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DETERMINATION OF THE UNCERTAINTIES AND COVARIANCES IN THE CALIBRATION OF THE SET OF WEIGHTS[1]

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Abstract

The paper shows a calibration model where a set of weights is calibrated in terms of the mass of a single reference weight. It is proved that under satisfying a simple condition the uncertainty of the reference mass value does not influence the mass values of the unknown weights, i.e. the calibration procedure is equivalent to a weighted least squares analysis. A simplified example demonstrates this calibration process.

1. Introduction

Estimated values of individual weights and their uncertainties are presented in a calibrating certificate of a set of weights. Covariances among estimated mass values of individual weights are quoted rarely. This fact does not introduce any problem for the use of individual weights but it can bring many problems in the common use of a calibrated set of weights. Namely, by common use of weights some new values can be realized (e.g. the sum of individual weights). Uncertainty of such a sum is then represented by uncertainties of the weights used, as well as by the covariances among them. Neglection of covariances is quite often explained by the orthogonality of the experiment's design. At the same time it is obvious that the use of an experiment with an orthogonal design matrix does not imply zero covariances (diagonal covariance matrix of the weights masses estimations) in the case of a common

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influence to all measurements. Although this facts has been extensively reported in the literature, and even in written standards (see e.g. [1], [5], [7], [8], [9]), the paper will present a new aspect in the assignment of mass values and associated covariances to a set of weights in term of the mass of a single reference weight. Under satisfying a simply condition the uncertainty of the reference mass value does not influence the mass values of the unknown weights, but only has to be taken into consideration, when calculating the covariance matrix of the estimated mass values. In Appendix is given the mathematical proof of this fact. So it is shown that a intuitively correct widely used procedure is in fact equivalent to a weighted least squares analysis, in which the variance of the mass value of the reference weight is included in the covariance matrix of the observations. On a simplified example dealing with the subdivision of the kilogram according to a specific design we demonstrate above mentioned facts.

2. Preliminary considerations

National standards laboratories need to perform calibration of the mass scale for a minimum range from 1 mgto 10 kg. The task is performed by the calibration of a set of weights using a one- kilogram standard (compared with a copy of the International Prototype). This procedure (so called *calibrating method*) is based on the performing of more comparisons than is the number of weights calibrated and the task can be solved using a mathematical statistics method (least-squares method). In the further standardization we can utilize the calibration method (using some of the standards) or the direct comparison of the calibrated weight with the standard. In this paper only calibration of the set of weights, not the calibration of individual weights will be discused.

3. Calibration model

A set of k weights with masses $\beta_1, \beta_2, \ldots, \beta_k$ is considered. They are to be calibrated by the standard with the mass m_E using n comparative weightings $(n \geq k)$ with different combinations of all weights from the set, according to the a priori selected calibration scheme. Values of the mass differences are $m_1, m_2, \ldots, m_n$. Further let $K_E, K_{\beta_1}, \ldots, K_{\beta_k}$ are the buoyancy corrections of the standard and calibrated weights, respectively. Of course,

$$K_E = V_E \rho_A, \;\; K_{\beta_i} = V_{\beta_i} \rho_A, \;\; i = 1, 2, \ldots, k,$$

where $V_E, V_{\beta_1}, \ldots, V_{\beta_k}$ are the volumes of the standard and calibrated weights, respectively and ρ_A is the air density.

For example let us have calibration scheme as follows

$$
\begin{array}{llllllllllllllllll}
m_1 & +m_E-K_E & = \beta_1 & +\beta_2 & +\beta_3 & +\beta_4 & & -K_{\beta_1} & -K_{\beta_2} & -K_{\beta_3} & -K_{\beta_4} & \\
m_2 & +m_E-K_E & = \beta_1 & +\beta_2 & +\beta_3 & & +\beta_5 & -K_{\beta_1} & -K_{\beta_2} & -K_{\beta_3} & & -K_{\beta_5} \\
m_3 & & = \beta_1 & -\beta_2 & -\beta_3 & -\beta_4 & & -K_{\beta_1} & +K_{\beta_2} & +K_{\beta_3} & +K_{\beta_4} & \\
m_4 & & = \beta_1 & -\beta_2 & -\beta_3 & & -\beta_5 & -K_{\beta_1} & +K_{\beta_2} & +K_{\beta_3} & & +K_{\beta_5} \\
m_5 & & = & \beta_2 & -\beta_3 & +\beta_4 & -\beta_5 & & -K_{\beta_2} & +K_{\beta_3} & -K_{\beta_4} & +K_{\beta_5} \\
m_6 & & = & \beta_2 & -\beta_3 & -\beta_4 & +\beta_5 & & -K_{\beta_2} & +K_{\beta_3} & +K_{\beta_4} & -K_{\beta_5} \\
m_7 & & = & \beta_2 & -\beta_3 & & & & -K_{\beta_2} & +K_{\beta_3} & & \\
m_8 & & = & \beta_2 & -\beta_3 & & & & -K_{\beta_2} & +K_{\beta_3} & & \\
m_9 & & = & \beta_2 & & -\beta_4 & -\beta_5 & & -K_{\beta_2} & & +K_{\beta_4} & +K_{\beta_5} \\
m_{10} & & = & \beta_2 & & -\beta_4 & -\beta_5 & & -K_{\beta_2} & & +K_{\beta_4} & +K_{\beta_5} \\
m_{11} & & = & & \beta_3 & -\beta_4 & -\beta_5 & & & -K_{\beta_3} & +K_{\beta_4} & +K_{\beta_5} \\
m_{12} & & = & & \beta_3 & -\beta_4 & -\beta_5 & & & -K_{\beta_3} & +K_{\beta_4} & +K_{\beta_5} \\
m_{13} & & = & & & \beta_4 & -\beta_5 & & & & -K_{\beta_4} & +K_{\beta_5} \\
m_{14} & & = & & & \beta_4 & -\beta_5 & & & & -K_{\beta_4} & +K_{\beta_5}
\end{array}
\tag{1}
$$

