10.7 Determination of available phosphorus

Reagents

- Aminonaphtholsulphonic acid: Place 195 ml of 15 per cent sodium bisulphate solution in a glass-stoppered cylinder. Add to it 0.5 g of 1, 2, 4- aminonaphtholsulphonic acid and 5 ml of 20 per cent sodium sulphite solution. Stopper the cylinder and shake well to dissolve the powder. If the powder is not dissolved completely add more sodium sulphite solution, 1 ml at a time, with shaking but avoid excess. Transfer the solution to a brown-glass bottle and store in the cold. The solution, if stored as described, may be used for about four weeks.
- Sodium bisulphite solution – 15 per cent: Weigh accurately 30 g of sodium bisulphate in a beaker and add 200 ml of water. Stir to dissolve, and if the solution is turbid allow to stand well-stoppered for several days and then filter. Keep the solution well stoppered.
- Sodium sulphite solution: Dissolve 20 g of anhydrous sodium sulphite in water and dilute to 100 ml, if necessary, filter the solution. Keep the solution well-stoppered.
- Calcium chloride solution: 10 per cent, saturated with calcium hydroxide at pH 8.8.
- Calcium chloride solution:20 per cent
- Hydrochloric acid: Dilute
- Molybdate I: Dissolve 25 g of reagent grade ammonium molybdate in about 200 ml of water. Place in one-litre graduated flask, 500 ml of 10 N sulphuric acid and add to it the molybdate solution. Dilute to one litre with water. Mix well. The solution keeps stable indefinitely.
- Molybdate II: Dissolve 25 g of reagent grade ammonium molybdate in about 200 ml of water. Place in one-litre graduated flask, 300 ml of 10 N sulphuric acid and add to it the molybdate solution. Dilute to one litre with water. Mix well. The solution keeps stable indefinitely.
- Phenolphthalein indicator solution: Dissolve 0.1 g of phenolphthalein in 100 ml of 95 per cent (m/v) ethyl alcohol.
- Sodium hydroxide solution - Saturated.
- Standard phosphate solution: Weigh exactly 0.351 g of pure dry mono-potassium phosphate and dissolve in water. Transfer quantitatively to a one-litre graduated flask. Add to it 10 ml of 10 N sulphuric acid and make up the volume to the mark. Shake thoroughly. This solution contains 0.4 mg of phosphorus in every 5 ml of the solution.
- Sulphuric acid – 10 N: Add carefully 450 ml of concentrated sulphuric acid to 1300 ml of water. To check, dilute 10 ml of this solution to 100 ml in graduated flask, mix and titrate a 10 ml portion of this solution with standard 1 N sodium hydroxide solution. From the titration results, adjust, if necessary, the normality of the original solution to make it exactly 10 N.
- Trichloroacetic acid – 5 per cent: Dissolve 5 g of the reagent grade trichloroacetic acid in water and dilute to 100 ml.

Apparatus

- Colorimeter

Procedure

1. Weigh accurately about 20 g of the ground material and transfer it to a 250 ml beaker. Add 100 ml of trichloroacetic acid (maintained at about 5ºC) and stir occasionally for 15 minutes. Allow it to stand for 2 hours.

2. Transfer the contents to a 250 ml graduated flask and make up the volume to the mark with trichloroacetic acid (maintain at about 5ºC). Stir the content of the flask thoroughly and allow to stand for 30 minutes. Filter about 120 ml of the supernatant liquid and transfer 100 ml of the filtrate to a 250 ml beaker. Neutralize with the sodium hydroxide solution using phenolphthalein as the indicator. Add to it 2 ml of calcium chloride solution and allow it to stand at room temperature for 10 minutes. Centrifuge the precipitate and wash with a small volume of water containing the calcium chloride solution. Filter and wash. Place the funnel containing the filter paper and the precipitate on an empty 100 ml graduated flask. Dissolve the precipitate with dilute hydrochloric acid, wash the filter paper, and then make up the volume of the filtrate to the mark.

3. Transfer 5 ml of the filtrate to a 10 ml graduated cylinder and add to it 1 ml of the molybdate II reagent. Shake thoroughly and add 0.4 ml of the aminonaphtholsulphonic acid reagent and mix again. Make up the volume to the 10 ml mark with water, mix, and allow the contents to stand for 5 minutes.

4. For taking the colorimetric measurement, compare in the colorimeter against a standard prepared at the same time as given below:

5. Transfer 5 ml of the standard phosphate solution containing 0.4 mg phosphate solution containing 0.4 mg phosphorus, to a 100 ml graduated flask and add 50 ml of water. Add 10 ml of molybdate I reagent. Mix thoroughly and add 4 ml of aminonaphtholsulphonic acid reagent. Dilute with water to the 100 ml mark, mix well and allow the contents of the flask to stand for 5 minutes. Compare the standard against itself in the colorimeter before taking a reading of the unknown solution. If the colour of the unknown is particularly strong, repeat the reading the unknown a few minutes later, to make sure that the maximum colour development has taken place.

6. Calculate the percentage of the available phosphorus in the material from the reading of the colorimeter.

Reference: IS 1374: 1992.