

Note on a Calibration Problem: Selected Results and Extensions of Professor Kubáček's Research*

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Dedicated to Lubomír Kubáček on the occasion of his 80th birthday

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Abstract

Professor Lubomír Kubáček has provided exceptional contributions to mathematical statistics and its applications. Because of his excellent knowledge in mathematical statistics as well as in the different fields of natural and especially technical sciences, he contributed to solution of a large number of real world problems. The continuation of Professor Kubáček's scientific work and his scientific school is demonstrated by the results of his numerous students. Here we present just one illustration of Professor Kubáček's original research and its extension by his followers, namely, the calibration problem.

Key words: calibration problem, multiple-use calibration

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1 Introduction

Calibration undoubtedly plays a crucial role in many applications of natural, technical, economical, as well as biomedical sciences. The most important contributions to proper mathematical-statistical formulation and solution of the calibration problem include the seminal works by Eisenhart [3], Krutchkoff [5], Scheffé [12], and Brown [1]. In this paper we deal with the parametric approach. In Section 2 we describe the calibration problem as considered by Kubáček and Kubáčková in [8], [9]. In Sections 2.1–2.3 we present some of our results, see e.g. [13], [14], originally motivated by the research work of Professor Kubáček and extending his research.

2 The calibration problem

Calibration curve, roughly speaking, expresses the relationship between the ideal (true, error-free) results of measuring the same object (substance, quantity) by two measuring devices (instruments) or measurement techniques, say A and B , respectively. Under the term *calibration problem* we understand here (1) the task of fitting the calibration curve based on well-designed *calibration experiment*, in particular, finding the proper estimators of the calibration curve coefficients, and (2) constructing the interval estimators for further determinations of the unknown true value of the measured object in units of the more precise measuring instrument, given measurement(s) in units of the less precise instrument, made by the help of the obtained calibration curve.

Let us have n different objects (substances, quantities) v_1, v_2, \dots, v_n and we measure them with two measuring devices A and B , respectively. The measurements with the device A constitute the normally distributed n -dimensional random vector \mathbf{X} (i.e. $\mathbf{X} \sim N(\boldsymbol{\mu}; \sigma_x^2 \mathbf{I}_{n,n})$) and measurements with the (reference) device B constitute the normally distributed n -dimensional random vector \mathbf{Y} (i.e. $\mathbf{Y} \sim N(\boldsymbol{\nu}; \sigma_y^2 \mathbf{I}_{n,n})$). These measurements are mutually independent. The vector of error-free measurement results made by the instrument A is $\boldsymbol{\mu} = (\mu_1, \dots, \mu_n)'$ and the vector of error-free measurement results made by the instrument B is $\boldsymbol{\nu} = (\nu_1, \dots, \nu_n)'$.

It is assumed that over the typical range of values of μ and ν (the range of interest) the true, however unknown, calibration curve is a linear function, i.e. $\nu_i = a + b\mu_i$, $i = 1, 2, \dots, n$ with (unknown) parameters a , b . So we have the following calibration model

$$\begin{pmatrix} \mathbf{X} \\ \mathbf{Y} \end{pmatrix} \sim N \left[\begin{pmatrix} \boldsymbol{\mu} \\ \boldsymbol{\nu} \end{pmatrix}, \begin{pmatrix} \sigma_x^2 \mathbf{I} & \mathbf{0} \\ \mathbf{0} & \sigma_y^2 \mathbf{I} \end{pmatrix} \right] \quad (1)$$

with nonlinear constraints on parameters

$$\boldsymbol{\nu} = a\mathbf{1} + b\boldsymbol{\mu}, \quad (2)$$

where $\mathbf{1} = \mathbf{1}_{n,1} = (1, \dots, 1)'$ is an n -dimensional vector of ones, \mathbf{I} is an $n \times n$ identity matrix, and a and b are unknown coefficients which specify the intercept

and the slope of the calibration line. The instrument B is here considered to be more precise than the instrument A ($\sigma_y^2 \leq \sigma_x^2$). The instrument A is said to be the calibrated device.

