SLOVENSKI STANDARD
oSIST prEN 15933:2011

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Blato, obdelani biološki odpadki in tla - Določevanje pH

Sludge, treated biowaste and soil - Determination of pH

Schlamm, behandelter Bioabfall und Boden - Bestimmung des pH-Werts

Boues, bio-déchets traités et sols - Détermination du pH

Ta slovenski standard je istoveten z: **prEN 15933**

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Sludge, treated biowaste and soil - Determination of pH

Boues, bio-déchets traités et sols - Détermination du pH

Schlamm, behandelter Bioabfall und Boden - Bestimmung des pH-Werts

This draft European Standard is submitted to CEN members for second enquiry. It has been drawn up by the Technical Committee CEN/TC 400.

If this draft becomes a European Standard, CEN members are bound to comply with the CEN/CENELEC Internal Regulations which stipulate the conditions for giving this European Standard the status of a national standard without any alteration.

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Foreword

This document (prEN 15933:2010) has been prepared by Technical Committee CEN/TC 400 “Project Committee - Horizontal standards in the fields of sludge, biowaste and soil”, the secretariat of which is held by DIN.

This document is currently submitted to the second CEN Enquiry.

This draft European Standard prEN 15933 was completely technically and editorially revised following the comments made during the 1st CEN-Enquiry in 2009 and the discussions from CEN/TC 400/WG 4 "Inorganic elements and compounds".

This European Standard is part of a modular horizontal approach in which this document belongs to the analytical step.

The preparation of this document by CEN is based on a mandate by the European Commission (Mandate M/330). The mandate considers standards on sampling and analytical methods for hygienic and biological parameters as well as inorganic and organic determinants. It was the aim of the mandate to develop standards that are applicable to sludge, treated biowaste and soil and lead to equivalent results as far as this is technically feasible.

Until now test methods determining properties of materials within the environmental area were prepared in Technical Committees (TCs) working on specific products/matrices (soil, waste, sludge etc). However, it is recognized that many steps in test procedures can be used in test procedures for other products/matrices. By careful determination of these steps and selection of specific questions within these steps, elements of the test procedure can be described in a way that can be used for more matrices and materials with certain specifications. This optimization is in line with the development among end-users of standards. A majority of routine environmental analyses are carried out by institutions and laboratories working under a scope which is not limited to one single environmental matrix but covers a wide variety of matrices. Availability of standards covering more matrices contributes to the optimization of laboratory procedures and standard maintenance costs, e.g. costs related to accreditation and recognition.

A horizontal modular approach was developed in the project 'Horizontal'. 'Modular' means that a test standard developed in this approach concerns a specific step in assessing a property and not the whole ”chain of measurement” (from sampling to analyses). A beneficial feature of this approach is that “modules” can be replaced by better ones without jeopardizing the standard “chain”.

The results of the desk study as well as the evaluation and validation studies have been subject to discussions with all parties concerned in the CEN structure during the development by project 'Horizontal'. The results of these consultations with interested parties in the CEN structure have been presented to and discussed in CEN/TC 400.

Based on data from interlaboratory studies and consultations with interested parties within CEN member bodies, it has been concluded that this draft standard prEN 15933 is acceptable for its intended use and is ready for the CEN enquiry.

It is recognized that standardization in the environmental field in most national standardization bodies is organized in national standardization committees that mirror the vertical structure of technical committees in the environmental field in CEN. The present CEN enquiry therefore asks for a special attention by the NSBs to assure that the relevant and interested parties are consulted during the CEN enquiry, i.e. to assure that one single consolidated enquiry reply on this draft standard prEN 15933 can be presented by the NSB that covers the entire scope of this draft standard.

Introduction

This European Standard is validated for several types of matrices as indicated below (see also Annex A for the results of the validation):

Table 1 — Matrices for which this European Standard is (applicable and) validated

Matrix	Validated for
Sludge	Municipal sludge
Biowaste	Compost
Soil	Arable soil
	Forest soil

WARNING — Persons using this European Standard should be familiar with normal laboratory practice. This European Standard does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user to establish appropriate safety and health practices and to ensure compliance with any national regulatory conditions.

IMPORTANT — It is absolutely essential that tests conducted according to this European Standard be carried out by suitably trained staff.

1 Scope

This European Standard describes a method for the determination of pH using a glass electrode in a 1:5-suspension (volume fraction) of sludge, treated biowaste or soil in either water (pH-H_2O), or a 0,01 mol/l calcium chloride solution (pH-$CaCl_2$).

The standard is applicable to sludge, treated biowaste or both fresh and air-dry soil samples.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

prEN WI 00400022*, Sludge, treated biowaste and soil — Guidance for sample pretreatment*

EN ISO 3696, *Water for analytical laboratory use — Specification and test methods*

3 Principle

The pH is measured in a suspension of a test portion which is made up in five times its volume of either water or a 0,01-mol/l-solution of calcium chloride solution.

NOTE To make the procedure generally applicable to all types of sludge, treated biowaste and soil samples, a volume fraction ratio is chosen because then all types of samples can be treated in the same way. For the purpose of this standard, taking the required volume of test portion with a measuring spoon is sufficiently accurate. Measurements in liquid sludge should only be made directly in the water suspension. No extra water should be added.

4 Interferences

In samples with high content of charged particles (e. g. organic matter, clay) the suspension effect can modify the potential difference between the electrodes, and thereby have an influence on the recorded pH value. This problem is minimized by gentle stirring of the suspension. For calcareous material, carbon dioxide may be absorbed by the suspension, which makes it difficult to reach an equilibrium value. Other sources of error are associated with materials containing sulphidic minerals or volatile acids.

