A CALIBRATION METHOD, BASED ON PIECEWISE RIDGE LS ESTIMATOR DESIGNED FOR DETERMINATION OF OLIVE OIL MIXTURES ON THE BASIS OF NIR SPECTRAL DATA

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Abstract − The spectrophotometric analysis of oil mixtures, containing olive oil, is the subject of this paper. Its objective is to propose and evaluate a new methodology for determination of a selected component of such a mixture. According to this methodology, the space of concentration values to be estimated is partitioned into subspaces and the calibration is performed separately for each subspace by means of a new *piecewise ridge least-squares estimator*. The estimation of concentrations is carried out, using the results of calibration, in two cycles: first, rough estimates of concentrations are found and a corresponding subspace is identified; next, the results of calibration, obtained for this subspace, are used for refining the estimates of concentrations. The performance of the proposed methodology is assessed using a criterion related to measurement uncertainty.

Keywords − least-squares estimators, NIR spectro-photometry, chemometrics, olive oil.

1. INTRODUCTION

The quality and purity of olive oil, extensively used in the food industry, is of significant commercial importance. According to the EU regulations, in force since 2002, a manufacturer of products, being based on or containing olive oil, must either indicate the share of olive oil in the total weight of the product or the percentage of olive oil as percentage of the total fat. That's why an increased interest in the methods for olive oil analysis has been observed for the last five years. Near-infrared (NIR) spectrophotometry, when combined with sophisticated procedures for spectrophotometric data processing, seems to be the most convenient and flexible tool for this application; consult, for example, [1–5] for more details. Numerous existing methods of estimation are potentially suitable for this application [6], in particular least-squares-type methods most frequently used in the domain of chemometrics. In [7], the authors have compared performance of six of them, when applied for estimation of the concentration of a selected component of an oil mixture, on the basis of the data representative of the NIR spectrum of this mixture, *viz.*: the ordinary least-squares estimator, the generalized least-squares estimator, the ridge least-squares estimator, the robust least-squares estimator, the total least-squares estimator and the partial least-squares estimator. Following the results of this comparison, the authors have decided to focus on the ridge least-squares estimator (RiLS), and to develop a methodology for its efficient application. This paper is devoted to such a methodology, being distinctive by three original elements:

− The space of concentration values to be estimated is partitioned into subspaces and the calibration is performed separately for each subspace by means of a ridge least-squares estimator.

− The regularisation parameter is selected in a way as to minimise the maximum error of estimation rather than to balance systematic and random errors of estimation.

− The estimation of concentrations is carried out, using the results of calibration, in two cycles: first, rough estimates of concentrations are found and a corresponding subspace is identified; next, the results of calibration, obtained for this subspace, are used for refining the estimates of concentrations.

The performance of the proposed methodology is assessed using a criterion related to the worst-case uncertainty of concentration measurement.

The following general rules are consistently used for generation of the mathematical symbols throughout this paper:

− x, y, … are real-valued scalar variables;

− $\dot{x}$, $\dot{y}$, … are exact values of x, y, …;

− $\hat{x}$, $\hat{y}$, … are estimated values of x, y, …;

− $\widetilde{x}$, $\widetilde{y}$, … are error-corrupted values of x, y, ….

The diacritical signs, whose meaning has been explained above in reference to scalar variables, are applied in an analogues way with respect to vectors ($\mathbf{x}$, $\mathbf{y}$, …) and matrices ($\mathbf{X}$, $\mathbf{Y}$, …) of real-valued variables.

2. PROBLEM FORMULATION

It is assumed that an oil mixture to be analyzed is composed of J known components, and that the vectors of exact data $\dot{\mathbf{s}}_j\big|_{M\times 1}$ ($j = 1,...,J < M$), representative of the absorbance spectra of all those components, are available. According to the Lambert-Beer's law, the vector of exact

absorbance data $\dot{\mathbf{s}}$, representative of the spectrum of the mixture, satisfy the equation:

$$\dot{\mathbf{s}} = \sum_{j=1}^{J} c_j \cdot \dot{\mathbf{s}}_j \tag{1}$$

where $\mathbf{c} = [c_1 ... c_J]^T$ is the vector of concentrations of components, subject to the following constraints:

$$\sum_{j=1}^{J} c_j = 1 \text{ and } c_j \in [0,1] \text{ for } j = 1, ..., J \tag{2}$$

It is assumed that the real-world absorbance data $\widetilde{\mathbf{s}}$, representative of the spectrum of a mixture, are corrupted by errors $\Delta\widetilde{\mathbf{s}}$ resulting both from inaccurate preparation of the mixture and imperfections of the spectrophotometer.

