The quantity of copper in the blank solution in mg Cu, p_1, is given by:

$$p_1 = \frac{Tr_1 \times V_1}{1000} \tag{33}$$

The quantity of copper in the test solution in mg Cu, p_2, is given by:

$$p_2 = \frac{Tr_2 \times V_2}{1000} \tag{34}$$

As one atom of sulfur combines with one atom of copper, the quantity of sulfur liberated in the reaction may be determined from the quantity of copper used. Determine this value in mg S to an accuracy of 0.01 mg and record as m_4.

$$m_4 = 0.504(p_1 - p_2) \tag{35}$$

This value is divided by the test sample mass, m_3, to obtain the monosulfide sulfur (MS). Record this value in % S to the nearest 0.01%.

$$\text{MS}(\%\text{S}) = \frac{m_4}{m_3} \times 100 \tag{36}$$

NOTE See 7.9 for the calculation of acid-soluble sulfate.

If the acid-soluble sulfate is also being determined, calculate the amount of sulfur in the digested solution in mg S and record as m_5.

$$m_5 = \frac{sr_1 \times dV_1}{1000} \tag{37}$$

Convert m_5 to the acid-soluble sulfur in the sample in % S to the nearest 0.01% and record as m_6.

$$m_6 = \left(\frac{m_5}{m_3}\right) \times 100 \tag{38}$$

Convert m_6 to the acid-soluble sulfate (AS) in % SO_4 to the nearest 0.01% and record as AS.

$$\text{AS} = 3 \times m_6 \tag{39}$$

7.12.6 Test report

The test report shall state that the test was carried out in accordance with BS 1377-3:2018, **7.12** and shall contain the following information:

a) the method of test used;

b) the monosulfide sulfur content (MS) to the nearest 0.01% (as S) of the oven-dry mass of soil passing a 2 mm test sieve;

c) the percentage by dry mass of the original sample passing a 2 mm test sieve;

d) the acid-soluble sulfate shall be reported as described in 7.9; and

e) the information required by BS 1377-1:2016, **10.1**.

8 Determination of the carbonate content

8.1 Types of test method

This clause describes three test methods for the determination of calcium carbonate content of soil and rocks:

a) Thermal decomposition of the carbonate method (**8.2**), which produces carbon dioxide and is measured with a detector. This is the preferred method and is suitable for rocks or soils.

b) Rapid titration method for determination of calcium carbonate content (**8.3**), which is a screening method suitable for rock and soils with more than 10% carbonate content (m/m), and where an accuracy of about 1% is sufficient. In this method the soil specimen is treated with a known quantity of acid to finality. The amount of excess acid is determined by titration against sodium hydroxide. The result is calculated in terms of the equivalent proportion of carbon dioxide.

c) Gravimetric method for determination of calcium carbonate content (**8.4**), as described for hardened concrete in BS 1881-124, to which reference is made. In the gravimetric method the carbon dioxide evolved when the rock or soil is treated with hydrochloric acid is passed through a granular absorbent which enables the mass of carbon dioxide to be determined gravimetrically.

The thermal decomposition method may be used to determine the carbonate content of siderite ($FeCO_3$), ankerite $Ca(Fe,Mg,Mn)(CO_3)_2$, dolomite $CaMg(CO_3)$ or other carbonate minerals as separate mineral species. However, the rapid titration and gravimetric methods only determine the calcium carbonate content.

NOTE The thermal decomposition of carbonate species may be undertaken which is particularly useful when evaluation of structural heave due to the expansion of fill containing pyrite and other sulfide minerals is being considered, where calcium carbonate buffers the products of sulfide oxidation to produce gypsum.

8.2 Total carbon analyzer, combustion method — Total inorganic carbon (TIC)

8.2.1 Principle

This procedure covers the determination of the percentage of total inorganic carbon (TIC) present in a sample by total carbon analyzer using the decomposition of carbonate and combustion of organic material method. The total carbon content (TC), i.e. the organic and inorganic carbon is determined on the whole sample. Another subsample is then treated with acid to remove inorganic carbon and the procedure repeated to give the total organic carbon, TOC, as described in Clause **5**. The total inorganic carbon, TIC, is then calculated as the difference:

$$\mathrm{TC} - \mathrm{TOC} = \mathrm{TIC} \qquad (40)$$

The carbonate content is then calculated from the total inorganic carbon (TIC).

8.2.2 Preparation of sample

Samples shall be prepared in accordance with BS 1377-1:2016, **8.3** and **8.5**.

