21GRD08 SoMMet

D1: Report on the calibration of soil-moisture measurement devices using a primary method, based on evolved water vapour detection, and transfer standards, such as “standardised” loss on drying (LoD) or fibre-coupled spectrometers, to ensure SI-traceable point-scale soil moisture measurements with uncertainties of 5 % under laboratory conditions

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Deliverable **D1** of project 21GRD08 SoMMet

Report on the calibration of soil-moisture measurement devices using a primary method, based on evolved water vapour detection, and transfer standards, such as "standardised" loss on drying (LoD) or fibre-coupled spectrometers, to ensure SI-traceable point-scale soil moisture measurements with uncertainties of 5 % under laboratory conditions

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Glossary

CETIAT: Centre Technique des Industries Aérauliques et Thermiques

CNN: Convolutional Neural Network

CMI: Český Metrologický Institut, Czech Metrology Institute

CNR: Italian National Research Council

DoE: Degree of Equivalence

DTI: Danish Technological Institute

DUT: Device Under Test

EC: Electrical Conductivity

ECV: Essential Climate Variable

EGD: European Green Deal

EWS: Evolved Water Vapour System

EWV: Evolved Water Vapour

FDR: Frequency Domain Reflectometry

fLoD: flow-based Loss-on-Drying

GCOS: Global Climate Observing System

ILC: Interlaboratory Comparison

ISO: International Organization for Standardization

KF: Karl Fischer (Titration)

LoD: Loss-on-Drying

LUCAS: Land Use/Cover Area frame Survey

MFC: Mass Flow Controller

NIST: National Institute of Standards and Technology

PCA: Principal Component Analysis

PLS: Partial Least Squares

PTSS: Proximal Soil Moisture Sensor

RMSE: Root Mean Square Error

RS: Remote Sensing

SM: Soil Moisture

SMF: Soil Moisture Fraction

SoMMet: Soil Moisture Metrology – full name of the project: Metrology for multi-scale monitoring of soil moisture (European Partnership on Metrology 21GRD08)

SSH: Sealed Sample Holder

SSL: Soil Spectral Library

SWC: Soil Water Content

SWIR: Shortwave Infrared

SVM: Support Vector Machine

TS: Transfer Standard

TDR: Time Domain Reflectometry

TUBITAK or TÜBİTAK: Türkiye Bilimsel ve Teknolojik Araştırma Kurumu, the Scientific and Technological Research Council of Türkiye

UNIBO: University of Bologna

UP DE: University of Potsdam, Germany

UV-Vis: Ultraviolet–Visible (Spectroscopy)

VIM: International Vocabulary of Metrology

Vis-NIR: Visible–Near Infrared

VWC: Volumetric Water Content

WMF: Water Mass Fraction

WMR: Water Mass Ratio

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1 Introduction

This document, deliverable D1, is the first of eight technical deliverables of the project 21GRD08 SoMMet (hereafter: SoMMet). The project has received funding from the European Partnership on Metrology, co-financed by the European Union's Horizon Europe Research and Innovation Programme and by the Participating States (Funder name: European Partnership on Metrology; Funder ID: 10.13039/100019599; Grant number: 21GRD08 SoMMet).

The aim is to outline the principles and methods for the calibration of soil moisture measurement devices. Soil moisture measurement devices, such as point-scale sensors, are attractive for several reasons, including ease of use, continuous monitoring, and relatively low prices. However, calibration is required to provide traceable, and thus reliable, measurements. This document describes several ways to ensure SI-traceable point-scale soil moisture measurements, including the use of a primary method based on evolved water vapour detection, and transfer standards, such as standardized Loss-on-Drying (LoD) or fibre-coupled spectrometers.

Soil moisture (SM) is one of the Essential Climate Variables (ECVs) as defined by the Global Climate Observing System (GCOS) of the World Meteorological Organization (WMO) [1]. Soil is a cross-cutting theme within the European Green Deal (EGD), with relevance to many inherently interdependent sectors such as water management, agriculture, forestry, and biodiversity. Soil moisture measurements with point scale sensors are performed by practical users in agriculture and hydrology (e.g., farmers and agronomists) or by scientists dealing with SM, for example in the calibration of cosmic-ray neutron sensing (CRNS) probes.

Soil moisture on a very local scale (10^{-1} m) is routinely measured using point scale sensors. Such a sensor is inserted into the soil and commonly measures the dielectric constant (ϵ) of its surroundings as a proxy for the water content of the soil. This technique works on the basis that, of the three major constituents of soil, two of them, i.e. soil minerals ($\epsilon < 10$) and air ($\epsilon \approx 1$), display a small value of ϵ, whereas the third, water, displays $\epsilon \approx 80$, and the relative dielectric constant of the soil as a medium is therefore largely determined by the water content of the soil.

A central focus of the work on SI-traceability of point-scale soil moisture sensors in the project SoMMet ("Metrology for multi-scale monitoring of soil moisture", European Partnership on Metrology 21GRD08) has been the establishment of calibration facilities in metrology laboratories for the sensors. An important aspect is the ability to define a body of soil with a normative value of volumetric water content to immerse the sensor in the soil and obtain an indication from it that may then be compared with a reference value. Specifically, the measurement uncertainty associated with soil moisture indications from point scale sensors has previously been highlighted as an important topic for investigation [2], and it impacts the uncertainty in derived parameters that are important for decision-making in agricultural water management [3].

1.1 Outline of the report

This report describes the efforts made in the SoMMet project towards providing metrological traceability for point-scale soil moisture sensors. The definitions that are important for establishing SI-traceability and the standards that support development of a metrological measurement

infrastructure are described in Section 2. In Section 3, the primary level of water content measurements, namely the instruments that realize an amount of water with specific detection of water, is described. Furthermore, uncertainty budgets for these instruments are presented, as well as an interlaboratory comparison that was conducted for gravimetric water content on a wet basis within the SoMMet project between several primary measurement standards. In Section 4, two secondary measurement standards—one based on Loss-on-Drying, and one based on UV-Vis spectroscopy—for the amount of water in soil are described, and their validation against the primary measurement standard from Section 3 is presented. In Section 5, a general procedure for point-scale soil moisture sensor calibration and an uncertainty budget based on this procedure is presented. Finally, in Section 6, some results of calibrating point-scale soil moisture sensors, along with an investigation of some factors that contribute to the measurement uncertainty, are presented.

2 Definitions and traceability

2.1 Definitions

The term “soil moisture” is an ambiguous term, which in many cases refers specifically to the amount of water that is in a certain amount of soil, i.e. the soil water content (SWC). Commonly, point-scale soil moisture sensors read out an indication of the volumetric water content (VWC, θ_{V}), which is the ratio between the volume of water (V_{water}) in a body of soil and the wet volume of that soil ($V_{\mathrm{soil,wet}}$), which is the volume occupied by the undisturbed wet soil. The result is expressed as a percentage in the dimensionless unit ($m^3\ m^{-3}$), which is calculated as

$$\theta_{\mathrm{V}} = \frac{V_{water}}{V_{soil,wet}} \times 100\ \%. \tag{1}$$

VWC may therefore also be called the volume fraction of water in the soil. A standard for determination of VWC is defined in ISO 11461 [4]. This quantity is also commonly reconstructed from CRNS and has been adopted as a standard unit for soil moisture of the community surrounding remote sensing (RS) [5].

Alternatively, the SWC may be expressed in terms of the mass of the water, m_{water}. One option is as the ratio of water *mass* to the *volume* of the wet soil,

$$\theta_v = \frac{m_{water}}{V_{soil,wet}}, \tag{2}$$

which, although it is not dimensionless, is a natural definition when a known volume of soil is dried, and the mass of water is determined either indirectly using the gravimetric technique of Loss-on-Drying, or directly with a metrologically traceable measurement of water mass determination such as those presented later in the text. Another option again is that the amount of water in the soil is expressed as the ratio of water *mass* compared to the dry soil *mass*,

$$\theta_{m,dry} = \frac{m_{water}}{m_{soil,dry}} \times 100\ \%. \tag{3}$$

This definition is natural if it is the masses of both the soil and the water that are measured, and it may be more straightforward to apply due to the challenge of defining soil volume. $\theta_{m,dry}$ is also called the *water mass ratio* (WMR). Alternatively, the *water mass fraction*, $\theta_{m,wet}$ (WMF) is defined as the ratio of the water mass to the wet mass of the soil as

$$\theta_{m,wet} = \frac{m_{water}}{m_{soil,wet}} \times 100\ \%. \tag{4}$$

Analogously to Equation (3) and Equation (4), it is useful to define the quantities Q_{dry}, the *soil moisture ratio*, and Q_{wet}, the *soil moisture fraction*. These quantities are important for the LoD technique that establishes a mass loss in a sample by drying the sample, and where it may be said that the mass loss is due to the evaporation of *moisture,* but it is not specifically shown that the mass difference before and after drying is due only to evaporated water. The moisture mass, $m_{moisture} = m_{wet} - m_{dry}$, is established as the mass difference between the initial, wet sample and the dried sample.

$$Q_{dry} = \frac{m_{moisture}}{m_{soil,dry}} \times 100\ \%, \tag{5}$$

$$Q_{wet} = \frac{m_{moisture}}{m_{soil,wet}} \times 100\ \%, \tag{6}$$

The quantities θ_V and $\theta_{m,dry}$ are related by the bulk density of the soil, ρ_b, and the density of water at a specified temperature and pressure, such that

$$\theta_{m,dry} = \frac{\rho_{water|T,\sigma}}{\rho_b}\theta_V, \tag{7}$$

where $\rho_{water|T,\sigma}$ is the water density at the given temperature (T) and salinity (σ), and ρ_b is the bulk density of the soil. ρ_b is the ratio between the dry mass and wet volume of the soil, and is calculated as

$$\rho_b = \frac{m_{soil,dry}}{V_{soil,wet}}. \tag{8}$$

Another measure of the compactness of the soil is the soil porosity, P, which is given by the ratio of the difference between the total volume (V_{total}) and solid volume (V_s) to the total volume

$$P = \frac{V_{total} - V_s}{V_{total}} = 1 - \frac{V_s}{V_{total}}. \tag{9}$$

The soil porosity thus measures the available space in the soil as a proportion of the total volume that the soil occupies, and the soil porosity and bulk density are related by

$$\rho_b = \rho_s(1 - P), \tag{10}$$

where ρ_s is the grain density of the solids in the soil.

2.2 Metrological traceability

The metrological traceability of a measurement result is paramount in SI-traceable calibration. The definition of "metrological traceability" of a measurement result is given in the International Vocabulary of Metrology (VIM, [6]). It applies to a measurement result which can be either metrologically traceable or not metrologically traceable. If it is the former, it means that the measurement provides a value that is known accurately with respect to the definition of the measurand that is being measured. Specifically, it means that the instrument used to perform a measurement must be calibrated with respect to a reference showing a smaller measurement uncertainty that itself may either be a primary standard, which is an expression of the highest level of measurement accuracy, or another instrument that has been calibrated. If such an unbroken chain of calibrations exists between a measurement and a definition, the measurement is said to be "metrologically traceable".

In the case of volumetric soil moisture, the definition is given by Equation (1). It means that to provide metrological traceability to volumetric water content, both the measurement of the amount of water and the measurement of the volume of soil need to be metrologically traceable.

2.2.1 Soil volume

A known volume of soil can be obtained, either by extracting soil with a sampling device with a known volume, or by measuring the empty volume left behind after removing the soil. However, soil volume cannot straightforwardly be measured with small uncertainty. Several methods do exist and are outlined below together with some of the related complications. However, a detailed investigation of the different methods and their uncertainties is not within the scope of this document but is instead deferred for future investigations.

A device with a known volume may be, for example, the *soil core ring*, which is a cylindrical apparatus with known dimensions that is pushed or hammered into the ground. The cylinder, thus filled with soil, is then extracted. A detailed description of a procedure using this instrument can be found in, for example, the European Union Land Use/Cover Area frame Survey (LUCAS) document "Sampling Instructions for Surveyors" [7]. This method, however, by being invasive, may itself change the density of the soil in the vicinity of the ring walls. This means that although the internal dimensions of the ring may be calibrated by comparison to a metrologically traceable length reference, the internal volume of the ring may not correspond to the volume that the soil within the ring would have if left undisturbed.

Other methods of obtaining the volume may be called *negative space* methods, where the space left behind after extraction of the soil is filled with a material of which the volume can easily be determined. Water and polyurethane foam may for example be used [8]. With water, a plastic lining is put into the hole, and the hole is then filled with a known amount of water. With polyurethane, the compound is left to set and fill the hole, and the hardened compound is taken away for a separate volume determination [9].

The *soil core ring* and *negative space* methods are standardized in ISO 11272 (ISO, 2017).

2.2.2 Amount of water

The metrological measurement infrastructure for the amount of water is well-established. Measurement of the amount of water in different matrices has been the focus of several previous projects under the EURAMET Metrology Partnership, including METefnet (EMRP SIB64 METefnet) [10] and BIOFMET (19ENG09 BIOFMET) [11]. The establishment of amount of water in soil under SoMMet has been built on the advances made in those projects by employing both the primary measurement standards that have been previously used, as well as newly implemented primary measurement standards. To validate these for the use of SWC determination, an interlaboratory comparison (Section 3.4) was conducted between the primary measurement standards. In addition to this, two new transfer standards have been built and validated for SWC using the results obtained with the primary measurement standards.

3 Primary measurement standards

Primary measurement standards realize a measurand without needing specific calibration with respect to that measurand [6]. Within the scope of SoMMet, primary measurement standards for measurement of the amount of water have been developed and adapted for measurements on soils.

3.1 Evolved Water Vapour using dew point – TUBITAK implementation

The instrument, an Evolved Water Vapour System (EWS), is based on measurement of the amount of water by evaporating all the water in a sample. In this configuration, as shown in Figure 1, dry gas (air or nitrogen) is selected via a Swagelok-type valve (V1) and directed to the inlet measurement block, then to a hermetically sealed sample holder (SSH) placed inside a temperature-controlled oven (Thermomac FDO30). The sample temperature can be adjusted from ambient up to 300 °C. The gas flow rate is precisely regulated using a mass flow controller (MFC2; Bronkhorst EL-Flow Select).

The humidified gas exiting the SSH passes through an outlet measurement block maintained at 65 °C to prevent condensation. Relative humidity and dew/frost point temperature are measured by a chilled mirror hygrometer (MBW 373-H, Switzerland) with SI-traceable calibration. This instrument operates within a dew/frost point temperature range of -40 °C to +60 °C and includes an automatic mirror heating function (every 30 min) to avoid contamination. All connecting lines between the SSH and the hygrometer are thermally regulated at 65 °C to prevent condensation.

Temperature is monitored using six PT100 resistance thermometers (Metronik, Türkiye), while gas pressure is measured by Sensotec pressure transducers. The humidity and temperature of the incoming gas are monitored using a Vaisala HMT315 hygrometer. System operation and data acquisition are performed via in-house software developed in the LabVIEW environment, which records all signals (from Vaisala, MFC2, MBW, and other measurements) and calculates the total water mass removed from the sample, including uncertainty analysis.

