

BS 1377-3:2018+A1:2021



BSI Standards Publication

Methods of test for soils for civil engineering purposes

Part 3: Chemical and electro-chemical testing

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Foreword

Publishing information

This part of BS 1377 is published by BSI Standards Limited, under licence from The British Standards Institution, and came into effect on 31 July 2018. It was prepared by Subcommittee B/526/3, *Site investigation and ground testing*, under the authority of Technical Committee B/526, *Geotechnics*. A list of organizations represented on these committees can be obtained on request to the committee manager.

Supersession

BS 1377-3:2018+A1:2021 supersedes BS 1377-3:2018, which is withdrawn.

Relationship with other publications

BS 1377 is published in the following parts:

- Part 1: General requirements and sample preparation;
- Part 2: Classification tests;
- Part 3: Chemical and electrochemical tests;
- Part 4: Compaction-related tests;
- Part 5: Compressibility, permeability and durability tests;
- Part 6: Consolidation and permeability tests in hydraulic cells and with pore pressure measurement;
- Part 7: Shear strength tests (total stress);
- Part 8: Shear strength tests (effective stress);
- Part 9: In-situ tests.

Information about this document

This part of BS 1377 is intended to be read in conjunction with BS 1377-1.

In this part of BS 1377, the tests described in the 1990 edition have been retained. Additional tests have been added to include the recommendations of BRE Special Digest 1 (BRE 2005) [1]. Also, analytical methods of chemical analysis have been included, i.e. total carbon analyzer, ion chromatography and inductively coupled plasma atomic emission spectroscopy. The two point resistivity method has been removed and additional four point tests included.

Text introduced or altered by Amendment No. 1 is indicated in the text by the tags A1 A1. Minor editorial changes are not tagged.

Amendment A1 introduces the following principal changes:

- technical changes to BS 1377-3 to remove an error in the wording and to improve clarity.

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Where websites and webpages have been cited, they are provided for ease of reference and are correct at the time of publication. The location of a webpage or website, or its contents, cannot be guaranteed.

Hazard warnings

WARNING. Persons using this British Standard are expected to be familiar with normal laboratory practice. This British Standard calls for the use of substances and/or procedures that can be injurious to health if adequate precautions are not taken. It refers only to technical suitability and does not absolve the user from legal obligations relating to health and safety at any stage. These include the use of fume cupboards or similar apparatus when using acids and other toxic chemicals. This standard does not purport to address the safety problems, if any, associated with its use. It is the responsibility of the user to establish appropriate safety and health practices and to ensure compliance with any national regulatory conditions.

It is expected that tests conducted in accordance with this British Standard will be carried out by suitably trained and experienced staff.

WARNING. It is dangerous to add water to concentrated acid.

Use of this document

It has been assumed in the preparation of this part of BS 1377 that the execution of its provisions will be entrusted to appropriately qualified and experienced people, for whose use it has been produced.

Presentational conventions

The provisions of this standard are presented in roman (i.e. upright) type. Its methods are expressed as a set of instructions, a description, or in sentences in which the principal auxiliary verb is “shall”.

Commentary, explanation and general informative material is presented in smaller italic type, and does not constitute a normative element.

Where words have alternative spellings, the preferred spelling of the Shorter Oxford English Dictionary is used (e.g. “organization” rather than “organisation”).

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1 Scope

This part of BS 1377 describes test methods for determining the amount of chemical substances in samples of soil and extremely weak and very weak rocks, as defined by BS 5930, and groundwater.

NOTE 1 Chemical tests in this part of BS 1377 may be used on other rocks if required.

It also describes test methods for the determination of some electrochemical and resistivity properties of solid samples.

NOTE 2 These tests provide data to assess the potential of the ground and solutes to damage construction materials, including cementitious materials and metals in the ground. They can also be used in assessment of the potential for volume change of the ground due to chemical reaction. Resistivity test results can also be used to assess in-situ resistivity results.

This British Standard is not written for testing samples from contaminated land or for soil quality assessment.

Procedures described in this part of BS 1377 are for the determination of the following:

- a) organic matter content in the material ([Clause 4](#));
- b) total organic carbon (TOC) content in the material ([Clause 5](#));
- c) loss on ignition of the material ([Clause 6](#));
- d) sulfur compounds ([Clause 7](#)):
 - 1) water-soluble sulfate content of the material by 2:1 extraction;
 - 2) sulfate content in groundwater;
 - 3) acid-soluble sulfate content of the material;
 - 4) total sulfur content of the material;
 - 5) total sulfide content (total reduced sulfur) content of the material;
 - 6) acid-soluble sulfide (monosulfides sulfur) content of the material;
- e) carbonate content of the material ([Clause 8](#));
- f) chloride content ([Clause 9](#)):
 - 1) water-soluble chloride content of the material;
 - 2) acid-soluble chloride content of the material.
- g) water-soluble magnesium content of the material ([Clause 10](#));
- h) total dissolved solids of the groundwater ([Clause 11](#));
- i) pH value ([Clause 12](#));
- j) electrical resistivity of the material ([Clause 13](#)); and
- k) redox potential of the material ([Clause 14](#)).

Brief guidance on the detrimental effects of sulfur compounds on engineering works and alternative methods of identifying the specific minerals is given in [Annex A](#).

NOTE 3 Good practice in chemical testing requires duplicate specimens to be tested. In each of the test methods the measurement of only one value of the overall result is described. It is recognized that it is necessary in many practical applications to make a number of tests in order to obtain a representative value and an indication of the reliability of the results. Guidance on the number of measurements required and the treatment of the results obtained are not provided in this standard.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes provisions 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.

BS 89, *Specification for direct acting indicating electrical measuring instruments and their accessories*

[BS 1377-1:2016](#), *Methods of test for soils for civil engineering purposes – Part 1: General requirements and sample preparation*

BS 1881-124:2015, *Testing concrete – Part 124: Methods for analysis of hardened concrete*

[BS 5930](#), *Code of practice for ground investigations*

BS EN ISO 3696:1995, *Water for analytical laboratory use – Specification and test methods*

[BS EN ISO 17034](#), *General requirements for the competence of reference material producers*

BS EN ISO 17892-1, *Geotechnical investigation and testing – Laboratory testing of soil – Part 1: Determination of water content*

BS EN ISO 22475-1:2016, *Geotechnical investigation and testing – Sampling methods and groundwater measurements – Part 1: Technical principles for execution*

3 Terms and definitions

For the purposes of this part of BS 1377, the terms and definitions given in BS 1377-1 and the following apply.

3.1 titration

addition of a solution from a graduated burette to a known volume of a second solution, until the chemical reaction between the two is completed

NOTE If the strength of one of the solutions is known, that of the other can be calculated from the volume of liquid added.

3.2 indicator

substance which is capable of giving a clear visual indication of the completion of a chemical reaction in a solution being titrated, usually by means of a change in colour

3.3 pH value

logarithm to base 10 of the reciprocal of the concentration of hydrogen ions in an aqueous solution

NOTE It provides a measure of the acidity or alkalinity of the solution on a scale reading from 0 to 14, on which 7 represents neutrality.

3.4 resistivity (of soil)

electrical resistance, in Ω (ohms) per unit length, of a column of soil of unit area of cross-section

NOTE In this part of BS 1377, resistivity is expressed in Ωm (ohm metres).

3.5 redox potential (reduction/oxidation potential)

relative measure, expressed in millivolts, of the reducing or oxidising capacity of soil, usually increasing with increasing oxygen content

3.6 calibration blank sample

extract solution used to make solutions

NOTE 1 For example, water or acid used for the preparation of extractions prior to analysis.

NOTE 2 The primary purpose of a blank is to trace sources of artificially introduced contamination and used to facilitate correction of the final test sample result.

3.7 stock solution

solution with accurately known analyte concentration(s) prepared with an appropriate purity

NOTE Stock solutions are reference materials within the meaning of PD ISO Guide 30.

4 Determination of the organic matter content

4.1 Principle

This procedure covers the determination of the percentage by dry mass of oxidisable organic matter present in a soil.

NOTE The method, which uses dichromate oxidation, is known as Walkley and Black's method.

Soils containing sulfides or chlorides have been found to give high results by this procedure.

Methods for checking for the presence of these compounds, and procedures for their removal before testing if they are present, are included.

The requirements of BS 1377-1, where appropriate, shall apply to this test method.

4.2 Reagents

4.2.1 All reagents shall be of recognized analytical reagent quality.

NOTE Where accurately standardized solutions are required it might be more convenient to obtain them already standardized in concentrated form and to dilute them as necessary in accordance with the manufacturer's instructions.

4.2.2 Potassium dichromate solution, $[c(K_2Cr_2O_7) = 0.167 \text{ mol/l}]$. Dissolve 49.035 g of potassium dichromate in distilled/de-ionized water (BS 1377-1:2016, 6.1) to make 1 l of solution.

