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L. Salguero Chaparro, B. Palagos, F. Peña Rodriguez, J.M. Roger

### ► To cite this version:

L. Salguero Chaparro, B. Palagos, F. Peña Rodriguez, J.M. Roger. Calibration transfer of intact olive NIR spectra between a pre-dispersive instrument and a portable spectrometer. *Computers and Electronics in Agriculture*, 2013, 96, p. 202 - p. 208. 10.1016/j.compag.2013.05.007 . hal-00864930

**HAL Id: hal-00864930**

**<https://hal.science/hal-00864930>**

Submitted on 23 Sep 2013

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1           **Calibration transfer of intact olive NIRS spectra between a pre-**  
2                           **dispersive instrument and a portable spectrometer**

3  
4           **Salguero-Chaparro, L. <sup>\*,a</sup>, Palagos, B. <sup>b</sup>, Peña-Rodríguez, F. <sup>a</sup> & Roger, J.M. <sup>b</sup>**

5           <sup>a</sup>IFAPA “Alameda del Obispo”, Avd. Menéndez Pidal s/n, 14004, Córdoba, Spain

6           <sup>b</sup>Irstea, UMR ITAP, Information-technologies-analyse environnementale-procédés agricoles, 361 rue J.F.  
7           Breton, BP 5095 F-34033 Montpellier Cedex 1, France

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13           \*Corresponding author: [salg\\_lou@hotmail.com](mailto:salg_lou@hotmail.com)

14   (+ 34) 957 01 61 41; Fax (+34) 957 01 60 43

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27 **Abstract**

28           The recent development of new portable devices enables the use of near infrared  
29 spectroscopy (NIRS) technologies in on-line industrial applications. However, the  
30 numerous existing NIRS databases have been constructed with off-line laboratory  
31 instruments which required a considerable effort in terms of time, labour and costs. For  
32 this reason, the transfer of calibrations between devices of different characteristics is a  
33 clearly crucial step. The three different standardization algorithms of Slope/Bias  
34 Correction (SBC), Piecewise Direct Standardization (PDS) and Transfer by Orthogonal  
35 Projection (TOP) were tested and evaluated for transferring olives quality databases  
36 from an off-line NIRS monochromator (FOSS NIRSystem 6500) to a portable NIRS  
37 diode-array spectrometer (CORONA 45 visNIR). The results obtained showed that the  
38 use of TOP yielded the best Standard Error of Prediction (SEP) values for the fat  
39 content (1.97 %) and free acidity (2.52 %) parameters, while PDS for moisture content  
40 (2.24%). These results suggest that good calibration models for quality evaluation in  
41 intact olives can be obtained, based on spectral databases transferred between diverse  
42 NIRS spectrometers.

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45 **Keywords**

46 Calibration transfer; slope/bias correction; PDS; TOP; Intact olives

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52        **1. Introduction**

53            Near Infrared Spectroscopy (NIRS) has been successfully used by the olive oil  
54 sector for the quantitative analysis of the major olive constituents such as fat content  
55 and moisture in the last years (Armenta *et al.* 2010). For this reason, the implementation  
56 of on-line control systems in these industries is desirable in order to determine quickly  
57 and accurately the quality parameters of the olives at an initial stage of the production  
58 process that could allow control the raw material (intact olives) and consequently the  
59 final product (olive oil). However, the major part of applications have been developed  
60 under controlled laboratory environments -i.e. off-line, a great distance away from the  
61 real process in the olive mills.

62            Nowadays, by establishment of new portable devices (such as diode array  
63 instruments) more robust, faster and better adapted to worse analysis conditions, the  
64 direct application of this technology in the olive industry is possible. However, until  
65 now, only a few studies have already been conducted for the on-line determination of  
66 chemical composition in olive oil, olive pomace and olive paste (Hermoso *et al.*, 1999;  
67 Jiménez-Márquez *et al.*, 2005; Gallardo-González *et al.*, 2005).

68            A key point concerning the implementation of NIR spectroscopy for olive  
69 process control is to demonstrate that the large data sets obtained from off-line analysis  
70 already existing, which have been developed during many years, may be used for on-  
71 line industrial purposes. The construction of calibration models requires considerable  
72 time, cost and effort for the collection and the measurement of the samples (Bouveresse  
73 *et al.*, 1998). For that reason, an easy and rapid calibration transfer (or standardisation)  
74 between instruments is necessary, in order to avoid re-measuring of the whole  
75 calibration procedure.

