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LINEAR COMPARATIVE CALIBRATION WITH CORRELATED MEASUREMENTS

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The paper deals with the linear comparative calibration problem, i. e. the situation when both variables are subject to errors. Considered is a quite general model which allows to include possibly correlated data (measurements). From statistical point of view the model could be represented by the linear errors-in-variables (EIV) model. We suggest an iterative algorithm for estimation the parameters of the analysis function (inverse of the calibration line) and we solve the problem of deriving the approximate confidence region for the parameters. The confidence limits are derived using the concept of Kenward and Roger [5]. Their performance is investigated by simulation. The simulation results show that under reasonable restrictions the proposed confidence regions are very satisfactory for practical use.

Keywords: linear calibration, analysis function, regression with errors-in-variables, Kenward–Roger type approximation

AMS Subject Classification: 60F05, 62E10

1. INTRODUCTION

We consider the linear comparative calibration problem, i. e. the situation when both variables are subject to errors. We suggest a procedure for fitting the linear analysis function and consider the problem of deriving the approximate confidence region for the parameters of the analysis function. From statistical point of view the analysis function expresses the ideal (true, errorless) values of the measurand (the measured object, substance, or quantity) in units of the measuring instrument $\mathcal{Y}$ (typically the more precise measuring instrument) as a function of the true values of the measurand in units of the measuring instrument $\mathcal{X}$ (typically the less precise instrument). In other words, the analysis function expresses the relationship between the ideal (true, errorless) values of measuring the same object (substance, quantity) by two measuring instruments $\mathcal{X}$ and $\mathcal{Y}$, respectively.

In particular, we are interested in finding the proper estimators of the coefficients of the linear analysis function. We also suggest a method for constructing the Scheffé-

type confidence region for the whole linear analysis function.

The problem discussed in this paper was motivated by and is closely related to the approach discussed in the international standard ISO 6143 [2]: *Gas analysis – Comparison methods for determining and checking the composition of calibration gas mixtures*. However, here we consider a model that allows to incorporate possibly correlated data and combines the type A as well as type B uncertainties of the measurements (for more details on metrological interpretation see the international standard *Guide to the expression of uncertainty in measurement*, ISO GUM [1]).

From statistical point of view the model could be represented by the linear errors-in-variables (EIV) model, see e. g. Casella and Berger [3]. In a standard situation, the estimators of the analysis 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 [5]. Their performance is investigated by simulation that show that under reasonable restrictions the proposed confidence regions are very satisfactory for practical use.

2. THE CALIBRATION MODEL

Throughout the paper we shall assume that the following assumptions and restrictions for the calibration model hold true:

(i) For estimation of analysis function parameters and obtaining their confidence regions we perform a pre-planned calibration experiment with replicated measurements made by both instruments $\mathcal{Y}$ (the more precise one) and $\mathcal{X}$ (the less precise one), on a set of $n \geq 4$ suitably chosen objects (substances, quantities of interest), say $V_1, V_2, \ldots, V_n$, such that their true values μ_i, $i = 1, 2, \ldots, n$, in units of instrument $\mathcal{X}$, span its (that is of instrument $\mathcal{X}$) appropriate calibration range, are approximately equally spaced, with one value bellow the lower limit and one value above the upper limit of the calibration range. The measurements are made repeatedly m_{1i} times, $i = 1, \ldots, n$, for each object measured by the measuring instrument $\mathcal{X}$ and m_{2i} times, $i = 1, \ldots, n$, for each object measured by the measuring instrument $\mathcal{Y}$.

(ii) For the less precise instrument $\mathcal{X}$ the measurement result $x_{i,j}$ is a realization of normally distributed random variable $X_{i,j}$, $i = 1, 2, \ldots, n$, $j = 1, 2, \ldots, m_{1i}$, with mean value μ_i, which is the ideal (true, errorless) value of measurand V_i given in units of the less precise instrument. The dispersion of $X_{i,j}$ is ${}_x\widetilde{\Delta}_i\sigma_x^2 + \{\mathbf{\Sigma}_x\}_{ii}$, $i = 1, 2, \ldots, n$, $j = 1, 2, \ldots, m_{1i}$, where σ_x^2 is an unknown scalar factor, ${}_x\widetilde{\Delta}_i$, $i = 1, 2, \ldots, n$, are known positive constants and $\mathbf{\Sigma}_x$ is an

