

NOTES

**Direct Spectrophotometric Determination of Vanadium with
3-hydroxy-3-phenyl-1-*p*-carboxyphenyltriazene**

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3-Hydroxy-3-phenyl-1-*p*-carboxyphenyltriazene was used for spectrophotometric determination of vanadium. The composition of the yellowish green complex is 1 : 1 (V : R) with maximum absorbance at 395 nm and working wavelength was chosen to be 420 nm using solvent as blank. Molar absorptivity and Sandell's sensitivity were found to be 9550 l/mole cm and 5.33 ng/cm² respectively in the pH range 6.5 to 7.0. Beer's law was obeyed in the range 0.50 to 3.05 ppm of vanadium. Standard deviation 'σ' (10 determinations) using 2.54 ppm of vanadium was 0.002. Interference of 22 diverse ions was studied.

The reagent 3-hydroxy-3-phenyl-1-*p*-carboxyphenyltriazene was prepared by Sogani and Bhattacharya¹ and used in spectrophotometric determination of zinc by Purohit and Humad². Purohit and Chauhan³ used it as a metallochromic indicator for complexometric determination of lanthanoids. The present communication deals with direct spectrophotometric determination of vanadium(V) with this reagent.

The reagent was prepared¹ as per the reported method and its 1 × 10⁻² M solution was prepared in ethanol. Solution of 1 × 10⁻² M solution of vanadium(V) was prepared by dissolving the requisite quantity of sodium metavanadate (A.R.) in water and was standardised with ferrous ammonium sulphate using diphenylamine as indicator⁴. Weaker solutions were prepared by appropriate dilutions. Solutions (1% v/v) of tris-buffer and (1% v/v) of perchloric acid were used to adjust the desired pH. Systronic UV-visible spectrophotometer-108 was used for absorbance measurements and systronic pH meter 324 was used for pH measurements.

The reagent forms an ethanol-soluble yellowish green complex with vanadium(V) in the pH range of 6.5 to 7.5. The λ_{max} of the complex was at 395 nm and working wavelength was chosen as 420 nm. The development of the colour is instantaneous and reaches maximum when reagent is in sixfold excess. All the solutions were made up to 10 ml and absorbance measurements were made against solvent blank. Composition of the complex was found to be 1 : 1 (V : R) by Job's method⁵, slope ratio⁶ method, Yoe and Jone's⁷ and Zolotov's mole ratio methods. Validity of Beer's law is observed in the range 0.50 to 3.05 ppm of vanadium(V).

Molar absorptivity and Sandell's sensitivity were found to be 9550 l/mole cm and 5.33 ng/cm² respectively. Standard deviation 'σ' using 2.54 ppm of vanadium was 0.002 (10 determinations). Conditional stability constant of the complex was found spectrophotometrically by Harvey and Manning's method⁹ using mole ratio curve as $\log \beta = 4.665$ and free energy of formation of the complex as $\Delta G = -6.40$ kcal/mole.

It was found that in determination of 2.54 ppm of vanadium(V), Na(I), K(I), NH₄⁺, Hg(II), Mg(II), Ba(II), Mn(II), F⁻, Br⁻, I⁻, CH₃COO⁻, NO₃⁻, CO₃²⁻, SO₄²⁻, Molybdate and oxalate did not interfere when present in 5 ppm level. However, Zn(II), Co(II), Ni(II), Cu(II), Cd(II) interfered when present in 5 ppm.

The solid complex was obtained by mixing in molar proportion the aqueous solution of vanadyl sulphate (AR) and ethanolic solution of the reagent. It was filtered and washed with 30% water-alcohol (cold) mixture to give light green platelet crystals (260°C). The elemental analysis of the complex, molecular formula VO(C₁₃H₁₀N₃O₃)₂ for carbon, hydrogen and nitrogen was found a:s

Theoretical			Experimental		
% C	% H	% N	% C	% H	% N
53.89	3.47	14.50	46.47	3.74	13.25

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(Received: 8 April 1993; Accepted: 15 May 1993)

AJC-627