

Spectrophotometric Determination of Phosphate in Soil and Detergents

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ARTICLE DETAILS	ABSTRACT
Research Paper	A simple and sensitive spectrophotometric method has been developed for the determination of phosphate in mg/lit. concentration range in soil and detergents samples. In this method a blue coloured complex is formed when samples is treated with ammonium molybdate in the presence of acid and excess of ascorbate ions. The system obeys Lambert Beer's law at 855nm. in the range of 0.4 to 0.5 ppm. Various parameters which affect the colour development and stability of complex were studied in soil and detergent samples.
Keywords : <i>Soil, Detergent, Spectrophotometer, Ammonium molybdate, ascorbic acid</i>	

Introduction

Phosphorous is the most abundant element of the earth surface and it is commonly found as phosphate. It is a key factors of eutrophication process¹. Due to the presence of calcium, iron, Aluminium and other organic matter animal & human waste decaying matter phosphate is usually insoluble. The chemical nature of soil is determined by the combination of minerals & organic matter that makes up the soil². Various methods have been reported for the determination of phosphate such as titrimetry³, complexometry⁴, colorimetry⁵, AAS⁶, Flow injection analysis⁷, HPLC⁸ and spectrophotometric method⁹⁻¹². Some of these methods are expensive, time taking and complicated. In the present method ascorbic acid has been used as reducing agent for the reduction of the molybdenum blue in acidic medium.

Experimental

Instruments:- UV- visible spectrophotometer, pH meter, Glass electrode, Mechanical shaker, centrifuge, water bath.

Preparation of soil sample solution¹³⁻¹⁶

1. Soil samples were collected from different areas of Chhattisgarh.
2. Samples were collected from different places by using auger at different depths (from 0-100cm.).The auger was screwed to the desired depth and the sample was withdrawn.
3. Soil samples were transferred to plastic bags and were labeled.
4. In the lab, the samples were air-dried; grass and any external objects were removed.
5. Sieving was done by mechanical sieving apparatus which consist of different sizes of meshes (10.0>0.075mm) after rolling the samples to break down the large masses of the soil particles.
6. The sieved samples (>2.0mm) were preserved in labeled bags.

Extraction of the samples

1. 50gm. of each of the soil samples were weighed transferred to 250ml stopper conical flask and was shaken with exactly 50ml of distilled water(1:1ratio).
2. After shaking , the equilibration was taken place by leaving the samples for 30minutes.
3. The samples were filtered into Buchner funnels by using filter papers Whatman no. 42.
4. In the case of turbid filtrates, they were centrifuged by using 3000 cycle/min. centrifuge for 5min.

Preparation of Detergent sample¹⁷

5gm. of washing powder was dried in an oven at about 350⁰C.ash was taken in a 150ml beaker. It was dissolved in about 50ml water and the solution was made acidic by adding dilute sulfuric acid. It was heated on water bath for about 10 minutes to expel the H₂S, nitrites, etc. It was filtered and the filtrate was transferred into a 100ml volumetric flask and diluted to the mark with water, later it was used for its phosphate analysis.

All chemicals were of analytical grade.

Analysis of phosphate



1. Extraction solution of phosphate:- 42gm. of NaHCO_3 was dissolved and was adjusted to pH of 8.5 with 50% NaOH and CH_3COOH .
2. Standard ammonium molybdate :- 12gm. of ammonium molybdate was dissolved in 250ml of distilled water.
3. Both of the above reagent (1&2) were added into 1000ml of 5N H_2SO_4 , mixed thoroughly, made to 2000ml with distilled water and were stored in a pyrex glass bottle in a dark compartment. This was labeled as reagent 'A'.
4. Ascorbic acid :- 1.056gm. of ascorbic acid was dissolved in 200ml of reagent 'A'. This was labeled as 'B'.
5. Stock phosphate solution (1000mg/L):- 4.3937gm. of oven dried KH_2PO_4 salt was dissolved in distilled water, 1ml of concentrated H_2SO_4 was added and the solution was diluted to 1L in a volumetric flask.
6. Intermediate phosphate solution (250mg/L):- It was prepared by diluting 25ml of the stock solution upto 100ml using redistilled water.
7. Standard phosphate solution (2.5mg/L) :- It was prepared by diluting 10ml of the intermediate solution upto 1L with extracting solution.

Procedure:- Take a 5ml of soil extract in a 25ml volumetric flask add 10ml distilled water and 4ml of reagent 'B', made upto the mark with distilled water. The colour was allowed to develop for 15min. and phosphate content was determined using a spectrometer at wavelength 855nm.

Result and Discussion:-

The developed method is based on the formation of phosphomolybdate complex due to the reaction between molybdate and phosphate followed by its reduction with ascorbic acid in aqueous acidic medium. The complex exhibits maximum absorbance at 855nm.

Various experimental parameters which affect the formation and stability of complex were carefully investigated and optimized for quantitative determination of phosphate in soil and detergents. In this experiment, the effect of concentration of ammonium molybdate in formation of phosphomolybdenum blue complex was investigated by varying the volume (0.1 to 8.0ml) of ammonium molybdate with addition of 0.5ml of H_2SO_4 , 2ml of phosphate solution and 1ml ascorbic acid in a 25ml volumetric flask and then made upto the mark with distilled water. After the time for maximum color development

absorbance of the solution were measured at 855nm. 0.20% ammonium molybdate is suitable for experiment for the determination of phosphate. For the maximum absorbance 0.1 to 2ml of 0.5ml of 0.5M H₂SO₄ is used. 0.5ml of 0.5M H₂SO₄ is suitable for throughout the experiment. The 1ml of 0.5ml ascorbic acid was sufficient for whole experiment.

At 855nm. wavelength the system obey's Lambert-Beer law in the concentration range of 0.5 -5ug/ml of phosphate and molar absorptivity was determined to be $3.2 \times 10^4 \text{ Lmol}^{-1}\text{cm}^{-1}$. Result obtained by using this methodology and instrumentation for the determination of phosphate in soil and detergent samples are reported in table.

Table:- Determination of phosphate in soil & detergent samples

Samples	Phosphate in mg/l in soil	Phosphate in mg/l in detergents
S ₁	0.91	63.5
S ₂	1.07	15.5
S ₃	1.12	19.2
S ₄	1.15	32.4
S ₅	1.50	54.3

Conclusion

The present method is very cheap and simple. It neither involve more reagents nor require more sophisticated instruments like HPLC. This method has been successfully applied for the determination of phosphate in various samples.

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