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Pyramidal Traceability Hierarchy for Pressure Measurements and Calibrations at NIS- Egypt

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Abstract: Pressure balances are excellent standards for measuring pressure with acceptable uncertainty and they are widely used at the primary pressure laboratories in the world. This study aims to study the propagation of uncertainty from primary standard piston cylinder assembly (PCA) up to 500 MPa. The hierarchy of pressure measurements at NIS is based on using large effective area PCA in defining the pressure of 1 MPa. Characterization of primary standard PCA is presented transferring the obtained results to other level pressure standard described. Uncertainty calculation method at each level was studied. Propagation of uncertainty from primary standards through national standards to digital pressure gauges, digital pressure calibrators, pressure sensors and pressure transducers were investigated. Study of the effect of each variable on the uncertainty calculation was discussed. *Copyright © 2012 IFSA.*

Keywords: Pressure balance, Calibration, Sensor, Uncertainty and traceability.

1. Introduction

When comparing results of measurements obtained by a measuring instrument against a standard whose traceability to the International System of units (SI) is assured with valid certificates this is called calibrations. In order to ensure the calibration of a measuring instrument traceable to (SI), a hierarchy of standards shall be established. So traceability is a concept of establishing a valid calibration of a measuring instrument or a standard step by step comparison with better standards, the national standard being the ultimate reference [1].

To achieve the traceability, a pyramid type structure of hierarchy of standards has been developed. Level (I) of hierarchy is the characterization of the National Primary Standards traced to the (SI).

When the National Primary Standards at the top level are used to calibrate the Reference Standards or transfer standards this is called level (II) of the hierarchy. At a level (III), there are calibrations that use reference standards or transfer standards calibrated against the National Primary Standards to calibrate other working standards or high level working instrument. So the concept of traceability is very important in measurements and any measurement taken with equipment which has no valid calibration is meaningless.

For mutual and international compatibility of measurement results between the countries, bilateral and/or international intercomparison of measurement, results of the national primary standards need to be performed regularly which also has become a necessity during this present global scenario.

Measurements in general are carried out with the objective of determining the physical parameter characteristic of an item or a measurement process using an equipment or assemblies of equipments called apparatus. A measurement process cannot give meaningful results unless all such assignable causes of variability are eliminated. The ability to reproduce a measurement with as little variability as possible is closely linked to the environmental factors that control the measurement process. The characteristic of a measurement process with controlled variables is called "Precision”.

In order to get meaningful and reliable results in measurements, there are certain steps which are to be followed in sequence applicable to all scientific measurements, irrespective of the field of measurements applied. These steps in general speaking way would be classified as identifying of measurement standard with all necessary supportive equipments, initial preparation of the environmental and working conditions required for the specific field of measurements, setting up of equipment under calibration, selection of measurement technique (method), developed measurement operations and conclusion of the results in their order of sequence. All these exercises have to be performed by a competent operator. In the present work, investigation of the hierarchy of pressure measurements, at National Institute of Standards NIS – Egypt, with example for each level will be presented associated with uncertainty calculation.

2. Pressure Measurement Hierarchy at NIS

The hierarchy of pressure measurements at NIS is based on using large effective area piston cylinder assembly (PCA) in defining the pressure of 1 MPa then transferring such pressure to the reference PCAs, the reference or transfer standards and the working instruments as shown in Fig. (1).

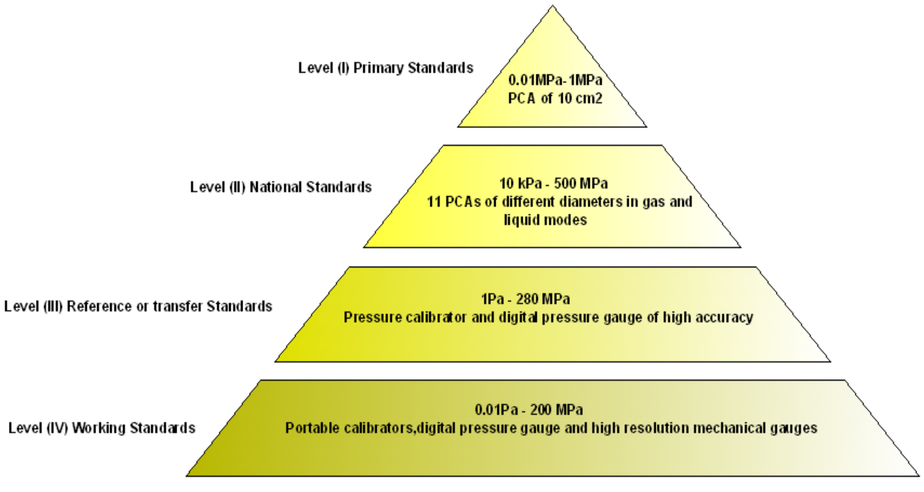

Fig. 1. Pyramidal hierarchy of traceability in pressure measurements at NIS.