$n \times n$ known positive semidefinite matrix which, in accordance with ISO GUM [1], corresponds to *the type B covariance matrix*. The covariances $\mathrm{cov}(X_{ij}, X_{kl})$ are also assumed to be known and are $\mathrm{cov}(X_{ij}, X_{kl}) = \{\mathbf{\Sigma}_x\}_{i,k}$, for all $i, k = 1, 2, \ldots, n$, $j = 1, 2, \ldots, m_{1i}$, $l = 1, 2, \ldots, m_{1k}$. Denoting $\bar{X}_i = \frac{1}{m_{1i}} \sum_{j=1}^{m_{1i}} X_{i,j}$, $i = 1, 2, \ldots, n$ it is supposed that the random vector $\bar{\boldsymbol{X}} = (\bar{X}_1, \ldots, \bar{X}_n)'$ is normally distributed with the mean $\boldsymbol{\mu} = (\mu_1, \ldots, \mu_n)'$ and covariance matrix $\sigma_x^2 \mathbf{\Delta}_x + \mathbf{\Sigma}_x$, i. e.

$$\bar{\boldsymbol{X}} \sim N\left(\boldsymbol{\mu}, \sigma_x^2 \mathbf{\Delta}_x + \mathbf{\Sigma}_x\right), \tag{1}$$

where, $\mathbf{\Delta}_x = \mathrm{diag}\left(\frac{{}_x\tilde{\Delta}_1}{m_{11}}, \cdots, \frac{{}_x\tilde{\Delta}_n}{m_{1n}}\right)$ is a (known) diagonal matrix.

The particular form of the matrix $\mathbf{\Sigma}_x$ depends on specific calibration problem, and usually is given in the instrument $\mathcal{X}$ calibration certificate, or is evaluated by other means, including expert judgement, see e. g. Gleser [4]. However, often the following very simple forms are used: $\mathbf{\Sigma}_x^I = \tau_x^2\, \mathrm{diag}\{\mu_1^2, \mu_2^2, \ldots, \mu_n^2\}$ and $\mathbf{\Sigma}_x^{II} = \tau_x^2\, \boldsymbol{\mu}\boldsymbol{\mu}'$, where τ_x^2 is a known positive constant.

Similarly, we will assume that

$$\bar{\boldsymbol{Y}} \sim N\left(\boldsymbol{\nu}, \sigma_y^2 \mathbf{\Delta}_y + \mathbf{\Sigma}_y\right). \tag{2}$$

(iii) The random vectors $\bar{\boldsymbol{X}}$ and $\bar{\boldsymbol{Y}}$ are independent.

(iv) We assume that over the typical range of values of ν and μ (the range of interest) the true, however unknown, analysis function is, in fact, a linear function, i. e. $\nu = a + b\mu$, given by the (unknown) parameters a and b.

The less precise instrument $\mathcal{X}$ is said to be the calibrated instrument.

Based on the above assumptions (i) – (iv) we get the statistical model of the calibration experiment:

$$\begin{pmatrix} \bar{\boldsymbol{X}} \\ \bar{\boldsymbol{Y}} \end{pmatrix} \sim N\left[\begin{pmatrix} \boldsymbol{\mu} \\ \boldsymbol{\nu} \end{pmatrix}, \begin{pmatrix} \sigma_x^2 \mathbf{\Delta}_x + \mathbf{\Sigma}_x & \mathbf{0} \\ \mathbf{0} & \sigma_y^2 \mathbf{\Delta}_y + \mathbf{\Sigma}_y \end{pmatrix}\right], \tag{3}$$

with linear restriction on the true means $\boldsymbol{\mu}$ and $\boldsymbol{\nu}$

$$\boldsymbol{\nu} = a\mathbf{1}_{n,1} + b\boldsymbol{\mu}, \tag{4}$$

where $\mathbf{1}_{n,1} = \mathbf{1} = (1, 1, \ldots, 1)'$. The (unknown) parameters of the model are a, b, $\boldsymbol{\mu}$, $\boldsymbol{\nu}$, σ_x^2 and σ_y^2, where a and b are the parameters of the linear analysis function.

As all the parameters a, b, $\boldsymbol{\mu}$, and $\boldsymbol{\nu}$ are unknown, the model (3) with nonlinear restrictions on the parameters (4) is a nonlinear regression model, that could be also interpreted as an error-in-variables (EIV) model, see e. g. Casella and Berger [3].

3. ESTIMATION OF THE PARAMETERS

To estimate the parameters of the model (3) – (4) we suggest an iterative algorithm. For similar approach see Kubáček and Kubáčková [6], Kubáček and Kubáčková [7], and Wimmer, Witkovský and Savin [10].

