SLOVENSKI STANDARD
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Tla, blato in obdelani biološki odpadki - Določevanje pH (ISO/DIS 10390:2020)

Soil, sludge and treated biowaste - Determination of pH (ISO/DIS 10390:2020)

Boden, Schlamm und behandelter Bioabfall - Bestimmung des pH-Wertes (ISO/DIS 10390:2020)

Terre, boues et biodéchets traités - Détermination du pH (ISO/DIS 10390:2020)

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Soil, sludge and treated biowaste – Determination of pH

Terre, boues et biodéchets traités — 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).

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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 15933:2012-11 were merged;

— The text was editorially revised.

Soil, sludge and treated biowaste – 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 water (pH in H_2O), in 1 mol/l potassium chloride solution (pH in KCl) or in 0,01 mol/l calcium chloride solution (pH in $CaCl_2$).

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

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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.

ISO 1770:1981, *Solid-stem general purpose thermometers*

3 Principle

A suspension of a testportion 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 ($CaCI_2$) 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 except liquid sludge, a volume-to-volume shaking ratio is chosen because then all soil 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 International Standard, taking the required volume of test portion with a measuring spoon is sufficiently accurate.

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 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.

4 Reagents

Use only reagents of recognized analytical grade.

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

4.2 Potassium chloride solution, c(KCl) = 1 mol/l

Dissolve 74,5 g of potassium chloride in water (4.1) and dilute to 1 000 ml.

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

Dissolve 1,47 g of calcium chloride dihydrate ($CaCl_2 \cdot 2H_2O$) in water (4.1) and dilute to 1 000 ml.

4.4 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 4.4.1, 4.4.2 and 4.4.3 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.

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

Dissolve 10,21 g of potassium hydrogen phthalate ($C_8H_5O_4K$) in water (4.1) and dilute to 1 000 ml.

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

4.4.2 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 (4.1) and dilute to 1 000 ml.

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

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

Dissolve 3,80 g of disodium tetraborate decahydrate ($Na_2B_4O_7 \cdot 10H_2O$) in water (4.1) and dilute to 1 000 ml .

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

5 Apparatus

5.1 Shaking or mixing machine

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

5.3 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 soil systems, the danger of deterioration of performance caused by breakage or contamination of the electrodes is increased.

5.4 Thermometer or temperature probe, capable of measuring to the nearest 1 °C, complying with type C according to ISO 1770:1981.

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

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

6 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 examples, 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.

Preparation of field moist samples is described in EN 16179.

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).

7 Procedure

7.1 Preparation of the suspension

7.1.1 Take a representative test portion of at least 5 ml from the laboratory sample using the spoon (5.6).

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.

7.1.2 Transfer the test portion in the sample bottle (5.5) and add five times its volume of either water (4.1), potassium chloride solution (4.2) or calcium chloride solution (4.3) 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.

7.1.3 Shake or mix the suspension for 60 min ± 10 min, using the mechanical shaker or mixer (5.1), 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 procedure and waiting time can be used if laboratory proves that comparable results are obtained.

7.2 Calibration of the pH-meter

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

Calibrate the pH-meter as specified in the manufacturer´s manual, using the buffer solutions (4.4) at 20 °C ± 2 °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

7.3 Measurement of the pH

Measure the pH in the suspension at 20 °C ± 2 °C immediately after or whilst being stirred. The stirring should be at such a rate to achieve a reasonably homogeneous suspension of the soil 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 9e).

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 quicker in a KCl or $CaCl_2$ medium than in water);

— the differences in pH between samples in a series;

— mechanical mixing before or while the measurement is performed may 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.

8 Repeatability

The repeatability, expressed in terms of the difference between the pH measurements in two separately prepared suspensions, shall satisfy the requirements given in Table 1.

A summary of the results of an interlaboratory trial for the determination of the pH of soils is given in Annex A.

Table 1 — Acceptable repeatability of pH measurement

pH range	Acceptable difference
pH < 7,00	0,15
7,00 < pH < 7,50	0,20
7,50 ⩽ pH ⩽ 8,00	0,30
pH > 8,00	0,40

9 Test report

The test report shall include the following information:

a) a reference to this document;

b) all information necessary for complete identification of the sample;

c) the aqueous medium used to make the suspension; whether the pH in H_2O, pH in KCl solution or pH in $CaCl_2$ solution has been determined;

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.