3. Estimation of Measurement Uncertainty

The present day quality society stress adherence to ISO/IEC17025–2005 requirement for improvement and assuring the quality of calibration result. Improvement in quality of calibration cannot be assessed unless there is an opportunity of enhancement of measurements. The measurements for most of the parameters can be carried out either using a single measuring equipment or a measurement set consisting of several equipments. Thus any measurement carried out with one to one correspondence without any functional relationship is called "direct measurement". But this is not so in many measurement circumstances, especially in pressure measurements where the parameter under measurement is functionally related to other parameters and has to be evaluated indirectly. In some situations, when using the equipment for a span of parameter ranges, it may not give linear relationship due to intrinsic changes in related parameters under different conditions.

The quality of measurements can be assessed by the estimation of any measurable physical quantity through statistical analysis if the measurement process adopted is under statistical control. The estimated value may be slightly less or more than the true value of the physical quantity and the range in which the true value is estimated to lie is called the uncertainty of measurement. The uncertainty calculations under direct method of measurements are simple. In indirect method of measurements, the uncertainty depends not only on the uncertainty of the input variable parameters but also on the functional relationship of the measurement parameter to its input variable parameters. The transfer of uncertainties from the input variables to the output variable through the functional relationship is the uncertainty transmission. The quantum of transmission depends on the uncertainties associated with the input variables and their statistical distributions and the nature of the functional relationship.

4. Uncertainty Transmission

Uncertainties are always expressed as a multiple of standard deviation of the variable parameter under measurement with some coverage factor. The output variable might be related to the input variables through a linear or nonlinear relationship continuous or non continuous. If the function f (x_i) is continuous and has derivatives around the mean value of the input variables, then the function can be written

$$y = y_0 + \sum_{i=1}^{n} \frac{\delta f}{\delta x_i}(x_i - u_i) + \sum_{i=1}^{n} \frac{1}{2} \frac{\delta^2 f}{\delta x_i^2}(x_i - u_i)^2 + \sum_{i=1}^{n-1} \sum_{j=1}^{n} \frac{\delta^2 f}{\delta x_i \delta x_j}(x_i - u_i)(x_j - u_j) + \ldots\ldots\ldots, \tag{1}$$

where

y_o is the value of function evaluated at the mean value of the input variables;
x_i is the ith input variable;
u_i is the mean of the input variables;
y is the output or the parameter of our interest.

The combined standard uncertainty thereby the expanded uncertainty are estimated for pressure by the analysis of uncertainty transmission. Generally first order approximations give satisfactory results in pressure measurements, then

$$y = y_0 + \sum_{i=1}^{n} \frac{\delta f}{\delta x_i}(x_i - u_i) \tag{2}$$

The derivatives are evaluated at the mean values of the input variables.

4.1. Type (A) Uncertainty

The random uncertainty depends upon the following factors:

- Method of measurement;
- Inconsistency of the measured parameter;
- Scales of the operator.

$$U_i = t.\frac{S}{\sqrt{n}}, \tag{3}$$

where S is an estimate of population standard deviation given by

$$S = s.\sqrt{\frac{n}{n-1}} \tag{4}$$

where s is the standard deviation of the n readings and t is the student's factor and n is the number of readings.

4.2. Procedure of Type A Uncertainty Calculation

If the number of readings was n: x_1, x_2, …. x_n. Then the following procedure is to be followed to calculate the type A uncertainty:

- Calculate the sample mean which is the point estimate of the true value and is the assigned value of the test result.
- Calculate standard deviation which is also the point estimate of the population standard deviation.
- Decide the confidence level at which the measurement uncertainty is to be evaluated.
- From the standard t table find out the student factor for the specified confidence level and number of degrees of freedom (n-1).
- Calculate type A uncertainty using equations (3) and (4).

4.3. Type B Uncertainty Calculation

In a measurement process, involving direct measurement method, identifying all the factors that contribute to the systematic uncertainty is a very involved task and needs a very clear understanding of the process. For determining the systematic uncertainty component (U_x), the uncertainty due to three sources namely measurement standard, method of measurement and the inherent characteristics of the instrument under test are taken into consideration. Some common type of systematic uncertainty components in measurements are the reported uncertainty of the standard used, uncertainty due to change in environmental conditions, uncertainty due to the characteristics of the measuring instrument and drift in measurements with time of the instrument etc. The combined uncertainty due to those systematic components which follow uniform or rectangular probability distribution is calculated by applying the proper probability distribution for each case.

5. Establishing of the Traceability

5.1. Level (I) Primary Standards

Piston gauge PC-NIS1 is a gas primary PCA that operate from 10 kPa to 1 MPa. It is deemed primary standard for pressure as its effective area is derived from dimensional measurements of the piston and cylinder diameters [3], along with force model of the gas flow in the gap between the piston and cylinder.

Fig. 2 shows a photographic view of the PCA. PC-NIS1 nominal diameter is approximately 35 mm and its length is 30 mm. The radial clearance between piston and cylinder is about 600 nm.

