

## Determination of Total Sulphate of Soils

Lab Test Reference 428  
British Standard Reference  
BS 1377 1975 Test 9

### Principal Apparatus

- i. A thermostatically controlled drying oven capable of maintaining a temperature of 75°-80°C (the normal drying oven can usually be adjusted to cover this range as well as the range 105°-110°C - Lab Inventory No. xxx.
- ii. Analytical balance Inventory Number 81.
- iii. A glass weighing bottle approximately 25mm in diameter, 50mm high and fitted with a ground glass stopper.
- iv. Two conical beakers of 500ml capacity and cover glasses to fit.
- v. A glass filter funnel about 100mm diameter.
- vi. A porcelain or silica crucible 35mm in diameter and 40mm in height or preferably a porous porcelain or silica filter and ignition crucible 35mm in diameter and 40mm in height with a suitable Gooch funnel and rubber cone.
- vii. A suitable means of igniting the precipitate preferably an electric muffle furnace capable of reaching and maintaining 800°C.
- viii. A desiccator containing anhydrous silica gel.
- ix. Whatman No. 541 filter papers 110mm diameter and Whatman No. 44 filter papers.
- x. BS Test Sieve 2mm and 425 micron.
- xi. Riffle boxes widths of opening of 7mm and 15mm.
- xii. A pestle and mortar or a suitable mechanical pulveriser.
- xiii. A filter pump.
- xiv. Two glass rods about 150mm to 200mm long and 3mm to 5mm in diameter.
- xv. A wash bottle containing distilled water.
- xvi. Rubber tubing.

### Reagents

The following reagents are required. They shall be of recognised analytical reagent quality.

1. Barium chloride 5% solution. Dissolve 5 grms of barium chloride in 1 litre of distilled water.

2. Dilute hydrochloric acid. Dilute 100ml of concentrated hydrochloric acid (relative density 1.18) to 1 litre with distilled water.
3. Dilute ammonia solution. Dilute 500ml of ammonia (relative density 0.88) to 1 litre with distilled water.
4. Blue litmus paper.
5. Silver nitrate 5% solution. Dissolve 0.5g of silver nitrate in 10ml of distilled water.
6. Bromine water. Shake 6ml of liquid bromine with 500ml of distilled water.

#### 1.0 Preliminaries

- 1.1 The chemistry laboratory shall be used to perform this test and clear working areas of bench must be prepared before testing proceeds.
- 1.2 Ensure the sample number and test schedule correspond.
- 1.3 Obtain the appropriate test work sheet from the cabinet.
- 1.4 Check the calibration status of the equipment and sieves.
- 1.5 Check the conditions of the sieves before testing commences.

#### 2.0 Standard Test Method

##### 2.1 Preparation of soil sample for analysis

The bulk sample obtained as described in the procedure for the preparation of disturbed samples testing (see Para 1.5 in BS 1377) shall be dried in the oven at a temperature not exceeding 80°C (see Note 1) and cooled.

- 2.2 It shall then be weighed to nearest 0.1% of its original mass and mass M1 recorded.
- 2.3 It shall then be sieved on a 2mm BS test sieve and all particles other than stones (see Note 2) crushed to pass through the sieve. The stones may be rejected and the mass of material passing the test sieve M2 shall be recorded to the nearest 0.1% of its total mass.
- 2.4 Through these and all subsequent operations care shall be taken to ensure that there is no loss of fines.
- 2.5 The material passing the 2mm BS test sieve shall be divided by successive riffing through the 15mm divider to produce a sample weighing approximately 100g. This sample shall then be pulverised so that it passes the 425 micron BS sieve.
- 2.6 The samples shall be subdivided by riffing through the 7mm divider until a sample weighing approximately 10g is obtained. Throughout this and any subsequent operation the material available for any division shall be mixed thoroughly and precautions taken to avoid segregation during riffing.

- 2.7 The sample shall be placed in the glass weighing bottle and dried at a temperature not less than 75° and not more than 80°C. The samples shall be deemed to be dry when the differences in successive weighings carried out at intervals of 4 hours do not exceed 0.1% of the original mass of the sample. Drying over night is usually sufficient.
- 2.8 Preparation of the acid extract
- The weighing bottles containing the dried soil shall be removed from the oven cooled in a desiccator and weighed to the nearest 0.001g. A sample about 2g (see Note 3) shall then be transferred to 500ml beaker, the weighing bottle reweighed, and the mass of soil M3 shall be calculated by difference.
- 2.9 200ml of 10% hydrochloric acid shall be added care being taken if effervescence occurs to ensure that no material is lost. The beaker shall be covered with a clockglass and contents boiled gently 4-5 minutes.
- 2.10 The underside of the clockglass shall be rinsed back into the beaker and 3ml of bromine water added whilst the solution continues to boil.
- 2.11 Ammonia solution shall be added slowly preferably from a burette with constant stirring to the boiling solution until the sesquioxides are precipitated and a liquid smells slightly of ammonia.
- 2.12 The suspension shall be filtered through an 110mm hardened filter paper (Whatman No. 541) into a conical 500ml beaker.
- 2.13 When the original beaker has been drained and when filtration has stopped the filter paper and contents shall be removed carefully from the funnel and transferred back to the same beaker.
- 2.14 20ml of 10% hydrochloric acid shall be added to the beaker and the mixture stirred until all the sesquioxides have gone into solution (more acid may be required if an undue excess of ammonia was added or the sesquioxide contents is high but the acid used should be kept to a minimum).
- 2.15 The filter paper should then be removed and washed with water until all traces of yellow coloration have disappeared. These washings should be collected in the beaker and when the washing is completed the filter shall be rejected and the contents of the beaker brought to the boil.
- 2.16 Ammonia solution shall be added as before to reprecipitate the sesquioxides and the content of the beaker filtered through a Whatman 541 filter paper into the conical beaker containing the washings from the first precipitation.
- 2.17 Analysis of the acid extract
- The extract shall be tested with litmus and made slightly acid by the addition of hydrochloric acid. It shall then be brought to boiling point and 25ml of 5% barium chloride solution shall be added drop by drop with stirring of the solution. The covered solution shall be kept hot but not boiling for at least 1 hour.

