

Soil quality —

Part 3: Chemical methods —

Section 3.11 Determination of water-soluble and acid-soluble sulfate

ICS 13.080

Committees responsible for this British Standard

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- 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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National foreword

This Section of BS 7755 has been prepared by Subcommittee EH/4/3 and is identical with ISO 11048:1995 *Soil quality — Determination of water-soluble and acid-soluble sulfate*, published by the International Organization for Standardization (ISO).

ISO 11048 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.*

Cross-references

International standard	Corresponding British Standard
ISO 3696:1987	BS 3978:1987 <i>Specification for water for laboratory use</i> (Identical)
ISO 9280:1990	BS 6068 <i>Water quality</i> Part 2 <i>Physical, chemical and biochemical methods</i> Section 2.39:1991 <i>Method for the determination of Sulfate using barium chloride and gravimetry</i> (Identical) BS 7755 <i>Soil quality</i> Part 3 <i>Chemical methods</i>
ISO 11464:1994	Section 3.5:1995 <i>Pretreatment of samples for physico-chemical analyses</i> (Identical)
ISO 11465:1993	Section 3.1:1994 <i>Determination of dry matter and water content on a mass basis by a gravimetric method</i> (Identical)

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Summary of pages

This document comprises a front cover, an inside front cover, pages i to iv, pages 1 to 16, 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.

Section 1. General

1.1 Scope

This International Standard specifies procedures for the preparation of water and acid extracts of air-dried soils and soil-like materials. The sulfate content of these extracts is determined by a gravimetric method in which barium chloride is added to the water or acid extract and the precipitate of barium sulfate is dried and weighed. The sulfate content is then calculated from the mass of the material used in the analysis and the mass of barium sulfate precipitated.

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

This International Standard consists of seven sections:

- *Section 1: General;*
- *Section 2: Pretreatment of samples and determination of dry matter contents;*
- *Section 3: Extraction of soil with water at a mass : volume ratio of 1 soil : 5 water;*
- *Section 4: Extraction of soil with water at a mass : volume ratio of 1 soil : 2 water;*
- *Section 5: Extraction of soil with dilute hydrochloric acid;*
- *Section 6: Determination of sulfate in solution by a gravimetric method using barium chloride;*
- *Section 7: Precision of method and test report.*

1.2 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 9280:1990, *Water quality — Determination of sulfate — Gravimetric method using barium chloride.*

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.*

1.3 Principle

Samples of air-dried soil are extracted with:

- dilute hydrochloric acid; or
- water in a mass : volume ratio of soil : water of 1 : 2 or 1 : 5.

The sulfate content of these extracts is determined by a gravimetric method in which barium chloride is added to the water or acid extract and the precipitate of barium sulfate is dried and weighed. The sulfate content is then calculated from the mass of soil used in the analysis and the mass of barium sulfate precipitated.

Section 6 is similar to ISO 9280.

It is recognized that other acceptable methods for the determination of sulfate are available but these are not covered by this International Standard. Suitable methods may include: direct barium titrimetry, inductively coupled plasma emission spectrometry, flow injection analysis using a turbidimetric method, continuous flow indirect spectrophotometry using 2-aminoperimidine, air-segmented continuous flow colorimetry using methylthymol blue, and ion chromatography. Reference should be made to the appropriate international and national standards for information regarding the applicability and limitations of such methods.

1.4 Applicability and limitations

The procedures in sections 3 and 4 are considered to be relevant to the extraction of sulfate from soils and may be relevant to the assessment of the “availability” of sulfate to plants (a variety of extractants have been used for this purpose and none were shown to be generally applicable to all soil types and climates). It should be noted that there is no verified relation between water-extractable sulfate and the need for sulfur fertilization. The procedure in section 4 is also relevant to the potential for the attack of sulfates on building materials, especially concrete.

The use of the procedures described in sections 4, 5 and 6 should enable a distinction to be made between the presence of sulfates that are highly soluble in water and those of limited solubility such as gypsum ($\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$).

Section 6 is applicable to concentrations of sulfate ion (SO_4^{2-}) in solution of 10 mg/l and above, i.e. in relation to the methods in sections 3 and 4 to minimum water-soluble concentrations in air-dried soil of about 50 mg/kg and 20 mg/kg [0,005 % (*m/m*) and 0,002 % (*m/m*)] respectively and, in relation to the method in section 5, to a minimum acid-soluble concentration in air-dried soil of about 500 mg/kg [0,05 % (*m/m*)].

If concentrations in solution are lower than this, a concentration step will be required, or a more sensitive analytical procedure than that described in section 6 should be used [e.g. ion chromatography, inductively coupled plasma (ICP) spectrometry or atomic emission spectrometry (AES)].

In most instances, complete characterization of a soil, in terms of the concentrations and nature of sulfates present, will require that determinations are made following the procedure in section 5 and in either section 3 or section 4 or both, whichever is the more appropriate for the purpose of investigation of the soil.

The methods described should be suitable for a range of “soils” including agricultural soils and materials from polluted sites. However, no method can be expected to be universally applicable, and users must recognize this fact and be prepared to deal with the analytical difficulties that may sometimes be encountered. Soils containing sulfides may require special attention (see note 7 to 5.1).

“Soils” from some polluted sites, for example former gasworks sites and colliery spoil tips, may contain a variety of sulfur species (e.g. elemental sulfur, sulfate, sulfite, thiosulfate, thiocyanate) that may be transformed from one to another under changing chemical conditions and/or microbial action. The presence of other ions may affect solubilities. Thus, in these circumstances, particular care may be required in sample handling and preparation, and in the interpretation of the results.

The limitations of the method in section 6, in terms of acceptable concentrations of other ions, are defined. The tests described are strictly empirical and thus cannot be considered to measure the true sulfate content in soil, although reproducibility should be good.

Because of the limited solubility of some sulfates (e.g. calcium sulfate) and possible interferences from other ions, a single extraction following the procedure described in section 3 may not give a true measure of the “total water extractable” sulfate. This potential problem can be tackled either by repeating the extraction one or more times on the same sample, or by substantially increasing the water : soil ratio. Another option is to use a “buffered” extraction solution which will enhance the solubility of the sulfates present, but such procedures are beyond the scope of this International Standard.

If calcium is the only cation present, sulfate ion in solution will not exceed 1 441 mg SO_4^{2-} /l at 20 °C. This means that the maximum amount of sulfate that can be extracted from such a soil by the procedure in section 3 is about 7 205 mg SO_4^{2-} /kg (10 g soil : 50 ml water) and by the procedure in section 4 is about 2 882 mg SO_4^{2-} /kg (50 g soil : 100 ml water).