($m_E \approx 1\,\mathrm{kg}$, $\beta_1 \approx 500\,\mathrm{g}$, $\beta_2 \approx 200\,\mathrm{g}$, $\beta_3 \approx 200\,\mathrm{g}$, $\beta_4 \approx 100\,\mathrm{g}$, $\beta_5 \approx 100\,\mathrm{g}$). Generally is assumed that all weights as well as measuring standard are manufactured from different materials. Practically set of weights is manufactured from one material and measuring standard from the different one most often. Then corrections on the air buoyancy are presented only in first two equations of (1). For set of weights and measuring standard being produced from the same material correction on the air buoyancy will not be presented in any equation (see example). Denoting

$$
\boldsymbol{\beta} = \begin{pmatrix} \beta_1 \\ \beta_2 \\ \beta_3 \\ \vdots \\ \beta_k \end{pmatrix}, \quad \boldsymbol{m} = \begin{pmatrix} m_1 \\ m_2 \\ m_3 \\ \vdots \\ m_n \end{pmatrix}, \quad \boldsymbol{c}_{n,1} = \begin{pmatrix} 1 \\ 1 \\ 0 \\ \vdots \\ 0 \end{pmatrix}, \quad \boldsymbol{k}_\beta = \begin{pmatrix} K_{\beta_1} \\ K_{\beta_2} \\ K_{\beta_3} \\ \vdots \\ K_{\beta_k} \end{pmatrix}, \tag{2}
$$

and

$$\boldsymbol{A}_{n,k} = \begin{pmatrix} 1 & 1 & 1 & 1 & 0 \\ 1 & 1 & 1 & 0 & 1 \\ 1 & -1 & -1 & -1 & 0 \\ 1 & -1 & -1 & 0 & -1 \\ 0 & 1 & -1 & 1 & -1 \\ 0 & 1 & -1 & -1 & 1 \\ 0 & 1 & -1 & 0 & 0 \\ 0 & 1 & -1 & 0 & 0 \\ 0 & 1 & 0 & -1 & -1 \\ 0 & 1 & 0 & -1 & -1 \\ 0 & 0 & 1 & -1 & -1 \\ 0 & 0 & 1 & -1 & -1 \\ 0 & 0 & 0 & 1 & -1 \\ 0 & 0 & 0 & 1 & -1 \end{pmatrix}, \tag{3}$$

($\boldsymbol{A}$ being $n \times k$ design matrix with only elements $0, 1$, or -1, for calibration scheme (1) $n = 14$, $k = 5$), above mentioned mathematical (theoretical) model of calibration can be written as

$$\boldsymbol{m} + \boldsymbol{c}m_E - \boldsymbol{c}K_E = \boldsymbol{A\beta} - \boldsymbol{AK}_\beta.$$

In general, for an arbitrary $n \times k$ design matrix $\boldsymbol{A}$ and $n \times 1$ vector $\boldsymbol{c}$

$$\boldsymbol{m} + \boldsymbol{c}(m_E - K_E) + \boldsymbol{AK}_\beta = \boldsymbol{A\beta} \tag{4}$$

is the *mathematical model* of calibration (no randomness in model (4) is supposed). It is seen that (4) is a little simplified model of calibration involving only buoyancy correction. Other influences e.g. heat expansion etc. are here neglected.

The values of the mass differences $m_1, m_2, \ldots, m_n$ are estimated as results of weighting. Let $\boldsymbol{X}_{n,1}$ be the random vector which realization is $\boldsymbol{x} = (x_1, x_2, \ldots, x_n)^T$, x_i being the result of $i-$th weighting, $i = 1, 2, \ldots, n$. We suppose the mean value

$$\mathrm{E}(\boldsymbol{X}) = \boldsymbol{m}$$

and its covariance matrix

$$\mathrm{cov}(\boldsymbol{X}) = \sigma^2 \mathrm{diag}(u_1^2, \ldots, u_n^2) = \sigma^2 \boldsymbol{H}_{n,n},$$

σ^2 is not required to be known, $\boldsymbol{H}_{n,n}$ is $n \times n$ known diagonal matrix (measurements are uncorrelated), u_i^2 is the square of uncertainty of the $i-$th used weighting (stated e.g. in the certificate). Further we consider independent unbiased measurements of the volumes $V_E, V_{\beta_1}, \ldots, V_{\beta_k}$ (e.g. hydrostatic weightings) and of ρ_A. If we denote $\boldsymbol{\xi} = (\xi_E, \xi_1, \ldots, \xi_k)^T$ the random vector which realization is $\boldsymbol{v} = (v_E, v_{\beta_1}, \ldots, v_{\beta_k})^T$, where $v_E, v_{\beta_1}, \ldots, v_{\beta_k}$ are the measured values of volumes $V_E, V_{\beta_1}, \ldots, V_{\beta_k}$), then

$$\mathrm{E}(\boldsymbol{\xi}) = \begin{pmatrix} V_E \\ V_{\beta_1} \\ \vdots \\ V_{\beta_k} \end{pmatrix} \quad \text{and} \quad \mathrm{cov}(\boldsymbol{\xi}) = \begin{pmatrix} \Delta_E^2 & 0 & & 0 \\ 0 & \Delta_{\beta_1}^2 & & 0 \\ & & \ddots & \\ & & & \Delta_{\beta_k}^2 \end{pmatrix} = \boldsymbol{\Delta}$$