4.2.3 Ferrous sulfate solution. Dissolve approximately 140 g of ferrous sulfate in sulfuric acid solution $[c(H_2SO_4) = 0.25 \text{ mol/l}]$ to make 1 l of solution. Add 14 ml of concentrated sulfuric acid to distilled/de-ionized water (BS 1377-1:2016, 6.1) to make 1 l of sulfuric acid solution $[c(H_2SO_4) = 0.25 \text{ mol/l}]$. Record the date the solution is made on the bottle. This solution is unstable in air. Keep it tightly stoppered and standardize against the potassium dichromate solution at least once a week.

4.2.4 Sulfuric acid, concentrated. Density 1.84 g/ml.

4.2.5 Orthophosphoric acid, 85% (v/v) solution. Density 1.70 g/ml to 1.75 g/ml.

4.2.6 Indicator solution. Dissolve 0.25 g of sodium diphenylamine sulfonate in 100 ml of distilled/de-ionized water (BS 1377-1:2016, 6.1) water.

4.2.7 Hydrochloric acid, 25% (v/v) solution. Add 250 ml of concentrated hydrochloric acid (density 1.18 g/ml) to 500 ml of distilled/de-ionized water (BS 1377-1:2016, 6.1), then make up to 1 l with distilled/de-ionized water (BS 1377-1:2016, 6.1).

4.2.8 Lead acetate paper. Filter paper that has been dipped in a 10% solution of lead acetate.

- 4.2.9** *Sulfuric acid, $[c(H_2SO_4)] = \text{approximately } 1 \text{ mol/l}]$.* Add 53 ml of concentrated sulfuric acid to about 500 ml of distilled/de-ionized water (BS 1377-1:2016, **6.1**) water, then make up to 1 l with distilled/de-ionized water (BS 1377-1:2016, **6.1**).

4.3 Apparatus

- 4.3.1** *Drying oven, capable of maintaining a temperature of $(50 \pm 2.5)^\circ\text{C}$.*
- 4.3.2** *Balance, readable to 1 g.*
- 4.3.3** *Balance, readable to 0.001 g.*
- 4.3.4** *1 l volumetric flask.*
- 4.3.5** *Two 25 ml burettes, graduated to 0.1 ml.*
- 4.3.6** *10 ml pipette and a 1 ml pipette, each fitted with a rubber teat.*
- 4.3.7** *Two conical flasks, of 500 ml capacity.*
- 4.3.8** *200 ml and 20 ml graduated measuring cylinders.*
- 4.3.9** *Desiccator, (a convenient size is about 200 mm to 250 mm in diameter) containing anhydrous silica gel.*
- 4.3.10** *Glass weighing bottle, approximately 25 mm in diameter, 50 mm high and fitted with a ground glass stopper.*
- 4.3.11** *Test sieves, 2 mm and 0.425 mm aperture sizes, with receiver.*
- 4.3.12** *Sample dividers of multiple-slot type (riffle boxes), having widths of opening of 7 mm and 15 mm.*
- 4.3.13** *Pestle and mortar, or a suitable mechanical crusher.*
- 4.3.14** *Wash bottle, preferably made of plastics, containing distilled/de-ionized water (BS 1377-1:2016, [6.1](#)).*
- 4.3.15** *Glass boiling tube.*
- 4.3.16** *Filter funnel, of approximately 100 mm diameter.*
- 4.3.17** *Filter papers, of a diameter appropriate to the size of the funnel: medium grade (e.g. Whatman No. 40 ¹⁾) and fine grade (e.g. Whatman No. 42 ¹⁾).*
- 4.3.18** *Blue litmus paper.*

4.4 Preparation of test specimen

- 4.4.1** Each test specimen shall be prepared for analysis from the laboratory sample as given in [4.4.2](#) to [4.4.11](#).
- 4.4.2** An initial sample shall be obtained as described in BS 1377-1:2016, **8.3**, and of the approximate size as specified in BS 1377-1:2016, **8.5**.
- 4.4.3** The sample shall be dried in the oven to constant mass at $(50 \pm 2.5)^\circ\text{C}$, and cooled to room temperature in the desiccator.
- 4.4.4** The sample shall be weighed to the nearest 0.1% and the mass m_1 (in g) recorded.

¹⁾ Whatman is a trademark of GE Healthcare. This information is given for the convenience of users of this document and does not constitute an endorsement by the British Standards Institution of the named product. Equivalent products may be used if they can be shown to lead to the same results.

- 4.4.5** The sample shall be sieved on a 2 mm test sieve (if appropriate, guarded by a test sieve of larger aperture) and crush retained particles other than stones to pass the 2 mm sieve.

NOTE It is assumed that any material retained on the 2 mm test sieve will not contain organic matter. If this is seen not to be true, the pieces of organic matter should be removed by hand, crushed to pass a 2 mm test sieve and incorporated in the fraction passing the sieve.

- 4.4.6** The stones shall be rejected, ensuring that no fine material adheres to them, e.g. by brushing. Record the mass of sample passing the 2 mm test sieve (in g) to the nearest 0.1% (m_2). Throughout these and subsequent operations take care to ensure that there is no loss of fines.

- 4.4.7** The material shall pass the 2 mm sieve by successive riffing through the 15 mm divider to produce a sample weighing approximately 100 g.

- 4.4.8** This sample shall be pulverized so that it passes the 425 μm sieve.

- 4.4.9** The sample shall be subdivided by riffing on the 7 mm divider to obtain the following test specimens, as appropriate:

- a) if sulfides and chlorides are known to be absent, two test specimens each weighing about 5 g;
- b) for a check test to determine whether sulfides are present, a specimen of about 5 g;
- c) for a check test to determine whether chlorides are present, a specimen of about 50 g;
- d) if sulfides and/or chlorides are present, a specimen of about 50 g.

NOTE Throughout this preparation and any subsequent operation mix the material available thoroughly before any division and take care to avoid segregation during riffing.

- 4.4.10** Each specimen shall be placed in a glass weighing bottle and dried in the oven at a temperature of $(50 \pm 2.5)^\circ\text{C}$. The specimens shall be deemed to be dry when the differences in successive weighings, carried out at intervals of 4 h, do not exceed 0.1% of the original mass of the sample.

- 4.4.11** The specimens shall be allowed to cool to room temperature in a desiccator containing dry self-indicating desiccant and each bottle and contents weighed to 0.001 g.

4.5 Procedure

4.5.1 Standardization of ferrous sulfate

- 4.5.1.1** 10 ml of the potassium dichromate solution ([4.2.2](#)) shall be added from a burette into a 500 ml conical flask.
- 4.5.1.2** 20 ml of concentrated sulfuric acid shall be added very carefully, the mixture swirled and allowed to cool for some minutes.
- 4.5.1.3** 200 ml of distilled/de-ionized water (BS 1377-1:2016, [6.1](#)) shall be added very carefully to the mixture followed by 10 ml of phosphoric acid and 1 ml of the indicator, and shaken to mix thoroughly.
- 4.5.1.4** Ferrous sulfate solution shall be added from the second burette in 0.5 ml increments, and the contents of the flask swirled until the colour of the solution changes from blue to green.
- 4.5.1.5** A further 0.5 ml of potassium dichromate solution shall be added, changing the colour back to blue.
- 4.5.1.6** Ferrous sulfate solution shall be added slowly drop by drop with continued swirling until the colour of the solution changes from blue to green after the addition of a single drop.
- 4.5.1.7** The total volume of ferrous sulfate solution used shall be recorded, x , to the nearest 0.05 ml.

4.5.2 Qualitative check for sulfides

- 4.5.2.1 The presence of sulfides in the soil shall be verified as described in [4.5.2.2](#) and [4.5.2.3](#).

NOTE A qualitative check might have been carried out in the tests for sulfide (see Clause 7, in particular 7.9).

- 4.5.2.2 The 5 g check sample (which need not be weighed) shall be placed in a boiling tube. 50 ml (approx.) of hydrochloric acid ([4.2.7](#)) shall be added. This shall be brought to the boil and the presence of hydrogen sulfide checked for by holding in the vapour a piece of filter paper that has been dipped in a 10% solution of lead acetate. This will turn black if hydrogen sulfide is present.

NOTE This should be carried out in a fume cupboard.

- 4.5.2.3 If the presence of sulfides is indicated it shall be removed from the test specimen as described in [4.5.3](#) before proceeding with the analysis for organic matter; otherwise a result that is too high will be obtained.
- 4.5.2.4 If the presence of sulfides is not indicated, omit the procedure given in [4.5.3](#).

