
**Soil, treated biowaste and sludge –
Determination of pH**

Sols, biodéchets traités et boues — Détermination du pH

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

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).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation of 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 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*.

This third edition cancels and replaces the second edition (ISO 10390:2005), which has been technically revised. The main changes compared to the previous edition are as follows:

- The content of ISO 10390:2005 and EN 15933:2012 were merged;
- The scope was widened to include sludge and treated biowaste;
- Additional validation data for soil, sludge and treated biowaste were included;
- The text was editorially revised.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Soil, treated biowaste and sludge – Determination of pH

1 Scope

This document specifies an instrumental method for the routine determination of pH within the range pH 2 to pH 12 using a glass electrode in a 1:5 (volume fraction) suspension of soil, sludge and treated biowaste in either water (pH in H₂O), in 1 mol/l potassium chloride solution (pH in KCl) or in 0,01 mol/l calcium chloride solution (pH in CaCl₂).

This document is applicable to all types of air-dried soil and treated biowaste samples.

NOTE For example, pretreated in accordance with ISO 11464 or EN 16179 or EN 15002.

2 Normative references

There are no normative references in this document.

3 Terms and definitions

No terms and definitions are listed in this document.

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/>

4 Principle

A suspension of a test portion is made up in five times its volume with one of the following solutions:

- water;
- a solution of potassium chloride (KCl) in water, $c = 1$ mol/l;
- a solution of calcium chloride (CaCl₂) in water, $c = 0,01$ mol/l.

The pH of the suspension is measured using a pH-meter.

NOTE 1 To make the procedure generally applicable to all types of soil, treated biowaste samples and sludges except liquid sludge, a volume-to-volume shaking ratio is chosen because all test samples can be treated in the same way. If a mass-to-volume ratio were chosen, the weighed amount of test sample would have to be adapted for e.g. soils with a low density, to enable the preparation of the suspension. For the purpose of this document, it is sufficiently accurate to measure the required volume of test portion with a measuring spoon.

NOTE 2 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 can 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.

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5.1 Water, with a specific conductivity not higher than 0,2 mS/m at 25 °C.

5.2 Potassium chloride solution, $c(\text{KCl}) = 1 \text{ mol/l}$

Dissolve 74,5 g of potassium chloride in water (5.1) and dilute to 1 000 ml.

5.3 Calcium chloride solution, $c(\text{CaCl}_2) = 0,01 \text{ mol/l}$

Dissolve 1,47 g of calcium chloride dihydrate ($\text{CaCl}_2 \cdot 2\text{H}_2\text{O}$) in water (5.1) and dilute to 1 000 ml.

5.4 Buffer solutions, for calibration of the pH-meter

Use at least two of the following buffer solutions for calibration. Buffer solutions having a similar or equivalent pH that are commercially available may also be used.

NOTE 1 The buffer solutions 5.4.1, 5.4.2 and 5.4.3 are stable for one month when stored in polyethylene bottles.

NOTE 2 If automated or semi-automated systems are used, buffers recommended by the manufacturer or commercially available buffers can be used.

5.4.1 Buffer solution, pH 4,00 at 20 °C

Dissolve 10,21 g of potassium hydrogen phthalate ($\text{C}_8\text{H}_5\text{O}_4\text{K}$) in water (5.1) and dilute to 1 000 ml.

The potassium hydrogen phthalate shall be dried before use for 2 h at $115 \text{ °C} \pm 5 \text{ °C}$.

5.4.2 Buffer solution, pH 6,88 at 20 °C

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

The potassium dihydrogen phosphate shall be dried before use for 2 h at $115 \text{ °C} \pm 5 \text{ °C}$.

5.4.3 Buffer solution, pH 9,22 at 20 °C

Dissolve 3,80 g of disodium tetraborate decahydrate ($\text{Na}_2\text{B}_4\text{O}_7 \cdot 10\text{H}_2\text{O}$) in water (5.1) and dilute to 1 000 ml.

