# **Experimental Study on Sample Size for Laboratory** Calibration Tests of Commercial Dielectric Soil Water Sensors

# Shinji SUZUKI<sup>1,3\*</sup>, Tomoyoshi HIROTA<sup>1</sup> and Yukiyoshi IWATA<sup>2</sup>

<sup>1</sup> National Agricultural Research Center for Hokkaido Region, National Agriculture and Food Research Organization (NARO) (Sapporo, Hokkaido 062-8555, Japan)

<sup>2</sup> National Agricultural Research Center for Hokkaido Region, NARO (Memuro, Hokkaido, 082-0081, Japan)

### Abstract

When calibration tests of dielectric soil water sensors are conducted in a laboratory, it is of great importance to know the appropriate sample size for each sensor used. In the current study, appropriate sample sizes for different dielectric sensors of time domain reflectometry (TDR) systems (TDR100 with CS605 probe and TRIME-EZ), water content reflectometer (WCR) systems (CS615 and CS616), and an ECH2O (EC20) were examined experimentally using air-dried packed soil (Andosol) columns with diameters ranging from 70 to 150 mm; surrounded initially by air and then water. Although the output values of all dielectric sensors increased independently of the sample diameters when the samples were surrounded by water, the significances of these increases depended on the sample diameters. The result showed that a sample diameter adequate for the effective sensor sampling area of the TDR100 with the CS605 was  $\geq$ 80 mm. Similarly, appropriate sample diameters were  $\geq$ 150 mm for the TRIME-EZ,  $\geq$ 100 mm for the CS615 and CS616, and  $\geq$ 70 mm for the EC20, respectively. The differences in the appropriate sample diameters among sensors are due to the distribution of the electromagnetic energy of probes linked to their design. The results suggest that approximately 70% of the electromagnetic energy or more must be within the sample diameter if the effective sampling area of the probe is to be included within the sample.

**Discipline:** Soils, fertilizers and plant nutrition **Additional key words:** capacitance sensor, dielectric constant, soil water content, TDR

### Introduction

In recent years, various sensors to measure soil water contents using dielectric properties of soils have been developed. These sensors are based on the fact that the dielectric constant of soils changes predominantly with changes in water content of soils, since the dielectric constant ( $\epsilon$ ) of water is much larger ( $\epsilon = 80.4$  at 20°C) than that of other substances in the soils ( $\epsilon = 1$  for air and  $\epsilon =$ 3-5 for soil particles). Time domain reflectometry (TDR) is used to determine the dielectric constant by analysis of the propagation time of electromagnetic waves, which travel along probe rods embedded in the soil<sup>11</sup>. Water content reflectometer (WCR) measures the oscillation frequency of the reflected rectangular electromagnetic waves traveling along the probe rods, which is a function of the dielectric constant of soils<sup>2</sup>. In the case of the ECH2O sensor, the probe rod and soil act as a capacitor; hence, the ECH2O measures the charge time of the capacitor when it is subject to a specific voltage. The charge time is a function of the probe capacitance, which, in

<sup>3</sup> Department of Bioproduction and Environment Engineering, Tokyo University of Agriculture

(Setagaya, Tokyo, 156-8502, Japan)

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Present address:

<sup>\*</sup>Corresponding author: e-mail s4suzuki@nodai.ac.jp

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turn, is directly related to the dielectric constant of the soil surrounding the probe<sup>7</sup>. These dielectric soil water sensors are now commercially available enabling in-situ, long-term, and multiple monitoring of soil water content.

