

1 **Effect of spiking strategy and ratio on calibration of on-line visible and**  
2 **near infrared soil sensor for measurement in European farms**

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9  
10 **Abstract**

11  
12 A previously developed on-line visible and near infrared (vis-NIR) spectroscopy-based soil  
13 measurement system was implemented for the measurement of soil organic carbon (OC),  
14 total nitrogen (TN) and moisture content (MC) in three fields at three European farms. The  
15 on-line sensor platform was coupled with a mobile, fibre type, vis-NIR spectrophotometer  
16 (AgroSpec from tec5 Technology for Spectroscopy, Germany), with a measurement range  
17 of 305 - 2200 nm, to acquire soil spectra in diffuse reflectance mode. A general calibration  
18 set of 425 soil samples, spiked with different number of spectra from the three validation  
19 fields were used to establish calibration models for the studied soil properties using partial  
20 least squares (PLS) regression analysis. Different spiking strategies and spiking ratios were  
21 investigated and results revealed that the best prediction accuracy was obtained after 20%  
22 spiking ratio with samples whose spectra were measured in the laboratory. Evaluated by the  
23 values of residual prediction deviation (RPD), which is the ratio of standard deviation to  
24 root mean square error of prediction (RMSEP), the accuracy of the on-line measurement

25 was classified as excellent for MC (RPD = 2.76 - 3.96), good to very good for OC (RPD =  
26 1.88 - 2.38) and good to excellent for TN (RPD = 1.96 - 2.52). Reducing the number of  
27 samples used for spiking resulted in deteriorating the prediction accuracy, although 1-2  
28 samples per ha were found to provide good predictions. There was a distinguishable spatial  
29 similarity between the on-line and laboratory measured maps for all studied properties,  
30 although the full-data point maps provided more detailed information about the spatial  
31 variation. This confirms that the on-line vis-NIR soil sensor provides correct and detailed  
32 information about soil OC, TN and MC at high sampling resolutions.

33

34 Keywords: on-line measurement, soil properties, spiking, European farms, accuracy.

35

## 36 **1. Introduction**

37

38 One of the strategies used in precision agriculture (PA) is to minimize the production costs  
39 through enhanced efficiency, which can increase profits and conserve the environment. It is  
40 proved that PA is an environmental friendly strategy, in which farmers can vary the use of  
41 inputs to respond to variable soil and crop conditions within a field (Srinivasan, 2006).  
42 Conventional determination of soil spatial variability usually involves manual soil  
43 sampling, sample pre-treatment, laboratory reference analyses and mapping. This procedure  
44 is very expensive, time consuming and provides scattered measurement points. Therefore,  
45 the development of a fast, robust, cost effective and environmental friendly detecting  
46 method of the soil spatial variability is a preliminary task for the implementation of PA.

47 Visible and near infrared (vis-NIR) spectroscopy became recently a proven technique for  
48 fast, cost effective and high resolution data collection on soil properties (Viscarra Rossel &  
49 McBratney, 1998; Shepherd & Walsh, 2002; Mouazen et al, 2010; Stenberg et al., 2010;).  
50 In comparison to non-mobile analysis, there is considerably less literature available about  
51 on-line vis-NIR spectroscopy analysis of soil properties. Generally, both the laboratory and  
52 *in situ* non-mobile vis-NIR methods provide better accuracy than the on-line method  
53 (Kuang et al., 2012). This might be attributed to other factors influencing the latter method  
54 only, such as noise associated with tractor vibration, sensor-to-soil distance variation  
55 (Mouazen et al., 2009), stones and plant debris and difficulties of matching the position of  
56 soil samples collected for validation with corresponding spectra collected from the same  
57 position (Mouazen et al., 2007). A review on the current status of on-line vis-NIR  
58 measurement systems confirms that only three systems are available today (Shibusawa et  
59 al., 2001; Mouazen et al., 2005; Christy et al., 2008). The beginning of these systems dates  
60 back to 1991, when Shonk et al., (1991) developed a system to measure soil organic matter  
61 (SOM) and moisture content (MC), which utilised a single wavelength (660 nm) of light,  
62 reporting a determination coefficient ( $R^2$ ) of 0.83 for SOM. Shibusawa et al., (2001)  
63 developed an on-line vis-NIR (400 – 1700 nm) sensor to predict organic carbon (OC), MC,  
64 pH and NO<sub>3</sub>-N. Although this system is highly technically instrumented, it is rather  
65 expensive. Christy et al., (2008) developed a prototype soil reflectance mapping unit  
66 equipped with a vis-NIR spectrophotometer, which is commercially available in the market  
67 today. The sapphire glass of the optical probe makes direct contact with soil and stones.  
68 They have reported that OC can be successfully measured with a RMSEP of 3.0 gkg<sup>-1</sup> in a  
69 high OC variability area (standard deviation = 5.1 gkg<sup>-1</sup> and range = 3.0-26.3 gkg<sup>-1</sup>). A

