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Homoscedastic controlled calibration model

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In the context of the usual calibration model, we consider the case in which the independent variable is unobservable, but a pre-fixed value on its surrogate is available. Thus, considering controlled variables and assuming that the measurement errors have equal variances we propose a new calibration model. Likelihood-based methodology is used to estimate the model parameters and the Fisher information matrix is used to construct a confidence interval for the unknown value of the regressor variable. A simulation study is carried out to asses the effect of the measurement error on the estimation of the parameter of interest. This new approach is illustrated with an example.

KEYWORDS: regression model; linear calibration model; measurement error model; Berkson model

1. INTRODUCTION

In the first stage of a calibration problem, a pair of data sample (x_i, Y_i), $i = 1, 2, \ldots n$ is observed. In the second stage, it is observed one or more values, which are the responses corresponding to a single unknown value of the regressor variable, X_0. The first and second stage equations of the usual linear calibration model are defined, respectively, as

$$Y_i = \alpha + \beta x_i + \epsilon_i, \quad i = 1, 2 \ldots, n \tag{1.1}$$

$$Y_{0i} = \alpha + \beta X_0 + \epsilon_i, \quad i = n+1, n+2, \ldots, n+k \tag{1.2}$$

It is considered the following assumptions:

- $x_1, x_2, \ldots, x_n$ take fixed values, which are considered as true values.
- $\epsilon_1, \epsilon_2, \ldots, \epsilon_{n+k}$ are independent and normally distributed with mean 0 and variance σ_ϵ^2.

The model parameters are α, β, X_0 and σ_ϵ^2 and the main interest is to estimate the quantity X_0.

The maximun likelihood estimators of the usual calibration model are given by

$$\hat{\alpha} = \bar{Y} - \hat{\beta}\bar{x}, \qquad \hat{\beta} = \frac{S_{xY}}{S_{xx}}, \qquad \hat{X}_0 = \frac{\bar{Y}_0 - \hat{\alpha}}{\hat{\beta}} \tag{1.3}$$

$$\sigma_\epsilon^2 = \frac{1}{n+k}\left[\sum_{i=1}^{n}(Y_i - \hat{\alpha} - \hat{\beta}x_i)^2 + \sum_{i=n+1}^{n+k}(Y_{0i} - \bar{Y}_0)^2\right] \tag{1.4}$$

where

$$\bar{x} = \frac{1}{n}\sum_{i=1}^{n} x_i, \quad \bar{Y} = \frac{1}{n}\sum_{i=1}^{n} Y_i, \quad S_{xY} = \frac{1}{n}\sum_{i=1}^{n}(x_i - \bar{x})(Y_i - \bar{Y})$$

$$S_{xx} = \frac{1}{n}\sum_{i=1}^{n}(x_i - \bar{x})^2, \quad \bar{Y}_0 = \frac{1}{n}\sum_{i=n+1}^{n+k} Y_{0i}$$

In Reference [1] an approximate expression is derived for the variance of the estimator $\hat{X}_0$, which is derived through the propagation error law. Another approximation for the variance of $\hat{X}_0$ is given by the Fisher information of $\theta = (\alpha, \beta, X_0, \sigma_\epsilon^2)$ which, after some length algebraic manipulations, it can be shown to be given by

$$I(\theta) = \frac{1}{\sigma_\epsilon^2}\begin{pmatrix} n+k & kX_0 + n\bar{x} & k\beta & 0 \\ kX_0 + n\bar{x} & kX_0^2 + \sum_{i=1}^{n} x_i^2 & \kappa\beta X_0 & 0 \\ k\beta & \kappa\beta X_0 & k\beta^2 & 0 \\ 0 & 0 & 0 & \frac{n+k}{2\sigma_\epsilon^2} \end{pmatrix} \tag{1.5}$$

The maximum likelihood estimator of $\hat{\theta} = (\hat{\alpha}, \hat{\beta}, \hat{X}_0, \hat{\sigma_\epsilon^2})$ has approximately normal distribution with mean θ and covariance matrix $I(\theta)^{-1}$, when $k = qn$, $q \in Q^+$ and $n \longrightarrow \infty$. Thus, the approximation of order n^{-1} for the variance of $\hat{X}_0$ is given by

$$V_1(\hat{X}_0) = \frac{\sigma_\epsilon^2}{\beta^2}\left[\frac{1}{k} + \frac{1}{n} + \frac{(\bar{X} - X_0)^2}{nS_{xx}}\right] \tag{1.6}$$

On the other hand, in Reference [2] the size k of the second stage is considered fixed, so that expanding $\hat{X}_0$ in Taylor series around the point (α, β) and ignoring terms of order less than n^{-2}, we can find the following approximations for the bias

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and variance of $\hat{X}_0$, respectively:

$$\text{Bias}(\hat{X}_0) = \frac{\sigma_\epsilon^2 (X_0 - \bar{x})}{n\beta^2 S_{xx}} \tag{1.7}$$

$$V_2(\hat{X}_0) = \frac{\sigma_\epsilon^2}{\beta^2}\left[\frac{1}{k} + \frac{1}{n} + \frac{(\bar{X} - X_0)^2}{nS_{xx}} + \frac{3\sigma_\epsilon^2}{nk\beta^2 S_{xx}}\right] \tag{1.8}$$

In order to construct a confidence interval for X_0, we consider that

$$\frac{\hat{X}_0 - X_0}{\sqrt{\hat{V}(\hat{X}_0)}} \xrightarrow{D} N(0,1) \tag{1.9}$$

where $\hat{V}(\hat{X}_0)$ is the estimated variance computed according to Equations (1.6) or (1.8). Hence, the approximated confidence interval for X_0 with a confidence level $(1-\alpha)$, is given by

$$\left(\hat{X}_0 - z_{\frac{\alpha}{2}}\sqrt{\hat{V}(\hat{X}_0)}, \hat{X}_0 + z_{\frac{\alpha}{2}}\sqrt{\hat{V}(\hat{X}_0)}\right) \tag{1.10}$$

where $z_{\frac{\alpha}{2}}$ is the quantile of order $(1 - \frac{\alpha}{2})$ of the standard normal distribution.

The usual calibration problem has been discussed in the literature for several decades (see References [2–7]). An illustration of this model is presented for example in Reference [8]. We can find a review of the literature on statistical calibration in Reference [9], where some approaches to the solution of the calibration problem are summarized.

This model encounters applications in different areas, but it is not well suited in some instances as, for example, in chemical analysis, where the preparation process of standard solutions are subject to measurement error [1].

There exist some situations, as mentioned above, where one is unable to observe x_i directly. In this case, Reference [10] defines two types of observations: *controlled* and *uncontrolled*.

In the *uncontrolled* situation, instead of observing x_i, one observes the sum

$$X_i = x_i + \delta_i, \quad i = 1, \ldots, n \tag{1.11}$$

where δ_i is a $(0, \sigma_\delta^2)$ random variable.

Assuming that x_i is a unknown fixed constant, the model defined by the classical linear regression Equations (1.1) and (1.11) is named as functional model [11,12], while the model defined by the Equations (1.1) and (1.11) with x_i regarded as random variable is called structural model [11,12]. On the other hand, the model defined by (1.1), (1.2), and (1.11) is called as the functional or structural calibration model if x_i is assumed as a fixed value or a random variable, respectively [12].