76 In the standardization process, a calibration model developed on a *master*  
77 instrument is modified in order to make it compatible with other multiple instruments  
78 (*slave*) by means of a set of mathematical and statistical procedures. However, direct  
79 transfer of calibration models obtained with one instrument on the same instrument after  
80 a period of time, or on another different equipment will usually result in erroneous  
81 predictions unless some adjustment is previously made (Andrew and Fearn, 2004). The  
82 problem of calibration transfer is a significant limitation of this technique that has been  
83 extensively reviewed (Wang *et al.*, 1991; De Noord, 1994; Bouveresse *et al.*, 1994;  
84 Bouveresse and Massart, 1996a; Bouveresse and Massart, 1996b; Park *et al.*, 2001;  
85 Fearn, 2001; Feudale *et al.*, 2002).

86 In the literature, several standardisation procedures have been developed to deal  
87 with this important problem and allow the transfer of calibration models (De Noord,  
88 1994; Bouveresse and Massart, 1996a; Fearn, 2001; Feudale *et al.*, 2002). There are  
89 some approaches than can be used to solve the transfer problems without the need for  
90 standardization (derivatives, multiplicative scatter correction (MSC), orthogonal signal  
91 correction (OSC), etc.) (Feudale *et al.*, 2002). However, when the problem is not due to  
92 intensity changes in the spectra if not that is related to wavelength shifts, different  
93 standardization procedures can be applied. According to the strategy described in  
94 Chauchard *et al.* (2004), a calibration transfer can be carried out following several  
95 modes: *a priori correction* consists in correcting the spectra before applying the existing  
96 calibration model; *model correction* consists in adapting the calibration model; *a*  
97 *posteriori correction* consists in correcting the predictions of the existing calibration  
98 model; *robust modelling* consists in building a model insensitive to the perturbation.

99 In the framework of transfer between instruments, *a priori* correction and model  
100 correction are based on multivariate correction of the spectra. In the former mode, slave

101 spectra are corrected to match the master ones and inputted in the existing model. In the  
102 latter mode, the spectra of the master calibration database are corrected to match the  
103 slave ones and the model is recalibrated. Spectra multivariate correction may use a large  
104 number of techniques, such as direct standardisation (DS) (Wang et al., 1991; Greensill  
105 and Walsh, 2002; Zamora-Rojas *et al.*, 2012), piecewise direct standardisation (PDS)  
106 (Bouveresse et al., 1996b; Park *et al.*, 2001; Bergman et al., 2006; Fernández-Ahumada  
107 *et al.*, 2008; Igne *et al.*, 2009; Fernández-Pierna *et al.*, 2010), wavelet transform (WT)  
108 (Park *et al.*, 2001; Greensill and Walsh, 2002), finite impulse response (FIR) filtering  
109 without standards (Black *et al.*, 1996), boxcar signal transfer (BST) (Oliveri *et al.*,  
110 2013) or the patented method proposed by Shenk and Westerhaus (1991).

111 In a posteriori correction, the existing master calibration is applied to a set of  
112 slave spectra for which the responses are known. A model of the prediction error is then  
113 calibrated and its inverse is applied to the future predictions. This model is generally  
114 performed by a simple univariate method such as bias/slope correction (BSC) of the  
115 predicted values (Osborne and Fearn, 1983; Jones *et al.*, 1993; Bouveresse *et al.*, 1998;  
116 Fearn, 2001 review; Greensill and Walsh, 2002; Bergman *et al.*, 2006).

117 Robust modelling consists in building the calibration in a subspace which is not  
118 affected by the influences which cause the problem of robustness. An efficient method  
119 for finding this subspace is to identify the influenced subspace and to remove it by  
120 means of an orthogonal projection. Depending on the way the detrimental subspace is  
121 identified, several methods have been proposed, as independent interference reduction  
122 (IIR) (Hansen, 2001), external parameter orthogonalization (EPO) (Roger *et al.*, 2003),  
123 transfer by orthogonal projection (TOP) (Andrew and Fearn, 2004), dynamic orthogonal  
124 projection (DOP) (Zeaiter *et al.*, 2006) and error removal by orthogonal subtraction  
125 (EROS) (Zhu *et al.*, 2008).