First, we propose to linearize the model (3) – (4) by using Taylor series expansion about $\boldsymbol{\mu}_0 = (\mu_{01}, \ldots, \mu_{0n})'$ and b_0 (the values chosen close to the true values of the parameters $\boldsymbol{\mu}$ and b). So, $\boldsymbol{\mu} = \boldsymbol{\mu}_0 + \delta\boldsymbol{\mu}$, $b = b_0 + \delta b$ and the new parameters of the approximate linear model are $\delta\boldsymbol{\mu} = (\delta\mu_1, \delta\mu_2, \ldots, \delta\mu_n)'$, $\boldsymbol{\nu}$, a, δb, σ_x^2, σ_y^2. The linearized regression model is:

$$\begin{pmatrix} \bar{\boldsymbol{X}} - \boldsymbol{\mu}_0 \\ \bar{\boldsymbol{Y}} \end{pmatrix} \sim N\left[\begin{pmatrix} \delta\boldsymbol{\mu} \\ \boldsymbol{\nu} \end{pmatrix}, \begin{pmatrix} \sigma_x^2\boldsymbol{\Delta}_x + \boldsymbol{\Sigma}_x & \boldsymbol{0} \\ \boldsymbol{0} & \sigma_y^2\boldsymbol{\Delta}_y + \boldsymbol{\Sigma}_y \end{pmatrix} \right], \tag{5}$$

with linear constraints

$$b_0\boldsymbol{\mu}_0 + b_0\delta\boldsymbol{\mu} - \boldsymbol{\nu} + a\boldsymbol{1}_{n,1} + \delta b\boldsymbol{\mu}_0 = \boldsymbol{0}. \tag{6}$$

Assuming that the model (5) with linear constraints (6) is (approximately) correct, the BLUEs (best linear unbiased estimators) of the parameters $\boldsymbol{\mu} = \boldsymbol{\mu}_0 + \delta\boldsymbol{\mu}$, $\boldsymbol{\nu}$, a and δb are, see e. g. Kubáčková [8], given by

$$\hat{\boldsymbol{\mu}} = \bar{\boldsymbol{X}} + b_0\left(\sigma_x^2\boldsymbol{\Delta}_x + \boldsymbol{\Sigma}_x\right)\boldsymbol{Q}\left(\bar{\boldsymbol{Y}} - b_0\bar{\boldsymbol{X}}\right), \tag{7}$$

$$\hat{\boldsymbol{\nu}} = \bar{\boldsymbol{Y}} - \left(\sigma_y^2\boldsymbol{\Delta}_y + \boldsymbol{\Sigma}_y\right)\boldsymbol{Q}\left(\bar{\boldsymbol{Y}} - b_0\bar{\boldsymbol{X}}\right), \tag{8}$$

$$\begin{pmatrix} \hat{a} \\ \hat{\delta b} \end{pmatrix} = \left([\boldsymbol{1}\ \boldsymbol{\mu}_0]'\boldsymbol{Z}^{-1}[\boldsymbol{1}\ \boldsymbol{\mu}_0]\right)^{-1}[\boldsymbol{1}\ \boldsymbol{\mu}_0]'\boldsymbol{Z}^{-1}\left(\bar{\boldsymbol{Y}} - b_0\bar{\boldsymbol{X}}\right), \tag{9}$$

with $\hat{b} = b_0 + \hat{\delta b}$. The matrix $\boldsymbol{Q}$ is given by

$$\boldsymbol{Q} = \left(\boldsymbol{M}_{[\boldsymbol{1}\ \boldsymbol{\mu}_0]}\boldsymbol{Z}\boldsymbol{M}_{[\boldsymbol{1}\ \boldsymbol{\mu}_0]}\right)^{+}, \tag{10}$$

($\boldsymbol{A}^{+}$ is the Moore–Penrose generalized inverse of the matrix $\boldsymbol{A}$, see e. g. Rao and Mitra [9]), where

$$\boldsymbol{M}_{[\boldsymbol{1}\ \boldsymbol{\mu}_0]} = \boldsymbol{I}_{n,n} - [\boldsymbol{1}\ \boldsymbol{\mu}_0]\begin{pmatrix} n & \boldsymbol{1}'\boldsymbol{\mu}_0 \\ \boldsymbol{\mu}_0'\boldsymbol{1} & \boldsymbol{\mu}_0'\boldsymbol{\mu}_0 \end{pmatrix}^{-1}[\boldsymbol{1}\ \boldsymbol{\mu}_0]', \tag{11}$$

$$\boldsymbol{Z} = \left[b_0^2\left(\sigma_x^2\boldsymbol{\Delta}_x + \boldsymbol{\Sigma}_x\right) + \left(\sigma_y^2\boldsymbol{\Delta}_y + \boldsymbol{\Sigma}_y\right)\right]. \tag{12}$$

The covariance matrix of $(\hat{a}, \hat{\delta b})'$ is

$$\operatorname{cov}\left\{\begin{pmatrix} \hat{a} \\ \hat{\delta b} \end{pmatrix}\right\} = \operatorname{cov}\left\{\begin{pmatrix} \hat{a} \\ \hat{b} \end{pmatrix}\right\} = \left([\boldsymbol{1}\ \boldsymbol{\mu}_0]'\boldsymbol{Z}^{-1}[\boldsymbol{1}\ \boldsymbol{\mu}_0]\right)^{-1}. \tag{13}$$

Assuming that the model (5) with linear constraints (6) is true, the estimators (7), (8), and (9) have multivariate normal distributions, respectively.

