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Soil quality — Screening method for water content — Determination by refractometry

Qualité du sol — Méthode de diagnostic applicable à la teneur en eau — Détermination par réfractométrie

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Foreword

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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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For an explanation on the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see the following URL: www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 190, *Soil quality*, Subcommittee SC 3, *Chemical and physical characterization*.

Introduction

On-site determination of water contents in soil helps geological and geotechnical research as well as commercial work in a variety of fields, especially in geotechnical engineering and agriculture. In the civil engineering field, water content tests have been required for the investigation or treatment of contaminated soils. At a disaster site, water contents specifying soil physical properties need to be rapidly determined in a reliable manner on-site, to enable the design of suitable civil engineering structures to relieve the damage caused or prepare appropriate remediation measures. Another potential example is for commercially grown vegetables in greenhouses or in the field. The most important need is to control the water content in the soil. In this type of scenario, quick, robust and simple methods are needed. The rapid screening method described in this document is a simple robust on-site test method for water contents of soils and has been developed to meet such a demand. It is based upon refractive index measurement of a sucrose solution after mixing with a soil sample.

In laboratories, water contents are normally determined by weighing soil samples before and after drying at a specified temperature (e.g. 105 °C). It is not practical, however, to apply this type of method to outdoor sites, since the method requires a time-consuming drying process. Furthermore, soil samples are conveyed from the sites to the laboratory with the need that the water content in a soil sample be maintained during sample transport to the laboratory. The proposed on-site method can be readily employed directly in the field and can be used to rapidly determine water contents at given sites.

One of the recent applications of water content tests is related to global environmental protection work, e.g. on the reduction of greenhouse gas emissions from soil. Management of water in soil to control greenhouse gas emission can help minimizing climate change issues, which depend on the conditions of microbial properties in soil. CO_2, CH_4 and N_2O are emitted from soil as a result of microbial activities that are activated by water at ambient temperatures.

The largest supply source of carbon dioxide is not necessarily from human activities, including industrial facilities and transportation services, but vital natural activities in soil. Increases in atmospheric temperature cause the frozen or cold ground to melt or warm up to change environmental conditions for the soil microbes. These are stimulated and activated at relatively low soil temperatures that will initiate their metabolisation systems. If there is moisture and biomass that can be digested by microbes in their environment, the microbes will immediately start metabolizing labile organic carbon, in biomass resulting in carbon dioxide that will be emitted into the air. This mechanism directly contributes to climate change since carbon dioxide is the most common greenhouse gas.

Investigations have been carried out to try and map land across the world for such risks. Two techniques can be used to monitor the target parameters. The first one is observation of the parameters with panoramic viewing using satellites and planes equipped with infrared or near-infrared spectroscopic detection devices and the second one is a screening method such as that described in this document. Data obtained using the two techniques can be compared to correct, improve and/or complement data from the less accurate panoramic view technique. This will allow more accurate and detailed mapping results of the potential risk from carbon dioxide emissions.

In this context, a rapid check screening method for determination of water contents of soil has become an international social demand in an effort to assist investigation of climate change issues. As the scale for investigation is very large, the refractive index measurement method with simple solvent extraction of water from soil is considered suitable for such situations.

Soil quality — Screening method for water content — Determination by refractometry

1 Scope

This document specifies a method for rapid determination of water content in soils. The method is based on refractive index measurement of a sucrose solution after it is mixed with a soil sample.

It is applicable to the determination of water content in geological or geotechnical research as well as geotechnical engineering. In addition, it can be used for commercial work in a variety of fields, e.g. agriculture and civil engineering.

The working range is up to approximately 50 % moisture content. The precision of the method typically ranges between 0,5 % and 1 % and depends on the type of refractometer that is used.

The result of this method is strongly influenced by soil matrices. Higher contents of clay and/or organic matter will lead to significant lower values for water content compared to standard methods such as that described in ISO 11465.

2 Normative references

There are no normative references in this document.

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at http://www.electropedia.org/

3.1
refractometer
device for measurement of a refractive index

3.2
saccharimeter
refractometer (3.1) that indicates a sucrose concentration instead of a refractive index

3.3
Brix degree
mass fraction percentage of sucrose in solution, which is related directly to a refractive index

3.4
temperature correction
function for a *refractometer* (3.1) or a *saccharimeter* (3.2) to correct measurement deviation caused by atmospheric temperature when measurement is carried out at a temperature other than 20 °C

4 Principle

When a soil sample is mixed with a sucrose solution, the sucrose solution is diluted with water contained in the soil.

The water content in the sucrose solution is determined by refractive index or Brix degree measurement with a refractometer. Both data are converted into water contents using a correlation curve established with sucrose solutions spiked with a known amount of water.

5 Apparatus and reagents

5.1 Portable refractometer, which has a function of output in refractive index or Brix degree and a function of temperature correction.

Both ordinary refractometer types and saccharimeter types are suitable.

5.2 Portable electronic balance, with an appropriate level of precision and accuracy to meet the requirements for measurement of water contents.

NOTE A balance capable of weighing to the nearest 0,1 g can be used in most cases.

5.3 Sucrose solution, 50 % in concentration.

5.4 Containers, such as plastic cups or glassware in dry condition, in which a soil sample and the sucrose solution are mixed.