126           The aim of this study was to transfer calibration models for predicting fat  
127 content, free acidity and moisture in intact olives from a NIRS monochromator (FOSS  
128 NIRSystem 6500) to a portable diode-array (CORONA 45 visNIR) instrument. In this  
129 paper, three different representative methods for calibration transfer were tested and  
130 evaluated: PDS, the most popular and widely used calibration transfer technique, for a  
131 priori and model correction; SBC, the simplest and most classical, for a posteriori  
132 correction and TOP, one of the most recently developed transfer method, for robust  
133 modeling.

134

135

## 136       **2. Materials and Methods**

137

### 138       *2.1. Samples, spectral acquisition and reference measurements*

139           A set of 174 batches of intact olive (*Olea Europaea* L.) samples, each between  
140 15 and 20 kg weight, were harvested over one crop season (October-March, 2010-2011)  
141 from different plots in Andalusia (Spain).

142           Once in the lab, the olive samples were kept under controlled refrigeration at 5°C  
143 and 90% relative humidity. Before spectroscopic measurements were acquired, the  
144 samples were equilibrated at room temperature (25°C).

145           Spectra were collected from samples in reflectance mode ( $\text{Log } 1/R$ ) using two  
146 NIRS-instruments: (1) FNS-6500 SY-II scanning monochromator (FOSS NIRSystems,  
147 Silver Spring, MD, USA); and (2) CORONA 45 visNIR diode-array spectrometer (Carl  
148 Zeiss, Jena, Germany).

149           A pre-dispersive FNS-6500 scanning monochromator provided with a transport  
150 module was used to measure all spectra in the scanning range of 400 to 2498 nm, at 2

151 nm interval (figure 1a). The instrument was equipped with a silicon detector in the  
152 wavelength range of 400-1100 nm and with a lead sulphide detector for the range 1100-  
153 2498 nm. A rectangular natural product cell with a window surface of 94.9 cm<sup>2</sup> was  
154 used in order to carried out the analysis of the samples. Each spectrum was the average  
155 of 32 scans. Three different charges of each sample were scanned as replicates and the  
156 average spectrum was used for calculations. Spectra were recorded using ISIScan v.  
157 1.26 software (Infrasoft International LLC, State College, PA, USA).

158         The second instrument was a post-dispersive single-beam diode array (DA)  
159 spectrometer, CORONA 45 visNIR, working in the range from 380 to 1690 nm, with a  
160 spectral wavelength interval of 2 nm (figure 1b). The DA device was equipped with a  
161 silicon diode array (Hamamatsu S 3904) for the range 380-950 nm and an InGaAs array  
162 for the range 950-1690. The NIRS instrument was mounted on a bracket over a  
163 conveyor belt set and spectra were obtained during the movement of the samples  
164 underneath the spectrometer. The optimisation of acquisition parameters were described  
165 in detail by Salguero-Chaparro *et al.* (2012). The distance from the sample surface to  
166 the sensing head was approximately 13 mm and the conveyor belt speed was fixed to  
167 0.1 ms<sup>-1</sup>. White and black spectral references were collected manually. With an  
168 integration time of 5 s, 10 scans were averaged for each measurement. A total of thirty  
169 spectra of the same sample were acquired and the mean spectrum was used for data  
170 processing. All spectra were recorded using CORA software version 3.2.2. (Carl Zeiss,  
171 Inc.).

172         Reference values for fat and moisture content in olive samples were determined  
173 using official analysis methods. Fat content was obtained on olive oil samples extracted  
174 by Soxhlet, according to the UNE 55030 procedure (AENOR, 1961), free acidity was  
175 determined by acid-base titration according to Regulation EEC/2568/91 of the European



176 Union Commission (EC, 2003) and the moisture content was measured by oven drying  
177 to constant weight at 105 °C (AENOR, 1973).

