DISSEMINATION OF THE NATIONAL STANDARD OF MASS FROM INACAL USING THE GAUSS MARKOV METHOD BY GENERALIZED LEAST SQUARES

Author: Donny Taipe A.

Instituto Nacional de Calidad, Calle de la Prosa N°150, San Borja, Lima- Peru

Summary: This article sustains the transfer of the national standard of mass (KP1) of INACAL to two reference standards ‘Weight 1’, ‘Weight 2’ and also KP2 (as witnessed mass standard and with known error). The dissemination was done using the Gauss Markov method by Generalized Least Squares. The uncertainty calculation was performed using Univariate Gaussian Distribution and Multivariate Gaussian Distribution; the latter was developed with the Monte Carlo method using a programming language called 'R software'.

Keywords: Dissemination, variance, univariate, multivariate, Monte Carlo.

1. INTRODUCTION

This article sustains the transfer of the National Standard of Mass of Peru (KP1) consisting of a high accuracy weight of nominal value of one kilogram, joined by a witness (KP2), both of austenitic stainless steel, with an uncertainty (k=2) of 50 µg , calibrated in PTB, where the mass and volume were determinated.

In order to give traceability to mass measurements in Peru, it was carried out the transfer of the KP1 by dissemination method. The dissemination was from KP1 mass to the other three; the first KP2 (as witnessed mass standard and with known error), the second called ' Weight 1' and the third called 'Weight 2'.

One way to evaluate the coherence of our results would compare them with the known values of KP2.

To transfer the mass unit, the Mass Laboratory of INACAL has a high accuracy mass automatic comparator, used as a transfer way; and also, a high accuracy weather control instrument, to know the effects of the magnitudes of influence of environmental conditions during the calibration process.

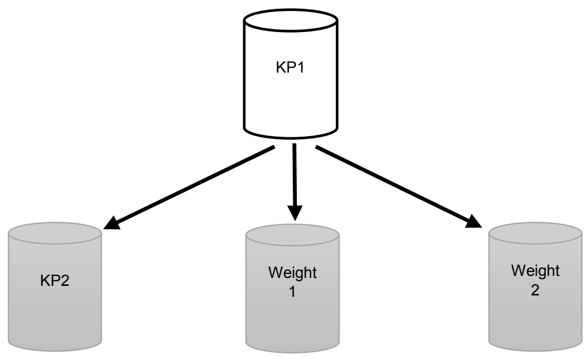

Fig. N°.1. Transfer Diagram KP1

2. DESCRIPTION OF THE TRANSFER OF THE NATIONAL STANDARD OF MASS

Since INACAL has a National Standard of Mass, it is necessary to transfer the mass unit to reference standard, witness standard and / or working standard (diagram in Figure N° 1).

The mass unit transfer is performed by dissemination method. To apply this method the technical specification of the used instruments must be known and evaluate its enough accuracy to achieve desired results. An automatic comparator, with a standard deviation of 2 µg (Mettler Toledo AX1006) was used for measurements, and a high accuracy weather control instrument, which has an standard deviation in temperature <0.01 ° C , was used to monitor the influence of environmental conditions.

FIG. 2 National Standard Mass of Peru (KP1) and control (KP2)

3. METHOD OF DISSEMINATION

The method of dissemination is performed by the Gaussian Markov method by generalized least squares.

The theoretical regression model is:

$$Y = X\hat{\beta} + e \tag{1}$$

The estimator of generalized least squares (GLS) is defined as:

$$\hat{\beta}_{GLS} = \left[X^T \phi^{-1} X\right]^{-1} X^T \phi^{-1} Y \tag{2}$$

We get the transformed model that satisfies all the assumptions of Markov Gauss theorem as follows:

$$V(\hat{\beta}) = \left[X^T \phi^{-1} X\right]^{-1} \tag{3}$$

Calculation of uncertainty of the components Y

For estimate and calculate measurement uncertainty we will use the guidelines established in the Guide to the Expression of Uncertainty in Measurement.