The above mentioned model (1)–(2) is a nonlinear regression model, that could be also interpreted as an error-in-variables model, (EIV), see e.g. Casella and Berger [2].

Kubáček and Kubáčková suggested another solution via EIV model: Linearize the model (1)–(2) by Taylor series expansion locally about $\boldsymbol{\mu}_0 = (\mu_{01}, \dots, \mu_{0n})'$ and b_0 (some values chosen near to the true parameters $\boldsymbol{\mu}$ and b) and neglect the terms of second and higher order. So, $\boldsymbol{\mu} = \boldsymbol{\mu}_0 + \delta\boldsymbol{\mu}$, $b = b_0 + \delta b$ and the new parameters of the approximate linear model are $\delta\boldsymbol{\mu} = (\delta\mu_1, \dots, \delta\mu_n)'$, $\boldsymbol{\nu}$, a , δb , σ_x^2 , σ_y^2 :

$$\begin{pmatrix} \mathbf{X} - \boldsymbol{\mu}_0 \\ \mathbf{Y} \end{pmatrix} \sim N \left[\begin{pmatrix} \delta\boldsymbol{\mu} \\ \boldsymbol{\nu} \end{pmatrix}, \begin{pmatrix} \sigma_x^2 \mathbf{I} & \mathbf{0} \\ \mathbf{0} & \sigma_y^2 \mathbf{I} \end{pmatrix} \right] \quad (3)$$

with linear constraints

$$b_0 \boldsymbol{\mu}_0 + (b_0 \mathbf{1}' - \mathbf{I}) \begin{pmatrix} \delta\boldsymbol{\mu} \\ \boldsymbol{\nu} \end{pmatrix} + (\mathbf{1}, \boldsymbol{\mu}_0) \begin{pmatrix} a \\ \delta b \end{pmatrix} = \mathbf{0}. \quad (4)$$

If the dispersions σ_x^2 and σ_y^2 are not known, Kubáček and Kubáčková suggested in [8] to estimate them using the (iterated) $(\sigma_{x0}^2, \sigma_{y0}^2)$ -MINQUE, i.e. the $(\sigma_{x0}^2, \sigma_{y0}^2)$ -locally minimum norm quadratic unbiased estimator. For more details on quadratic estimation of variance components see e.g. [10] and also [6, 8]. However, it is necessary to change the measuring model, as the MINQUEs of the parameters σ_x^2 and σ_y^2 in model (3)–(4) do not exist. The common and relatively easy way is to repeat the whole experiment m -times independently. The replicated measurements are $\mathbf{X}_j = (X_{j1}, \dots, X_{jn})'$, $\mathbf{Y}_j = (Y_{j1}, \dots, Y_{jn})'$, $j = 1, \dots, m$.

If we denote $\mathbf{M}_{[1, \boldsymbol{\mu}_0]} = \mathbf{I} - [\mathbf{1}, \boldsymbol{\mu}_0] ([\mathbf{1}, \boldsymbol{\mu}_0]' [\mathbf{1}, \boldsymbol{\mu}_0])^{-1} [\mathbf{1}, \boldsymbol{\mu}_0]'$ then the BLUEs (best linear unbiased estimators) of $\boldsymbol{\mu}$, $\boldsymbol{\nu}$, a and δb (see also in [14]) in replicated model are

$$\hat{\boldsymbol{\mu}} = \bar{\mathbf{X}} + \frac{b_0 \sigma_x^2}{b_0^2 \sigma_x^2 + \sigma_y^2} \mathbf{M}_{[1, \boldsymbol{\mu}_0]} (\bar{\mathbf{Y}} - b_0 \bar{\mathbf{X}}), \quad (5)$$

$$\hat{\boldsymbol{\nu}} = \bar{\mathbf{Y}} - \frac{\sigma_y^2}{b_0^2 \sigma_x^2 + \sigma_y^2} \mathbf{M}_{[1, \boldsymbol{\mu}_0]} (\bar{\mathbf{Y}} - b_0 \bar{\mathbf{X}}), \quad (6)$$