178

## 179 *2.2. Sample sets selection*

180 Before the calibration, test and standards sets from the two instruments were  
181 selected, the spectra obtained from the FNS and CORONA equipments were previously  
182 reduced to the same 646 data points (400-1690 nm). No wavelength interpolation was  
183 needed because the wavelength intervals of the two devices matched. Then, raw spectral  
184 data were corrected for baseline and scattering effect using the Savitzky-Golay  
185 (SavGol) algorithm (21-point window, 3<sup>rd</sup>-order polynomial and 2<sup>nd</sup>-order derivative)  
186 (Savitzky and Golay, 1964).

187 After spectral data preprocessing, the 174 samples obtained from each  
188 spectrophotometer, forming the original dataset  $X_o$  (174 x 646),  $Y_o$  (174 x 3), were divided into  
189 three groups: calibration ( $X_c$ ,  $Y_c$ ), standards ( $X_s$ ,  $Y_s$ ) and test ( $X_t$ ,  $Y_t$ ). For the  
190 construction of these sets, Principal Component Analysis (PCA) was used. This  
191 algorithm was applied on the centred  $Y_o$  matrix and then, the scores of the first  
192 component was sorted in increasing order. Calibration, standards and test samples were  
193 then drawn regularly from this ranking, in the following proportions: 61% (106  
194 samples), 6% (10 samples) and 33% (58 samples), respectively.

195

## 196 *2.3. Development of NIRS calibration models*

197 Partial least squares regression (PLSR) with leave-one-out cross validation  
198 (LOOCV) was used for model calibration on  $X_c$  and  $Y_c$ . Models having as many as 20  
199 latent variables (LVs) were considered and the optimal model was determined by  
200 choosing the number of LVs that gave the minimum in the standard error of cross-

201 validation (SECV). Coefficient of determination ( $R^2$ ) between lab-measured and  
202 predicted values was also reported for the optimal model.

203         Once the best calibration model for the prediction of fat, free acidity and  
204 moisture content constructed based on one instrument were selected, it was then applied  
205 on  $X_t$ ,  $Y_t$  of the other instrument. Here, quantitative PLS models were carried out with  
206 and without (models 1, *MI*) the prior performance of standardization procedures.

207

#### 208         2.4. Calibration transfer techniques

209         Slope/bias correction (SBC), piecewise direct standardization (PDS) and transfer  
210 by orthogonal projection (TOP) methods were evaluated to transfer calibration models  
211 from one instrument to another. In this study, FNS-6500 instrument was used as the  
212 *master* and the CORONA 45 visNIR as the *slave* device.

213

##### 214         2.4.1. Slope/bias correction

215         Using the standard sets ( $X_s$  and  $Y_s$ ), an SBC was carried out (models 2, *M2*). For  
216 each response (fat content, free acidity and moisture), the model calibrated on the  
217 master was applied on  $X_s$  and  $y_s$  ( $174 \times 1$ ) of the slave instrument, yielding prediction  $\hat{y}_s$ .  
218 Slope and bias coefficients ( $b$ ,  $b_0$ ) were calculated by a linear regression between  
219 predicted and actual values of  $y_s$ . The model was then tested on the test sets ( $X_t$ ,  $y_t$ ) of  
220 the slave device and the  $y_t$  predicted ( $\hat{y}_t$ ) was corrected ( $\hat{y}_{tc}$ ) as follows (1):

221

$$222 \qquad \qquad \qquad \hat{y}_{tc} = (\hat{y}_t - b_0) / b \qquad (1)$$

223

##### 224         2.4.2. Piecewise Direct Standardization

225           In this work, two different PDS approaches were evaluated. In the first of them,  
226 called as PDS1,  $X_tX_s$  spectra from the slave instrument were corrected, yielding  
227 *standardized*  $X_tX_s$ , in order to become closer to the output from the master device  
228 (models 3, *M3*). For that, a transformation matrix was constructed using  $X_s$  of both  
229 equipments. A new LOOCV-PLS model was calibrated on  $X_cX_s$  and  $Y_cY_s$  of the master  
230 device and then, it was tested on the *standardized*  $X_tX_s$  and  $Y_tY_s$  from the slave  
231 instrument.

232           In the second approach, considered as PDS2,  $X_s$  spectra of the two instruments  
233 were again applied to correct the  $X_cX_s$  spectra from the master device.  $X_s$  spectra from  
234 the slave and the *standardized*  $X_cX_s$  spectra obtained were then used to build a new  
235 LOOCV-PLS model. Finally, the model obtained was tested on  $X_tY_t$  from the slave  
236 device (models 4, *M4*).

