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Two models for linear comparative calibration

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Abstract. We consider the comparative calibration problem in the case when linear relationship is assumed between two considered measuring devices with possibly different units and precisions. The first method for obtaining the approximate confidence region for unknown parameters of the calibration line applies the maximum likelihood estimators of the unknown parameters. The second method is based on estimation of the calibration line via replicated errors-in-variables model. Essential point in this approach is approximation of the small sample distribution of the Wald-type test statistic. This enables to construct the interval estimators for the multiple-use calibration case.

Keywords: Calibration problem; multiple-use calibration; maximum likelihood estimator; errors-in-variables model; Kenward-Roger approximation

1 Introduction

We consider the problem of comparative calibration in small sample case. This paper presents two methods for obtaining the approximate confidence region for unknown parameters of the calibration line and to construct the interval estimators for the multiple-use calibration case. This is useful for linear univariate comparative calibration problem with possibly different and unknown precisions of both measuring devices and enables to construct the interval estimators for the unknown quantity in multiple-use calibration case.

The first method for obtaining the approximate confidence region for unknown parameters of the calibration line applies the maximum likelihood estimators (MLE) of the unknown parameters and relies on the asymptotic properties of the MLEs which are used for construction of the Scheffé-type confidence region for the calibration line. From statistical point of view, the second method is based on the linear errors-in-variables (EIV) model. In a standard situation, the estimators of the calibration function parameters are based on minimization of the weighted total sum of squares in the orthogonal regression with weights inversely proportional to the true standard deviations. If the true standard deviations are (partially or completely) unknown, and should be estimated from the measurements, we suggest to use an alternative iterative algorithm based on locally linearized model for parameter estimation that allows to consider the problem of deriving the approximate confidence region for the parameters. The confidence limits are derived using the concept of Kenward and Roger, see [1] and also [2–5].

The suggested estimation methods enables to construct the interval estimators for the multiple-use calibration case. In this paper we present and illustrate both methods for construction the approximate confidence interval for the true value of the measurand (in units of the more precise device) in the multiple-use calibration case.

2 Methods

2.1 Estimation of the calibration line parameters via maximum likelihood method

Let us have measurements $X_{11}, Y_{11}, \ldots, X_{n1}, Y_{n1}$. We suppose that the measurements are normally distributed, independent and it is valid that the mean value of Y_{i1} is

$$\mathcal{E}(Y_{i1}) = a + b\mu_i, \quad i = 1, 2, \ldots, n,$$

where μ_i is the mean value of X_{i1} and a, b are the unknown parameters of the calibration line (more see in [6]). We consider this group of $2n$ measurements as one (single) experiment. The likelihood function of the random vector

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$(X_{11}, Y_{11}, \ldots, X_{n1}, Y_{n1})'$ is

$$\begin{aligned} L(a, b, \sigma_x^2, \sigma_y^2, \mu_1, \ldots, \mu_n | x_{11}, y_{11}, \ldots, x_{n1}, y_{n1}) \\ = \prod_{i=1}^{n} f_i(x_{i1}; \mu_i, \sigma_x^2) \prod_{j=1}^{n} g_j(y_{j1}; a + b\mu_j, \sigma_y^2) \\ = \frac{1}{(2\pi)^n \sigma_x^n \sigma_y^n} \exp\left\{ -\frac{1}{2\sigma_x^2} \sum_{i=1}^{n} (x_{i1} - \mu_i)^2 \right. \\ \left. - \frac{1}{2\sigma_y^2} \sum_{j=1}^{n} (y_{j1} - a - b\mu_j)^2 \right\}. \end{aligned}$$

We shall assume that this experiment is repeated independently m-times. The r-th experiment is modeled by the random vector $(X_{1r}, Y_{1r}, \ldots, X_{nr}, Y_{nr})'$. The likelihood function of the whole calibration experiment (consisting of m experiments) is

$$\begin{aligned} L(a, b, \sigma_x^2, \sigma_y^2, \mu_1, \ldots, \mu_n | x_{11}, y_{11}, \ldots, x_{nm}, y_{nm}) \\ = \frac{1}{(2\pi)^{nm} \sigma_x^{nm} \sigma_y^{nm}} \exp\left\{ -\frac{1}{2\sigma_x^2} \sum_{i=1}^{n} \sum_{j=1}^{m} (x_{ij} - \mu_i)^2 \right. \\ \left. - \frac{1}{2\sigma_y^2} \sum_{i=1}^{n} \sum_{j=1}^{m} (y_{ij} - a - b\mu_i)^2 \right\}. \end{aligned}$$