($\Delta_e, \Delta_{\beta_1}, \ldots, \Delta_{\beta_k}$ are known or estimated standard uncertainties). Finally let ζ be a random variable which realization ρ is the measured value of ρ_A, i.e. $\mathrm{E}(\zeta) = \rho_A$ and its dispersion is Δ_ρ^2 (known). As $\boldsymbol{\xi}$ and ζ are uncorrelated, we can (e.g. using [3] or [10]) establish the mean value and covariance matrix of the random vector $\boldsymbol{\eta} = (\xi_E\zeta, \xi_1\zeta, \ldots, \xi_k\zeta)^T$ as

$$\mathrm{E}(\boldsymbol{\eta}) = \begin{pmatrix} V_E\rho_A \\ V_{\beta_1}\rho_A \\ \vdots \\ V_{\beta_k}\rho_A \end{pmatrix} = \begin{pmatrix} K_E \\ K_{\beta_1} \\ \vdots \\ K_{\beta_k} \end{pmatrix} = \begin{pmatrix} K_E \\ \boldsymbol{K}_\beta \end{pmatrix}, \quad \mathrm{cov}(\boldsymbol{\eta}) = \Delta_\rho^2 \boldsymbol{v}\boldsymbol{v}^T + \rho^2 \boldsymbol{\Delta} = \boldsymbol{N}.$$

The set of weights $\beta_1, \beta_2, \ldots, \beta_k$ is calibrated by the standard weight. Value reproduced by the standard weight is referred to as x_E (known) with given standard uncertainty (standard deviation) u_E. So x_E can be considered as the realization of the random variable X_E with $\mathrm{E}(X_E) = m_E$ and its dispersion is u_E^2. For our calibration are available

realization of the random vector $\boldsymbol{X}$

realization of the random vector $\boldsymbol{\eta}$ and

realization of the random variable X_E.

We only note that $\boldsymbol{X}$, $\boldsymbol{\eta}$ and X_E are uncorrelated.

From the mathematical model of calibration (4) we obtain using $\boldsymbol{X}$, $\boldsymbol{\eta}$ and X_E instead of $\boldsymbol{m}$, $(K_E, \boldsymbol{K}_\beta^T)^T$ and m_E so called *stochastic model* of calibration, where the observed random vector

$$\boldsymbol{Y} = \boldsymbol{X} + \boldsymbol{c}X_E + (-\boldsymbol{c} \vdots \boldsymbol{A})\boldsymbol{\eta} \tag{5}$$

has its mean value

$$\mathrm{E}(\boldsymbol{Y}) = \boldsymbol{A}\boldsymbol{\beta}$$

and covariance matrix

$$\begin{aligned}\operatorname{cov}(\boldsymbol{Y}) &= (\boldsymbol{I}_{n,n} \vdots \boldsymbol{c} \vdots - \boldsymbol{c} \vdots \boldsymbol{A}) \begin{pmatrix} \sigma^2 \boldsymbol{H} & 0 & \mathbf{o} \\ 0 & u_E^2 & 0 \\ \mathbf{o}^T & 0 & \boldsymbol{N} \end{pmatrix} \begin{pmatrix} \boldsymbol{I} \\ \boldsymbol{c}^T \\ -\boldsymbol{c}^T \\ \boldsymbol{A}^T \end{pmatrix} \\ &= \sigma^2 \boldsymbol{H} + u_E^2 \boldsymbol{c}\boldsymbol{c}^T + (-\boldsymbol{c} \vdots \boldsymbol{A}) \boldsymbol{N} \begin{pmatrix} -\boldsymbol{c}^T \\ \boldsymbol{A}^T \end{pmatrix} = \boldsymbol{U}_{\boldsymbol{Y}}. \end{aligned} \qquad (6)$$

(We only note that this stochastic model corresponds to model (3.1) in [4].)

4. Estimation of the mass values, their uncertainties and the covariances among them

According to [10], (see also e.g. in [2], [4]) the BLUE (best linear unbiased estimator) of $\boldsymbol{\beta}$ is

$$\boldsymbol{Z} = (\boldsymbol{A}^T \boldsymbol{U}_{\boldsymbol{Y}}^{-1} \boldsymbol{A})^{-1} \boldsymbol{A}^T \boldsymbol{U}_{\boldsymbol{Y}}^{-1} \boldsymbol{Y}$$

(obtained by weighted least squares method). Its covariance matrix is

$$\boldsymbol{U}_{\boldsymbol{Z}} = (\boldsymbol{A}^T \boldsymbol{U}_{\boldsymbol{Y}}^{-1} \boldsymbol{A})^{-1}.$$