4.5.3 Elimination of sulfides

- 4.5.3.1 About 50 g of the soil shall be weighed, after cooling as described in [4.4.10](#) and [4.4.11](#), to 0.01 g and placed in a 500 ml conical flask.
- 4.5.3.2 Sulfuric acid solution [$c(\text{H}_2\text{SO}_4) = 1.0 \text{ mol/l}$] shall be added until no further evolution of hydrogen sulfide occurs, as determined by testing with lead acetate paper.
- 4.5.3.3 The contents of the conical flask shall be filtered on a medium grade filter paper, taking care to retain all solid particles. This shall be washed several times with hot distilled/de-ionized water (BS 1377-1:2016, [6.1](#)) until the washings do not indicate acidity when tested with blue litmus.
- 4.5.3.4 The soil retained on the filter paper shall be dried to constant mass at a temperature of $(50 \pm 2.5)^\circ\text{C}$ and cooled in the desiccator.
- 4.5.3.5 The soil shall be carefully removed from the filter paper and its mass determined to 0.01 g.
- 4.5.3.6 The treated sample shall be subdivided as described in [4.4.9](#), item a) and each test specimen shall be dried and cooled as described in [4.4.10](#) and [4.4.11](#).

4.5.4 Qualitative check for chlorides

- 4.5.4.1 The presence of chlorides in the soil shall be verified by following the procedure described in [9.2.3](#).
- 4.5.4.2 If the presence of chlorides are indicated they shall be removed from the test specimen as described in [4.5.5](#) before proceeding with the analysis for organic matter; otherwise a result that is too high will be obtained.

NOTE 1 Alternatively, the effect of chlorides on the organic matter determination can be partly eliminated by using concentrated sulfuric acid in which silver sulfate has been dissolved in place of the concentrated sulfuric acid specified in [4.2.4](#). If the ratio of carbon to chloride does not exceed unity, 25 g of silver sulfate per litre of sulfuric acid will be sufficient to precipitate the chloride.

NOTE 2 If the presence of both sulfides and chlorides is indicated, the procedures described in [4.5.3](#) and [4.5.5](#) should both be carried out on the sample of soil used for determination of the organic content.

- 4.5.4.3 If the presence of chlorides is not indicated, omit the procedure given in [4.5.5](#).

4.5.5 Elimination of chlorides

- 4.5.5.1 About 50 g of the soil shall be weighed, after cooling as described in [4.4.10](#) and [4.4.11](#), to 0.01 g.

- 4.5.5.2** The soil shall be placed on a medium-grade filter paper in a funnel and washed with distilled/de-ionized water (BS 1377-1:2016, **6.1**) water.
- 4.5.5.3** Washing shall continue until no turbidity is observed when a drop of the wash water is tested with silver nitrate solution.
- 4.5.5.4** The soil retained on the filter paper shall be dried to constant mass at a temperature of $(50 \pm 2.5)^\circ\text{C}$ and cooled in the desiccator.
- 4.5.5.5** All the soil shall be carefully removed from the filter paper and its mass determined to 0.01 g.
- 4.5.5.6** The treated sample shall be subdivided as described in [4.4.9](#), item a) and each test specimen shall be dried and cooled as described in [4.4.10](#) and [4.4.11](#).

4.5.6 Analysis for organic matter

- 4.5.6.1** Each weighing bottle containing soil, obtained as described in [4.4.4](#), shall be weighed to 0.001 g.
- 4.5.6.2** A small quantity, from 5.0 g to 0.2 g, depending on the organic content (see note), shall be transferred to a dry 500 ml conical flask, the weighing bottle reweighed and the mass of soil removed (m_3) calculated by difference.

NOTE The size of the sample for chemical analysis varies with the amount of organic matter present in the soil. As much as 5 g might be required for soil low in organic matter, and as little as 0.2 g with a very peaty soil. After a number of determinations have been made experience will indicate the most suitable size of sample to be taken. Where this is not so, it is suggested that a series of samples of varying sizes should be tested. The determination giving a total of 5 ml to 8 ml of potassium dichromate solution reduced should be taken as the one giving the correct result.

- 4.5.6.3** 10 ml of the potassium dichromate solution ([4.2.2](#)) shall be run from a burette into the conical flask, and 20 ml concentrated sulfuric acid added very carefully from a measuring cylinder. The mixture shall be swirled for about 1 min, and then allowed to stand on a heat-insulating surface for 30 min to allow oxidation of the organic matter to proceed. During this period the flask shall be protected from cold air and draughts.
- 4.5.6.4** 200 ml of distilled/de-ionized water (BS 1377-1:2016, **6.1**) shall be added very carefully to the mixture, followed by 10 ml of orthophosphoric acid and 1 ml of indicator, and the mixture thoroughly shaken. If the indicator is absorbed by the soil a further 1 ml of the solution shall be added.
- 4.5.6.5** Ferrous sulfate solution shall be added from the second burette in 0.5 ml increments and the contents of the flask swirled until the colour of the solution changes from blue to green.
- 4.5.6.6** A further 0.5 ml of potassium dichromate solution shall be added, changing the colour back to blue.
- 4.5.6.7** Ferrous sulfate solution shall be added slowly drop by drop with continued swirling until the colour of the solution changes from blue to green after the addition of a single drop.
- 4.5.6.8** The total volume of ferrous sulfate solution used, y , shall be recorded to the nearest 0.05 ml.

4.6 Calculations

- 4.6.1** The total volume V (in ml) of potassium dichromate solution used to oxidise the organic matter in the soil sample shall be calculated from the equation:

$$V = 10.5 \left(1 - \frac{y}{x} \right) \quad (1)$$

where

- y is the total volume of ferrous sulfate solution used (see 4.5.6.8) (in ml);
 x is the total volume of ferrous sulfate solution used in the standardization test (see 4.5.1) (in ml).

- 4.6.2** The percentage of the original soil sample passing the 2 mm test sieve shall be calculated from the equation:

$$\text{fraction finer than 2 mm} = \frac{m_2}{m_1} \times 100 \quad (2)$$

where

- m_1 is the initial dry mass of sample (in g);
 m_2 is the mass of the sample passing the 2 mm test sieve (in g).

- 4.6.3** The percentage of organic matter present in the fraction of the soil specimen finer than 2 mm for each determination shall be calculated from the equation (see note):

$$\text{percentage organic matter content} = \frac{0.67V}{m_3} \quad (3)$$

where

- m_3 is the mass of soil used in the test (in g).

NOTE The method is based on wet oxidation of the organic content of the soil, and assumes that soil organic matter contains an average of 58% (m/m) of carbon. The method employed oxidises approximately 77% of the carbon in the organic matter, and these factors are included in the equation given. The factors will give correct results only for soil containing natural organic matter.

- 4.6.4** If duplicate specimens have been tested, and if the individual results expressed as a percentage of organic matter differ by no more than 2%, the mean result shall be calculated. If they differ by more than 2%, the test shall be repeated starting with two new representative portions of soil.

4.7 Test report

The test report shall state that the test was carried out in accordance with **BS 1377-3:2018+A1:2021**, **Clause 4** and shall contain the following information:

- the method of test used;
- the organic matter content present in the soil fraction passing a 2 mm test sieve to the nearest 0.1% of the original oven dry mass of soil;
- the percentage by dry mass of the original sample passing the 2 mm test sieve to the nearest 1%;
- if sulfides or chlorides have been identified in the soil;
- the information required by BS 1377-1:2016, **10.1**.

5 Determination of total organic carbon (TOC)

5.1 Principle

This procedure covers the determination of the percentage of total organic carbon present in a sample by total carbon analyzer using the combustion method. Total organic carbon, which measures

just carbon, is different from organic matter, which includes all the elements (hydrogen, oxygen, nitrogen, etc.) that are components of organic compounds. The inorganic carbon, as carbonate, is removed prior to testing for total organic carbon.

NOTE An example of a routine method for the determination of TOC is described using a total carbon analyzer controlled by a computer running appropriate software. The sample is washed with acid prior to analysis to remove inorganic carbon so the result obtained from the instrument is only organic carbon. The sample is heated in a high temperature combustion furnace (typically around 1 050 °C), through which a carrier gas, such as helium, is constantly flowing. Once in the combustion furnace oxygen is introduced to combust the sample. The carrier gas then conveys the combustion products via other furnace tubes containing materials to aid complete combustion and remove halogens, then via a suitable desiccant to remove moisture. The remaining stream of gas passes into a detector (e.g. infra-red) where carbon dioxide is determined as a function of time. Carbonate minerals are ubiquitous in natural deposits and will affect the TOC determination. All metal carbonate minerals are unstable and break down in the presence of hydrogen ions available in hydrochloric acid releasing the mineral carbon as CO₂. Most show reaction with cold dilute hydrochloric acid, but some such as dolomite, siderite, magnesite and other less common forms are only very slowly soluble. The solubility increases significantly in hot hydrochloric acid. Pre-treatment of soil and powdered rock samples with hot hydrochloric acid is essential to remove any carbonate minerals and to ensure that the value of TOC determined accounts only for the total organic carbon present.