The *controlled* observation is defined by a pre-fixed value X_i according to the experimenter convenience and a procedure is established in order to attain the pre-fixed value. The experiment gives the unobserved x_i and it is such that

$$x_i = X_i - \delta_i, \quad i = 1, \ldots, n \tag{1.12}$$

where δ_i is a $(0, \sigma_\delta^2)$ random variable. The model defined by Equations (1.1) and (1.12) is known as Berkson regression model [13]. The model defined by (1.1), (1.2), and (1.12) has not been considered before in the measurement error literature and in this work it will be called as the *controlled calibration model*.

In the functional and structural calibration model, the regressor X_i is random variable, whereas in the controlled calibration model it is assumed as pre-fixed by the experimenter.

This work is organized as follows. In Section 2, we derive the maximum likelihood estimators of the homoscedastic controlled calibration model by considering both cases: σ_δ^2 *unknown* and *known*. In Section 3, a simulation study is undertaken to investigate the sensitivity of parameter estimates of the proposed model (Proposed-M). In Section 4, an example is presented to illustrate our new approach. In Section 5, the concluding remark is presented.

2. PARAMETER ESTIMATION

In this section we study the controlled calibration model. From the Equations (1.1), (1.2), and (1.12) we can write

$$Y_i = \alpha + \beta X_i + (\epsilon_i - \beta\delta_i), \quad i = 1, 2 \ldots, n \tag{2.1}$$

$$Y_{0i} = \alpha + \beta X_0 + \epsilon_i, \quad i = n+1, n+2, \ldots, n+k \tag{2.2}$$

with the following assumptions for the random errors:

- ϵ_i are independent $N(0, \sigma_\epsilon^2)$ random variables.
- $E(\delta_i)$=0, $(V\delta_i)$= $\sigma_{\delta_i}^2$.
- $\text{cov}(\delta_i, \delta_j)$=0 for any $i \neq j$.
- $\text{cov}(\epsilon_i, \delta_j)$=0 for all i, j.

Some comments are in order here. The variable X_i in Equation (2.1) is controlled and the error model $(\epsilon_i - \beta\delta_i)$ is independent of X_i. The error model in Equation (2.2) is only in function of error measure ϵ_i related to Y_{0i}, this model assumes that there is no error in the preparation sample related to parameter X_0. We define the homoscedastic controlled calibration model by considering that the errors δ_i are independent and normally distributed with mean 0 and constant variance, σ_δ^2. The study of this model is carried out following similar analysis to the usual calibration model as summarized above.

The maximum likelihood estimator for the homoscedastic controlled calibration model is derived in the following. The logarithm of the likelihood function is given by:

$$l\left(\alpha, \beta, X_0, \sigma_\epsilon^2, \sigma_\delta^2\right) \propto -\frac{n}{2}\log\left(\sigma_\epsilon^2 + \beta^2\sigma_\delta^2\right) - \frac{k}{2}\log\left(\sigma_\epsilon^2\right) - \frac{1}{2}\left[\frac{1}{\sigma_\epsilon^2 + \beta^2\sigma_\delta^2}\sum_{i=1}^{n}(Y_i - \alpha - \beta X_i)^2 + \frac{1}{\sigma_\epsilon^2}\sum_{i=n+1}^{n+k}(Y_{0i} - \alpha - \beta X_0)^2\right] \tag{2.3}$$

Solving $\partial l/\partial\alpha = 0$ and $\partial l/\partial X_0 = 0$ we have the maximum likelihood estimator of α and X_0, which are given, respectively, by

$$\hat{\alpha} = \bar{Y} - \hat{\beta}\bar{X} \quad \text{and} \quad \hat{X}_0 = \frac{\bar{Y}_0 - \hat{\alpha}}{\hat{\beta}} \tag{2.4}$$

From Equations (2.3) and (2.4), it follows that the likelihood for $(\beta, \sigma_\epsilon^2, \sigma_\delta^2)$ can be written as:

$$l\left(\beta, \sigma_\epsilon^2, \sigma_\delta^2\right) \propto -\frac{n}{2}\log\left(\sigma_\epsilon^2+\beta^2\sigma_\delta^2\right)-\frac{k}{2}\log\left(\sigma_\epsilon^2\right) -\frac{1}{2}\left[\frac{1}{\sigma_\epsilon^2+\beta^2\sigma_\delta^2}\sum_{i=1}^{n}[(Y_i-\bar{Y})-\beta(X_i-\bar{X})]^2 + \frac{1}{\sigma_\epsilon^2}\sum_{i=n+1}^{n+k}(Y_{0i}-\bar{Y}_0)^2\right] \quad (2.5)$$

Next, we consider two cases for σ_δ^2. First, we obtain the maximum likelihood estimator of β, σ_ϵ^2, and σ_δ^2 from Equation (2.5). In the second case we assume that the variance σ_δ^2 is known and obtain the maximum likelihood estimators for β and σ_ϵ^2.

Case 1. *Unknown* variance σ_δ^2.

The maximum likelihood estimators of β, σ_δ^2, and σ_ϵ^2 (the proof is given in Appendix A) are given, respectively, by

$$\hat{\beta}=\frac{S_{XY}}{S_{XX}}$$

$$\hat{\sigma}_\delta^2=\frac{(S_{YY}-2\hat{\beta}S_{XY}+\hat{\beta}^2S_{XX})-\hat{\sigma}_\epsilon^2}{\hat{\beta}^2}$$

$$\hat{\sigma}_\epsilon^2=S_{Y_0Y_0}$$

The variance of $\hat{X}_0$, derived from the Fisher information matrix (see Appendix B), is given by:

$$V_1(\hat{X}_0)=\frac{\sigma_\epsilon^2}{\beta^2}\left[\frac{1}{k}+\frac{\gamma}{n\sigma_\epsilon^2}+\frac{\gamma}{\sigma_\epsilon^2}\frac{(\bar{X}-X_0)^2}{nS_{XX}}\right] \quad (2.6)$$

where

$$\gamma=\beta^2\sigma_\delta^2+\sigma_\epsilon^2 \quad (2.7)$$

Considering k fixed and expanding $\hat{X}_0$ in a Taylor series around (α, β) and ignoring terms of order less than n^{-2}, it can be shown that the bias and variance of $\hat{X}_0$ (the proof is given in Appendix C), are given by:

$$\text{Bias}(\hat{X}_0)=\frac{\gamma(\bar{X}-X_0)}{n\beta^2S_{XX}} \quad (2.8)$$

$$V_2(\hat{X}_0)=\frac{\sigma_\epsilon^2}{\beta^2}\left[\frac{1}{k}+\frac{\gamma}{n\sigma_\epsilon^2}+\frac{\gamma(\bar{X}-X_0)^2}{n\sigma_\epsilon^2S_{XX}}+\frac{3\gamma}{nk\beta^2S_{XX}}\right] \quad (2.9)$$

We can observe that the estimator of X_0 is biased, but it is asymptotically unbiased.

With relation to the variance of the estimator $\hat{X}_0$, let us notice that when $k=qn$, $q\in Q^+$, and ignoring the terms of order less than n^{-1} the variance in Equation (2.9) coincide with the variance given in Equation (2.6), which was found through the Fisher information. Equation (2.6) considers large sample sizes in the first and second stages (n and k), whereas (2.9) considers large sample sizes in the first stage and a fixed sample size in the second stage.

Notice that when $\sigma_\delta^2=0$, Equations (2.6) and (2.9) coincide with (1.6) and (1.8) of the usual model (Usual-M), respectively.

Case 2. *Known* variance σ_δ^2.