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

6 Apparatus

6.1 Shaking or mixing machine

6.2 pH-meter, with slope adjustment and temperature control, readable to two decimals.

6.3 Glass electrode and reference electrode, or combined electrode of equivalent performance

In the case of pH values greater than 10, an electrode specifically designed for that range should be used or an additional buffer solution at pH 12 should be used.

NOTE In case of soils, the danger of deterioration of performance caused by breakage or contamination of the electrodes is increased.

6.4 Thermometer or temperature probe, capable of measuring to the nearest 1 °C.

6.5 Sample bottle, of capacity at least 50 ml, a suitable bottle allowing effective shaking or mixing and measurement made of (borosilicate) glass or polyethylene where appropriate fitted with a tightly fitting cap or stopper.

6.6 Spoon, of known capacity of at least 5,0 ml.

7 Laboratory sample

Use the fraction of particles of air-dried samples, or samples dried at a temperature not higher than 40 °C, which passes through a sieve with a 2 mm mesh size.

NOTE 1 For example, samples pretreated according to ISO 11464 or EN 16179 can be used (see bibliography).

NOTE 2 Drying can influence the pH of the soil. In some soil samples, particularly those containing sulfides, drying can lower the pH substantially.

NOTE 3 For treated biowaste, particle sizes can be between 10 mm and 40 mm. Therefore, these treated biowaste samples are measured without pretreatment.

For the determination in a field moist sample, a homogenized sample is used.

NOTE For example, samples pretreated according to EN 16179 can be used (see Bibliography).

Liquid sludges (samples with low dry matter content) are measured directly without pretreatment.

NOTE For example, samples pretreated according to EN 15002 can be used (see Bibliography).

8 Procedure

8.1 Preparation of the suspension

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8.1.1 Take a representative test portion of at least 5 ml from the laboratory sample using the spoon (6.6).

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

8.1.2 Transfer the test portion into the sample bottle (6.5) and add five times its volume of either water (5.1), potassium chloride solution (5.2) or calcium chloride solution (5.3) according to purpose.

For treated biowaste without pretreatment, 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.

8.1.3 Shake or mix the suspension for 60 min ± 10 min, using the mechanical shaker or mixer (6.1), and wait at least 1 h but not longer than 3 h.

Ingress of air during standing after shaking should be avoided.

NOTE Different shaking or mixing procedure and waiting time can be used if laboratory proves that comparable results are obtained.

8.2 Calibration of the pH-meter

Adjust the pH-meter as indicated in the manufacturer's manual.

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Calibrate the pH-meter as specified in the manufacturer's manual, using the buffer solutions (5.4) at $20\text{ °C} \pm 2\text{ °C}$.

NOTE Using electrodes that are in good condition, equilibrium is usually reached within 30 s. Temperature compensation can be used when measuring the sample.

8.3 Measurement of the pH

Measure the pH in the suspension at $20\text{ °C} \pm 2\text{ °C}$ whilst being stirred or immediately after. The stirring should be at such a rate to achieve a reasonably homogeneous suspension of the sample particles, but entrainment of air should be avoided. Read the pH after stabilization of the value is reached. Note the recorded value to two decimal places. In case stabilization of the pH value is not reached a note shall be added to the report (see 10e).

NOTE 1 The reading can be considered stable for example when the pH measured over a period of 5 s varies by not more than 0,02 pH units. The time required for stabilization is usually 1 min or less, but can depend on a number of factors including:

- the value of the pH (at high pH-values, it is more difficult to reach stabilization);
- the quality of the glass electrode (differences in manufacture between electrodes) and its age;
- the medium in which the pH is measured (stabilization is reached faster in a KCl or CaCl₂ medium than in water);
- the differences in pH between samples in a series;
- mechanical mixing before or while the measurement is performed can help to achieve stable readings in a shorter time.