70 simpler design to the one of Shibusawa et al., (2001) without sapphire window optical  
71 configuration was developed by Mouazen (2006). So far, the system provided variable  
72 degrees of success for the measurement of MC, total nitrogen (TN), total carbon (TC), pH  
73 and available P in different soils in Belgium and northern France (Mouazen et al., 2005;  
74 Mouazen et al., 2007; Mouazen et al., 2009). More recently, employing the on-line system  
75 developed by Christy et al. (2008), Brickley et al. (2010) reported on the on-line  
76 measurement for soil OC and clay content. Although authors did not report quantitative  
77 estimation of accuracy for OC, they calculated a standard error of prediction (SEP) of 3.4  
78  $\text{gkg}^{-1}$  and a RPD value of 1.4 for clay content. Using the same system, coupled with  
79 topography and aerial photograph data, Munoz & Kravchenko (2011) reported low to  
80 moderate accuracy of soil OC measurement with  $R^2$  and RMSEP ranged from 0.44 to 0.66  
81 and from 1.41 to 1.51  $\text{gkg}^{-1}$ , respectively. Applying this vis-NIR system in combination  
82 with electrical conductivity (EC) and temperature sensors in a Danish field, Knadel et al.  
83 (2011) obtained moderate prediction accuracy (RMSEP = 59.4  $\text{gkg}^{-1}$  and RPD = 2.3) for  
84 soil OC. However, the vis-NIR sensor alone only achieved a relatively low accuracy  
85 (RMSEP = 59.8  $\text{gkg}^{-1}$ , RPD = 1.9). This brief review reveals that the existing on-line  
86 sensors do not provide sufficiently accuracy and stability to recommend them for site  
87 specific application of different inputs. The variable degrees of performance of these on-  
88 line sensors might be attributed to the fluctuation in model performance, with the majority  
89 of them established for field-scale analysis (e.g. Shibusawa et al., 2001; Mouazen et al.,  
90 2005; Christy et al., 2008; Munoz & Kravchenko, 2011), or for regional- and country-scale  
91 analysis (e.g. Mouazen et al., 2007; Mouazen et al., 2009). Some studies suggested spiking  
92 local samples into the general calibration models can improve prediction accuracy under

93 laboratory or *in situ* measurement conditions (Shepherd & Walsh, 2002; Janik et al., 2007;  
94 Waiser et al. 2007; Sankey et al. 2008; Viscarra Rossel et al. 2008; Minasny et al. 2009;  
95 Wetterlind and Stenberg, 2010; Guerrero et al. 2010). As yet, no general calibration  
96 procedure that included spiking strategy have been reported for establishing calibration  
97 models of on-line sensors, which has led to stable performance of on-line sensors, at the  
98 lowest model production cost.

99 This paper aims at reporting on a methodology for the calibration of a vis-NIR on-line  
100 measurement system (Mouazen, 2006), including spiking concept for automatic data  
101 collection of OC, TN and MC at farm scale in three European farms.