The maximum likelihood estimators $\tilde{a}(X_{11}, \ldots, Y_{nm})$, $\tilde{b}(X_{11}, \ldots, Y_{nm})$, $\tilde{\sigma}_x^2(X_{11}, \ldots, Y_{nm})$, $\tilde{\sigma}_y^2(X_{11}, \ldots, Y_{nm})$, $\tilde{\mu}_1(X_{11}, \ldots, Y_{nm}), \ldots, \tilde{\mu}_n(X_{11}, \ldots, Y_{nm})$ are solutions of the likelihood equations

$$\sum_{i=1}^{n} \sum_{j=1}^{m} (Y_{ij} - \tilde{a} - \tilde{b}\tilde{\mu}_i) = 0,$$

$$\sum_{i=1}^{n} \sum_{j=1}^{m} (Y_{ij} - \tilde{a} - \tilde{b}\tilde{\mu}_i)\tilde{\mu}_i = 0,$$

$$\sum_{i=1}^{n} \sum_{j=1}^{m} (X_{ij} - \tilde{\mu}_i)^2 = mn\tilde{\sigma}_x^2,$$

$$\sum_{i=1}^{n} \sum_{j=1}^{m} (Y_{ij} - \tilde{a} - \tilde{b}\tilde{\mu}_i)^2 = mn\tilde{\sigma}_y^2,$$

$$\tilde{\sigma}_y^2 \sum_{j=1}^{m} (X_{1j} - \tilde{\mu}_1) = -\tilde{\sigma}_x^2 \tilde{b} \sum_{j=1}^{m} (Y_{1j} - \tilde{a} - \tilde{b}\tilde{\mu}_1)$$

$$\vdots$$

$$\tilde{\sigma}_y^2 \sum_{j=1}^{m} (X_{nj} - \tilde{\mu}_n) = -\tilde{\sigma}_x^2 \tilde{b} \sum_{j=1}^{m} (Y_{nj} - \tilde{a} - \tilde{b}\tilde{\mu}_n).$$

It is valid

$$\sqrt{m} \begin{pmatrix} \tilde{a} - a \\ \tilde{b} - b \end{pmatrix} \xrightarrow{\mathcal{D}} N\left(\begin{pmatrix} 0 \\ 0 \end{pmatrix}, \mathbf{\Sigma} \right)$$

(convergence in distribution). The asymptotic covariance matrix of the likelidood estimator $(\tilde{a}, \tilde{b})'$ is

$$\mathbf{\Sigma} = \frac{b^2\sigma_x^2 + \sigma_y^2}{m\left(n\sum_{i=1}^{n} \mu_i^2 - \left(\sum_{j=1}^{n} \mu_j \right)^2 \right)} \times \begin{pmatrix} \sum_{i=1}^{n} \mu_i^2 & -\sum_{i=1}^{n} \mu_i \\ -\sum_{i=1}^{n} \mu_i & n \end{pmatrix}.$$

We use the approximative $\tilde{\mathbf{\Sigma}}$

$$\tilde{\mathbf{\Sigma}} = \frac{\tilde{b}^2\tilde{\sigma}_x^2 + \tilde{\sigma}_y^2}{m\left(n\sum_{i=1}^{n} \tilde{\mu}_i^2 - \left(\sum_{j=1}^{n} \tilde{\mu}_j \right)^2 \right)} \times \begin{pmatrix} \sum_{i=1}^{n} \tilde{\mu}_i^2 & -\sum_{i=1}^{n} \tilde{\mu}_i \\ -\sum_{i=1}^{n} \tilde{\mu}_i & n \end{pmatrix}.$$

Further, it holds

$$\chi^2 = \begin{pmatrix} \tilde{a} - a \\ \tilde{b} - b \end{pmatrix}' \tilde{\mathbf{\Sigma}}^{-1} \begin{pmatrix} \tilde{a} - a \\ \tilde{b} - b \end{pmatrix} \overset{approx.}{\sim} \chi_2^2,$$

and

$$\Pr\left\{ \begin{pmatrix} \tilde{a} - a \\ \tilde{b} - b \end{pmatrix}' \tilde{\mathbf{\Sigma}}^{-1} \begin{pmatrix} \tilde{a} - a \\ \tilde{b} - b \end{pmatrix} \leqq \chi_2^2(1-\alpha) \right\} \overset{approx.}{=} 1-\alpha,$$

($\chi_2^2(1-\alpha)$ is the $(1-\alpha)$-quantile of the χ^2 distribution with 2 degrees of freedom).

From that we obtain the ML (approximative, asymptotic) $(1-\alpha)$-confidence region for $(a, b)'$

$$\mathcal{C}_{(1-\alpha)}^{ML} = \left\{ \begin{pmatrix} a \\ b \end{pmatrix} : \begin{pmatrix} \tilde{a} - a \\ \tilde{b} - b \end{pmatrix}' \tilde{\mathbf{\Sigma}}^{-1} \begin{pmatrix} \tilde{a} - a \\ \tilde{b} - b \end{pmatrix} \leqq \chi_2^2(1-\alpha) \right\}.$$

2.2 Estimation of the calibration line parameters via replicated errors-in-variables model

Calibration experiment we can model using EIV model

$$Y_i = \alpha + \beta\mu_i + \varepsilon_i, \quad X_i = \mu_i + \delta_i$$

($\varepsilon_i \sim N(0, \sigma_Y^2), \delta_i \sim N(0, \sigma_X^2)$ independent). Written in another way

$$\mathcal{E}(Y_i) = \alpha + \beta\mu_i, \quad \mathcal{E}(X_i) = \mu_i.$$

Vectors of errorless measurements realized using instruments A and B are $\boldsymbol{\mu} = (\mu_1, \mu_2, \ldots, \mu_n)'$ and $\boldsymbol{\nu} = (\nu_1, \nu_2, \ldots, \nu_n)'$. Vector of measurements with instrument A is $\mathbf{X}_{n,1} \sim N(\boldsymbol{\mu}; \sigma_x^2\mathbf{I}_{n,n})$. Vector of measurements with instrument B is $\mathbf{Y}_{n,1} \sim N(\boldsymbol{\nu}; \sigma_y^2\mathbf{I}_{n,n})$. We obtain the model

