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INTERNATIONAL STANDARD

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Soil quality — Determination of pH

Qualité du sol — Détermination du pH



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Foreword

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International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. 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.

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.

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

This second edition cancels and replaces the first edition (ISO 10390:1994), which has been technically revised.

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 (volume fraction) suspension of soil in 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 International Standard is applicable to all types of air-dried soil samples, for example pretreated in accordance with ISO 11464.

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.

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

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

3 Principle

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

- 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 To make the procedure generally applicable to all types of soil samples, a volume-to-volume shaking ratio is chosen because then all soils 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 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 in accordance with ISO 3696:1987).

ISO 10390:2005(E)**4.2 Potassium chloride solution**, $c(\text{KCl}) = 1 \text{ mol/l}$

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

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

Dissolve 1,47 g of calcium chloride dihydrate ($\text{CaCl}_2 \cdot 2\text{H}_2\text{O}$) in water (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 The buffer solutions 4.4.1, 4.4.2 and 4.4.3 are stable for one month when stored in polyethylene bottles.

4.4.1 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 (4.1) and dilute to 1 000 ml.

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

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 $115 \text{ °C} \pm 5 \text{ °C}$.

4.4.3 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 (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**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.

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, made of borosilicate glass or polyethylene 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 soil samples, or soil samples dried at a temperature not higher than 40 °C, which passes through a sieve with a 2 mm mesh size. For example, soil samples pretreated according to ISO 11464 can be used.

NOTE Drying can 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 for 60 min \pm 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.

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 \pm 2 °C.

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

7.3 Measurement of the pH

Measure the pH in the suspension at 20 °C \pm 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.

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

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 CaCl₂ 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.

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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 |
|---------------------------------|-----------------------|
| $\text{pH} \leq 7,00$ | 0,15 |
| $7,00 < \text{pH} < 7,50$ | 0,20 |
| $7,50 \leq \text{pH} \leq 8,00$ | 0,30 |
| $\text{pH} > 8,00$ | 0,40 |

9 Test report

The test report shall include the following information:

- a reference to this International Standard;
- all information necessary for complete identification of the sample;
- the aqueous medium used to make the suspension; whether the pH in H₂O, pH in KCl solution or pH in CaCl₂ solution has been determined;
- the results of the determinations, to the nearest 0,1 pH-unit;
- any difficulties experienced in establishing equilibrium conditions;
- details of any operation not specified in this International Standard or regarded as optional, as well as any factor which may have affected the results.

Annex A (informative)

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 International Standard.

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 ($r = 2,8 s_r$) | 0,08 | 0,12 | 0,10 | 0,10 |
| Reproducibility value ($R = 2,8 s_R$) | 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 ($r = 2,8 s_r$) | 0,09 | 0,08 | 0,08 | 0,14 |
| Reproducibility value ($R = 2,8 s_R$) | 0,47 | 0,37 | 0,36 | 0,25 |

ISO 10390:2005(E)**Table A.3 — Results of the interlaboratory trial for the determination of the pH in CaCl_2**

| 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 ($r = 2,8 s_r$) | 0,07 | 0,06 | 0,06 | 0,09 |
| Reproducibility value ($R = 2,8 s_R$) | 0,37 | 0,25 | 0,32 | 0,33 |

Bibliography

- [1] ISO 5725-2:1994, *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*
- [2] ISO 11464:1994, *Soil quality — Pretreatment of samples for physico-chemical analyses*

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