The estimators (7), (8), (9), and the covariance matrix (13) strongly depend on the chosen initial values b_0 and $\boldsymbol{\mu}_0$, the unknown parameters σ_x^2 and σ_y^2, and on the quality of linearization of the originally nonlinear model (3) – (4) at these values that, on the other hand, strongly depends on the experimental design of the calibration experiment. If there is no specific prior information on the true value of the

parameters b, $\boldsymbol{\mu}$, σ_x^2 and σ_y^2, a natural choice of the initial values, estimated from the measured data, could be the following:

$$\hat{\boldsymbol{\mu}}_0 = \bar{\boldsymbol{X}}, \tag{14}$$

$$\hat{b}_0 = \frac{n\bar{\boldsymbol{X}}'\bar{\boldsymbol{Y}} - (\mathbf{1}'\bar{\boldsymbol{X}})(\mathbf{1}'\bar{\boldsymbol{Y}})}{n\bar{\boldsymbol{X}}'\bar{\boldsymbol{X}} - (\mathbf{1}'\bar{\boldsymbol{X}})^2}. \tag{15}$$

The natural estimator of σ_x^2 is

$$\hat{\sigma}_x^2 = \frac{1}{(\sum_{i=1}^n m_{1i} - n)} \sum_{i=1}^n \sum_{j=1}^{m_{1i}} \frac{\left(X_{i,j} - \bar{X}_i\right)^2}{m_{1i}\{\boldsymbol{\Delta}_x\}_{i,i}}, \tag{16}$$

which is an unbiased estimator of σ_x^2 distributed as $(\sigma_x^2/p)\chi_p^2$, where $p = \sum_{i=1}^n m_{1i} - n$ and χ_p^2 represents a random variable with chi-square distribution with p degrees of freedom, $\{\boldsymbol{\Delta}_x\}_{i,i}$ stands for the ith diagonal element of the matrix $\boldsymbol{\Delta}_x$, $i = 1, \ldots, n$.

The natural estimator of σ_y^2 is

$$\hat{\sigma}_y^2 = \frac{1}{(\sum_{i=1}^n m_{2i} - n)} \sum_{i=1}^n \sum_{k=1}^{m_{2i}} \frac{\left(Y_{i,k} - \bar{Y}_i\right)^2}{m_{2i}\{\boldsymbol{\Delta}_y\}_{i,i}}, \tag{17}$$

which is an unbiased estimator of σ_y^2 distributed as $(\sigma_y^2/q)\chi_q^2$, where $q = \sum_{i=1}^n m_{2i} - n$ and χ_q^2 represents the random variable with chi-square distribution with q degrees of freedom, $\{\boldsymbol{\Delta}_y\}_{i,i}$ stands for the ith diagonal element of the matrix $\boldsymbol{\Delta}_y$, $i = 1, \ldots, n$. The estimators (16) and (17) are independent with the covariance matrix

$$\boldsymbol{W} = \operatorname{cov}\left\{\begin{pmatrix} \hat{\sigma}_x^2 \\ \hat{\sigma}_y^2 \end{pmatrix}\right\} = \begin{pmatrix} \frac{2\sigma_x^4}{p} & 0 \\ 0 & \frac{2\sigma_y^4}{q} \end{pmatrix}. \tag{18}$$

The parameters $\boldsymbol{\mu}$, $\boldsymbol{\nu}$, a, and b could be estimated by (7), (8), and (9) where, instead of the unknown values $\boldsymbol{\mu}_0$, b_0, σ_x^2, and σ_y^2, we use their estimates: $\boldsymbol{\mu}_0 = \hat{\boldsymbol{\mu}}_0^{(est)}$, $b_0 = \hat{b}_0^{(est)}$, $\sigma_x^2 = \hat{\sigma}_x^{2(est)}$, and $\sigma_y^2 = \hat{\sigma}_y^{2(est)}$ given by equations (14) – (17), respectively.

The estimation procedure could be iterated in such a way until convergence is reached according to the following algorithm:

Algorithm.

