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Sludge, Treated Biowaste and Soil - Determination of Specific Electrical Conductivity

Einführendes Element — Haupt-Element — Ergänzendes Element

Élément introductif — Élément central — Élément complémentaire

ICS:

Descriptors: soil, sewage sludge, **treated** biowaste, tests, specific electrical conductivity

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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, Treated 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		Johnsson, L., Nilsson, S.I. & Jennische, P. (2005). Desk study to asses the feasibility of a draft horizontal standard for electrical conductivity
Soil		- " -
Soil improvers		
Sediment		
Treated Biowaste		- " -

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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 specific electrical conductivity in sludge, treated biowaste, soil 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 a 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 analysis).

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 routine determinations of specific electrical conductivity in aqueous extracts of sludge (fresh), treated biowaste (fresh) or soil (fresh or air-dry). Please note that soil improvers and growing media are not included in this standard. The EC determination is carried out to obtain an indication of the content of water-soluble electrolytes in the materials mentioned. The standard is based on ISO 11265 (soils). There is presently no international standard for sludge or treated 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, except for those containing sulphidic minerals or volatile acids. In both cases fresh soil should be used to avoid either sulphide oxidation resulting in the formation of sulphuric acid, or volatilisation of low-molecular organic acids. Regarding sludge and treated biowaste, fresh samples are recommended. In these materials air-drying may introduce artefacts due to a stimulation of oxidation processes and should therefore be avoided.

Fresh samples versus air-dry, depending on sample category:

	Fresh	Dry
Sample category		
Sludge	X	
Treated biowaste	X	
Soils containing sulphide minerals	X	
Soils containing volatile acids	X	
Other soils	X*	X*

* Optional depending on whether comparisons are to be made with previous measurements on fresh or air-dry samples.

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).

ISO 1770:1981, Solid-stem general purpose thermometers

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 – Determination of the specific electrical conductivity.

ISO 11464:1994, Soil quality – Pretreatment of samples for physico-chemical analyses.

EN 13040: 1999, Soil improvers and growing media – Sample preparation for chemical and physical tests, determination of dry matter content, moisture content and laboratory compacted bulk density.

EN 12176:1998, Characterization of sludge - Determination of pH-value.

EN 13038: 1999, Soil improvers and growing media – Determination of electrical conductivity.

3 Terms and definitions

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

Specific 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 sludge, treated biowaste or soil is expressed as milli-Siemens per meter (mS/m).

4 Principle

A suspension of either fresh sludge, fresh treated biowaste or air-dried (or fresh) soil is made up in 5 times its weight 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 sewage sludge, treated biowaste or soil). The specific electrical conductivity of the filtered extract is measured and the result is corrected to a temperature of 25°C. Temperature corrections of the measured values are made by adding 2 % of the measured value (measurement temperature < 25°C) or subtracting 2 % of the measured value (measurement temperature > 25°C) for each degree's difference (Bower & Wilcox 1965).

5 Interferences and sources of errors

The measured values of the specific 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. Other sources of error are associated with materials containing sulphidic minerals or volatile acids (see section 1).

6 Reagents

Use only reagents of recognised analytical grade.

6.1 Water, with a specific electrical conductivity not higher than 0,2 mS/m at 25 °C (grade 2 water according to EN ISO 3696).

6.2 Potassium chloride solution

c(KCl) = 0,1 M.

Dissolve in water 7,456 g of potassium chloride, previously dried for 24 h at 220 °C ± 10°C (6.1) and dilute to 1000 ml at 25°C. The specific electrical conductivity of this solution is 1290 mS/m at 25°C.

6.3 Potassium chloride solution

c(KCl) = 0,0200 M.

Pour 200,0 ml of the potassium chloride solution (6.2) into a 1000 ml volumetric flask and dilute to volume with water at 25°C. The specific electrical conductivity of this solution is 277 mS/m at 25°C.

6.4 Potassium chloride solution

c(KCl) = 0,0100 M.

Pour 100,0 ml of the potassium chloride solution (6.2) into a 1000 ml volumetric flask and dilute to volume with water at 25°C. The specific electrical conductivity of this solution is 141 mS/m at 25°C.

All the potassium chloride solutions (6.2, 6.3, 6.4). 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 specific electrical conductivity of the solutions.

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

7 Apparatus

7.1 Equipment for sample preparation

According to EN 12176 (sludge), EN 13040 (treated biowaste) and ISO 11464 (soil samples). The latter standard refers to air-dry soil. Except for the drying procedure, it is applicable to fresh soil samples as well.

7.2 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. Preferrably, the conductivity meter should also be equipped with a cell-constant control.

7.3 Analytical balance, with an accuracy of at least 0,01 g.

7.4 Thermometer, capable of measuring to the nearest 0,1 °C.

7.5 Shaking machine, with a horizontal movement sufficiently vigorous to produce and maintain 1:5 substrate-water suspensions. It should be placed in a constant room, where the temperature is maintained at 25°C ± 1°C.

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

7.7 Shaking bottles, of sufficient capacity, made of polyethylene.

8 Sampling and sample pre-treatment

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

Samples should be pretreated and preserved according to EN 12176 (liquid or pastelike sludge; fresh), EN 13040 (treated biowaste; fresh) or ISO 11464 (soil; fresh or air-dry). The particle size of soils should be ≤ 2 mm, while that of treated biowaste should be 20-40 mm.

NOTE 1: Sample preparation for liquid sludge is unnecessary. EC should be determined directly according to EN 12176. Pastelike sludge may need a breakdown of solid particles before the preparation of a suspension.

NOTE 2: ISO 11464 refers to air-dry soil. Except for the drying procedure, it is applicable to fresh soil samples as well.

9 Procedure

9.1 Extraction

Weigh 20,00 g of the laboratory sample (sewage sludge or soil) and transfer to a shaking bottle **(7.7)**. Add 100 ml of water **(6.1)** at a temperature of 25°C ± 1°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 **(7.6)**. For treated biowaste the procedure and weight ratio water :sample should be similar. For a detailed description see EN 13038.

NOTE 1: Extraction is not applicable to liquid sludge. Measurements should be made directly.

9.2 Calibration by checking the cell constant.

9.2.1 Measure the specific conductivity (EC_M) of the potassium chloride solutions (6.2, 6.3 and 6.4) according to the instruction manual of the instrument.

9.2.2 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 specific 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.

9.2.3. Adjust the cell constant on the conductivity meter.

9.3 Measurement of the specific electrical conductivity of the filtrates

Measure the specific electrical conductivity of the filtrates (EC_M) according to the instructions provided by the manufacturer of the conductivity meter (9.2). Carry out the measurements with the temperature corrected to 25 ^oC.

9.4 Blank determination

Carry out a blank determination in each batch of samples by treating water (6.1) 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.

9,5 Quality Assurance of the overall procedure

If duplicate measurements are made (optional), the average value should be reported. A reference sample should be included in each batch of samples. The reference could be either “home made” or consist of a certified reference material.

10 Expression of results

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

11 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 **10**.

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.

12 Performance characteristics

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

Specific conductivity (mS/m at 25°C)	Accepted variation
0 to 50	5 mS/m
> 50 – 200	20 mS/m
> 200	10%

*) This table is preliminary, and will be adjusted during validation step 1.

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

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

Bower, C.A. & Wilcox, L. V. 1965. Soluble salts by electrical conductivity. In Black, C. A. (Editor-in Chief) Methods of Soil Analysis Part 2. Chemical and Microbiological Properties, pp. 936-940. American Society of Agronomy, Inc., Publisher Madison, Wisconsin, USA.