The research problem, studied in this paper, consists in estimation of the concentration of only one component of the mixture, *viz.* c_1, by means of a linear estimator of the form:

$$\hat{c}_1 = \mathbf{p}_1^T \cdot \widetilde{\mathbf{s}} \tag{3}$$

where $\mathbf{p}_1 = [p_{1,1} ... p_{1,M}]^T$ is a vector of parameters to be determined on the basis of a set of calibration data:

$$\widetilde{\mathbb{D}}^{cal} = \left\{ \widetilde{\mathbf{s}}_n^{cal}, \left\langle \dot{c}_{1,n}^{cal}, \dot{c}_{2,n}^{cal} \right\rangle \middle| n = 1, ..., N \right\} \tag{4}$$

which – for the sake of convenience of numerical manipulations – are organized in a matrix:

$$\widetilde{\mathbf{S}}^{cal} \equiv \left[\widetilde{\mathbf{s}}_1^{cal} \quad ... \quad \widetilde{\mathbf{s}}_N^{cal} \right]^T \tag{5}$$

and two vectors:

$$\dot{\mathbf{c}}_1^{cal} \equiv \left[\dot{c}_{1,1}^{cal} \quad ... \quad \dot{c}_{1,N}^{cal} \right]^T \text{ and } \dot{\mathbf{c}}_2^{cal} \equiv \left[\dot{c}_{2,1}^{cal} \quad ... \quad \dot{c}_{2,N}^{cal} \right]^T \tag{6}$$

The parameters to be determined are assumed to satisfy the following approximate equality:

$$\widetilde{\mathbf{S}}^{cal} \cdot \mathbf{p}_1 \cong \dot{\mathbf{c}}_1^{cal} \tag{7}$$

which is a basis for development of all the methods that may be used for their estimation. The performance of the proposed methodology is assessed on the basis of an indicator of worst-case uncertainty of the final result of measurement, using a set of validation data:

$$\widetilde{\mathbb{D}}^{val} = \left\{ \widetilde{\mathbf{s}}_n^{val}, \left\langle \dot{c}_{1,n}^{val}, \dot{c}_{2,n}^{val} \right\rangle \middle| n = 1, ..., N' \right\} \tag{8}$$

which – for the sake of convenience of numerical manipulations – are organized in a matrix:

$$\widetilde{\mathbf{S}}^{val} \equiv \left[\widetilde{\mathbf{s}}_1^{val} \quad ... \quad \widetilde{\mathbf{s}}_{N'}^{val} \right]^T \tag{9}$$

and two vectors:

$$\dot{\mathbf{c}}_1^{val} \equiv \left[\dot{c}_{1,1}^{val} \quad ... \quad \dot{c}_{1,N'}^{val} \right]^T \text{ and } \dot{\mathbf{c}}_2^{val} \equiv \left[\dot{c}_{2,1}^{val} \quad ... \quad \dot{c}_{2,N'}^{val} \right]^T \tag{10}$$

The study presented in this paper is based on semi-synthetic data representative of NIR spectra of trinary mixtures of edible oils.

3. SYNTHESIS OF DATA

The study has been based on the semi-synthetic data, generated using the real-world data representative of nut oil, corn oil and olive oil ($J = 3$). The sequences of the latter data, each containing $N = 501$ points, are shown in Fig. 1.

The data for calibration and validation have been synthesised in a way imitating the procedure used for obtaining the real-world data. First, the reference (exact) values of the concentrations of nut oil ($\dot{c}_1$) and of corn oil ($\dot{c}_2$) have been selected, and the value of the concentration of olive oil has been calculated:

$$\dot{c}_3 = 1 - \dot{c}_1 - \dot{c}_2 \tag{11}$$

Next, the error-corrupted values of all concentrations have been determined by emulation of the process of sample preparation which consists in mixing precisely measured out volumes of the components. The error-free values of concentration are related to the error-free values of those volumes in the following way:

$$\dot{c}_k = \frac{\dot{V}_k}{\dot{V}} \text{ for } k = 1, 2, 3 \tag{12}$$

where $\dot{V} = \dot{V}_1 + \dot{V}_2 + \dot{V}_3$. The error-corrupted values of all concentrations are related to the error-corrupted values of those volumes in the following way:

$$\widetilde{c}_k = \frac{\widetilde{V}_k}{\widetilde{V}} \text{ for } k = 1, 2, 3 \tag{13}$$

where:

$$\widetilde{V} = \widetilde{V}_1 + \widetilde{V}_2 + \widetilde{V}_3 \text{ and } \widetilde{V}_k = \dot{V}_k(1 + \vartheta_k) \text{ for } k = 1, 2, 3 \tag{14}$$

with ϑ_k being relative errors of measuring out the volumes. The resulting concentrations $\widetilde{c}_k$ are subject to relative errors ζ_k:

$$\widetilde{c}_k = \dot{c}_k(1 + \zeta_k) \text{ for } k = 1, 2, 3 \tag{15}$$

which are related to the errors in the volume errors ϑ_k in the following way:

$$\begin{aligned} \widetilde{c}_k = \dot{c}_k(1 + \zeta_k) &= \frac{\widetilde{V}_k}{\widetilde{V}_1 + \widetilde{V}_2 + \widetilde{V}_3} \\ &= \frac{\dfrac{\dot{V}_k(1 + \vartheta_k)}{\dot{V}}}{\dfrac{\dot{V}_1(1 + \vartheta_1) + \dot{V}_2(1 + \vartheta_2) + \dot{V}_3(1 + \vartheta_3)}{\dot{V}}} \\ &= \frac{\dot{c}_k(1 + \vartheta_k)}{\dot{c}_1(1 + \vartheta_1) + \dot{c}_2(1 + \vartheta_2) + \dot{c}_3(1 + \vartheta_3)} \end{aligned} \tag{16}$$
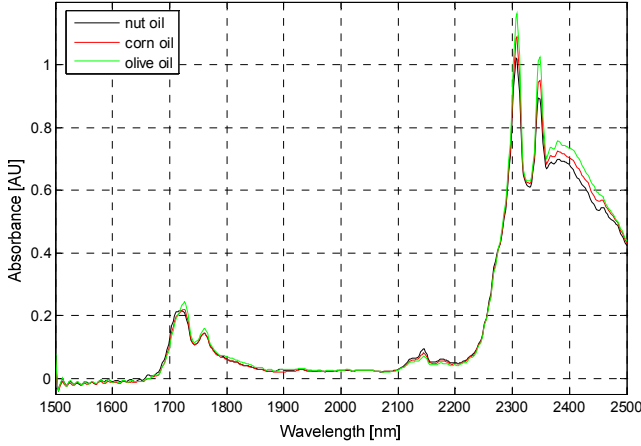
Fig. 1. The denoised and baseline-corrected spectrophotometric data used for experimentation.

The spectral data $\widetilde{\mathbf{s}} \equiv \left[\widetilde{s}_1\ \widetilde{s}_2\ ...\right]^T$, corresponding to a given triplet of concentrations – $\widetilde{c}_1$, $\widetilde{c}_2$ and $\widetilde{c}_3$ determined after Eq.(16) – have been generated after the formulae:

$$\overline{\mathbf{s}} = \widetilde{c}_1 \dot{\mathbf{s}}_1 + \widetilde{c}_2 \dot{\mathbf{s}}_2 + \widetilde{c}_3 \dot{\mathbf{s}}_3 \tag{17}$$

$$\widetilde{s}_n = \overline{s}_n \left(1 + \xi_n\right) \text{ for } n = 1, 2, ... \tag{18}$$

where $\dot{\mathbf{s}}_1$, $\dot{\mathbf{s}}_2$ and $\dot{\mathbf{s}}_3$ are the vectors of denoised and baseline-corrected real-world data representative of nut oil, corn oil and olive oil – respectively; ξ_n are realisations of random variables modelling the relative errors of spectrum measurement.

For generation of the relative errors of the volumes (ϑ_k), uncorrelated pseudorandom numbers following the zero-mean normal distribution with the standard deviation $\sigma_\vartheta = 0.002$, truncated outside of the interval $[-3\sigma_\vartheta, +3\sigma_\vartheta]$, have been used; for generation of the relative errors of spectral data (ξ_n) – uncorrelated pseudorandom numbers following the zero-mean normal distribution with the standard deviation $\sigma_\xi = 10^{-4}$, truncated outside of the interval $[-3\sigma_\xi, +3\sigma_\xi]$.

