

13. DELETERIOUS CONTENTS OF SOILS.

I. Determination of Total Soluble Sulphates (IS : 2720 – Part – 27)

A. By Precipitation Method:

Object:

Determination of total soluble sulphate content of soil by precipitation method.

Apparatus:

1) Analytical Balance – sensitive to 0.001 g , 2) Glass Beaker – 250 ml capacity, 3) Glass Funnel – 50 mm diameter, 4) Glass Bottle – 250 ml capacity with a rubber cork, 5) Crucible – 50 ml capacity, 6) Pipette – 25 ml, 7) Burette – 50 ml with 0.1 ml graduation, 8) Filter paper – Whatman No.42, 9) Heating equipment, Muffle furnace, Mechanical shaker, Drying oven and A Mortar with rubber-covered pestle.

Reagents:

- 1. Phenolphthalein Indicator Solution:** Dissolve 0.1 g of phenolphthalein in 60 ml of rectified spirit and dilute with distilled water to 100 ml.
- 2. Concentrated Hydrochloric Acid:** Specific gravity 1.18 (conforming to IS:265-1976)
- 3. Barium Chloride Solution:** 5 percent. Dissolve 5 g of barium chloride in 100 ml of distilled water.
- 4. Silver Nitrate Indicator Solution:** 0.5 percent. Dissolve 500 mg of silver nitrate in 100 ml of distilled water.

Procedure:

a) Soil Sample: The soil sample shall be brought to a state in which it may be crumbled if necessary, by drying it in an oven maintained at 105 to 110⁰C. The aggregations of particles shall be broken up in mortar with rubber-covered pestle or the mechanical device. The sample shall be thoroughly mixed and then sub-divided by quartering.

b) Procedure: Take 10 g of soil from the sample prepared as above in a 250 ml bottle with 100 ml of distilled water. Give occasional shaking for 2 hours by means of the mechanical shaker. Allow the soil suspension to stand overnight. Filter and take 25 ml of filtrate in a beaker and add concentrated hydrochloric acid to just neutralize the solution if it is found alkaline to phenolphthalein indicator. Add further 4 ml concentrated hydrochloric acid to make the solution acidic. Boil the soil suspension and add barium chloride filter it. The precipitation shall be wash free from chloride ions. The filtration mass also done through a preheated with sintered glass crucible. In the case of filter paper after drying ashing shall be done on a low flame and the precipitate then ignited over a burner or in a muffle furnace at 600 to 700⁰C for half an hour cool in a desiccator weigh

and note the weight of the residue. This is the weight of barium sulphate. A corresponding weight of sodium sulphate should be calculated and thus its percentage determined.

Note: To check whether the residue is free of chloride ions, collect the washings in a separate test tube at different time intervals and a drop of 0.5 percent silver nitrate solution to it. The formation of white cloudy precipitate shows the presence of chloride ions in the precipitate. Continue washing until the white precipitation is not formed in the washings by the addition of silver nitrate solution. Hot water may be used for washing.

Calculations:

$$\text{Sulphates (as SO}_4\text{), by mass} = 41.15 \frac{W1}{W2}$$

$$\text{Sulphates (as Na}_2\text{SO}_4\text{), by mass} = 60.85 \frac{W1}{W2}$$

Where, W1 = mass in g of the precipitation and
W2 = mass in g of the soil contained in the solution taken for precipitation.

B. Volumetric Method:

Reagents:

- 1. Barium Chloride Solution:** N/4. Dissolve 30.54 g of barium chloride in one litre of distilled water.
- 2. Potassium Chromate Solution:** N/4. Dissolve 24.275 g of potassium chromate in a small amount of distilled water. Add a few drops of silver nitrate solution to it to remove any chloride, filter and dilute to 250 ml.
- 3. Silver Nitrate Indicator Solution:** 0.5 percent. Dissolve 500mg of silver nitrate in 100 ml of distilled water.
- 4. Dilute Solution of Ammonium Hydroxide:** Sp.Gr.0.888. Mix ammonium hydroxide and distilled water in the ratio of 1:2.
- 5. Concentrated Hydrochloric Acid:** Sp.Gr.1.11 (conforming to IS:265-1976).

Procedure:

Weigh 10 g of the soil specimen obtained by the method specified in above, in a beaker and add about 50 ml water. Stir well, allow to decant, filter, wash the soil on filter paper with a small quantity of water and make the filtrate to 100-ml. Pipette out 10 ml of the

water extract in a conical flask, make it slightly acidic by adding concentrated hydrochloric acid and heat to boiling. While boiling, add barium chloride solution (N/4) from the burette till the precipitation is complete and barium chloride solution is in slight excess.

Neutralize the solution with ammonium hydroxide and titrate the excess of barium chloride against potassium chromate solution (N/4). The end point may be confirmed, if considered necessary, by using silver nitrate solution as an external indicator.

Calculations:

Sulphates as sodium sulphate in soil, percent by mass = $0.0177 \times 100 (x - y)$

Where, x = volume of N/4 barium chloride added in ml.

y = volume of N/4 potassium chromate solution used in back titration in ml.

**DELETERIOUS CONTENT TEST
(TOTAL SOLUBLE SULPHATE CONTENT)
BY VOLUMETRIC METHOD (IS : 2720 - PART - 27)**

Lab Ref No : _____

Date of Sampling : _____

Type of Material : _____

Date of Testing : _____

Source : _____

1	Mass of Precipitate in gms - (W1)	
2	Mass of soil contained in the solution taken for precipitation in gms - (W2)	
3	Sulphates as (SO ₄) percent by mass = 41.15 (W1 / W2)	
4	Sulphates as (Na ₂ SO ₄) percent by mass = 60.85 (W1 / W2)	

Remarks : _____

Tested by : _____
(For Contractor)

Checked by : _____
(For Contractor)

(For Engineer)

**DELETERIOUS CONTENT TEST
(TOTAL SOLUBLE SULPHATE CONTENT)
BY PRECIPITATION METHOD (IS : 2720 - PART - 27)**

Lab Ref No : _____

Date of Sampling : _____

Type of Material : _____

Date of Testing : _____

Source : _____

Determination No.	1	2	3
Weight of specimen, gm			
Volume of N/4 Barium Chloride Added (x), ml			
Volume of N/4 Potassium Chromate Solution Used in Back Titration (y), ml.			
N/4 Barium Chloride Actually Used for Precipitating Sulphate			
Sulphate as Sodium Sulphate in Soil, % by mass = $0.0177 (100) (x - y)$			

Remarks : _____

Tested by : _____
(For Contractor)

Checked by : _____
(For Contractor)

(For Engineer)