Since these sensors measure the dielectric constant or relevant dielectric properties of soils dependent on the amount of soil water, a relationship between the output value of the sensors and the soil water content (i.e. a calibration equation) is required to determine the soil water content. However, the sample size used when the calibration equations are determined in a laboratory is of great concern, because the dielectric properties of the materials surrounding soil samples subject to the test might affect the measurement, resulting in a critical error during in-situ monitoring of the soil water dynamics. Ferré et al.4 defined the sensor sampling volume (i.e. the necessary sample size) as the region of the porous medium that contributes to the measurement, namely; "changes in the properties of the porous medium outside this volume have no significant influence on the response of the instrument". To clarify this, Knight<sup>5</sup> and Knight et al.<sup>6</sup> proposed numerical solutions to analyze the spatial sensitivity of TDR probes associated with the two-dimensional electromagnetic energy distribution in the plane perpendicular to the longitudinal axis of the TDR probe rods. Ferré et al.<sup>4</sup> applied these analysis results to determine the sampling area in the plane perpendicular to TDR probes with various designs. Baker and Lascano<sup>1</sup> experimentally visualized a three-dimensional measurement sensitivity distribution around a two-rod TDR probe. These approaches determine the sensor sample size to be the region where a certain proportion of the measurement sensitivity or the electromagnetic energy concentrates around the TDR probe (i.e. the effective sensor sampling area). For example, Baker and Lascano<sup>1</sup> reported that the effective sensor sampling area with minimal loss of accuracy was that included approximately 40% of the measurement sensitivity. Similarly, Petersen et al.<sup>10</sup> reported that the effective sensor sampling area should contain approximately 95% of the electromagnetic energy. However, the distribution of the electromagnetic energy changes greatly depending on the probe designs<sup>4</sup>. Because there is no evidence regarding the effect of changes in the dielectric constant outside the soil sample on the measurement, the ability of these approaches to specify an appropriate sample size for each type of dielectric soil water sensor remains uncertainty.

In the current study, we experimentally examined the effect of changes in the dielectric constant outside the soil samples on the outputs of dielectric soil water sensors, and assessed these experimental results in relationship to the electromagnetic energy concentrated in the samples. The objective of our study was to find the appropriate soil sample size for several commercially available water content sensors, which is particularly important when calibration tests are undertaken in a laboratory. Provided that the soil sample volume is sufficient, measurement values should remain constant, even if the dielectric constant around the sample changes greatly. Therefore, measurements were performed using soil columns surrounded by two different media with very different dielectric constants (air,  $\varepsilon = 1$ , and water,  $\varepsilon = 80.4$ ) after verifying the methodology.

# Materials and methods

TDR100 (Campbell Scientific, Logan, UT, USA) and TRIME-EZ (Imko GmbH, Ettlingen, Germany) were used as the TDR systems. The wave guide used for the TDR100 was a CS605 (Campbell Scientific, Logan, UT, USA). The CS605 consists of three parallel stainless steel rods, each 300 mm long and 4.75 mm in diameter, spaced 22 mm apart. The TRIME-EZ had two rods, 160 mm long and 6 mm in diameter, spaced at 40 mm. The TDR100 with CS605 outputs a dielectric constant, while the result of the TRIME-EZ measurement is a voltage (mV) which is proportional to the dielectric constant. CS615 and CS616 (Campbell Scientific) were used as the WCR, and an EC20 (Decagon Devices, Inc., Pullman, WA, USA) was used as the ECH2O. Both the CS615 and

	TDR100 with CS605	TRIME-EZ	CS615	CS616	ECH2O
Output value and unit	Dielectric constant (-)	Voltage (mV)	Wave period (ms)	Wave period (µs)	Voltage (mV)
Number of rods	3	2	2	2	1
Length (mm)	300	160	300	300	200
Diameter (mm)	4.75	6	3.2	3.2	1(thickness)
Rod spacing (mm)	22	40	32	32	32(width)
	(44 mm from side to side)				

Table 1. Specifications of the soil water content sensors and probes used

Particle size distribution <sup>1</sup> (g kg <sup>-1</sup> )			Texture <sup>1</sup>	$EC_{1:5}^{2}$	Organic
Sand	Silt	Clay		(dS m <sup>-1</sup> )	carbon (g kg <sup>-1</sup> )
432	354	214	Clay Loam	0.09	46.5

 
 Table 2. Selective soil physical and chemical properties of an Andosol (Melanaquand)

<sup>1</sup> International Soil Science Society classification.

<sup>2</sup> Electrical conductivity measured on a 1:5 soil-water solution.

CS616 consist of two parallel stainless steel rods, 300 mm long and 3.2 mm in diameter, with spacing of 32 mm. The CS616 is an improved version of the CS615<sup>3</sup>. The results for both the CS615 and CS616 are reported in terms of the wave period (ms and  $\mu$ s for the CS615 and CS616, respectively), which is the inverse of the oscillation frequency. The EC20 consists of a 32 × 200 mm plate with 1 mm thickness, which outputs a voltage (mV) proportional to the charge time measured. The specifications of the sensors and probes used are summarized in Table 1.