5 Reagents

Only use reagents of recognized analytical grade.

5.1 Water, grade 2 as specified in EN ISO 3696.

The water shall have a specific electric conductivity not greater than 0,2 mS/m at 25 °C, and a pH not greater than 5,6.

5.2 Calcium chloride solution, $c(CaCl_2)$ = 0,01 mol/l.

Dissolve 1,47 g calcium chloride dihydrate ($CaCl_2 \cdot 2H_2O$) in water (5.1) and dilute to 1 000 ml.

This solution may be stored for several months in a refrigerator in a closed volumetric flask or other type of closed glass vessel.

5.3 Buffer solution, for calibration of the pH-meter.

Use at least two of the following buffer solutions for calibration. Commercially available buffer solutions of similar or equivalent pH may also be used.

Freshly prepared, the buffer solutions 5.3.1, 5.3.2 and 5.3.3 are stable for one month when stored in polyethylene bottles.

5.3.1 Buffer solution pH = 4,00, at 20 °C

Dissolve 10,21 g potassium hydrogen phthalate ($C_8H_5O_4K$) in water (5.1) and dilute to 1 000 ml.

Potassium hydrogen phthalate shall be dried before use for 2 h at 115 °C± 5 °C.

5.3.2 Buffer solution pH = 6,88, at 20 °C

Dissolve 3,39 g potassium dihydrogen phosphate (KH_2PO_4) and 3,53 g disodium hydrogen phosphate (Na_2HPO_4) in water (5.1) and dilute to 1 000 ml.

Potassium dihydrogen phosphate shall be dried before use for 2 h at 115 °C± 5 °C.

5.3.3 **Buffer solution pH = 9,22,** at 20 °C

Dissolve 3,80 g disodium tetraborate decahydrate ($Na_2B_4O_7 \cdot 10H_2O$) in water (5.1) and dilute to 1 000 ml.

NOTE Disodium tetraborate decahydrate may lose water of crystallization when stored for a long time.

6 Apparatus

6.1 pH-meter, with slope adjustment and temperature control, readable to two decimal points.

6.2 Combined glass electrode

6.3 Shaking device

6.4 Measuring spoon

6.5 Bottle, polyethylene (PE) or glass with tightly fitting cap or stopper.

7 Sample pretreatment

Samples should be pretreated according to prEN WI 00400022.

For treated biowaste particle sizes can be between 10 mm and 40 mm. Therefore, treated biowaste samples are measured without pretreatment.

8 Procedure

8.1 Preparation of a suspension

Take a test portion of at least 5 ml from the test sample using a measuring spoon (6.4). Place the test portion in a bottle (6.5). Add water (5.1) or calcium chloride solution (5.2) at an amount which is 5 times the volume of the test portion.

For treated biowaste the procedure and volume ratio (1:5) is the same, except that at least 60 ml sample volume is added to 300 ml of either water or calcium chloride solution.

Shake or mix the suspension for 60 min ± 10 min using a shaking device. Allow the suspension to settle for at least 1 h, but not longer than 3 h.

Ingress of air during standing after shaking should be avoided.

8.2 Calibration of the pH-meter

Adjust the pH-meter (6.1) as indicated in the manufacturer's manual. Calibrate the pH-meter as specified in the manufacturer's manual, using buffer solutions (5.3) at (20 ± 2) °C.

Depending on the expected pH-range start the calibration of the pH-meter with buffer solution pH 6,88 (5.3.2) and use then either buffer solution pH 4,00 (5.3.1) for acidic suspensions or buffer solution pH 9,22 (5.3.3) for alkaline suspensions.

Commercially available standards within the same pH range can also be used.

8.3 Measurement of the pH

Measure the pH in the suspension at (20 ± 2) °C immediately after or whilst being stirred. The stirring should be at such a rate to achieve a reasonably homogenous suspension of the particles, but entrainment of air should be avoided. Note the recorded values to 2 decimal places.

9 Expression of results

The results of pH-value should be reported to one decimal place.

10 Precision

The performance characteristics of the method have been evaluated (see annex A.3).

11 Test report

The test report shall contain the following information:

a) a reference to this European Standard;

b) complete identification of the sample;

c) sample preparation (e.g. fresh or air dried);

d) the aqueous used to make the suspension: water or $CaCl_2$ solution;

e) expression of results, according to clause 9;

f) any details not specified in this European Standard or which are optional, as well as any factor which may have affected the results.

Annex A
(informative)

Repeatability and reproducibility data

A.1 Materials used in the interlaboratory comparison study

The interlaboratory comparison of the determination of pH in sludge, treated biowaste and soil was carried out with 14 to 16European laboratories on 6 materials. Detailed information can be found in the final report on the interlaboratory comparison study mentioned in the Bibliography.

Table A.1 lists the types of materials tested.

Table A.1 — Materials tested analysed in the interlaboratory comparison of the determination of pH in sludge, treated biowaste and soil

Grain size	Sample	Material
Sludge (< 0,5 mm)	Sludge 1	Mix 1 of municipal waste water treatment plant sludges from North Rhine Westphalia, Germany
	Sludge 2	Mix 2 of municipal waste water treatment plant sludges from North Rhine Westphalia, Germany
Fine grained (< 2,0 mm)	Compost 1	Fresh compost from Vienna, Austria
	Compost 2	Compost from Germany
	Soil 4	Sludge amended soil from Hohenheim, Germany
	Soil 5	Agricultural soil from Reading, United Kingdom