Fig. 2. Photographic view of the PC-NIS1 (Piston on the left and cylinder on the right).

The effective area A_e of PC-NIS1 is given by the linear distortion equation:

$$A_e = A_0\left(1+\lambda p\right) \tag{5}$$

Here, A_0 is the effective area at atmospheric pressure and the reference temperature, 20 ºC, λ is the pressure coefficient and P is the applied pressure.

5.2. Characterization from Dimensional Measurements

Diameters were measured along two directrices (two longitudes, 0° to 180° and 90° to 270°) for both pieces. Diameters were obtained at two places in both vertical planes, or four diameters on the piston and four diameters on the cylinder. All diameters were measured at about 20 °C and adjusted to the reference temperature of 20 °C. A full set of straightness data, as shown in Fig. 3 was obtained for both piston and cylinder. Roundness data is shown in Fig. 4.

5.3. Direct Averages

The effective area of PC_NIS1 was calculated from the average diameters of the piston and cylinder that yielded values for the areas of each component at the reference temperature of 20 °C:

$$A_{op,20} = \pi D_p^2 / 4 \tag{6}$$

and

$$A_{oc,20} = \pi D_c^2 / 4 \tag{7}$$

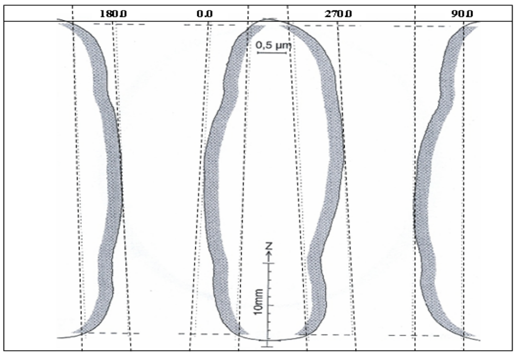

Fig. 3. Straightness traces of PC-NIS1 measured at 0°, 90°, 180°, and 270°.

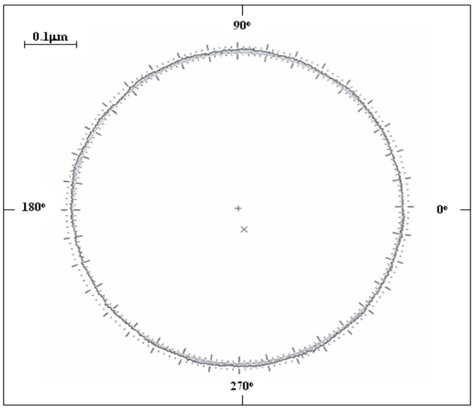

Fig. 4. Roundness traces of PC-NIS1 measured at different elevations from the bottom.

Here D_p and D_c are the average diameters of the piston and cylinder, respectively. The zero pressure effective area of the assembly derived from these measurements at 20 °C is:

$$(980.5774983 \pm 0.0038)\ \text{mm}^2 \quad = A_{o,20} = \left(A_{0p,20} + A_{0c,20}\right) / 2 \tag{8}$$

The uncertainty listed is approximately 4 ppm at (1σ) at zero pressure which is obtained from the dimensional measurements uncertainty. The uncertainty calculated from the standard deviations of piston and cylinder diameters were divided by the square root of the number of measurements points; this is taken as the uncertainty due to diameters variations. The calculations of the effective area at zero pressure from dimensional measurements were validated by solving equation (9) [1].

$$A_0 = \pi r_0^2 \left\{ 1 + \frac{h_0}{r_0} + \frac{1}{r_0} \frac{\int_0^l \frac{(u + U)}{h^3} dz}{\int_0^l \frac{1}{h^3} dz} \right\},$$

where

r_0 : radius of the piston at the bottom of the engagement length (z=0).
h_0: difference between piston radius at any given (z) and cylinder radius at (z=0).
h : difference between cylinder radius at any given (z) and cylinder radius at (z=0).
u : difference between piston radius along the engagement length at any given (z) and piston radius at (z=0).
U: difference between cylinder radius along the engagement length at any given (z) and cylinder radius at (z=0).

Fig. 5 shows a schematic diagram of the PCA with x- dependent radii [1] where all the above notations are illustrated.

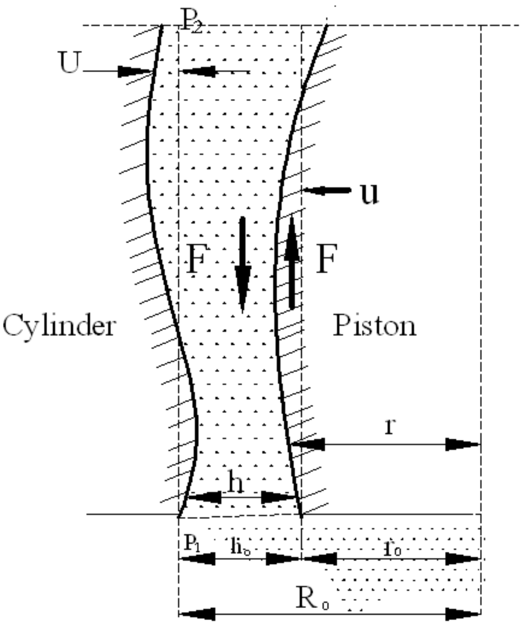

Fig. 5. Schematic diagram of the PCA with x- dependent radii.