The soil used for the current study was an Andosol collected from the experimental field of the National Agricultural Research Center for the Hokkaido Region, Sapporo, Japan (43° 00' N, 141° 25' E). The soil was classified as a Melanaquand<sup>12</sup>. Selected soil physical and chemical properties are given in Table 2. Since the magnitude of electrical conductivity (EC<sub>1.5</sub>) was 0.09 dS m<sup>-1</sup>, we assumed that the measurements would not be affected by the dissolved solutes in the soil. PVC tubes of 3 mm in thickness and 340 mm in length with inner diameters of 70, 80, 100, and 150 mm were prepared. The dielectric constant of the PVC is 2.8 to 3.113 which is close to that of air. The bottom end of each tube was closed, and the top end was left open. In the current study, we used air-dried soil to facilitate the experiment, for example, eliminating the need to prevent evaporation and enhancing the uniformity of soil packing. The air-dried soil (0.08 kg kg<sup>-1</sup> in mass water content) was sieved with a mesh size of 2 mm and packed into the tubes to a height of 335 mm so that the bulk density of the samples corresponded to  $0.900 \pm$ 0.004 Mg m<sup>-3</sup>.

Soil columns with three replications of each of the different diameters were prepared, and a probe was inserted vertically into each. Each soil column with the probe was then placed into a container ( $640 \times 440$  mm in cross-sectional area and 380 mm in height) (Fig. 1). The sensors were connected to the CR10X (Campbell Scientific, Logan, UT, USA) and measurements were performed at 20°C. Afterward, the container was filled with





A soil column with a dielectric water content sensor probe is surrounded by air (a) and water (b). The diameter of the soil column (D) ranged from 70 to 150 mm. For the measurement using EC20, a soil column with diameter of 55 mm was also incorporated into a set of columns.

distilled deionized water until the water level reached a height equivalent to that of the surface of the soil sample, fully surrounding the PVC tube by water, whereupon the measurements were repeated (Fig. 1). For the measurement using EC20, a soil column of equivalent length with diameter of 55 mm was also incorporated into a set of the columns. The effect of environmental surroundings with different dielectric constants (air vs. water) on the output of each of the soil water sensors was assessed by the ttest, and the standard error of the means was also calculated. The t-test differences were considered significant at P < 0.05.

Among the sensors used in the current study, only the TDR100 with the CS605 outputs a dielectric constant which is quantitatively comparable between different media. Therefore, the methodology adopted in the current study was verified using the TDR100 with the CS605. Namely, the same PVC tubes with diameters of 70, 80, 100, and 150 mm used in the above mentioned measurements were filled with distilled deionized water, and three replications of each of the different diameters were prepared. Each water column was placed into the same container as previously described. The CS605 was vertically inserted into the column, and the dielectric conductivity of the water in the column was measured at a temperature of 20°C (Fig. 2). This test was conducted to evaluate the effect of the dielectric constant outside the columns on the measurements when the materials inside the tubes had high dielectric constant (i.e. water). The effect of differences in the diameter of water columns on the measurement of the dielectric constant was assessed using the analysis of variance (ANOVA) with a signifiS. Suzuki, T. Hirota & Y. Iwata



# Fig. 2. Schematic diagram of the experiment to assess the effect of sample diameter on measurement of the dielectric conductivity of water

The diameter of the soil column (*D*) ranged from 70 to 150 mm.

cant level of P < 0.05. Means were compared by the least significant difference (LSD) test when the *F*-test was significant.