The set of data for calibration $\widetilde{\mathbb{D}}^{cal}$ has been assumed to contain all the pairs ($N = 121$) of the following values of concentrations:

$$\dot{c}_1^{cal}, \dot{c}_2^{cal} \in \left\{ k \cdot 0.01 \mid k = 0, 1, ..., 10 \right\} \tag{19}$$

and the corresponding triplets of spectral data.

The set of data for validation $\widetilde{\mathbb{D}}^{val}$ has been assumed to contain all the pairs ($N' = 100$) of the following values of concentrations:

$$\dot{c}_1^{val}, \dot{c}_2^{val} \in \left\{ 0.005 + k \cdot 0.01 \mid k = 0, 1, ..., 9 \right\} \tag{20}$$

and the corresponding triplets of spectral data. Thus, the validation has been carried out over an area of the c_1-c_2 plane slightly smaller that the area covered by the

calibration data. In this way, the impact of border effects has been mitigated.

Taking into account that the fourth singular value of the 121×501 matrix $\widetilde{\mathbf{S}}^{cal}$, composed of all spectral data available for calibration, is:
– $ca.\ 5 \cdot 10^4$ times smaller than the first singular value,
– $ca.\ 15$ times smaller than the third singular value,
three wavelength values have been assumed sufficient for this study, viz. $\lambda_{n1} = 1762\ \text{nm}$, $\lambda_{n2} = 2146\ \text{nm}$ and $\lambda_{n3} = 2330\ \text{nm}$. They have been selected via an exhaustive search of a triplet which minimises the condition number of the denoised matrix $\widetilde{\mathbf{S}}^{cal}$.

4. METHODOLOGY OF STUDY

Both sets of data, $\widetilde{\mathbb{D}}^{cal}$ and $\widetilde{\mathbb{D}}^{val}$, have been generated in $R = 100$ versions, corresponding to R realisations of the errors. For each version $\widetilde{\mathbb{D}}^{cal}(r)$, $r = 1, ..., R$, of the set $\widetilde{\mathbb{D}}^{cal}$, the full calibration has been performed, and its results have been validated by means of R versions $\widetilde{\mathbb{D}}^{val}(r')$ of $\widetilde{\mathbb{D}}^{val}$, $r' = 1, ..., R$. The worst-case error of estimation, calculate for each concentration value used for validation:

$$\Delta \hat{c}_1 = \sup \left\{ \left| \hat{c}_1^{val}(r, r') - \dot{c}_1^{val} \right| \; \middle| \; \begin{matrix} r = 1, ..., R \\ r' = 1, ..., R' \end{matrix} \right\} \tag{21}$$

has been used as a performance indicator of the estimation methods under study. Three calibration methods have been compared using this indicator, viz. the ordinary least-squares method (OLS), the ridge least-squares method (RiLS), and a new method, called piecewise least-squares method (pRiLS), proposed in the next section. The implementation of OLS has been based on the formula:

$$\hat{\mathbf{p}}_1^{OLS} = \left[\widetilde{\mathbf{S}}^{cal} \right]^+ \dot{\mathbf{c}}_1^{cal} \tag{22}$$

and an unstructured set of data $\widetilde{\mathbb{D}}^{cal}$ (where the symbol $^+$ is for the operator of matrix Moore-Penrose pseudo-inversion). The implementation of RiLS has been based on the formula:

$$\hat{\mathbf{p}}_1^{RiLS}(\alpha) = \left[\left(\widetilde{\mathbf{S}}^{cal} \right)^T \cdot \widetilde{\mathbf{S}}^{cal} + \alpha \cdot \mathbf{I} \right]^+ \left(\widetilde{\mathbf{S}}^{cal} \right)^T \cdot \dot{\mathbf{c}}_1^{cal} \tag{23}$$

and the set of data $\widetilde{\mathbb{D}}^{cal}$ split into two subsets: $\widetilde{\mathbb{D}}^{cal,1}$ and $\widetilde{\mathbb{D}}^{cal,2}$. The first of them has been assumed to contain all the pairs of the following values of concentrations:

$$\dot{c}_1^{cal}, \dot{c}_2^{cal} \in \left\{ 2k \cdot 0.01 \mid k = 0, 1, ..., 5 \right\} \tag{24}$$

and the corresponding triplets of spectral data included in the matrix $\widetilde{\mathbf{S}}^{cal}$. The subset $\widetilde{\mathbb{D}}^{cal,2}$, used for optimisation of the regularisation parameter α, has been assumed to contain all the pairs of the following values of concentrations:

$$\dot{c}_1^{cal}, \dot{c}_2^{cal} \in \left\{ (2k-1) \cdot 0.01 \mid k = 0, 1, ..., 5 \right\} \tag{25}$$