$$\begin{pmatrix} \hat{a} \\ \hat{\delta b} \end{pmatrix} = \begin{pmatrix} n & \mathbf{1}' \boldsymbol{\mu}_0 \\ \boldsymbol{\mu}'_0 \mathbf{1} & \boldsymbol{\mu}'_0 \boldsymbol{\mu}_0 \end{pmatrix}^{-1} \begin{pmatrix} \mathbf{1}' (\bar{\mathbf{Y}} - b_0 \bar{\mathbf{X}}) \\ \boldsymbol{\mu}'_0 (\bar{\mathbf{Y}} - b_0 \bar{\mathbf{X}}) \end{pmatrix}, \quad (7)$$

with the covariance matrix

$$\text{cov} \begin{pmatrix} \hat{a} \\ \hat{\delta b} \end{pmatrix} = \frac{b_0^2 \sigma_x^2 + \sigma_y^2}{m} \begin{pmatrix} n & \mathbf{1}' \boldsymbol{\mu}_0 \\ \boldsymbol{\mu}'_0 \mathbf{1} & \boldsymbol{\mu}'_0 \boldsymbol{\mu}_0 \end{pmatrix}^{-1}$$

$$(\bar{\mathbf{X}} = \frac{1}{m} \sum_{j=1}^m \mathbf{X}_j, \bar{\mathbf{Y}} = \frac{1}{m} \sum_{j=1}^m \mathbf{Y}_j).$$

Kubáček and Kubáčková in [8] derived also the $(\sigma_{x0}^2, \sigma_{y0}^2)$ -MINQUEs of σ_x^2 and σ_y^2 in the replicated model. The estimators are as follows

$$\begin{pmatrix} \hat{\sigma}_x^2 \\ \hat{\sigma}_y^2 \end{pmatrix} = \frac{1}{n(m-1)} \left[\mathbf{I}_{2,2} - c_0 \begin{pmatrix} b_0^4 \sigma_{x0}^4 & b_0^2 \sigma_{x0}^4 \\ b_0^2 \sigma_{y0}^4 & \sigma_{y0}^4 \end{pmatrix} \right] \begin{pmatrix} \hat{\kappa}_1 \\ \hat{\kappa}_2 \end{pmatrix}, \quad (8)$$

where

$$c_0 = \frac{n-2}{(b_0^4 \sigma_{x0}^4 + \sigma_{y0}^4)(mn-2) + 2b_0^2 \sigma_{x0}^2 \sigma_{y0}^2 (m-1)n},$$

$$\hat{\kappa}_1 = \sum_{j=1}^m (\mathbf{X}_j - \bar{\mathbf{X}})' (\mathbf{X}_j - \bar{\mathbf{X}}) + m(\bar{\mathbf{X}} - \hat{\boldsymbol{\mu}})' (\bar{\mathbf{X}} - \hat{\boldsymbol{\mu}}),$$

$$\hat{\kappa}_2 = \sum_{j=1}^m (\mathbf{Y}_j - \bar{\mathbf{Y}})' (\mathbf{Y}_j - \bar{\mathbf{Y}}) + m(\bar{\mathbf{Y}} - \hat{\boldsymbol{\nu}})' (\bar{\mathbf{Y}} - \hat{\boldsymbol{\nu}}).$$

The covariance matrix (correct locally at $(\sigma_{x0}^2, \sigma_{y0}^2)$) of the estimator (8) is

$$\mathbf{W} = \begin{pmatrix} w_{11} & w_{12} \\ w_{21} & w_{22} \end{pmatrix} = \frac{2}{n(m-1)} \left[\mathbf{I}_{2,2} - c_0 \begin{pmatrix} b_0^4 \sigma_{x0}^4 & b_0^2 \sigma_{x0}^4 \\ b_0^2 \sigma_{y0}^4 & \sigma_{y0}^4 \end{pmatrix} \right] \begin{pmatrix} \sigma_{x0}^4 & 0 \\ 0 & \sigma_{y0}^4 \end{pmatrix}. \quad (9)$$

Kubáček and Kubáčková in [8] suggested to use the $(\sigma_{x0}^2, \sigma_{y0}^2)$ -MINQUE estimates instead of the true (unknown) values σ_x^2 and σ_y^2 in (5)–(7).