Assuming now that σ_δ^2 is known and equating to zero the partial derivative of Equation (2.5) with respect to the parameters β and σ_ϵ^2, we have the following equations, respectively:

$$\hat{\beta}\sigma_\delta^2(\hat{\sigma}_\epsilon^2+\hat{\beta}^2\sigma_\delta^2-S_{YY}+\hat{\beta}S_{XY})=(S_{XY}-\hat{\beta}S_{XX})\hat{\sigma}_\epsilon^2 \quad (2.10)$$

and

$$\frac{kS_{Y_0Y_0}}{(\hat{\sigma}_\epsilon^2)^2}-\frac{k}{\hat{\sigma}_\epsilon^2}=\frac{n}{\hat{\sigma}_\epsilon^2+\hat{\beta}^2\sigma_\delta^2}-\frac{S_{YY}-2\hat{\beta}S_{XY}+\hat{\beta}^2S_{XX}}{(\hat{\sigma}_\epsilon^2+\hat{\beta}^2\sigma_\delta^2)^2} \quad (2.11)$$

The estimates of β and σ_ϵ^2 are obtained using some iterative method to solve Equations (2.10) and (2.11).

The variance of $\hat{X}_0$, derived from the Fisher information matrix (see Appendix D), is given by

$$V(\hat{X}_0)=\frac{\sigma_\epsilon^2}{\beta^2}\left[\frac{1}{k}+\frac{\gamma}{n\sigma_\epsilon^2}+\frac{\gamma}{\sigma_\epsilon^2}E\right] \quad (2.12)$$

where γ is defined in Equation (2.7) and

$$E=\frac{nX_0^2\sigma_\epsilon^4+kX_0^2\gamma^2-2nX_0\bar{X}\sigma_\epsilon^4-2kX_0\bar{X}\gamma^2+n\bar{X}^2\sigma_\epsilon^4+k\bar{X}^2\gamma^2}{(n\sigma_\epsilon^4+k\gamma^2)\sum_{i=1}^{n}X_i^2+2nk\beta^2\gamma\sigma_\delta^4-n^2\bar{X}^2\sigma_\epsilon^4-nk\bar{X}^2\gamma^2}$$

Notice that if $\sigma_\delta^2=0$, the Expression (2.12) is reduced to (1.6).

To construct a confidence interval for X_0, for both cases σ_δ^2 *unknown* and *known*, we consider the interval (1.10), where $\hat{V}(\hat{X}_{0C})$ is the estimated variance that follows from (2.6), (2.9), or (2.12).

3. SIMULATION STUDY

In this section we present a simulation study for both cases of the homoscedastic controlled calibration model: σ_δ^2 known and unknown. The objective of this section is to study the performance of the estimators of the Proposed-M and verify the impact by considering erratically the Usual-M.

It was considered 5000 samples generated from the homoscedastic controlled calibration model. In all samples, the value of the parameters α and β were 0.1 and 2, respectively. The range of values for the controlled variable was [0,2]. The fixed values for the controlled variable were $x_1=04$, $x_i=x_{i-1}+2/n-1$, $i=2,\ldots,n$, and the parameter values X_0 were 0.01 (extreme inferior value), 0.8 (near to the central value), and 1.9 (extreme superior value). It was considered $\sigma_\epsilon^2=0.04$ and the parameter values of σ_δ^2 were 0.01 and 0.1, which are named, respectively, as small and large variances. For the first and second stages we consider the sample of sizes $n=5, 20, 100$ and $k=2, 20, 100$, respectively.

The empirical mean bias is given by $\sum_{j=1}^{5000}(\hat{X}_0-X_0)/5000$ and the empirical mean squared error (MSE) is given by $\sum_{j=1}^{5000}(\hat{X}_0-X_0)^2/5000$. The mean estimated variance of $\hat{X}_0$ is given by $\sum_{j=1}^{5000}\hat{V}(\hat{X}_0)/5000$, with $\hat{V}(\hat{X}_0)=\hat{V}_1(\hat{X}_0)$ or $\hat{V}_2(\hat{X}_0)$, where $\hat{V}_1(\hat{X}_0)$ is the estimated variance of (1.6), (2.6), or (2.12) and $\hat{V}_2(\hat{X}_0)$ is the estimated variance of (2.9). The theoretical variances of $\hat{X}_0$ denoted as $V_1(\hat{X}_0)$ and $V_2(\hat{X}_0)$ are referred, respectively, to the Expressions (1.6), (2.6) or (2.12) and (2.9) evaluated on the relevant parameter values. In Appendix E it is presented the simulation results.

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Tables E1, E2, E5, and E6 present the empirical bias, the empirical MSE, the theoretical variance, and the estimated variance of X_0. In these tables, it is considered only the variance (1.6) of the Usual-M, because based on a simulation study in Reference [15] it was shown that the variances (1.6) and (1.8) give similar results.

Tables E3, E4, and E7 present the covering percentages and the confidence interval amplitudes constructed with a 95% confidence level for the parameter X_0. In Table E3, the covering percentages $\%_1$ and $\%_2$ and amplitudes A_1 and A_2 are referred to the confidence intervals constructed using the Equations (2.6) and (2.12).

Tables E1–E4 consider the homoscedastic controlled calibration model assuming that σ_δ^2 is unknown.

In Table E1 the empirical bias and MSE of $\hat{X}_0$ are little and an addition in the size of the variance σ_δ^2, described in Table E2, causes an increase in the bias and MSE. Moreover, we have that the bias and MSE of $\hat{X}_0$ are smaller when X_0 is near to the center value of the variation interval of the variable X. These tables show that for all n, k, and X_0, the theoretical variances obtained using the Expressions (2.12) and (2.9) are equal. This fact occurs also for the mean estimated variances. We verify also that when $n \geq 20$ and $k \geq 20$ the theoretical variances and the mean estimated variances from the Proposed-M are approximately equal. Observing these tables, we can also notice that there exist differences between the mean estimated variances of the Usual- and Proposed-Ms.

Analyzing Tables E3 and E4, we observe that for all n and X_0 when it is adopted erratically the Usual-M, the amplitudes decrease very much as the size of k increases. This causes the covering percentage to decrease moving away from 95%. Whereas, adopting the Proposed-M it is observed that when k increases the confidence interval amplitude decreases, but the covering percentages increase approaching 95%. Notice that the covering percentage $\%_1$ and $\%_2$ and the amplitudes A_1 and A_2 are approximately equal, the amplitudes are very small for $X_0 = 0.8$. In these tables, we observe that when $k = 20$ or 100 and when n increases, the amplitudes of the intervals decrease and the covering percentages approach 95%. In most cases, the covering percentages obtained through the Proposed-M are greater than that for the Usual-M results and are close to 95%.

Tables E5 and E7 describe the results for the controlled homoscedastic calibration model with σ_δ^2 known. The iterative method Quasi-Newton [14] has been used.

In Tables E5 and E6 we have that the empirical bias and SME decrease as the size of n or k increases and they are small when X_0 is near to the central value of the variation interval, $X_0 = 0.8$. When σ_δ^2 is small (Table E5), for all n and k, the empirical values of MSE from the Usual- and Proposed-M are close to the theoretical variance, but only the mean estimated variance from the Proposed-M is close to the theoretical variance. When σ_δ^2 is large (Table E6), in general, the empirical MSE and the mean estimated variance from the Usual- and Proposed-M are different, but the values supplied by the Proposed-M are very close to the theoretical variance.

Analyzing Table E7, we can make similar comments to the ones we made about Tables E3 and E4.