102

## 103 **2. Material and methods**

104

### 105 ***2.1 Soil samples and laboratory analyses***

106

107 A total of 425 soil samples were used as a general dataset to be used to establish calibration  
108 models for the measurement of OC, TN and MC in European soils (Table 1). They were  
109 collected from four different farms in Europe, namely Mespol Medlov, A.S. (Czech  
110 Republic, <http://farmsubsidy.org/CZ>), Wimex (Germany, <http://www.wimex-online.de>),  
111 Bramstrup Estate (Denmark, <http://www.bramstrup.dk>) and Silsoe Farm (The UK). Bulk  
112 samples from 16 cores were collected from the upper soil layer (0-30 cm) in the spring of  
113 2008 (Czech Republic and Germany), spring of 2009 (Denmark) and summer of 2009 (UK)  
114 and covered diverse soil conditions (Kuang and Mouazen, 2011). A total of 128, 97 and 48

115 soil samples were, respectively, collected from seven fields in Mespol Medlov, A.S. farm,  
116 six fields in Bramstrup Estate farm and two fields in Silsoe farm. Further 152 soil samples  
117 were collected from four sub-areas belong to Wimex farm, with four samples from two  
118 fields at Reppichau, 50 samples from nine fields at RAG, 20 samples from ten fields at  
119 Aken and 54 samples from fifteen fields at the Wulfen farm.

120 Apart from these soil samples, another 113 soil samples were collected from three selected  
121 fields, where on-line measurement was carried out, namely, in Mespol Medlov,  
122 Bramstrump Estate and Silsoe farms (Table 2). These were considered as validation fields.  
123 They were collected during the on-line measurement from the bottom of trenches at a depth  
124 of 15 cm. The validation field in Silsoe farm is 500 m away from the nearest fields, where  
125 samples used for general data set were collected, whereas the validation fields in Mespol  
126 Medlov and Bramstrump Estate farms are 2.5 km 3 km far from the nearest fields,  
127 respectively. A principal component analysis (PCA), performed on 425 samples of general  
128 calibration dataset and 113 validation data set shows clear separation between validation  
129 samples of each individual farm from the general dataset samples (Fig. 1).

130 Around 200 g of soil from each sample was kept deep frozen (-18 °C) until analysis. After  
131 careful mixing, half of each sample from Czech Republic, Germany and Denmark was sent  
132 to Leibniz Centre for Agricultural Landscape Research (ZALF) in Germany for soil  
133 chemical analyses for TN and OC and the second half was sent to Cranfield University for  
134 optical measurement and data analysis. Samples collected from the UK and 113 soil  
135 samples collected during the on-line measurement in 2010, were subjected to both optical  
136 and chemical measurement at Cranfield University. Sample statistics of laboratory

137 reference measurements is summarised in Table 3 for the calibration and validation  
138 datasets.

139

## 140 *2.2 On-line measurement*

141

142 The on-line measurement system designed and developed by Mouazen (2006) was used  
143 (Fig. 2) to measure three fields in the three European farms. It consists of a subsoiler, which  
144 penetrates the soil to the required depth, making a trench, whose bottom is smoothed by  
145 the downwards forces acting on the subsoiler. The optical probe is housed in a steel lens  
146 holder. This is attached to the backside of the subsoiler chisel in order to acquire soil  
147 spectral reflectance data from the smooth bottom of the trench. The subsoiler was  
148 retrofitted with the optical unit and attached to a frame. This was mounted onto the three  
149 point linkage of the tractor (Mouazen et al., 2005a). An AgroSpec mobile, fibre type, vis-  
150 NIR spectrophotometer (Tec5 Technology for Spectroscopy, Germany) with a  
151 measurement range of 305-2200 nm was used to measure soil spectra in diffuse reflectance  
152 mode. The spectrometer was an IP 66 model, protected for harsh working environments. A  
153 differential global positioning system (DGPS) (EZ-Guide 250, Trimble, USA) was used to  
154 record the position of on-line measured spectra with sub-meter accuracy. A Panasonic  
155 semi-rugged laptop was used for data logging and communication. The spectrometer  
156 system, laptop and DGPS were powered by the tractor battery.