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

with condition on parameters

$$\boldsymbol{\nu} = a\mathbf{1}_{n,1} + b\boldsymbol{\mu},$$

where $\mathbf{1}_{n,1} = (1,1,\dots,1)'$. First we linearize the model using Taylor series in a neighborhood of $\boldsymbol{\mu}_0 = (\mu_{01}, \mu_{02}, \dots, \mu_{0n})'$ a b_0 (some values near the reality $\boldsymbol{\mu}$ a b). Now $\boldsymbol{\mu} = \boldsymbol{\mu}_0 + \delta\boldsymbol{\mu}$, $b = b_0 + \delta b$ and the new model parameters are $\delta\boldsymbol{\mu} = (\delta\mu_1, \delta\mu_2, \dots, \delta\mu_n)', \boldsymbol{\nu}, a, \delta b, \sigma_x^2, \sigma_y^2$. We get the (approximative) linear regression model

$$\begin{pmatrix} \boldsymbol{X} - \boldsymbol{\mu}_0 \\ \boldsymbol{Y} \end{pmatrix} \sim N\left[\begin{pmatrix} \delta\boldsymbol{\mu} \\ \boldsymbol{\nu} \end{pmatrix}, \begin{pmatrix} \sigma_x^2 \boldsymbol{I} & \mathbf{0} \\ \mathbf{0} & \sigma_y^2 \boldsymbol{I} \end{pmatrix}\right] \tag{1}$$

with (linear) conditions on parameters

$$b_0\boldsymbol{\mu}_0 + (b_0\boldsymbol{I} \vdots - \boldsymbol{I}) \begin{pmatrix} \delta\boldsymbol{\mu} \\ \boldsymbol{\nu} \end{pmatrix} + (\mathbf{1}, \boldsymbol{\mu}_0) \begin{pmatrix} a \\ \delta b \end{pmatrix} = \mathbf{0}. \tag{2}$$

Dispersions σ_x^2 and σ_y^2 are unknown. One possibility to estimate them are the $(\sigma_{x0}^2, \sigma_{y0}^2)$-MINQUE estimators (minimum norm quadratic unbiased estimator). As this estimators do not exist in model (1)–(2), we need to repeat the whole experiment m times independently. The repeated measurements are $\boldsymbol{X}_j = (X_{j1}, \dots, X_{jn})'$, $\boldsymbol{Y}_j = (Y_{j1}, \dots, Y_{jn})'$, $j = 1, \dots, m$. The best linear unbiased estimators $\boldsymbol{\mu}, \boldsymbol{\nu}$, $a, \delta b$ in replicated model are (see [2])

$$\hat{\boldsymbol{\mu}} = \bar{\boldsymbol{X}} + \frac{b_0\sigma_x^2}{b_0^2\sigma_x^2 + \sigma_y^2} \boldsymbol{M}_{[1,\mu_0]}(\bar{\boldsymbol{Y}} - b_0\bar{\boldsymbol{X}}), \tag{3}$$

$$\hat{\boldsymbol{\nu}} = \bar{\boldsymbol{Y}} - \frac{\sigma_y^2}{b_0^2\sigma_x^2 + \sigma_y^2} \boldsymbol{M}_{[1,\mu_0]}(\bar{\boldsymbol{Y}} - b_0\bar{\boldsymbol{X}}), \tag{4}$$

$$\begin{pmatrix} \hat{a} \\ \hat{\delta b} \end{pmatrix} = \begin{pmatrix} n & \mathbf{1}'\boldsymbol{\mu}_0 \\ \boldsymbol{\mu}_0'\mathbf{1} & \boldsymbol{\mu}_0'\boldsymbol{\mu}_0 \end{pmatrix}^{-1} \begin{pmatrix} \mathbf{1}'(\bar{\boldsymbol{Y}} - b_0\bar{\boldsymbol{X}}) \\ \boldsymbol{\mu}_0'(\bar{\boldsymbol{Y}} - b_0\bar{\boldsymbol{X}}) \end{pmatrix}, \tag{5}$$

with the covariance matrix

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

where

$$\bar{\boldsymbol{X}} = \frac{1}{m}\sum_{j=1}^m \boldsymbol{X}_j, \quad \bar{\boldsymbol{Y}} = \frac{1}{m}\sum_{j=1}^m \boldsymbol{Y}_j$$

and

$$\boldsymbol{M}_{[1,\mu_0]} = \boldsymbol{I} - [\mathbf{1}, \boldsymbol{\mu}_0]\left([\mathbf{1}, \boldsymbol{\mu}_0]'[\mathbf{1}, \boldsymbol{\mu}_0]\right)^{-1}[\mathbf{1}, \boldsymbol{\mu}_0]'.$$

