1). Each laboratory shall use its own electronics to measure the resistance of the thermometer. The maximum current supplied to the resistance thermometer should not exceed 5 mA to avoid its self-heating.

The reference temperature of the comparison is 20 °C. If measurements are performed at a temperature deviating from 20 °C, the effective area of TS should be referred to 20 °C using the piston-cylinder thermal expansion coefficient given in Annex 1.

TS is recommended to be located close to the laboratory's reference standard to keep the pressure line between the two instruments as short as possible.

It is also recommended to adjust the height position of TS to minimise the height difference between the reference level of TS defined in Annex 1 and the reference level of the laboratory standard.

Piston displacement indicator can be used to control the piston position.

In the equilibrium state, the piston of TS should not deviate from its working position by more than 0.5 mm.

During a cross-float equilibrium between the reference standard and TS, both the low and the high pressure shut off valves shall be closed to avoid the effect of possible oil leak in the variable volume screw press.

The direction and the rotation speed of the piston is predefined by the motor of the pressure balance model PG7302. The rotation is clockwise with the speed 32 rpm.

It is assumed that the motor is switched on during the cross-float equilibrium measurement. From experience of the pilot laboratory the motor does not produce any visible disturbance if TS is cross-floated against another pressure balance. However, if a high-resolution electronic pressure monitor is used to measure the pressure of TS, disturbances might occur when the piston is hit by the rotating pin. In such a case the laboratory may operate TS with the motor being switched off. Then the piston should be rotated by hand in the clockwise direction with the rotation speed lying between 30 and 35 rpm. The piston free rotation time is given in Annex 1.

As the motor is the main source of heat (s. Annex 1), the piston rotation should be switched on at least 30 minutes before starting the measurement to get a quasi-stationary distribution of temperatures in the pressure balance. In the time between taking measurement points, the motor is recommended to stay switched on. Only if the time till the next measurement exceeds approximately 2 hours, the motor can be switched off. However, in such a case, it should be switched on at least 30 minutes prior to the next measurement.

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 shall include five cycles each with nominal pressures generated in the following order (10, 100, 200, 250, 350, 500, 500, 350, 250, 200, 100, 10) MPa. The generated pressures should not deviate from these nominal values by more than 0.1 MPa Thus, 60 measurements shall be performed in total. It should be waited for at least 15 minutes between two consequent measurements at 500 MPa and between two measurement cycles.

The masses of the piston and the carrying bell are given in Annex 1.

9. Report of results

The participant laboratory should provide the pilot laboratory with the data of its own pressure standard filling in the form in Annex 4 as well as with additional information, if useful.
It should be reported how TS was connected to LS and how their pressure equilibrium was achieved and controlled. When LS was operated with another pressure-transmitting medium than used in TS, details of separating the two different liquids should be included.

To prepare the results of the measurements in the form convenient for the further processing, the participant should use the tables presented in Annexes 5 and 6.

The effective area of TS determined from a particular measurement (A'_p) referred to 20 °C can be calculated with the equation:

$$A'_p = \frac{\sum_i m'_i g \left(1 - \frac{\rho_a}{\rho'_i}\right) + 2\sigma\sqrt{\pi A'_{0,\mathrm{nom}}}}{p'\left[1 + \left(\alpha'_p + \alpha'_c\right)\left(t' - t'_0\right)\right]}, \quad \text{where} \tag{1}$$

m'_i are true masses of the piston, the weight carrier and the mass pieces placed on the weight carrier of TS;
ρ'_i are densities of the parts with masses m'_i;
ρ_a is air density;
g is local gravity acceleration;
σ is surface tension of the TS oil;
$A'_{0,\mathrm{nom}}$ is nominal effective area of TS;
p' is pressure generated by the laboratory standard at the TS reference level;
α'_p and α'_c are thermal expansion coefficients of the piston and cylinder materials, respectively;
t' is temperature of TS;
t'_0 is reference temperature, $t'_0 = 20$ °C.

The values of p', ρ_a and t' as well as the masses of the participating laboratory are to be calculated or measured by the laboratory. All other parameters can be found in Annex 1.

Each laboratory should report whether equation (1) or an alternative equation was used for A'_p determination. The formula to calculate p' should be given. In particular, it should be reported whether p- or Δp-method was used.

Additionally, the zero-pressure effective of TS (A'_0) and its pressure distortion coefficient (λ') which satisfy equation

$$A'_p = A'_0(1+\lambda' p) \tag{2}$$

and are based on the results of all 60 measurements, the combined standard uncertainties of A'_0 and λ' as well as a description of how they were calculated should be included.

Reports with the results of the measurements are expected to be prepared as a WORD file with the tables from Annexes 5 and 6 being additionally provided in an EXCEL file. The reports should be sent to the pilot laboratory by e-mail to the address

jtorres@cenam.mx

within two weeks after finishing the measurements.

ANNEX 1

Technical data of the transfer standard

All uncertainties in this Annex are the standard ones.

Piston-cylinder assembly

The serial number, 2279, is engraved on the upper cylinder face and the upper piston cap face. The nominal effective area of the assembly is
$A'_{0,nom} = 1.96\ mm^2$.