They made also some basic investigations for determination, whether such plug-in estimators are proper for practical purposes, i.e. for measuring with calibrated (less precise) device A. If the future measurement realized with this instrument, say x , is a realization of a random variable X , distributed as $X \sim N(\mu_x, \sigma_x^2)$, (μ_x represents the unobservable true value of measurand) then the suggested estimator of $\nu_x = a + b\mu_x$ is

$$\hat{\nu}_x = \hat{a} + \hat{b}X \quad (10)$$

which is a nonlinear estimator. In [7], Kubáček has suggested a method to determine the bias and dispersion of the estimator (10) and a simple rule to decide whether it is suitable for practical purposes or not.

In the next sections we present some extensions of the Kubáček's results, as originally suggested in [13] and [14]: derivation of the approximate distribution of the test statistics about the regression parameters of the calibration line, derivation of the Scheffé-type confidence region for the calibration line, as well as derivation of new approximate multiple-use calibration intervals for a series of future determinations based on the approximate (linearized) calibration model suggested by Kubáček.

2.1 Estimation of the calibration line parameters

If there is no specific prior information on the true values of the calibration line parameters $\boldsymbol{\mu}$, b , and the variance components σ_{x0}^2 and σ_{y0}^2 the following set is

a natural choice of the initial values, estimated from the measured data:

$$\begin{aligned}\hat{\boldsymbol{\mu}}_0 &= \bar{\mathbf{X}}, \\ \hat{b}_0 &= \frac{n\bar{\mathbf{X}}'\bar{\mathbf{Y}} - (\mathbf{1}'\bar{\mathbf{X}})(\mathbf{1}'\bar{\mathbf{Y}})}{n\bar{\mathbf{X}}'\bar{\mathbf{X}} - (\mathbf{1}'\bar{\mathbf{X}})^2}, \\ \hat{\sigma}_{x0}^2 &= \frac{1}{n(m-1)} \sum_{i=1}^n \sum_{j=1}^m (X_{ji} - \bar{X}_i)^2, \\ \hat{\sigma}_{y0}^2 &= \frac{1}{n(m-1)} \sum_{i=1}^n \sum_{j=1}^m (Y_{ji} - \bar{Y}_i)^2.\end{aligned}\tag{11}$$

Further we compute \hat{a} , \hat{b} from (7), $\hat{\boldsymbol{\mu}}$ from (5), $\hat{\boldsymbol{\nu}}$ from (6), $\hat{\sigma}_x^2$ and $\hat{\sigma}_y^2$ from (8). The estimation procedure can be iterated until convergence is reached. After reaching the convergence we calculate the covariance matrix \mathbf{W} according to (9).

For testing the null hypothesis $H_0 : (a, b)' = (a_*, b_*)'$ and for construction of the confidence region for the parameters $(a, b)'$ we suggest to use the F -statistic

$$F = \frac{1}{2} \frac{m}{b_0^2 \hat{\sigma}_x^2 + \hat{\sigma}_y^2} \begin{pmatrix} \hat{a} - a \\ \hat{b} - b \end{pmatrix}' \begin{pmatrix} n & \mathbf{1}'\boldsymbol{\mu}_0 \\ \boldsymbol{\mu}'_0 \mathbf{1} & \boldsymbol{\mu}'_0 \boldsymbol{\mu}_0 \end{pmatrix} \begin{pmatrix} \hat{a} - a \\ \hat{b} - b \end{pmatrix},\tag{12}$$

with the values of the parameters given from the last iteration stipulated above.