5.2 Reagents

- 5.2.1 All reagents shall be of recognized analytical reagent quality.
- 5.2.2 Concentrated hydrochloric acid (density 1.18 g/ml).
- 5.2.3 Hydrochloric acid, 10% (v/v) solution. Dilute 100 ml of concentrated hydrochloric acid (density 1.18 g/ml) to 1 ml with distilled/de-ionized water (BS 1377-1:2016, 6.1).
- 5.2.4 High purity gaseous oxygen.
- 5.2.5 Combustion catalyst, as specified by the instrument manufacturer.
- 5.2.6 Certified carbon standards, a range of carbon levels are required to provide a calibration over the required range.

5.3 Apparatus

- 5.3.1 Drying oven, capable of maintaining a temperature of (50 ±2.5) °C.
- 5.3.2 Balance, readable to 0.01 g.
- 5.3.3 Balance, readable to 0.0001 g.
- 5.3.4 Conical flask.
- 5.3.5 Funnel.
- 5.3.6 Desiccator with dry self-indicating desiccant.
- 5.3.7 GF/C glass fibre filters.
- 5.3.8 Filterable crucibles and vacuum bath.
- 5.3.9 Beakers.
- 5.3.10 Pasteur pipette.
- 5.3.11 Hot plate.
- 5.3.12 Test sieves, 2 mm and 425 µm aperture sizes, with receiver.

- 5.3.13** *Sample dividers of multiple-slot type (riffle boxes)*, having widths of opening of 7 mm and 15 mm.
- 5.3.14** *Pestle and mortar*, or a suitable mechanical crusher.
- 5.3.15** *Carbon combustion analyzer* and associated catalyst.
- 5.3.16** *Appropriate pipework and regulator*, as required, to convey the gasses to the carbon combustion analyzer.

5.4 Preparation of test specimen

- 5.4.1** Each test specimen shall be prepared for analysis from the laboratory sample as given in [5.4.2](#) to [5.4.10](#).
- 5.4.2** An initial sample and of approximate size shall be obtained as described in BS 1377-1:2016, **8.3**.
- 5.4.3** The sample shall be dried in the oven to constant mass at $(50 \pm 2.5)^\circ\text{C}$, and cooled to room temperature in the desiccator containing dry desiccant.
- 5.4.4** The sample shall be weighed to the nearest 0.1% and the mass recorded, m_1 (g).
- 5.4.5** The sample shall be sieved on a 2 mm test sieve (if appropriate, guarded by a test sieve of larger aperture) and retained particles other than stones crushed to pass the 2 mm sieve.
- NOTE* It is assumed that any material retained on the 2 mm test sieve will not contain organic matter. If this is seen not to be true, the pieces of organic matter should be removed by hand, crushed to pass a 2 mm test sieve and incorporated in the fraction passing the sieve.
- 5.4.6** The stones shall be rejected, ensuring that no fine material adheres to them, e.g. by brushing. The mass of sample passing the 2 mm test sieve shall be recorded (in g) to the nearest 0.1% (m_2). Throughout these and subsequent operations care shall be taken to ensure that there is no loss of fines.
- 5.4.7** The material passing the 2 mm sieve shall be divided by successive riffing through the 15 mm divider to produce a sample weighing approximately 100 g.
- 5.4.8** This sample shall be pulverized so that it passes the 425 μm test sieve.
- 5.4.9** Each specimen shall be placed in a glass weighing bottle and dried in the oven at a temperature of $(50 \pm 2.5)^\circ\text{C}$. The specimens shall be deemed to be dry when the differences in successive weighings, carried out at intervals of 4 h, do not exceed 0.1% of the original mass of the sample.
- 5.4.10** The specimen shall be cooled to room temperature in the desiccator and each bottle and contents weighed to 0.001 g.

5.5 Procedure

5.5.1 Determination of test sample mass

The glass fibre and filter or filterable cuvette shall be weighed, m_3 , recorded to the nearest 0.001 g. The sample shall be added to the glass fibre filter or into the cuvette and reweighed, m_4 , to the nearest 0.001 g.

NOTE 1 The mass of the sample tested depends on the organic carbon content, apparatus used and the manufacturer's recommendations for that apparatus. Samples with high organic content, such as peaty material, require lower test mass than for instance material containing high carbonate and low organic carbon.

NOTE 2 The size of the sample for chemical analysis may be reduced when there is a large amount of organic matter present in the soil. After a number of determinations have been made experience will indicate the most suitable size of sample to be taken.

5.5.2 Removal of inorganic carbon

- 5.5.2.1** The carbonate content shall be removed from the sample as described in [5.5.2.2](#) to [5.5.2.10](#).
- 5.5.2.2** The glass fibre filter containing the sample shall be placed into a funnel supported in a conical flask, or the filterable crucible placed into its vacuum bath.
- 5.5.2.3** Cold 10% hydrochloric acid shall be added drop by drop to the sample until each sample is wet, if using filterable crucibles turn on the vacuum.
- NOTE* This process should be carried out in a fume cupboard, beneath an extractor hood or similar equipment.
- 5.5.2.4** The addition of acid might cause the samples to effervesce, if so, the 10% hydrochloric acid shall be added until no further reaction can be seen.
- 5.5.2.5** Hot $[(90 \pm 5) ^\circ\text{C}]$ 10% hydrochloric acid shall then be added drop by drop until no reaction can be seen.
- 5.5.2.6** Washing with hot concentrated hydrochloric acid ([5.2.2](#)) shall be repeated. Each aliquot shall be allowed to drain through the filter or crucible before the next addition is made.
- 5.5.2.7** The sample shall be washed with six washings of a Pasteur pipette full (approximately 1 ml) of hot $[(90 \pm 5) ^\circ\text{C}]$ distilled/de-ionized water (BS 1377-1:2016, [6.1](#)). The water shall be allowed to drain through between each addition.
- 5.5.2.8** Once the liquid has drained through, fold the filters to ensure no sample is lost and placed in a crucible.
- 5.5.2.9** The crucible shall be placed into the oven at $(105 \pm 5) ^\circ\text{C}$ for at least 2 h or until the sample is dry.
- 5.5.2.10** The crucible shall be removed from the oven and placed in a desiccator with dry self-indicating desiccant to cool until ready for analysis.

5.5.3 Analysis for total organic carbon

A representative sub sample of suitable mass as recommended by the apparatus manufacturers, shall be placed into a ceramic crucible, and then placed on the auto-sampler of the analyzer. The crucible is mechanically lowered into the combustion furnace and the test shall proceed as in the apparatus manufacturer's recommendations.

5.5.4 Calibration

- 5.5.4.1** The performance of the instrument shall be verified before each batch of analyzes and after each service.
- 5.5.4.2** The combustion analyzer shall be calibrated using a multi-point calibration of certified carbon standards with different organic carbon contents following the manufacturer's instructions.
- 5.5.4.3** The analysis of samples and check standards shall be as per manufacturer's instructions.
- 5.5.4.4** Procedural blanks shall be run in duplicate. The instrumental software should automatically register that the carbon signal for a procedural blank is below the specified calibration range. If both of these duplicates values exceed this value, remedial action shall be taken to identify and correct it.

NOTE 1 This method does not measure soluble organic compounds as they are removed during the acidification and washing procedure.

NOTE 2 Any chloride or water not removed by the instrument can corrode analyzer components and/or interfere with the determination of carbon dioxide.

5.6 Calculations

5.6.1 The weight of sample prior to acid washing, m_5 (which is equal to $m_4 - m_3$) shall be input into the apparatus computer data for that sample as required and the results from the instrument are as % carbon. Because the acid washing has removed inorganic carbon this will equate to the % total organic carbon present in the dry soil.

5.6.2 The percentage of the original soil sample passing the 2 mm test sieve shall be calculated from the equation:

$$\text{fraction finer than 2 mm} = \frac{m_2}{m_1} \times 100 \quad (4)$$

where

m_1 is the initial dry mass of sample (in g);

m_2 is the mass of the sample passing the 2 mm test sieve (in g).

5.7 Test report

The test report shall state that the test was carried out in accordance with

BS 1377-3:2018+A1:2021, [Clause 5](#) and shall contain the following information:

- a) the method of test used, BS 1377-3, test 5;
- b) the apparatus used;
- c) the total organic carbon present in the soil fraction passing a 2 mm test sieve to the nearest 0.1% of the original oven dry mass of soil;
- d) the percentage by dry mass of the original sample passing the 2 mm test sieve to the nearest 1%; and
- e) the information required by BS 1377-1:2016, **10.1**.

6 Determination of the mass loss on ignition

6.1 Principle

This clause describes the procedure for determining the proportion by mass that is lost from a soil by ignition at a specified temperature.

The mass loss only relates to the organic content of peat, organic sand and materials that do not contain minerals that decompose or dehydrate at the test temperature such as clay minerals, calcium carbonate and gypsum. Such minerals might be responsible for the major proportion of the mass loss on ignition.

