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Sludge, treated biowaste and soil — Determination of pH

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National foreword

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The UK participation in its preparation was entrusted to Technical Committee H/-/4, Environmental testing programmes.

A list of organizations represented on this committee can be obtained on request to its secretary.

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Sludge, treated biowaste and soil - Determination of pH

Boue, biodéchet traité et sol - Détermination du pH

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

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EUROPEAN COMMITTEE FOR STANDARDIZATION
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EUROPÄISCHES KOMITEE FÜR NORMUNG

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Foreword

This document (EN 15933:2012) 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 European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by February 2013, and conflicting national standards shall be withdrawn at the latest by February 2013.

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This document has been prepared under a mandate given to CEN by the European Commission and the European Free Trade Association.

The preparation of this document by CEN is based on a mandate by the European Commission (Mandate M/330), which assigned the development of standards on sampling and analytical methods for hygienic and biological parameters as well as inorganic and organic determinants, aiming to make these standards applicable to sludge, treated biowaste and soil as far as this is technically feasible.

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Introduction

This European Standard is applicable and validated for several types of matrices as indicated in Table 1 (see also Annex A for the results of the validation).

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

Matrix	Materials used for validation
Sludge	Municipal sludge
Biowaste	Compost
Soil	Arable soil
	Forest soil

WARNING — Persons using this European Standard should be familiar with usual 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 specifies a method for the determination of pH within the range pH 2 to pH 12 in a suspension of sludge, treated biowaste or soil in either water (pH-H_2O), or a 0,01 mol/l calcium chloride solution (pH-$CaCl_2$).

This European Standard is applicable to sludge, treated biowaste and fresh or air-dry soil samples.

2 Normative references

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

EN 16179*, Sludge, treated biowaste and soil — Guidance for sample pretreatment*

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

3 Principle

The pH is measured in a suspension of a test portion which is made up in 5 times its volume of either water or a 0,01-mol/l-solution of calcium chloride.

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.

4 Interferences

In samples with a 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 sulfidic minerals or volatile acids.

5 Reagents

Use only reagents of recognized analytical grade, unless otherwise specified.

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

The water shall have a specific electric conductivity ≤ 0,2 mS/m at 25 °C, and pH ≤ 5,6.

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

Dissolve 1,47 g calcium chloride dihydrate ($CaCl_2 \cdot 2\ H_2O$) 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 (120 ± 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 (120 ± 5) °C.

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

Dissolve 3,80 g disodium tetraborate decahydrate ($Na_2B_4O_7 \cdot 10\ H_2O$) 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, readable to two decimal points. The device shall allow calibration of the pH-electrode for zero and the slope of the function. A normalisation to the standard temperature (25 °C) shall be applied.

6.2 Combined glass electrode, pH/temperature combination electrode consisting of a measurement sensor and a temperature sensor.

6.3 Shaking device

6.4 Measuring spoon

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

7 Sample pretreatment

Pretreat the samples according to EN 16179, if not otherwise specified.

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

Liquid sludge is measured directly without pretreatment.

8 Procedure

8.1 Preparation of a suspension

For solid samples 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) to a volume 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.

For liquid sludge the suspension is prepared without the addition of water. Measurements in liquid sludge shall be made directly in the liquid suspension.

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

NOTE For soils with high contents of calcium carbonate and/or magnesium carbonate the equilibration time can be longer than 3 h.

Ingress of air during standing after shaking should be avoided.

8.2 Calibration of the pH-meter

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

NOTE Temperature compensation can be used when measuring the sample.

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 may 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.

9 Expression of results

The results of the pH determinations should be reported to one decimal place.

10 Precision

The performance characteristics of the method have been evaluated, see A.2.

11 Test report

The test report shall contain at least the following information:

a) a reference to this European Standard (EN 15933);

b) complete identification of the sample;

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

d) the solution 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 16 European laboratories on six materials. Detailed information can be found in the final report on the interlaboratory comparison study mentioned in [8].

Table A.1 lists the types of materials tested.

Table A.1 — Materials tested in the interlaboratory comparison for 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

A.2 Interlaboratory comparison results

The statistical evaluation was conducted according to ISO 5725-2. The average values, the repeatability standard deviation (s_r) and the reproducibility standard deviation (s_R) were obtained (Table A.2).

Table A.2 — Results of the interlaboratory comparison studies of the determination of pH in sludge, treated biowaste and soil

Matrix	l	n	n_o	$\bar{\bar{x}}$	s_r	s_R
pH $CaCl_2$						
Sludge 1	11	52	1	6,59	0,02	0,12
Sludge 2	12	63	0	7,09	0,03	0,22
Compost 1	13	64	0	7,68	0,02	0,15
Compost 2	13	52	3	7,68	0,02	0,17
Soil 4	12	58	0	6,48	0,04	0,15
Soil 5	12	55	2	6,64	0,04	0,14
pH aq						
Sludge 1	16	69	2	6,74	0,05	0,10
Sludge 2	18	80	2	7,48	0,04	0,18
Compost 1	16	70	2	8,13	0,04	0,08
Compost 2	18	80	3	8,39	0,05	0,16
Soil 4	16	66	2	7,05	0,04	0,18
Soil 5	14	51	3	7,12	0,03	0,15

Explanation of symbols

l	number of participating laboratories
n	number of analytical results after outlier rejection
n_o	number of outliers
$\bar{\bar{x}}$	total mean of results (without outliers)
s_r	repeatability standard deviation
s_R	reproducibility standard deviation
pH $CaCl_2$	pH in 0,01 mol/l calcium chloride solution
pH aq	pH in water suspension

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[4] ISO 1770, *Solid-stem general purpose thermometers*

[5] ISO 5725-2, *Accuracy (trueness and precision) of measurement methods and results — Part 2: Basic method for the determination of repeatability and reproducibility of a standard measurement method*

[6] ISO 10390, *Soil quality — Determination of pH*

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