157 Three fields were measured in summer 2010, namely, in Mespol Medlov, A.S. farm in  
158 Czech Republic, Bramstrup Estate farm in Denmark and Silsoe farm in the UK. In each

159 field, blocks of 150 m by 150 m, covering about 2 ha of land were measured. Each  
160 measured line was 150 m long with 10 m intervals between adjacent transects (Fig. 3). The  
161 travel speed of the tractor was around 2 km/h and the measurement depth was set at 15 cm.  
162 During the measurement at each line, 2 or 3 soil samples were collected from the bottom of  
163 the trench and the sampling positions were carefully recorded with a DGPS. Each of those  
164 samples was equally divided into two parts. One half used to carry out the laboratory  
165 reference measurements of soil OC, TN and MC and the other half used for optical  
166 scanning.

167

### 168 *2.3 Laboratory reference analyses*

169

170 Soil OC and TN were measured by a TrusSpecCNS spectrometer (LECO Corporation, St.  
171 Joseph, MI, USA), using the Dumas combustion method. Soil MC was determined by oven  
172 drying of the soil samples at 105 °C for 24 h. The results of the textural analysis of the three  
173 on-line measured fields were based on a mixed sample from each field. Each sample was  
174 subjected to wet sieving and a hydrometer test in order to determine the particle size  
175 distribution. Texture classes were determined according to the United State Department of  
176 Agriculture (USDA) classification system (Table 2).

177

### 178 *2.4 Optical measurement*

179



180 Each soil sample was put into a glass container and mixed well. Big stones and plant  
181 residue were removed (Mouazen et al., 2007). Soil from each sample was placed into three  
182 Petri dishes, which were 2 cm deep and 2 cm in diameter. The soil in the Petri dish was  
183 shaken and pressed gently before levelling with a spatula. A smooth soil surface ensures  
184 maximum light reflection and a high signal to noise ratio (Mouazen et al., 2007). The soil  
185 samples were scanned by the same AgroSpec portable spectrophotometer (Tec5  
186 Technology for Spectroscopy, Germany), used during the on-line measurement. A 100 %  
187 white reference was used before scanning. A total of 10 scans were collected from each cup  
188 and these were averaged in one spectrum.

189

## 190 *2.5 Sample pre-treatment and development of calibration models*

191

### 192 *2.5.1 Pre-treatment of spectra*

193

194 The spectral range of the soil spectra was first reduced to 371 - 2150 nm to eliminate the  
195 noise at both edges of each spectrum. The number of wavelengths was then reduced by  
196 averaging three successive wavelengths in the visible range, and 15 points in the NIR  
197 range. The Savitzky-Golay smoothing, maximum normalisation and first derivation  
198 (Martens & Naes, 1989) were successively implemented using Unscrambler 7.8 software  
199 (Camo Inc.; Oslo, Norway). The pre-treated spectra and the laboratory chemical  
200 measurement values were used to develop the calibration models.

201

202 **2.5.2 Spiking**

203

204 The purpose of spiking is to introduce variability of a new set of data into a general  
205 calibration dataset. Three different spiking strategies of soil samples collected from the  
206 target validation fields were selected. These included - no spiking of soil samples, - spiking  
207 with laboratory non-mobile scanned spectra and - spiking with on-line (mobile) spectra  
208 recorded during the on-line measurement. When spiking was considered with the latter 2  
209 cases, 20% of a randomly selected set of samples (21 samples from the three fields) from  
210 the total 113 soil samples collected during the on-line measurement were spiked into the  
211 general data set of 425 samples, collected from the 4 European farms (see also 2.1).  
212 Furthermore, smaller ratios of spiking samples of 5% (8 samples) and 10% (12 samples)  
213 were randomly selected from the laboratory measured spectra only, and were used as  
214 spiking sets to envisage the influence of different spiking ratios (sample numbers) on the  
215 on-line prediction accuracy. By adopting these spiking strategies and ratios we obtained  
216 five calibration sets (Tables 4), namely, without spiking, 5%, 10% and 20% spiking ratios  
217 of laboratory measured spectra and 20% spiking ratio of on-line measured spectra.