The requirements of BS 1377-1, where appropriate, shall apply to this test method.

6.2 Apparatus

- 6.2.1** *Drying oven*, capable of maintaining a temperature of $(50 \pm 2.5)^\circ\text{C}$.
- 6.2.2** *Balance*, readable to 1 g.
- 6.2.3** *Balance*, readable to 0.001 g.
- 6.2.4** *Desiccator*, containing anhydrous silica gel.
- 6.2.5** *Test sieves*, 2 mm and 425 μm aperture sizes, with receiver.

6.2.6 *Pestle and mortar*, or a suitable mechanical crusher.

6.2.7 *Sample dividers of the multiple-slot type (riffle boxes)*, having widths of opening of 7 mm and 15 mm.

6.2.8 *Crucible or similar container*, of about 30 ml capacity.

6.2.9 *Electric muffle furnace*, capable of maintaining a temperature of $(440 \pm 25)^\circ\text{C}$.

6.3 Procedure

6.3.1 Preparation of crucible

6.3.1.1 Before starting each series of tests, a test shall be carried out on the empty crucible or container as described in [6.3.1.2](#) to [6.3.1.4](#).

6.3.1.2 The crucible shall be placed in the muffle furnace, heated to $(440 \pm 25)^\circ\text{C}$, and maintained for 1 h.

6.3.1.3 The crucible shall be removed from the furnace and allowed to cool to room temperature in the desiccator.

6.3.1.4 The crucible shall be weighed to the nearest 0.001 g (m_c).

6.3.2 Preparation of test specimen

6.3.2.1 Each test specimen shall be prepared for analysis from the laboratory sample as described in [6.3.2.2](#) to [6.3.2.11](#).

6.3.2.2 The initial sample shall be obtained as described in BS 1377-1:2016, **8.3**, and of the approximate mass as specified in BS 1377-1:2016, Table 5.

6.3.2.3 The sample shall be dried in the oven at $(50 \pm 2.5)^\circ\text{C}$, and cooled to room temperature in the desiccator.

6.3.2.4 The sample shall be weighed to the nearest 0.1% and the mass recorded, m_1 (in g).

6.3.2.5 The sample shall be sieved on a 2 mm test sieve (if appropriate, guarded by a test sieve of larger aperture), and retained particles other than stones shall be crushed to pass the 2 mm test sieve.

6.3.2.6 Fine material adhering to stones shall be brushed off into the sieve. The stones shall then be rejected. The mass of the sample passing the 2 mm test sieve shall be recorded to the nearest 0.1% (m_2). Throughout these and subsequent operations care shall be taken to ensure that there is no loss of fines.

6.3.2.7 The material passing the 2 mm sieve shall be divided by successive riffing through the 15 mm divider to produce a sample weighing at least 10 g.

6.3.2.8 This sample shall be pulverized so that it passes the 425 μm test sieve.

6.3.2.9 The sample shall be subdivided by riffing on the 7 mm divider to obtain test specimens each weighing approximately 5 g. Throughout this and any subsequent operation the material shall be mixed thoroughly before any division and to avoid segregation during riffing.

6.3.2.10 Each specimen shall be placed in a prepared crucible and dried in the oven at a temperature of $(50 \pm 2.5)^\circ\text{C}$. The specimens shall be deemed to be dry when the differences in successive weighings, carried out at intervals of 4 h, do not exceed 0.1% of the original mass of the sample.

6.3.2.11 The specimen shall be allowed to cool to room temperature in the desiccator and each crucible weighed to 0.001 g (m_3).

6.3.3 Ignition of soil

6.3.3.1 Ignite each test specimen as described in [6.3.3.2](#) to [6.3.3.4](#).

6.3.3.2 The crucible with soil of known weight, shall be placed in the unheated muffle furnace, and then heated to $(440 \pm 25)^\circ\text{C}$, and this temperature shall be maintained for not less than 3 h.

NOTE The period required for ignition will vary with the type of soil and size of sample.

6.3.3.3 The crucible and contents shall be removed from the furnace and allowed to cool to room temperature in the desiccator.

6.3.3.4 The crucible and contents shall then be weighed to the nearest 0.001 g (m_4).

6.4 Calculations

6.4.1 The percentage of the original soil sample passing the 2 mm test sieve shall be calculated from the equation:

$$\text{fraction finer than 2 mm} = \frac{m_2}{m_1} \times 100 \quad (5)$$

where

m_1 is the initial dry mass of sample (in g);

m_2 is the mass of the sample passing the 2 mm test sieve (in g).

6.4.2 The mass loss on ignition, LOI, as a percentage of the dry mass of soil passing a 2 mm test sieve shall be calculated from the equation:

$$\text{LOI} = \frac{m_3 - m_4}{m_3 - m_c} \times 100\% \quad (6)$$

where

m_3 is the mass of the crucible and oven-dry soil specimen (in g);

m_4 is the mass of the crucible and specimen after ignition (in g);

m_c is the mass of the crucible (in g).

6.5 Test report

The test report shall state that the test was carried out in accordance with

A1 BS 1377-3:2018+A1:2021 **A1**, [Clause 6](#) and shall contain the following information:

- the method of test used;
- the mass loss on ignition as a percentage of the soil fraction passing the 2 mm test sieve, to two significant figures;
- the percentage by dry mass of the original sample passing the 2 mm test sieve, to the nearest 1%; and
- the information required by BS 1377-1:2016, **10.1**.

7 Determination of sulfur compounds

7.1 General

7.1.1 Principle

This clause describes procedures for determining sulfur species in soils and extremely weak and very weak rocks, as defined in BS 5930, and sulfate in groundwater.

NOTE The tests for sulfur species in this part of BS 1377 may be used on other rocks if required. Sulfur species include sulfate minerals such as gypsum ($\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$), anhydrite (CaSO_4), barite (BaSO_4), jarosite [$\text{KFe}_3(\text{OH})_6(\text{SO}_4)_2$] and sulfide minerals, such as pyrrhotite ($\text{Fe}_{(1-x)}\text{S}$), pyrite (FeS_2), marcasite (FeS_2), together with complex organic sulfur compounds. They are commonly found in both natural ground and in anthropogenic materials. The oxidation of sulfide minerals produces sulfuric acid, which reacts with calcium carbonate (CaCO_3) to produce gypsum. This reaction occurs as a result of natural weathering and also where natural deposits are disturbed by construction work (see Czerewko et al. 2016) [7]. Where there is insufficient calcium carbonate or other mineral to buffer the reaction, acid groundwater might be produced. Further information and references are given in [Annex A](#).

7.1.2 Types of test

Procedures are described for the determination of the following:

- the water-soluble sulfate content of a specimen, for which a water extract is first prepared (see [7.3](#));
- the dissolved sulfate in groundwater (see [7.8](#));
- the acid-soluble sulfate content of a specimen for which an acid extract is first prepared (see [7.9](#));
- the total sulfur content of a specimen ([7.10](#));
- the total reduced sulfur (sulfide) content of a specimen for which an extract is prepared ([7.11](#));
- the acid-soluble sulfide content of a specimen for which an acid extract is prepared ([7.11](#)).

NOTE Guidance is given in BS EN 1744-1 on testing for sulfur compounds in aggregates.

7.2 Sampling, sample storage and sample preparation

7.2.1 General

Samples shall be taken in accordance with the recommendations in BS 1377-1:2016, **8.3** with modifications herein to preserve the original chemistry of the materials (see BRE [2005] [1], TRL 447 [2005] [2]).

The sampling, storage, sub-sampling and testing operations shall be carefully planned at the outset of investigations so changes in the type and amounts of sulfur compounds in the ground are preserved in the samples.

NOTE The use of water or drilling fluid during recovery that might come into contact with the sample should be avoided or, if necessary, kept to a minimum for sample recovery and noted for the samples, or material taken from within undisturbed samples or core that has not been exposed to drilling fluids. Guidance for appropriate sampling, handling and labelling procedures are given in BS EN 1997-2, BS EN ISO 22475-1 and BS 5930. The colour of the sample should be recorded on receipt as described in BS 5930.

Sampling tools shall be clean. Samples shall be taken as soon as practicable with the delay between excavation and sampling kept to a minimum. The time of sampling shall be noted and used to limit the time until tested for the sulfur species. Care shall be taken to avoid samples becoming contaminated with overlying or surface materials. If water is present, this shall also be sampled, as specified in BS EN ISO 22475-1. Notes shall be made concerning the nature of the samples, for example, whether a bulk representative sample or a sample of a specific feature, such as a particular horizon or zone, has been taken. The dates and environmental conditions to which samples are subjected during transportation and storage shall be recorded.