Piston-cylinder material properties

The cylinder of the assembly is made of tungsten carbide, and the piston is made of steel with the following linear thermal expansion coefficient (α), Young's modulus (E) and Poisson's coefficient (μ):

	Material	α / °C	E / N/m^2	μ
Piston	Tungsten Carbide	(0.45 ± 0.05)·10-5	5.6E-11	0.218
Cylinder	Tungsten Carbide	(0.45 ± 0.05)·10-5	6.2E-11	0.218

The thermal expansion coefficient of the piston-cylinder unit can be taken as
$\alpha'p + \alpha'c = (0.9 \pm 0.05)\cdot 10^{-5}\ °C^{-1}$.

The cylinder cap is of stainless steel.

Piston mass and density

	True mass / g	Density g/cm^3
Piston	200.000 8 ± 0.000 2	7.230 ± 0.02

Reference level and piston working position

The piston working position is 5 mm above its rest position.
The reference level of the TS coincides with the piston lower face; the total piston length (distance from the piston lower face to the upper piston cup edge) is 84.47 mm.
Herewith, the reference level lies 0.06 mm above the middle of the reference level line shown on the housing of the pressure balance.
The pilot laboratory measurements in which the piston was located within [-1; +1] mm around its normal working position have not shown any systematic change in the effective area.

Typical cross-float sensitivity and reproducibility

The change in the TS piston load by
20 mg at 50 MPa,
50 mg at 150 MPa and
100 mg at 350 MPa
led to a reproducible reverse of the TS piston motion when it was compared with the reference standard. This corresponds to the relative sensitivity in pressure of $2 \cdot 10^{-6}$, $1.7 \cdot 10^{-6}$ and $1.4 \cdot 10^{-6}$ at pressures (50, 150 and 350) MPa, respectively.
The relative experimental standard deviations of single values of the effective areas measured at the pressures specified for the comparison lie typically between (3 and 7)$\cdot 10^{-6}$.

Piston-cylinder temperature drift

When the driving motor continuously works, the temperature of the piston-cylinder assembly increases with a typical rate between (0.05 and 0.15) K/h during first 4 h.

Piston fall rates

Piston fall rates (v_f) measured by the pilot laboratory at temperatures around 20 °C are:

p / MPa	v_f / (mm/min)
10	0.02
100	0.14
200	0.21
250	0.30
350	0.40
500	0.40

It should be waited minimum 10 minutes after generating the pressure in the TS measurement system prior to starting the piston fall rate measurements in order to stabilise the TS temperature. When measuring v_f, both the low and the high pressure shut off valves should be closed to avoid the effect of possible oil leak from the variable volume screw press.

Piston free rotation time

When piston rotates freely (without motor) at a pressure of 50 MPa and a temperature of 20 °C, the rotation speed descends from 35 rpm to 30 rpm within 15,5 minutes.

Stability of the transfer standard

It will be estimated during the comparison process.

Carrying bell

The carrying bell, serial number 1201, is made of stainless steel.
Its true mass is m_b = (800,014 7 ± 0,000 8) g.
Its density is ρ_b = 6 058 ± X·10^{-3}) kg/m^3.

Temperature probe

The temperature of the piston-cylinder assembly (t) is determined with a platinum resistance thermometer using the formula
t = [R/Ω - (100.00)] / 0.3896 °C,
where R is the resistance of the probe at temperature t. This equation results from the calibration by the pilot laboratory in the temperature range (18.5 to 24.5) °C. The maximum current sent through the resistance thermometer should not exceed 5 mA to avoid its self-heating.

Pressure transmitting medium

The working liquid is di(2-ethylhexyl) sebacate (DHS). According to the literature data its density (ρ_l) in dependence on pressure (p) and temperature (t) is
$\rho_l = [912.7 + 0.752(p/\mathrm{MPa}) - 1.65\cdot 10^{-3}(p/\mathrm{MPa})^2 + 1.5\cdot 10^{-6}\,(p/\mathrm{MPa})^3] \times$
$\times\, [1 - 7.8\cdot 10^{-4}\,(t/°\mathrm{C} - 20)] \times (1 \pm 0.01)\ \mathrm{kg/m^3}$.
The surface tension (σ) of DHS as determined by the pilot laboratory is:
σ = 31.2 x (1 ± 0.05) mN/m

ANNEX 2

Arrival check protocol

Laboratory name:

Arrival of TS

Date:

From:

TS is free of damage (yes/no):

if not, describe the damage

TS set is complete (yes/no):

if not, indicate the missing part from the list below

- Piston-cylinder assembly serial no. ________ in carrying case with a special mounting key
- Carrying bell, serial no. ________
- Pressure balance model ________ serial no. ________
- 4 foot rests for the pressure balance
- 3 piston travel limit pins
- Cylinder retaining nut
- 2 quick connectors no. ________
- Plug for quick-connecting head
- 2 glass bottles with sebacate
- Oil run-off cup
- Temperature probe serial no. ________ (built in pressure balance)
- Temperature probe output cable
- Power supply, AC input (100-240) V, (50-60) Hz
- Power supply cord
- User’s Manual Reference Pressure Balance, ________ Series, Model ________
- Handle of variable volume screw press
- Plug for mounting post