218

219 **2.5.3 Development of calibration models**

220

221 The five calibration sets were subjected to a partial least squares (PLS) regression analysis  
222 with the leave-one-out cross validation using an Unscrambler 7.8 software (Camo Inc.;  
223 Oslo, Norway), which resulted in five groups of PLS models for each soil property (Table  
224 4). For all five spiking strategies and ratios the same on-line data set of 80 %, was used as

225 the validation set for the three soil properties in each field. This resulted in three validation  
226 sets of 39, 30 and 23 samples in Czech Republic, Denmark and UK validation fields,  
227 respectively (Table 5). These three data sets were used to validate the on-line measurement  
228 and to develop maps comparing the laboratory reference with the corresponding on-line  
229 measured values.

230 The number of latent variables for a model was determined by examining a plot of the  
231 leave-one-out cross-validation residual variance against the number of latent variables  
232 obtained from the PLS. The latent variable of the first minimum value of residual variance  
233 was selected. Outliers were detected using the residual sample variance plot after PLS.  
234 Samples located far from the zero line of residual variance were considered outliers and  
235 excluded from the cross-validation sample set.

236 The prediction performance of the PLS models was evaluated by means of root mean  
237 square error of prediction (RMSEP) and the residual prediction deviation (RPD), which is  
238 the ratio of standard deviation (SD) of the measured values to RMSEP. Viscarra Rossel et  
239 al. (2006) classified RPD values as follows:  $RPD < 1.0$  indicates very poor  
240 model/predictions and their use is not recommended; RPD between 1.0 and 1.4 indicates  
241 poor model/predictions where only high and low values are distinguishable; RPD between  
242 1.4 and 1.8 indicates fair model/predictions which may be used for assessment and  
243 correlation; RPD values between 1.8 and 2.0 indicates good model/predictions where  
244 quantitative predictions are possible; RPD between 2.0 and 2.5 indicates very good,  
245 quantitative model/predictions, and  $RPD > 2.5$  indicates excellent model/predictions. This  
246 classification system was adopted in this study.

247

## 248 ***2.6 Development of soil maps***

249

250 Two types of maps were developed, namely, comparison and full-data points maps. The  
251 comparison maps were developed to compare on-line predicted with laboratory reference  
252 measurement of a soil property based on randomly selected points in the field. This  
253 comparison also included maps of predicted values based on laboratory scanned spectra of  
254 the same randomly selected samples. The full-point maps were developed using all on-line  
255 predicted data points (~1500 - 2000 point per ha). ArcGis 10 (ESRI, USA) software was  
256 used to generate the comparison maps, using the inverse distance weighing (IDW)  
257 interpolation methods. To produce the latter maps, Vesper 1.6 software, developed by  
258 Australian Centre for Precision Agriculture, was used to develop semivariogram models for  
259 OC, TN and MC using the entire field on-line data. Based on semivariogram parameters  
260 and kriging interpolation method, ArcGis 10 (ESRI, USA) was used to produce the full-  
261 data point maps.

262 A flow diagram illustrating the different steps taken into account in this study, from the  
263 collection of soil sample to the development of different maps is shown in Fig. (4). This  
264 Figure illustrates clearly that the vis-NIR calibration models were developed using soil  
265 spectra scanned under non-mobile laboratory environment, except for spiking with 20% on-  
266 line measured spectra.

267

## 268 **3. Results and discussion**

269

### 270 ***3.1 Accuracy of calibration models in cross-validation***

271

272 Based on spiking strategies and ratio of samples spiked in the general calibration set, the  
273 accuracy of calibration models achieved based on cross-validation proves promising results  
274 (Table 4). Examining RPD values reveals that the models with 20 % spiking ratio of  
275 laboratory-scanned spectra were the most accurate with RPD values of 2.82, 2.61 and 4.28  
276 for OC, TN and MC, respectively. Similar results were reported by Kuang & Mouazen  
277 (2011) but for a larger range (350-2500 nm) vis-NIR spectrophotometer. Furthermore, RPD  
278 values of all models were equal or larger than 2.5. According to Viscarra Rossel et al.  
279 (2006), an RPD values above 2.5 indicates excellent prediction results. Results also show  
280 that models without spiking provide smaller accuracy (larger RMSEP and smaller RPD  
281 values) than models with spiking for all spiking strategies and ratios (Table 4), except for  
282 models with 20% spiking ratio of on-line samples for OC and TN.

