INTERNATIONAL STANDARD

ISO 11263

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Soil quality — Determination of phosphorus — Spectrometric determination of phosphorus soluble in sodium hydrogen carbonate solution

Qualité du sol — Dosage du phosphore — Dosage spectrométrique du phosphore soluble dans une solution d'hydrogénocarbonate de sodium

Reference number
ISO 11263:1994(E)

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 11263 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 phosphorus — Spectrometric determination of phosphorus soluble in sodium hydrogen carbonate solution

1 Scope

This International Standard specifies an extraction method and analytical conditions to determine the content of soil phosphorus soluble in sodium hydrogen carbonate solution. After the extraction step, two different methods of colour development are possible. Subclause 4.2 specifies colour development at room temperature. Subclause 4.3 specifies colour development after heating to a high temperature. This International Standard is applicable to all types of soils.

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

ISO 11464:1994, *Soil quality — Pretreatment of samples for physico-chemical analyses.*

ISO 11465:1993, *Soil quality — Determination of dry matter and water content on a mass basis — Gravimetric method.*

3 Principle

A soil, pretreated according to ISO 11464, is treated with a 0,5 mol/l sodium hydrogen carbonate solution at pH 8,50 to reduce the concentration of calcium, aluminium and iron(III) ions by precipitation of calcium carbonate and aluminium and iron(III) hydroxides and to release phosphate ions into solution.

The clear extract is analysed for phosphorus by a spectrometric method involving the formation of an antimony-phosphate-molybdate complex (at room temperature, 4.2) or a phosphate-molybdate complex (at a high temperature, 4.3) both reduced with ascorbic acid to form a deep-coloured, blue complex.

4 Reagents

All reagents shall be of recognized analytical grade. Use water complying with grade 2 of ISO 3696.

4.1 Reagents used in both colour developments

4.1.1 Sodium hydroxide solution, c(NaOH) = 1 mol/l.

Dissolve 40,0 g ± 0,4 g of sodium hydroxide (NaOH) pellets in water. Cool and dilute to 1 000 ml with water. Store in an inert and hermetically sealed bottle.

4.1.2 Extracting solution.

Dissolve 42,0 g ± 0,1 g of sodium hydrogen carbonate ($NaHCO_3$) in 800 ml of water. Adjust the pH to 8,50 ± 0,02 with sodium hydroxide solution (4.1.1). Transfer the solution into a 1 000 ml volumetric flask and make up to the mark with water.

NOTE 1 This reagent shall be used within 4 h of preparation.

4.1.3 Carbon, activated, allowing the absorbance of the blank, A_B, to be less than 0,015. Otherwise prepare carbon as follows.

Weigh 100 g ± 1 g of carbon in a 1 000 ml beaker and add 400 ml of extracting solution (4.1.2). Stir on a magnetic stirrer for 2 h. Filter on phosphorus-free paper and repeat the washing, using the same volume of extracting solution. Filter again and wash the carbon with water until the pH of the washings is 7,0 ± 0,1. Dry the carbon at 105 °C ± 2 °C.

4.1.4 Sulfuric acid, ρ = 1,84 g/ml.

4.1.5 Sulfuric acid, dilute, $c(H_2SO_4)$ = 5 mol/l.

Pour 400 ml ± 10 ml of water into a 1 000 ml beaker. Cautiously add 278 ml ± 5 ml of sulfuric acid (4.1.4) while stirring continuously. Cool to 20 °C ± 5 °C and make up to the mark with water.

4.1.6 Sulfomolybdic reagent.

Pour about 400 ml of water into a 1 000 ml beaker. Cautiously add 278 ml ± 5 ml of sulfuric acid (4.1.4) while stirring continuously. Cool to 50 °C. Then add 49,08 g ± 0,01 g of ammonium heptamolybdate tetrahydrate $[(NH_4)_6Mo_7O_{24}\cdot 4H_2O]$ and stir until dissolved. Cool to 20 °C ± 5 °C and make up to the mark with water.

NOTE 2 If stored in an amber glass bottle, this reagent is stable for many years.

4.1.7 Orthophosphate standard stock solution, containing 450 mg/l of phosphorus.

Weigh 1,976 g ± 0,001 g of potassium dihydrogen phosphate (KH_2PO_4), dried in an oven for 2 h at 105 °C ± 1 °C, into a 1 000 ml volumetric flask. Dissolve and make up to the mark with water.

NOTE 3 If stored at 4 °C, this solution is stable for 3 months.

4.1.8 Standard solutions.

Pipette volumes (as shown in table 1) of the standard stock solution (4.1.7) into a set of flasks of 1 000 ml nominal volume. Make up to volume with the extracting solution (4.1.2). These solutions are stable for 1 month.

Table 1 — Standard stock solutions and concentrations of phosphorus

Standard stock solution ml	Concentration of phosphorus mg/l
0,000	0,000
1,000	0,45
5,00	2,25
10,00	4,50
20,00	9,00

4.2 Reagents used for colour development at room temperature

4.2.1 Sodium thiosulfate solution, 12 g/l.

Dissolve 1,20 g ± 0,01 g of sodium thiosulfate pentahydrate ($Na_2S_2O_3\cdot 5H_2O$) in 100 ml of water. Add 50 mg ± 1 mg of sodium carbonate (Na_2CO_3) as a preservative. This solution shall be freshly prepared before use.

4.2.2 Sodium metabisulfite solution, 200 g/l.

Dissolve 20,0 g ± 0,1 g of sodium metabisulfite ($Na_2S_2O_5$) in 100 ml of water. This solution shall be freshly prepared before use.

WARNING — Sodium metabisulfite produces gas that is dangerous when inhaled.

4.2.3 Potassium antimony(III) oxide tartrate solution, 0,5 g/l.

Dissolve 0,500 g ± 0,01 g of potassium antimony(III) oxide tartrate hemihydrate $[K(SbO)C_4H_4O_6\cdot 0{,}5H_2O]$ in 1 000 ml of water.

WARNING — Antimony compounds are highly toxic.

4.2.4 Colour reagent.

Dissolve 1,00 g ± 0,01 g of ascorbic acid ($C_6H_8O_6$) in 525 ml of water. Then add

— 10 ml of dilute sulfomolybdic reagent (4.1.6),

— 15 ml of dilute sulfuric acid (4.1.5), and

— 50 ml of potassium antimony(III) oxide tartrate solution (4.2.3).

Mix well. The volume obtained is approximately 600 ml.