

NOTE

Photometric Determination of vanadium(V) after Adsorption of its 1-Hydroxy-1-(*o*-Chlorophenyl)-3-Phenyl-2-Thiourea Complex of Microcrystalline Naphthalene

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A water insoluble stable complex of vanadium(V) with 1-hydroxy-1-(*o*-chlorophenyl)-3-phenyl-2-thiourea is separated from aqueous phase by its adsorption on microcrystalline naphthalene. The adsorbed complex is dissolved in dimethylformamide. The absorbance of the solution is measured at 395 nm. A linear relationship exists between absorbance and concentration of vanadium(V) in the concentration range of 4-110 µg of vanadium in 10 mL of DMF. The molar absorptivity was found to be $3.46 \times 10^4 \text{ l mol}^{-1} \text{ cm}^{-1}$ at 395 nm and sensitivity being $1.13 \times 10^{-2} \text{ µg cm}^{-2}$ of vanadium for the absorbance of 0.001.

Numerous photometric reagents¹⁻⁴ have been reported for the photometric determination of vanadium(V). Thiourea and its derivatives⁵⁻¹² have been employed for the photometric determination of different metal ions. The substituted hydroxy thioureas indicate their reactivities towards most of the metal ions due to the presence of >C=S group. The considerable difference in colour in these compounds is attributed to the presence of alkyl or aryl group.

1-hydroxy-1-(*o*-chlorophenyl)-3-phenyl-2-thiourea has been prepared¹³ by reacting equimolar quantities of N-(*o*-chlorophenyl) hydroxylamine and phenylisothiocyanate in petroleum ether (b.p. 60-80°C). A 0.1% solution of reagent was prepared by dissolving 0.1 g of it in 100 mL of ethanol.

A stock solution of vanadium(V) (1000 ppm) was prepared by dissolving requisite amount of ammonium vanadate in double distilled water. Standard solution of vanadium(V) (10 ppm) was made by diluting 10 mL of stock solution to 1000 mL in a volumetric flask.

Buffer solutions of pH range 3-6 were prepared by mixing 1 M CH₃COOH and 1M CH₃COONH₄ solution and for pH range 8-11. 1M aqueous NH₃ and 1 M CH₃COONH₄ solution were mixed.

A 20% naphthalene solution was prepared by dissolving 20 g of naphthalene in 100 mL of acetone.

All reagents used were of spectrograde quality.

Procedure

Small aliquots of standard vanadium(V) containing 4–110 µg of vanadium(V) was taken in a flask and diluted to 50 mL pH 4.5 was maintained by buffer solution and 4 mL of 0.1% reagent solution was mixed in it. After heating at 50–55°C in a water bath, 2 mL of 20% naphthalene solution was added. On vigorous shaking, the vanadium(V) complex of 1-hydroxy-1-(*o*-chlorophenyl)-3-phenyl-2-thiourea was adsorbed on microcrystalline naphthalene. It was then separated from aqueous phase by filtration, washed, dried and was dissolved in 10 mL of DMF. The absorbance of this solution was made with systronics spectrophotometer model 106.

The vanadium(V) complex formed by the recommended procedure was adsorbed on microcrystalline naphthalene and the dry, solid mixture of naphthalene and complex was dissolved in dimethyl formamide. The absorbance of the solution so obtained was measured from 350–590 nm wavelength. It was found that the vanadium(V) complex has maximum absorbance at 395 nm wavelength. Therefore, all consequent absorbance measurements were carried out at 395 nm wavelength. The effect of different factors *viz.* reagent concentration, naphthalene concentration, pH was studied and found that 4.0 mL of 0.1% reagent solution, 2.0 mL of 20% naphthalene solution, buffer solution of pH 4.5, 210 seconds shaking time, 25 minutes standing time, 20 minutes digestion time were found to be suitable for all measurements.

The precision of proposed method was determined by taking the absorbance measurements of ten sample solutions of vanadium(V) complex obtained by recommended procedure from a series of vanadium solution containing 65 µg of vanadium(V). The standard deviation was calculated as 0.065%.

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