This configuration enables accurate and repeatable water content measurements under controlled conditions of temperature, gas flow, and pressure, without the need for a sample conditioning (humidification) subsystem.

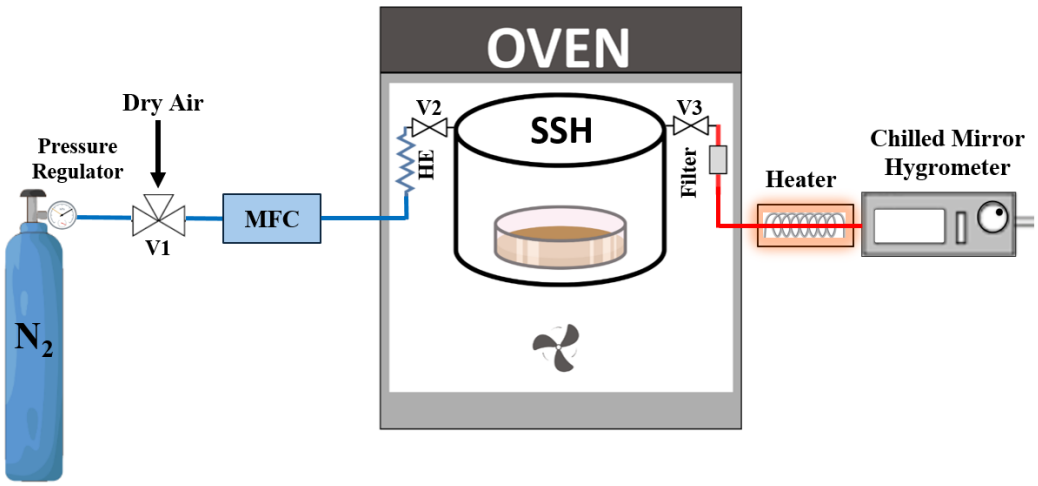


Figure 1. Block diagram of the developed Evolved Water Vapour System (EWS).

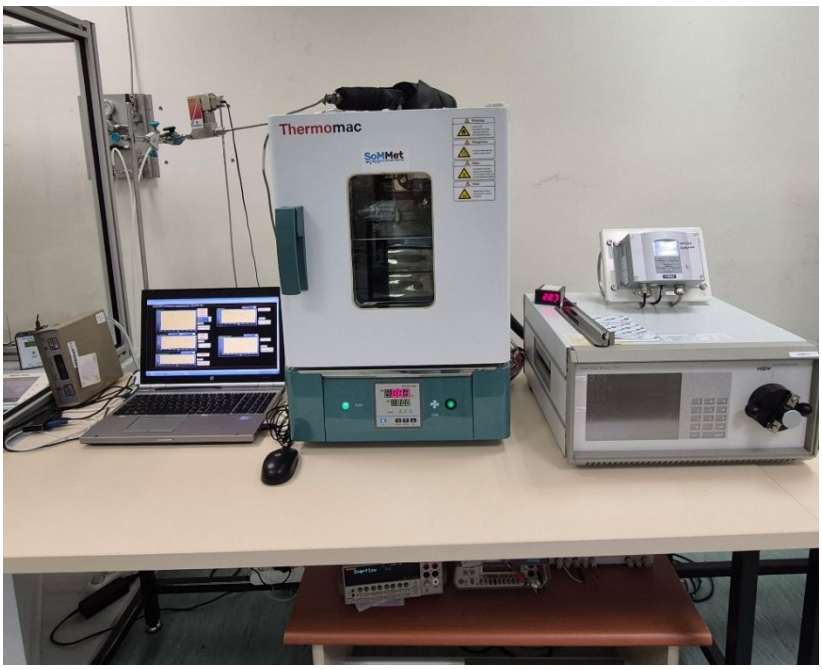


Figure 2. Primary-level Evolved Water Vapour System (EWS) developed by TUBITAK.

Figure 2 shows the developed primary-level evolved water vapour system. During EWS experiments, high-purity nitrogen gas (99.999 %), supplied by the local provider Habaş (Türkiye), was utilized. The dryness level of each nitrogen cylinder was verified using another chilled-mirror hygrometer device, model MBW 373-HXLX, capable of operating within a dew/frost point range of -95 °C to 95 °C. Typically, the frost point readings varied between -69 °C and -71 °C depending on the cylinder.

Prior to initiating measurements, the entire gas pathway of the system was conditioned to reach a frost point of -38 °C, while maintaining the SSH at a temperature of 105 °C.

3.1.1 Flow-based Loss-on-Drying (fLoD)

The working principle of the EWS consists of determining a sample's total water content by drying it with a dry gas flow at elevated temperatures. Once the process is complete, the sample is considered fully dried, and the reduction in mass corresponds to its moisture content. This concept closely aligns with the traditional Loss-on-Drying (LoD) method outlined in Section 4.1. The main distinction is that EWS performs drying in a single cycle, whereas the conventional LoD method requires multiple cycles of drying and weighing until the dry weight stabilizes. Additionally, the LoD method relies on natural air convection or vacuum for moisture removal, while EWS uses a directed gas flow. In this project, this approach is referred to as "flow-based Loss-on-Drying (fLoD)" to differentiate it from the traditional LoD technique.

The SSH has an empty mass of about 2800 g, which makes it impractical for direct use in fLoD measurements, as precision analytical balances cannot detect the small weight changes with sufficient accuracy. To overcome this limitation, smaller glass containers such as Petri dishes, wide-mouth jars, or laboratory vials can be placed inside the SSH to hold the sample. During SoMMet, pre-dried Petri dishes were mainly utilized. The empty mass of a Petri dish m_{epd}, along with the masses of the wet sample in the Petri dish, M_{wet}, and the dried sample in the Petri dish, M_{dry}, were measured accurately using an analytical balance. By establishing $m_{soil,wet} = M_{wet} - m_{epd}$ and $m_{soil,dry} = M_{dry} - m_{epd}$ as well as $m_{moisture} = M_{wet} - M_{dry}$, the soil moisture ratio and soil moisture fraction may be calculated using the standard formulas Equation (5) and Equation (6).

3.1.2 Validation

The system was optimized, validated, and verified using double-distilled water and an inorganic reference salt material chosen for its lack of organic volatile components [12]. The verification experiments were done at 105 °C. Salt samples were dried, hydrated with known water quantities, and stored at 4 °C before undergoing EWS measurements with controlled temperature increases. Results from both EWS and fLoD methods shown in Table 1 are in strong agreement, confirming accurate water retrieval. Additional tests with double-distilled water in Petri dishes shown in Table 2 further validate the system's repeatability and resolution, with dew-point tracking confirming complete evaporation. Across all experiments, post-drying weight checks indicated negligible residual moisture, supporting the reliability of the EWS method.

Table 1. Results of experiments with the salt samples.

Dry Mass (g)	Sprayed Water (g)	EWS Results (g)	WC (%)	U_{EWS} (k=1) (%)	fLOD result (%)	U_{fLoD} (k=2) (%)	fLoD – WC (%)
47.524	2.8	2.757	5.48	0.16	5.49	0.08	0.01
47.593	3	3.019	5.97	0.16	6.01	0.07	0.04
46.85	3.8	3.699	7.30	0.16	7.39	0.07	0.09
46.442	5	4.901	9.53	0.17	9.51	0.07	-0.02

System validation was performed using salt samples and double-distilled water, which contain only water as a volatile component. These tests showed excellent agreement between the added and measured water, with discrepancies below 0.1 %. Finally, the EWS system is used for the SI-traceable calibration of a Vis-NIR transfer standard spectrometer designed for surface soil water content measurement. The results of this calibration study will be reported in an upcoming publication.

Table 2. Results of experiments using distilled water.

Nominal Amount of Water (g)	Weighted Amount of Water (g)	U_{weigh} (k=1) (g)	EWS Results (g)	U_{EWS} (k=1) (g)	Difference (Weight - EWS) (g)
1	0.914	0.001	0.924	0.007	–0.002
4	3.964	0.001	4.047	0.029	–0.009
7	6.804	0.002	6.955	0.051	0.011
10	9.783	0.002	10.005	0.071	–0.004

3.1.3 Uncertainty Analysis

Table 3. Typical uncertainty budget for EWS measurements in a single point.

Parameter	Nominal Value	SI-Traceable Calibration Uncertainty	Sensitivity Coefficients	Standard Uncertainty
Flow rate	20 L h^{-1}	0.1 L h^{-1}	13.245 µg (L h^{-1})$^{-1}$	1.324 µg
P_1	110000 Pa	26 Pa	2.408 × 10^{-3} µg Pa^{-1}	0.063 µg
T_1	293.15 K	0.050 K	0.904 µg K^{-1}	0.045 µg
P_{atm}	101325 Pa	30 Pa	2.752·10^{-3} µg Pa^{-1}	0.083 µg
Dew Point	308.15 K	0.070 K	13.929 µg K^{-1}	0.975 µg
			Combined uncertainty (k=1)	**1.65 µg**
			Expanded uncertainty (k=2)	**3.30 µg**

Table 4. Typical uncertainty budget for EWS measurements in assessing WMF, the water content of the soil as a fraction of the wet mass of the soil used.

Parameters	Value (g)	Uncertainty (g)	Sensitivity Coefficient	Standard Uncertainty (%WC)
m_W	**2.640**	**0.075**	**1.510 %mc g^{-1}**	**0.113**
Gas Flow		0.060	1.000 g g^{-1}	
SSH Gas Pressure		0.003	1.000 g g^{-1}	
Inlet Dry Gas Temperature		0.002	1.000 g g^{-1}	
Atmospheric Pressure		0.004	1.000 g g^{-1}	
Dew/Frost Point		0.045	1.000 g g^{-1}	
M_{wet}	**104.586**	**0.056**	**0.276 %mc g^{-1}**	**0.015**
Certificate		0.002	1.000 g g^{-1}	
Dehydration		0.053	0.577 g g^{-1}	
Repeatability		0.018	1.000 g g^{-1}	
m_{epd}	**50.792**	**0.007**	**0.276 %mc g^{-1}**	**0.002**
Certificate		0.001	1.000 g g^{-1}	
Repeatability		0.007	1.000 g g^{-1}	
		Combined uncertainty (k=1)		**0.114**
		Expanded uncertainty (k=2)		**0.23**

Table 5. Typical uncertainty budget for fLoD measurements in assessing SMF, the moisture content as a fraction of the wet mass of the soil used.

Parameters	Value	Uncertainty (g)	Sensitivity Coefficient	Standard Uncertainty (%MC)
M_{wet}	**117.012**	**0.056**	**1.227 %mc g^{-1}**	**0.069**
Certificate		0.002	1.000 g g^{-1}	
Dehydration		0.053	0.577 g g^{-1}	
Repeatability		0.018	1.000 g g^{-1}	
M_{dry}	**104.586**	**0.020**	**1.510 %mc g^{-1}**	**0.030**
Certificate		0.002	1.000 g g^{-1}	
Rehydration		0.011	0.577 g g^{-1}	
Repeatability		0.016	1.000 g g^{-1}	
m_{epd}	**50.792**	**0.007**	**0.283 %mc g^{-1}**	**0.002**
Certificate		0.001	1.000 g g^{-1}	
Repeatability		0.007	1.000 g g^{-1}	
		Combined uncertainty (k=1)		**0.075**
		Expanded uncertainty (k=2)		**0.15**

3.2 Evolved Water Vapour using dew point – DTI implementation

The primary measurement standard for water content measurement at DTI has previously been described in detail in P. Østergaard and J. Nielsen (2018) [13]. Since that initial implementation, some changes have been made to the system. Most notably, the carrier gas in the current setup is dry air, which is dried to a dew point temperature below -50 °C before being used in the experiment. The measurement principle is the same as the EWS system at TUBITAK described in the preceding section. A schematic of the setup is shown in Figure 3.

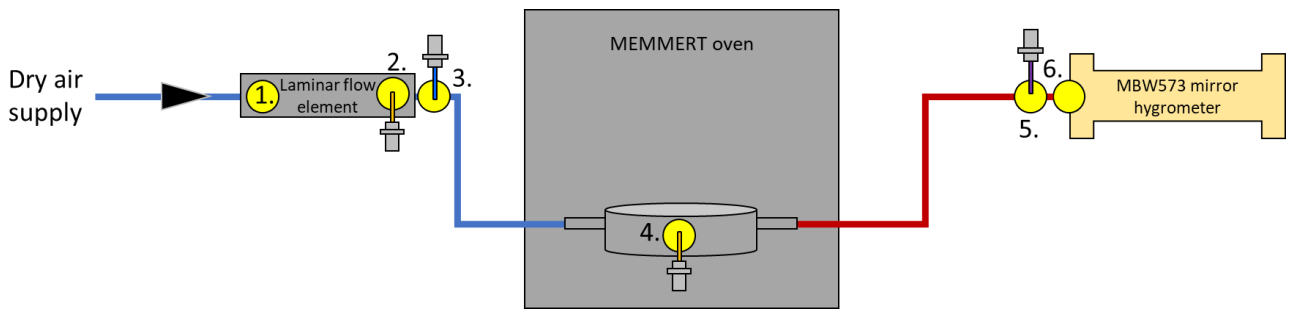


Figure 3. Schematic of the primary measurement standard at DTI. Cold (blue) and warm (red) gas lines are indicated in the figure. During the experiment, data is collected for the ingoing flow of carrier gas (1), the temperature of the laminar flow element (2), the gauge pressure at the laminar flow element (3), the temperature in the sample chamber (4), the humidity (5), and the dew point (6) of the carrier gas after interaction with the sample as well as the ambient temperature and ambient pressure (not shown).

3.2.1 Measurement uncertainty

The uncertainty budget of the DTI primary standard is given in Table 6 for determining water mass fraction. The contributions have been sorted according to the magnitude of the contribution.

Table 6. Uncertainty budget for water content determination with the DTI primary standard.