8.2.2.1 Reagents

The reagents as listed in **5.2** shall be used.

8.2.2.2 Apparatus

The apparatus used shall be that described in **5.3**.

8.2.3 Preparation of test specimen

The sample shall be prepared as in 5.4.

8.2.4 Test procedure

8.2.4.1 A representative sample of the dried prepared material (5.4) shall be placed into the total carbon analyzer (5.5.2).

8.2.4.2 The total carbon (TC) shall be measured as given in 5.5. The total inorganic carbon (TIC) shall be removed using the method described in **5.5.1.2** to **5.5.1.10** and analyze for total organic carbon (TOC) as described in 5.5.2.

NOTE If it is known that there is no organic content then TC = TIC and the acidification stage may be omitted.

8.2.5 Calibration

8.2.5.1 The performance of the instrument shall be checked before each batch of analyzes and after each service.

8.2.5.2 The combustion analyzer shall be calibrated using a multi-point calibration of certified carbon standards with different inorganic carbon and organic contents following the manufacturer's instructions.

8.2.5.3 The analysis of samples and check standards shall be as per manufacturer's instructions.

8.2.5.4 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 and if both of these duplicate values exceed this value, remedial action shall be taken to identify and correct it.

NOTE Any chloride in the sample might corrode the analyzer.

8.2.6 Calculation

8.2.6.1 Sample

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

$$\text{fraction finer than 2 mm} = \frac{m_2}{m_1} \times 100\% \qquad (41)$$

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).

8.2.6.2 Carbonate content as CO_2

The carbonate content shall be calculated from total inorganic carbon = total carbon – total organic carbon;

Or, in the absence of organic matter;

Total inorganic carbon = total carbon;

Carbonate content as CO_2 % = total inorganic carbon × 3.67.

8.2.7 Test report

The test report shall state that the test was carried out in accordance with BS 1377-3:2018, Clause **8** and shall contain the following information:

a) the method of test used;

b) the average total inorganic carbon as CO_2 (present in the soil fraction passing a 2 mm test sieve to the nearest 0.1% of the original oven dry mass of soil);

c) the percentage by dry mass of the original sample passing the 2 mm test sieve to the nearest 1%; and

d) the information required by BS 1377-1:2016, **10.1**.

8.3 Rapid titration method for determination of calcium carbonate content

8.3.1 Preparation of sample

8.3.1.1 Apparatus

8.3.1.1.1 *Drying oven*, controlled to a temperature range of 75 °C to 80 °C, as well as 105 °C to 110 °C.

8.3.1.1.2 *Balance*, readable to 0.001 g.

8.3.1.1.3 *Desiccator*, containing anhydrous silica gel.

8.3.1.1.4 *Test sieves*, 2 mm and 425 µm aperture sizes, with receiver.

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

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

8.3.1.1.7 *Glass weighing bottle*, approximately 50 mm diameter, 25 mm high and fitted with a ground glass stopper.

8.3.1.1.8 *Glass or plastics funnel*, about 100 mm diameter.

8.3.1.2 Preparation of soil sample

8.3.1.2.1 An initial sample as described in BS 1377-1:2016, **8.3.1**, and of the appropriate size specified in BS 1377-1:2016, **8.5** shall be obtained.

8.3.1.2.2 This sample shall be dried in an oven at 105 °C to 110°C and then cooled to room temperature in the desiccator.

8.3.1.2.3 The sample shall be sieved on a 2 mm test sieve (if appropriate, guarded by a sieve of a larger aperture).

8.3.1.2.4 All the retained particles shall be crushed to pass the 2 mm sieve and mixed thoroughly with the material already passing the sieve.

8.3.1.2.5 The material passing the 2 mm sieve shall be divided by successive riffling through the 15 mm divider to produce a representative sample of approximately 50 g.

8.3.1.2.6 This sample shall be sieved on a 425 µm test sieve.

8.3.1.2.7 All the retained particles shall be crushed to pass the 425 µm test sieve, and mixed thoroughly with the material already passing the sieve.

8.3.1.2.8 This sample shall be used for preparing the specimens for testing (see **8.3.2.6** and also **8.4.4**).

8.3.2 Rapid titration test procedure

8.3.2.1 General

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

8.3.2.2 Reagents

8.3.2.2.1 All reagents shall be of recognized analytical reagent quality.

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

8.3.2.2.2 *Hydrochloric acid*, [c(HCl) = approximately 1 mol/l]. Dilute 88 ml of concentrated hydrochloric acid with distilled/de-ionized water (BS 1377-1:2016, **6.1**) water to make 1 l of solution.