$(\sigma_{x0}^2, \sigma_{y0}^2)$-MINQUE estimators of σ_x^2 and σ_y^2 in replicated model are

$$\begin{pmatrix} \hat{\sigma}_x^2 \\ \hat{\sigma}_y^2 \end{pmatrix} = \frac{1}{n(m-1)}\left[\boldsymbol{I}_{2,2} - c_0\begin{pmatrix} b_0^4\sigma_{x0}^4 & b_0^2\sigma_{x0}^4 \\ b_0^2\sigma_{y0}^4 & \sigma_{y0}^4 \end{pmatrix}\right]\begin{pmatrix} \hat{\kappa}_1 \\ \hat{\kappa}_2 \end{pmatrix}, \tag{6}$$

where

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

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

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

The covariance matrix of the estimators (6) (local in the values $(\sigma_{x0}^2, \sigma_{y0}^2)$) is

$$\boldsymbol{W} = \begin{pmatrix} w_{11} & w_{12} \\ w_{21} & w_{22} \end{pmatrix} = \frac{2}{n(m-1)}\left[\boldsymbol{I}_{2,2} - c_0\begin{pmatrix} b_0^4\sigma_{x0}^4 & b_0^2\sigma_{x0}^4 \\ b_0^2\sigma_{y0}^4 & \sigma_{y0}^4 \end{pmatrix}\right]\begin{pmatrix} \sigma_{x0}^4 & 0 \\ 0 & \sigma_{y0}^4 \end{pmatrix}. \tag{7}$$

A natural choice of the initial values resulting from the measurements can be as follows

$$\boldsymbol{\mu}_0 = \bar{\boldsymbol{X}}, \qquad b_0 = \frac{n\bar{\boldsymbol{X}}'\bar{\boldsymbol{Y}} - (\mathbf{1}'\bar{\boldsymbol{X}})(\mathbf{1}'\bar{\boldsymbol{Y}})}{n\bar{\boldsymbol{X}}'\bar{\boldsymbol{X}} - (\mathbf{1}'\bar{\boldsymbol{X}})^2},$$

$$\sigma_{x0}^2 = \frac{1}{n(m-1)}\sum_{i=1}^n\sum_{j=1}^m (X_{ji} - \bar{X}_i)^2,$$

$$\sigma_{y0}^2 = \frac{1}{n(m-1)}\sum_{i=1}^n\sum_{j=1}^m (Y_{ji} - \bar{Y}_i)^2.$$

Further the estimates are computed as follows: $\hat{a}$, $\hat{b}$ from (5), $\hat{\boldsymbol{\mu}}$ from (3), $\hat{\boldsymbol{\nu}}$ from (4), $\hat{\sigma}_x^2$ and $\hat{\sigma}_y^2$ from (6). The estimation procedure is iterative till the convergence is reached (usually in 4-5 steps). After the procedure is finished, computed is the covariance matrix $\boldsymbol{W}$ according to (7).

We have obtained

$$\begin{pmatrix} \hat{a} - a \\ \hat{b} - b \end{pmatrix} \sim N\left(\begin{pmatrix} 0 \\ 0 \end{pmatrix}, \frac{b_0^2\sigma_x^2 + \sigma_y^2}{m}\begin{pmatrix} n & \mathbf{1}'\boldsymbol{\mu}_0 \\ \boldsymbol{\mu}_0'\mathbf{1} & \boldsymbol{\mu}_0'\boldsymbol{\mu}_0 \end{pmatrix}^{-1}\right).$$

To emphasize dependence of the distribution on the parameters (σ_x^2, σ_y^2), we will alternatively denote the covariance matrix of the distribution also by $\boldsymbol{\Phi}(\sigma_x^2, \sigma_y^2)$. If the parameters σ_x^2, σ_y^2 are known, $(1-\alpha)$-confidence region for parameters a, b is

$$\mathcal{C}^*_{(1-\alpha)} = \left\{\begin{pmatrix} a \\ b \end{pmatrix} : \frac{m}{b_0^2\sigma_x^2 + \sigma_y^2}Q^{(EIV)} \leqq \chi_2^2(1-\alpha)\right\},$$

where

$$Q^{(EIV)} = \begin{pmatrix} \hat{a} - a \\ \hat{b} - b \end{pmatrix}'\begin{pmatrix} n & \mathbf{1}'\boldsymbol{\mu}_0 \\ \boldsymbol{\mu}_0'\mathbf{1} & \boldsymbol{\mu}_0'\boldsymbol{\mu}_0 \end{pmatrix}\begin{pmatrix} \hat{a} - a \\ \hat{b} - b \end{pmatrix}.$$