If the measurement model (calibration model) fulfils the condition

$$\boldsymbol{c} = \boldsymbol{A}\boldsymbol{q}, \qquad (7)$$

according to Corollary 2 in Appendix the BLUE $\boldsymbol{Z}$ of $\boldsymbol{\beta}$ is

$$\boldsymbol{Z} = (\boldsymbol{A}^T \boldsymbol{H}^{-1} \boldsymbol{A})^{-1} \boldsymbol{A}^T \boldsymbol{H}^{-1} \boldsymbol{Y} \qquad (8)$$

(uncertainty of the reference mass value does not influence the estimates of the unknown weights) and

$$\boldsymbol{U}_{\boldsymbol{Z}} = \sigma^2 (\boldsymbol{A}^T \boldsymbol{H}^{-1} \boldsymbol{A})^{-1} + u_E^2 \boldsymbol{q}\boldsymbol{q}^T + (-\boldsymbol{q} \vdots \boldsymbol{I}_{k,k}) \boldsymbol{N} \begin{pmatrix} -\boldsymbol{q}^T \\ \boldsymbol{I}_{k,k} \end{pmatrix}. \qquad (9)$$

If σ^2 is unknown, it is estimated according to Lemma 5 in Appendix as

$$\hat{\sigma}^2 = \frac{(\boldsymbol{X} - \boldsymbol{A}(\boldsymbol{A}^T \boldsymbol{H}^{-1} \boldsymbol{A})^{-1} \boldsymbol{A}^T \boldsymbol{H}^{-1} \boldsymbol{X})^T \boldsymbol{H}^{-1} (\boldsymbol{X} - \boldsymbol{A}(\boldsymbol{A}^T \boldsymbol{H}^{-1} \boldsymbol{A})^{-1} \boldsymbol{A}^T \boldsymbol{H}^{-1} \boldsymbol{X})}{n-k} \qquad (10)$$

and so

$$\hat{\sigma}^2 (\boldsymbol{A}^T \boldsymbol{H}^{-1} \boldsymbol{A})^{-1} + u_E^2 \boldsymbol{q}\boldsymbol{q}^T + (-\boldsymbol{q} \vdots \boldsymbol{I}_{k,k}) \boldsymbol{N} \begin{pmatrix} -\boldsymbol{q}^T \\ \boldsymbol{I}_{k,k} \end{pmatrix} \qquad (11)$$

is the unbiased estimator of the covariance matrix $\boldsymbol{U}_{\boldsymbol{Z}}$ of the estimator $\boldsymbol{Z}$.

5. Example

It is assumed that the calibration model is in the form (1) and the buoyancy correction is equal to zero (all weights as well as standard are made from the same material) and all uncertainties of weightings are equal, i.e. $\boldsymbol{H} = \boldsymbol{I}$. In this case K_E and $\boldsymbol{K}_\beta$ in (4) are zero and also so are $\boldsymbol{\eta}$ and $\boldsymbol{N}$. The observed random vector $\boldsymbol{Y}$ (see (5)) is

$$\boldsymbol{Y} = \boldsymbol{X} + \boldsymbol{c}X_E$$

($\boldsymbol{c}$ is given in (2)). Its mean value and covariance matrix are

$$\mathrm{E}(\boldsymbol{Y}) = \boldsymbol{A\beta}, \quad \mathrm{cov}(\boldsymbol{Y}) = \sigma^2\boldsymbol{I} + u_E^2\boldsymbol{cc}^T$$

($\boldsymbol{A}$ is given in (3), so $n = 14$ and $k = 5$).

As for $\boldsymbol{q} = (5/10, 2/10, 2/10, 1/10, 1/10)^T$ is $\boldsymbol{Aq} = \boldsymbol{c}$, the calibration model fulfils the condition (7). According to (8) the BLUE of $\boldsymbol{\beta}$ is $\boldsymbol{Z} = (\boldsymbol{A}^T\boldsymbol{A})^T\boldsymbol{A}^T\boldsymbol{Y}$, so the estimators of individual weights are

$$\begin{aligned}
Z_1 &= 0.25(X_1 + X_2 + X_3 + X_4 + 2X_E)\\
Z_2 &= 0.1(X_1 + X_2 - X_3 - X_4 + X_5 + X_6 + X_7 + X_8 + X_9 + X_{10} + 2X_E)\\
Z_3 &= 0.1(X_1 + X_2 - X_3 - X_4 - X_5 - X_6 - X_7 - X_8 + X_{11} + X_{12} + 2X_E)\\
Z_4 &= 0.1(X_1 - X_3 + X_5 - X_6 - X_9 - X_{10} - X_{11} - X_{12} + X_{13} + X_{14} + X_E)\\
Z_5 &= 0.1(X_2 - X_4 - X_5 + X_6 - X_9 - X_{10} - X_{11} - X_{12} - X_{13} - X_{14} + X_E). \qquad (12)
\end{aligned}$$

The standard uncertainties of the calculated values of the masses and their covariances, i.e. the covariance matrix $\boldsymbol{U_Z}$ is (according to (9) with $\boldsymbol{N} = \mathbf{o}$)

$$\boldsymbol{U_Z} = \sigma^2(\boldsymbol{A}^T\boldsymbol{A})^{-1} + u_E^2\boldsymbol{qq}^T.$$