(i) Set $k = 0$ and the initial estimates of the parameters $\boldsymbol{\mu}$ and b: $\hat{\boldsymbol{\mu}}^{(0)} = \hat{\boldsymbol{\mu}}_0^{(est)}$, $\hat{b}^{(0)} = \hat{b}_0^{(est)}$ given by equations (14) – (15). Further, set $\sigma_x^2 = \hat{\sigma}_x^{2(est)}$ and $\sigma_y^2 = \hat{\sigma}_y^{2(est)}$ given by (16) – (17).

(ii) For given $\hat{\boldsymbol{\mu}}^{(k)}$ and $\hat{b}^{(k)}$ set $\boldsymbol{\mu}_0 = \hat{\boldsymbol{\mu}}^{(k)}$, $b_0 = \hat{b}^{(k)}$ and estimate the parameters $\boldsymbol{\mu}$, $\boldsymbol{\nu}$, a, and b as $\hat{\boldsymbol{\mu}}^{(k+1)}$, $\hat{\boldsymbol{\nu}}^{(k+1)}$, $\hat{a}^{(k+1)}$, and $\hat{b}^{(k+1)}$ by (7), (8), and (9). Set $k = k + 1$ and repeat the point (ii) until convergence is reached (we do not specify here the explicite form of the stopping rule).

(iii) By using $\sigma_x^2 = \hat{\sigma}_x^{2(est)}$ and $\sigma_y^2 = \hat{\sigma}_y^{2(est)}$ calculate the estimate $\hat{\boldsymbol{W}}^{(est)}$ of the covariance matrix $\boldsymbol{W}$, according to (18).

(iv) After reaching the convergence report the final results of the estimation procedure: $\hat{a}^{(est)}$, $\hat{b}^{(est)}$, $\hat{\boldsymbol{\mu}}^{(est)}$, $\hat{\boldsymbol{\nu}}^{(est)}$, $\hat{\sigma}_x^{2(est)}$, $\hat{\sigma}_y^{2(est)}$, and $\hat{\boldsymbol{W}}^{(est)}$.

Remark 1. The general calibration model (3) – (4) covers different specific submodels. For instance, if repeated measurements give the same numerical result for each measurement of the same object or, when we have only one measurement for each object, then we set ${}_y\widetilde{\Delta}_i = 0$, $i = 1, 2, \ldots, n$ (and consequently $\sigma_y^2 = 0$).

Remark 2. If some of the covariance matrices depend on the true, however unknown parameters $\boldsymbol{\mu}$ or $\boldsymbol{\nu}$, respectively, we suggest to approximate those parameters by their natural estimates $\bar{\boldsymbol{x}} = (\bar{x}_1, \ldots, \bar{x}_n)'$ and $\bar{\boldsymbol{y}} = (\bar{y}_1, \ldots, y_n)'$, respectively. In particular, the type B covariance matrix $\boldsymbol{\Sigma}_x^I = \tau_x^2 \operatorname{diag}\{(\mu_1^2, \mu_2^2, \ldots, \mu_n^2)'\}$ is approximated by $\tilde{\boldsymbol{\Sigma}}_x^I = \tau_x^2 \operatorname{diag}\{(\bar{x}_1^2, \bar{x}_2^2, \ldots, \bar{x}_n^2)'\}$ and $\boldsymbol{\Sigma}_x^{II} = \tau_x^2 \boldsymbol{\mu}\boldsymbol{\mu}'$ is approximated by $\tilde{\boldsymbol{\Sigma}}_x^{II} = \tau_x^2 \bar{\boldsymbol{x}}\bar{\boldsymbol{x}}'$. In the same way we could obtain the approximations of the covariance matrices $\boldsymbol{\Sigma}_y^I$ and $\boldsymbol{\Sigma}_y^{II}$.

4. CONFIDENCE REGIONS FOR THE PARAMETERS AND ANALYSIS LINE

In this section we will derive the approximate confidence region for the analysis line parameters $(a, b)'$ and the approximate Scheffé-type confidence region for whole analysis line. In derivation of the approximate distribution of the suggested F statistic (a scaled Wald statistic) we will follow steps suggested by Kenward and Roger [5] for making small sample inferences for fixed effects in linear mixed models from restricted maximum likelihood (REML). This approximate distribution is later used to construct the confidence regions. Under assumption that the linear (linearized) model (5) with linear constraints (6) is correct it could be interpreted as a special form of the linear mixed model with constraints on the parameters and with two variance components σ_x^2 and σ_y^2. Instead of using the REML estimators we will use the estimates that are obtained from the proposed estimation procedure given by the Algorithm.