Disturbances due to: repeatability, eccentricity of the balance, scale resolution, sensitivity factor, standard weight, derived from the standard weight, air density and volume of the weights have been considered in the following equation.

$$y = I.f_s + (m_P + \delta_P).X_{Pi} + \rho_{ai}.\Delta V + \varepsilon_i \tag{4}$$

Analyzing each contribution of uncertainty of the equation (4), we obtain:

$$\begin{aligned} u^2(y) &= f_s^{\,2}.\left(u^2(resl) + u^2(rept)\right) + I^2.\,u^2(f_s) + \\ &\left(u^2(m_P) + u^2(\delta_P)\right)X_P^2 + \Delta V^2.\,u^2(\rho_a) + \\ &\rho_a^{\,2}(u^2(V_+) - u^2(V_-)) + \sigma^2 \end{aligned} \tag{5}$$

Turning the equation (4) and (5) into its matrix form:

$$Y = I.f_s + (m_P + \delta_P).X_P + \boldsymbol{\rho}_\mathbf{a} \otimes \boldsymbol{\Delta} V + \boldsymbol{\varepsilon} \quad (6)$$

$$\begin{aligned} Var(Y) &= f_s^{\,2}.\left(Var(resl) + Var(rept)\right) + \\ &(\mathbf{I}\ \mathbf{I}^\mathbf{T}).\,Var(f_s) + \mathbf{X_P X_P}^T\left(Var(m_P) + Var(\delta_P)\right) + \\ &\boldsymbol{\Delta}\mathbf{V}\ \boldsymbol{\Delta}\mathbf{V}^\mathbf{T}.\,Var(\rho_a) + \rho_a^{\,2}(Var(\Delta V)) + \sigma^2\mathbf{I} \end{aligned} \quad (7)$$

where:

- $Var(Y) = \phi$
- f_s : sensitivity factor
- $Var(resl) = diag\ matrix(d_i^2/12)$
- $Var(rept) = diag\ matrix(s_i^2/\sqrt{n_i})$
- $Var(f_s) = diag\ matrix(u^2(f)) \rightarrow$ Negligible
- $\left(Var(m_P) + Var(\delta_P)\right)X_P = = (u^2(p) + \delta/\sqrt{3})\begin{bmatrix} 0 & 0 & \cdots & 0 \\ 0 & 0 & & 0 \\ \vdots & & \ddots & \vdots \\ 0 & 0 & \cdots & 1 \end{bmatrix}$
- $\Delta V = diag\ matrix\left[V_{Pi} + V_{Xi}\right]$
- $Var(\rho_a) = matriz\ diag(u^2(\rho_{ai}))$
- If air density in each comparison measurement is obtained then the variance is as follows $Var(\Delta V) = Cov(\Delta V_i;\Delta V_j)$

$$\sigma^2 I = \frac{(Y_i - X_i\beta)(Y_i - X_i\beta)^T}{n-p}.I$$

In case of no covariance effects in $V(\hat{\beta})$, then it is reduced to a Univariate distribution function and it is given by the square root of the diagonal matrix.

If there are covariance effects, it is assigned as a multivariate Gaussian distribution $N(\beta, U_\beta)$ a $\hat{\beta}$.

where math expectation and covariance matrix are:

$E(\hat{\beta}) = \beta$ and $V(\hat{\beta}) = U_\beta$

Then we need to sample $N(\beta, U_\beta)$, and for this we require $n = 4$ independent values for $\boldsymbol{z}$ from the typical Gaussian distribution $N(0,1)$, which it is formed by:

$$\varepsilon_i = \beta_i + R^T z_i \quad (8)$$

where $\boldsymbol{z} = (z_1, z_2, z_3, z_4)^T$ and R is the upper triangular matrix given by the Cholesky decomposition $U_\beta = R^T R$

where the $z_1, z_2, z_3\ y\ z_4$ are generated independent random numbers with r_1 and r_2 from a rectangular distribution $R(0,1)$ and thereby we calculate

$$z_i = \sqrt{-2ln\, r_1}\cos 2\pi r_2 \qquad (9)$$

It is therefore advisable to use a programming language. In our case we use and recommend the 'software R'.