If σ_x^2, σ_y^2 are unknown, we apply the procedure suggested by Kenward and Roger, see [1], to obtain the adjusted Wald-type statistic and its approximate F-distribution. This procedure was suggested for small range of measured data (in our case small m, n). Kenward and Roger proposed a modified estimator of the matrix $\boldsymbol{\Phi}$ of the form

$$\hat{\boldsymbol{\Phi}}_A = \hat{\boldsymbol{\Phi}} - \sum_{i=1}^2\sum_{j=1}^2 w_{ij}\frac{\partial^2\boldsymbol{\Phi}}{\partial\sigma_i^2\partial\sigma_j^2}$$

where $\hat{\mathbf{\Phi}} = \mathbf{\Phi}(\hat{\sigma}_x^2, \hat{\sigma}_y^2), w_{ij} = \{\mathbf{W}\}_{ij}$ ($\mathbf{W}$ is given in (7)) and $\sigma_1^2 = \sigma_x^2, \sigma_2^2 = \sigma_y^2$. After computations we obtain

$$\sum_{i=1}^{2}\sum_{j=1}^{2} w_{ij}\frac{\partial^2\mathbf{\Phi}}{\partial\sigma_i^2\partial\sigma_j^2} = 0,$$

so $\hat{\mathbf{\Phi}}_A = \hat{\mathbf{\Phi}}$. The modified estimator $\hat{\mathbf{\Phi}}_A$ is recommended to use in the statistics

$$F = \frac{1}{2}\begin{pmatrix}\hat{a}-a\\ \hat{b}-b\end{pmatrix}' \hat{\mathbf{\Phi}}_A^{-1}\begin{pmatrix}\hat{a}-a\\ \hat{b}-b\end{pmatrix}.$$

Further approximation of F is in such a way that λF is $F_{2,u}$ distributed (Fisher-Snedecor distribution with 2 and u degrees of freedom). Analogically as Kenward and Roger considerations after tedious computations we obtain

$$\lambda = 1$$

and

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

If the true values of calibration line coefficients are a and b, then the following (approximative) distribution is valid:

$$F = \frac{1}{2}\frac{m}{b_0^2\hat{\sigma}_x^2 + \hat{\sigma}_y^2}\begin{pmatrix}\hat{a}-a\\ \hat{b}-b\end{pmatrix}'\begin{pmatrix} n & \mathbf{1}'\boldsymbol{\mu}_0\\ \boldsymbol{\mu}_0'\mathbf{1} & \boldsymbol{\mu}_0'\boldsymbol{\mu}_0\end{pmatrix} \times \begin{pmatrix}\hat{a}-a\\ \hat{b}-b\end{pmatrix} \overset{approx.}{\sim} F_{2,u}.$$

From that we get

$$\Pr\left\{\frac{1}{2}\frac{m}{b_0^2\hat{\sigma}_x^2 + \hat{\sigma}_y^2}\begin{pmatrix}\hat{a}-a\\ \hat{b}-b\end{pmatrix}'\begin{pmatrix} n & \mathbf{1}'\boldsymbol{\mu}_0\\ \boldsymbol{\mu}_0'\mathbf{1} & \boldsymbol{\mu}_0'\boldsymbol{\mu}_0\end{pmatrix} \times \begin{pmatrix}\hat{a}-a\\ \hat{b}-b\end{pmatrix} \leq F_{2,u}(1-\alpha)\right\} \overset{approx.}{=} 1-\alpha,$$

($F_{t,u}(1-\alpha)$ is the $(1-\alpha)$-quantile of $F_{t,u}$ distribution).

So,

$$\mathcal{C}_{(1-\alpha)}^{(EIV)} = \left\{\begin{pmatrix}a\\ b\end{pmatrix} : \frac{m}{2(b_0^2\hat{\sigma}_x^2 + \hat{\sigma}_y^2)}Q^{(EIV)} \leqq F_{2,u}(1-\alpha)\right\}$$

is the EIV (approximative) $(1-\alpha)$-confidence region for $(a, b)'$.

3 Results

3.1 Scheffé-type confidence region for the calibration line

By applying the Scheffé's method, see [7], we directly get the $100 \times (1-\alpha)\%$-confidence region for the calibration line $a + b\mu$ for all μ. In the case of the maximum likelihood method we get

$$\Pr\left\{\left|(\tilde{a}+\tilde{b}\mu) - (a+b\mu)\right| \leq \sqrt{\chi_2^2(1-\alpha)\frac{\tilde{b}^2\tilde{\sigma}_x^2 + \tilde{\sigma}_y^2}{m}\left(\frac{1}{n} + \frac{(\mu-\bar{\tilde{\mu}})^2}{\tilde{\boldsymbol{\mu}}'\tilde{\boldsymbol{\mu}} - n\bar{\tilde{\mu}}^2}\right)}\right\} = 1-\alpha, \tag{8}$$

where $\tilde{\boldsymbol{\mu}} = (\tilde{\mu}_1, \ldots, \tilde{\mu}_n)$ and $\bar{\tilde{\mu}} = (\mathbf{1}'\tilde{\boldsymbol{\mu}})/n$.

In the case of using the errors-in-variables model and the Kenward-Roger approximation we get

$$\Pr\left\{\left|(\hat{a}+\hat{b}\mu) - (a+b\mu)\right| \leq \sqrt{2F_{2,u}(1-\alpha)\frac{b_0^2\hat{\sigma}_x^2 + \hat{\sigma}_y^2}{m}\left(\frac{1}{n} + \frac{(\mu-\bar{\mu}_0)^2}{\boldsymbol{\mu}_0'\boldsymbol{\mu}_0 - n\bar{\mu}_0^2}\right)}\right\} = 1-\alpha, \tag{9}$$

where $\bar{\mu}_0 = (\mathbf{1}'\boldsymbol{\mu}_0)/n$.