The relative difference between result of the numerical calculation of the zero pressure effective area from equation (9) and the obtained result from experimental work according to equation (8) was found to be 3.5 ppm which complies with the standard uncertainty calculated from cross floating experiment. Such accepted difference between the two calculated and experimental methods was a good validation of the obtained zero pressure effective area of the primary standard.

5.4. Level (II) National Standards. Calculations of Effective Area from Cross Floating Experiment

The effective area of the piston-cylinder assembly under test, as a function of pressure A_T, could be calculated using the equation

$$\frac{W_s}{A_s} = \frac{W_T}{A_T} \tag{10}$$

where W_S is the applied weights on the standard PCA, W_T is the applied weights on the under test PCA and A_S is the effective area of the standard PCA. Knowing both W_S and W_T and using the effective area of the standard PCA, as obtained from primary standard characterization or from a calibration certificate, A_T could be calculated.

At ideal condition, a PCA under test is hydrostatically balanced against a primary standard PCA of known effective cross-sectional area. The basis of the comparison is determination of the loads at which each PCA would individually barostat the system at precisely the same pressure. State of equilibrium is identified by rate-of-fall technique [4], which means that load of each assembly is carefully adjusted by means of trimming weights until both pistons are falling in their natural rates. Fig. 6 shows the used system for crossfloat experiment.

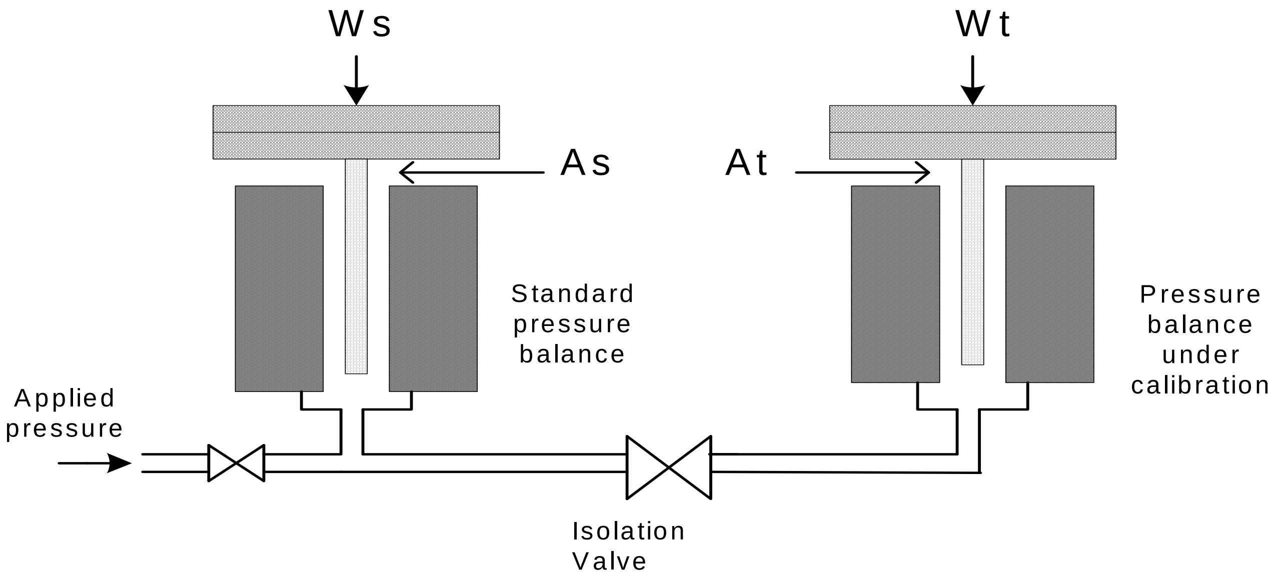

Fig. 6. Schematic diagram illustrating the cross floating experiment set up.

