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Study on New Sensitive Method of Determination of Phosphorus by Solid Phase Spectrophotometry

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Abstract: The use of solid phase spectrophotometry for the determination of trace phosphorus in the system of phosphomolybdate-fructose is described. The adsorption of the system on anion-exchange resin is reported.

Keywords: Phosphorus, solid phase spectrophotometry, anion-exchanger resin, adsorption.

Introduction

The spectrophotometric determination of phosphorus with colour system composed of phosphomolybdate-fructose in aqueous phase has been reported[1,2]. It was found that this colour system was easily adsorbed on the anion-exchange resin (20-40mesh,Cl^- form), hence, a new method of determination of trace phosphorus was established by solid phase spectrophotometry. In solution of sulfuric acid, phosphorus reacts with ammonium molybdate to form phosphomolybdate yellow, which is reduced into phosphomolybdate blue by fructose, the phosphomolybdate blue can be quantitatively adsorbed by Cl- exchange resin. The absorbance of the resin-phase after adsorption is then measured. The apparent molar absorptivity was found to 1.6×10^5 L mol^{-1} cm^{-1} at 810 nm, the sensitivity is 80 times higher than the method of phosphomolybdate yellow and 10 times higher than the method of phosphomolybdate blue. Beer's law is obeyed for 0-12 μg of phosphorus. The advantages of the method are high sensitivity, selective adsorption, ease of operation, good reproducibility, and simplicity of apparatus.

Experimental

Apparatus and reagents

Absorbances and absorption spectra were measured on a Model 751-GW spectrophotometer with 0.5 cm cell.

Standard phosphorus stock solution (50 μ g/mL): Prepared by dissolving 0.2195 g KH_2PO_4 in 25 mL sulfuric acid (1∶5) and diluting to 1L with water.

Standard phosphorus solution (1 μ g/mL): Dilute 10 ml of the standard phosphorus stock solution to volume in a 500 mL standard flask with water.

Anion-exchange resin: The resin (20-40mesh, Cl^- form) used was no.717, produced by the Shanghai Resin Factory; it is a strong-base resin, equivalent to Dowex 1 or Deacidite FF. Before new resin was used, it was soaked in solution of 4 mol/L hydrochloric acid for 24hrs and then washed repeatedly with water. All small particles and inclusions should be removed and the resin was dried with filter paper and stored in a polyethylene container.

Sulfuric acid solution (2.5mol/L); 4% ammonium molybdate solution;4% fructose solution.

Analytical-grade chemicals and demineralized water were used.

General procedure

Transfer a solution containing not more than 15 μ g of phosphorus into a 25 mL calibrated flask. Add 5.0 mL sulfuric acid, 1.0mL ammonium molybdate solution and 2.0 mL fructose solution, mix well. Place the flask in a boiling-water bath for 8 min, cool, transfer the solution into a 50 mL beaker with 0.70 g anion-exchange resin, shake the beaker mechanically for 10 min. Withdraw the resin with a dropper and put it into a 0.5-cm glass cell, ensuring that it is evenly packed. Measure the absorbance at 810 nm against a resin blank.

Results and discussion

Absorption spectrum of the resin.

Anion-exchange resin was found to adsorb only the phosphomolybdate blue. A single-beam spectrophotometer was suitable for the present work. The absorption spectrum of the phosphomolybdate blue resin was the same as that of the aqueous solution, exhibiting maximum absorption at 810 nm, so the absorbance of resin was measured at 810 nm.

Optimization of conditions for phosphomolybdate blue formation.

The suitable acidity for producing phosphomolybdate blue was in the range of 0.7-1.1 mol/L and 1.0 mol/L of acidity was selected.

In 25 mL of solution, 0.5-2.0 mL of 4% ammonium molybdate gave maximum and constant absorbance, thus 1.0 mL ammonium molybdate solution was used.

.In 25 mL of solution,1~4 mL of 4% fructose solution gave maximum and constant absorbance, thus 2.0 mL fructose solution was selected.

Optimization of conditions of absorption of phosphomolybdate blue on the anion – exchange resin.

The effect of stirring time on adsorption of phosphomolybdate blue on the anion-exchange resin was investigated. The adsorption was almost complete within 4 min, 6 min being required for complete adsorption. A stirring time of 6 min was chosen to keep the analysis time reasonably short.

The absorbance was dependent of the amount of resin used. With the increase of amount of resin, the absorbance was decreased. The amount of 0.70 g resin was recommended (considering use of 0.5 cm cell).

Constant absorbance was obtained at 20-30^{0}C. Below 20^{0}C the absorbance decreased, because of the lower adsorption of the sample species on the resin. Above 30^{0}C the adsorption is faster and may be complete within 6 min.

Effect of foreign ions.

An amount of 2.0 μ g of phosphorus was examined. When a change of 5% in absorbance was set as the tolerance limit, the following ions did not interfere (amount given in μ g): CO_3^{2-}, NO_3^- (8000); SO_4^{2-} (10000); SiO_3^{2-} (150); Ca^{2+}, Cu^{2+}, Al^{3+}, Fe^{3+}, Bi^{3+}, Mg^{2+} (500); Ag^+, Na^+, K^+, Mn^{2+}, Cr^{3+}, $Cr_2O_7^{2-}$, Pb^{2+}, ClO_3^-, PO_4^{3-}, V (V), and Ce (IV) (800). Most ions did not interfere with the determination, but AsO_4^{3-} (2) and AsO_3^{3-} (0.5) interfered.

Calibration graph, sensitivity and precision.

A calibration graph was constructed in the usual way according to the general procedure. Beer's law was obeyed for 0-12 μ g of phosphorus in 25 mL solution. The molar absorptivity was calculated from the slope of the calibration graph to be 1.6×10^5 L mol^{-1} cm^{-1}. Eight replicate analyses of a test solution containing 2.0 μ g of phosphorus gave a relative standard deviation of 3.6%. The recoveries of phosphorus averaged 98.2%, with ranges of 97.1-102.4%.

References

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