Report date:

Name of inspecting person:

Address, phone, fax, e-mail, if deviating from those given in section 2 “Participants”

ANNEX 3

Departure check protocol

Laboratory name:

Departure of TS

Date:

To:

TS is free of damage (yes/no):

if not, describe the damage

TS set is complete (yes/no):

if not, indicate the missing part from the list below

- Piston-cylinder assembly serial no. 2279 in carrying case with a special mounting key
- Carrying bell, serial no. 1201
- Pressure balance model PG7302 serial no. 1177
- 3 foot rests for the pressure balance
- Cylinder retaining nut
- 2 quick connectors no.
- Plug for quick-connecting head
- 2 glass bottles with sebacate
- Oil run-off cup
- Temperature probe serial no. 1102 (built in pressure balance)
- Temperature probe output cable
- Power supply, AC input (100-240) V, (50-60) Hz
- Power supply cord
- User's Manual Reference Pressure Balance, PG7000 Series, Model PG7302
- Handle of variable volume screw press
- Plug for mounting post

Report date: 28/01/2021

Name of inspecting person: Thulani Khoza

Address, phone, fax, e-mail, if deviating from those given in section 2 “Participants”

ANNEX 4

Laboratory standard and measurement conditions

The uncertainties here should be expressed as the standard ones.

Manufacturer	Fluke
Measurement range in MPa	10; 100; 200; 250; 350; 500
Material of piston	Tungsten Carbide
Material of cylinder	Tungsten Carbide
Operation mode, free-deformation or controlled-clearance	Free deformation
Zero-pressure effective area (A_0) at reference temperature, in mm^2	1,96 073
Relative uncertainty of A_0 in 10^{-6}	0,000 14
Pressure distortion coefficient (λ) in MPa^{-1}	1,013 * 10^{-6}
Uncertainty of λ in MPa^{-1}	1,013 * 10^{-7}
Relative uncertainty of mass pieces in 10^{-6}	105
Linear thermal expansion coefficient of piston (α_p) in °C^{-1}	0,000 000 225
Linear thermal expansion coefficient of cylinder (α_c) in °C^{-1}	0,000 000 225
Reference temperature (t_0) in °C	0,1
Local gravity (g) in m/s^2	9,786 099
Relative uncertainty of g in 10^{-6}	0,000 005
Height difference between laboratory standard (LS) and TS (h, positive if LS is higher than TS) in mm	0
Uncertainty of h in mm	2

In addition, traceability of LS to SI units should be explained. Details of how A_0 and λ and their uncertainties were determined should be reported.

ANNEX 5

Results in individual cycles

Laboratory name :

Date (period) :

Cycle number :

Average relative air humidity and its uncertainty:

Meas. no.	Nominal pressure (MPa)	t (°C)	t_{amb} (°C)	p_{amb} (kPa)	p' (MPa)	t' (°C)	A'_p (mm^2)
1	10						
2	100						
3	200						
4	250						
5	350						
6	500						
7	500						
8	350						
9	250						
10	200						
11	100						
12	10						

t is temperature of the laboratory standard;
t_{amb} is temperature of ambient air;
p_{amb} is pressure of ambient air;
p' is pressure generated by the laboratory standard at the TS reference level;
t' is temperature of TS
A'_p is effective area of TS at the reference temperature 20 °C.

The formula to calculate p' must be reported.

ANNEX 6

Summary of all cycles

Laboratory name :

Date (period) :

Nominal pressure (MPa)	Typical min. adjusted mass (mg) 1)	Average of A'_p, $\langle A'_p \rangle$ (mm²) 2)	Relative standard deviation of $\langle A'_p \rangle$ (10^{-6}) 3)	Relative standard uncertainty of p' (10^{-6}) 4)	Standard uncertainty of t' (°C) 5)	Rel. standard uncertainty of $\langle A'_p \rangle$ (10^{-6}) 6)
10						
100						
200						
250						
350						
500						

1) the smallest mass adjusted on the piston of the transfer standard to reach the equilibrium between it and LS, if the classical fall rate method to control the equilibrium is used. If another method is applied, the typical uncertainty of the pressure difference between the reference standard and TS (in MPa) due to this method should be given. In this case the heading of this column shall be appropriately changed;

2) average of the values measured at the same nominal pressure;

3) standard deviation of the mean value;

4) type B uncertainty including the uncertainty of the pressure at the reference level of TS, which includes uncertainty of pressure generated by the laboratory standard, of the height difference between the laboratory standard and TS, of the density of the pressure transmitting oil, etc.;

5) type B uncertainty of the temperature measurement on TS

6) combined uncertainty of the mean values in 2);

In addition, a list of the main uncertainty sources and their contributions to $\langle A'_p \rangle$ for pressures 10 MPa and 500 MPa must be presented.

All the uncertainties should be expressed as the standard ones.