7.2.2 Storage

Samples shall be packed and retained in clean, airtight, individual containers with no or minimal airspace as soon after sampling as is practicable. They shall be stored at between 0 °C and 4 °C for no longer than 14 days after sampling before testing. If storage is anticipated for longer, the samples

shall be dried in a ventilated oven at between 40 °C and 60 °C until a constant mass is reached and placed in airtight containers, or vacuum pouches or under a vacuum and stored under refrigerated conditions (at between 0 °C and 4 °C).

The water loss of material during the drying process shall be recorded and presented with the test results.

7.2.3 Sample preparation

7.2.3.1 Apparatus

- 7.2.3.1.1 *Drying oven*, capable of being maintained at a temperature of between 40 °C and 60 °C.
- 7.2.3.1.2 *Balance*, readable to 1 g.
- 7.2.3.1.3 *Balance*, readable to 0.001 g.
- 7.2.3.1.4 *Sieves*, 5 mm, 2 mm and 212 µm aperture sizes, with receiver.
- 7.2.3.1.5 *Pestle and mortar*, agate or ceramic.
- 7.2.3.1.6 *Mechanical crusher*.
- 7.2.3.1.7 *Binocular microscope*, of magnification x35 or greater.
- 7.2.3.1.8 *Sample dividers of multiple-slot type (riffle boxes)*, having widths of opening of 7 mm and 15 mm.
- 7.2.3.1.9 *Desiccator*, containing anhydrous silica gel.
- 7.2.3.1.10 *Glass weighing-bottle*, approximately 50 mm in diameter, 25 mm high and fitted with a ground-glass stopper.
- 7.2.3.1.11 *Tungsten carbide disc shatter mill*, or equivalent.

NOTE A steel hammer and plate may also be used.

7.2.3.2 Drying of samples

Samples shall be dried in a clean, suitable container at a temperature of between 40 °C and 60 °C in a fan-assisted or conventional ventilated oven to a constant weight. The specimens shall be deemed to be dry when the differences in successive weighings, carried out at intervals of 4 h, do not exceed 0.1% of the original mass of the sample. The samples shall then be allowed to cool to room temperature in the desiccator. The dried sample shall be weighed to the nearest 0.1 g and the mass recorded as m_1 in g.

7.2.3.3 Mechanical processing

7.2.3.3.1 Coarse-grained material

The dried coarse-grained material shall be dry sieved using 5 mm and 2 mm sieves. Material retained on the sieves shall be examined using a binocular microscope of magnification $\times 35$ or similar. If the >5 mm retained fraction consists of inert material such as quartz then the material shall be brushed clean of any adhering particles, which shall be added to the <2 mm fraction, and the inert fragments shall be retained for weighing. If the material consists of weakly cemented grains, they shall be broken down by gentle pounding using a clean agate or ceramic pestle and mortar and re-sieved. If the retained material consists of dried clay, silt, mudstone or siltstone or any material considered to be a potential host for sulfur minerals as identified by a suitably experienced person, it shall be crushed and added to the fraction passing the 2 mm sieve, which is retained for testing.

The <2 mm fraction shall be weighed to the nearest 0.1 g and the mass m_2 shall be recorded in g.

7.2.3.3.2 Fine-grained material

The dried fine-grained material shall be broken down using a clean ceramic mortar and rubber pestle. Any gravel size (>2 mm) material shall be removed for examination under a binocular microscope. Any fragments of inert material shall be brushed clean of any adhering particles, which shall be added to the disaggregated material and retained for weighing. If the retained material consists of clay, mudstone, silt, siltstone or any material considered to be a potential host for sulfur minerals, as identified by a suitably experienced person, it shall be added to the disaggregated fraction which is retained for testing.

The <2 mm fraction shall be weighed to the nearest 0.1 g and the mass m_2 shall be recorded in g.

7.2.3.4 Extremely weak and very weak rock

After drying, samples shall be brushed clean of any loose particles and then broken down into fragments of less than 10 mm using a clean industrial fly press; alternatively, a hammer and steel plate or a steel pestle and mortar or other similar equipment may be used. The material is retained for testing. Care shall be taken when using mechanical crushing devices that samples do not become heated above 60 °C by the pounding action.

NOTE For intact rock samples the tests are carried out on m_1 . The fraction finer than 2 mm, m_2 , is 0.

7.2.3.5 Subdividing

The material retained for testing shall be mixed thoroughly and subdivided by cone and quartering or riffing until a representative sub-sample of between 200 g and 300 g is obtained.

7.2.3.6 Particle size reduction

The particle size reduction shall be accomplished using a clean tungsten carbide disc shatter mill, or an equivalent type of mill. The samples shall be subjected to successive episodes of between 10 and 15 seconds of milling. Longer periods shall be avoided as they might cause alteration to sulfur species. The powdered sample shall be passed through a 212 μm sieve, with any material retained on the 212 μm sieve re-ground and the process repeated.

7.2.3.7 Storage of processed material

When samples are to be stored before testing, the powdered samples shall be oven dried at a temperature of between 40 °C and 60 °C for about 24 hours. The samples shall then be allowed to cool to room temperature in a desiccator with dry desiccant and stored as described in [7.2.2](#).

7.3 Determination of water-soluble sulfate in soil (WS)

7.3.1 Principle

The method measures the water-soluble sulfate of solid samples. The prepared dry sample is mixed with de-ionized water with a ratio of 2:1 water to solid by mass ratio and the resultant filtered solution is tested for sulfate ions. The results obtained give the sulfate content at the time of sampling only.

NOTE 1 The test methods in [7.4](#), [7.5](#), [7.6](#) and [7.7](#) may also be used to determine the sulfate content of samples of groundwater and surface water provided they have been filtered using the method given in [7.3.3.5](#) and [7.3.3.6](#) as required.

NOTE 2 Water-soluble sulfate is an index value that is used for the assessment of the potentially aggressive sulfate that might be readily leached from soil and rocks. The most common sulfate, hydrated calcium sulfate ($\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$, gypsum) has a relatively low solubility of about 1 500 mg/l SO_4 at room temperature. However, other sulfate minerals, particularly those containing magnesium or sodium, have much higher solubility and, hence, pose greater risk to concrete, steel and construction materials. Therefore, it might be necessary to test the water extract for

cations; such as magnesium (Clause 10). The total amount of potentially reactive sulfate is given by the acid-soluble sulfate test in 7.9.

7.3.2 Preparation of water: soil extract

7.3.2.1 General

The requirements of BS 1377-1:2016, where appropriate, shall apply to this test method.

7.3.2.2 Reagents

7.3.2.2.1 *Nitric acid*, analytical grade concentrated nitric acid, 1.42 g/ml (solution = 70%) for use if water samples are to be stored for ICP-AES or gravimetric chemical determination methods only.

7.3.2.2.2 *Hydrochloric acid*, analytical grade concentrated hydrochloric acid, 1.19 g/ml (solution = 40%). Used if samples need to be stored for ICP-AES or gravimetric chemical determination methods only.

7.3.2.3 Apparatus

7.3.2.3.1 For test sample preparation prior to water:soil extraction see 7.2.3.

7.3.2.3.2 *Extraction bottle*, a 250 ml capacity clean and dry, rigid plastic such as high-density polyethylene (HDPE) container with a water tight screw cap. Glass containers should not be used as they are susceptible to damage from the soil particles during the extraction.

7.3.2.3.3 *Mechanical shaker*, a rotary tumbler or mechanical shaker capable of keeping 50 g of soil in suspension in 100 ml of water.

7.3.2.3.4 *Sample bottles*, 30 ml to 50 ml capacity sterile polystyrene or polypropylene sample bottles with leak-proof caps.

7.3.2.3.5 *Vacuum filtration unit with filter funnel, flask and filter paper* (e.g. Whatman No. 542®²⁾).

7.3.2.3.6 *Syringe filter*, 0.45 µm membrane syringe filter.

7.3.2.3.7 *pH meter*, calibrated, electronic pH meter.

7.3.2.3.8 *Laboratory glassware*, including 75 mm diameter watch glass and 50 ml and 25 ml pipettes.

7.3.3 Procedure

7.3.3.1 A 30 g to 50 g representative portion of the oven-dried powdered sample from 7.2.3.6 shall be weighed and put into the 250 ml capacity extraction bottle to an accuracy of 0.001 g and record as m_3 .

7.3.3.2 Between 60 ml and 100 ml of distilled/de-ionized water (BS 1377-1:2016, 6.1) water shall be added, recording the amount to make a 2:1 water to sample mixture (by mass) as V_1 assuming that 1 ml has a mass of 1 g.

7.3.3.3 The extraction bottle shall be tightly stoppered. A blank of around 10 ml of the distilled/de-ionized water (BS 1377-1:2016, 6.1) water for each batch of extractions shall be retained for chemical analysis.

7.3.3.4 The extraction vessel shall be placed in a mechanical shaker or mechanical rotator and agitated for at least 16 h (overnight).