Further, there was concern that the dielectric constant of the air-dried soil used was too low for measurements by the dielectric soil water sensors used in the current study. For TDR100, for example, the propagation time of the electromagnetic wave shortens with decreasing soil water content, hampering analysis of the electromagnetic waves. To evaluate the accuracy of measuring the dielectric constant of air-dried soil, the dielectric constants of the benzene, toluene, diethyl ether, chloroform, and acetic acid contained in glass vessels of 350 mm in length with diameters of 100 mm were measured by the TDR100 with CS605 at a temperature of 25°C. The diameter of the glass vessels (100 mm) was determined based on the results of previously described tests (see Figs. 3 and 5) and the laboratory facilities. The dielectric constants of those solvents are very small compared to water. The dielectric constant of the benzene, toluene, diethyl ether, chloroform, and acetic acid is reported as 2.3 (20°C), 2.4 (25°C), 4.3 (20°C), 4.8 (20°C), and 6.2, respectively<sup>8</sup> (the temperature at which the dielectric constant of the acetic acid was measured is not listed in the literature). By comparing the measured and published data regarding the dielectric constants of those solvents, the applicability of the use of air-dried soil was tested.

### **Results and discussion**

When the soil samples were surrounded by air, there was little difference in the dielectric constants measured by the TDR100 with the CS605 among the samples of various diameters (Fig. 3). When the samples were surrounded by distilled-deionized water, a significant in-

crease in the dielectric constant was observed for the sample diameter of 70 mm (P < 0.05, Fig. 3). Although the dielectric constant of the samples with diameters of 80, 100, and 150 mm also increased with the change in the surrounding medium, the increases were slight and insignificant (P > 0.05 for each diameter, Fig. 3). In addition, the results indicate that the dielectric constant of the packed air-dried samples was 4 (Fig. 3). Further, the dielectric constants of solvents measured by TDR100 with CS605 ranged from 2.3 to 6.2 and remarkably agreed with the literature data (Fig. 4). The coefficient of determination  $(r^2)$  and the root mean square error (RMSE) between the measured and published (i.e. in literature) dielectric constants of the solvents were 0.9998 and 0.04, respectively, thus indicating that the dielectric constant of the air-dried soil was measured accurately. As shown in Fig. 5, the dielectric constant of the water column with diameter of 70 mm was significantly small (P < 0.05,  $\varepsilon$  < 80) compared with other sample diameters, while those of water columns with diameters of 80, 100, or 150 mm were close to 80.4 which is the dielectric constant of water. If the soil sample diameter is sufficiently larger than the effective sampling area of the sensor, changes in the dielectric constant of the surrounding medium should not affect the measurement. For a soil sample diameter of 70 mm, the effective sampling area of the TDR100 with CS605 included some of the surrounding medium, indicating that changes in the dielectric constant of the surrounding medium from 1 (air) to 80.4 (water) resulted in a significant increase in the measurement value (Fig. 3). In the case of soil samples surrounded by air, the small difference in dielectric constant among the soil sample diameters was due to the similarity of the dielectric constants between the air-dried soil sample and the surrounding air (Fig. 3). For the same reason, measurement of the dielectric constant of water in the column of 70 mm diameter resulted in underestimation (Fig. 5). Consequently, the results showed that the area of samples with diameters  $\geq 80$  mm exceeded the effective sampling area of the TDR100 with CS605.

For the other sensors, output values increased for all soil sample diameters when the samples were surrounded by distilled-deionized water (Fig. 6). Since these changes in output values resembled the case of the TDR100 with the CS605 shown in Fig. 3, which accuracy to measure the dielectric constant of the air-dried soil was verified, it is suggested that measurements on the air-dried soil samples performed by the TRIME-EZ, CS615, CS616, and EC20 were also reliable. Further, the significances of the increases of the output values depended on the sample diameters, indicating that the soil sample diameters that contained effective sensor sampling area were  $\geq$ 150 mm





Vertical bars represent standard error of the means. '\*' indicates that there is a significant difference between samples surrounded by air and water at each diameter (P < 0.05). 'ns' represents not significant.



