# Spectrophotometric Determination of Phosphate

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## Introduction

The determination of phosphate has a vast literature extending into different areas. Gravimetric methods were based on the precipitation of phosphate as ammonium phosphomolybdate[1], ammonium magnesium phosphate[2] and silver phosphate[3]. Organic bases[4] have also been used for the gravimetric determination of phosphate. Alternatively the precipitate could be dissolved and determined volumetrically[5]. Recently, there are many spectrophotometric methods used for the determination of phosphate. Earlier work is based on the measurement of the absorbance of molybdophosphoric acid or its reduction products[6]. Abbott. et al[7] used the vanadomolybdophosphate for the colorimetric determination of phosphate. The colour formed was measured at 370 nm. Ramirez-Munos[8] has automated two molybdenum blue procedures for the determination of phosphate, in which stannous salts and ascorbic acid were used as reductants. This paper describes a spectrophotometric method based on the precipitation of manganese ammonium phosphate and oxidation of the precipitate to permanganate which can be determined colorimetrically.

## Experimental

A.D.M.R. II, recording spectrophotometer, Zeiss, was used throughout this work. Diammonium hydrogen phosphate, manganese sulphate, ammonium chloride, ammonia, hydrochloric acid, nitric acid, phosphoric acid and potassium periodate were all supplied by BDH, except diammonium hydrogen phosphate which was supplied by Hopkin and Williams.

## General Procedure

0.2 g of $MnSO_4$ is dissolved in 100 ml of distilled water, placed in a 200 ml beaker and made slightly acidic with nitric acid, 5 g of $NH_4Cl$ is added followed by 3 ml of the sample containing about $7 \times 10^{-3}$ M $(NH_4)_2HPO_4$. The mixture is heated to 95°C with adding drops of ammonia until a precipitate appears. After complete precipitation the mixture was left for two hours at 0°C then filtered using porcelain filtering crucible and washed with 3 % ammonium nitrate solution. The precipitate is dissolved in 25 ml of 1 : 3 nitric acid and transferred quantitatively into 150 ml conical flask. 10 ml of phosphoric acid were added followed by 0.7g potassium periodate. The mixture was boiled for 5 minutes and completed to 100 ml with distilled water and the absorbance was measured at 545 nm.

## Results and Discussion

The precipitation of ammonium manganese phosphate has been utilised for the gravimetric determination of manganese. The method was adapted to the spectrophotometric determination of small amounts of phosphate by oxidising this precipitate with potassium periodate in acidic solution which yields the well-known permanganate colour which can be measured colorimetrically at 545 nm. In the beginning, it was very difficult to precipitate and transfer such small amounts of the ammonium manganese phosphate. Moreover, the precipitate was found to dissolve in the excess of ammonia, therefore, careful precipitation is necessary to avoid the loss of precipitate.

Calibration graph was prepared by direct oxidation of ammonium manganese phosphate and found to be linear from 0.05 – 2 x $10^{-5}$ M $MnNH_4PO_4$ (Fig. 1). The method was applied to the determination of phosphate in two authentic samples, the results obtained are shown in Table (1).

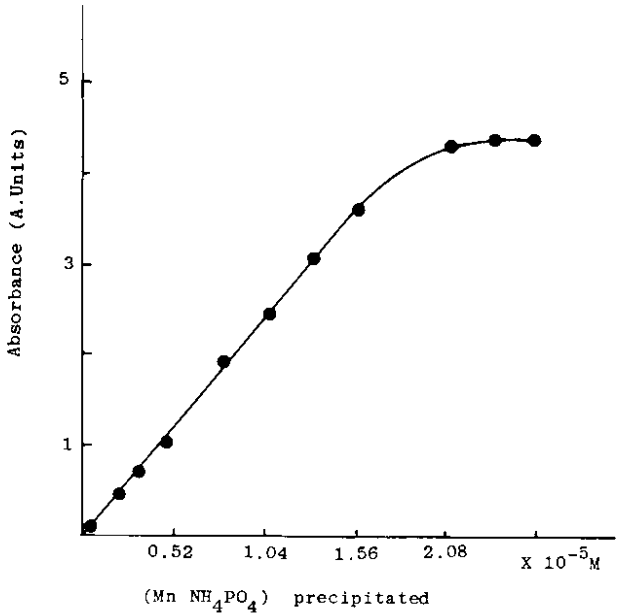


Fig (1). Calibration graph for phosphate

Table (1)

| Calculated $(NH_4)_2HPO_4$ | Found $(NH_4)_2HPO_4$ |
|---|---|
| 2.23 x $10^{-5}$ M | 2.24 x $10^{-5}$ M |
| 2.23 x $10^{-5}$ M | 2.22 x $10^{-5}$ M |

The method was also applied to the determination of phosphate in urine 0.5 ml of the sample were taken and diluted to 100 ml with distilled water and treated as before. Results obtained are shown in Table (2).

The effect of interfering ions such as arsenate, silicate, molybdate on the absorbance of 3.79 x $10^{-4}$ M

Table 2

| Sample | Calculated $PO_4^{-3}$ from the graph | Actual $PO_4^{-3}$ | Average |
|---|---|---|---|
| 1. | 0.254 x $10^{-5}$M | 5.08 x $10^{-5}$M | 5.18 x $10^{-5}$M |
| 2. | 0.264 x $10^{-5}$M | 5.28 x $10^{-5}$M | |

$MnNH_4PO_4 . H_20$ was studied and found to give positive interference because they all precipitate manganese. Magnesium also interferes by precipitating phosphate and should be separated.

The method is in general very simple and reasonably sensitive. The use of cheap reagents is also another advantage.

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