This is directly used for *the multiple-use linear univariate calibration*, i.e. for measuring with calibrated device.

3.2 Multiple-use calibration – measuring with calibrated device

We will assume 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 the unobservable true value of the measurand.

First, we suggest to construct the approximate $(1-\alpha)$-confidence region for the calibration line, for small significance level $\alpha \in (0, 1)$, chosen by the user, according to (8) or (9).

Second, for small significance level $\gamma \in (0, 1)$, we suggest to construct the approximate $(1-\gamma)$-confidence interval for μ_x. For that we suggest to construct t-statistic with approximate t_v Student's t distribution

$$t = \frac{X-\mu_x}{\tilde{\sigma}_x} \overset{approx}{\sim} t_{\tilde{v}}, \quad \text{resp.} \quad t = \frac{X-\mu_x}{\hat{\sigma}_x} \overset{approx}{\sim} t_{\hat{v}},$$

where the degrees of freedom are approximated by the Satterthwaite's approximation, see [8]. In the case of ML method

$$\tilde{v} = nm,$$

see [6]. In the case of EIV approach

$$\hat{v} = \frac{2\hat{\sigma}_x^4}{w_{11}},$$

where w_{11} is element of the matrix $\boldsymbol{W}$ given in (7).

This leads to the approximate $(1-\gamma)$-confidence interval for unobservable value μ_x:

$$\mu_x \in \langle x - \tilde{\sigma}_x t_{\tilde{v}}(1-\gamma/2), x + \tilde{\sigma}_x t_{\tilde{v}}(1-\gamma/2)\rangle = \langle \tilde{\mu}_{xl}, \tilde{\mu}_{xu}\rangle$$

resp.

$$\mu_x \in \langle x - \hat{\sigma}_x t_{\hat{v}}(1-\gamma/2), x + \hat{\sigma}_x t_{\hat{v}}(1-\gamma/2)\rangle = \langle \hat{\mu}_{xl}, \hat{\mu}_{xu}\rangle,$$

$t_v(1-\gamma/2)$ is the $(1-\gamma/2)$-quantile of Student's t distribution with v degrees of freedom.

The suggested interval estimator for ν_x is given as the intersection of the bounds of the Scheffé-type $(1-\alpha)$-confidence region for the whole calibration line $a+b\mu$ and the limits of the $(1-\gamma)$-confidence interval for μ_x, and is given as $\langle \tilde{\nu}_{xl}, \tilde{\nu}_{xu}\rangle$ (ML method) or $\langle \hat{\nu}_{xl}, \hat{\nu}_{xu}\rangle$ (EIV method). In fact,

$$\begin{aligned}\tilde{\nu}_{xl} &= \tilde{a} + \tilde{b}\tilde{\mu}_{xl} \\ &\quad - \sqrt{\chi^2_2(1-\alpha)\frac{\tilde{b}^2\tilde{\sigma}^2_x + \tilde{\sigma}^2_y}{m}\left(\frac{1}{n} + \frac{(\tilde{\mu}_{xl} - \bar{\tilde{\mu}})^2}{\tilde{\boldsymbol{\mu}}'\tilde{\boldsymbol{\mu}} - n\bar{\tilde{\mu}}^2}\right)} \\ \tilde{\nu}_{xu} &= \tilde{a} + \tilde{b}\tilde{\mu}_{xu} \\ &\quad + \sqrt{\chi^2_2(1-\alpha)\frac{\tilde{b}^2\tilde{\sigma}^2_x + \tilde{\sigma}^2_y}{m}\left(\frac{1}{n} + \frac{(\tilde{\mu}_{xu} - \bar{\tilde{\mu}})^2}{\tilde{\boldsymbol{\mu}}'\tilde{\boldsymbol{\mu}} - n\bar{\tilde{\mu}}^2}\right)}, \quad (10)\end{aligned}$$

for the ML method and

$$\begin{aligned}\hat{\nu}_{xl} &= \hat{a} + \hat{b}\hat{\mu}_{xl} \\ &\quad - \sqrt{2F_{2,u}(1-\alpha)\frac{\hat{b}^2\hat{\sigma}^2_x + \hat{\sigma}^2_y}{m}\left(\frac{1}{n} + \frac{(\hat{\mu}_{xl} - \bar{\mu}_0)^2}{\boldsymbol{\mu}_0'\boldsymbol{\mu}_0 - n\bar{\mu}_0^2}\right)} \\ \hat{\nu}_{xu} &= \hat{a} + \hat{b}\hat{\mu}_{xu} \\ &\quad + \sqrt{2F_{2,u}(1-\alpha)\frac{\hat{b}^2\hat{\sigma}^2_x + \hat{\sigma}^2_y}{m}\left(\frac{1}{n} + \frac{(\hat{\mu}_{xu} - \bar{\mu}_0)^2}{\boldsymbol{\mu}_0'\boldsymbol{\mu}_0 - n\bar{\mu}_0^2}\right)}, (11)\end{aligned}$$

for the EIV based approach. Using Bonferroni's inequality, the intervals (10) and (11) are (approximative) at least $(1-\alpha-\gamma)$-confidence intervals for the (unobservable) value ν_x. Preliminary simulation study indicated that the suggested confidence intervals are conservative, i.e. "safe" and appropriate for metrological applications.

