

Geotechnical Engineering Laboratory  
CIVIL ENGINEERING VIRTUAL LABORATORY

Experiment no 7

Total Sulphate Test

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## OBJECTIVE

To determine the total soluble sulphates in soils by Precipitation Method.

## APPARATUS USED

- i) Silica Crucible-50 ml capacity
- ii) Electric stirrer with a dispersion cup
- iii) Glass Beaker
- iv) Analytical balance accurate to 0.0002 g
- v) Glass funnel
- vi) Glass bottle
- vii) Heating apparatus
- viii) Pipette-25 ml, Burette
- ix) Drying oven

## REAGENTS

Barium chloride solution (5%)-Dissolve 5 g of barium chloride in 100 ml of distilled water.

Con.HCl- Specific gravity 1.18

Phenolphthalein indicator solution

Silver nitrate indicator (0.5%)

## THEORY

In this method an aqueous extract of the soil is prepared and the sulphate content of the extract or an aliquot part of it is determined by the precipitation of sulphate as barium sulphate, filtering the precipitate and weighing it.

## PROCEDURE

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i) Take 10 g of representative soil from the sample prepared and transfer the same in a dispersion cup. Add 100 ml distilled water to it and fit the cup in the electric stirrer. Stir the solution in the dispersion cup for 30 minutes and leave the suspension undisturbed for about 24 hours.

ii) Filter the above solution and take 50 ml of the filtrate in a beaker. Add a few drops of phenolphthalein indicator and if the extract is found alkaline, pour continuously conc. HCl in it just to neutralise the solution. Add further 4 ml conc. HCl to make the solution acidic. Heat the solution to boiling.

iii) Take out the boiling source from the heat source and add hot barium chloride slowly and stir the solution continuously till there is no precipitation by further addition. Now place the beaker on a steam bath for about 4 to 5 hours so that precipitate may settle. Filter the solution using ashless filter paper in a glass funnel. Wash sample in filter paper by adding distilled water continuously till the water coming out of the glass funnel is free from chloride ions. As a check collect the washing in a test tube at different intervals and add a drop of 0.5% silver nitrate solution. The formation of white precipitates indicates the presence of chloride ions in the precipitate. Continue the white process till white precipitates are not formed in the washings by addition of silver nitrate solution.

Alternatively filtration may also be done using a pre-weighed sintered glass crucible or a Gooch crucible.

In case of filter paper, dry the filter paper containing precipitates in an oven at 60 degree C for about 2-3 hours. Now keep this filter paper in a pre-weighed silica crucible and ignite the filter paper in a muffle furnace at 700 degree C to 800 degree C for about half an hour. Cool the crucible in a desiccator, weigh and note the weight of the residue in it. The difference of weights i.e. the weight of residue is the mass of barium sulphate

## **CALCULATIONS**

Let weight of empty silica crucible =  $m_1$  g

Weight of crucible + residue ( $BaSO_4$ ) =  $m_2$  g

Weight of residue i.e. precipitates of  $BaSO_4$  =  $(m_2 - m_1)$  g

The initial extract was taken 50 ml which contains 5g of soil (say  $m_3$ )

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Hence  $BaSO_4$  in 1 g soil  $= \frac{m_2 - m_1}{m_3}$

1mole of  $BaSO_4$  contains 1 mole of  $SO_4$

Thus, ( 137.36 + 32.066+64)g of  $BaSO_4$  contains (32.006+64)g of  $SO_4$

or, 233.426 g of  $BaSO_4$  contains 96.066 g of  $SO_4$

1g of  $BaSO_4$  contains 96.066/233.426 g of sulphate

i.e.=0.4115 g of sulphate

Hence (a) sulphate (as  $SO_4$ ) % by mass=  $\frac{m_2 - m_1}{m_3} \times 0.4115 \times 100$

$$=41.15 \times \frac{m_2 - m_1}{m_3}$$

(b) Sulphate as  $Na_2SO_4$  % by mass=  $60.85 \times \frac{m_2 - m_1}{m_3}$

## PRECAUTIONS

1) Acid/Alkalis should be handled very carefully. Wash the body organ immediately if same acid or alkali falls on it.

2) Unless specified otherwise pure chemicals and distilled water should be used in tests. Pure chemical means “The chemicals that do not contain impurities, which effect the results of analysis”.

## REFERENCES

Soil Testing for Engineers.

## QUIZ

1) What are the sources of sulphate ions in soil?

2) What is the effect of sulphates on foundations?

3) What is the principle of Precipitation method or standard solution?

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