BRITISH STANDARD

BS 7755-3.6:
1995
ISO 11263:
1994

Soil quality —

Part 3: Chemical methods —

Section 3.6: Determination of phosphorus — Spectrometric determination of phosphorus soluble in sodium hydrogen carbonate solution

Committees responsible for this British Standard

The preparation of this British Standard was entrusted by Technical Committee EPC/48, Soil quality, to Subcommittee EPC/48/3, Chemical methods, upon which the following bodies were represented:

Association of Consulting Scientists
Association of Public Analysts
British Ceramic Research Ltd.
British Gas plc
British Society of Soil Science
Department of the Environment (Drinking Water Inspectorate)
Department of Trade and Industry (Laboratory of the Government Chemist)
National Rivers Authority
Royal Society of Chemistry
Society of Chemical Industry
Soil Survey and Land Research Centre
Water Research Centre
Water Services Association of England and Wales

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Contents

National foreword

This Section of BS 7755 has been prepared by Subcommittee EPC/48/3. It is identical with ISO 11263:1994 *Soil quality — Determination of phosphorus — Spectrometric determination of phosphorus soluble in sodium hydrogen carbonate solution*, published by the International Organization for Standardization (ISO). ISO 11263 was prepared by Subcommittee 3, Chemical methods, of Technical Committee ISO/TC 190, Soil quality, with the active participation and approval of the UK.

BS 7755 is being published in a series of Parts subdivided into Sections and Subsections that will generally correspond to particular international standards. Other Parts of BS 7755 are, or will be, as follows.

— *Part 1: Terminology and classification;*

— *Part 2: Sampling;*

— *Part 3: Chemical methods;*

— *Part 4: Biological methods;*

— *Part 5: Physical methods.*

Textual error. When adopting the text of the international standard, the following textual error was discovered. It has been marked in the text and has been reported to ISO in a proposal to amend the text of the international standard.

In **6.4.1**, line 2, “for 30 min” should read “within 30 min.”

Additional information

In **4.1.3**, it is important that low phosphorus activated charcoal is used as charcoal derived from bone contains large quantities of phosphorus and is unsuitable for use in this method.

In **6.5**, if the absorbance is greater than that of the highest standard solution, then the solution needs diluting accordingly thereby necessitating the use of the dilution factor d in the calculation.

Cross-references

International standard	British Standard
ISO 3696:1987	BS 3978:1987 *Specification for water for laboratory use* (Identical)
	BS 7755 *Soil quality*
ISO 11464:1994	Section 3.5:1995 *Pretreatment of samples for physico-chemical analyses* (Identical)
ISO 11465:1993	Section 3.1:1994 *Determination of dry matter and water content on a mass basis by a gravimetric method* (Identical)

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Summary of pages

This document comprises a front cover, an inside front cover, pages i and ii, pages 1 to 4, an inside back cover and a back cover.

This standard has been updated (see copyright date) and may have had amendments incorporated. This will be indicated in the amendment table on the inside front cover.

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*, $c(\text{NaOH}) = 1$ 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*, $\rho = 1{,}84$ 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.

This reagent must be used within 30 min of preparation.

4.3 Reagents used for colour development at high temperatures

4.3.1 *Colour reagent*

Dissolve 1,00 g ± 0,01 g of ascorbic acid ($C_6H_8O_6$) and 50 mg ± 0,5 mg of sodium thiosulfate pentahydrate ($Na_2S_2O_3 \cdot 5H_2O$) in 720 ml of water. Then add

- 15 ml of sulfomolybdic reagent (**4.1.6**), and
- 65 ml of dilute sulfuric acid (**4.1.5**).

Mix well. The volume obtained is approximately 800 ml.

This reagent must be used within 30 min of preparation.

5 Apparatus

Usual laboratory equipment and

5.1 Apparatus used in both colour developments

5.1.1 *Analytical balance*, with a readability of ± 0,001 g.

5.1.2 *Shaker* (to prevent settling of the soil in the extracting solution).

5.1.3 *pH-meter*, with a readability of ± 0,01 pH units.

5.1.4 *Spectrometer*, capable of measuring the absorbance in wavelengths up to 900 nm and accepting optical cells of path length 10 mm (readability of 0,001 units of absorbance).

5.1.5 *Optical cells*, of path length 10 mm.