By using the method suggested by Kenward and Roger in [4], under H_0 the distribution of the F -statistic could be approximated by the Fisher-Snedecor F -distribution with 2 and u degrees of freedom, i.e. $F \sim F_{2,u}$, where u is given by

$$u = (mn - 2) + \frac{2n(m-1)b_0^2 \hat{\sigma}_x^2 \hat{\sigma}_y^2}{b_0^4 \hat{\sigma}_x^4 + \hat{\sigma}_y^4}.\tag{13}$$

For more details see [14] and [13].

2.2 Scheffé-type confidence region for the calibration line

Let $(a, b)'$ represents the true vector of the calibration line parameters $\nu = a + b\mu$, $\mu \in \langle \mu_l, \mu_u \rangle$, where the interval $\langle \mu_l, \mu_u \rangle$ represents the typical range of the calibration experiment.

By applying the Scheffé's Theorem, i.e. a method for adjusting significance levels in a linear regression analysis to account for multiple comparisons, see e.g. [11] and also [6, 8], we directly get from (12) the $100 \times (1 - \alpha)\%$ -confidence region for the calibration line $a + b\mu$, for all $\mu \in \langle \mu_l, \mu_u \rangle$:

$$\begin{aligned}\Pr \left\{ \left| (\hat{a} + \hat{b}\mu) - (a + b\mu) \right| \leq \sqrt{2F_{2,u}(1 - \alpha) \frac{b_0^2 \hat{\sigma}_x^2 + \hat{\sigma}_y^2}{m} \left(\frac{1}{n} + \frac{(\mu - \bar{\mu}_0)^2}{\boldsymbol{\mu}'_0 \boldsymbol{\mu}_0 - n\bar{\mu}_0^2} \right)} \right\} \\ = 1 - \alpha,\end{aligned}\tag{14}$$

where $\bar{\mu}_0 = (\mathbf{1}'\boldsymbol{\mu}_0)/n$ and $F_{2,u}(1 - \alpha)$ is the $(1 - \alpha)$ -quantile, $\alpha \in (0, 1)$ of the Fisher-Snedecor F -distribution with 2 and u degrees of freedom.

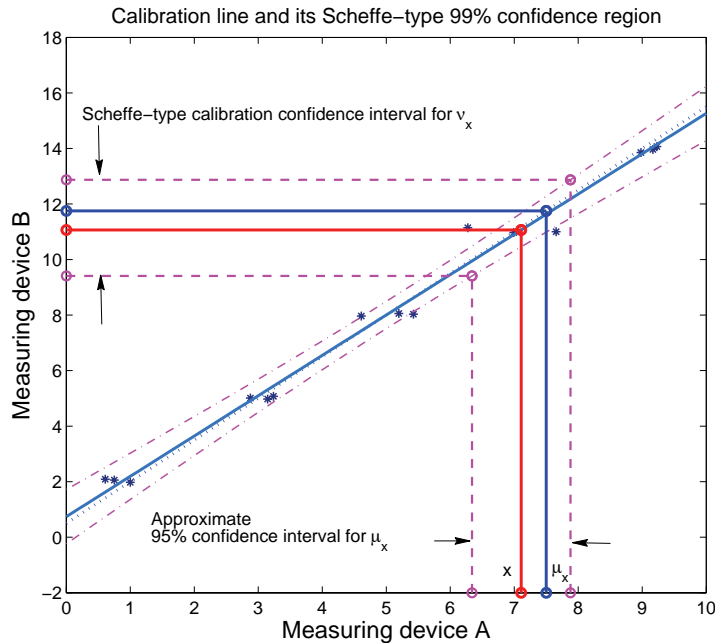


Figure 1: Calibration line and its Scheffé-type confidence region. The thick dotted line represents the true calibration line, the solid line is the estimated calibration line together with the limits of the 99% confidence region (dashed-dotted lines). The dashed lines represents the Scheffé-type interval estimator for $\nu_x = a + b\mu_x$, where $\mu_x = 7.5$, based on $x = 7.1097$, the realization of random variable $X \sim N(7.5, 0.15)$.