Table I. Concentration (mg/g) and intensity of the standard solutions of cromo and cadmium element

Cromo element		Cadmium element	
X_i	Intensity	X_i	Intensity
0.05	6 455 900	0.05	489 733
0.11	13 042 933	0.10	9 706
0.26	32 621 733	0.25	2 341 333
0.79	97 364 500	0.73	6973
1.05	129 178 100	1.01	9 685 667

4. APLICATION

In this section we test our model, considering both cases σ_δ known and unknown, using the data supplied by the chemical laboratory of the 'Instituto de Pesquisas Tecnológicas (IPT)'—Brasil. We also consider the Usual-M in order to observe the performance of the Proposed-M. Our main interest is to estimate the unknown concentration value X_0 of two samples A and B of the chemical elements cromo and cadmium.

Table I presents the fixed values of concentration of the standard solutions and the corresponding intensities for the cromo and cadmium element, which are supplied by the plasma spectrometry method. These data are referred to as the first stage of the calibration model.

Table II. Intensity of the sample solutions A and B of cromo and cadmium element

Intensity for cromo element		Intensity for cadmium element	
Sample A	Sample B	Sample A	Sample B
1465.0	10173.6	0.679	5066
1351.0	10516.9	0.6837	5027
1495.6	10352.2	0.6846	5085

Table III. Estimates of α, β, X_0, $V(\hat{X}_0)$, and the confidence interval amplitude $U(X_0)$ from the usual and proposed model for the samples A and B of cromo element

	Sample A			Sample B		
		Proposed-M			Proposed-M	
Parameters	Usual-M	Unknown σ_δ^2	Known σ_δ^2	Usual-M	Unknown σ_δ^2	Known σ_δ^2
α	123 574	123 574	123 889	123 574	123 574	124 021
β	1.23E+05	1.23E+05	1.23E+05	1.23E+05	1.23E+05	1.23E+05
X_0	0.011	0.011	0.011	0.083	0.083	0.083
$V(\hat{X}_0)$	9.80E-07	9.15E-07	1.35E-06	1.16E-06	1.13E-06	1.71E-06
σ_δ^2	—	1.60E-06	—	—	5.48E-07	—
$U(X_0)$	2.55E-03	2.46E-03	2.99E-03	2.77E-03	2.73E-03	3.36E-03

Table IV. Estimates of α, β, X_0, $V(\hat{X}_0)$, and the confidence interval amplitude $U(X_0)$ from the usual and proposed model for the samples A and B of cadmium element

	Sample A			Sample B		
		Proposed-M			Proposed-M	
Parameters	Usual-M	Unknown σ_δ^2	Known σ_δ^2	Usual-M	Unknown σ_δ^2	Known σ_δ^2
α	−0.156	−0.156	−0.158	−0.156	−0.156	−0.158
β	95.828	95.828	95.831	95.828	95.828	95.831
X_0	8.75E-03	8.75E-03	8.77E-03	0.054	0.054	0.054
$V(\hat{X}_0)$	4.06E-06	3.72E-06	1.26E-06	3.81E-06	3.32E-06	1.17E-06
σ_δ^2	—	8.31E-06	—	—	8.24E-06	—
$U(X_0)$	5.18E-03	4.96E-03	2.89E-03	5.02E-03	4.68E-03	2.78E-03

Table II presents the intensities corresponding to three sample solutions from the samples A and B. These data are referred to as the second stage of the calibration model.

Tables III and IV describe the estimates of α, β, X_0, $V(\hat{X}_0)$, σ_δ^2, and the confidence interval amplitude $U(X_0)$ from the homoscedastic controlled calibration model of the samples A and B for the chemical elements cromo and cadmium. The values of the variance σ_δ^2 considered as known are obtained from an external study carried out by the IPT, which are $\sigma_\delta^2 = 2.5865E-06$ for the cromo element and $\sigma_\delta^2 = 0.0017E+02$ for the cadmium element. As seen in Section 2, in order to obtain the estimates of the parameters β and σ_ϵ^2 of the Proposed-M when σ_δ^2 is known, iterative methods are required. In order to solve the system of Equations (2.10) and (2.11) it was used the Quasi-Newton iterative method. It is also presented the estimates from the Usual-M. The estimates of the variance of $\hat{X}_0$ are computed using the relevant Expressions (1.6), (2.6), or (2.12). The amplitude $U(X_0)$ is given by the product of the squared root of the estimated variance of $\hat{X}_0$ and 1.96.

In Tables III and IV we can observe that the estimates of α and β supplied by the Usual-M are equal to the Proposed-M when σ_δ^2 is unknown and they are equal for samples A and B, this occurs because the expressions of the estimators $\hat{\alpha}$ and $\hat{\beta}$ of both models are equal and they only depend on the first stage of the calibration model. These estimates are slightly different when compared with the estimates from the Proposed-M when σ_δ^2 is known. With respect to the estimate of X_0, we observe that there is no difference in the estimates supplied by the Usual- and the Proposed-Ms of the cromo and cadmium element in both samples A and B, respectively. The estimates of the concentration of the sample A, of the elements cromo and cadmium, are outside of the variation range of the standard solution concentrations. We verify that, except to the known σ_δ^2 case of the cromo element, the estimates of the variance of $\hat{X}_0$ and the amplitude $U(X_0)$ from the Usual-M are greater than the estimates supplied by the both Proposed-Ms.

5. CONCLUDING REMARKS

In general, the simulation study reveals that the Proposed-M is sensible to the presence of error related to the independent variable and gives better results in contrast to the Usual-M results. It was noticed that when the error variance σ_δ^2 increases, the mean estimated variance of $\hat{X}_0$ obtained using the Usual-M moves away from the theoretical value. In the example above, the confidence interval amplitude from the Proposed-Ms are supplied by the incorporation of error due to the lecture of equipment and the preparation of the standard solutions. It is observed that despite the classical model only considers the error originated from the lecture of the equipment, the amplitude is greater than obtained by the new approach.

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APPENDIX A

We derive the maximum likelihood estimator for β, σ_ϵ^2, and σ_δ^2.

Taking the partial derivative of Equation (2.5) with respect to β, σ_ϵ^2, and σ_δ^2 and equating to zero we obtain, respectively:

$$\hat{\beta}\hat{\sigma}_\delta^2(\hat{\sigma}_\epsilon^2 + \hat{\beta}^2\hat{\sigma}_\delta^2 - S_{YY} + \hat{\beta}S_{XY}) = (S_{XY} - \hat{\beta}S_{XX})\hat{\sigma}_\epsilon^2 \quad \text{(A-1)}$$

$$\hat{\sigma}_\epsilon^2 + \hat{\beta}^2\hat{\sigma}_\delta^2 = S_{YY} - 2\hat{\beta}S_{XY} + \hat{\beta}^2 S_{XX} \quad \text{(A-2)}$$

$$\frac{kS_{Y_0Y_0}}{(\hat{\sigma}_\epsilon^2)^2} - \frac{k}{\hat{\sigma}_\epsilon^2} = \frac{n}{\hat{\sigma}_\epsilon^2 + \hat{\beta}^2\hat{\sigma}_\delta^2} - \frac{n(S_{YY} - 2\hat{\beta}S_{XY} + \hat{\beta}^2 S_{XX})}{(\hat{\sigma}_\epsilon^2 + \hat{\beta}^2\hat{\sigma}_\delta^2)^2} \quad \text{(A-3)}$$

where $S_{XX} = \frac{1}{n}\sum_{i=1}^{n}(X_i - \bar{X})^2$, $S_{XY} = \frac{1}{n}\sum_{i=1}^{n}(X_i - \bar{X})(Y_i - \bar{Y})$, $S_{YY} = \frac{1}{n}\sum_{i=1}^{n}(Y_i - \bar{Y})^2$, and $S_{Y_0Y_0} = \frac{1}{k}\sum_{i=n+1}^{n+k}(Y_{0i} - \bar{Y}_0)^2$, and the relevant estimator notation has been introduced. From Equations (A-1) and (A-2) we have the following equations:

$$(\hat{\beta}S_{XX} - S_{XY})(S_{YY} - 2\hat{\beta}S_{XY} + \hat{\beta}^2 S_{XX}) = 0$$