8.3.2.2.3 *Sodium hydroxide solution*, [c(NaOH) = approximately 1 mol/l]. Dissolve about 20 g of sodium hydroxide in 500 ml of distilled/de-ionized water (BS 1377-1:2016, **6.1**) and store in an airtight plastic container. Determine the concentration (B) of this solution by preparing a 0.1 B diluted solution (pipette 25 ml of the concentrated solution into a 250 ml volumetric flask and dilute to 250 ml with distilled/de-ionized water (BS 1377-1:2016, **6.1**). Determine the concentration (B/10) of the diluted solution as described in 7.7.2.4 and multiply by 10 to obtain the concentration (B) of the concentrated solution.

8.3.2.2.4 *Screened methyl orange indicator*.

NOTE Screened methyl orange gives a more distinct end-point than the unscreened indicator but the latter may be used if preferred. Methyl red or bromcresol green are also suitable.

8.3.2.3 Apparatus

8.3.2.3.1 *250 ml tall-form beaker and watch glass cover.*

8.3.2.3.2 *Two 100 ml burettes reading to 0.1 ml.*

8.3.2.3.3 *25 ml pipette.*

8.3.2.3.4 *250 ml conical flask.*

8.3.2.3.5 *1 l volumetric flask.*

8.3.2.4 Standardization of the hydrochloric acid

8.3.2.4.1 25 ml of the hydrochloric acid shall be pipetted into a 250 ml conical flask.

8.3.2.4.2 The conical flask shall be placed on a white background and the sodium hydroxide solution added slowly from a burette (see Note). During this operation, the flask shall be rotated constantly with one hand while the stopcock on the burette is controlled with the other.

NOTE The burette that contained sodium hydroxide should be cleaned soon after use by thoroughly rinsing with water. The burette can be damaged and the tap seize if this is not done.

8.3.2.4.3 The sodium hydroxide shall be added until the acid is neutralized as indicated by the colour change.

8.3.2.4.4 The volume of sodium hydroxide used shall be recorded, V_1.

8.3.2.4.5 8.3.2.4.1 to 8.3.2.4.4 shall be repeated using two more 25 ml aliquots of acid solution. The volumes of sodium hydroxide used for each titration shall not differ by more than 0.1 ml.

8.3.2.4.6 The following shall be calculated; the mean volume of sodium hydroxide used V_1 (in ml) and the concentration (H) of the hydrochloric acid solution (in mol/l), from the following equation:

$$H = \frac{V_1 B}{25} \qquad (42)$$

where

B is the concentration of the sodium hydroxide solution (in mol/l).

8.3.2.5 Preparation of the test sample

8.3.2.5.1 The test sample shall be prepared in accordance with **8.3.1.2**, taking representative portions containing a little over 5 g of oven-dry soil.

8.3.2.5.2 This proportion shall be dried in the oven at 105 °C to 110 °C . The specimen shall be deemed to be dry when the difference in successive weighings at intervals of 4 h do not exceed 0.1% of the specimen mass.

8.3.2.5.3 The specimen shall be allowed to cool to room temperature in the desiccator.

8.3.2.5.4 Approximately 5 g of dry soil shall be weighed as the test specimen and its mass (m) recorded to the nearest 0.001 g.

8.3.2.6 Analysis of test specimen

8.3.2.6.1 The weighed specimen shall be placed into the 250 ml tall-form beaker.

8.3.2.6.2 100 ml of the hydrochloric acid solution shall be added slowly from the burette.

8.3.2.6.3 The beaker shall be covered with the watch glass and allowed to stand for 1 h, stirring occasionally.

8.3.2.6.4 When the soil has settled after the final stirring, 25 ml of the supernatant liquid shall be removed with the pipette and transfer to a conical flask.

8.3.2.6.5 Six drops of the indicator solution shall be added to the liquid in the conical flask and titrated with the sodium hydroxide solution as described in 8.3.2.4 until the same colour change as was observed in the standardization procedure (**8.3.2.4.3**) occurs. The volume (V_2) of sodium hydroxide solution used shall be recorded, to the nearest 0.1 ml.

8.3.2.7 Calculations

8.3.2.7.1 Sample

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

$$\text{fraction finer than 2 mm} = \frac{m_2}{m_1} \times 100 \qquad (43)$$

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).