The pressure balance under test, T, is connected to a standard pressure balance, S, and a pressure generator. The loads, M, on both pistons are adjusted until they are hydrostatically balanced at the required pressure. Difference in altitude, Δh, between the reference levels of the S and T balances as well as the temperatures of the environment, tested and standard PCA units are measured. For each calibration point the applied pressure, p_e, measured at the reference level of the S is calculated, using the known characteristic of the standard piston/cylinder assembly according to

$$P_S = \frac{\left\{ \left[\sum_i m_i \left(1 - \frac{\rho_a}{\rho_{mi}} \right) \right] + (h.A_0 - \upsilon) * (\rho_f - \rho_a) \right\}_S g + \Gamma.C_S}{A_{0S}.(1 + \lambda.p_s).[1 + (\alpha_p + \alpha_C)_S (t_S - 20)]} + \rho_f.\Delta h.g \tag{11}$$

where:

A_{0s} is the zero pressure effective area of standard PCA;
P_S is the gauge pressure measured at the bottom of the standard piston;
m_i is the mass of i-th weight applied on the piston, including all floating elements;
g is the local acceleration of gravity;
ρ_a is the density of surrounding air;
ρ_{mi} is the density of i-th weight;
α_p is the linear thermal expansion coefficient of the piston;
α_c is the linear thermal expansion coefficient of the cylinder;
t_S is the measured temperature of the standard PCA at the time of balancing;

λ is the pressure distortion coefficient;
ρ_f is the density of the measuring fluid;
Δh is the difference in altitude between the bottoms of the two PCAs;
Γ is the surface tension of the measuring fluid;
C_s is the circumference of the standard piston.

At each calibration point, the effective area, A_{eT}, of the test PCA can be calculated as:

$$A_{eT} = \frac{\sum_i m_i \left(1 - \frac{\rho_a}{\rho_{mi}}\right)_T g + \Gamma C_T}{(p_S + \rho_f . g . \Delta h).[1 + (\alpha_p + \alpha_C)_T (t_T - 20)]} \tag{12}$$

where A_{eT} is the effective area of the under test as a function of applied pressure. All other symbols have the same definition as in equation (11) by replacing the index S “standard” with the index “T” under test.

The effective area is a linear function of the pressure as given by equation (5). The aim of calibration is to determine the PCA effective area at null pressure A_0. The experimental results from crossfloat calibration have the independent variable p_e, represented by X– axis, while the dependent variable $A_{eT}(p_e)$ is represented by the Y-axis. A graph of the effective areas, A_{eT}, is plotted against the applied pressure and the method of list squires straight line is used to fit a straight line to the results. The gradient gives the difference in the distortion coefficients and the intercept with the abscissa gives A_0, the effective area extrapolated to zero applied pressure. For *n* calibration points intercept of the best-fit straight line with the abscissa i.e. A_0 is calculated as:

$$A_{0T} = \frac{1}{n}.[\sum_{i=1}^{n} A_{Si} - b.\sum_{i=1}^{n} P_{Si}] \tag{13}$$

where *b* is the gradient of best fit straight line given by:

$$b = \frac{n\sum_{i=1}^{n}(p_{Si}.A_{Si}) - \sum_{i=1}^{n} p_{Si}.\sum_{i=1}^{n} A_{Si}}{n.\sum_{i=1}^{n}(P_{Si}^2) - (\sum_{i=1}^{n} P_{Si})^2} \tag{14}$$

Then the pressure distortion coefficient could be obtained from

$$\lambda = b / A_{0T} \tag{15}$$

A calibration of 500 MPa PCA has been carried out using other 500 MPa PCA as previously described. Results of calibrations are shown in Fig. 7. The zero pressure effective area of the unit under test, A_{0T}, as a result of the fit, including the standard uncertainty of the fitting u_{fit} (A_{0T}), the gradient of best fit straight line b and the associated uncertainty are:

$A_{0T} = 1.961760321$ mm^2
$u_{fit}(A_{0T}) = 2.07053\text{x}10^{-6}$ mm^2
$\lambda = 9.07\text{x}10^{-7}$ MPa^{-1}
$u_{fit}(\lambda) = 8.0\text{x}10^{-9}$ MPa^{-1} .

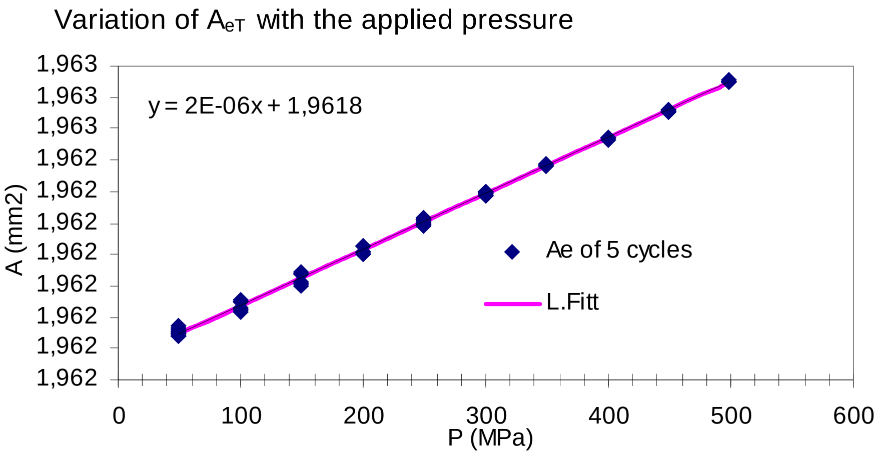

Fig. 7. AeT vs. PS in the range from 50 MPa to 500 MPa.