Description	Estimate	Standard Uncertainty	Unit	Sensitivity coefficient	Contribution, 10^{-2} g g^{-1}
Dew point temperature	50	0.09	°C	$1.5 \cdot 10^{-2}$	$1.4 \cdot 10^{-1}$
Laminar flow element, difference pressure	42	0.15	Pa	$6.5 \cdot 10^{-3}$	$9.5 \cdot 10^{-2}$
Weighing, wet	5600	0.2	g	$-6.8 \cdot 10^{-4}$	$-1.4 \cdot 10^{-2}$
Weighing, empty	5200	0.2	g	$6.8 \cdot 10^{-4}$	$1.4 \cdot 10^{-2}$
Ambient pressure	102000	100	Pa	$-7.5 \cdot 10^{-7}$	$-7.5 \cdot 10^{-3}$
Gauge pressure	8000	13.40	Pa	$4.9 \cdot 10^{-6}$	$6.6 \cdot 10^{-3}$
Temperature of laminar flow element	23	0.031	°C	$-1.6 \cdot 10^{-3}$	$-5.1 \cdot 10^{-3}$
Temperature, ambient	23	0.026	°C	$2.4 \cdot 10^{-5}$	$6.4 \cdot 10^{-5}$
Temperature in sample chamber	80	0.026	°C	0	0
				Combined uncertainty (k=1), 10^{-2} g g^{-1}	0.17
				Expanded uncertainty (k=2), 10^{-2} g g^{-1}	0.34

3.3 Evolved water vapour (EWV) using KF (CMI)

The volumetric Karl-Fischer V30 titrator with Stromboli oven (Mettler Toledo) was used to determine the water content in soil samples. This method is based on the chemical reaction of released water from the sample and iodine from the titrant. The soil sample (0.1 g – 2 g) is placed into the vial which is placed into the Stromboli oven automatic sample holder. Each vial is warmed to 150 °C for at least 10 min. Released vapours are transported to a titration cell using nitrogen gas and tempered transfer line. The released water immediately reacts with the titration reagent according to the reaction $H_2O + I_2 + SO_2 + 3\ C_5H_5N \rightarrow 2\ C_5H_5NH^+I^- + C_5H_5N \cdot SO_3$, and the device quantifies the water content with an accuracy of 0.01% by weight based on the volume of titration solution consumed.

The automated V30 system allows continuous dosing of the titrant until the water is completely depleted, ensuring high reproducibility and minimal influence from organic interferences. The total analysis time is typically 5-15 minutes per sample, which, together with excellent sensitivity, makes the method ideal for accurate moisture determination in both small and large volumes of soil samples.

3.4 Interlaboratory comparison of gravimetric soil moisture

An interlaboratory comparison (ILC) was conducted with 4 participants to investigate the agreement between the primary measurement standards of the 4 laboratories about soil water content. The full report including all calculations will be available from EURAMET [14], and a brief recapitulation of the results will be given here.

The pilot study was targeting the measurement of gravimetric water content as in Equation (4) and was conducted on samples of both organic and inorganic soil. The four participants in the comparison were the Danish Technological Institute (DTI, Denmark, pilot), Centre Technique des Industries Aérauliques et Thermiques (CETIAT, France), Český Metrologický Institut (CMI, Czech Republic), and Türkiye Bilimsel ve Teknolojik Araştırma Kurumu, Ulusal Metroloji Enstitüsü (TUBITAK-UME, Türkiye).

To perform the comparison, a batch of soil for each soil type was prepared according to the procedure in Section 5.2.1 and divided into samples that were packed individually in sealed aluminium bags. The bags were kept cold until ready to measure and were distributed amongst the participating laboratories. From each of the participating partners, 3 samples of each soil type were returned to the pilot laboratory for stability analysis.

Homogeneity and stability of the water content was assessed by the pilot laboratory using Loss-on-Drying (Section 4.1) and is shown in Table 7. The assessments confirmed that the sample materials were suitable for interlaboratory comparison, with very low standard deviations (≤ 0.15 % w/w). The reported measurement results from the participating laboratories for both soil types are shown in Figure 4 and Figure 5.

Table 7. Homogeneity and stability of the WMF of the soil samples used in the interlaboratory comparison.

	Organic soil	**Inorganic soil**
Homogeneity, 10^{-2} g g^{-1}	0.03	0.15
Stability, 10^{-2} g g^{-1}	0.02	0.05

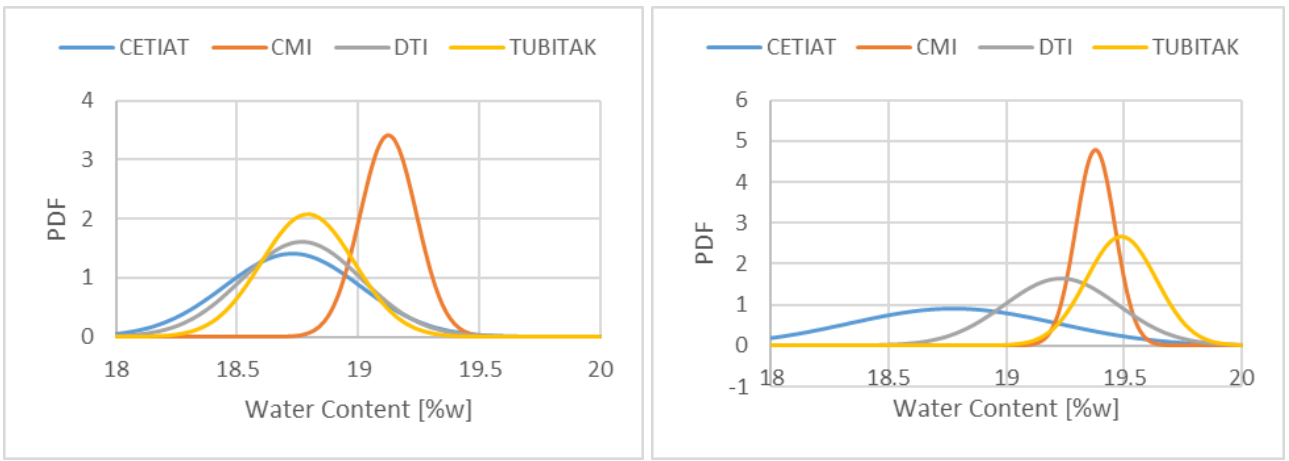


Figure 4. Probability density functions (PDF) of the reported results in the interlaboratory comparison. Left: PDF for organic soil. Right: PDF for inorganic soil.

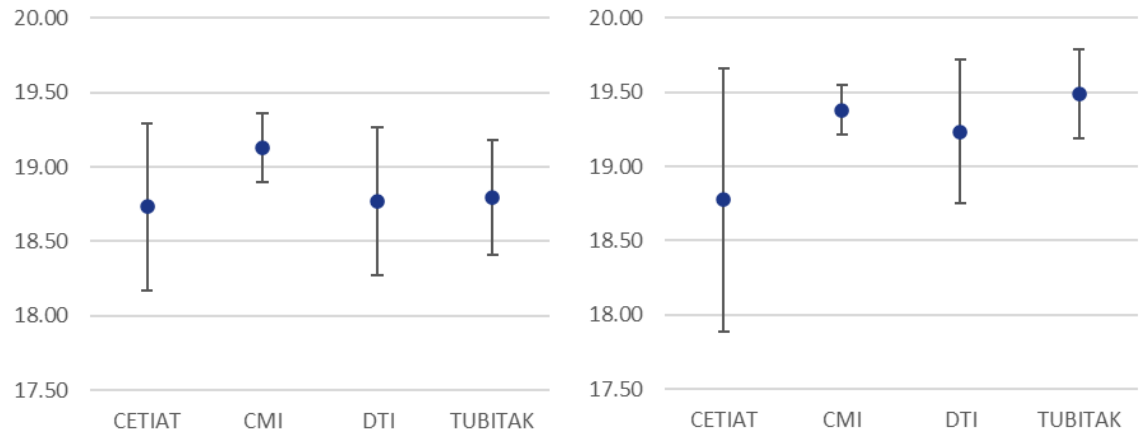


Figure 5. Reported mean values and expanded uncertainties for organic soil (left) and inorganic soil (right).

Based on these results, bilateral degree of equivalence (DoE) showed that there are no significant differences between any pair of participants for either of the soil types. Three different ways of calculating a consensus value (weighted mean, mean, and median) and a corresponding E_n-value for each laboratory revealed that for inorganic soil all laboratories were consistent with each other for all reference values. For organic soil all laboratories were also consistent with each other, with the exception that one laboratory displayed a significant deviation ($E_n > 1$) in the analysis based on the median as a consensus value.

3.4.1 Conclusions from the interlaboratory comparison

The interlaboratory comparison confirmed that samples can be prepared with a high degree of stability and homogeneity (standard deviations < 0.15 % g g^{-1}) and showed no differences between any pairs of laboratories based on DoE. Differences between the methods of calculating a reference value should be interpreted with caution due to the low number of participants. Future comparisons may benefit from a higher number of participants, and future work may include a more detailed analysis of participants' uncertainty budgets and development of a guideline on how to develop the uncertainty analysis.

4 Secondary measurement standards and techniques

4.1 Loss-on-Drying (LoD)

The LoD method is a straightforward technique for gravimetrically measuring the moisture content of solids and is extensively documented across various materials and applications. The method is also known as *the gravimetric method*. This method is standardized for soil in ISO 11465 [15]. The process consists of three primary steps: first, weighing the wet soil sample to obtain the mass of the wet soil sample, m_{wet}; second, drying the sample to eliminate moisture; and finally, weighing the dried soil to obtain the mass of the dry soil, m_{dry}. The moisture content may then be calculated according to Equations (5) and (6).

4.2 Transfer standard based on fLoD

DTI has built and validated a transfer standard based on LoD, which works on the fLoD-principle described in Section 3.1.1. The system is shown in Figure 6. The DTI transfer standard consists, first, of a compressor with a fitted membrane drier. This system supplies a flow of dry air to a sample chamber, into which the sample with an unknown water content is placed. The dry air, in combination with raised temperature, removes water from the sample. The experiment continues while there is still water in the sample, as determined by the humidity of the air leaving the sample. When the humidity drops below a preset threshold, the experiment is stopped. The mass difference between the sample before and the sample after the experiment, i.e. the Loss-on-Drying, is the mass of water in the original sample. The use of a flow of dry air permits drying the sample at a lower temperature, which reduces the risk of thermal degradation of the sample. The system and its intended use in on-site calibration of point-scale soil moisture sensors have previously been described in detail [16].

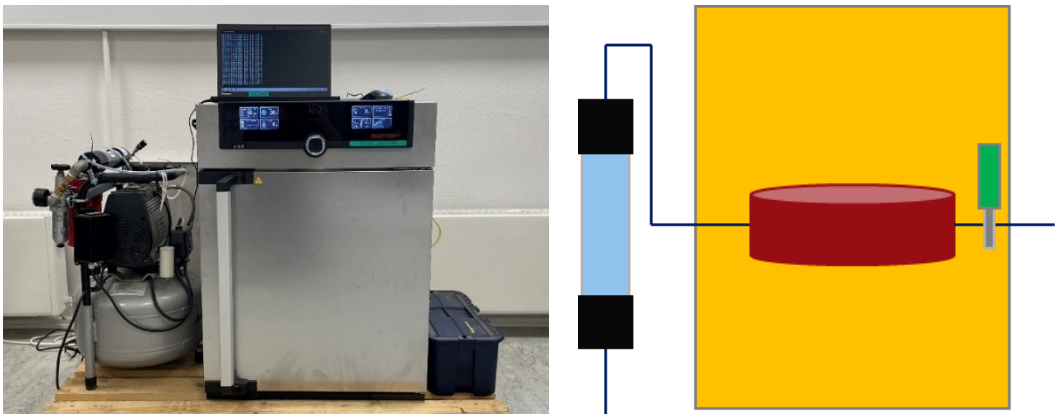


Figure 6. The DTI transfer standard based on Loss-on-Drying. Left: Picture of the final implementation of the setup. Right: Schematic of the setup. Compressed air flowing from left to right moves through a membrane drier (light blue) into a heated oven and sample chamber (orange, red). The humidity in the gas line is monitored with a thermohygrometer (green) to automatically end the experiment when the humidity drops below a threshold, indicating that all water has evaporated from the sample.

4.2.1 Validation of the DTI transfer standard

The fLoD-based transfer standard was validated by comparison to the DTI primary standard using soil samples from the same batch as those used for the interlaboratory comparison described in Section 3.4. The DTI transfer standard was validated on both organic and inorganic soil samples. The measurement results from the transfer standard are given as soil moisture fraction defined as in Equation (6). The measurement results are shown in Table 8 and Table 9. The given uncertainties are based on weighing of the empty sample chamber, the wet sample, and the dry sample.

Table 8. Measurement results obtained on organic soil samples with the DTI transfer standard for the validation of the transfer standard. “Result” is the soil moisture fraction with the corresponding uncertainty “u”.

Sample ID	Result, 10^{-2} g g^{-1}	u (k=1), 10^{-2} g g^{-1}
107	19.10	0.03
121	18.88	0.03
128	19.22	0.03
135	18.94	0.03
149	19.04	0.03
156	18.89	0.03
170	19.23	0.03

Table 9. Measurement results obtained on inorganic soil samples with the DTI transfer standard for the validation of the transfer standard. “Result” is the soil moisture fraction with the corresponding uncertainty “u”.

Sample ID	Result, 10^{-2} g g^{-1}	u (k=1), 10^{-2} g g^{-1}
7	19.39	0.03
14	19.45	0.03
21	19.52	0.03
28	19.35	0.03
35	19.44	0.03
56	19.48	0.03
70	19.32	0.03

These results were used to validate the transfer standard for measurement of water mass fraction on soils according to the procedure outlined in ILAC G8: 2019 “Guidelines on Decision Rules and Statements of Conformity” [17]. The combined result for the transfer standard was obtained by taking the average of the individual measurements to give Q_{wet}, and the corresponding uncertainty is given by the combination of the standard deviation of the mean and the average method uncertainty. The reference value was realized using the DTI primary standard described in Section 3.2. The calculation results are given in Table 10. The validation results are shown in Figure 7.

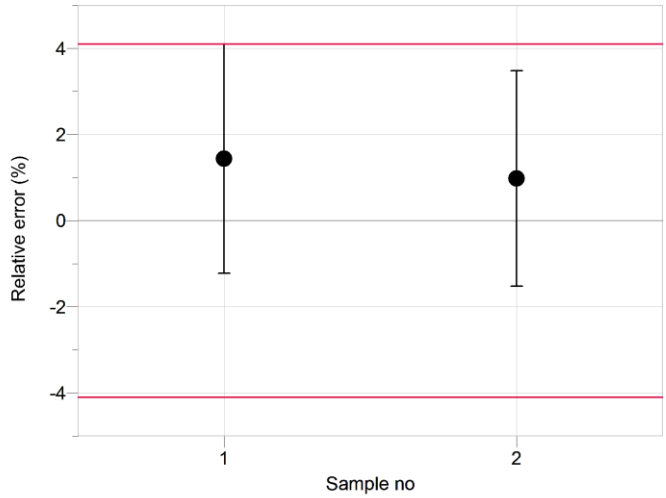


Figure 7. Error observed for organic (sample no. 1) and inorganic (sample no. 2) soil types (black points and error bars). For comparison, the claimed uncertainty of the transfer standard is indicated (red lines).

Table 10. Summary of the data and calculations used in the validation of the LoD based transfer standard.