For given $\boldsymbol{A}$ and $\boldsymbol{q}$ it holds

$$\boldsymbol{U_Z} = \frac{\sigma^2}{100}\begin{pmatrix} 25 & 0 & 0 & 0 & 0\\ 0 & 10 & 0 & 0 & 0\\ 0 & 0 & 10 & 0 & 0\\ 0 & 0 & 0 & 10 & 0\\ 0 & 0 & 0 & 0 & 10 \end{pmatrix} + \frac{u_E^2}{100}\begin{pmatrix} 25 & 10 & 10 & 5 & 5\\ 10 & 4 & 4 & 2 & 2\\ 10 & 4 & 4 & 2 & 2\\ 5 & 2 & 2 & 1 & 1\\ 5 & 2 & 2 & 1 & 1 \end{pmatrix}. \qquad (13)$$

If σ^2 is unknown, it is estimated from (10). In this case

$$\hat{\sigma}^2 = \frac{1}{14-5}\sum_{i=1}^{14}(X_i - \tilde{X}_i)^2, \qquad (14)$$

where $\tilde{X}_i$ is the $i-$th element of the random vector $\boldsymbol{AZ} - \boldsymbol{c}X_E$ (according to (10) and Lemma 4 in Appendix).

Presented procedure can be illustrated using data in Table 1. The calibration certificate introduces following values for the mass standard: $x_E = (1000 - 2.82 \cdot 10^{-3})\,\mathrm{g}$, standard uncertainty $u_E = 0.07 \cdot 10^{-3}\,\mathrm{g}$.

i	1	2	3	4	5	6	7
x_i (mg)	-4.80	-3.94	4.07	3.30	-4.77	-3.06	-3.95
i	8	9	10	11	12	13	14
x_i (mg)	-3.95	-4.64	4.66	-0.73	-0.73	-0.76	-0.77

The estimated mass values (i.e. realization $\boldsymbol{z} = (z_1, z_2, \ldots, z_5)$ of the estimator $\boldsymbol{Z}$) are

$z_1 = 500\,\mathrm{g} + 1.067\,\mathrm{mg}$, $z_2 = 200\,\mathrm{g} - 3.550\,\mathrm{mg}$, $z_3 = 200\,\mathrm{g} + 0.380\,\mathrm{mg}$,

$z_4 = 100\,\mathrm{g} + 0.147\,\mathrm{mg}$, $z_5 = 100\,\mathrm{g} + 0.958\,\mathrm{mg}$ (calculated from (12)).

For our measured values is the estimate of σ^2 (its numerical value, i.e. the realization of the estimator (14)) equal to $(0.0361\,\mathrm{mg})^2$. So from (11) we obtain the covariance matrix of the estimated mass values for a reference mass uncertainty $u_E = 0.07\,\mathrm{mg}$ and $\sigma^2 = (0.0361\,\mathrm{mg})^2$ as

$$\boldsymbol{U_Z} = \frac{0,0361^2}{100}\begin{pmatrix} 25 & 0 & 0 & 0 & 0 \\ 0 & 10 & 0 & 0 & 0 \\ 0 & 0 & 10 & 0 & 0 \\ 0 & 0 & 0 & 10 & 0 \\ 0 & 0 & 0 & 0 & 10 \end{pmatrix} + \frac{0.07^2}{100}\begin{pmatrix} 25 & 10 & 10 & 5 & 5 \\ 10 & 4 & 4 & 2 & 2 \\ 10 & 4 & 4 & 2 & 2 \\ 5 & 2 & 2 & 1 & 1 \\ 5 & 2 & 2 & 1 & 1 \end{pmatrix}$$

$$= 10^{-4}\begin{pmatrix} 15.51 & 4.90 & 4.90 & 2.45 & 2.45 \\ 4.90 & 2.26 & 1.96 & 0.98 & 0.98 \\ 4.90 & 1.96 & 3.26 & 0.98 & 0.98 \\ 2.45 & 0.98 & 0.98 & 1.79 & 0.49 \\ 2.45 & 0.98 & 0.98 & 0.49 & 1.79 \end{pmatrix}.$$

(In fact it is the estimate of the covariance matrix $\boldsymbol{U_Z}$.)

Uncertainties of the estimated masses (calibrated weights) are:

$u_{500} = 0.040\,\mathrm{mg}$, $u_{200} = 0.018\,\mathrm{mg}$, $u_{200^*} = 0.018\,\mathrm{mg}$, $u_{100} = 0.014\,\mathrm{mg}$, $u_{100^*} = 0.014\,\mathrm{mg}$.

Covariances among the estimated masses are:

$u_{500,200} = 0.000\,49\,\mathrm{mg}^2$, $u_{500,200^*} = 0.000\,49\,\mathrm{mg}^2$, $u_{500,100} = 0.000\,245\,\mathrm{mg}^2$,

$u_{500,100^*} = 0.000\,245\,\mathrm{mg}^2$,

$u_{200,200} = 0.000\,196\,\mathrm{mg}^2$, $u_{200,100} = 0.000\,098\,\mathrm{mg}^2$, $u_{200,100^*} = 0.000\,098\,\mathrm{mg}^2$,

$u_{200^*,100} = 0.000\,098\,\mathrm{mg}^2$, $u_{200^*,100^*} = 0.000\,098\,\mathrm{mg}^2$,
$u_{100,100^*} = 0.000\,049\,\mathrm{mg}^2$.