8.3.2.7.2 Carbonate content

The carbonate content of the soil, as a percentage of CO_2, shall be calculated from the equation:

$$\text{carbonate (as } CO_2\text{)} = \frac{8.8(25H - BV_2)}{m} \qquad (44)$$

where

H is the concentration of the hydrochloric acid (in mol/l);

B is the concentration of the sodium hydroxide solution (in mol/l);

m is the mass of the soil specimen (in g);

V_2 is the volume of sodium hydroxide used (in ml).

8.3.2.8 Test report

The test report shall state that the test was carried out in accordance with BS 1377-3:2018 and shall contain the following information:

a) the method of test used;

b) the percentage carbonates in the soil sample, expressed as CO_2, to two significant figures;

c) the percentage by dry masss of the original sample passing the 2 mm test sieve to the nearest 1%; and

d) the information required by BS 1377-1:2016, **10.1**.

8.4 Gravimetric method for determination of calcium carbonate content

8.4.1 General

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

8.4.2 Apparatus

8.4.2.1 Apparatus shall be as described in BS 1881-124:2015, **6.3**.

8.4.2.2 *Balance*, readable to 0.0001 g.

8.4.3 Reagents

The reagents shall be as described in BS 1881-124:2015, **6.2**.

8.4.4 Preparation of test specimen

8.4.4.1 From the sample prepared as described in 8.3.1 a representative test specimen of the required mass shall be taken.

NOTE As an approximate guide, the amount of soil required will range from about 0.2 g for a pure limestone or chalk to 1 g for a relatively non-calcareous soil. If in doubt the appropriate quantity should be ascertained from preliminary trial tests.

8.4.4.2 The specimen shall be dried in the oven at 105 °C to 110 °C. The specimen shall be deemed to be dry when the difference in successive weighings at intervals of 4 h do not exceed 0.1% of the specimen mass.

8.4.4.3 The specimen shall be allowed to cool to room temperature in the desiccator.

8.4.4.4 The mass of the specimen shall be determined to 0.0001 g.

8.4.5 Analysis of test specimen and calculations

The procedure followed shall be as described in BS 1881-124:2015, **6.7**.

8.4.6 Calculation

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

$$\text{fraction finer than 2 mm} = \frac{m_2}{m_1} \times 100 \qquad (45)$$

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).

8.4.7 Test report

The test report shall state that the test was carried out in accordance with BS 1377-3:2018, **8.4** and shall contain the following information:

a) the method of test used;

b) the percentage carbonate in the soil sample, expressed as CO_2;

c) the percentage of dry mass of the original sample passing a 2 mm test sieve; and

d) the information required by BS 1377-1:1990, **10.1**.

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9 Determination of the chloride content

9.1 General

9.1.1 Principle

This clause describes the preparation of a sample for the determination of the chloride content and the analysis of the prepared sample. Analysis for water soluble and groundwater soluble chloride of the test material might be done using ion chromatography, which is preferred and water soluble, groundwater soluble chloride acid soluble chloride salt content may be done using test procedures based on Volhard's method.

NOTE 1 The principle can also be used for determining chlorides in ground water for which Mohr's method is more suitable than Volhard's method, see BRE Report 279 [3].

NOTE 2 Alternative methods based on potentiometric titration may be used as found in BS EN 1744-1.

For the determination of water-soluble chlorides (see **9.2**) the chlorides are extracted from a dry soil sample by solution in a mass of water equal to twice that of the sample. Results are expressed as the chloride ion content.

A qualitative test for checking for the presence of chlorides is included, which if negative obviates the need for the quantitative analysis.

For the determination of acid-soluble chlorides (see **9.3**), which includes chlorides not extracted by water, chlorides are extracted from a dry soil sample with dilute nitric acid.

Results are expressed as the chloride content.

9.1.2 Applications

The water-extract chloride content is applicable only to materials in which the chloride content derives directly from recent contact with, or immersion in, saline water.

NOTE The principle can also be used for determining chlorides in ground water for which Mohr's method is more suitable, see BRE Report 279 [3].

The acid-extract method is applicable to the determination of the chloride content of soils from desert areas or where the origin of the chlorides is uncertain.

9.2 Determination of water-soluble chloride content

9.2.1 General

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

9.2.2 Apparatus

9.2.2.1 *Balance*, readable to 1 g.

9.2.2.2 *Balance*, readable to 0.001 g.

9.2.2.3 *1 l volumetric flask.*

9.2.2.4 *10 ml graduated glass measuring cylinder.*

9.2.2.5 *500 ml graduated glass measuring cylinder.*

9.2.2.6 *100 ml pipette.*

9.2.2.7 *25 ml pipette.*

9.2.2.8 *Two 50 ml burettes.*

9.2.2.9 *Stoppered conical flasks*, 250 ml capacity. (At least four.)