283

### 284 *3.2 Validation of on-line measurement*

285

286 The same spectra pre-treatment was used for the on-line collected spectra as that used for  
287 the development of calibration models. The on-line collected spectra after spectra pre-  
288 treatment were used to predict MC, TN and OC using calibration models developed in  
289 advance, as explained above. The laboratory reference measurement values were compared  
290 with the on-line predicted concentration values at the same positions. Table 5 summarises  
291 the accuracy of the on-line measurement for studied soil properties based on different  
292 spiking strategies (no spiking, spiking with laboratory or on-line collected spectra) and  
293 spiking ratios. Table 5 reveals that RPD values were above 2 for all soil properties in all

294 fields, except for 3 cases. Out of these cases, RPD in one case was 1.98 for OC in the field  
295 in Denmark. The other two cases (OC and TN in the Danish and the UK fields,  
296 respectively) are for models developed based on 20% spiking ratio with on-line collected  
297 spectra. Adopting Viscarra Rossel et al. (2006) classification system for the prediction  
298 accuracy reveals that the on-line prediction of OC is good/very good and good/excellent for  
299 TN and is excellent for MC, for all modelling strategies without and with spiking (Table 5).  
300 As SOM or OC are essential for soil management and carbon sequestration, they were the  
301 main properties considered for on-line vis-NIR measurement (Shonk et al., 1991; Hummel  
302 et al., 2001; Shibusawa et al., 2001; Mouazen et al., 2007; Christy et al., 2008; Brickleyer  
303 et al., 2010). Although there are some significant absorbance peaks associated with C=O,  
304 C-H + C-H and C-H + C-C overtones and combinations in the NIR spectral ranges, only  
305 few moderate successful cases for on-line measurement have been reported so far (Hummel  
306 et al., 2001; Christy et al., 2008). This might be attributed to the several affecting factors  
307 during on-line measurement (Mouazen et al., 2007; Mouazen et al., 2009; Stenberg et al.,  
308 2010). Compared to the on-line sensing of OC, there are even fewer studies on TN can be  
309 found in the literature (Christy et al., 2008; Mouazen et al., 2007). Only Christy et al.,  
310 (2008) archived high accuracy for TN ( $R^2 = 0.86$ ) for a field scale calibration. This was not  
311 confirmed stable for different fields, as reported in the current study where on-line  
312 validation is classified as good to excellent in the three measured fields (Table 5). Actually,  
313 there is high correlation between OC and TN, because the N content in soil is almost  
314 entirely dependent on SOM content, and the overall TN:OC ratio is 1:10 (Martin et al.,  
315 2002). This high correlation explains the successful measurement of TN, although no direct  
316 spectral response exists for TN.

317 Soil MC is the most successfully on-line measured soil property and higher accuracies as  
318 compared to OC and TN were reported in the literature (Hummel et al., 2001; Mouazen et  
319 al., 2005; Christy et al., 2008). This is due to significant O-H absorbance peaks at 950,  
320 1450 and 1950 nm overtones. Although the range of MC was relative narrow in the current  
321 study ( $SD = 12.8 - 23.4 \text{ gkg}^{-1}$ ) (Table 5), the RMSEP for on-line prediction after 20%  
322 spiking with laboratory scanned spectra is very small ( $3.23 \text{ gkg}^{-1} - 7.40 \text{ gkg}^{-1}$ ), proving the  
323 excellent performance of the sensor for on-line sensing of MC.

324

### 325 ***3.3 Soil maps***

326

#### 327 ***3.3.1 Comparison maps***

328

329 Figure 5 compares maps of on-line spectra-based predicted values (e.g. models of 20%  
330 spiking ratio with laboratory scanned samples), laboratory spectra-based predicted values  
331 and laboratory reference measured values for OC (5a), TN (5b) and MC (5c) in the field of  
332 Bramstrup Estate farm in Denmark, shown as an example. In order to allow for meaningful  
333 comparisons between reference and on-line measured maps, the same number of classes (7  
334 classes) was considered for all maps with each class has identical range in the three maps  
335 (Mouazen et al., 2007). A comparison between maps of measured and predicted soil  
336 properties investigated shows large spatial similarity, with high and low zones match  
337 almost perfectly. No spatial differences can be observed between validation maps  
338 developed with on-line measured spectra and the corresponding maps developed with