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hence

$$\hat{\beta}S_{XX} - S_{XY} = 0 \quad \text{or} \tag{A-4}$$

$$S_{YY} - 2\hat{\beta}S_{XY} + \hat{\beta}^2 S_{XX} = 0 \tag{A-5}$$

Therefore, from Equation (A-4), we have that $\hat{\beta} = S_{XY}/S_{XX}$. But, according to the Cauchy–Schwarz inequality, $S_{XX}S_{YY} \geq S_{XY}^2$, hence Equation (A-5) has real roots if and only if $Y_i = cX_i$, where c is a constant.

The estimator of σ_δ^2 can be obtained from the Equation (A-2)

$$\hat{\sigma}_\delta^2 = \frac{(S_{YY} - 2\hat{\beta}S_{XY} + \hat{\beta}^2 S_{XX}) - \hat{\sigma}_\epsilon^2}{\hat{\beta}^2}$$

Likewise, from Equations (A-2) and (A-3) we obtain the estimator of the variance σ_ϵ^2

$$\hat{\sigma}_\epsilon^2 = S_{Y_0Y_0} \tag{A-6}$$

APPENDIX B

In order to find the variance of $\hat{X}_0$, we need to derive the Fisher information matrix of $\theta = (\alpha, \beta, X_0, \sigma_\delta^2, \sigma_\epsilon^2)$, which can be shown to be given by:

$$I(\theta) = \begin{pmatrix} \frac{n}{\gamma} + \frac{k}{\sigma_\epsilon^2} & \frac{n\bar{X}}{\gamma} + \frac{kX_0}{\sigma_\epsilon^2} & \frac{k\beta}{\sigma_\epsilon^2} & 0 & 0 \\ \frac{n\bar{X}}{\gamma} + \frac{kX_0}{\sigma_\epsilon^2} & \frac{\sum_{i=1}^n X_i^2}{\gamma} + \frac{2n\beta^2\sigma_\delta^4}{\gamma^2} + \frac{kX_0^2}{\sigma_\epsilon^2} & \frac{k\beta X_0}{\sigma_\epsilon^2} & \frac{n\beta^3\sigma_\delta^2}{\gamma^2} & \frac{n\beta\sigma_\delta^2}{\gamma^2} \\ \frac{k\beta}{\sigma_\epsilon^2} & \frac{k\beta X_0}{\sigma_\epsilon^2} & \frac{k\beta^2}{\sigma_\epsilon^2} & 0 & 0 \\ 0 & \frac{n\beta^3\sigma_\delta^2}{\gamma^2} & 0 & \frac{n\beta^4}{2\gamma^2} & \frac{n\beta^2}{2\gamma^2} \\ 0 & \frac{n\beta\sigma_\delta^2}{\gamma^2} & 0 & \frac{n\beta^2}{2\gamma^2} & \frac{n}{2\gamma^2} + \frac{k}{2\sigma_\epsilon^4} \end{pmatrix}$$

When $k = qn$, $q \in Q^+$, and $n \longrightarrow \infty$, the estimator $\hat{\theta}$ is approximately normally distributed with mean θ and variance $I(\theta)^{-1}$, thus we have that the approximate variance to order n^{-1} for $\hat{X}_0$ is given by

$$V_1(\hat{X}_0) = \frac{\sigma_\epsilon^2}{\beta^2}\left[\frac{1}{k} + \frac{\gamma}{n\sigma_\epsilon^2} + \frac{\gamma}{\sigma_\epsilon^2}\frac{(\bar{X} - X_0)^2}{nS_{XX}}\right] \tag{B-1}$$

APPENDIX C

In the following we derive the bias (2.8) and the variance (2.9) of the estimator $\hat{X}_0$ from the homoscedastic controlled calibration model when σ_δ^2 is unknown.

Considering the models (2.1) and (2.2), the estimator $\hat{X}_0 = (\bar{Y}_0 - \hat{\alpha})/\hat{\beta}$ can be expressed as

$$\hat{X}_0 = \bar{X} + \frac{\beta(X_0 - \bar{X}) + \bar{\epsilon}_0 - \bar{\phi}}{\hat{\beta}} \tag{C-1}$$

where $\bar{\epsilon}_0 = \sum_{i=n+1}^{n+k} \epsilon_i/k$ and $\bar{\phi} = \sum_{i=1}^{n}(\epsilon_i - \beta\delta_i)/n$.

Considering k fixed, expanding $1/\hat{\beta}$ in a Taylor series around β and ignoring terms of order less than n^{-2}, we obtain the expected value of Equation (C-1), given by:

$$E(\hat{X}_0) = X_0 + \frac{\gamma(\bar{X} - X_0)}{n\beta^2 S_{XX}} \tag{C-2}$$

From this last equation we get the bias (2.8).

To derive the variance (2.9) we take the variance of Equation (C-1), which is given by

$$V(\hat{X}_0) = \beta^2(X_0 - \bar{X})^2 V\left(\frac{1}{\hat{\beta}}\right) + V\left(\frac{\bar{\epsilon}_0}{\hat{\beta}}\right) + V\left(\frac{\bar{\phi}}{\hat{\beta}}\right) \tag{C-3}$$

We call attention to the fact that Equation (C-3) is only expressed as a function of the related variances because the corresponding covariances are zero. The variances $V(1/\hat{\beta})$, $V(\bar{\epsilon}_0/\hat{\beta})$, and $V(\bar{\phi}/\hat{\beta})$ can be obtained by expanding $1/\hat{\beta}$, $\bar{\epsilon}_0/\hat{\beta}$, and $\bar{\phi}/\hat{\beta}$ in a Taylor series around β and ignoring terms of order less than n^{-2}. They are given by:

$$V(1/\hat{\beta}) = \frac{V(\hat{\beta})}{\beta^4} \tag{C-4}$$

$$V(\bar{\epsilon}_0/\hat{\beta}) = \frac{\sigma_\epsilon^2}{k\beta^2} + 3\frac{\sigma_\epsilon^2}{k\beta^4}V(\hat{\beta}) \tag{C-5}$$

$$V(\bar{\phi}/\hat{\beta}) = \frac{\gamma}{n\beta^2} \tag{C-6}$$

Substituing Equations (C-4), (C-5), and (C-6) in Equation (C-3), then, the variance (2.9) is obtained.