No.	Parameter	Value, organic soil	Value, inorganic soil	Remarks
1	Q_{wet}, 10^{-2} g g^{-1}	19.04	19.42	TS*
2	Uncertainty of Q_{wet}, 10^{-2} g g^{-1}	0.04	0.03	TS*, k = 1
3	$\theta_{m,wet}$, 10^{-2} g g^{-1}	18.77	19.23	Reference*
4	Uncertainty of $\theta_{m,wet}$, 10^{-2} g g^{-1}	0.25	0.24	Reference*, k = 1
5	Error, 10^{-2} g g^{-1}	0.27	0.19	[TS] – [Reference]
5a	**Relative error, %**	**1.44**	**0.98**	**[Error]/[Reference]**
6	u: Combined standard uncertainty (2 & 4), 10^{-2} g g^{-1}	0.25	0.24	u (k = 1)
7	U: Combined expanded uncertainty, (2 & 4), 10^{-2} g g^{-1}	0.51	0.48	U (k = 2)
7a	Relative expanded uncertainty	2.66 %	2.50 %	[U (k=2)]/[Reference]
8	**Claimed method uncertainty**	**4.10 %**	**4.10 %**	**TS, relative**
9	Max. allowed error	1.44 %	1.60 %	Relative
10	**Pass/fail**	**Pass**	**Pass**	

*TS: transfer standard, Reference: DTI primary standard

4.2.1.1 Conclusion

The results show that the validation of the transfer standard supports with a probability of 97.5 % a claimed method uncertainty for water mass fraction determination with the transfer standard of 4.1 % (relative, k = 2).

4.3 Portable transfer standard in the Vis-NIR range based on fibre-coupled spectrometers - Proximal Soil Moisture Sensor (PTSS)

Spectroscopic techniques in the visible–Near Infrared (VIS–NIR) and Shortwave Infrared (SWIR) ranges enable non-contact, rapid, and non-destructive soil analysis, supporting multi-criteria evaluation from both colour and spectral data. These methods provide critical insights into soil chemical composition, facilitating accurate classification and are applied in remote sensing (UAV, aerial, satellite) and proximal sensing.

Three primary technologies, listed below, underpin the development of soil spectral libraries (SSLs) worldwide, through both local and global initiatives. The Global SSL, combined with remote sensing techniques, is now extensively applied for soil surface mapping, enabling detection of dynamic processes and supporting large-scale monitoring of soil degradation and health. Many degradation processes - such as organic carbon loss, biodiversity decline, contamination, and acidification - primarily impact the topsoil horizon (≈30 cm depth). As these changes occur at the surface, the importance of proximal sensing continues to grow, offering increasingly valuable data for timely assessment and management.

1. VIS–NIR–SWIR spectrometers offer the highest spectral resolution via dispersive optics and sensitive detectors but are limited to point-based measurements with restricted spatial coverage.

2. Hyperspectral imaging systems capture both spatial and spectral data, including surface moisture patterns, but suffer from lower spatial resolution, reduced SWIR signal-to-noise ratio, and high data-processing demands.

3. Multispectral sensors provide lower spectral resolution but are cost-effective, compact, lightweight, energy-efficient, and simpler to process.

The first one, VIS–NIR–SWIR spectrometers, high-resolution spectrometer-based systems, covering 350–2500 nm in a single scan, have transformed soil analysis, enabling precise, non-destructive chemical characterization in both laboratory and field settings. Data from such instruments have, for an extended period, served as the foundation of SSL databases (both regional and global) containing paired spectral and chemical profiles.

Recently, considerable effort has been devoted by the soil spectroscopy research community to standardizing in-situ surface soil reflectance measurements in both laboratory and field settings. For example, ref. [18] proposed a standardized protocol, and ref. [19] introduced a dedicated apparatus designed to acquire soil reflectance data in the field without disturbing the soil surface. These methodologies have been formally adopted by the IEEE SA P4005 Working Group “Standards and Protocols for Soil Spectroscopy,” and are documented in “The IEEE SA Standard and Protocol Scheme for Soil Spectral Measurement in Both Laboratory and Field Conditions.”

Field soil reflectance measurements are affected by atmospheric conditions, solar angle, operator skills, protocol settings, surface heterogeneity, and measurement geometry. To reduce variability and align local and field SSLs for integration into a global SSL, a patented soil probe SoilPRO® (U.S.

Patent Office Serial No. 15407295) device is the benchmark in the domain. This portable device connects to any fibre-optic spectrometer and has shown strong field performance. In recognition of the need for standardized procedures, the IEEE Standards Association formed Working Group P4005 in 2020 to establish global protocols for soil spectral reflectance measurements across the VIS–NIR–SWIR spectrum.

Based on the defined requirements in these standards, within the scope of the SoMMet project, TÜBİTAK has successfully developed, tested, and SI-traceably calibrated a novel system for the soil moisture measurements based on the soil reflectance data. The system is designed to comply with the P4005 protocol. The system has been validated in both laboratory and field environments. When the Transfer Standard spectrometer was designed, the objectives were to:

1. Accurately assess soil surface moisture based on soil reflectance in the Visible, NIR, and SWIR regions.
2. Serve as a transfer standard for the calibration of other remote sensing and proximal sensors.
3. Be integrated into laboratory soil moisture measurement routines.
4. Be portable for field measurements.

An additional requirement, not directly connected to the SoMMet project scope, was to enable isolation of the soil's spectral signature from moisture-induced variations—removing or correcting the influence of soil moisture on spectral data to improve the accuracy of soil property predictions and support the development of soil spectral libraries. This was successfully achieved in the system shown in Figure 8 which currently has the following main features:

1. It is based on a high-precision spectrometer.
2. It covers the full spectral range from 350 nm to 2500 nm within a single device.
3. It fully complies with the requirements of the IEEE Standards Association Working Group P4005.
4. It can be calibrated traceable to SI units using TÜBİTAK’s primary-level reference system for soil water content measurements (which was developed also in the frame of the SoMMet project).
5. The device is robust and portable, enabling easy transportation and long-term field measurements by a single person.

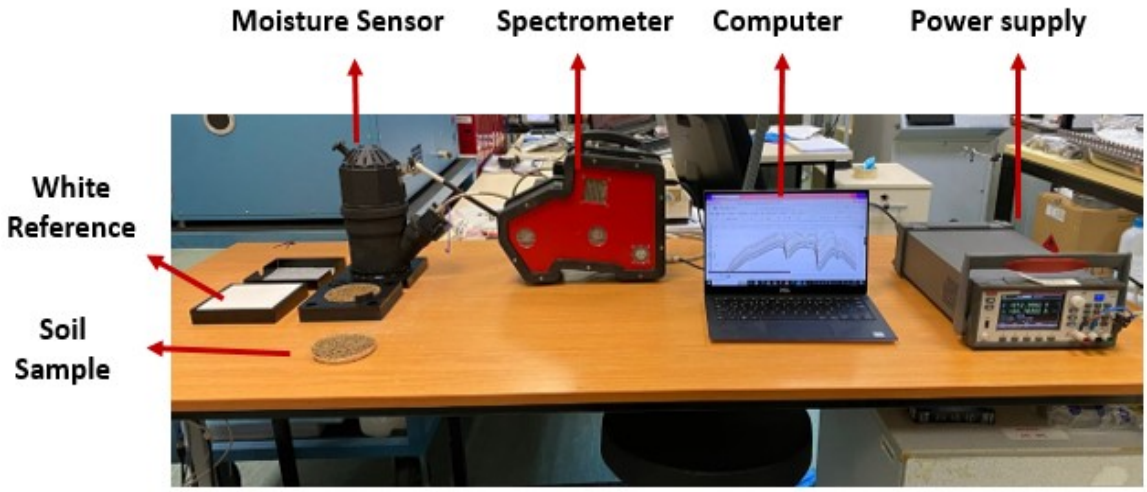


Figure 8. Reflectance-based soil moisture measurement system.

The system is composed of a high-resolution spectrometer, a custom-designed measurement chamber manufactured in-house using 3D printing, a lamp holder, a measurement table, and a reference white plate with its dedicated holder.

The spectrometer used was a commercially available VIS-NIR-SWIR device, specifically designed for field measurements. It was obtained from Spectral Evolution (USA) and is the NaturaSpec™ Spectroradiometer model. Table 11 summarizes its key specifications.

Table 11. Technical specification of the spectrometer.

Spectral range	350-2500 nm
Spectral resolution	**2.7 nm @ 700 nm** 5.5 nm @ 1500 nm **5.8 nm @ 2100 nm**
Spectral sampling bandwidth (nm)	Data output in 1nm increments 2151 channels reported
Calibration	Spectral and radiometric calibration for radiance/irradiance measurements using NIST traceable sources.
Noise equivalence radiance $W/cm^2/nm/sr$ (1.2 m fiber optic)	$0.3x10^{-9}$ @ 400 nm $0.1x10^{-9}$ @ 1500 nm $2.5x10^{-9}$ @ 2100 nm
Minimum scan speed	100 ms per scan
Wavelength reproducibility	0.1 nm
Wavelength accuracy	±0.5 nm bandwidth
Input	1.5 m fibre optic (25° field of view). Optional fore optics and longer fibre optic cables available.

The lamp holder has been specially designed and rigidly fixed to the measurement chamber to ensure uniform illumination of the soil surface. A diffuser is positioned in front of the lamp to (i) generate diffuse illumination, thereby eliminating specular reflections, and (ii) protect the soil surface from direct heating caused by lamp radiation, as shown in Figure 9. The system employs a tungsten

halogen lamp (25 or 50 W) with an aluminium reflector and diffuser to provide bright and uniform illumination.

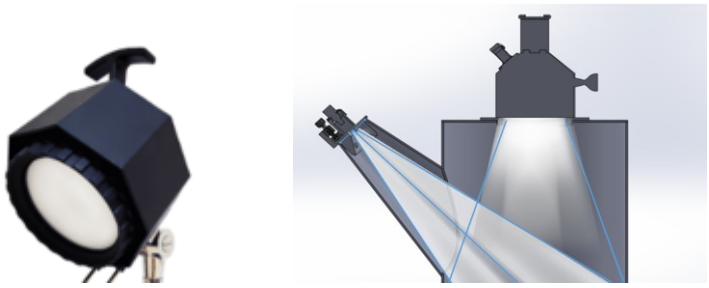


Figure 9. Left: The lamp holder used in the system. Right: Working principle of the portable Vis-NIR spectrometer.

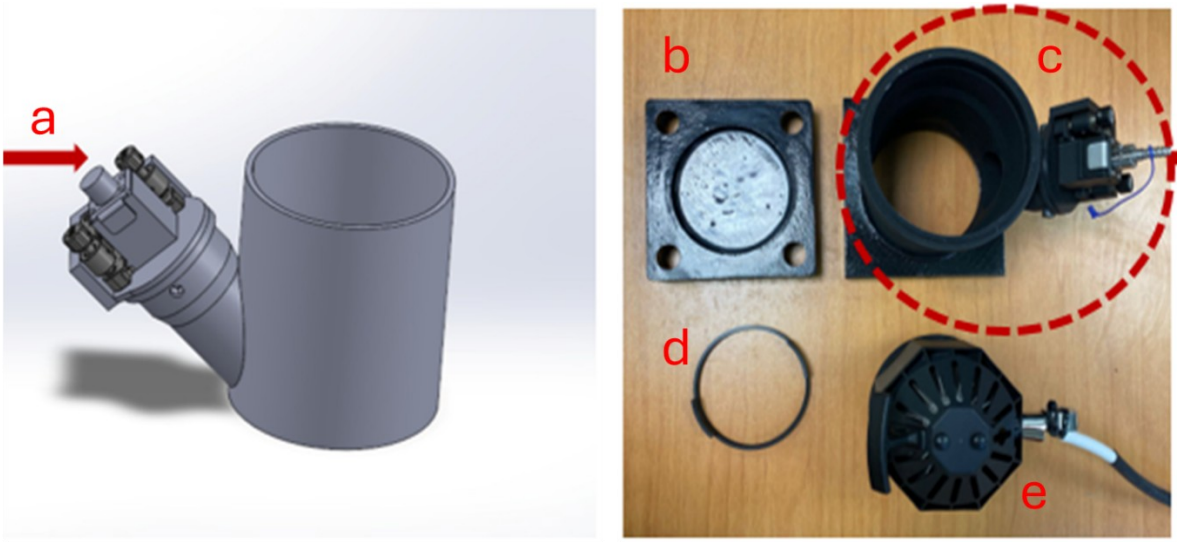


Figure 10. Components of the developed measurement chamber: (a) 3D CAD view of the assembled measurement chamber, (b) sample holder, (c) measurement chamber, (d) aluminium can sample adapter, and (e) tungsten–halogen lamp.

The main distinguishing characteristic of the developed system shown in Figure 10 is its use of vertical illumination combined with a 45° detection configuration. Unlike conventional designs, the system does not employ any pre-optics such as lenses or mirrors. Instead, the light reflected from the soil surface is directly collected by a bare optical fibre and guided to the spectrometer's measurement channel.

The field of view of the detection system has been designed such that it collects only the reflected light from a circular area with a 75 mm diameter. This configuration enables calibration and use under laboratory conditions by placing the soil sample into a standard glass Petri dish holder. Such Petri dishes are readily available in all chemical laboratories, easily purchasable, and commonly used in other soil research applications, including LoD tests and laboratory moisture analysers.

Soil samples were collected from twelve points on the TÜBİTAK campus (Izmit Gulf), with examples shown in Figure 11. Over ten soil types varying in organic matter, clay, and sand content were

selected for preliminary experiments. Topsoil samples (organic soils) were collected from the upper 10 cm using a 250 cm^3 custom Kopecky ring, mixing soil from five points per site. The surface 3–5 cm layer, crop residues, roots, and stones were removed. Inorganic soils were collected from 50–60 cm depth and cleaned of stones. Samples were sealed in airtight bags; some were measured immediately, while others were either stored at 4 °C for later laboratory analysis or dried and sieved using meshes ranging from 0.5 mm to 3 mm.

Figure 11. Left: Soil sampling area. Right: Examples of the collected soil types.

Figure 12. Representative soil samples selected from the study area: T0–T4, showing the variation in colour and texture among the five soil types.

Five representative soil samples (T0, T1, T2, T3, and T4) were selected from the twelve collected across the study area to capture the diversity of soil properties. As illustrated in Figure 12, these soils exhibit distinct colours ranging from light yellowish-brown to reddish-brown, reflecting variations in mineral composition and texture. This variability provides a basis for subsequent physical and chemical characterization under controlled measurement conditions.

Table 12. Physicochemical properties of three representative soils (T0, T1, and T2) selected from the study area, including soil texture, and organic matter content.

Performed analyses	T0	T1	T2
Lime (%)	1.81	0.66	0.82
Sand (%)	41.12	39.12	51.12
Silt (%)	24.00	24.00	16.00
Clay (%)	34.88	36.88	32.88
Soil texture	Clay-loam	Clay-loam	Sandy-clay-loam
Organic Matter (%)	1.67	0.58	1.08

From these five samples, three (T0, T1, and T2) were selected for detailed physicochemical analysis. Table 12 summarizes the principal parameters, including lime content, particle-size distribution (sand, silt, clay), and organic matter content. The samples differ markedly in texture—from clay-loam to sandy-clay-loam—and in chemical composition. These contrasts are expected to influence their optical, physical, and hydrological behaviour and therefore justify their selection for controlled measurements.