The comparison of OLS and RiLS performance is shown in Fig. 2. It follows from this figure that, despite expectation, the RiLS performance is worse than that of OLS. There are two reasons of this surprising behaviour of RiLS:

- The right-hand-side vector of Eq.(7) is error-free, and both the errors ϑ_k and ξ_n influence the left-hand-side matrix of that equation.
- The values of the regularisation parameter α, optimal for the calibration data, used for its optimisation, are not always optimal for the corresponding validation data.
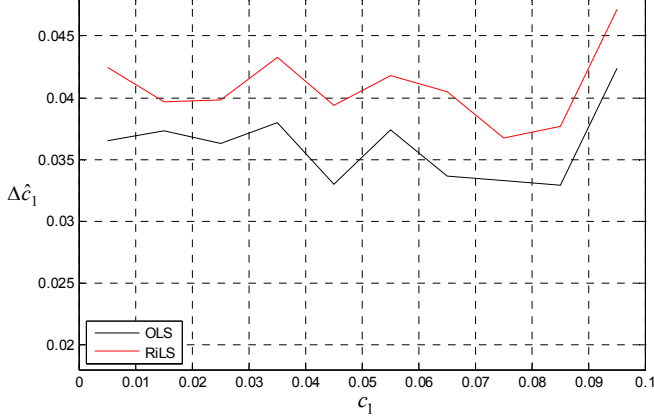

Fig. 2. The dependence of the performance indicator, defined by Eq.(21), on the value of the measurand, for OLS and RiLS.

5. PROPOSED SOLUTION AND ITS PERFORMANCE

The main idea of the proposed method of calibration is to improve the performance of RiLS by making it dependent on the concentration c_2. The implementation of this idea requires, consequently, the solution of an auxiliary problem of rough estimation of c_2. It has turned out that OLS is not sufficient for this purpose, and that a more precise method, called piecewise OLS, has to be applied. The latter consists in determination of the vectors of parameters $\hat{\mathbf{p}}_{2,i}^{OLS}$ optimised for the consecutive values of $\dot{c}_2^{cal} = i \cdot 0.01$ ($i = 0, 1, ..., 10$). In this case, the set of calibration data $\widetilde{\mathbb{D}}^{cal}$ is split into 11 subsets $\widetilde{\mathbb{D}}_i^{cal}$, each containing all the pairs of:

$$\dot{c}_1^{cal} \in \left\{ k \cdot 0.01 \mid k = 0, 1, ..., 10 \right\} \text{ and } \dot{c}_2^{cal} = i \cdot 0.01 \quad (26)$$

and the corresponding triplets of spectral data. The resulting vectors $\hat{\mathbf{p}}_{2,i}^{OLS}$ are approximated by means of a linear function:

$$\hat{\mathbf{p}}_2^{pOLS}(c_2) = \hat{\mathbf{a}}_2^{pOLS} \cdot c_2 + \hat{\mathbf{b}}_2^{pOLS} \quad (27)$$

This function is used by pRiLS together with the OLS result:

$$\hat{\mathbf{p}}_2^{OLS} = \left[\widetilde{\mathbf{S}}^{cal} \right]^+ \dot{\mathbf{c}}_2^{cal} \quad (28)$$

obtained on the basis of $\widetilde{\mathbb{D}}^{cal}$, and with another linear function:

$$\hat{\mathbf{p}}_1^{pRiLS}(c_2) = \hat{\mathbf{a}}_1^{pRiLS} \cdot c_2 + \hat{\mathbf{b}}_1^{pRiLS} \quad (29)$$

approximating the vectors of parameters $\hat{\mathbf{p}}_{1,i}^{pRiLS}$. The latter are obtained by means of RiLS for the subsets $\widetilde{\mathbb{D}}^{cal,1}$ of the calibration data $\widetilde{\mathbb{D}}^{cal}$, containing all the pairs of:

$$\dot{c}_1^{cal} \in \left\{ 2k \cdot 0.01 \mid k = 0, 1, ..., 5 \right\} \text{ and } \dot{c}_2^{cal} = i \cdot 0.01 \quad (30)$$

and the subsets $\widetilde{\mathbb{D}}^{cal,2}$ of the calibration data $\widetilde{\mathbb{D}}^{cal}$, containing all the pairs of:

$$\dot{c}_1^{cal} \in \left\{ (2k-1) \cdot 0.01 \mid k = 0, 1, ..., 5 \right\} \text{ and } \dot{c}_2^{cal} = i \cdot 0.01 \quad (31)$$

with the corresponding triplets of spectral data (in both cases).