APPENDIX D

Similarly, as in Case 1, the Fisher information matrix of $\theta = (\alpha, \beta, X_0, \sigma_\epsilon^2)$ is given by

$$I(\theta) = \begin{pmatrix} \frac{n}{\gamma} + \frac{k}{\sigma_\epsilon^2} & \frac{n\bar{X}}{\gamma} + \frac{kX_0}{\sigma_\epsilon^2} & \frac{k\beta}{\sigma_\epsilon^2} & 0 \\ \frac{n\bar{X}}{\gamma} + \frac{kX_0}{\sigma_\epsilon^2} & \frac{\sum_{i=1}^n X_i^2}{\gamma} + \frac{2n\beta^2\sigma_\delta^4}{\gamma^2} + \frac{kX_0^2}{\sigma_\epsilon^2} & \frac{k\beta X_0}{\sigma_\epsilon^2} & \frac{n\beta\sigma_\delta^2}{\gamma^2} \\ \frac{k\beta}{\sigma_\epsilon^2} & \frac{k\beta X_0}{\sigma_\epsilon^2} & \frac{k\beta^2}{\sigma_\epsilon^2} & 0 \\ 0 & \frac{n\beta\sigma_\delta^2}{\gamma^2} & 0 & \frac{n}{2\gamma^2} + \frac{k}{2\sigma_\epsilon^4} \end{pmatrix} \tag{D-1}$$

The large sample variance of $\hat{X}_0$ follows by inverting the Fisher information matrix and is given by:

$$V(\hat{X}_0) = \frac{\sigma_\epsilon^2}{\beta^2}\left[\frac{1}{k} + \frac{\gamma}{n\sigma_\epsilon^2} + \frac{\gamma}{\sigma_\epsilon^2}E\right] \tag{D-1}$$

where,

$$E = \frac{nX_0^2\sigma_\epsilon^4 + kX_0^2\gamma^2 - 2nX_0\bar{X}\sigma_\epsilon^4 - 2kX_0\bar{X}\gamma^2 + n\bar{X}^2\sigma_\epsilon^4 + k\bar{X}^2\gamma^2}{(n\sigma_\epsilon^4 + k\gamma^2)\sum_{i=1}^{n} X_i^2 + 2nk\beta^2\gamma\sigma_\delta^4 - n^2\bar{X}^2\sigma_\epsilon^4 - nk\bar{X}^2\gamma^2}$$

APPENDIX E

Table E1. Empirical bias and mean squared error, theoretical variance, and the mean estimated variance of $\hat{X}_0$, for $\sigma_\delta^2 = 0.01$ and unknown

			Empirical		Theoretical Proposed-M		Mean of $\hat{V}(\hat{X}_0)$		
							Usual-M	Proposed-M	
X_0	n	k	Bias	MSE	$V_1(\hat{X}_0)$	$V_2(\hat{X}_0)$	$\hat{V}_1(\hat{X}_0)$	$\hat{V}_1(\hat{X}_0)$	$\hat{V}_2(\hat{X}_0)$
0.01	5	2	−0.0060	0.0180	0.0170	0.0170	0.0120	0.0100	0.0100
		20	−0.0087	0.0130	0.0120	0.0120	0.0072	0.0120	0.0120
		100	−0.0060	0.0130	0.0120	0.0120	0.0065	0.0120	0.0120
	20	2	−0.0038	0.0086	0.0087	0.0087	0.0120	0.0052	0.0053
		20	−0.0028	0.0043	0.0042	0.0042	0.0033	0.0040	0.0040
		100	−0.0032	0.0038	0.0038	0.0038	0.0022	0.0036	0.0036
	100	2	−0.0023	0.0058	0.0058	0.0058	0.0100	0.0027	0.0027
		20	−0.0002	0.0013	0.0013	0.0013	0.0016	0.0012	0.0012
		100	−0.0007	0.0008	0.0009	0.0009	0.0007	0.0009	0.0009
0.8	5	2	−0.0011	0.0094	0.0093	0.0094	0.0079	0.0045	0.0046
		20	−0.0034	0.0050	0.0048	0.0048	0.0029	0.0045	0.0046
		100	−0.0007	0.0047	0.0044	0.0044	0.0024	0.0045	0.0045
	20	2	0.0005	0.0063	0.0061	0.0061	0.0095	0.0028	0.0029
		20	0.0007	0.0016	0.0016	0.0016	0.0015	0.0015	0.0015
		100	−0.0001	0.0012	0.0012	0.0012	0.0008	0.0012	0.0012
	100	2	0.0005	0.0050	0.0052	0.0052	0.0099	0.0021	0.0021
		20	−0.0001	0.0007	0.0007	0.0007	0.0011	0.0007	0.0007
		100	−0.0003	0.0003	0.0003	0.0003	0.0003	0.0003	0.0003
1.9	5	2	0.0041	0.0160	0.0150	0.0160	0.0120	0.0093	0.0094
		20	0.0026	0.0110	0.0110	0.0110	0.0065	0.0110	0.0110
		100	0.0076	0.0110	0.0110	0.0110	0.0058	0.0110	0.0110
	20	2	0.0006	0.0079	0.0082	0.0082	0.0110	0.0049	0.0049
		20	0.0040	0.0039	0.0037	0.0037	0.0030	0.0035	0.0035
		100	0.0008	0.0033	0.0033	0.0033	0.0019	0.0031	0.0031
	100	2	0.0020	0.0057	0.0057	0.0057	0.0100	0.0025	0.0025
		20	0.0003	0.0012	0.0012	0.0012	0.0015	0.0011	0.0011
		100	0.0003	0.0008	0.0008	0.0008	0.0006	0.0008	0.0008

Table E2. Empirical bias and mean squared error, theoretical variance, and the mean estimated variance of $\hat{X}_0$, for $\sigma_\delta^2 = 0.1$ and unknown

			Empirical		Theoretical Proposed-M		Mean of $\hat{V}(\hat{X}_0)$		
							Usual-M	Proposed-M	
X_0	n	k	Bias	MSE	$V_1(\hat{X}_0)$	$V_2(\hat{X}_0)$	$\hat{V}_1(\hat{X}_0)$	$\hat{V}_1(\hat{X}_0)$	$\hat{V}_2(\hat{X}_0)$
0.01	5	2	−0.0510	0.1000	0.0700	0.0710	0.0770	0.0680	0.0690
		20	−0.0500	0.0950	0.0660	0.0660	0.0220	0.0660	0.0660
		100	−0.0510	0.0950	0.0650	0.0650	0.0130	0.0730	0.0730
	20	2	−0.0180	0.0280	0.0250	0.0250	0.0670	0.0240	0.0240
		20	−0.0160	0.0230	0.0210	0.0210	0.0140	0.0210	0.0210
		100	−0.0170	0.0230	0.0200	0.0200	0.0055	0.0210	0.0210
	100	2	−0.0046	0.0094	0.0093	0.0093	0.0580	0.0069	0.0069
		20	−0.0026	0.0048	0.0048	0.0048	0.0082	0.0048	0.0048
		100	−0.0033	0.0043	0.0044	0.0044	0.0029	0.0044	0.0044
0.8	5	2	−0.0084	0.0370	0.0290	0.0290	0.0460	0.0250	0.0250
		20	−0.0095	0.0270	0.0240	0.0240	0.0071	0.0200	0.0200
		100	−0.0072	0.0290	0.0240	0.0240	0.0037	0.0200	0.0200
	20	2	−0.0030	0.0120	0.0110	0.0110	0.0530	0.0085	0.0086
		20	−0.0040	0.0068	0.0066	0.0066	0.0061	0.0064	0.0064
		100	−0.0031	0.0063	0.0062	0.0062	0.0017	0.0060	0.0060
	100	2	−0.0011	0.0063	0.0062	0.0063	0.0550	0.0038	0.0038
		20	−0.0015	0.0017	0.0017	0.0017	0.0057	0.0017	0.0017
		100	−0.0011	0.0014	0.0013	0.0013	0.0013	0.0013	0.0013

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Table E2. (Continued)

