CEN/TF

Date: 2005 - 10 - 04

TC WI

CEN/TF

Secretariat: DS

Determination of electrical conductivity in soil, sewage sludge and biowaste

Einführendes Element — Haupt-Element — Ergänzendes Element

Élément introductif — Élément central — Élément complémentaire

ICS:

Descriptors: soil, sewage sludge, biowaste, tests, electrical conductivity

Document type: European Standard
Document subtype:
Document stage: Working Document
Document language: E

Foreword

This document is a working document.

This document TF WI has been prepared by CEN/BT/Task Force 151 – Horizontal Standards in the Field of Sludge, Biowaste and Soil, the secretariat of which is held by Danish Standards.

This document has been prepared under a mandate given to CEN by the European Commission and the European Free Trade Association, and supports essential requirements of EU Directive(s).

For relationship with EU Directive(s), see informative Annex A, B, C or D, which is an integral part of this document.

This standard is applicable and validated for several types of matrices. The table below indicates which ones.

[table to be filled and amended by the standards writer]

Material	**Validated for** **(type of sample, e.g. municipal sludge, compost)**	**Document**
Sludge	X	Desk study to asses the feasibility of a draft horizontal standard for electrical conductivity Lars Johnsson, S Ingvar Nilsson & Per Jennische
Soil	X	- " -
Soil improvers		
Sediment		
Waste	X	- " -

Contents

Page

Introduction

This document is developed in the project 'Horizontal'. It is the result of a desk study prepared by Lars Johnsson, S. Ingvar Nilsson & Per Jennische entitled “Desk study to assess the feasibility of a draft horizontal standard for electrical conductivity” and aims at an evaluation of the latest developments in assessing electrical conductivity in sludge, soil, treated biowaste and neighbouring fields. After discussion with all parties concerned in CEN and selection of a number of test methods described in this study the standard has been developed further as an modular horizontal method and has been validated within in the project 'Horizontal' .

A horizontal modular approach is being investigated and developed in the project 'Horizontal'. 'Horizontal' means that the methods can be used for a wide range of materials and products with certain properties. 'Modular' means that a test standard developed in this approach concerns a specific step in a test procedure and not the whole test procedure (from sampling to analyses).

The use of modular horizontal standards implies the drawing of test schemes as well. Before executing a test on a certain material or product to determine certain characteristics it is necessary to draw up a protocol in which the adequate modules are selected and together form the basis for the test procedure.

The texts of the chapters 1 to 12 are normative; annexes are normative or informative, as stated in the top lines of the annexes.

1 Scope

This European Standard describes an instrumental method for the routine determination of the specific electrical conductivity in an aqueous extract of soil (fresh or air-dry), sludge (fresh) or biowaste (fresh). The determination is carried out to obtain an indication of the content of water-soluble electrolytes in the materials mentioned. This European standard is based on ISO 11265 (soils). There is presently no international standard for sludge or biowaste. For practical reasons, for instance if there is a need to make strict comparisons with previous measurements, soils should generally be air-dried. Air-drying can be used for all soils with one important exception: EC in soils that contain sulphidic material should be measured on fresh samples to avoid sulphide oxidation resulting in the formation of sulphuric acid. Whenever sulphidic soils are to be compared with other soils the comparison should be made on fresh samples. For sludge and biowaste, fresh samples are recommended. Air-drying may introduce artefacts due to stimulation of oxidation processes and should therefore be avoided.

2 Normative references

This European Standard incorporates by dated or undated reference, provisions from other publications. These normative references are cited at appropriate places in the text and the publications are listed hereafter. For dated references, subsequent amendments to or revisions of any of these publications apply to this European Standard only when incorporated in it by amendment or revision. For undated references the latest edition of the publication referred to applies (including amendments).

EN ISO 3696: 1997, Water for analytical laboratory use - Specification and test methods.

ISO 7888:1985, Water quality – Determination of electrical conductivity.

ISO 11464 Soil quality – Pretreatment of samples for physico-chemical analyses.