NOTE 2 In samples with a high content of organic material (peat soils, pot soils, etc.), the suspension effect can play a role. For calcareous soils, it is possible that carbon dioxide is absorbed by the suspension. Under these circumstances, it is difficult to reach an equilibrium pH-value.

9 Precision

The performance characteristics of the method have been evaluated and a summary of the results of interlaboratory trials for the determination of the pH are given in [Annex A](#).

10 Test report

The test report shall include the following information:

- a) a reference to ISO 10390:-;
- b) all information necessary for complete identification of the sample;
- c) the aqueous medium used to make the suspension or in case of liquid sludge direct measurement in the test portion;
- d) the results of the determinations, to the nearest 0,1 pH-unit;
- e) any difficulties experienced in establishing equilibrium conditions;
- f) details of any operation not specified in this document or regarded as optional, as well as any factor which may have affected the results.

Annex A (informative)

Repeatability and reproducibility data

A.1 General

Following are repeatability and reproducibility data of interlaboratory trials carried out by ISO/TC 190 for the validation of ISO 10390:2005 (see A.2), and those carried out by CEN/TC 400 for the validation of EN 15933:2012 (see A.3). Whilst the validation trial for ISO 10390 included pH determinations based on suspensions with water, CaCl₂ and KCl, validation for EN 15933 did not consider suspensions made with KCl.

A.2 Results of an interlaboratory trial for the determination of the pH in soils

An interlaboratory trial was organized in 2004 to test the procedures specified in this document.

For this interlaboratory trial, the determination of the pH of four soils was carried out by 35 laboratories.

The summary of the results of the interlaboratory trial is presented in Tables A.1 to A.3.

Sample 1 (cultivated sandy soil) and sample 2 (clayey cultivated soil) came from the Czech Republic. Samples 3 and 4 (both loamy cultivated soils) came from France.

The repeatability value, r , and the reproducibility value, R , listed in Tables A.1 to A.3, were calculated according to ISO 5725-2.

Table A.1 — Results of the interlaboratory trial for the determination of the pH in H₂O

Sample No.	1	2	3	4
Number of laboratories retained after eliminating outliers	35	33	32	33
Number of outliers (laboratories)	0	2	3	2
Number of accepted results	70	66	64	66
Mean value	5,72	7,60	8,08	6,40
Repeatability value ($r = 2,8 \cdot s_r$)	0,08	0,12	0,10	0,10
Reproducibility value ($R = 2,8 \cdot s_R$)	0,79	0,45	0,42	0,36

Table A.2 — Results of the interlaboratory trial for the determination of the pH in KCl

Sample No.	1	2	3	4
Number of laboratories retained after eliminating outliers	35	35	34	33
Number of outliers (laboratories)	0	0	1	2
Number of accepted results	70	70	68	66
Mean value	5,00	7,13	7,38	5,67
Repeatability value ($r = 2,8 \cdot s_r$)	0,09	0,08	0,08	0,14
Reproducibility value ($R = 2,8 \cdot s_R$)	0,47	0,37	0,36	0,25

Table A.3 — Results of the interlaboratory trial for the determination of the pH in CaCl₂

Sample No.	1	2	3	4
Number of laboratories retained after eliminating outliers	33	33	32	34
Number of outliers (laboratories)	2	2	3	1
Number of accepted results	66	66	64	68
Mean value	5,15	6,98	7,45	5,81
Repeatability value ($r = 2,8 \cdot s_r$)	0,07	0,06	0,06	0,09
Reproducibility value ($R = 2,8 \cdot s_R$)	0,37	0,25	0,32	0,33

A.3 Results of an interlaboratory trial for the determination of the pH in soils, sludge and treated biowaste

A.3.1 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 18 European laboratories on six materials.

[Table A.4](#) lists the types of materials tested.

Table A.4 — Materials tested in the interlaboratory comparison for the determination of pH in soil, sludge and treated biowaste

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.3.2 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.5](#)).