3.3 Example

In order to illustrate numerically the suggested methods for multiple-use linear calibration case, we have generated a set of artificial calibration data – a possible outcome of simple linear calibration experiment with replicated measurements, see Table 1.

Based on the calibration data from the considered experiment we get the estimated values of the model parameters:

- Based on the MLE method we get $\tilde{a} = 0.7405$, $\tilde{b} = 1.4522$, $\tilde{\sigma}^2_x = 0.1091$, $\tilde{\sigma}^2_y = 0.0038$, $\tilde{\mu}_1 = 0.8938$, $\tilde{\mu}_2 = 2.9491$, $\tilde{\mu}_3 = 5.0120$, $\tilde{\mu}_4 = 7.0903$, $\tilde{\mu}_5 = 9.1047$, and $\tilde{v} = 15$.

Table 1. Example data from comparative linear calibration experiment. The values x_{ij}, $i = 1,\dots n$, $j = 1,\dots,m$, represent m-times ($m = 3$) replicated measurements of n measurands ($n = 5$) with true values $\mu_i = (\nu_i - a)/b$ measured in units of the less precise measurement device. The values y_{ij}, $i = 1,\dots n$, $j = 1,\dots,m$, are the measurements of the measurands with their true values $\nu_i = a + b\mu_i$, measured in units of the more precise measurement device. Here, the true (unobservable) values of the model parameters are $a = 0.5$, $b = 1.5$, $\mu_1 = 1$, $\mu_2 = 3$, $\mu_3 = 5$, $\mu_4 = 7$, and $\mu_5 = 9$, and $\nu_1 = 2$, $\nu_2 = 5$, $\nu_3 = 8$, $\nu_4 = 11$, and $\nu_5 = 14$. The observed values x_{ij} and y_{ij} are realizations of mutually independent random variables $X_{ij} \sim N(\mu_i, \sigma^2_x)$ and $Y_{ij} \sim N(\nu_i, \sigma^2_y)$, respectively, with the true variances $\sigma^2_y = 0.15$ and $\sigma^2_y = 0.01$.

i	μ_i	x_{i1}	x_{i2}	x_{i3}	ν_i	y_{i1}	y_{i2}	y_{i3}
1	1	0.6086	0.7507	1.0000	2	2.0896	2.0569	1.9766
2	3	3.2380	3.1473	2.8769	5	5.0731	4.9744	5.0118
3	5	5.1966	4.6092	5.4241	8	8.0578	7.9623	8.0315
4	7	7.6555	6.9924	6.2742	11	11.0040	10.9704	11.1444
5	9	9.2290	8.9813	9.1658	14	14.0677	13.8525	13.9649

For chosen $\alpha = 0.01$, the $(1-\alpha)$-quantile of χ^2-distribution with 2 degrees of freedom is $\chi^2_2(1-\alpha) = 9.2103$. For chosen $\gamma = 0.05$, the $(1-\gamma/2)$-quantile of t-distribution with $\tilde{v}$ degrees of freedom is $t_{\tilde{v}}(1-\gamma/2) = 2.1314$.

- Based on the EIV approach (linearized EIV model with application of the Kenward-Roger method for estimation of the model parameters) we get $\hat{a} = 0.7405$, $\hat{b} = 1.4522$, $\hat{\sigma}^2_x = 0.1264$, $\hat{\sigma}^2_y = 0.0057$, $\hat{\mu}_1 = 0.8933$, $\hat{\mu}_2 = 2.9497$, $\hat{\mu}_3 = 5.0123$, $\hat{\mu}_4 = 7.0897$, $\hat{\mu}_5 = 9.1048$, and $u = 13.4244$. The element w_{11} of the matrix $\boldsymbol{W}$ is $w_{11} = 0.0025$, and from that we get $\hat{v} = 12.8762$.
  For chosen $\alpha = 0.01$, the $(1-\alpha)$-quantile of F-distribution with 2 and u degrees of freedom is $F_{2,u}(1-\alpha) = 6.6178$. For chosen $\gamma = 0.05$, the $(1-\gamma/2)$-quantile of t-distribution with $\hat{v}$ degrees of freedom is $t_{\hat{v}}(1-\gamma/2) = 2.1625$.

After calibration, the less precise device can be used (multiple-times) for estimation of the true value of measurand ν_x together with its (approximate) confidence interval, based on observed value x, which is considered to be a realization of random variable $X \sim N(\mu_x, \sigma^2_x)$.

Consider measurement of the measurand whose true (unobservable) value in units of the more precise device is $\nu_x = 11.75$. For estimation of the value ν_x we can use only the less precise device (in possibly different units). For example, here we have generated measurement $x = 7.1097$ as a realization of $X \sim N(\mu_x, \sigma^2_x)$ with $\mu_x = (11.75 - 0.5)/1.5 = 7.5$ and $\sigma^2_x = 0.15$. For chosen $\alpha = 0.01$ and $\gamma = 0.05$ we get the following confidence intervals for the true value ν_x:

- Based on the MLE method for estimation of the model parameters from (10) we get $\langle \tilde{\nu}_{xl}, \tilde{\nu}_{xu}\rangle = \langle 9.6223, 12.6138\rangle$ as an approximate, at least 94%, confidence interval for the (unobservable) value $\nu_x = 0.5 + 1.5 \times 7.5 = 11.75$.