The system was calibrated using SI-traceable measurements performed on various soil types that were sieved into defined particle-size fractions and conditioned at multiple moisture levels. For this purpose, a dedicated soil humidification system was designed and implemented. This system includes a soil moisturizing unit consisting of a hermetically sealed chamber designed to hold soil samples. Inside the unit, four nebulizers generate water aerosol droplets, which are directed into the soil chamber to uniformly increase the moisture content of the sample.

The moisturized soil samples were subsequently placed in a refrigerator and stored at 4 °C for a minimum of two days.

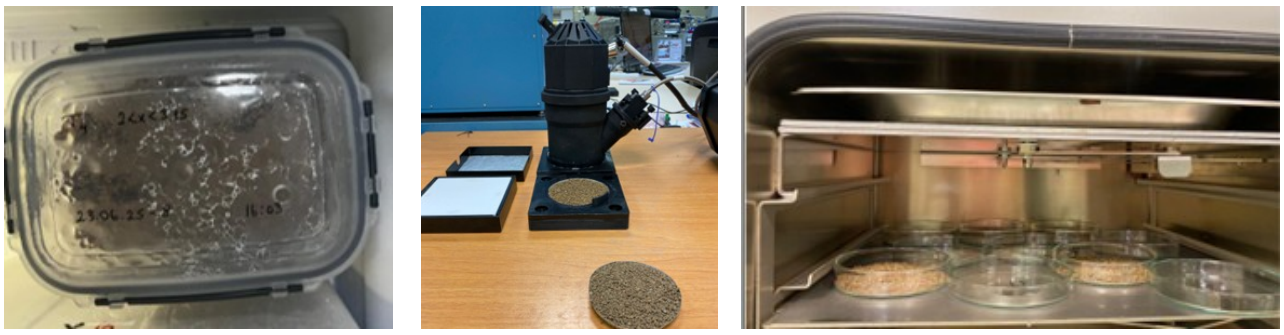


Figure 13. Left: Example of moisturized soil. Middle: Use of the transfer standard spectrometer in the laboratory. Right: View of the oven-based LoD system.

After conditioning, the samples were transferred into the prepared Petri dishes and measured using the developed system. Immediately afterwards, the samples were placed in both the primary-level reference system and the vacuum-capable Loss-on-Drying (LoD) system, as illustrated in Figure 13. At each soil moisture level, the conditioned soil was divided into three separate Petri dishes to ensure measurement repeatability and statistical reliability.

For each Petri dish, an initial “white” reference measurement was recorded, followed by three independent reflectance measurements of the same soil sample. Each individual reflectance measurement was obtained by averaging 40 spectra (internal option of the used spectrometer), after which the results were stored in the database. Figure 14 depicts a view from this measurement procedure in the laboratory.

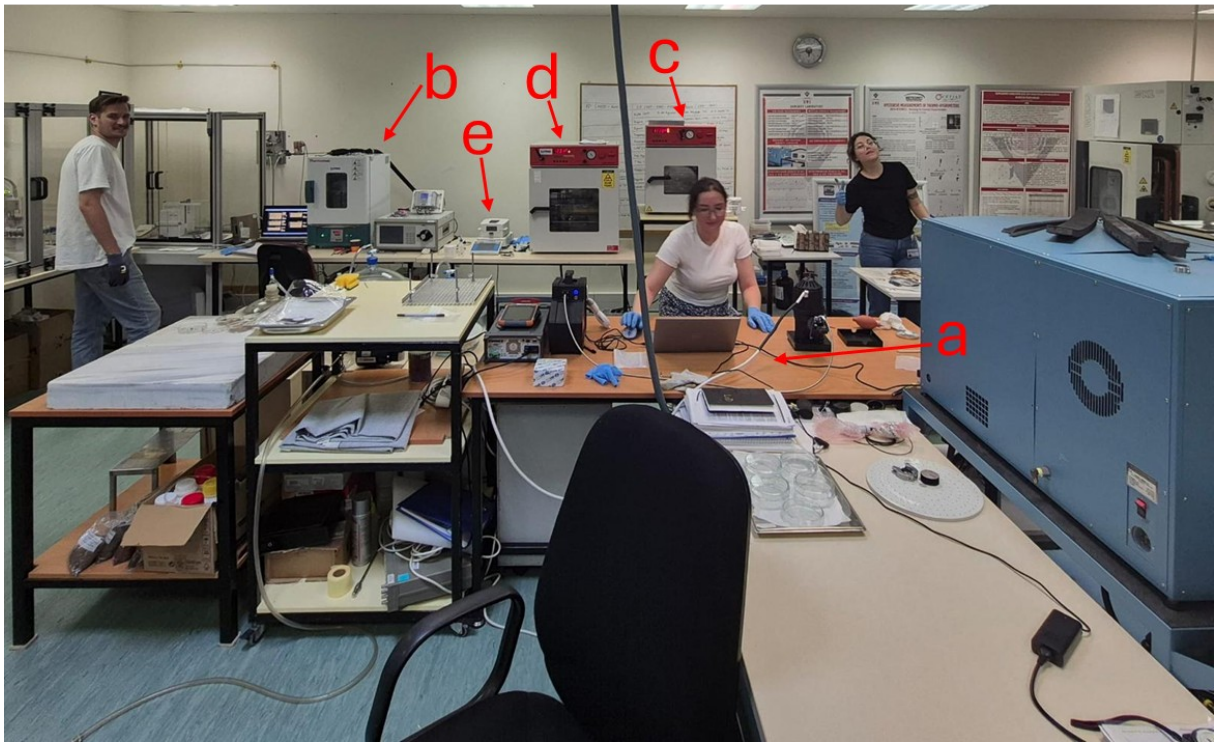


Figure 14. Laboratory setup for simultaneous measurements of soil samples using: (a) spectroscopic system, (b) primary-level soil water content system, (c) LoD system, (d) vacuum-capable LoD system, and (e) laboratory moisture analyser.

Figure 15 illustrates the use of the spectrometer-based portable transfer standard during field measurements. A representative set of measurement results, showing the spectral variation of light reflected from the soil surface as a function of soil moisture, is presented in Figure 16 . Only a subset of spectra is shown for clarity.

Figure 15. Field measurements with the spectrometer-based portable transfer standard.

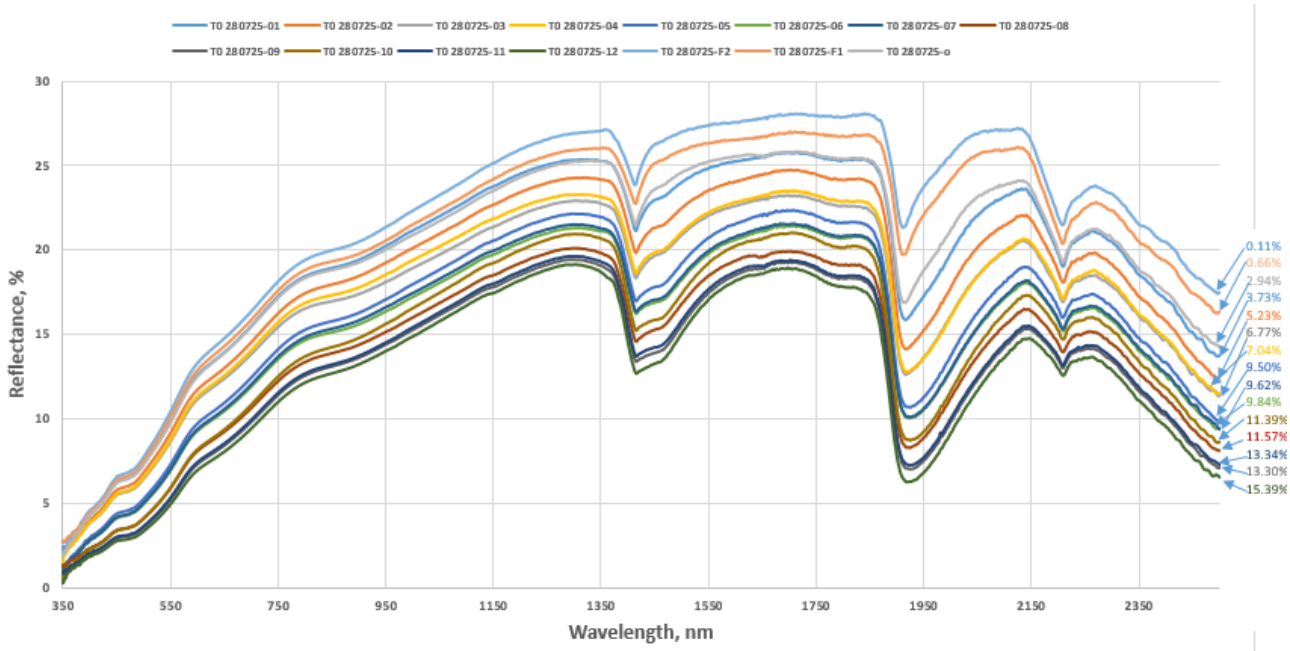


Figure 16. Reflectance results of soil T0 (2 < x < 3.15 mm).

To establish the relationship between reflected light from the soil surface and soil moisture content, two independent algorithms and corresponding software were developed, and their results were compared. The first algorithm is based on widely used techniques in the literature, consisting of a

sequence of methods including signal processing, Principal Component Analysis (PCA), Partial Least Squares (PLS), and Support Vector Regression (SVR).

For this purpose, the reflectance obtained by the developed spectral system were normalized using a reference white plate (whose reflection spectrum is traceable to NIST photometric standards). The reflectance values are normalized by dividing each spectrum by its maximum value. To ensure that specific soil and moisture combinations remain grouped within training, testing, or cross-validation sets, the normalized data is saved in a special format that preserves soil classification features (e.g., organic vs. inorganic type, particle size, moisture level). This approach prevents overfitting and data leakage and allows group-based logic to guide the selection of the number of latent components in the models.

Software developed in the Python environment was used for learning and training, and data splitting was performed using the GroupShuffleSplit method, which randomly divides the data while maintaining group integrity —each soil and moisture combination appears only in either the training or testing set. For each number of latent components, a PLS model is fitted, and the latent scores (X scores) are extracted. These scores are then input into an SVR pipeline, which includes standardization followed by regression. During cross-validation, the GroupKFold method ensures that no group is split across folds.

SVR hyperparameters are optimized using GridSearchCV, and the model achieving the highest cross-validation R^2 score is selected as optimal. Predictions are made using this model, with negative values clipped to zero. R^2 and RMSE metrics are calculated for both training and testing sets to assess performance. Additionally, various smoothing functions and alternative models are being tested to improve the R^2 score across datasets containing all soil types. Residuals were calculated as the difference between predicted and actual values. Figure 17 and Figure 18 show the model development results for the five soil types. The error between the model-predicted values and the actual soil moisture values (determined independently using the primary-level system and LoD) was within 3 %, which is well below the SoMMet project target of approximately 5 %.

Field measurements performed using the developed spectral system on native soil surfaces, followed by packing and transporting the soil samples to the laboratory (over 20 measurements were conducted on the TÜBİTAK campus area), resulted in a maximum discrepancy of approximately 4.5 % between in-field and laboratory measurements, remaining below the project target value of 5 %.

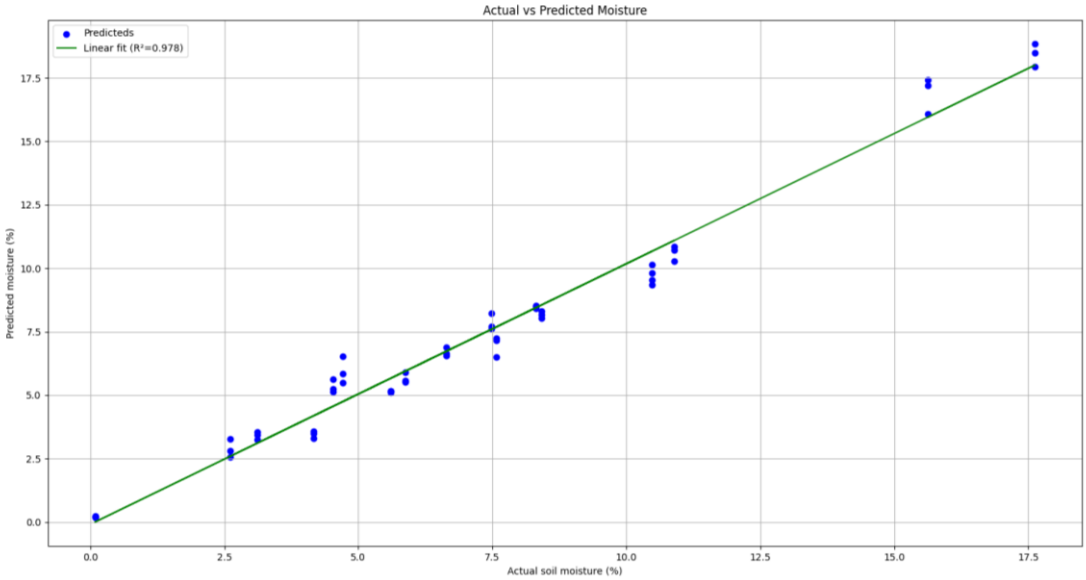


Figure 17. Actual vs predicted moisture plot for T0, T1, T2, T3, and T4 type soils.

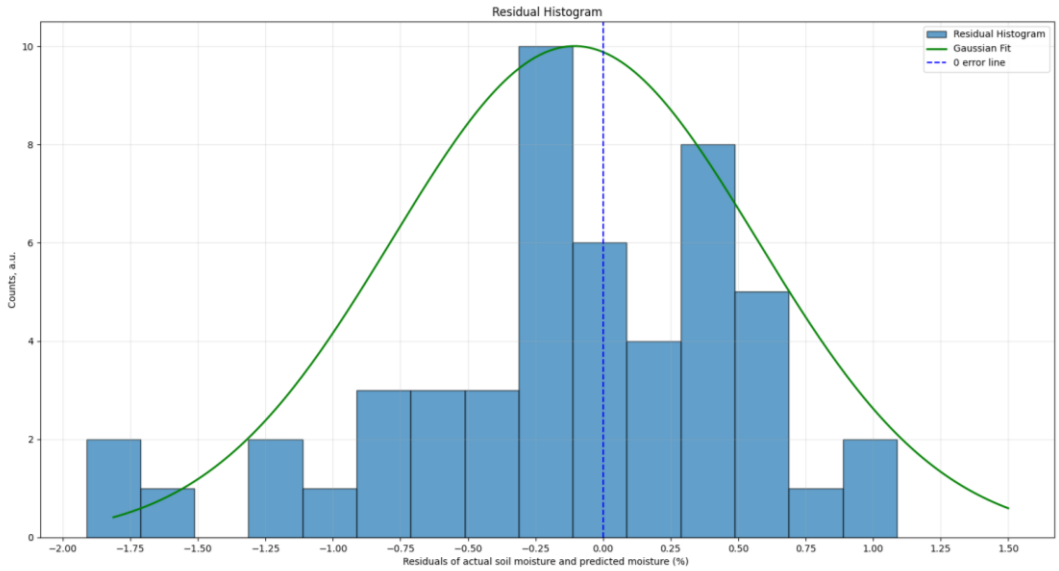


Figure 18. Residual histogram for T0, T1, T2, T3, and T4 type soils.