339 laboratory (non-mobile) measured soil spectra. This proves the high quality of on-line  
340 measured spectra, which reflects the sensor stability and robustness during on-line  
341 measurement. Figure 6 shows the error maps between laboratory reference measurement  
342 and on-line prediction values of OC, TN and MC in the Denmark field. It can be observed  
343 that the largest error in the three maps occur at the boarder of each field, which might be  
344 attributed to errors associated with placing or removing the optical probe in the soil,  
345 respectively, at the start or end of some measured lines. Another reason might be the  
346 irregularity of the soil surface at the field circumference.

347

### 348 ***3.3.2 Full-data point maps***

349

350 The semivariogram parameters of the three properties in the Denmark field are summarised  
351 in Table 6. Maps show high spatial variability of the three soil properties, although the field  
352 area is remarkably small of 2 ha (Fig. 7). This high variability encourages the need for on-  
353 line soil sensor for the characterisation of within field spatial variability of soil properties,  
354 as zones with different levels of concentration should be managed differently in PA,  
355 particularly for site specific fertilisation. In particular, high similarity between OC and TN  
356 maps can be observed, which can be attributed to the high correlation exist between OC and  
357 TN in the soil. Comparing between laboratory reference analyses maps produced based on  
358 few measurement points (Figure 5) with corresponding full-data point maps based all on  
359 on-line measurement points (Figure 7), more detailed characterisation of within field



360 variation of the three soil properties can be observed in the latter maps. This detailed  
361 variation is essential for precise management of input applied to the soil-crop system.

362

### 363 *3.4 Effect of spiking strategy and spiked sample number*

364

365 Comparing the effect of different spiking strategies and ratios, revealed that the best  
366 accuracy of on-line prediction of the three soil properties is achieved with models  
367 developed after 20% spiking ratio with laboratory scanned spectra. However, it can be  
368 clearly seen that even the smallest 5% spiking ratio with laboratory scanned spectra can  
369 improve the on-line prediction for all properties in all fields, comparing to on-line  
370 prediction using models without spiking. Furthermore, the prediction accuracy decreases as  
371 the number of spiked samples decreases and vice versa. However, for spiking with on-line  
372 measured spectra (e.g. 20% spiking ratio), the prediction accuracies are smaller than the  
373 corresponding accuracies obtained from models without spiking. This may suggest that  
374 spiking with on-line collected spectra is not the best strategy to follow. This might be  
375 attributed to difficulties associated with precision of matching sampling position of on-line  
376 collected spectra with corresponding soil sample used for laboratory reference analysis,  
377 which is an issue highlighted by Mouazen et al. (2007).

378 In order to investigate the effect of sample number per hectare spiked into the general  
379 calibration set on prediction accuracy, variations in RPD values obtained from model  
380 validation of on-line set versus sample number spiked in the general data set (425 samples)

381 is shown in Fig. (8). Clear increases in RPD values with spiked sample number per ha can  
382 be observed. However, the degree of increase differs according to soil property considered,  
383 with larger increases is observed for OC and TN, and smaller increases for MC. This is also  
384 affected by the field measured. On the basis of average values of the RPD of the three  
385 fields, an increase in spiked sample number from 1/1.5 to 3.5/4.5 per ha, leads to an  
386 average increase in % RPD of 9.1% and 11.1% for OC and TN, respectively, whereas an  
387 increase of 10.4% is observed for MC. This quantitative evaluation of % difference of RPD  
388 values may suggest that spiking of laboratory scanned spectra with a sampling rate of 1 to 2  
389 samples per ha is sufficient to obtain accurate on-line prediction of soil properties. **Figure**  
390 **(8) proves that spiking with 1-2 sample per ha results in a very good model performance for**  
391 **OC (RPD = 2.13-2.32) and TN (RPD = 2.15-2.27) and excellent model performance for**  
392 **MC (RPD = 2.91-3.62).** However, this number of samples is only valid for the degree of  
393 spatial variation similar to those encountered in the three validation fields of this study.  
394 This number might increase or decrease for other study cases, if the spatial variation is  
395 larger or smaller, respectively, as compared to those of the fields of the current study. This  
396 sampling rate is almost identical to the sampling rate considered for conventional analysis  
397 of soil properties, adopted today by laboratories to provide fertilisation recommendations  
398 (Mouazen et al., 2007 & 2009). This reveals that with only slight decrease in accuracy, the  
399 consumable cost of on-line measurement of OC, TN and MC would be approximately  
400 identical to that of laboratory conventional methods, after excluding the costs of on-line  
401 sensor and general dataset. However, traditional laboratories provide fertilisation  
402 recommendations based on 1 sample per ha, where the spatial variation in soil properties is  
403 ignored. Furthermore, the on-line measurement system enables the collection of high