237           Different window sizes (WS) were tested (5, 9 and 31) and optimized in order to  
238 compute the transformation matrix using the  $X_s$  samples.

239

#### 240           2.4.3. Transfer by orthogonal projection

241           TOP method was also evaluated as a standardization procedure (models 5, *M5*).  
242 Firstly, the difference between the same  $X_s$  samples measured on both instruments was  
243 used to build a matrix of differences (D). Then, a singular value decomposition (SVD)  
244 of D yielded its  $k$  main principal directions, in the matrix P; spectra from master  
245 instrument were projected orthogonally to P; a LOOCV-PLS model was then built on  
246 the orthogonalized spectra, using a maximum of 20 latent variables. The optimum  
247 number  $k$  of loadings and  $l$  of latent variables were selected on the basis of the  
248 minimum value obtained for SECV. Once selected the  $k$  and  $l$  values, the master  
249 database was orthogonalized to the  $k$  first directions of D and a PLS model was

250 recalibrated on this base using  $l$  latent variables and tested on the samples of the slave  
251 instrument.

252

### 253 *2.5. Computing and model evaluation*

254 Computation procedure was performed using the chemometric software Matlab  
255 ver. 7.0 (The Mathworks Inc., Natick, MA, USA) and several routines created by the  
256 authors, based on algorithms from the PLS Toolbox ver. 3.5 (EigenvectorResearch, Inc.,  
257 Manson, WA, USA).

258 The performance of the prediction models was evaluated by the  $R^2$ , bias and  
259 standard error of prediction (SEP). The higher  $R^2$  and the lower SEP, the better the  
260 robustness.

261

262

## 263 **3. Results and discussion**

264

265 Figure 2 presents the raw spectra of 9 samples drawn randomly from the original  
266 database, and measured by the CORONA (fig.2A) and the FNS (fig. 2B) spectrometers,  
267 respectively. On the common part of both figures, some major bands can be observed.  
268 The main one appears around 1450 nm. It is attributed to the first overtone of the O-H  
269 bond stretching in Osborne *et al.* (1993), and is mainly due to water absorption. The  
270 second one is very sharp, but appears only on some spectra, at 675 nm. It is classically  
271 attributed to the chlorophyll (a and b), as in Solovchenko *et al.* (2010). At 1215 nm, one  
272 can observe a band attributed in Osborne *et al.* (1993) to the second overtone of the C-H  
273 bond stretching and is certainly due to the oleic acids of the olive oil. The last  
274 significant band is located around 980 nm, and is assigned to the second overtone of the

275 O-H bond stretching in Osborne *et al.* (1993), and also due to water absorption. One can  
276 also notice that the spectra recorded by both devices are impacted by a strong baseline  
277 addition, which is due to the light scattering. Indeed, a large part of the photons which  
278 are not collected by the sensors, and consequently considered as absorbed, are actually  
279 scattered. This phenomenon, which causes a multiplicative effect on the signal, appears  
280 additive after the log transform. Considering the visible part of the spectra on both  
281 figures, two groups of spectra can be clearly distinguished. Some spectra present a very  
282 high and relatively flat absorption. They correspond to very mature fruits, mainly black  
283 and very absorbent in the whole visible range. The other ones present two main  
284 absorbance bands, in the blue (450 nm) and in the far red (675 nm) which can be  
285 assigned both to chlorophyll, and producing the green color of the non mature olives.

286 Comparing the two figures provides some other rough observations. The main  
287 bands (1450, 675, 1215 and 980 nm) are very similar on both devices. On the contrary,  
288 the vertical ordering of the spectra differs. The visible part differs between the two  
289 devices. Some bands appear on the FNS spectra for the more mature fruits at 560 and  
290 630 nm. They could traduce a balance between pigments related to chlorophyll and  
291 anthocyanin. Contrarily, the spectra collected by the CORONA appear in the same zone  
292 flatter and noisier. One can see a sharp break on both figures; between 980 and 982 nm  
293 for the CORONA and between 1098 and 1100 nm for the FNS. Both are due to the  
294 change of sensor inside the spectrometers and would cause a strong difference to be  
295 compensated in the transfer procedure.