The second method is based on the simple machine learning algorithm, that involves SVM, PCA, SVM kernel comparison, K-fold cross-validation, and the application of PCA and SVM after data preprocessing using the ratio of wavelengths. Using over 900 spectra for machine learning and testing the results on more than 400 spectra revealed that this machine learning algorithm outperforms the standard linear approach described below. More advanced machine learning methods, such as those based on CNN technology, can provide even more robust results; however, they require greater computational resources, high-speed processors, and specialized hardware.

Within the scope of this project, since the goal was to develop a portable transfer standard that could also be used under field conditions, the focus was on creating an algorithm that can run efficiently on tablet-compatible hardware, which was achieved through low-resource calculations.

5 Calibration of point scale sensors

This section presents an outline of procedures required for calibrating point scale SM sensors. The SI traceability of input factors for establishing a reference volumetric water content is revisited, and an outline is given of procedures for preparing field-moist soil for calibration, and for establishing the relationship between a sensor indication and a reference value. An uncertainty budget for the calibration of point scale soil water content sensors is provided.

5.1 Traceability chain of point-scale sensors

The point scale sensors, that are assumed here to show an indication of volumetric water content (Equation (1)), will be made traceable through calibration by comparison. This means that it is necessary to obtain a traceable measurement of both water volume and soil volume. The water volume can be realized from measurements of water mass if the density of water is known. The density of water is often taken to be 1 g cm^{-3} [20], which is close to the free-water density at laboratory temperatures (0.997 g cm^{-3}) [21]. Some work indicates that this value may in fact be at the lower end of the water density range found in soil pores [22], but that is not explored further here. The water mass traceability is based on the primary measurement standards and the interlaboratory comparison of those standards presented in Section 3, as well as the use of validated portable secondary measurement standards presented in Section 4. The soil volume can be made traceable through SI traceable calibration of the equipment and measurement apparatus used in techniques such as those presented in Section 2.2.1.

5.2 Calibration procedure

5.2.1 Preparation of soil for sensor calibration

This section describes how soil samples for calibration are prepared at the DTI calibration facility. Some specific description of the preparation of soil samples at the individual laboratories is given in Section 6.

1. Soil is collected from the field and air dried.

2. The dried soil is sieved to remove grass, stones, roots, and other elements from the soil used for calibration. Aggregates of soil may be gently crushed to allow passage through the sieve.

3. Soil and water are mixed by manual, forced mixing. The relative masses of soil and water that are required to give a target volumetric water content can be calculated using the equations given in Section 2.1 by assuming a target bulk density for the soil.

4. The soil-water mixture may be covered and left for hours to days to homogenize.

Ideally, the soil that will be used for calibration is packed to a known bulk density matching the bulk density of the soil at the location of sampling.

5.2.2 Procedure for comparing a device indication and reference value

This section describes how a relationship between the indication from a Device Under Test (DUT, typically a point-scale sensor) and a reference value are established at the DTI calibration facility. Some specific description of this step at the individual laboratories is given in Section 6. The procedure is outlined in the following:

1. Pack the prepared soil mixture (Section 5.2.2) in a calibration container. Importantly, the container should be large enough to completely cover the DUT. A known mass of prepared soil may be packed to known dimensions to control the bulk density of the soil.
2. Note the time of beginning the calibration as the time at which prepared soil is taken from the mixing container.
3. Insert the DUT into the middle of the soil in the calibration container. For a DUT model that has not been encountered before, an investigation of the signal compared to depth should be undertaken to ensure a plateau in the indication. This indicates sufficient immersion of the DUT in the soil.
4. Measure the temperature of the soil in the calibration container.
5. Read the indication from the DUT for an extended time to assess the indication value and stability.
6. Extract the DUT from the soil.
7. Extract soil samples for homogeneity assessment using Kopecky rings. Extract two samples from the top of the container, diametrically opposite each other, and two samples from the lower half of the container, diametrically opposite each other, and with the connecting line between the last two samples being perpendicular to the connecting line between the first two samples.
8. Measure the WMF of the 4 samples and provide a reference value for VWC based on the expressions in Section 2.1.

5.3 Uncertainty estimate

The uncertainty in the calibration procedure outlined in Section 5.2.2 can be calculated given the following model equation which is derived from the expressions in Section 2.1.

$$VWC = \frac{m_{water}}{\rho_{water|T,\sigma} \cdot V_{soil,wet}} + VWC_{homogen.} + VWC_{stability} + VWC_{res.}, \quad (11)$$

where m_{water} is the mass of water obtained from a metrological reference, $\rho_{water|T,\sigma}$ is the density of water at the temperature in the laboratory and given a specific salt content (σ), and $V_{soil,wet}$ is the volume of the wet soil extracted for example, using a standardized method such as those presented in Section 2.2.1. $VWC_{homogen.}$ is the homogeneity of the volumetric water content in the calibration container, $VWC_{stability}$ is the stability of the DUT indication, and $VWC_{res.}$ is the resolution of the DUT indication. A similar analysis of the uncertainty has been undertaken in [16]. The numbers are calculated based on a volumetric water content of 25 % and assuming a relative uncertainty for the

determination of water mass of 3.0 %. The standard uncertainty due to the water density determination is based on the density change associated with a change in water temperature from 18 °C to 28 °C (based on a controlled laboratory temperature of 23 ± 5 °C) [21], and the density change due to salinity levels between 0 and 1 g kg^{-1}. The uncertainty budget is shown in Table 13.

The calculations show that at a volumetric water content of 25 % cm^3 cm^{-3} it is possible to realize an expanded calibration uncertainty (k=2) of 2.7 % cm^3 cm^{-3}. This uncertainty is dependent on the level of volumetric water content, and calculations for 10 % and 40 % VWC give expanded uncertainties (k=2) of 2.1 % VWC and 3.5 % respectively. The largest contributors to the measurement uncertainty are soil homogeneity in the container, the determination of water mass, and the determination of soil volume.

Table 13. Uncertainty budget for the calibration of a point scale sensor for volumetric water content.

Description	Estimate	Standard Uncertainty	Unit	Sensitivity coefficient	Contribution, 10^{-2} g g^{-1}
Water mass (m_{water})	25.56	0.77	g	$9.8 \cdot 10^{-3}$	$7.5 \cdot 10^{-1}$
Water density (ρ_{water})	0.99741	$7.2 \cdot 10^{-4}$	g cm^{-3}	$-2.5 \cdot 10^{-1}$	$1.8 \cdot 10^{-2}$
Soil volume ($V_{soil,wet}$)	102.5	2.0	cm^3	$-2.4 \cdot 10^{-3}$	$4.9 \cdot 10^{-1}$
Homogeneity* ($VWC_{homogen.}$)	0	0.01	cm^3 cm^{-3}	1	1.0
Stability* ($VWC_{stability}$)	0	0.001	cm^3 cm^{-3}	1	$1.0 \cdot 10^{-1}$
Resolution* ($VWC_{res.}$)	0	0.0001	cm^3 cm^{-3}	1	$1.0 \cdot 10^{-2}$
			Combined uncertainty (k=1), 10^{-2} cm^3 cm^{-3}		1.3
			Expanded uncertainty (k=2), 10^{-2} cm^3 cm^{-3}		2.7

* The homogeneity contribution is based on experience from the maximum homogeneity value encountered in calibrating sensors at DTI. Stability is similarly based on experience from the SoMMet project. Resolution is based on the observation that sensors encountered in the test of sensors (Section 6) all report the volumetric water content to 0.01 % cm^3 cm^{-3}.

6 Examples of calibration results

The results shown herein are the outcome of a collaborative effort during the SoMMet-project to test some important aspects of the use of point scale sensors for soil volumetric water content determination.

6.1 Influence of soil material

Using the calibration procedure presented in Section 5.2, DTI has investigated the influence of soil composition on the indications of a point scale sensor by calibrating a SMT100-sensor (Trübner, Germany) in 4 different inorganic soils. The four different soils were from the three SoMMet high-level test sites at Apulian Tavoliere hosted by the Italian National Research Council (CNR) (Italy, site located at 41.460 latitude, 15.50 longitude), Bondeno hosted by the University of Bologna (UNIBO) (Italy, site located at 44.8552 latitude, 11.3772 longitude), and Potsdam-Marquardt hosted by the University of Potsdam (UP DE) (Germany, site located at 52.466571 latitude, 12.95874 longitude), as well as a test field in Skejby hosted by the Danish Technological Institute (DTI) (Denmark, site located at 56.202 latitude, 10.150 longitude). The soils were sampled from a single location at each

site. However, the sampling was from one location at the site only and is therefore not a representative sampling for the site. The same specific SMT100-sensor was used for calibration against homogenized soil from the four sampling locations. The calibration data are shown in Figure 19. Calibration using the soil from Apulian Tavoliere was unsuccessful due to issues observed during the collection of soil cores for reference measurements. Figure 19 indicates that, for all other soil locations, the soil moisture sensor readings are lower than the reference value within the expanded uncertainty (k=2).

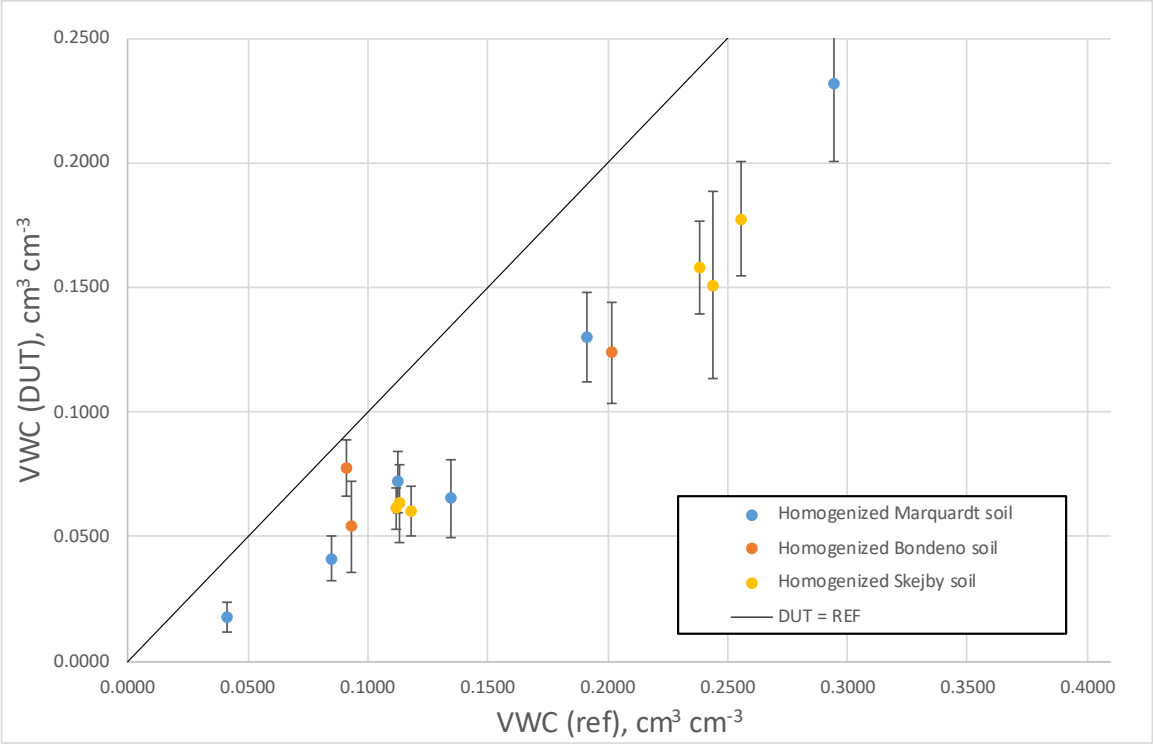


Figure 19. Calibration data of VWC given for the SMT100 sensor as a function of reference measurements with homogenized soils sampled at four different locations. The combined expanded uncertainty (k=2) in the calibration is shown. The full black line corresponds to DUT equal to REF.

The individual sets of calibration data as well as the combination of all data were fitted with a linear model giving the coefficients and fit statistics shown in Table 14. The model function is

$$[\mathrm{REF}] = \mathrm{Slope} \cdot [\mathrm{DUT}] + \mathrm{Intercept}$$

where $[\mathrm{REF}]$ is the reference value and $[\mathrm{DUT}]$ is the DUT indication. The model on all the calibration data excludes the data point with [REF] > 0.35 $cm^3\ cm^{-3}$. The standard error of the fit is calculated by the expression

$$s_e^2 = \frac{1}{n-2}\sum_{i=1}^{n}\left(VWC_{ref.,i} - VWC_{model,i}\right)^2, \qquad (12)$$

where n is the number of data points, $VWC_{ref.}$ is the reference value for volumetric water content that was measured by the reference instrument, and VWC_{model} is the reference value calculated from the linear model using the DUT reading as input. The standard error measures the spread of the model fit with respect to the reference measurements. The standard error of the fit is between 0.9 % and 3.2 % as shown in Table 14.

Table 14. Fits of a linear model to the calibration data of an SMT100-sensor in different soil materials.

	Skejby	**Marquardt**	**Bondeno**	**All**
Calibration points	6	6	3	15
Slope, [REF %] / [DUT %]	1.29	1.14	1.67	1.24
Intercept	3.5 %	3.8 %	-1.4 %	3.2 %
Standard error*	0.9 %	1.5 %	3.2 %	1.7 %

*Standard error calculated from Equation (12).

6.1.1 Conclusion on soil material

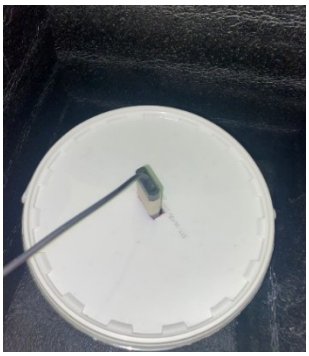
An SMT100 sensor was calibrated in three different soil materials collected at the SoMMet high-level test sites. The calibration data did not show any dependence on soil material: the full set of calibration points from all four soil types could be fitted equally well with a linear model, as when fitting one soil type at a time. The sensor (DUT) values were generally lower than the reference values, even when considering the measurement uncertainty.