Fig. 8 shows the residual of the results from the straight line was less than 15 ppm which confirms a good performance of the cross floating experiment.

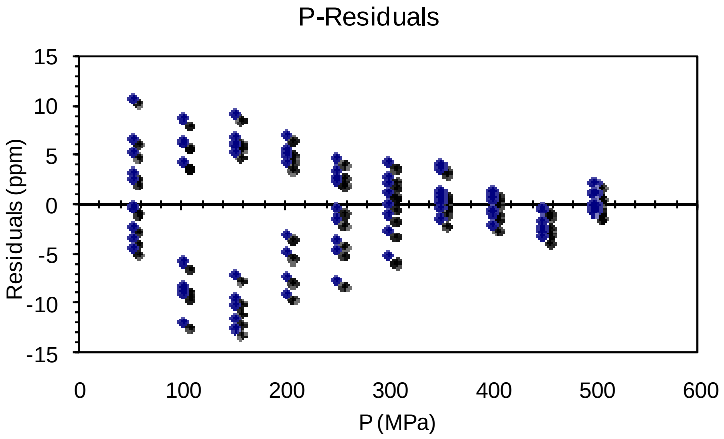

Fig. 8. Residuals of AeT from Linear fit.

5.5. Uncertainty Estimation

According to the mathematical model the uncertainty of the A_{0T}, $u(A_{0T})$, is estimated first as type A and type B. Type A is taken from the results of the experiment as described previously. Type B is calculated taking into account the following quantities:

- uncertainty of the masses, $u(m)_S$;
- uncertainty of the standard PCA effective area, $u(A)_S$;
- uncertainty of the pressure distortion coefficient, $u(\lambda)_S$;
- uncertainty of the temperature of the standard $u(\Delta t)_S$;
- uncertainties of thermal expansion coefficients of piston and cylinder, $u(2\alpha)_S$;
- uncertainty of the density of the load, $u(\rho m)_S$;
- uncertainty of the air buoyancy, $u(\rho a)_S$;
- uncertainty of the head correction, $u(\Delta h)$;
- uncertainty of the surface tension, $u(\sigma)_S$.

Most of the above mentioned uncertainties are taken from calibration report except uncertainty values of thermal expansion coefficient and densities of the used masses which are obtained from manufacturer or could be found in literature. Table 1 gives type B uncertainty budget for calibration of PCA of range 500 MPa. It could be noticed that there are many contributions which are not significant values and could be ignored from the calculations such as ρmi, ρPCA, ρa, Verticality, Δh, piston position and g. It is clear that such insignificant circumstance is not a must but required much of care in order to be achieved.

Table 1. Type B uncertainty budget for calibration of PCA of range 500 MPa.

Parameter	**Uncertainty of the quantity**	**Distribution**	**Standard uncertainty u(xi)**	**Sensitivity coefficient**	**Standard uncertainty ui(p)=c_i*u(xi)**
m_i	1.50E-04	2	p/m	5.00E+06	3.75E+02
Drift in m_i	5.00E-06	1.732	p/m	5.00E+06	1.44E+01
ρ_{mi}	3.96E+00	1.732	- A_{0s} / ρ_{mi}^2x P	1.56E-05	3.57E-05
ρ_{PCA}	9.94E+00	1.732	- A_{0s} / ρ_{PCA}^2x P	9.93E-06	5.70E-05
ρ_a	2.00E-04	2	ρ_{mi}	7.90E+03	7.90E-01
A_{0s}	3.53E-11	2	- p/ A_{0s}	2.55E+14	4.50E+03
Drift in A_{0s}	1.96E-11	1.732	- p/ A_{0s}	2.55E+14	2.89E+03
λ	1.00E-13	1.732	-P^2 / (1+ λ *p)	2.50E+17	1.44E+04
T	3.40E-02	1.732	-(α_p+α_c)*p/ (1+ (α_p+α_c))*(t-20))	4.50E+03	8.83E+01
αp+ αc	9.00E-08	1.732	-(t-20)*p/ (1(α_p+ α_c)*(t-20))	0.00E+00	0.00E+00
Verticality	2.90E-04	1.732	tanθ x p	1.45E+05	2.43E+01
Δh	1.00E-03	1.732	ρ_f x g	9.12E+03	5.27E+00
Piston position	1.00E-04	1.732	ρ_f x g	9.12E+03	5.27E-01
g	1.96E-07	2	p/g	5.11E+07	5.00E+00

Fig. 9 shows histogram of the different contributions having significant effect on the expanded uncertainty namely the distortion coefficient, zero pressure effective area of the standard PCA and its drift, used masses and sensitivity of cross floating.

The elastic distortion coefficient is the main source of uncertainty at high pressure, from 350 MPa up to 500 MPa. In the other range the zero pressure effective area is also an important source of uncertainty.