The final result of calibration – *i.e.* the vectors $\hat{\mathbf{p}}_2^{OLS}$, $\hat{\mathbf{a}}_2^{pOLS}$, $\hat{\mathbf{b}}_2^{pOLS}$ $\hat{\mathbf{a}}_1^{pRiLS}$ and $\hat{\mathbf{b}}_1^{pRiLS}$ – are used in the following way for estimation of $\dot{c}_1$ on the basis of a given triplet of spectral data $\widetilde{\mathbf{s}}$:

$$\hat{c}_2^{OLS} = \widetilde{\mathbf{s}}^T \cdot \hat{\mathbf{p}}_2^{OLS} \quad (32)$$

$$\hat{c}_2^{pOLS} = \widetilde{\mathbf{s}}^T \cdot \left(\hat{\mathbf{a}}_2^{pOLS} \cdot \hat{c}_2^{OLS} + \hat{\mathbf{b}}_2^{pOLS} \right) \quad (33)$$

$$\hat{c}_1^{pRiLS} = \widetilde{\mathbf{s}}^T \cdot \left(\hat{\mathbf{a}}_1^{pRiLS} \cdot \hat{c}_2^{pOLS} + \hat{\mathbf{b}}_1^{pRiLS} \right) \quad (34)$$

or:

$$\hat{c}_1^{pRiLS} = \widetilde{\mathbf{s}}^T \cdot \left(\hat{\mathbf{a}}_1^{pRiLS} \cdot \widetilde{\mathbf{s}}^T \cdot \left(\hat{\mathbf{a}}_2^{pOLS} \cdot \widetilde{\mathbf{s}}^T \cdot \hat{\mathbf{p}}_2^{OLS} + \hat{\mathbf{b}}_2^{pOLS} \right) + \hat{\mathbf{b}}_1^{pRiLS} \right) \quad (35)$$

The performance of the above described method is compared with that of OLS in Fig. 3.
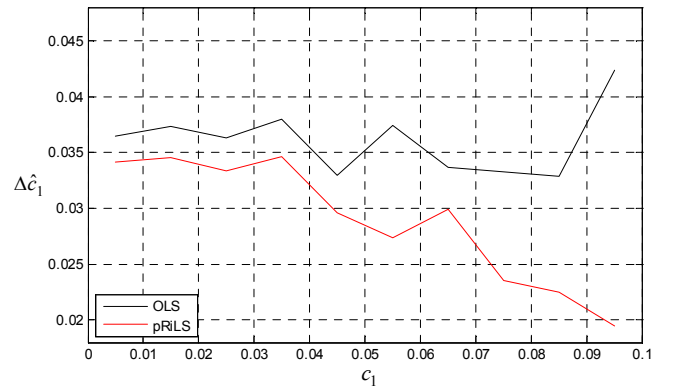

Fig. 3. The dependence of the performance indicator, defined by Eq.(21), on the value of the measurand, for OLS and pRiLS.

6. CONCLUSIONS

The main contribution of this paper to the development of methodology for solving difficult numerical problems, related to the determination of the mixtures of edible oils on the basis of NIR spectrophotometric data, consists of the following elements:

- a more realistic method for generation of synthetic data, better reflecting the procedures applied in analytical laboratories;
- a worst-case uncertainty procedure for evaluation and comparison estimators used for determination of the mixtures of edible oils;
- a new method of calibration, called piecewise ridge least-squares.

Although the reduction of worst-case uncertainty of estimation, attained by means of the latter method is not very spectacular, it should be viewed in the context of an extreme difficulty of the task under study, the difficulty implied by a very close similarity of the NIR spectra of nut oil, corn oil and olive oil. Moreover, further optimisation of independent parameters of the estimator, defined by Eq.(35), seems to be possible.

ACKNOWLEDGEMENTS

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