- 2.18 This suspension shall be allowed to settle and a few drops of barium chloride solution shall be added to the supernatant liquid to ensure completed precipitation of the barium sulphate. If precipitation is incomplete more barium chloride solution should be added until no further sulphate is precipitated. The solution shall again be kept hot but not boiling for about 1 hour.
- 2.19 The precipitate of barium sulphate shall be transferred with extreme care to a previously ignited and weighed porous porcelain filter and ignition crucible using suction. Alternatively, the precipitate shall be transferred with extreme care to a suitable filter paper (Whatman No. 44) in the glass funnel and filtered. In either case the precipitate shall be washed several times with hot distilled water until the washings are free from chloride as indicated by an absence of turbidity when a drop is tested with a solution of silver nitrate.
- 2.20 If a porous porcelain filter and ignition crucible is used it shall be removed from the filter flask and dried at 105-110°C for approximately 30 minutes and the temperature gradually raised to 800°C in a muffle furnace until no further loss in mass occurs (15 minutes at 800°C should suffice see Note 4).
- 2.21 The crucible shall be cooled in a desiccator and weighed to the nearest 0.001g. The mass of the precipitate M4 shall be calculated from the increase in mass of the crucible.
- 2.22 If the precipitate is filtered through a filter paper the filter paper and precipitate shall be transferred to a previously ignited and weighed porcelain and silica crucible dried slowly and ignited. If an electric muffle furnace is used the crucible and contents shall be placed in it at room temperature and the temperature shall be raised gradually to red heat 800°C.
- 2.23 The crucible and contents when cool shall be moistened with a few drops of concentrated hydrochloric acid followed by a few drops of concentrated sulphuric acid brought to constant mass by ignition for 15 minutes and then cooled in a desiccator for 30 minutes (see Note 5).
- 2.24 The mass of the precipitate obtained M4 is calculated from the increase in mass of the crucible (to the nearest 0.001g).
- 3.0 Calculations
- 3.1 The percentage of sulphate (as SO<sub>3</sub>) present in the original soil sample shall be calculated from the equation:-

$$\text{Percentage of SO}_3 = \frac{34.3 \times M_2 \times M_4}{M_1 \times M_3}$$

where M<sub>1</sub> is the mass of soil before sieving, M<sub>2</sub> is the mass of sample passing a 2mm BS test sieve, M<sub>3</sub> is the mass of soil used, M<sub>4</sub> is the mass of the ignited precipitate.

- 4.0 Reporting of Results
- 4.1 The sulphate content of the soil shall be reported to the nearest 0.01% as SO<sub>3</sub> of the original oven dry soil mass (see Note 6).

5.0 Notes on Test

NOTE 1

Soils containing sulphates in the form of gypsum lose water of crystallization if heated above the specified temperature.

NOTE 2

It is assumed that any material retained on the 2mm BS test sieve will not contain sulphates. This is generally true but certain soils may contain lumps of gypsum larger than 2mm diameter and in such cases the gypsum should be removed by hand, crushed to pass the 2mm BS test sieve and incorporated in the fraction passing the sieve.

NOTE 3

The mass of sample to be used depends on the amount of sulphate present. Ideally a mass of soil should be chosen that will produce a precipitate of barium sulphate weighing approximately 0.2g.

NOTE 4

If a rapid estimation of sulphate is required and a furnace is not available the crucible may be dried in an oven at 105-110°C until no further loss in mass occurs. The result in this case will usually be less accurate than that obtained by ignition of the crucibles.

NOTE 5

Treatment with acids after ignition.

During the ignition some of the barium sulphate precipitate may be reduced to sulphide by the carbon of the filter paper. The addition of a few drops of hydrochloric acid converts any sulphide acid reprecipitates barium sulphate. Any excess acid is driven off in a subsequent ignition. The effect may be ignored for most practical purposes.

NOTE 6

In cases where the sulphate content exceeds 0.5% it is suggested that as an additional test the water soluble sulphate content should be determined by the method given in test 10.