6.2 Sensor-sensor variation

6.2.1 Methodology

In the initial phase, sensor-to-sensor variability tests were conducted using specially prepared soil samples with well-defined moisture content, bulk density, and soil type. The samples were stored for a week in a controlled environment to ensure homogenization and stabilization. In total, approximately ten soil samples were prepared and conditioned at various moisture levels, ranging from 5 % to 40 %, as determined gravimetrically.

Each soil moisture sensor was immersed in these samples for at least one full day with measurements recorded at one-minute intervals. The sensors were placed in the soil in a consistent manner as shown in Figure 20 (a) and (b), and data acquisition followed the same procedure across all tests. For CS650 sensors, the results showed good consistency, with differences within 2 % as shown in Figure 21. In contrast, SMT100 sensors exhibited higher variability, with results spanning a range of about 7 %, as can be seen in Figures 23–27.



a. Only sensor immersed, vertical.

b. Only sensor immersed, horizontal.

c. Complete immersion, vertical.

d. Complete immersion, horizontal.

Figure 20. Insertion of soil moisture sensors during calibration.

Further experiments were performed on the same soil samples using the same sensors. In this procedure, a sensor was removed from the soil after one day and re-immersed under identical conditions. CS650 sensors again demonstrated stable and repeatable results, whereas SMT100 sensors showed significant changes between repeated measurements. This variability was attributed primarily to differences at the soil-sensor interface, immersion depth, and handling.

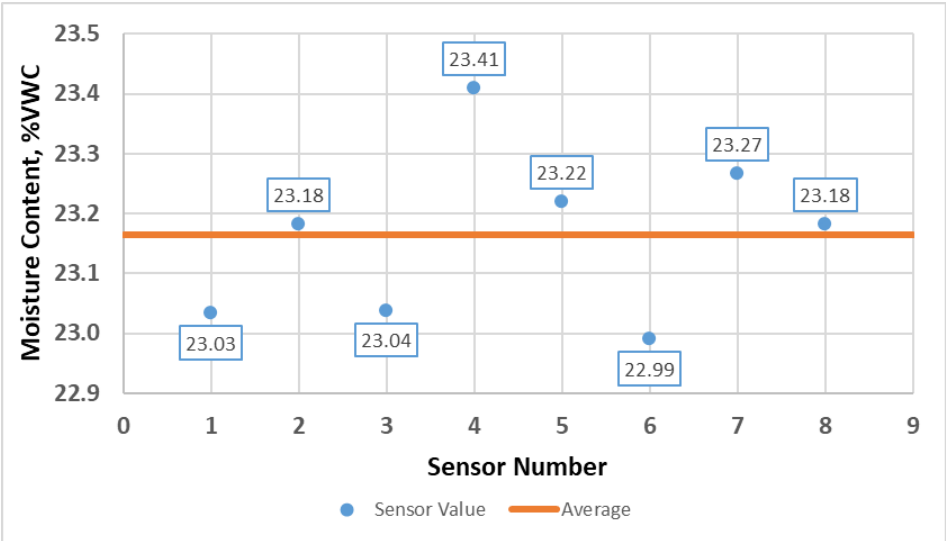


Figure 21. Sensor-to-sensor variability measurements for CS650 volumetric water content probes with T0 soil.

Additionally, tests were conducted under two different configurations: (i) when only the sensing area was immersed in the soil (Figure 20 (a) and (b)) and (ii) when both the sensing area and the

electronic block were fully immersed (Figure 20 (c) and (d)). In both cases, the measurement-to-measurement variability of the SMT100 sensors exceeded 5 %.

Figure 22. Left: SMT100 sensors. Right: Performing the soil wetting process.

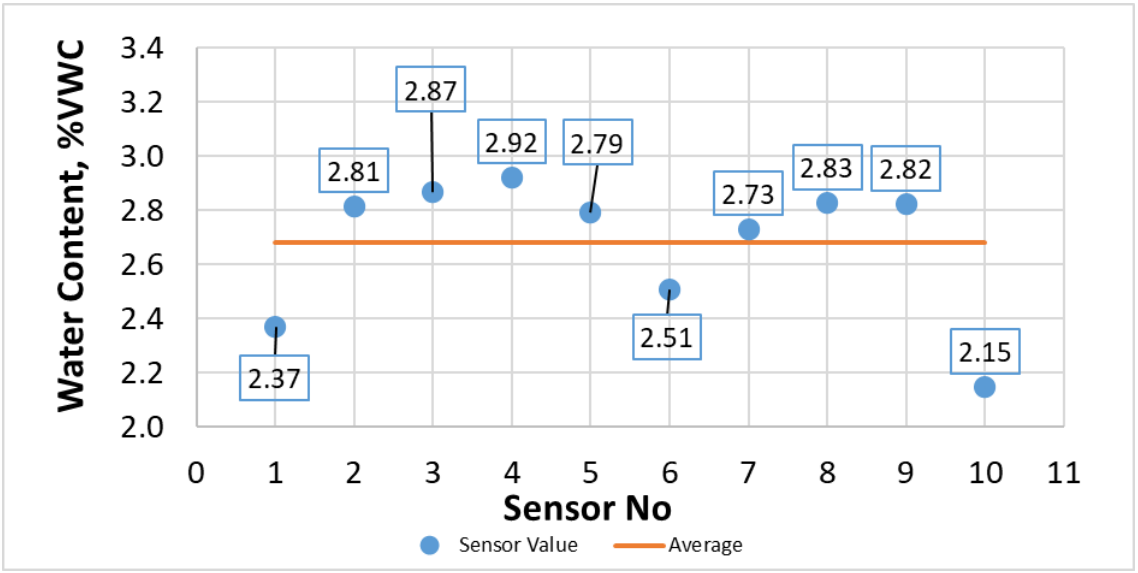


Figure 23. Volumetric water content (VWC) measurements obtained from ten SMT100 probes in the same soil sample (T0, 2.02 ± 0.10 %MC). The graph presents both the individual probe readings and the mean value (2.68 %VWC).

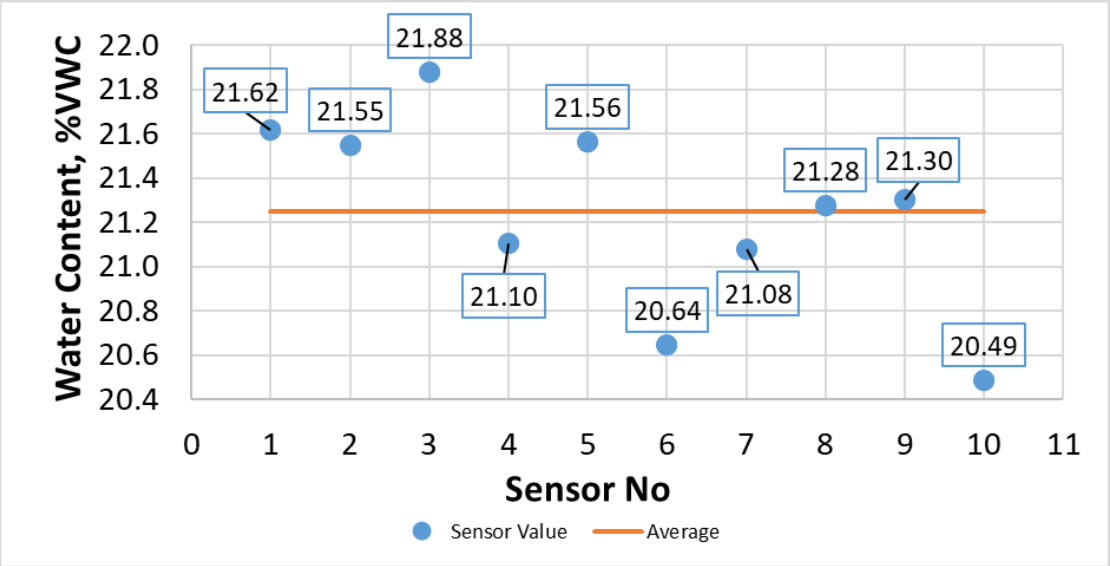


Figure 24. Volumetric water content (VWC) measurements obtained from ten SMT100 probes in the same soil sample (T0, 8.22 ± 0.15 %MC). The graph presents both the individual probe readings and the mean value (21.25 %VWC).

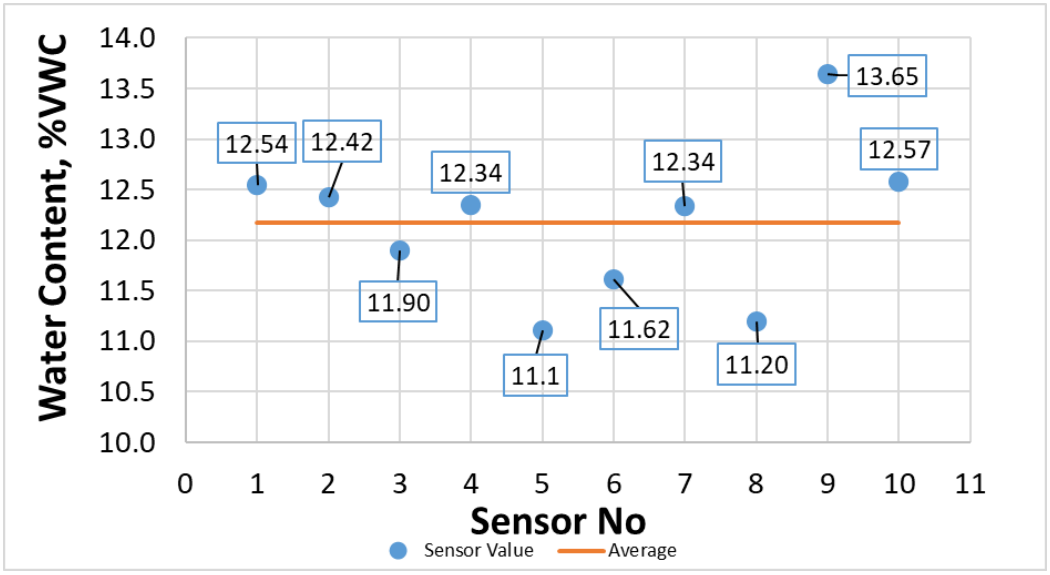


Figure 25. Volumetric water content (VWC) measurements obtained from ten SMT100 probes in the same soil sample (T0, 11.85 ± 0.18 %MC). The graph presents both the individual probe readings and the mean value (12.17 %VWC).

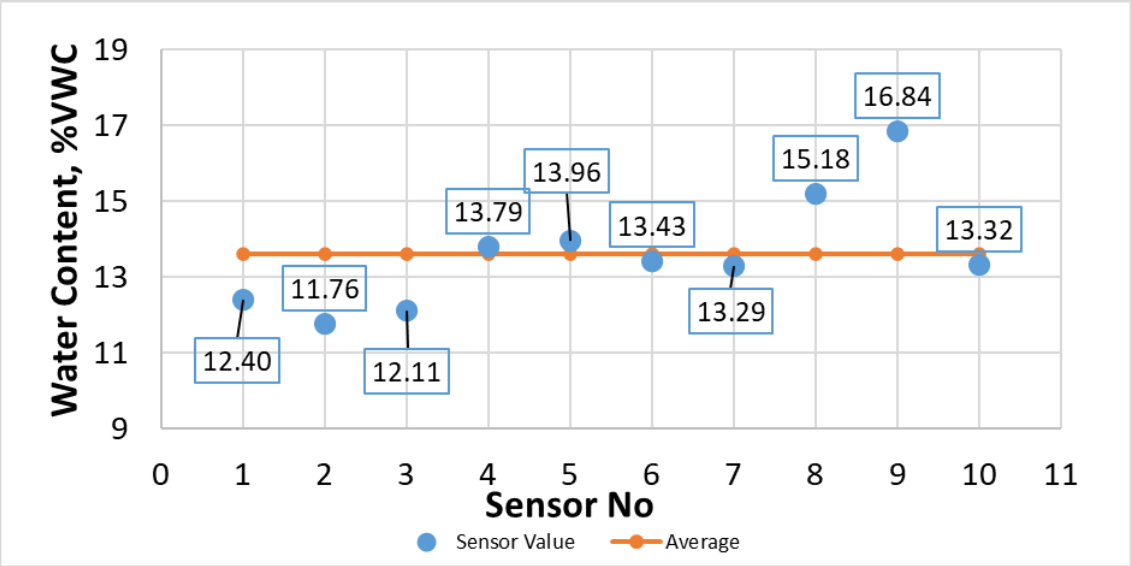


Figure 26. Volumetric water content (VWC) measurements obtained from ten SMT100 probes in the same soil sample (T0, 16.19 ± 0.21 %MC). The graph presents both the individual probe readings and the mean value (13.61 %VWC).

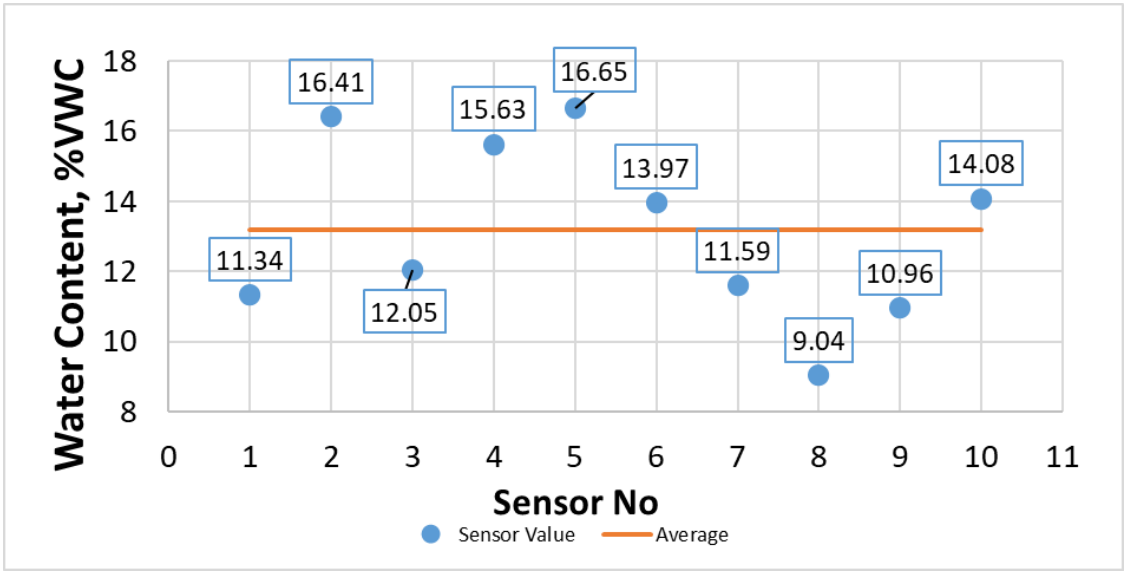


Figure 27. Volumetric water content (VWC) measurements obtained from ten SMT100 probes in the same soil sample (T0, 15.87 ± 0.20 %MC). The graph presents both the individual probe readings and the mean value (13.17 %VWC).