Calculated covariances are important and cannot be neglected. Even when the orthogonal calibration scheme has been used (orthogonal matrix $\boldsymbol{A}$), the covariance matrix of the calibrated weights estimations is not diagonal so that estimations are correlated. It is caused by the use of the same standard for the calibration of each weight in the set. When three calibrated weights from the set are used (for example 500, 200, 100), value of the sum of the weights will be

$$\begin{aligned} z_{500+200+100} &= 500\,\mathrm{g} + 1.067\,\mathrm{mg} + 200\,\mathrm{g} - 3.550\,\mathrm{mg} + 100\,\mathrm{g} + 0.147\,\mathrm{mg} \\ &= 800\,\mathrm{g} - 2.336\,\mathrm{mg} \end{aligned}$$

and the resulting uncertainty (calculated from the law of the uncertainties propagation)

$$\begin{aligned} u_{500+200+100} &= \sqrt{u_{500}^2 + u_{200}^2 + u_{100}^2 + 2u_{500,200} + 2u_{500,100} + 2u_{200,100}} \\ &= \sqrt{0,040^2 + 0,018^2 + 0,014^2 + 2(0.000\,49 + 0.000\,245 + 0.000\,098)} \\ &= 0.062\,\mathrm{mg}. \end{aligned}$$

In the case that covariances among the weights values are neglected, the resulting uncertainty is as follows

$$\begin{aligned} u_{500+200+100} &= \sqrt{u_{500}^2 + u_{200}^2 + u_{100}^2} \\ &= \sqrt{0.040^2 + 0.018^2 + 0.014^2} \\ &= 0.046\,\mathrm{mg}. \end{aligned}$$

6. Discussion and conclusion

The presented contribution shows an evaluation procedure used for the calibration of a set of weights (similar considerations have been done by the authors in [9]). It respects uncertainties and covariances presented in the calibration process. The procedure can be used for any set of measurement standards, not only for a set of weights. The (estimated) covariance matrix of the estimated weight masses (expression (11)) can be divided into two parts. The matrix

$$\hat{\sigma}^2(\boldsymbol{A}^T\boldsymbol{H}^{-1}\boldsymbol{A})^{-1} + (-\boldsymbol{q}\vdots\boldsymbol{I}_{k,k})\boldsymbol{N}\begin{pmatrix} -\boldsymbol{q}^T \\ \boldsymbol{I}_{k,k} \end{pmatrix}$$

is evaluated by the type A method, if the volumes $V_E, V_{\beta_1}, \ldots, V_{\beta_k}$ and density ρ_A are measured. If all quantities are obtained from certificates or tables, the second matrix of the sum is evaluated by the type B. It is also possible that some quantities are measured and others are obtained from certificates, tables, etc. The matrix

$$u_E^2\boldsymbol{q}\boldsymbol{q}^T$$

is always evaluated by the type B method (see [6], [7], [8]). The presented numerical example shows the known fact that covariances calculated using described procedure can obtain non neglectable values and they should be introduced in the calibration certificates. But the contribution will focus reader's attention to condition (7). When the measuring model (calibration model) fulfills this condition, the uncertainty of the reference mass value does not influence the estimate of the unknown mass values, i.e. the calibration procedure is equivalent to weighted least squares analysis. It is easy to see that condition (7) could be without any problems generalized to $\boldsymbol{C}_{n,p} = \boldsymbol{A}_{n,k}\boldsymbol{Q}_{k,p}$ in the case of using p reference weights for the calibration.

Appendix: Mathematical – statistical assertions

Lemma 1. *If the mean of the random vector $\boldsymbol{W}_{n,1}$ is $E(\boldsymbol{W}) = \boldsymbol{A\beta}$ ($\boldsymbol{A}$ is a known $n \times k$ matrix of rank k, $k \leq n$, $\boldsymbol{\beta}$ is an unknown $k \times 1$ vector of parameters) and the covariance matrix of the vector $\boldsymbol{W}$ is of the form cov$(\boldsymbol{W}) = \boldsymbol{U_W} = \sigma^2\boldsymbol{H}_{n,n} + \boldsymbol{ARA}^T$ with diagonal matrix $\boldsymbol{H}$ and nonsingular matrix $\boldsymbol{R}$ then*

$$(\boldsymbol{A}^T\boldsymbol{U}_{\boldsymbol{W}}^{-1}\boldsymbol{A})^{-1}\boldsymbol{A}^T\boldsymbol{U}_{\boldsymbol{W}}^{-1} = (\boldsymbol{A}^T\boldsymbol{H}^{-1}\boldsymbol{A})^{-1}\boldsymbol{A}^T\boldsymbol{H}^{-1}$$

and

$$(\boldsymbol{A}^T\boldsymbol{U}_{\boldsymbol{W}}^{-1}\boldsymbol{A})^{-1} = \sigma^2(\boldsymbol{A}^T\boldsymbol{H}^{-1}\boldsymbol{A})^{-1} + \boldsymbol{R}.$$

Proof. It is valid that for nonsingular matrices $\boldsymbol{C},\ \boldsymbol{D}$

$$(\boldsymbol{C} + \boldsymbol{BDB}^T)^{-1} = \boldsymbol{C}^{-1} - \boldsymbol{C}^{-1}\boldsymbol{B}(\boldsymbol{B}^T\boldsymbol{C}^{-1}\boldsymbol{B} + \boldsymbol{D}^{-1})^{-1}\boldsymbol{B}^T\boldsymbol{C}^{-1}$$

and

$$(\boldsymbol{C} - \boldsymbol{BDB}^T)^{-1} = \boldsymbol{C}^{-1} - \boldsymbol{C}^{-1}\boldsymbol{B}(\boldsymbol{B}^T\boldsymbol{C}^{-1}\boldsymbol{B} - \boldsymbol{D}^{-1})^{-1}\boldsymbol{B}^T\boldsymbol{C}^{-1}$$