			Empirical		Theoretical Proposed-M		Mean of $\hat{V}(\hat{X}_0)$		
							Usual-M	Proposed-M	
X_0	n	k	Bias	MSE	$V_1(\hat{X}_0)$	$V_2(\hat{X}_0)$	$\hat{V}_1(\hat{X}_0)$	$\hat{V}_1(\hat{X}_0)$	$\hat{V}_2(\hat{X}_0)$
1.9	5	2	0.0430	0.1090	0.0630	0.0630	0.0830	0.0750	0.0750
		20	0.0450	0.0860	0.0580	0.0580	0.0210	0.0650	0.0650
		100	0.0410	0.0860	0.0580	0.0580	0.0110	0.0600	0.0600
	20	2	0.0160	0.0260	0.0230	0.0230	0.0650	0.0210	0.0210
		20	0.0140	0.0190	0.0180	0.0180	0.0130	0.0180	0.0180
		100	0.0170	0.0200	0.0180	0.0180	0.0048	0.0180	0.0180
	100	2	0.0050	0.0088	0.0087	0.0088	0.0570	0.0063	0.0063
		20	0.0030	0.0043	0.0042	0.0042	0.0078	0.0042	0.0042
		100	0.0020	0.0039	0.0038	0.0038	0.0026	0.0038	0.0038

Table E3. Covering percentage (%) and amplitude (A) of the intervals with a 95% confidence level for the parameter X_0, when $\sigma_\delta^2 = 0.01$ and unknown

			Usual-M		Proposed-M			
X_0	n	k	%	A	$\%_1$	A_1	$\%_2$	A_2
0.01	5	2	83.04	0.40	79.34	0.36	79.37	0.36
		20	84.95	0.32	91.15	0.41	91.15	0.41
		100	83.72	0.31	92.24	0.42	92.24	0.42
	20	2	96.46	0.42	83.48	0.28	83.52	0.28
		20	90.19	0.22	92.47	0.24	92.47	0.24
		100	86.16	0.18	92.78	0.23	92.78	0.23
	100	2	98.84	0.40	73.16	0.19	73.16	0.19
		20	96.71	0.16	94.14	0.14	94.14	0.14
		100	91.90	0.11	94.68	0.12	94.68	0.12
0.8	5	2	85.43	0.32	74.33	0.24	74.39	0.24
		20	85.16	0.21	91.34	0.26	91.34	0.26
		100	85.04	0.19	92.50	0.25	92.50	0.25
	20	2	97.90	0.38	73.55	0.20	73.55	0.20
		20	93.55	0.15	93.89	0.15	93.89	0.15
		100	86.53	0.11	93.12	0.13	93.12	0.13
	100	2	99.41	0.39	65.05	0.16	65.05	0.16
		20	98.54	0.13	94.05	0.10	94.05	0.10
		100	94.56	0.07	94.86	0.07	94.86	0.07
1.9	5	2	82.47	0.39	78.05	0.35	78.13	0.35
		20	84.50	0.31	90.92	0.39	90.92	0.39
		100	84.75	0.29	92.83	0.39	92.83	0.39
	20	2	96.79	0.41	83.09	0.26	83.11	0.26
		20	91.32	0.21	93.29	0.23	93.31	0.23
		100	86.43	0.17	93.32	0.22	93.32	0.22
	100	2	98.88	0.40	73.06	0.19	73.06	0.19
		20	97.12	0.15	94.31	0.13	94.31	0.13
		100	92.56	0.10	94.74	0.11	94.74	0.11

Table E4. Covering percentage (%) and amplitude (A) of the intervals with a 95% confidence level for the parameter X_0, when $\sigma_\delta^2 = 0.1$ and unknown

			Usual-M		Proposed-M			
X_0	n	k	%	A	$\%_1$	A_1	$\%_2$	A_2
0.01	5	2	84.89	0.94	80.73	0.85	80.79	0.86
		20	64.10	0.51	82.75	0.86	82.75	0.86
		100	52.09	0.39	82.42	0.86	82.42	0.86
	20	2	99.40	0.99	91.04	0.58	91.10	0.58
		20	87.10	0.45	92.60	0.55	92.62	0.55
		100	65.70	0.28	92.16	0.55	92.18	0.55
	100	2	100.00	0.94	87.60	0.32	87.66	0.32
		20	98.82	0.36	94.88	0.27	94.88	0.27
		100	89.12	0.21	94.84	0.26	94.84	0.26

(Continued)

Table E4. (Continued)

			Usual-M		Proposed-M			
X_0	n	k	%	A	$\%_1$	A_1	$\%_2$	A_2
0.8	5	2	90.27	0.73	80.63	0.52	80.75	0.52
		20	64.39	0.31	82.68	0.51	82.74	0.51
		100	50.57	0.23	84.08	0.50	84.08	0.50
	20	2	99.92	0.88	87.30	0.35	87.50	0.35
		20	91.80	0.30	92.78	0.31	92.82	0.31
		100	67.46	0.16	92.38	0.30	92.38	0.30
	100	2	100.00	0.91	77.88	0.22	77.98	0.22
		20	99.94	0.29	94.98	0.16	95.04	0.16
		100	94.60	0.14	94.92	0.14	94.92	0.14
1.9	5	2	85.63	0.91	81.42	0.81	81.42	0.81
		20	61.71	0.49	81.89	0.82	81.89	0.82
		100	50.71	0.37	81.24	0.81	81.24	0.81
	20	2	99.54	0.97	91.12	0.54	91.18	0.55
		20	88.10	0.43	92.74	0.52	92.76	0.52
		100	66.44	0.26	92.86	0.51	92.86	0.51
	100	2	100.00	0.93	86.38	0.30	86.38	0.30
		20	99.12	0.35	94.76	0.25	94.76	0.25
		100	89.40	0.20	95.00	0.24	95.00	0.24

Table E5. Empirical bias and mean squared error, theoretical variance, and the mean estimated variance of $\hat{X}_0$, for $\sigma_\delta^2 = 0.01$ and known

			Empirical				Theoretical		
			Usual-M		Proposed-M		Proposed-M	Mean of $\hat{V}(X_0)$	
X_0	n	k	Bias	MSE	Bias	MSE	$V(\hat{X}_0)$	Usual-M	Proposed-M
0.01	5	2	−0.0290	0.0210	−0.0280	0.0210	0.0170	0.0180	0.0160
		20	−0.0290	0.0140	−0.0320	0.0140	0.0120	0.0081	0.0130
		100	−0.0240	0.0140	−0.0270	0.0140	0.0120	0.0070	0.0130
	20	2	−0.0081	0.0091	−0.0064	0.0090	0.0086	0.0130	0.0076
		20	−0.0060	0.0043	−0.0072	0.0043	0.0041	0.0034	0.0041
		100	−0.0038	0.0038	−0.0060	0.0038	0.0037	0.0022	0.0038
	100	2	−0.0011	0.0056	−0.0005	0.0056	0.0058	0.0100	0.0053
		20	−0.0002	0.0013	−0.0001	0.0013	0.0013	0.0016	0.0012
		100	−0.0009	0.0009	−0.0012	0.0009	0.0009	0.0007	0.0009
0.8	5	2	−0.0074	0.0110	−0.0072	0.0100	0.0093	0.0120	0.0085
		20	−0.0046	0.0051	−0.0051	0.0052	0.0048	0.0032	0.0049
		100	−0.0076	0.0048	−0.0082	0.0048	0.0044	0.0025	0.0046
	20	2	−0.0034	0.0063	−0.0031	0.0063	0.0061	0.0100	0.0052
		20	0.0001	0.0016	0.0000	0.0016	0.0016	0.0015	0.0016
		100	−0.0009	0.0012	−0.0013	0.0012	0.0012	0.0008	0.0012
	100	2	0.0000	0.0053	0.0002	0.0053	0.0052	0.0099	0.0048
		20	0.0000	0.0007	0.0000	0.0007	0.0007	0.0011	0.0007
		100	−0.0001	0.0003	−0.0002	0.0003	0.0003	0.0003	0.0003
1.9	5	2	0.0200	0.0180	0.0200	0.0180	0.0150	0.0170	0.0140
		20	0.0240	0.0120	0.0260	0.0130	0.0110	0.0071	0.0120
		100	0.0200	0.0130	0.0230	0.0130	0.0100	0.0062	0.0110
	20	2	0.0037	0.0082	0.0021	0.0081	0.0082	0.0120	0.0071
		20	0.0059	0.0037	0.0066	0.0037	0.0037	0.0030	0.0036
		100	0.0033	0.0032	0.0051	0.0032	0.0032	0.0020	0.0033
	100	2	0.0020	0.0058	0.0015	0.0057	0.0057	0.0100	0.0053
		20	0.0003	0.0012	0.0002	0.0012	0.0012	0.0015	0.0011
		100	0.0003	0.0008	0.0006	0.0008	0.0008	0.0006	0.0008