6.2.2 Bulk density calculation

For bulk density determination, a ring (E16) with a length of 19.73 mm, a diameter of 15.98 mm, and a volume of 3.96 cm³ was used, as illustrated in Figure 28. Soil samples were collected from each soil type without applying compaction. The collected bulk soil samples were subsequently dried using a Radwag MA 60.3y moisture analyser, and the bulk density values were calculated according to the expressions in Section 2.1.

Figure 28. Left: Bulk soil sampling. Right: Ring E16 used to determine the bulk density of the soils used for testing sensor-to-sensor variability.

6.3 Sensor-type variation

Four different sensors with different measuring principles were compared using several different types of soil with varying water content and bulk density. The aim was to evaluate the accuracy of each device and identify factors that could distort the measurement.

The SMT100 sensor works on the principle of Frequency Domain Reflectometry (FDR), which measures the dielectric constant at high frequencies. This method significantly reduces the influence of salinity and temperature on the results, compared to conventional capacitive sensors.

The CS655 sensors use the Time Domain Reflectometry (TDR) and, in addition to volumetric water content (VWC), also measure soil temperature and electrical conductivity (EC). These additional parameters enable better compensation for environmental influence. Their ability to measure EC is particularly important in soils with high salinity, where other sensors may fail.

The simplest of the devices tested, the 10HS is capacitance sensor whose outputs are strongly influenced by soil salinity and organic content. Factory calibration often did not provide reliable results. This demonstrates the need for local calibration for each soil type.

The last device tested was Soil Moisture Meter FieldScout TDR 350 which also uses the TDR technology and measures volumetric water content, electrical conductivity and surface temperature. Its robust design and fast data collection are advantageous for field use, but the results showed greater variation compared to other sensors, especially in soils with higher heterogeneity.

The testing was focused on the following aspects:

1. Comparison of the outputs of individual sensors with gravimetric moisture determination, which served as the reference method.
2. Evaluation of the influence of soil texture and bulk density on measurement accuracy. It was found that heavier clay soils and soils with higher organic matter content increase the variability of results.
3. Evaluation of the adequacy of factory calibration – it turned out that only some sensors (e.g., CS655, SMT100) provide satisfactory results without the need for local calibration, while sensors such as 10HS require it almost always.

An exemplary test setup is shown in Figure 29, and a summary of the obtained results is visualised in Figures 30–32.

Figure 29. The setup for SMT100 sensors testing – three different soil types.

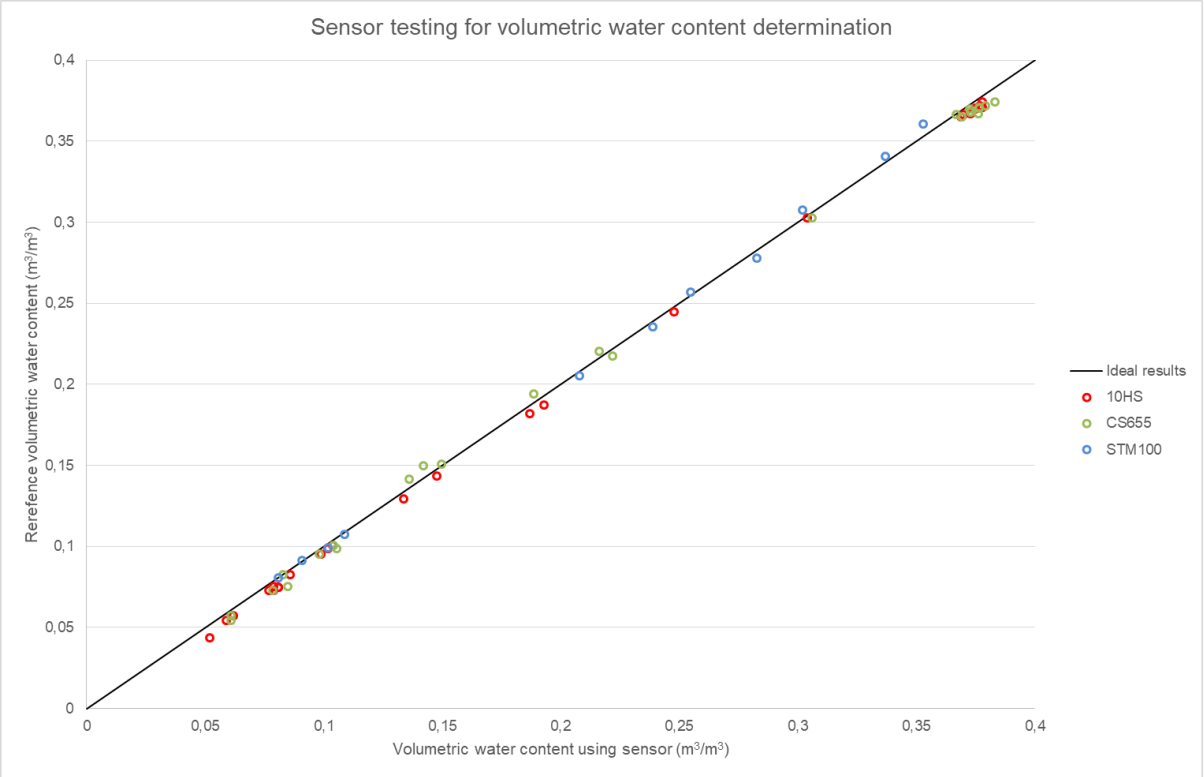


Figure 30. Comparison of measured and reference VWC for three different sensor types.

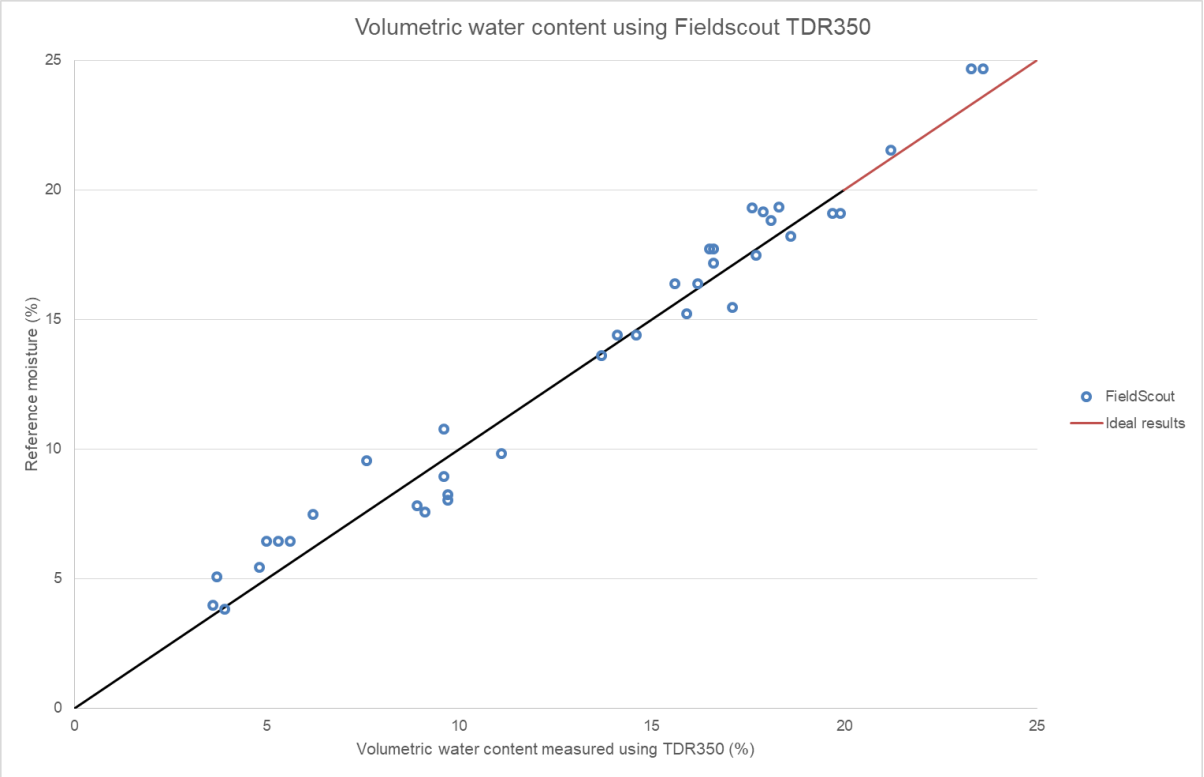


Figure 31. Comparison of measured and reference VWC for FieldScount TDR350.

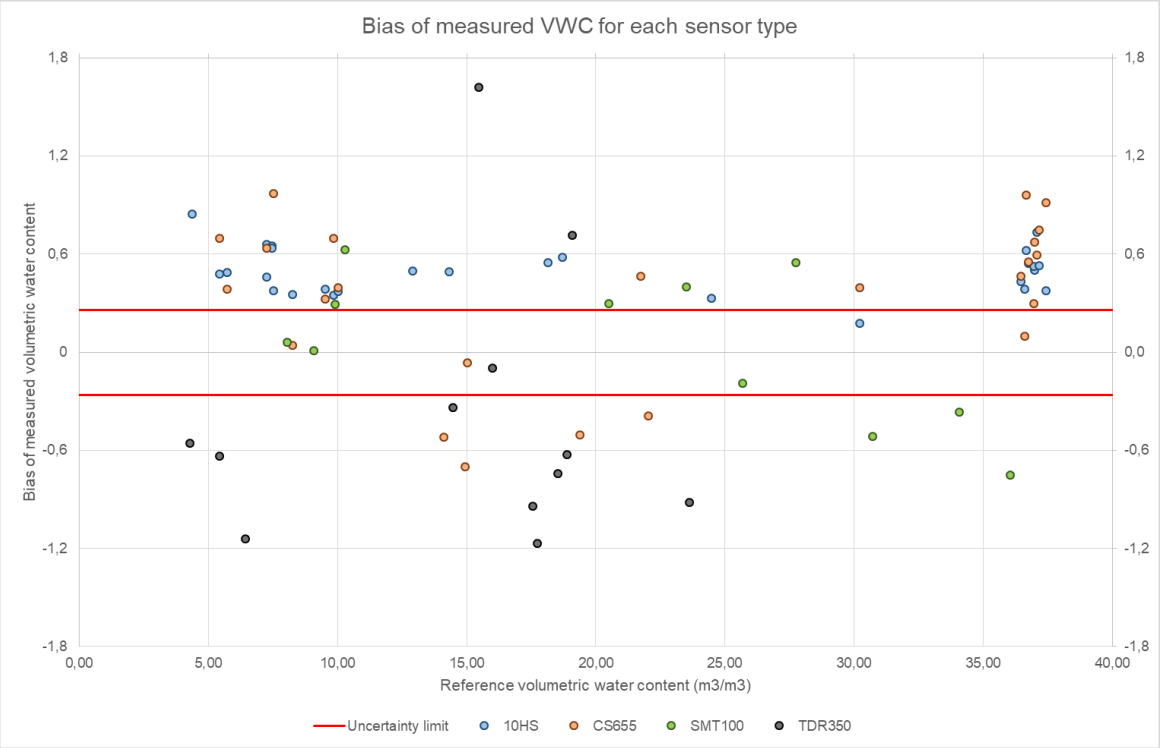


Figure 32. Comparison of Bias ([sensor] – [reference]) vs. Reference VWC for all types of sensors with the uncertainty of reference VWC limits.

All sensors showed some sensitivity to temperature, which required a stabilization period of at least 10 minutes before each measurement. Underestimating this phase led to significant deviations. In addition, it was found that sensors are affected to varying degrees by the salinity of the environment, which significantly affects the measurements of capacitive sensors. The exception was the CS655, which allows for better data interpretation thanks to its integrated EC measurement. 10HS sensors are ideal for homogenous, mineral soils after a local calibration. In terms of practical field use, sensors based on TDR and FDR technologies proved to be the most reliable, as they cope better with changing conditions. In the case of capacitive sensors, thorough calibration for each specific soil is necessary.

Table 15. Comparison of the key parameters of tested sensors.

Sensor	Technology	Parameters measured	Local calibration	Salinity sensitivity	Drift stability
SMT100	FDR	VWC	No	Low	Very High
CD655	TDR	VWC, T, EC	No	Low	High
10HS	Capacitance	VWC	Yes	High	Medium
TDR 350	TDR	VWC, T, EC	No	Medium	Medium

7 Conclusion

Accurate measurement of soil water content is critical for a wide range of applications, including smart irrigation, soil health assessment, and hydrological studies. Although commonly used, the term “soil moisture” can be ambiguous—particularly in organic-rich soils, where water coexists with volatile organic compounds. This highlights the importance of precise, SI-traceable determination of soil water content, especially given recent advances in sensor technology and growing interest in soil chemistry.

The primary measurement standard of the EWC system developed at TÜBITAK in this project, along with the primary measurement standards already existing at DTI and CMI, provide an accurate approach for measuring soil water content. The dual functionality of the EWC allows simultaneous assessment of both soil moisture via thermogravimetric analysis and water content through direct vapour extraction from a single sample. Through the interlaboratory comparison EURAMET Pilot study P1685, it has been shown that the primary standards all show bilateral agreement. Furthermore, the soil samples used for the comparison show excellent homogeneity and stability during transport, providing further opportunity for future comparison studies.

During the project, two secondary measurement transfer standards have been developed at DTI and TÜBITAK. The DTI transfer standard is based on the fLoD-principle, and the TÜBITAK transfer standard is based on UV-Vis spectrometry. Both transfer standards have been tested in their respective operating environments. The DTI transfer standard was validated against the DTI primary measurement standard, and the validation of the TÜBITAK transfer standard will be presented in a separate publication.

The metrology laboratories at TÜBITAK and DTI developed and implemented procedures and facilities for calibrating point scale soil water content sensors. Similar facilities exist at CMI. Through a combined effort, these facilities were employed to test four types of soil water content sensors, the Trübner SMT100, Campbell Scientific CS655, the METER 10 HS and the Fieldscout TDR350. The sensors were chosen based on the sensor models already being employed at some of the high-level sites of SoMMet. The tests were planned to assess sensor-to-sensor variability for SMT100-sensors, differences between the three models of sensor, and the SMT100 response in different soils. The results for differences in the SMT100-sensor between soils show that the sensor generally reads a smaller value for soil moisture content than the reference measurements. However, the calibrations do not reveal a dependence on the soil material. We note that all four soils in which the sensor was tested were inorganic soils.

The test of sensor-to-sensor variability showed that the tested CS650 sensors have an inter-sensor variability of roughly 0.4 % VWC. The tested SMT100 sensors showed a much larger inter-sensor variability of up to ~7.6 % VWC for a soil with a reference VWC of ~16 % VWC. The test of variations between sensor types shows that there is a good agreement between sensor indications and reference value for the CS650 and SMT100 sensor types. On the other hand, the FieldScout TDR350 shows a higher variability with respect to the reference value.

The tests altogether highlight the importance of SI-traceable reference values in the calibration of point scale soil moisture measurement devices.

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