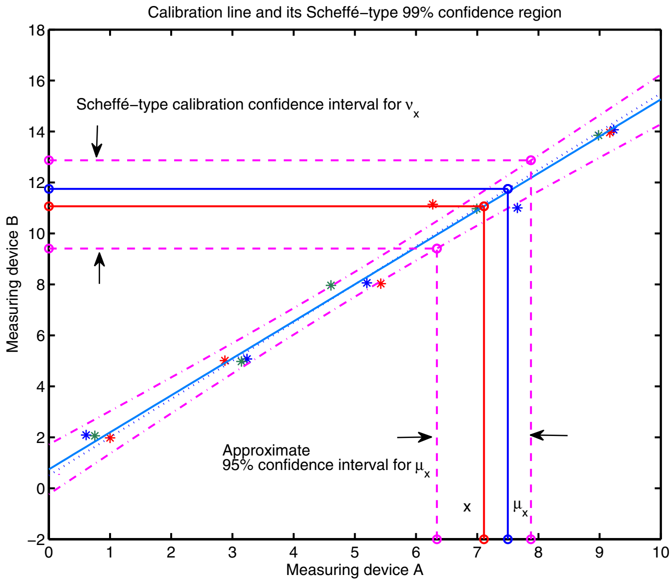

Fig. 1. Calibration line and its Scheffé-type confidence region constructed by the EIV approach. The thick dotted line represents the true calibration line, the solid line is the estimated calibration line together with the limits of the 99% confidence region (dashed-dotted lines). The dashed lines represent 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 $X \sim N(7.5, 0.15)$.

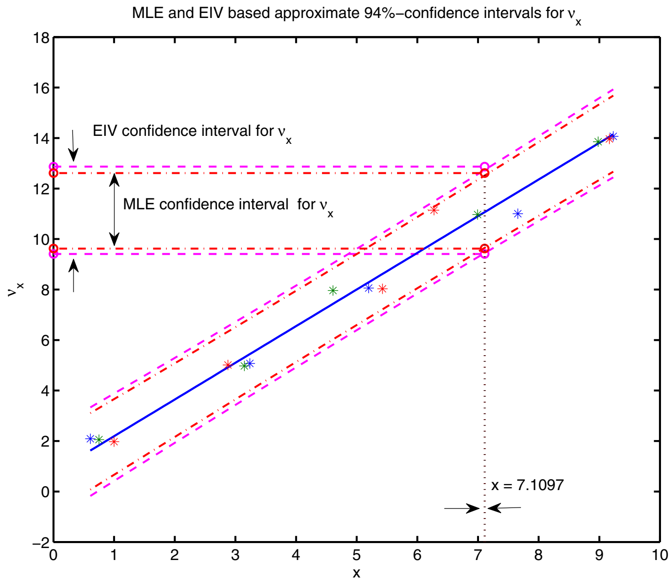

Fig. 2. Estimated calibration line and the approximate MLE and EIV confidence bounds for ν_x. The solid line is the estimated calibration line. The dashed-dotted lines represent the interval estimator for $\nu_x = a + b\mu_x$ based on the MLE method and the dashed lines represent the interval estimator for $\nu_x = a + b\mu_x$ based on the EIV method. Here $x = 7.1097$ is the realization of random variable $X \sim N(7.5, 0.15)$. Based on the MLE method, see (10), we get $\langle \tilde{\nu}_{xl}, \tilde{\nu}_{xu} \rangle = \langle 9.6223, 12.6138 \rangle$. Based on the EIV model and the Kenward-Roger method, see (11), we get $\langle \hat{\nu}_{xl}, \hat{\nu}_{xu} \rangle = \langle 9.4096, 12.8699 \rangle$.

– Based on the EIV model and the Kenward-Roger method for estimation of the model parameters from (11) we get $\langle \hat{\nu}_{xl}, \hat{\nu}_{xu} \rangle = \langle 9.4096, 12.8699 \rangle$ as an approximate, at least 94%, confidence interval for the (unobservable) value $\nu_x = 0.5 + 1.5 \times 7.5 = 11.75$.

Figure 1 illustrates construction of the approximate 94%-confidence interval for the (unobservable) value $\nu_x = 11.75$ based on the observed value $x = 7.1097$ by the EIV approach. In Figure 2 are plotted the bounds of the approximate MLE and EIV 94%-confidence regions for ν_x, see equations (10) and (11), for arbitrary observation x.

4 Discussion

As expected, the confidence interval for the true value of measurand, by using the calibrated measuring device and obtained via the EIV method, is in the considered example wider than the confidence interval obtained via the ML method. The EIV method with the Kenward-Roger approximation is suitable for small sample case (as it was suggested), while the ML method is based on asymptotic theory, i.e. is suitable for large samples. But this phenomenon still needs further statistical investigations, as well as the study of the statistical properties of suggested estimators with regard to true values of the calibration model parameters.

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