Fig. 4. Comparison of dielectric constants of solvents between data referred to in literature<sup>7</sup> and measured by TDR100 with CS605  $r^2$  and RMSE are the coefficient of determination and root mean square error, respectively.  $\bigcirc$  : Benzene,  $\blacksquare$  : Toluene,  $\blacktriangle$ : Diethyl ether,  $\Box$ : Chloroform,  $\triangle$ : Acetic acid

for the TRIME-EZ, ≥100 mm for the CS615 and CS616, and  $\geq$ 70 mm for the EC20 (Fig. 6a–d). It is noteworthy that the necessary sample size for the TRIME-EZ was approximately twice that for the TDR100 with the CS605 despite similar rod diameters and spacings (Fig. 2 and Ta-



Sample Size of Dielectric Soil Water Sensors



Fig. 5. Changes in the dielectric constant of the deionized distilled water column with diameters of the sample Vertical bars represent standard error of the means. Means denoted with the same letters do not differ significantly at the P = 0.05 level.

ble 1). Ferré et al.<sup>4</sup> demonstrated that the sensor sampling area increases with both diameter and rod spacing and that two-rod probes have a much larger sampling area than tree-rod designs. Therefore, this large difference in the necessary sample size between the TRIME-EZ and TDR100 with CS605 is attributable to the differing number of rods between these probes<sup>4</sup>. Further, the smaller sample sizes required for the CS615 and CS616 compared with the TRIME-EZ were due to differences in the diameter and spacing of the rods<sup>4</sup> (Table 1).

In the case of a two-rod probe placed into a cylindrical medium of given diameter, Knight<sup>5</sup> demonstrated that the ratio of the electromagnetic energy (J) included in the cylindrical medium relative to total electromagnetic energy can be expressed by the following equation:

$$J = 1 - \frac{\ln\left[\left(\rho^{2} + 1 - \beta^{2}\right) / \left(\rho^{2} - 1 + \beta^{2}\right)\right]}{2\ln\left[\beta^{-1} + \left(\beta^{-1} - 1\right)^{\frac{1}{2}}\right]}, \quad \rho = \frac{D}{d}, \qquad \beta = \frac{B}{d}, \qquad (1)$$

where D is the sample diameter (mm), d is the rod diameter (mm), and B is the rod spacing (mm). By applying the d and B values of Table 1 to Eq. (1), J is estimated to be 0.59 for the TRIME-EZ when D = 100, whereas J =0.82 when D = 150. For the CS615 and CS616, J = 0.52when D = 80, and J = 0.70 when D = 100. As described previously, the sample diameters satisfying the effective sensor sampling area were  $\geq$ 150 mm for the TRIME-EZ and  $\geq 100$  mm for the CS615 and CS616. Consequently, the results indicate that approximately 70% or more of

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Vertical bars represent standard error of the mean. '\*' indicates that there is a significant difference between samples surrounded by air and water at each diameter (P < 0.05). 'ns' represents not significant.

the electromagnetic energy must be included within the cylindrical soil sample if the effective sampling area of these two-rod probe sensors is to be included within the sample. Since the effective sensor sampling area of different probe designs depends on the distribution of electromagnetic energy<sup>4</sup>, the results of the current experiment and this assessment of two-rod probe sensors suggest that the sample sizes used for the TDR100 with CS605 and EC20 also need to contain >70% of the electromagnetic energy. The sensor sampling areas are independent of the magnitude of the soil dielectric constant, provided that the dielectric constant is distributed homogeneously around the probe<sup>9</sup>. Figs. 3 and 5 indicate that

the effective sensor sampling area was the same for both cases of the columns filled with air-dried soil ( $\varepsilon = 4$ ) and with water ( $\varepsilon = 80.4$ ). Therefore, it is argued that the results of the current study are independent of soil type and soil water content.

### Conclusion

In the current study, appropriate soil sample sizes for TDR systems (TDR100 with CS605 and TRIME-EZ), WCR systems (CS615 and CS616), and an ECH2O (EC20) were examined experimentally by measuring airdried soil, first when surrounded by air and then by water. The output values of all dielectric sensors increased independent of soil sample diameters when the samples were surrounded by distilled-deionized water. However, statistical analysis (t-test) revealed that the sample diameter containing an effective sensor sampling area was  $\geq$ 80 mm for the TDR100 with CS605. The measurement of the dielectric constants of solvents and the measurement of the dielectric constant of water contained in columns with different diameters verified the results. Similarly, appropriate sample diameters were revealed as  $\geq 150$ mm for the TRIME-EZ, ≥100 mm for the CS615 and CS616, and  $\geq$ 70 mm for the EC20, respectively. We suggest that these soil sample diameters included >70% of the electromagnetic energy of the probes. The differences in sampling area among sensors result from the differences in probe design.

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