5.6. Level (III) Reference or Transfer Standards and Level (IV)

Calibration of transfer standard was carried out using the National Standard in the range from 0 to 100 MPa. The sensing element of the monitor is a precision quartz crystal resonator. The frequency of oscillation varies with pressure induced stress. Nominal accuracy of the sensor was indicated as 0.008 % of full scale.

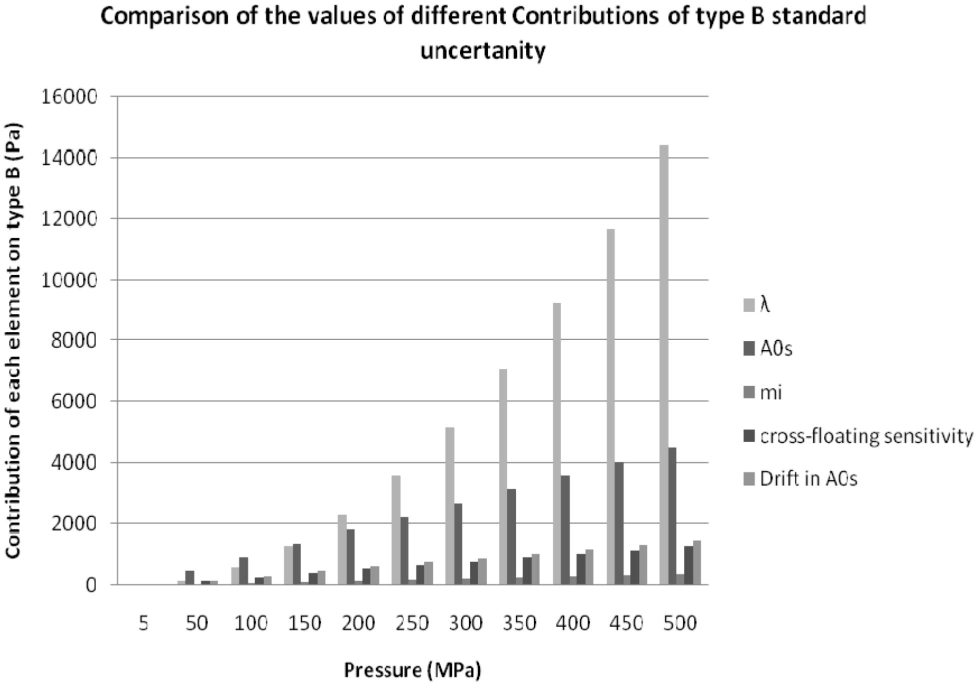

Fig. 9. Comparison of the values of the five significant sources contributed to type B uncertainty at applied pressures.

At nominal pressures of 0, 10, 20, 30, 40, 50, 60, 70, 80, 90, and 100 MPa, the differences between the pressure applied and the reading of the pressure monitor were examined. The values of the difference, together with the respective measurement uncertainties, were obtained. Atmospheric temperature, relative humidity and atmospheric pressure are measured. After applying the pressure, keep the pressure to stabilize for 20 minutes. The position of the piston of the National Standard was kept in the floating range. After a waiting time of 10 minutes, the reading of each pressure monitor, which is the average of 20 measurements and its corresponding standard deviation σ, are obtained.

The applied pressure with the associated standard uncertainty [*k*=1] at the reference level of the transfer standard is to be calculated. The correction by the differential height of the reference level between the national standard and the transfer standard was considered. One complete measurement cycle consists of recording the pressure monitor readings for the 11 points from 0 to 100 MPa for ascending pressures, then 11 points from 100 to 0 MPa descending pressures. The ascending pressure measurement cycle must start from 0 MPa while the descending pressure measurement cycle must start from 100 MPa. For one cycle a number of 23 measurement points was obtained. The complete calibration was considered to be five measurements cycles in order to obtain better uncertainty.

Fig. 10 shows the variation of relative error for the five measurement cycles. The maximum relative variation was found to be 6 ppm**.** Uncertainty was calculated as type A and Type B. Type A uncertainty is calculated from the standard deviation of the measurements then dividing the standard deviation by the number of measurements (5 in our case). Type B is calculated at each calibration point. Fig. 11 shows the variation of the relative error of the mean measured pressure associated with the uncertainty of measurements.

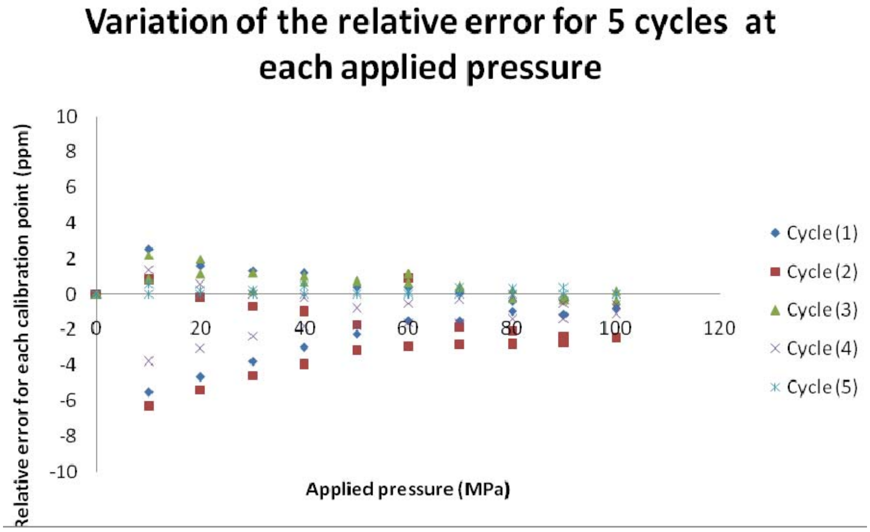

Fig. 10. Variation of relative error of the five measurement cycles.