3 Terms and definitions

For the purpose of this European Standard, the following definition applies:

3.1 Electrical conductivity (EC) is a numerical expression of the ability of an aqueous solution to carry an electrical current. EC at equilibrium in a water suspension of soil, sludge or biowaste is expressed as milli-Siemens per meter (mS/m).

4 Safety remarks

None

5 Principle

A suspension of air-dried (or fresh) soil, fresh sludge or fresh biowaste is made up in 5 times (soil) or 10 times (sludge and biowaste) its volume with water, to dissolve the electrolytes. Concerning liquid sludge the measurements are made without adding any water (cf. the procedure used for determination of pH in soil, sewage sludge and biowaste). The specific electrical conductivity of the filtered extract is measured and the result is corrected to a temperature of 20^o *C*.

6 Interferences and sources of errors

The measured values of the electrical conductivity can be influenced by contamination of the electrodes. Air bubbles on the electrodes perturb the measurements. Measurements < 1 mS/m are influenced by gaseous carbon dioxide (CO_2) or ammonia (NH_3) coming from the atmosphere. In these cases, measurements are carried out in an adapted measuring cell. This aspect is not further treated in this European Standard.

7 Reagents

Use only reagents of recognised analytical grade.

7.1 Water, with a specific electrical conductivity not higher than 0.2 mS/m at 20 °C (grade 2 water according to EN ISO 3696).

7.2 Potassium chloride solution

c(KCl) = 0.1 mol/l.

Dissolve 7.456 g of potassium chloride, previously dried for 24 h at 220 °C ± 10°C in water (7.1) and dilute to 1000 ml at 20°C. The specific electrical conductivity of this solution is 1161 mS/m at 20°C.

7.3 Potassium chloride solution

c(KCl) = 0.0200 mol/l.

Pour 200.0 ml of the potassium chloride solution (7.2) into a 1000 ml volumetric flask and dilute to volume with water at 20°C. The specific electrical conductivity of this solution is 249 mS/m at 20°C.

7.4 Potassium chloride solution

c(KCl) = 0.0100 mol/l.

Pour 100.0 ml of the potassium chloride solution (7.2) into a 1000 ml volumetric flask and dilute to volume with water at 20°C. The specific electrical conductivity of this solution is 127 mS/m at 20°C.

All the potassium chloride solutions (7.2, 7.3, 7.4). used for calibration shall be stored in tightly sealed bottles which do not release alkali or alkali-earth metals in amounts that would compromise the electrical conductivity of the solutions.

NOTE: Polyethylene bottles could be used. The use of commercially available conductivity standards is also permitted.

8 Apparatus

8.1 Equipment for sample preparation

According to ISO 11464 (soil samples), EN 12176 (sludge) and EN 13037 (biowaste)

8.2. Conductivity meter , fitted with a conductivity cell, equipped with an adjustable measuring range setting and automatic temperature correction and having an accuracy of 1 mS/m at 20°C. Preferably, the conductivity meter should also be equipped with a cell-constant control.

8.3 Analytical balance, with an accuracy of at least 0.01 g.

8.4 Thermometer, capable of measuring to the nearest 0.1 °C.

8.5 Shaking machine, with a horizontal movement sufficiently vigorous to produce and maintain 1:5 or 1:10 substrate-water suspensions, placed in for instance a constant room, where the temperature is maintained at 20°C ± 1°C.

8.6 Filter paper, with low ash content and high retentive properties.

8.7 Shaking bottle, of sufficient capacity, made of polyethylene.

9 Sampling and sample pre-treatment

Sampling should be carried out in accordance with EN yyyy:2003 (Horizontal standard module(s) for sampling of sludge, soil and waste).

Samples should be pretreated according to ISO 11464 (soil samples), EN 12176 (sludge) and EN 13037 (biowaste). The particle size should be < 2mm. Air-dried samples should not be exposed to a temperature higher than 40 ^{o}C. The pretreated samples should be stored in well aerated but closed polyethylene containers. Samples should be kept cold (< 8°C) and in the dark. The sample pre-treatment should take place within 24 hours of sampling.

NOTE: No pretreatment is needed for liquid sludge.