296 Table 1 gives a statistical summary of the chemical composition of the subsets  
297 formed. The main goal was to build sets with comparable ranges for all the 3 responses.  
298 As can be observed, the mean and standard deviation (SD) for fat content and moisture  
299 parameters showed very similar results in all three sets of calibration, test and standards.

300 However, for acidity property, significant differences were appreciated between the  
301 standards set and the calibration-test sets.

302 The calibration statistics of all the models developed are shown in table 2, which  
303 presents for each parameter measured the best prediction in each situation, showing the  
304 number of PLS factors or latent variables (LVs), the window-size (WS), the  $k$  value, the  
305 coefficient of calibration ( $r^2$ ) and prediction ( $R^2$ ), the bias, the standard error of  
306 calibration (SEC), of cross-validation (SECV), the standard error of prediction (SEP)  
307 corrected for bias (SEPC) and the SEP.

308 As can be observed in table 2, the LOOCV-PLS equations for the prediction of  
309 fat and moisture content based on spectra collected with the FNS instrument and applied  
310 to the spectra collected with the CORONA device (*models 1*), without the performance  
311 of any standardization procedure, gave a model with 4 LVs and a SEP=2.67 % to the fat  
312 content parameter and a model with 7 LVs and a SEP=3.45 % to the moisture content.  
313 However, for free acidity, it was observed that the *model 1* provided a very poor  
314 predictive capacity for this parameter ( $R^2 < 0.5$  and SEP value  $> 11\%$ ). It should be  
315 noted that the high SEP and bias values (1.61 % for fat content, 10.3 % for free acidity  
316 and 1.75 % for moisture content) obtained in these models reveal the need for applying  
317 a standardization procedure between spectrophotometers.

318 Table 2 also shows the results of the SBC, PDS and TOP methods applied in  
319 order to transfer of calibrations for the three parameters.

320 The effect of slope/bias correction using 10 samples for the transfer is shown in  
321 *models 2*. As expected, the values of bias for the predictions achieved for all properties  
322 were considerably lower in these models than those obtained in the previous *models 1*.  
323 For fat content, a bias of 0.41 % and a SEP of 2.54 % were obtained. For free acidity,  
324 SEP and bias values were enormously reduced in both models, achieving a bias of -1.18

325 % and a SEP of 3.61 %. Finally, for moisture parameter a bias of 0.34 % and a SEP of  
326 3.00 % were obtained.

327 For PDS1 and PDS2, the results obtained from *models 3* and *4* are shown, in the  
328 same way, in table 2. As mentioned above (section 2.4.2), the window size (WS) was  
329 optimized in order to compute the transformation matrix using the standards samples.  
330 For the calibration transfer using PDS1, the lowest SEP values in *models 3* for fat  
331 content, free acidity and moisture were found when a WS of 31, 9 and 5 wavelengths  
332 were chosen, respectively. Concerning the results obtained in PDS2 (*models 4*), similar  
333 values were obtained. For fat content, the lowest SEP value was obtained with a WS of  
334 31 and it was slightly higher (2.78 %) than the ones obtained in PDS1 (SEP=2.31 %).  
335 For acidity parameter, although a higher  $R^2$  value was obtained in *model 4*, the lowest  
336 SEP value was achieved in *model 3* (SEP=2.92 %). Finally, in moisture case, the SEP  
337 value in *model 4* result in 2.36 % using a WS of 31 wavelengths.

338 Both PDS transfer methods have been applied using the standards set (10  
339 samples) and applied to the test set (58 samples). Figure 3 shows the mean square error  
340 (reconstruction error) before and after the PDS processing for the test set using a WS of  
341 5. The discontinuous line corresponds to the reconstruction error between the spectra  
342 measured with both devices before standardization, whereas the other line shows the  
343 result after the standardization process. As can be observed from the figure 3, PDS  
344 transfer allows reconstruction error to be reduced for the test set, indicating the correct  
345 performance of the proposed methodology.