7.3.3.5 The soil suspension shall be filtered under vacuum through the filter funnel, such as a Buchner funnel, into the filter flask, using the filter paper.

²⁾ Whatman is a trademark of GE Healthcare. This information is given for the convenience of users of this document and does not constitute an endorsement by the British Standards Institution of the named product. Equivalent products may be used if they can be shown to lead to the same results.

- 7.3.3.6** If the filtrate is still cloudy it shall be further filtered through a 0.45 µm membrane syringe filter without the addition of water.
- 7.3.3.7** An aliquot of 50 ml (approx.) shall be retained for chemical determination and the measurement of pH (see [Clause 12](#)), however, this measurement is made on a 2:1 extract and shall not to be confused with the pH determination in [Clause 12](#), which is carried out on a 2.5:1 extract.

7.3.4 Methods of chemical analysis

- 7.3.4.1** The chemical tests used for the determination of water-soluble sulfate are ion chromatography (IC), inductively coupled plasma atomic emission spectroscopy (ICP-AES), gravimetric or ion exchange column methods.
- 7.3.4.2** If ion chromatography (IC) method (see [7.4](#)) is used the samples shall not have any chemicals added to preserve them. Dilution might be necessary to bring the sulfate content within the validated range of the IC apparatus or within acceptable levels for the total anion content of the sample and correction made for the dilution. For the procedure see [7.4](#).
- 7.3.4.3** If ICP-AES or similar apparatus (see [7.5](#)) is to be used and the water sample is to be stored then it shall be preserved using 1% v/v nitric acid and 0.5% v/v hydrochloric acid with respect to concentrated analytical grade reagents. An aliquot of the sample shall be taken for analysis and diluted, if necessary, with a 1% nitric acid and 0.5% hydrochloric acid blank, which forms the blank used as part of the analytical run.
- 7.3.4.4** If the gravimetric method of analysis (see [7.6](#)) is to be used, 50 ml of the filtered sample shall be transferred into a clean, dry 500 ml conical beaker and diluted to 300 ml with distilled/de-ionized water (BS 1377-1:2016, [6.1](#)).
- 7.3.4.5** If the ion-exchange column method of analysis (see [7.7](#)) is to be used, no additional water shall be added at any stage and the soil remaining on the filter paper is not washed. Exactly 100 ml of the filtered water shall be transferred to each of two clean dry 250 ml conical beakers.

7.3.5 Test report

The test report shall state that the test was carried out in accordance

[A1](#) BS 1377-3:2018+A1:2021 [A1](#), [7.3](#) and relevant chemical test method and shall contain the following information:

- the method of test used;
- the water-soluble sulfate content to the nearest 10 mg/l as SO₄;
- the percentage by dry mass of the original sample passing a 2 mm test sieve, to the nearest 1%;
- the pH of the water extract to the nearest 0.1 pH unit; and
- the information required by BS 1377-1:2016, [10.1](#).

7.4 Ion Chromatography (IC) method for analysis of water extract or groundwater sulfate

7.4.1 General

Ion chromatography is used in this standard to analyze 2:1 water soluble sulfate and chloride ions and groundwater sulfate. Laboratories shall follow BS EN ISO 17034.

7.4.2 Principle

Anions are determined by injecting a known volume of sample as a solution, into a mobile eluent phase, which passes through a separating column containing a stationary phase (absorbent). This partitions the anions between the stationary absorbent, which differentially retards the ion according

to charge and charge mass, and the moving eluent/sample mixture. A detector analyzes the output at the end of the column and produces a time vs detected anion output. The result is usually given by the equipment computer; alternatively, it is on a printed chromatogram. The values, which are for sulfate and chloride ions are in mg/l.

7.4.3 Reagents

7.4.3.1 Only reagents of analytical grade shall be used. Weigh the reagents with an accuracy of $\pm 1\%$ of the normal mass.

7.4.3.2 *Water*, BS EN ISO 3696:1995, grade 1.

7.4.3.3 *Sodium chloride*.

7.4.3.4 *Sodium sulfate*.

7.4.3.5 *Chemicals for eluents*.

NOTE 1 The choice of eluent depends on the choice of column and detector; seek advice from the column supplier.

NOTE 2 Stock solutions may be made up by the laboratory (see BS EN ISO 17034) or bought from suppliers to laboratories requirements. BS EN ISO/IEC 17025 requires tractability for all reference standards used.

7.4.4 Apparatus

7.4.4.1 *Ion chromatography system*, typically consisting of a gradational pump, an electrochemical detector, a guard column, an absorbance detector, an autosampler and a thermal compartment. The system is controlled and data capture is by a dedicated computer installed with proprietary software. The system shall be regularly serviced by an appropriately qualified service technician.

7.4.4.2 *Eluent reservoir*.

7.4.4.3 *Refrigerator for storing solutions*.

7.4.4.4 *Equipment for making and storing chemical solutions*.

NOTE The concentration of the analyte should be limited to that recommended by the manufacturer and by the laboratory as in [Table 1](#). High concentrations of anions might not be accurately measured and shorten the life of the separating column.

7.4.4.5 The autosampler door shall not be opened during sample injection sequence.

WARNING. There are various hazards associated with ion chromatographs such as operation at high pressure and ultra-violet radiation from the absorbance detector.
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7.4.5 Performance checks

7.4.5.1 General

The required instrument performance, system performance, detector performance and guard column performance checks shall be carried out before each sample run and after servicing.

7.4.5.2 Instrument performance checks

Prior to a run of samples and after each service the instrument shall be checked by monitoring the back pressure and any background conductivity of the system. If these checks show that the check values are out of specification for the instrument as provided by the manufacturer, remedial action shall be taken as required by the manufacturer's instructions.

7.4.5.3 System performance checks

The laboratory shall have a system performance check in which known solutions of mixed anions of interest, such as 50 mg/l chloride (Cl) and 50 mg/l sulfate (SO₄) are used.

7.4.5.4 Detector performance

The detector performance shall be checked before each sample run and after each service, by running the detector performance check solution at the start of the run. The peak areas for the anions in the system performance check on solutions shall be within the required tolerances, otherwise, if outside the required performance, remedial action shall be taken and the test run again.

7.4.5.5 Guard column performance check

The performance of the guard column is checked by running the system performance check solution through the guard column. The capacity factor, k , is calculated as the difference in sulfate and fluoride retention times, $t_2 - t_1$, divided by the fluoride retention time, t_1 . The column shall be cleaned according to the manufacturer's instructions if k is less than 50% of its value when the machine was first brought into service.

7.4.6 Calibration

7.4.6.1 Solutions covering the high and low ranges of the calibration shall be prepared from analytical grade solid reagents including sodium chloride and sodium sulfate. The anion content of the calibration solution covers the range as in [Table 1](#). However, if other anions are to be analyzed at the same time in addition to chloride and sulfate, then they shall be added to the working standard. Freshly prepared high purity water blanks, as used in the preparation of the samples shall also be analyzed. All the calibration solutions and blanks shall be traceable to a primary standard, e.g. BS EN ISO 17034.

NOTE 1 Higher concentration stock solutions for each anion might be prepared from which the calibration solutions are made.

NOTE 2 The typical concentration of calibration ranges defined in this method for inorganic anions are in [Table 1](#).

Table 1 — Example concentration of calibration ranges

Anions	Calibration range (mg/l)
Chloride (Cl ⁻)	2 – 100
Sulfate (SO ₄ ²⁻)	2 – 50

7.4.6.2 At least five working standards shall be used, typical concentration of ions are given in [Table 2](#).

Table 2 — Example anion concentrations in calibration standards

Working standard	Chloride Cl ⁻	Sulfate SO ₄ ²⁻
Unit	mg/l	mg/l
Standard 1	0.25	0.5
Standard 2	5	25
Standard 3	10	50
Standard 4	20	75
Standard 5	50	100

NOTE Other anions may be added if they are also to be analyzed.

7.4.6.3 The reagents, such as sodium chloride and sodium sulfate solid shall be heated in clean containers at (105 ± 4) °C to constant weight and allowed to cool in a desiccator containing dry self-indicating

desiccant. Each solution shall be made up for the required concentration using high purity water typically with the resistivity of about 18.2 MΩcm at 25 °C.

- 7.4.6.4** A calibration shall be performed for each batch of analyses. The calibrations shall be acceptable if the coefficient of determination (r^2) values are 99.7% or better and their offsets are better than ± 0.05 . If the instrument is out of calibration and recalibration is required the instrument shall be shut down and restarted.
- 7.4.6.5** A calibration drift check shall be carried out at the end of the analytical run using the midpoint working standard for each anion (in the case of [Table 2](#) it is Standard 3). The data shall be within 10% of the target value.
- 7.4.6.6** The concentration of the anions in the test solutions shall not be greater than the calibration range.
- 7.4.6.7** The ions of interest shall be separated and measured using a system typically comprising of inline ultrafiltration, guard column, analytical column, suppressor device and conductivity detector (CD).