404 number of data points (around 1500 - 2000 readings per ha), with an average of 2 points per  
405 meter travel distance. Assuming that laboratory methods of soil analysis do not prone to  
406 measurement error, which is not the case in practice, the detailed information about the  
407 spatial variation in soil properties obtained with the on-line sensor, even with a smaller  
408 accuracy than that of the laboratory methods can be considered a pronounced advancement  
409 in the field of proximal soil sensing. In addition to the high accuracy achieved for the  
410 measurement of the three properties, the performance illustrates stability (Table 5) among  
411 the three fields, which is the ultimate objective of the vis-NIR calibration of soil properties,  
412 prone to fluctuation in performance among different sites.

413 Considering the wide geographical and texture variation of the three measured fields (Table  
414 2), the concept of general data set spiked with a small number of samples (1-2 samples per  
415 ha) from each new measured field seems to be a successfully calibration procedure for on-  
416 line vis-NIR measurement of soil OC, TN and MC. Generally, the general model concept  
417 proposed by Mouazen et al (2007) is based on a sufficient number of soil samples to  
418 account for the soil spatial variability in a new target site, where the prediction will be  
419 carried out (Viscarra Rossel et al., 2008; Guerrero et al., 2010). Spiking local (target site)  
420 soil samples into global or continental models for non-mobile calibrations proved to be an  
421 efficient way to improve the prediction accuracy of target field for some soil constituents  
422 (Shepherd & Walsh, 2002; Janik et al., 2007; Waiser et al. 2007; Guerrero et al., 2010;  
423 Minasny et al. 2009; Viscarra Rossel et al. 2009; Wetterlind and Stenberg, 2010). The  
424 successful implementation of spiking general calibration models with field specific samples  
425 confirms the sample spiking technique is successfully applied for on-line vis-NIR

426 spectroscopy sensing, similar to that of laboratory and *in situ* non-mobile vis-NIR  
427 spectroscopy.

428

#### 429 **4. Conclusions**

430

431 This paper reports on the performance of vis-NIR spectroscopy-based calibration models  
432 spiked with different sampling strategies and ratios for the on-line prediction of soil TN,  
433 OC and MC. The results reported allow the following conclusions to be drawn:

434 1- The on-line measurement system is robust that enables the collection of data on  
435 several soil properties, simultaneously.

436 2- This system enables the collection of large data points per field (around 1500-2000  
437 readings per ha). The large number of points will open new possibilities for the  
438 management of soil-water-plant system, which was not achievable so far.

439 3- The accuracy of on-line prediction of OC, TN and MC was classified as good to  
440 excellent prediction performance with RPD values range between 1.88 and 3.96.

441 4- The concept of general calibration models, spiked with 1-2 samples per ha from  
442 new measured fields is a successful procedure for the calibration of the on-line vis-  
443 NIR sensor. However, a smaller or larger number of spiking samples might be  
444 required for fields with a smaller or larger spatial variability, respectively.

445 Further work is being undertaken to improve the prediction accuracy of the system by  
446 accounting for the effect of MC and texture on the result obtained. The concept of spiking

447 of general calibration models needs to be tested for other soil properties than those reported  
448 in this study.

449

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# Effect of spiking strategy and ratio on calibration of on-line visible and near infrared soil sensor for measurement in European farms

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