5.5 Filtering device, in dry condition, such as a funnel with a filter paper to separate soil from the sucrose solution.

6 Procedure

6.1 Selection of a temperature correction mode

Confirm that a temperature correction mode has been selected in the refractometer. If temperature correction parameters are required for correction, input required the parameters by following the instruction manual given by the manufacturer.

6.2 Creation of a correlation curve

A correlation curve is necessary to convert the measured refractive indexes or Brix degrees of the sucrose solution to water contents of the unknown soil. The correlation curve should be prepared prior to on-site determination of water contents.

Establish a correlation curve between refractive indexes (or Brix degrees) and water contents as follows.

a) Prepare a sucrose solution by carefully dissolving an exact amount of sucrose into measured water and dilute to 50 % in concentration.

   NOTE In case a correlation curve has a gentler slope or solutions are difficult to handle because of high viscosity, sucrose solutions at lower or higher concentrations can be used, unless there is a problem with inaccurate water content quantification.

b) Set up a correlation curve by adding different amounts of water to the sucrose solutions, for the relevant range. For instance, if 10 g sucrose solution is used, add in the range of 0 to 4 g water to the 10 g of sucrose solution. Plot a curve with the amount of water added on the horizontal axis and the measured refractive index or Brix degree on the vertical axis.

6.3 Confirmation of refractometer performance

Before determination, set up and condition the refractometer to be used, according to the instructions of the instrument manufacturer.

Measure the following samples to confirm the basic performance of the refractometer:

a) pure water;

b) sucrose solution with a concentration adjusted to 50 %.

Confirm that refractive indexes or Brix degrees match well with the expected readings of the refractometer.

6.4 Procedure for on-site determination of water contents

a) Take a representative soil sample and homogenize if necessary.

b) Weigh 10 g to 30 g of the sampled soil with a portable electronic balance (5.2).

c) Weigh out the sucrose solution with a portable electronic balance (5.2). The amount of the sucrose solution can be the same amount as the amount of soil or larger/smaller but the amount of the sucrose solution shall be the same as that taken for calibration in 6.2.

d) If taking a certain volume of a sucrose solution is convenient, use a pipette instead of a balance. In this case, the correlation curve shall be prepared in the same manner described in 6.2 b) but using a pipette to take a certain volume of a sucrose solution.

e) Mix the soil with the sucrose solution and completely stir the mixture.

f) Separate the soil from the sucrose solution with a filter to obtain the sample solution to be measured for refractive indexes. When the mixture is too viscous to filter, increase the amount of the sucrose solution and then follow the procedure after c). Apply the procedure in 6.2 using the changed amount of the sucrose solution to prepare the corresponding correlation curve.

g) Measure the refractive index or Brix degree of the sample solution.

7 Calculation of results

Read the mass of water extracted from soil directly from the horizontal axis of the correlation curve. Calculate the mass fraction percentage of water in soil as analysed according to Formula (1)

$$w_{\text{fm}} = \frac{m_{\text{w}} \times 100}{m_{\text{s}}} \tag{1}$$

where

w_{fm} is the water content of the soil as a mass fraction percentage;

m_{w} is the mass of water extracted from soil

m_{s} is the mass of soil.

NOTE The mass fraction percentage of water based on dry soil (w_{dm}) can be calculated according to Formula (2)

$$w_{\text{dm}} = \frac{m_{\text{w}} \times 100}{m_{\text{s}} - m_{\text{w}}} \tag{2}$$

The water content shall be rounded off to the next percent without any decimals.

Information on validation results and statistical evaluation of the method is given in Annex A.

8 Quality control

8.1 General performance

Periodically check the refractometer in accordance with the instructions of the instrument manufacturer. Use pure water and at least three sucrose solutions with a known concentration at different levels.

8.2 Site-specific performance

Periodically check the refractometer under conditions specific to a site (e.g. outdoor atmospheric conditions in a winter or summer season) in accordance with the instructions of the instrument manufacturer. Use pure water and at least one sucrose solution at a known concentration.

9 Test report

The test report shall contain the following information:

a) a reference to this document, i.e. ISO 20244:2018;

b) any site-specific information;

c) complete identification of the samples;

d) identification of the operation mode of the instrument;

e) the results of the determination;

f) any details not specified in this document or that are optional as well as any factor that may have affected the results.

Annex A
(informative)

Validation results and statistical evaluation

Table A.1 — Validation results and statistical evaluation

Sample	**Water content according to ISO 11465** %	*l*	*n*	**Outliers** %	*x*	s_R	s_r	$C_{V,R}$	$C_{V,r}$
1	11,6	11	44	0	10,3	0,88	0,35	8,6 %	3,3 %
2	18,6	11	44	9,1	13,4	2,64	0,67	19,8 %	5,0 %
3	5,5	11	44	9,1	5,0	0,49	0,27	9,7 %	5,3 %
4	14,6	11	44	0	9,5	2,02	0,82	21,3 %	8,6 %

l is the number of laboratories participating;

n is the number of submitted results;

Outliers is the percentage of results eliminated based on the Cochran and Grubb 's test;

x is the mean value, in % based on wet sample;

s_R is the reproducibility standard deviation, % based on wet sample;

s_r is the repeatability standard deviation, in % based on wet sample;

$C_{V,R}$ is the reproducibility coefficient of variation, in percent;

$C_{V,r}$ is the repeatability coefficient of variation, in percent.