INTERNATIONAL STANDARD

ISO
10390

Soil quality — Determination of pH

Qualité du sol — Détermination du pH

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.

Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

International Standard ISO 10390 was prepared by Technical Committee ISO/TC 190, *Soil quality*, Subcommittee SC 3, *Chemical methods and soil characteristics*.

Annex A of this International Standard is for information only.

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Soil quality — Determination of pH

1 Scope

This International Standard specifies an instrumental method for the routine determination of pH using a glass electrode in a 1:5 (V/V) suspension of soil in water (pH-H_2O), in a solution of 1 mol/l potassium chloride (pH-KCl) or in a solution of 0,01 mol/l calcium chloride (pH-$CaCl_2$).

This International Standard is applicable to all types of air-dried soil samples, for example pretreated according to ISO 11464.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

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

ISO 3696:1987, *Water for analytical laboratory use — Specification and test methods.*

ISO 11464:—[1], *Soil quality — Pretreatment of samples for physico-chemical analyses.*

3 Principle

A suspension of soil is made up in five times its volume of one of the following:

— water;

— a 1 mol/l solution of potassium chloride (KCl) in water;

— a 0,01 mol/l solution of calcium chloride ($CaCl_2$) in water.

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

NOTE 1   To make the procedure generally applicable to all types of soil samples, a V/V shaking ratio is chosen because then all soils can be treated in the same way. If a m/V ratio were chosen, the weighed amount of test sample would have to be adapted for 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.

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 and a pH greater than 5,6 (grade 2 water according to ISO 3696).

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 at 20 °C.

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

Dissolve 1,47 g of calcium chloride dihydrate ($CaCl_2.2H_2O$) in water (4.1) and dilute to 1 000 ml at 20 °C.

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1) To be published.

4.4 Solutions for the calibration of the pH-meter.

Use at least two of the following calibration solutions.

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 at 20 °C.

The potassium hydrogen phthalate shall be dried before use for 2 h at 110 °C to 120 °C.

4.4.2 Buffer solution, pH 7,00 at 20 °C.

Dissolve 3,800 g of potassium dihydrogen phosphate (KH_2PO_4) and 3,415 g of disodium hydrogen phosphate (Na_2HPO_4) in water (4.1) and dilute to 1 000 ml at 20 °C.

Potassium dihydrogen phosphate shall be dried before use for 2 h at 110 °C to 120 °C.

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

Dissolve 3,80 g of disodium tetraborate decahydrate ($Na_2B_4O_7.10H_2O$) in water (4.1) and dilute to 1 000 ml at 20 °C.

NOTES

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

3 The buffer solutions 4.4.1, 4.4.2 and 4.4.3 are stable for one month if stored in polyethylene bottles.

4 Buffer solutions that are commercially available may also be used.

5 Apparatus

5.1 Shaking or mixing machine.

5.2 pH-meter, with slope adjustment and temperature control

5.3 Glass electrode and a reference electrode, or a combined electrode of equivalent performance.

NOTES

5 In the case of pH-values greater than 10, an electrode specifically designed for that range should be used.

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

5.4 Thermometer, capable of measuring to the nearest 1 °C, complying with type C according to ISO 1770.

5.5 Sample bottle, of capacity at least 50 ml, made of borosilicate glass or polyethylene with a tightly fitting cap or stopper.

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

6 Laboratory sample

Use the fraction of particles of air-dried soil samples, or soil samples dried at a temperature not higher than 40 °C, which passes through a square hole sieve with a 2 mm aperture. For example, soil samples pretreated according to ISO 11464 can be used.

NOTE 7 Drying may influence the pH of the soil. In some soil samples, particularly those containing sulfides, drying can lower the pH substantially.

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).

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

7.1.3 Shake or mix the suspension vigorously, for 5 min, using the mechanical shaker or mixer (5.1), and wait at least 2 h, but not longer than 24 h.

NOTES

8 In most soils, equilibrium is reached within 2 h. When this is the case, measurements can be made after 2 h.

9 In some contaminated soils, recently limed soils or carbonate-containing soils, the equilibrium pH may not be reached within the time specified in 7.1.3. Consequently, values which are too low or too high, compared to naturally achieved equilibrium situations, will be obtained due to slow changes in the buffering system. To check whether this is the case, pH-measurements should be made according to this International Standard at least two different times between 2 h and 24 h of shaking. In this case, two or more pH-values should be reported to indicate the approximate pH of the soil, and the fact that the measurement is not stable should be reported in item e) of the test report.

7.2 Calibration of the pH-meter

Calibrate the pH-meter as prescribed in the manufacturer's manual, using the buffer solutions given in 4.4.

NOTE 10   Using electrodes that are in good condition, equilibrium is normally reached within 30 s.