346 For TOP method (*models 5*), a low-SECV basin, with SECV level of 1.80 % fat,  
347 was described by  $k=3$  and  $lv=8$ , achieving a  $R^2$  and a SEP value of 0.87 and 1.97 %, respectively.  
348 For acidity property, the optimal SECV close to 3.00 % oleic acid, was  
349 seen for  $k=5$  and  $lv=5$ . In this case, a  $R^2$  and SEP values of 0.66 and 2.52 %, respectively.

350 respectively, were obtained. Finally, the best moisture prediction ( $R^2 = 0.92$  and SEP =  
351 2.93 %) was carried out with  $k=5$  using  $lv=8$  in this *model 5*.

352 Figure 4 shows the spectra of two standards samples (#141 and #56), collected  
353 by the two devices, before (original spectra) and after the TOP processing (to  $k=1$  and  
354  $k=5$ ). The effect was remarkable. As can be observed in figure 4, the two original  
355 spectra (2<sup>nd</sup> derivative) of a same sample acquired with both spectrometers, showed  
356 several differences in the visible region, specially between wavelengths of 675-775 nm  
357 and between the wavelengths of 960-980 nm and 1450-1550 nm. After TOP, most were  
358 corrected using a  $k$  value of 1. Only, for #56, differences observed in visible region were  
359 not completely corrected with  $k=1$ . These differences totally disappeared when a  $k=5$   
360 was used.

361

### 362 3.1. Comparison of transfer methods

363 All three transfer methods (SBC, PDS and TOP) worked well for transferring of  
364 the present NIRS calibrations for fat, free acidity and moisture content in intact olives.

365 For fat parameter, the best calibration transfer performance was found when the  
366 TOP procedure was applied (*model 5*). Note that the SEP value after TOP  
367 standardization was significantly lower (1.97 %) using 8 latent variables than the error  
368 obtained in the original calibration (*model 1*) with LVs=4 (SEP=2.67 %). Additionally,  
369 as can be observed in table 2, the  $R^2$  values increased from 0.84 in *M1* to 0.87 in *M5*  
370 and the bias values decreased from 1.61 % in *M1* to 0.26 % in *M5*.

371 In the same way, the application of TOP algorithm allowed to obtain the best  
372 prediction results for the free acidity property. The SEP value obtained (table 2)  
373 indicated that the prediction of acidity was greatly improved using TOP transferred



374 rather than untransferred models. The prediction performed in *M5* gave a SEP of 2.52  
375 %, a bias of -0.36 % and a  $R^2$  of 0.66, using only 5 latent variables.

376 Finally, for moisture parameter, it can be seen that although the fitting of the  
377 models was slightly better using TOP algorithm, the predictive ability of the model  
378 developed with the PDS1 method (*model 3*), was better. The initial SEP of 3.45 % and  
379 the bias value of 1.75 % obtained in *MI* were highly reduced until reach a SEP=2.24 %  
380 and a bias= -0.57 with a WS of 5 and using only 7 latent variables.

381 Figure 5 shows the test results of the three best models achieved from the FNS  
382 master instrument for each one of the parameters evaluated.

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384

## 385 **Conclusions**

386 Slope/bias correction (SBC), piecewise direct standardization (PDS) or transfer  
387 by orthogonal projection (TOP) can be used for transferring multivariate calibrations  
388 models for the prediction of fat content, free acidity and moisture on intact olives  
389 between a pre- and a post- dispersive NIRS spectrophotometer.

390 From this study, it can be possible to affirm that the use of these standardization  
391 procedures allows that the huge databases on intact olives built over several years with  
392 an off-line instrument can be successfully transferred to new hand-held devices that  
393 could be implanted at mill level.

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## 396 **Acknowledgements**

397 This work is part of a PhD thesis which is being carried out by the first author, at  
398 the Postharvest and Food Technology Area in the olive culture station IFAPA 'Alameda

399 del Obispo' (Córdoba, Spain), with a grant from the National Institute for Agronomic  
400 Research (INIA).

401 The authors gratefully acknowledge the financial support by the Project Number  
402 DEX-560630-2008-16 (Application of NIRS technology for the on-line determination  
403 on an olive mill of control, quality and food safety parameters), funded by the  
404 Andalusian Federation of Cooperative Agrarian Companies (FAECA) and IFAPA. The  
405 authors also would like to FMP/ffee "Andalucía se mueve con Europa" for research  
406 support of this study.

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