Following Kenward and Roger [5] the following terms are required for derivation of the approximate distribution of the F statistic:

$$\begin{aligned}
\hat{\boldsymbol{\Phi}} &= \left([\mathbf{1}\ \boldsymbol{\mu}_0]'\hat{\boldsymbol{Z}}^{-1}[\mathbf{1}\ \boldsymbol{\mu}_0]\right)^{-1}, \\
\hat{\boldsymbol{P}}_1 &= -b_0^2[\mathbf{1}\ \boldsymbol{\mu}_0]'\hat{\boldsymbol{Z}}^{-1}\boldsymbol{\Delta}_x\hat{\boldsymbol{Z}}^{-1}[\mathbf{1}\ \boldsymbol{\mu}_0], \\
\hat{\boldsymbol{P}}_2 &= -[\mathbf{1}\ \boldsymbol{\mu}_0]'\hat{\boldsymbol{Z}}^{-1}\boldsymbol{\Delta}_y\hat{\boldsymbol{Z}}^{-1}[\mathbf{1}\ \boldsymbol{\mu}_0], \\
\hat{\boldsymbol{Q}}_{11} &= b_0^4[\mathbf{1}\ \boldsymbol{\mu}_0]'\hat{\boldsymbol{Z}}^{-1}\boldsymbol{\Delta}_x\hat{\boldsymbol{Z}}^{-1}\boldsymbol{\Delta}_x\hat{\boldsymbol{Z}}^{-1}[\mathbf{1}\ \boldsymbol{\mu}_0], \\
\hat{\boldsymbol{Q}}_{22} &= [\mathbf{1}\ \boldsymbol{\mu}_0]'\hat{\boldsymbol{Z}}^{-1}\boldsymbol{\Delta}_y\hat{\boldsymbol{Z}}^{-1}\boldsymbol{\Delta}_y\hat{\boldsymbol{Z}}^{-1}[\mathbf{1}\ \boldsymbol{\mu}_0],
\end{aligned} \tag{19}$$

where

$$\hat{\boldsymbol{Z}} = \left[b_0^2\left(\hat{\sigma}_x^{2(est)}\boldsymbol{\Delta}_x + \boldsymbol{\Sigma}_x\right) + \left(\hat{\sigma}_y^{2(est)}\boldsymbol{\Delta}_y + \boldsymbol{\Sigma}_y\right)\right], \tag{20}$$

with $\boldsymbol{\mu}_0 = \hat{\boldsymbol{\mu}}^{(est)}$ and $b_0 = \hat{b}^{(est)}$. Further,

$$\hat{\boldsymbol{\Lambda}} = \hat{\boldsymbol{\Phi}} \left\{ \hat{W}_{11} \left(\hat{\boldsymbol{Q}}_{11} - \hat{\boldsymbol{P}}_1 \hat{\boldsymbol{\Phi}} \hat{\boldsymbol{P}}_1 \right) + \hat{W}_{22} \left(\hat{\boldsymbol{Q}}_{22} - \hat{\boldsymbol{P}}_2 \hat{\boldsymbol{\Phi}} \hat{\boldsymbol{P}}_2 \right) \right\} \hat{\boldsymbol{\Phi}}, \tag{21}$$

where $\hat{W}_{ij}$ is the (i,j)th element of the matrix $\hat{\boldsymbol{W}}^{(est)}$, and finally

$$\hat{\boldsymbol{\Phi}}_A = \hat{\boldsymbol{\Phi}} + 2\hat{\boldsymbol{\Lambda}}. \tag{22}$$

Further, we will use

$$\begin{aligned} A_1 &= \hat{W}_{11} \left(\operatorname{tr} \left(\hat{\boldsymbol{P}}_1 \hat{\boldsymbol{\Phi}} \right) \right)^2 + \hat{W}_{22} \left(\operatorname{tr} \left(\hat{\boldsymbol{P}}_2 \hat{\boldsymbol{\Phi}} \right) \right)^2, \\ A_2 &= \hat{W}_{11} \operatorname{tr} \left(\hat{\boldsymbol{P}}_1 \hat{\boldsymbol{\Phi}} \hat{\boldsymbol{P}}_1 \hat{\boldsymbol{\Phi}} \right) + \hat{W}_{22} \operatorname{tr} \left(\hat{\boldsymbol{P}}_2 \hat{\boldsymbol{\Phi}} \hat{\boldsymbol{P}}_2 \hat{\boldsymbol{\Phi}} \right), \\ B &= \frac{1}{4} \left(A_1 + 6A_2 \right), \end{aligned} \tag{23}$$

$$c_1 = \frac{g}{8 - 2g}, \quad c_2 = \frac{2 - g}{8 - 2g}, \quad c_1 = \frac{1}{2}, \tag{24}$$

where

$$g = \frac{3A_1 - 6A_2}{4A_2}, \tag{25}$$

and

$$E^* = \frac{1}{1 - A_2/2}, \quad V^* = \frac{2(1 + c_1 B)}{(1 - c_2 B)^2 (2 - B)}, \quad \varrho = \frac{V^*}{2(E^*)^2}. \tag{26}$$