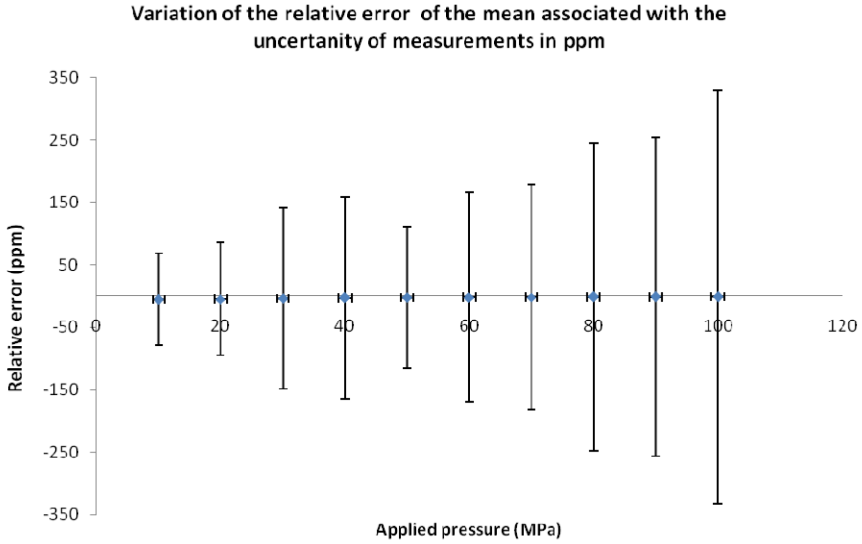

Fig. 11. Relative error of the mean measured pressure from the applied pressure.

6. Conclusions

- Two separate methods were successfully implemented for the determination of A_0 of PCA from dimensional data with excellent agreement; within 3.5 ppm.
- It could be demonstrated that uncertainty calculation procedure was used in different traceability levels, primary level, national standards level and working or transfer standards level with successful application at each level.
- To reduce uncertainty below the obtained limits, specially at high pressure calibration, it is highly required to study the pressure distortion coefficient deeply since it is almost the dominant value in high pressure calibration. Using controlled clearance PCA would be a good selection to reduce the effect of pressure distortion coefficient.

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Guide for Contributors

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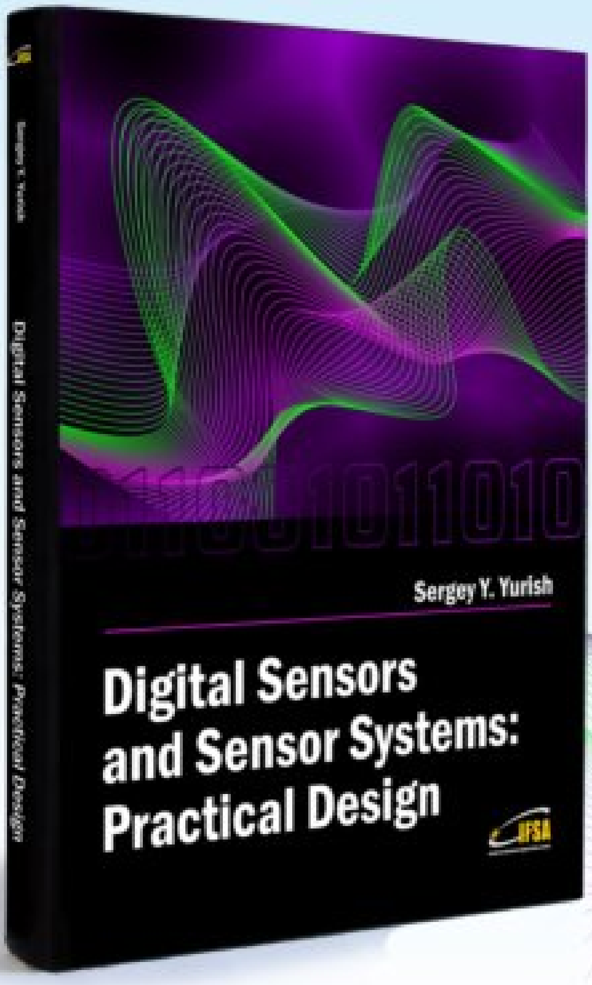
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