(see e.g. Exercise 2.9, paragraph 1b, in [10]). So

$$\boldsymbol{U}_{\boldsymbol{W}}^{-1} = \sigma^{-2}\boldsymbol{H}^{-1} - \sigma^{-4}\boldsymbol{H}^{-1}\boldsymbol{A}(\sigma^{-2}\boldsymbol{A}^T\boldsymbol{H}^{-1}\boldsymbol{A} + \boldsymbol{R}^{-1})^{-1}\boldsymbol{A}^T\boldsymbol{H}^{-1},$$

$$\boldsymbol{A}^T\boldsymbol{U}_{\boldsymbol{W}}^{-1}\boldsymbol{A} = \sigma^{-2}\boldsymbol{A}^T\boldsymbol{H}^{-1}\boldsymbol{A} - \sigma^{-2}\boldsymbol{A}^T\boldsymbol{H}^{-1}\boldsymbol{A}(\sigma^{-2}\boldsymbol{A}^T\boldsymbol{H}^{-1}\boldsymbol{A} + \boldsymbol{R}^{-1})^{-1}\boldsymbol{A}^T\boldsymbol{H}^{-1}\boldsymbol{A}\sigma^{-2}$$

and

$$(\boldsymbol{A}^T\boldsymbol{U}_{\boldsymbol{W}}^{-1}\boldsymbol{A})^{-1} = \sigma^2(\boldsymbol{A}^T\boldsymbol{H}^{-1}\boldsymbol{A})^{-1} + \boldsymbol{R}.$$

Finally

$$(\boldsymbol{A}^T\boldsymbol{U}_{\boldsymbol{W}}^{-1}\boldsymbol{A})^{-1}\boldsymbol{A}^T\boldsymbol{U}_{\boldsymbol{W}}^{-1} =$$

$$
\begin{aligned}
&= [\sigma^2(\boldsymbol{A}^T\boldsymbol{H}^{-1}\boldsymbol{A})^{-1}+\boldsymbol{R}]\boldsymbol{A}^T[\sigma^{-2}\boldsymbol{H}^{-1}-\sigma^{-4}\boldsymbol{H}^{-1}\boldsymbol{A}(\sigma^{-2}\boldsymbol{A}^T\boldsymbol{H}^{-1}\boldsymbol{A}+\boldsymbol{R}^{-1})^{-1}\boldsymbol{A}^T\boldsymbol{H}^{-1}]\\
&= (\boldsymbol{A}^T\boldsymbol{H}^{-1}\boldsymbol{A})^{-1}\boldsymbol{A}^T\boldsymbol{H}^{-1}+\sigma^{-2}\boldsymbol{R}\boldsymbol{A}^T\boldsymbol{H}^{-1}-\sigma^{-2}(\sigma^{-2}\boldsymbol{A}^T\boldsymbol{H}^{-1}\boldsymbol{A}+\boldsymbol{R}^{-1})^{-1}\boldsymbol{A}^T\boldsymbol{H}^{-1}\\
&\quad -\sigma^{-4}\boldsymbol{R}\boldsymbol{A}^T\boldsymbol{H}^{-1}\boldsymbol{A}(\sigma^{-2}\boldsymbol{A}^T\boldsymbol{H}^{-1}\boldsymbol{A}+\boldsymbol{R}^{-1})^{-1}\boldsymbol{A}^T\boldsymbol{H}^{-1}\\
&= (\boldsymbol{A}^T\boldsymbol{H}^{-1}\boldsymbol{A})^{-1}\boldsymbol{A}^T\boldsymbol{H}^{-1}+\sigma^{-2}\boldsymbol{R}\boldsymbol{A}^T\boldsymbol{H}^{-1}-\sigma^{-2}(\sigma^{-2}\boldsymbol{A}^T\boldsymbol{H}^{-1}\boldsymbol{A}+\boldsymbol{R}^{-1})^{-1}\boldsymbol{A}^T\boldsymbol{H}^{-1}\\
&\quad -\sigma^{-2}\boldsymbol{R}(\sigma^{-2}\boldsymbol{A}^T\boldsymbol{H}^{-1}\boldsymbol{A}+\boldsymbol{R}^{-1}-\boldsymbol{R}^{-1})(\sigma^{-2}\boldsymbol{A}^T\boldsymbol{H}^{-1}\boldsymbol{A}+\boldsymbol{R}^{-1})^{-1}\boldsymbol{A}^T\boldsymbol{H}^{-1}\\
&= (\boldsymbol{A}^T\boldsymbol{H}^{-1}\boldsymbol{A})^{-1}\boldsymbol{A}^T\boldsymbol{H}^{-1}. \qquad \square
\end{aligned}
$$

Corollary 2. *Let $\boldsymbol{Y}$ given in (5) be the observed vector in (stochastic) calibration model which fulfils condition (7). The BLUE $\boldsymbol{Z}$ of $\boldsymbol{\beta}$ is*

$$(\boldsymbol{A}^T\boldsymbol{H}^{-1}\boldsymbol{A})^{-1}\boldsymbol{A}^T\boldsymbol{H}^{-1}\boldsymbol{Y}.$$

and its covariance matrix is

$$\boldsymbol{U_Z}=\sigma^2(\boldsymbol{A}^T\boldsymbol{H}^{-1}\boldsymbol{A})^{-1}+u_E^2\boldsymbol{q}\boldsymbol{q}^T+(-\boldsymbol{q}\vdots\boldsymbol{I})\boldsymbol{N}\begin{pmatrix}-\boldsymbol{q}^T\\ \boldsymbol{I}\end{pmatrix}.$$