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Table E6. Empirical bias and mean squared error, theoretical variance, and the mean estimated variance of $\hat{X}_0$, for $\sigma_\delta^2 = 0.1$ and known

			Empirical				Theoretical		
			Usual-M		Proposed-M		Proposed-M	Mean of $\hat{V}(X_0)$	
X_0	n	k	Bias	MSE	Bias	MSE	$V(\hat{X}_0)$	Usual-M	Proposed-M
0.01	5	2	−0.4330	0.5590	−0.3830	0.4580	0.0590	0.3650	0.2310
		20	−0.1140	0.1310	0.4140	1.0880	0.0540	0.0250	0.0760
		100	−0.1890	0.1540	−0.0290	0.7730	0.0540	0.0200	0.1250
	20	2	−0.0930	0.0430	−0.0770	0.0360	0.0210	0.0950	0.0380
		20	−0.0490	0.0210	−0.0510	0.0210	0.0160	0.0150	0.0180
		100	−0.0430	0.0190	−0.0250	0.0640	0.0150	0.0058	0.0170
	100	2	−0.0200	0.0110	−0.0160	0.0097	0.0084	0.0630	0.0110
		20	−0.0077	0.0041	−0.0085	0.0039	0.0037	0.0084	0.0038
		100	−0.0066	0.0037	−0.0087	0.0037	0.0033	0.0029	0.0033
0.8	5	2	−0.0500	0.0620	−0.0430	0.0550	0.0280	0.1150	0.0620
		20	−0.0450	0.0380	0.0820	0.0810	0.0240	0.0086	0.0320
		100	−0.0530	0.0510	−0.0069	0.0980	0.0230	0.0055	0.0360
	20	2	−0.0280	0.0150	−0.0240	0.0140	0.0110	0.0740	0.0210
		20	−0.0079	0.0068	−0.0085	0.0069	0.0064	0.0065	0.0067
		100	−0.0055	0.0065	−0.0030	0.0079	0.0060	0.0018	0.0062
	100	2	−0.0030	0.0067	−0.0021	0.0065	0.0062	0.0600	0.0091
		20	−0.0018	0.0018	−0.0018	0.0017	0.0017	0.0058	0.0017
		100	−0.0021	0.0013	−0.0025	0.0013	0.0013	0.0013	0.0013
1.9	5	2	0.3480	0.3770	0.3070	0.3000	0.0530	0.2850	0.1760
		20	0.1400	0.1630	−0.3310	0.9370	0.0490	0.0300	0.0780
		100	0.0410	0.0180	0.0270	0.0510	0.0140	0.0051	0.0150
	20	2	0.0970	0.0430	0.0790	0.0350	0.0190	0.0930	0.0350
		20	0.1400	0.1630	−0.3310	0.9370	0.0490	0.0300	0.0780
		100	0.1670	0.1300	−0.0005	0.7090	0.0480	0.0160	0.1120
	100	2	0.0200	0.0093	0.0160	0.0087	0.0080	0.0620	0.0110
		20	0.0120	0.0039	0.0130	0.0037	0.0033	0.0080	0.0035
		100	0.0072	0.0031	0.0089	0.0031	0.0029	0.0027	0.0030

Table E7. Covering percentage (%) and amplitude (A) of the intervals with a 95% confidence level for the parameter X_0, when $\sigma_\delta^2 = 0.01$ and 0.1 and known

			$\sigma_\delta^2 = 0.01$				$\sigma_\delta^2 = 0.1$			
			Usual-M		Proposed-M		Usual-M		Proposed-M	
X_0	n	k	%	A	%	A	%	A	%	A
0.01	5	2	92.10	0.51	91.20	0.48	95.06	1.89	92.40	1.49
		20	87.32	0.34	95.18	0.44	63.38	0.59	64.47	1.09
		100	84.50	0.32	95.19	0.44	52.69	0.48	89.24	1.18
	20	2	97.46	0.43	90.12	0.33	99.87	1.17	96.27	0.71
		20	91.00	0.23	94.03	0.25	92.76	0.48	94.48	0.52
		100	86.73	0.19	95.21	0.24	70.38	0.30	90.35	0.51
	100	2	97.01	0.43	90.00	0.33	100.00	0.98	93.41	0.40
		20	92.34	0.23	95.26	0.25	99.77	0.36	95.09	0.24
		100	85.93	0.19	94.55	0.24	92.70	0.21	94.42	0.23
0.8	5	2	94.33	0.41	89.28	0.35	98.77	1.31	97.54	0.98
		20	86.84	0.22	95.03	0.27	61.85	0.34	81.25	0.69
		100	85.65	0.19	95.56	0.27	49.29	0.27	87.82	0.69
	20	2	98.35	0.39	88.44	0.27	100.00	1.03	94.09	0.53
		20	93.84	0.15	94.41	0.15	94.44	0.32	95.11	0.32
		100	85.98	0.11	94.32	0.14	70.38	0.17	92.41	0.31
	100	2	98.08	0.39	88.22	0.27	100.00	0.95	92.25	0.34
		20	92.75	0.15	93.96	0.15	99.89	0.30	94.89	0.16
		100	87.17	0.11	94.60	0.14	95.00	0.14	94.88	0.14

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Table E7. (*Continued*)

			$\sigma_\delta^2 = 0.01$				$\sigma_\delta^2 = 0.1$			
			Usual-M		Proposed-M		Usual-M		Proposed-M	
X_0	n	k	%	A	%	A	%	A	%	A
1.9	5	2	92.30	0.50	90.61	0.46	96.26	1.78	92.14	1.45
		20	86.96	0.33	95.13	0.42	63.58	0.56	67.24	1.03
		100	85.84	0.30	94.73	0.42	49.30	0.46	83.75	1.21
	20	2	97.61	0.43	89.92	0.32	100.00	1.17	96.27	0.70
		20	91.60	0.21	94.27	0.23	94.26	0.46	94.95	0.49
		100	86.16	0.17	94.99	0.23	72.44	0.28	94.18	0.48
	100	2	97.04	0.43	89.70	0.32	100.00	0.97	94.27	0.39
		20	91.21	0.21	93.85	0.23	99.59	0.35	95.18	0.23
		100	87.04	0.17	95.11	0.23	93.38	0.20	94.98	0.21

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