4 DEVELOPMENT:

Corresponding data for each weight:

	VN (kg)	Corr. (mg)	u(k=1) (mg)	Vol. (cm^3)	U (Vol.) (cm^3)
KP1	1	-0,029	0,025	124,8199	0,0010
KP2	1	-0.076	0.025	124,8208	0,0010
Weight1	1			124,8000	0,0020
Weight2	1			124,7716	0,0015

Table 1

Weighing the following scheme where has 6 equations weighing and 3 unknown mass (KP2, Weight 1 and Weight 2) are used.

$$\begin{array}{cc} & \begin{array}{cccc} \textbf{KP1} & \textbf{KP2} & \textbf{Weight1} & \textbf{Weight 2} \end{array} \\ X\,\hat{\beta} = & \begin{pmatrix} -1 & 1 & 0 & 0 \\ -1 & 0 & 1 & 0 \\ -1 & 0 & 0 & 1 \\ 0 & -1 & 1 & 0 \\ 0 & -1 & 0 & 1 \\ 0 & 0 & -1 & 1 \end{pmatrix} \begin{pmatrix} \beta_1 \\ \beta_2 \\ \beta_3 \\ \beta_4 \end{pmatrix} \end{array}$$

The '-1' represents the pattern and '1' represents the sample for a weighing sequence.

6 series were performed (each series has weighing sequence "ABBA"), these 6 series correspond to an equation weighing.

Recording of the data taken in the calibration process (table 2):

$\Delta\bar{m}$ mg	ρ_a mg/cm3	u_{ρ_a} mg/cm^3	$V_P - V_S$ cm^3	Y_i mg	$S_{\Delta\bar{m}}$
-0,0421	1,1656	94,5x10^{-5}	0,0009	-0,0411	0,00061
0,1343	1,1654	94,5x10^{-5}	-0,0199	0,1112	0,00094
-0,1196	1,1653	94,5x10^{-5}	-0,0483	-0,1760	0,00156
0,1738	1,1646	94,5x10^{-5}	-0,0208	0,1496	0,00075
-0,0703	1,1638	94,5x10^{-5}	-0,0492	-0,1276	0,00076
-0,2427	1,1634	94,5x10^{-5}	-0,0284	-0,2758	0,00049

Table 2.

$\Delta\bar{m}_i$: Average of the differences of weights of each series.

$S_{\Delta\bar{m}}$:Standard deviation of the measurements of the differences of the weights.

For each Δm_i, we perform the air correction buoyancy, this will be given by the following equation:

Were: $Y_i = \Delta\bar{m}_i + \rho_{ai}(V_p - V_s)_i$

We estimate ∅ according to equation (7), considering the reference value correction KP1, then we obtain the equation (2) y (3).

Substituting, we have:

$$\hat{\beta}_{GLS} = \left[X^T \phi^{-1} X\right]^{-1} X^T \phi^{-1} Y = \begin{pmatrix} \beta_1 = -0.02900 \text{ mg} \\ \beta_2 = -0.07136 \text{ mg} \\ \beta_3 = 0.07858 \text{ mg} \\ \beta_4 = -0.20050 \text{ mg} \end{pmatrix}$$

- o The univariate Gaussian distribution for uncertainty is given by equation (3)
- o Figuring multivariate Gaussian distribution for uncertainty, we do the following:

We find R, where R is the upper triangular matrix decomposition Cholesky ($V(\hat{\beta}) = R^T R$)

We can use a *command* in a programming language that allows us to get that value. In our case we use the 'Software R'

Independently generate random numbers r_1 and r_2 and obtain the 4 separate values for $\boldsymbol{z}$, Replaced in the following equation (9) and obtain $\varepsilon_1, \varepsilon_2, \varepsilon_3 \ y \ \varepsilon_4$:

The uncertainty is given by the standard deviation of ε_i

$$u_{\beta_1}(k = 1) = 0{,}02623 \text{ mg}$$

$$u_{\beta_2}(k = 1) = 0{,}02628 \text{ mg}$$

$$u_{\beta_3}(k = 1) = 0{,}02635 \text{ mg}$$

$$u_{\beta_4}(k = 1) = 0{,}02632 \text{ mg}$$

5. CONCLUSION:

- The difference between the result obtained of KP2 and its calibration certificate is less than 5 µg, which is less than its uncertainty specified in the calibration certificate.
- The Gauss Markov method allows the Mass Laboratory of INACAL to obtain appropriate uncertainties of class E1 (the uncertainty obtained for the ‘weight 1’ and ‘weight 2’ is 26 µg)
- The Mass Laboratory of INACAL is capable of calibrating weights of Class E1.

6. REFERENCIAS

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