Proof. The mean of the vector $\boldsymbol{Y}$ is $\boldsymbol{A}\boldsymbol{\beta}$. Its covariance matrix is (according to (6) and (7))

$$
\begin{aligned}
\boldsymbol{U_Y} &= \sigma^2\boldsymbol{H}+u_E^2\boldsymbol{c}\boldsymbol{c}^T+(-\boldsymbol{c}\vdots\boldsymbol{A})\boldsymbol{N}\begin{pmatrix}-\boldsymbol{c}^T\\ \boldsymbol{A}^T\end{pmatrix}\\
&= \sigma^2\boldsymbol{H}+u_E^2\boldsymbol{A}\boldsymbol{q}\boldsymbol{q}^T\boldsymbol{A}^T+(-\boldsymbol{A}\boldsymbol{q}\vdots\boldsymbol{A})\boldsymbol{N}\begin{pmatrix}-\boldsymbol{q}^T\boldsymbol{A}^T\\ \boldsymbol{A}^T\end{pmatrix}\\
&= \sigma^2\boldsymbol{H}+\boldsymbol{A}\left[u_E^2\boldsymbol{q}\boldsymbol{q}^T+(-\boldsymbol{q}\vdots\boldsymbol{I})\boldsymbol{N}\begin{pmatrix}-\boldsymbol{q}^T\\ \boldsymbol{I}\end{pmatrix}\right]\boldsymbol{A}^T\\
&= \sigma^2\boldsymbol{H}+\boldsymbol{A}\boldsymbol{R}\boldsymbol{A}^T
\end{aligned}
$$

The regularity of the matrix $\boldsymbol{R}$ is guaranted by the regularity of the matrix $\boldsymbol{N}=\Delta_\rho^2\boldsymbol{v}\boldsymbol{v}^T+\rho^2\boldsymbol{\Delta}$. The assertion is now an easy consequence of Lemma 1. □

Lemma 3. *Let $\boldsymbol{Y}$ given in (5) be the observed vector in (stochastic) calibration model which fulfils condition (7) and $\boldsymbol{Z}$ is the BLUE of $\boldsymbol{\beta}$. The random vector $\boldsymbol{Y}-\boldsymbol{A}\boldsymbol{Z}$ has the mean value $\mathbf{o}$ and the covariance matrix $\sigma^2(\boldsymbol{H}-\boldsymbol{A}(\boldsymbol{A}^T\boldsymbol{H}^{-1}\boldsymbol{A})^{-1}\boldsymbol{A}^T)$.*

Proof. Using Lemma 1 and condition (7) the proof can be easily obtained. □

Following assertions can also be proved similarly.

Lemma 4. *Let $\boldsymbol{Y}$ given in (5) be the observed vector in (stochastic) calibration model which fulfils condition (7) and $\boldsymbol{Z}$ is the BLUE of $\boldsymbol{\beta}$. Then*

$$\boldsymbol{Y}-\boldsymbol{A}\boldsymbol{Z}=\boldsymbol{X}-\boldsymbol{A}(\boldsymbol{A}^T\boldsymbol{H}^{-1}\boldsymbol{A})^{-1}\boldsymbol{A}^T\boldsymbol{H}^{-1}\boldsymbol{X}.$$

Lemma 5. *Let $\boldsymbol{Y}$ given in (5) be the observed vector in (stochastic) calibration model which fulfils condition (7) Then*

$$\begin{aligned}\hat{\sigma}^2 &= \frac{(\boldsymbol{X} - \boldsymbol{A}(\boldsymbol{A}^T\boldsymbol{H}^{-1}\boldsymbol{A})^{-1}\boldsymbol{A}^T\boldsymbol{H}^{-1}\boldsymbol{X})^T\boldsymbol{H}^{-1}(\boldsymbol{X} - \boldsymbol{A}(\boldsymbol{A}^T\boldsymbol{H}^{-1}\boldsymbol{A})^{-1}\boldsymbol{A}^T\boldsymbol{H}^{-1}\boldsymbol{X})}{n-k} \\ &= \frac{\boldsymbol{X}^T(\boldsymbol{H}^{-1} - \boldsymbol{H}^{-1}\boldsymbol{A}(\boldsymbol{A}^T\boldsymbol{H}^{-1}\boldsymbol{A})^{-1}\boldsymbol{A}^T\boldsymbol{H}^{-1})\boldsymbol{X}}{n-k}\end{aligned}$$

is an unbiased estimator of σ^2.

Lemma 6. *Let $\boldsymbol{Y}$ given in (5) be the observed vector in (stochastic) calibration model which fulfils condition (7) and $\boldsymbol{Z}$ is the BLUE of $\boldsymbol{\beta}$. Then*

$$\hat{\sigma}^2 = \frac{(\boldsymbol{Y} - \boldsymbol{A}\boldsymbol{Z})^T\boldsymbol{H}^{-1}(\boldsymbol{Y} - \boldsymbol{A}\boldsymbol{Z})}{n-k}$$

is an unbiased estimator of σ^2.
When the vector $\boldsymbol{X}$ is normally distributed, $\hat{\sigma}^2$ has $\sigma^2\chi^2_{n-k}/(n-k)$ distribution.

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