9.2.2.10 *Wash bottle*, preferably made of plastics, containing distilled/de-ionized water (BS 1377-1:2016, **6.1**).

9.2.2.11 *Amber-coloured glass reagent bottle*.

9.2.2.12 *Wide mouth screw-capped plastics or metal bottle*, of 2 l capacity. The cap shall be watertight when closed.

9.2.2.13 *Mechanical shaking apparatus*, capable of keeping 500 g of soil and 1 000 ml of water contained in the bottles in continuous suspension.

NOTE A device which rotates the containers end over end at 30 r/min to 60 r/min is satisfactory. Shaking machines giving a vibrating motion are also suitable.

9.2.2.14 *Drying oven*, capable of being controlled to maintain temperatures of (105 ±5) °C and (150 ±5) °C, conforming to BS 1377-1:2016, **4.2.2.1**.

9.2.2.15 *Desiccator containing anhydrous silica gel*.

9.2.2.16 *Filter funnel*, of approximately 100 mm diameter.

9.2.2.17 *Filter papers*, of a diameter appropriate to the size of the funnel: medium grade (e.g. Whatman No. 40[7]) and fine grade (e.g. Whatman No. 42[7]).

9.2.2.18 *Test sieve*, of 2 mm aperture size, with receiver.

9.2.2.19 *Sample dividers of multiple-slot type (riffle boxes),* conforming to BS 1377-1:2016, having widths of opening of 7 mm and 15 mm.

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

9.2.2.21 *500 ml volumetric flask.*

9.2.2.22 *Two beakers*, of about 250 ml capacity.

9.2.3 Preparation of test specimen

9.2.3.1 A specimen for analysis shall be prepared from the laboratory sample as given in 9.2.3.2.

9.2.3.2 An initial sample shall be obtained, as described in 9.2.3.3 **to** 9.2.3.8, and of the approximate size specified in BS 1377-1:2016, Table 5.

9.2.3.3 This sample shall be dried in an oven at 105 °C to 110 °C, and allowed to cool to room temperature in the desiccator containing dry desiccant.

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

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[7] 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.

9.2.3.5 The stones shall be rejected, ensuring that no fine material adheres to them, e.g. by brushing. The mass m_2 shall be recorded in g of sample passing the 2 mm test sieve to the nearest 0.1%. Throughout these and subsequent operations there shall be no loss of fines.

9.2.3.6 The material passing the 2 mm test sieve shall be divided by successive riffling through the 1 mm divider to produce the following:

a) a test specimen of about 500 g; and

b) a specimen of about 50 g for a qualitative check test to determine whether chlorides are present.

9.2.3.7 The specimens shall be dried in the oven at 105 °C to 110 °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.

9.2.3.8 The specimens shall be allowed to cool to room temperature in the desiccator containing dry desiccant.

9.2.4 Qualitative check for chlorides

9.2.4.1 Reagents

9.2.4.1.1 All reagents shall be of recognized analytical reagent quality.

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

9.2.4.1.2 *Silver nitrate solution* [$c(AgNO_3) = 0.100$ mol/l]. Dry about 20 g of silver nitrate at not more than 150 °C for 1 h to 2 h and allow to cool in the desiccator. Weigh out 16.987 g of the dried silver nitrate, dissolve in distilled/de-ionized water (BS 1377-1:2016, **6.1**) and dilute to 1 l in a volumetric flask. Store the solution in the amber-coloured glass reagent bottle and protect from prolonged exposure to sunlight.

9.2.4.2 Procedure

9.2.4.2.1 The presence of chlorides in the soil shall be verified as described in 9.2.4.2.2 to 9.2.4.2.5.

9.2.4.2.2 50 g check specimen shall be placed in a 500 ml conical flask and to it an approximately equal mass of distilled/de-ionized water shall be added (BS 1377-1:2016, **6.1**).

9.2.4.2.3 The contents shall be agitated intermittently for 4 h, allowed to settle and some of the supernatant solution poured into a beaker.

9.2.4.2.4 About 25 ml of clear solution shall be obtained by filtering, if necessary, through a medium grade filter paper, e.g. Whatman No. 40®8).

9.2.4.2.5 The liquid shall be acidified with nitric acid, add about five drops of the silver nitrate solution shall be added and allowed to stand for 10 min.

NOTE If no turbidity is apparent after this time the soluble chloride ion content of the soil is not likely to cause harm to construction materials and the quantitative test for chloride content is not necessary.

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[8] 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.