10 Procedure

10.1Extraction

Weigh 20.00 g of the laboratory sample and transfer to a shaking bottle (8.7). Add 100 ml of water (7.1) to soil samples and 200 ml to sewage sludge or biowaste samples at a temperature of 20^{o}C $\pm$ 1^{o}C. Close the bottle and place it in a horizontal position in the shaking machine. Shake for 30 minutes. Filter directly through a filter paper (8.6).

NOTE: Extraction is not applicable to liquid sludge. Measurements should be made directly.

10.2Calibration by checking the cell constant.

10.2.1 Measure the conductivity (EC_M) of the potassium chloride solutions (7.2, 7.3 and 7.4) according to the instruction manual of the instrument.

10.2.2 Calculate for each potassium chloride solution, a cell constant according to

$$K = EC_S/EC_M$$

where

K is the cell constant in reciprocal metres (1/m)

EC_S is the specific electrical conductivity of one of the potassium chloride solutions in mS/m, according to its concentration

EC_M is the measured electrical conductivity of the same potassium chloride solution, in mS/m.

Use the average of the calculated values as the cell constant of the instrument. The calculated cell constant should not differ by more than 5% from the value given by the manufacturer.

10.2.3 Adjust the cell constant on the conductivity meter.

10.3Measurement of the electrical conductivity of the filtrates

Measure the electrical conductivity of the filtrates (EC_M) according to the instructions provided by the manufacturer of the conductivity meter (8.2). Carry out the measurements with the temperature corrected to 20^{o}C. Temperature corrections of the measured values are made by adding 2 % of the measured value (measurement temperature < 20^{o}C) or subtracting 2 % of the measured value (measurement temperature > 20^{o}C) for each degree's difference.

10.4Blank determination

Carry out blank determination in each batch of samples by treating water (7.1) in the same way as the samples. The value of the blank should not exceed 1 mS/m. If the EC of the blank > 1 mS/m, the extraction should be repeated from the beginning. Carry out at least two blank determinations in each series and use the average blank value for subsequent calculations.

10,5Quality Assurance of the overall procedure

10.5.1 Duplicate determination

Analyse two individual test samples of each (dried or fresh), homogenised sample submitted for analysis. Establish a control limit for the difference between results for the two sub-samples based on for example laboratory precision data.

10.5.2 Analysis of spiked natural samples

Not relevant for routine EC measurements

11 Expression of results

Note the results to 1 decimal place, expressed in mS/m.

12 Test report

The test report shall contain the following information:

a) a reference to this European Standard including its date of publication;

b) precise identification of the sample;

c) type of sample preparation: fresh, air-dry or (concerning liquid sludge) no preparation

d) expression of results, according to 11.

e) any deviation from this standard, and any facts which may have influenced the result. Where the test is not carried out in accordance with this standard, reference may only be made to EN xxxx:2003 in the report in case all deviations from the procedures prescribed in this standard are indicated in the report stating the reason for deviation.

13 Performance characteristics

Performance data in terms of repeatability of the electrical conductivity measurements in two separately prepared filtrates shall satisfy the requirements shown in the following table.

Electrical conductivity (mS/m *at 20^{o}C*)	Accepted variation
0 to 50	5 mS/m
> 50 – 200	20 mS/m
> 200	10%

Annex A
(informative)

Validation of methods

Annex B
(informative)

The modular horizontal system

Annex C
(informative)

Information on WP xx and the project Horizontal

Bibliography

EN 12176 (1998) Characterization of sludge- Determination of pH-value. European committee for standardization. Brussels, Belgium.

EN 13037 (1999). Soil improvers and growing media – Determination of pH. European committee for standardization. Brussels, Belgium.

EN ISO 3696: 1997, Water for analytical laboratory use - Specification and test methods.

ISO 7888:1985, Water quality – Determination of electrical conductivity.

ISO 11265 (1994) Soil quality – Determining of the specific electrical conductivity

ISO 11464 (1994) Soil quality – Pretreatment of samples for physico-chemical analyses

Johnsson, L., Nilsson, S.I. & Jennische, P. 2005. Desk study to assess the feasibility of a draft horizontal standard for electrical conductivity.