2.3 Multiple-use calibration—measuring with calibrated device

In this section we will derive a new approximate multiple-use calibration intervals for a series of future determinations based on the approximate (linearized) calibration model (3) with linear constraints (4).

We will assume as in (10) that the future measurement realized by the calibrated (less precise) measurement device A, say x , is a realization of a random variable X , distributed as $X \sim N(\mu_x, \sigma_x^2)$, where μ_x represents unobservable true value of measurand given in units of the less precise instrument and further $\mu_x \in \langle \mu_l, \mu_u \rangle$.

Based on the observed value x we suggest the estimate and a simple derivation of the confidence interval for $\nu_x = a + b\mu_x$ (the unobservable true value of measurand in units given by the more precise (reference) measuring device B).

First, we suggest to construct the approximate $(1 - \gamma)$ -confidence region for the calibration line, for small significance level $\gamma \in (0, 1)$, chosen by the user, according to (14).

Second, for small significance level $\alpha \in (0, 1)$, we suggest to construct the approximate $(1 - \alpha)$ -confidence interval for μ_x , given x and the estimated value (realization of) $\hat{\sigma}_x^2$. For that we suggest to construct t -statistic $t = (X - \mu_x)/\hat{\sigma}_x$ with approximate Student's t distribution with v degrees of freedom, where v is approximated by $v = 2\hat{\sigma}_x^4/w_{11}$, with w_{11} being the first element of the matrix \mathbf{W} given by (9). This leads to the approximate $(1 - \alpha)$ -confidence interval for unobservable value μ_x :

$$\mu_x \in \{x \pm \hat{\sigma}_x t_v(1 - \alpha/2)\}, \quad (15)$$

where $t_v(1 - \alpha/2)$ is the $(1 - \alpha/2)$ -quantile of the Student's t distribution with v degrees of freedom. Let μ_{xl} and μ_{xu} denote the lower and upper limit of the approximate $(1 - \alpha)$ -confidence interval for μ_x .

The suggested interval estimator for $\nu_x = \langle \nu_{xl}, \nu_{xu} \rangle$ is then given as the intersection of the bounds of the $(1 - \gamma)$ -confidence region for the whole calibration line $a + b\mu$ and the limits of the $(1 - \alpha)$ -confidence interval $\langle \mu_{xl}, \mu_{xu} \rangle$ for μ_x . In fact,

$$\begin{aligned} \nu_{xl} &= \hat{a} + \hat{b}\mu_{xl} - \sqrt{2F_{2,u}(1 - \gamma) \frac{b_0^2 \hat{\sigma}_x^2 + \hat{\sigma}_y^2}{m} \left(\frac{1}{n} + \frac{(\mu_{xl} - \bar{\mu}_0)^2}{\boldsymbol{\mu}'_0 \boldsymbol{\mu}_0 - n\bar{\mu}_0^2} \right)} \\ \nu_{xu} &= \hat{a} + \hat{b}\mu_{xu} + \sqrt{2F_{2,u}(1 - \gamma) \frac{b_0^2 \hat{\sigma}_x^2 + \hat{\sigma}_y^2}{m} \left(\frac{1}{n} + \frac{(\mu_{xu} - \bar{\mu}_0)^2}{\boldsymbol{\mu}'_0 \boldsymbol{\mu}_0 - n\bar{\mu}_0^2} \right)}. \end{aligned} \quad (16)$$

Using Bonferroni's inequality, $\langle \nu_{xl}, \nu_{xu} \rangle$ is at least $1 - (\alpha + \gamma)$ -confidence interval for the (unobservable) value ν_x . Simulations indicate that the confidence interval $\langle \nu_{xl}, \nu_{xu} \rangle$ is conservative, "safe" and appropriate for metrology.

3 Conclusion

In this paper we have illustrated small part of broad scientific interests of professor Kubáček's research work. The continuation of his scientific school is well demonstrated by the results of his students and co-workers. Here we have tried to emphasize Kubáček's contribution in the field of statistics in measurement science and metrology, namely his contribution to solving the calibration problem, and to illustrate extensions of Kubáček's research by his students.

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