Finally we get

$$m = \frac{8\varrho}{2\varrho - 1}, \quad \lambda = \frac{m}{E^*(m-2)}. \tag{27}$$

The statistical inference about parameters $(a, b)'$, of the analysis line will be based on the following scaled Wald-type statistic

$$F = \frac{\lambda}{2} \begin{pmatrix} \hat{a} - a_* \\ \hat{b} - b_* \end{pmatrix}' \hat{\boldsymbol{\Phi}}_A^{-1} \begin{pmatrix} \hat{a} - a_* \\ \hat{b} - b_* \end{pmatrix}, \tag{28}$$

where the scale parameter λ is given in (27) and $(a_*, b_*)'$ represent the hypothetical values of the parameters of the analysis line. If the parameters $(a_*, b_*)'$ are true, then, using the arguments given by Kenward and Roger [5], the distribution of the scaled Wald-type F-statistic given by (28) is approximately given by

$$F \sim \mathcal{F}_{2,m}, \tag{29}$$

where $\mathcal{F}_{2,m}$ represents the Fisher–Snedecor F-distribution with 2 and m degrees of freedom, m given by (27).

Under the assumption that the approximation (29) is acceptable, the set

$$\mathcal{C}_1 = \left\{ \begin{pmatrix} a \\ b \end{pmatrix} : \begin{pmatrix} \hat{a} - a \\ \hat{b} - b \end{pmatrix}' \hat{\boldsymbol{\Phi}}_A^{-1} \begin{pmatrix} \hat{a} - a \\ \hat{b} - b \end{pmatrix} \leq \frac{2}{\lambda} F_{2,m,(1-\alpha)} \right\} \tag{30}$$

is the approximate $(1-\alpha)\times 100\%$-confidence region for the (unknown) parameters a, b of the linear analysis function. ($F_{2,m,(1-\alpha)}$ denotes the $(1-\alpha)$-quantile, $\alpha \in (0,1)$,

of the Fisher–Snedecor F-distribution with 2 and m degrees of freedom). It means that confidence region $\mathcal{C}$ covers the true values of the parameters, a_* and b_*, with probability approximately equal to $(1-\alpha)$, i. e.

$$\Pr\left\{\begin{pmatrix} a_* \\ b_* \end{pmatrix} \in \mathcal{C}_1\right\} \approx 1-\alpha. \tag{31}$$

By applying the Scheffé's Theorem, we directly get the $(1-\alpha)\times 100\%$-confidence region $\mathcal{C}_2$ for the whole linear analysis function, i. e. $a+b\mu$ for all $\mu \in \langle \mu_l, \mu_u\rangle$ (the interval represents the typical range of the calibration experiment):

$$\begin{aligned} \mathcal{C}_2 \quad = \quad & \Big\{(a+b\mu) : \Big|(\hat{a}+\hat{b}\mu) - (a+b\mu)\Big| \\ & \leq \sqrt{\tfrac{2}{\lambda} F_{2,m,(1-\alpha)}(1,\mu)\widehat{\boldsymbol{\Phi}}_A(1,\mu)'},\ \forall \mu \in \langle \mu_l, \mu_u\rangle\Big\}, \end{aligned} \tag{32}$$

with

$$\Pr\left\{(a+b\mu) \in \mathcal{C}_2,\ \forall \mu \in \langle \mu_l, \mu_u\rangle\right\} \approx 1-\alpha. \tag{33}$$

5. SIMULATION STUDY

The approximate confidence regions (30) and (32) are based on a sequence of assumptions and approximations. We have checked the basic statistical properties (the empirical coverage probabilities) of the proposed approximate confidence region (30) for the parameters a and b of the analysis function $\nu = a + b\mu$. This was done by Monte Carlo simulations for a wide spectrum of experimental designs.

We have assumed the model (3) – (4), with $\boldsymbol{\Delta}_x = \frac{1}{m}\boldsymbol{I}$, $\boldsymbol{\Sigma}_x = \boldsymbol{0}$, $\boldsymbol{\Delta}_y = \frac{1}{m}\boldsymbol{I}$, and $\boldsymbol{\Sigma}_y = \tau_y^2\,\boldsymbol{\nu}\boldsymbol{\nu}'$, then, 10000 independent replications of the calibration experiment were generated, and the empirical coverage probability of the nominal 95 % confidence region (30) was estimated, for each combination of the following values of the parameters:

- $n \in \{5, 10\}$ and $m \in \{2, 5, 10\}$.
- $a = 0$ and $b \in \{10, \sqrt{10}, 1\}$.
- $\sigma_x \in \{0.01, 0.05, 0.1\}$, $\sigma_y \in \{0, 0.05, 0.1\}$, and $\tau_y \in \{0.01, 0.005, 0.001\}$.

