

Photometric determination of vanadium(V) after adsorption of its schiff's base (derived from 2-aminopyridine and naphthalene-1-carbaldehyde) complex on polyurethane foam

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Received 7 May 1996 revised 10 October 1996

The absorbance of vanadium(V) complex of schiff's base derived from 2-aminopyridine and naphthalene-1-carbaldehyde after preliminary adsorption of polyurethane foam has been studied. It is found that Beer's law is obeyed over the concentration range 10-85 μg of vanadium(V) in 10 ml chloroform. Molar absorptivity and sensitivity are calculated to be $6.6 \times 10^4 \text{ l mol}^{-1} \text{ cm}^{-1}$ and $0.061 \mu\text{g cm}^{-2}$ of V(V) respectively at 495 nm. The effects of pH, reagent concentration, absorbent, diverse metal ions on photometric determination of vanadium have been studied. The method has been applied satisfactorily for determination of vanadium in OHNS steel alloy sample.

Chelating behaviour of schiff bases has been extensively investigated¹⁻⁴. Not only these are employed as complexing ligands for a number of metals but these have been used as reagents for their estimation. There have been many efforts for identifying reagents for the determination of vanadium and a few reagents have been proposed for the photometric determination of vanadium(V)⁵⁻⁸. The present note reports the use of schiff base synthesised from 2-aminopyridine and naphthalene-1-carbaldehyde (SB₁) in the photometric determination of vanadium(V). The newly synthesised reagent has been found to be selective and sensitive. Since long, a number of pre-concentration techniques have been employed^{9,10}, but the use of polyurethane foam as an adsorbent has gained significance due to its being inexpensive, sensitive, selective and reproducible. The proposed method is more sensitive as compared to the liquid-liquid extraction technique (sensitivity $0.018 \mu\text{g cm}^{-2}$). It is evident that the proposed method (sensitivity $0.0161 \mu\text{g cm}^{-2}$) is more sensitive in comparison to the liquid-liquid extraction technique for spectrophotometric determination

of vanadium(V). Conditions have been optimised for the determination of vanadium(V) in an alloy.

Experimental

A standard stock solution of vanadium(V) was prepared by dissolving requisite amount of ammonium metavanadate in doubly distilled water and the volume was made to one litre. A 0.2% solution of schiff base derived from 2-aminopyridine and naphthalene-1-carbaldehyde was prepared by dissolving 0.2 g of the reagent in 100 ml ethanol. Polyurethane foam pieces were prepared by the method of Hamon *et al.*¹¹ The foam was cut into pieces of about 1 cm³ size and thoroughly cleaned before use. All foam pieces were soaked in 1.0 M HCl for about 10-12 h to remove possible inorganic contaminants. These pieces were then extensively rinsed with distilled water. Finally, they were squeezed and dried in air at room temperature. The dried pieces were ready for use. A GS 5701 EC spectrophotometer and Systronics digital pH meter 335 were used for absorbance and pH measurements respectively.

General procedure

An aliquot of standard vanadium(V) solution (2 ml) was taken in a flask. To this, 0.2% reagent solution (2.5 ml) was added and the pH adjusted to 4.5 using acetate buffer solution. The volume was made to 10 ml using distilled water. The resultant mixture was shaken and kept for a few minutes to ensure complete colour formation. Four prepared foam pieces were then added to the coloured complex and the flask was stoppered and shaken vigorously for 200 s to allow complete adsorption of the metal complex on foam pieces. The foam pieces were squeezed and transferred to another beaker. Excess of reagent was further squeezed out and the complex eluted from the foam pieces by using two portions of 5.0 ml chloroform. Traces of water were removed by using anhydrous sodium sulphate. Finally, absorbance of the solution was measured at 495 nm against reagent blank.

Results and discussion

The absorption spectrum of the sample solution containing 50 μg of vanadium(V) and 0.2% reagent solution (2.5 ml) at pH 4.5 was investigated

in the wavelength region 380-620 nm against reagent blank. Maximum absorbance was observed at 495 nm, whereas reagent blank showed negligible absorbance at this wavelength. Hence, all absorbance measurements were carried out at 495 nm.

The absorbance studies were found to be pH dependent. The absorbance increased up to pH 3.0, remained maximum and almost constant in the pH range 3.0-6.0, beyond which it decreased. Hence, 4.5 was taken as a standard pH for all absorbance measurements. The effect of reagent concentration was studied by varying the amount of reagent and maintaining the other conditions constant. Maximum and almost constant absorbance values were observed with 1.5-5.0 ml of 0.2% reagent solution. So, 2.5 ml of 0.2% reagent solution was considered to be most suitable for absorbance studies. The absorbance was maximum with 3-7 pieces of polyurethane foam. So, four foam pieces were found to be appropriate for extraction studies. The optimum shaking time for complete adsorption of metal complex on foam was found to be 200 s, since the absorbance remained unchanged during the shaking period of 180-240 s.

Calibration curve for vanadium(V) was plotted by taking absorbance at λ_{\max} of a series of sample solution having different concentrations, each was taken at 495 nm. The curve was found to be linear, thereby obeying Beer's law in the concentration range 10-85 μg of vanadium(V). The molar absorptivity and sensitivity were found to be $6.16 \times 10^4 \text{ l mol}^{-1} \text{ cm}^{-1}$ and $0.0161 \mu\text{g cm}^{-2}$ respectively at 495 nm. The standard deviation was calculated to be 0.83%.

Effect of diverse metal ions

Possible interference due to the presence of diverse metal ions were observed using a solution of vanadium(V) complex of schiff base (SB₁) con-

taining 50 μg of vanadium(V). Tolerance limits (in μg) are given in parentheses. Co(II)(100), Sn(II)(100), Fe(III)(100), Ti(IV)(100), Pt(IV)(100), U(VI)(100), Mn(II)(150), Cu(II)(200), Pd(II)(200), Mg(II)(250), Cr(III)(250).

Determination of vanadium(V) in an alloy

The method has been applied for the determination of vanadium(V) in an alloy. An alloy sample weighing 0.4 g was dissolved in 10-15 ml of aqua regia. The volume of this was made to 4 ml by evaporation. This solution was transferred to a 250 ml flask and the volume was made up to the mark using distilled water. An aliquot of this solution was boiled for a few minutes and then cooled. The required pH value was finally adjusted. Vanadium(V) was determined in OHNS steel alloy. The certified composition for the alloy is Si: 0.25, Cr: 0.30, V: 0.15, C: 0.85, M: 1.90, Fe: 96.55. For a sample containing 50 μg of vanadium(V) the amount of vanadium(V) found was 49.76 μg (average of five determinations). The percentage error in the above determination was -0.48%.

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