5.2 Apparatus used in the colour development at high temperatures

5.2.1 *Vortex stirrer*

5.2.2 *Water bath*

6 Procedure

6.1 Determination of dry mass

Pretreat the soil sample according to ISO 11464. Determine the dry mass of the pretreated sample according to ISO 11465.

6.2 Extraction

Weigh 5,00 g ± 0,01 g of pretreated soil into a 250 ml flask. Add 1,0 g of activated carbon (**4.1.3**) and 100 ml ± 0,5 ml of extracting solution (**4.1.2**). Stopper the flask and place it immediately on the shaker (**5.1.2**). Shake for exactly 30 min at 20 °C ± 1 °C (shaking has to prevent settling of the soil in the extracting solution). Within 1 min after shaking has ended, filter immediately into a dry vessel using a phosphorus-free paper.

Prepare a blank by following the above procedure and excluding soil.

6.3 Colour development

6.3.1 *Colour development at room temperature*

Into a set of 50 ml volumetric flasks, transfer 5,00 ml of either

- — a blank solution prepared according to **6.2**, or
- — a soil extract prepared according to **6.2**, or
- — standard solutions according to **4.1.8**.

Then carefully add 0,5 ml of dilute sulfuric acid (**4.1.5**) to each unstoppered flask and gently swirl to liberate the carbon dioxide.

Add 4,0 ml of sodium metabisulfite solution (**4.2.2**) and 6,0 ml of sodium thiosulfate solution (**4.2.1**). Stopper the flask immediately. Mix well and wait for 30 min. Then add 30,0 ml of colour reagent (**4.2.4**), make up to the mark with water, stopper the flask and mix well. Wait for 60 min to allow the colour to develop.

6.3.2 *Colour development at high temperatures*

Into a set of tubes, transfer 2,00 ml of either

- — a blank solution prepared according to **6.2**, or
- — a soil extract prepared according to **6.2**, or
- — standard solutions according to **4.1.8**.

Then add 8,0 ml of colour reagent (**4.3.1**) to each tube and mix well. Wait for 60 min.

Place the tubes in the water bath (**5.2.2**) at 90 °C for 10 min to allow the colour to develop. Cool the tubes to 20 °C and mix using a vortex stirrer (**5.2.1**).

6.4 Spectrometric measurement

Measurement shall be carried out within the linear part of the calibration curve.

6.4.1 *Spectrometric measurement after colour development at room temperature*

Measure the absorbance of each solution against water at 880 nm using optical cells (**5.1.5**) for 30 min[1] after colour development. Record the values.

NOTE 4 A wavelength of 710 nm can be used if a loss of sensitivity can be accepted and after checking linearity and freedom from bias error.

6.4.2 *Spectrometric measurement after colour development at high temperatures*

Measure the absorbance of each solution against water at 825 nm using optical cells (**5.1.5**). Record the values.

6.5 Calculation

The content of phosphorus soluble in sodium hydrogen carbonate, in milligrams per kilogram of dried soil, is calculated using the following equation

$$w_P = \frac{\rho_P(A_{ES} - A_B)}{A_S - A_0} \times \frac{20d \cdot m_1}{m_2}$$

where

ρ_P	is the phosphorus concentration, in milligrams per litre, in the chosen standard solution (see Table 1);
A_{ES}	is the absorbance of the soil extract;
A_B	is the absorbance of the blank;
A_S	is the absorbance of the chosen standard solution;
A_0	is the absorbance of the 0,000 mg P/l standard solution (see Table 1);
d	is the dilution factor of the soil extract (if required);
m_1	is the mass, in grams, of air-dried soil;
m_2	is the mass, in grams, of oven-dried soil.

7 Repeatability

The repeatability of the determination of phosphorus soluble in sodium hydrogen carbonate solution carried out following colour development at room temperature, or colour development after heating to a high temperature, shall satisfy the conditions given in Table 2.

Table 2 — Repeatability

Phosphorous content mg/kg	Acceptable variation
≤ 10	3 mg P/kg
> 10 to 25	40 % of the value
> 25 to 100	15 mg P/kg
> 100	25 % of the value

8 Test report

The test report shall contain the following information:

a) a reference to this International Standard;

b) a reference to the method used;

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

d) the results of the determination in whole numbers, in milligrams of phosphorus per kilogram, calculated on the basis of oven-dried soil;

e) any details of operations riot specified in this International Standard or which are regarded as optional, as well as any other factor which may have affected the results.

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[1] See national foreword for details of textual error.

Annex A (informative)
Bibliography

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List of references

See national foreword.

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