For each fixed n the vector $\boldsymbol{\mu}$ was randomly generated with its values uniformly distributed over the interval $(n, 2n)$. Then the vector $\boldsymbol{\nu}$ was calculated, $\boldsymbol{\nu} = a\boldsymbol{1}_{n,1} + b\boldsymbol{\mu}$ and the measurements $(X_{i,j}, Y_{i,j})$, $i = 1, \ldots, n$, $j = 1, \ldots, m$, were generated according to (3). The Algorithm was used to estimate the parameters of the model. The Kenward–Roger's method was used to estimate $\hat{\boldsymbol{\Phi}}_A$, m and λ, see (22) and (27). Finally, for given true values of a and b the validity of the inequality in (30) (the coverage of the true values) was checked. The empirical coverage probability was calculated based on independent replications of the calibration experiment.

In Figure we present the empirical coverage probabilities of the nominal 95 % confidence region (30) for the parameters a and b of the analysis function $\nu = a + b\mu$,

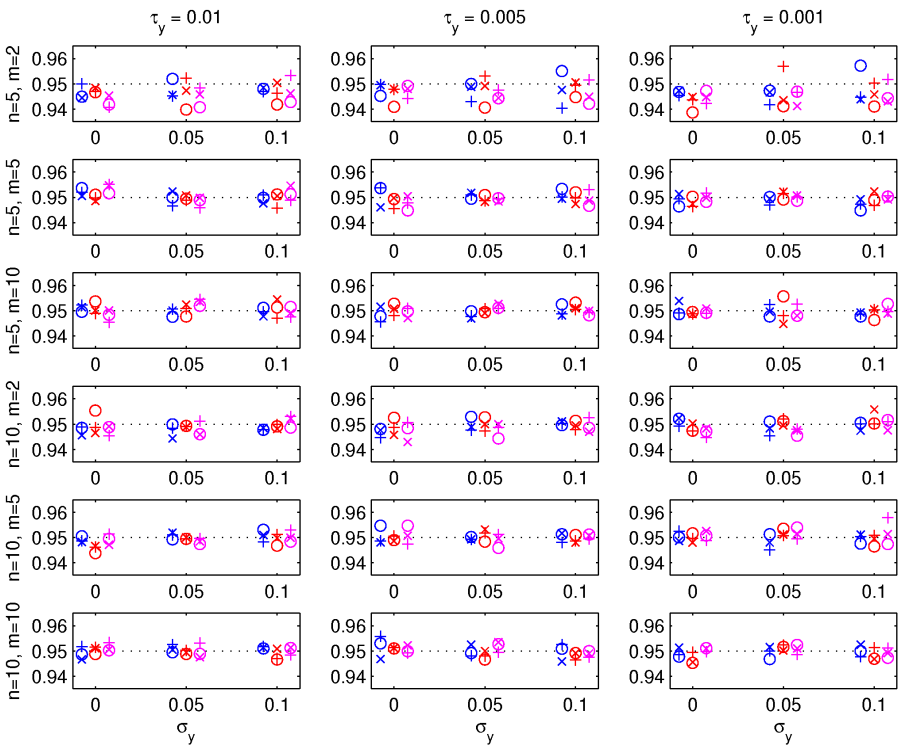

Fig. The empirical coverage probabilities of the nominal 95% confidence region (30) for the parameters a and b of the analysis function $\nu = a + b\mu$, based on 10 000 Monte Carlo runs for each specific design. Here we use the symbol $\circ$ for designs with $b = 10$, $\times$ for designs with $b = \sqrt{10}$, and $+$ for designs with $b = 1$. The left tripple of symbols for each σ_y denotes the designs with $\sigma_x = 0.01$, the middle tripple denotes the designs with $\sigma_x = 0.05$, and the right tripple denotes the designs with $\sigma_x = 0.1$.

based on 10 000 Monte Carlo runs for each specific design. We could conclude that for considered class of experimental designs the empirical coverage probabilities of the confidence region (30) are very close to the nominal 0.95 level. The minimum observed value was 0.9386 and the maximum observed value was 0.9579.

6. CONCLUSIONS

In this paper we have suggested an iterative algorithm for estimation of the parameters of the linear analysis function and considered the problem of deriving the approximate confidence region for the parameters of the analysis line using the concept of Kenward and Roger [5]. The covariance structure of the considered calibration

model allows to consider possibly correlated data.

The performance of the proposed confidence region was investigated by simulation. The simulation results imply that the empirical coverage probabilities of this confidence region for the parameters of the analysis line are very close to the nominal level and consequently are very satisfactory for practical use.

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