7.3 Measurement of the pH

Adjust the pH-meter as indicated in the manufacturer's manual. Measure the temperature of the suspension and take care that the temperature of the buffer solution and the soil suspension do not differ by more than 1 °C. Shake the suspension thoroughly just before measurement of the pH. Measure the pH in the settling suspension. Read the pH after stabilization is reached. Note the recorded values to two decimal places.

NOTES

11   If a swinging needle pH-meter is used, the second decimal place should be estimated.

12   The reading may be considered stable when the pH measured over a period of 5 s varies by not more than 0,02 pH-unit. The time required for stabilization is usually 1 min or less, but may 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 of manufacture between electrodes) and its age;

— the medium in which the pH is measured (stabilization is reached more quickly in a KCl or $CaCl_2$ medium than in water);

— the differences in pH between samples in a series;

— mechanical mixing before the measurement is performed which may help to achieve stable readings in a shorter time.

13   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 and reproducibility

The repeatability of the pH-measurement in two separately prepared suspensions shall satisfy the following demands:

Table 1 — Repeatability

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

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

9 Test report

The test report shall include the following information:

a)   a reference to this International Standard;

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

c)   the aqueous medium used to make the suspension: whether the pH-H_2O, pH-KCl or pH-$CaCl_2$ 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 operations not specified in this International Standard or regarded as optional, as well as any factor which may have affected the results.

Annex A

(informative)

The results of an interlaboratory trial for the determination of the pH of soils

An interlaboratory trial was organized in 1990 by the Wageningen Agricultural University, to test the procedures specified in this International Standard.

For this interlaboratory trial, the determination of the pH of five soils was carried out by 30 laboratories.

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

Samples 1 and 4 included in these tables were duplicate samples collected in France from a soil originat-

ing from granitic rock. Sample 2, a löess soil, also came from France. Samples 3 and 5 were taken in the Netherlands; sample 3 was a peaty soil and sample 5 was a sandy soil.

The repeatability, r, and the reproducibility, R, listed in these tables were calculated according to ISO 5725:1986, *Precision of test methods — Determination of repeatability and reproducibility for a standard test method by inter-laboratory tests.*

Table A.1 — The results of the interlaboratory trial for the determination of the pH-H$_2$O

Sample No.	1	2	3	4	5
Number of laboratories retained after eliminating outliers	30	30	30	30	30
Number of outliers (laboratories)	—	—	—	—	—
Number of accepted results	60	60	60	60	60
Mean value	8,066	8,259	5,469	8,086	4,500
Standard deviation of the repeatability (S_r)	0,066	0,071	0,102	0,091	0,078
Relative standard deviation of the repeatability (%)	0,819	0,854	1,867	1,122	1,735
Repeatability limit ($r = 2,8 \times S_r$)	0,185	0,198	0,286	0,254	0,219
Standard deviation of the reproducibility (S_R)	0,276	0,232	0,182	0,259	0,177
Relative standard deviation of the reproducibility (%)	3,423	2,809	3,327	3,198	3,927
Reproducibility limit ($R = 2,8 \times S_R$)	0,773	0,650	0,509	0,724	0,495

Table A.2 — The results of the interlaboratory trial for the determination of the pH-KCl

Sample No.	1	2	3	4	5
Number of laboratories retained after eliminating outliers	30	30	30	30	30
Number of outliers (laboratories)	—	—	—	—	—
Number of accepted results	60	60	60	60	60
Mean value	7,487	7,683	4,883	7,506	4,159
Standard deviation of the repeatability (S_r)	0,076	0,054	0,066	0,067	0,056
Relative standard deviation of the repeatability (%)	1,013	0,706	1,355	0,888	1,343
Repeatability limit ($r = 2,8 \times S_r$)	0,212	0,152	0,185	0,187	0,156
Standard deviation of the reproducibility (S_R)	0,190	0,179	0,129	0,185	0,127
Relative standard deviation of the reproducibility (%)	2,544	2,334	2,637	2,464	3,055
Reproducibility limit ($R = 2,8 \times S_R$)	0,533	0,502	0,361	0,518	0,356

Table A.3 — The results of the interlaboratory trial for the determination of the pH-CaCl$_2$

Sample No.	1	2	3	4	5
Number of laboratories retained after eliminating outliers	30	30	30	30	30
Number of outliers (laboratories)	—	—	—	—	—
Number of accepted results	60	60	60	60	60
Mean value	7,374	7,410	4,927	7,381	4,260
Standard deviation of the repeatability (S_r)	0,075	0,058	0,039	0,075	0,052
Relative standard deviation of the repeatability (%)	1,022	0,787	0,785	1,022	1,231
Repeatability limit ($r = 2,8 \times S_r$)	0,211	0,163	0,108	0,211	0,147
Standard deviation of the reproducibility (S_R)	0,260	0,240	0,173	0,236	0,178
Relative standard deviation of the reproductibility (%)	3,520	3,234	3,513	3,198	4,183
Reproducibility limit ($R = 2,8 \times S_R$)	0,727	0,671	0,485	0,661	0,499

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