7.4.7 Test procedure

- 7.4.7.1** The system shall be configured and run according to the manufacturer's instructions. The availability of sufficient volumes of eluent and helium or other suitable gas as identified by the manufacturer of the system for the time of the analytical run shall be confirmed. The test shall be carried out as in the manufacturer's instructions.
- 7.4.7.2** Freshly prepared water blanks shall be included in the run.
- 7.4.7.3** Where necessary, the sample shall be diluted with high purity water either to bring the individual determinand into the calibrated analytical range or the sample matrix to an acceptable level (for instance below a maximum total anion content, e.g. 1 000 mg/l total anion content). Details of sample dilution shall be recorded.

NOTE Samples that contain moderate or high levels of aluminium, iron or zinc are liable to result in column contamination, so suspect samples should be pre-analyzed, such as with an ICP method.

- 7.4.7.4** The test shall proceed as required by the apparatus manufacturer's instructions.
- 7.4.7.5** All chromatograph data and chromatograms shall be saved on a computer system and inspected for quality after completion of the analysis. If errors are identified then the value may be changed by a qualified operator and the reason for the change shall be documented and reported with the results.

7.4.8 Calculations – Corrections for dilutions and final concentration

Dilution factor (DF) shall be calculated from equation (9) and is the ratio of the volume of the initial concentration solution to the volume of the final, diluted solution.

$$DF = \frac{V_2}{V_1} \quad (7)$$

where

- V_1 is the volume of the undiluted solution;
- V_2 is the volume of the diluted solution that is tested.

$$\rho_c = \rho_i \times DF \quad (8)$$

where

ρ_c is the concentration of the initial solution;
 ρ_i is the concentration of the diluted solution that is tested.

The dilution factor = 1 if not diluted.

The final concentration, ρ_f is:

$$\rho_f = \rho_c - \rho_b \quad (9)$$

where

ρ_b is the concentration of the blank solution.

The blank solution shall not be diluted, but if it is, the sample shall be diluted and this shall be noted.

7.5 Inductively coupled plasma atomic emission spectrometer (ICP-AES) method for analysis of acid or water extract or groundwater sulfate

7.5.1 General

This method of analysis is suitable for determination of the concentration of sulfur species ([Clause 7](#)) and magnesium ([Clause 10](#)) and many other elements in solution. The general method for ICP-AES, also known as ICP-OES, is covered in BS EN ISO 11885, which is intended primarily for water quality. ICP-AES measures the concentration of elemental sulfur (S), magnesium (Mg) and copper (Cu), rather than the concentration of the SO_4 anion per se. The maximum concentration of the different elements that can be tested are instrument specific. Where the extracted sample is expected to contain higher concentrations, dilution shall be diluted.

NOTE Inductively coupled plasma mass spectrometer (ICP-MS) method may also be used. This equipment is designed to measure very low concentrations, which are not relevant for the purposes of this British Standard.

7.5.2 Principle

The prepared liquid sample is converted into an aerosol (nebulized), which is transported by a plasma torch where excitation occurs. Characteristic emission spectra are produced by an inductively coupled plasma (ICP). The spectra are dispersed by a grating spectrometer and the intensities of the lines are measured with a detector. The signals from the detector(s) are processed and controlled by a computer system.

7.5.3 Interferences

To avoid interference with other ions, the method used shall be carefully reviewed to identify if a new method of sample preparation should be developed to validate the method.

NOTE Several spectrum interference effects at different wavelengths can contribute to inaccuracies in the determination of elements. For sulfur the interference at a wavelength of 180.669 nm is caused by arsenic (As) and calcium (Ca) and at a wavelength of 181.975 nm the interference is caused by chromium (Cr) and molybdenum (Mb). For magnesium the interference at wavelength 279.078 and 279.553 nm is caused by iron (Fe) and interference at wavelength 285.213 nm caused by chromium (Cr). For copper the interference at wavelength 324.754 nm is caused by molybdenum (Mo), cobalt (Co) and iron (Fe) and at 327.393 nm it is cobalt (Co) and titanium (Ti). Comparison tests using methods other than ICP-AES may also be used.

7.5.4 Calibration

7.5.4.1 General

All the calibration solutions and blanks shall be traceable to a primary standard, e.g. BS EN ISO 17034.

7.5.4.2 Preparation of calibration solutions

A minimum of five calibration solutions shall be used covering the range of elements in test solutions.

Typical calibration standards as a % of the upper limit of the apparatus for the various sulfate and sulfur extractions, and groundwater sulfur, copper and magnesium extractions are given in [Table 3](#).

Table 3 — *The concentration as a % of the upper limit of the apparatus of the different calibration standards for ICP-AES*

Standard 1	Standard 2	Standard 3	Standard 4	Standard 5
0.5	2.5	25	50	100

Examples of five calibration standards are given in Table 4.

Table 4 — *Example of five calibration standards for copper, magnesium and sulfur*

Analyte	Standard 1	Standard 2	Standard 3	Standard 4	Standard 5
	mg/l	mg/l	mg/l	mg/l	mg/l
Cu	2.5	12.5	125	250	500
Mg	2.1	10.6	106	213	425
S	6	30	300	600	1200

7.5.4.3 Preparation of calibration blank

A 100 ml volumetric flask shall be filled with high purity water. The calibration standards shall be labelled and the expiry date of the solution shall match the expiry date of the standard(s) used to prepare the solution.

7.5.4.4 Calibration procedure

Calibration shall be performed at the start of the analytical run and validated by Continued Calibration Verification (CVV) standard (see [7.5.5](#)). The correlation coefficient of the validation shall be 0.99 or better. Check standards shall be within prescribed limits as determined by the laboratory during method validation and shall be within 10% of the specified values.

NOTE Calibration charts might be automatically created by the apparatus software.

7.5.5 Quality control

The elements of interest shall be analyzed during an analytical run against a relevant working quality control (QC) check standard, and this shall be prepared in accordance with laboratories operating procedures. Additionally, the Continuing Calibration Verification (CCV) standard shall be measured at the beginning and at the end of each run, and at regular intervals throughout the run. Verification shall be carried out in accordance with the requirements of the laboratory operating procedures. Non-conformities shall be documented.

7.5.6 Procedure

7.5.6.1 The apparatus shall be checked to ensure that it is in good working order and that checks as required by the manufacturer and the laboratory's requirements and procedures have been carried out.

7.5.6.2 The method and calibration files covering all the elements to be determined and the list of solutions to be analyzed shall be loaded into the computer software.

7.5.6.3 The auto sampler, if used, shall be loaded ensuring that there is sufficient volume of sample to carry out the analysis as required from the prepared solution from [Clause 7](#) and [Clause 10](#) as required. The software that starts the test procedure shall then be run.

7.5.6.4 On completion of the run any specific comments regarding analysis as required by the laboratory quality system shall be made.

7.5.7 Data manipulation

7.5.7.1 All the data, mean calculated concentrations and unit information, shall be stored in suitable software.

7.5.7.2 The raw data shall be checked to ensure that it is within the calibrated range. Any samples that are over range shall be diluted as required and reanalyzed.

7.5.7.3 Median concentrations obtained from blanks analyzed throughout the analytical run shall be calculated and subtracted from sample data as shown in [7.5.8.1](#).

7.5.7.4 If results on one sample differ by no more than 50 mg/l (S), the mean result shall be calculated. If they differ more than 50 mg/l the test shall be repeated with two new analytical portions of the soil.

7.5.8 ICP Calculations

7.5.8.1 The correction for dilution shall be calculated from:

$$\rho_c = \rho_i \times DF \quad (10)$$

The dilution factor (see [7.4.8](#)) = 1 if not diluted.

The final concentration, ρ_p is $\rho_f = \rho_c - \rho_b$.

where

ρ_b is the concentration of the blank solution.

The blank solution shall not be diluted, but if it is, this shall be corrected by using the dilution factor.

Record the final concentration of sulfur, ρ_p (mg/l).

7.5.8.2 The conversion from elemental sulfur to sulfate concentration shall be calculated from equation (11) and shall be in mg/l:

$$SO_4 = \rho_f \times 3 \quad (11)$$

where

ρ_f is the measured sulfur in mg/l.

7.5.8.3 As appropriate, the results shall be reported as water-soluble sulfate in soil (WS) or groundwater (GWS) in mg/l using equation [12].

$$\text{Water-soluble sulfate (mg/l)} = SO_4 \text{ (mg/l)} \quad (12)$$

As appropriate, the results shall be reported as acid-soluble sulfate in soil (AS) in % using equation [13]

$$\text{Acid-soluble sulfate (\%)} = SO_4 \text{ (mg/l)} \times 0.01/m_3 \quad (13)$$

where