

Extraction & Spectrophotometric Determination of Vanadium(V) with Salicylaldehyde Thiosemicarbazone

N SUBBA RAMI REDDY & D VENKATA REDDY*

Department of Chemistry, Sri Krishnadevaraya University,
Anantapur 515 003

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Vanadium(V) forms a 1:1 (metal:ligand) yellow coloured complex with salicylaldehyde thiosemicarbazone (SAT) in *n*-butanol. The yellow coloured complex is quantitatively extracted from acetic acid medium by *n*-butanol. Beer's law is obeyed in the range 0.5-6.5 ppm of metal. Large number of foreign ions do not interfere.

Most of the methods used for the extractive spectrophotometric determination of vanadium(V) suffer from deficiencies such as narrow ranges of determination, lengthy procedures, and interference by common ions¹⁻³. The method described herein overcomes some of the difficulties, is simple, selective and highly sensitive.

Solution (1×10^{-2} M) of salicylaldehyde thiosemicarbazone (SAT) was prepared in *n*-butanol. A stock solution of vanadium(V) was prepared from ammonium metavanadate in doubly distilled water and standardized against a standard solution of iron(II). The stock solution thus prepared was diluted to give a solution containing 10 µg/ml of vanadium.

All the other chemicals used were of BDH (AR) grade.

Recommended procedure – The extractions were carried out by shaking 15 ml of the aqueous solution containing 1 ml of acetic acid (1 M) and the metal ion solution (5-65 µg) with a mixture of 7.5 ml of *n*-butanol and 2.5 ml of butanolic solution of SAT for about 2 min. The organic layer was separated, dried over anhydrous sodium sulphate and the absorbance measured at 375 nm against the reagent blank, employing an ECIL GS 865 spectrophotometer fitted with 1 cm matched cell.

The yellow vanadium-SAT complex extracted into *n*-butanol showed maximum absorbance at 375 nm. The reagent however showed least absorbance at this wavelength. Fifteen fold molar excess of the reagent was required for complete extraction of vanadium(V). However even 50-fold molar excess of the reagent had no adverse effect. The colour was stable for 48 hr and an equilibration period of 2 min was sufficient for complete extraction. Order of addition of reagent had no effect on λ_{max} or the absorbance of the extract.

Maximum absorbance was observed in the acetic acid concentration range of 0.06-0.08 M. However, an acid concentration of 0.07 M was chosen for the present study.

The system obeyed Beer's law in the range 0.5-6.5 ppm of vanadium. The optimum concentration range for the determination of vanadium was evaluated from Ringbom plot and found to be 1.5-5.00 ppm. The Sandell's sensitivity was 11.84 ng V cm⁻². The molar absorptivity of the complex at 375 nm was 4300 litre mol⁻¹ cm⁻¹. The sensitivity of the proposed method is more than that of some of the methods reported⁴⁻⁶.

Among various solvents tried (benzene, toluene, dichloroethane, nitrobenzene, chloroform, carbon tetrachloride, methyl isobutyl ketone, amyl alcohol, cyclohexanol, *n*-butanol) only *n*-butanol was found suitable. This is probably due to the fact that the reagent is fairly soluble in this solvent.

The effect of some of the ions which are often found in association with vanadium in soils and plant materials was studied. For these studies different amounts of ionic species were added to 50 µg of vanadium in 15 ml solution and V(V) estimated by the recommended procedure. 20 mg each of phosphate, nitrate, sulphate, perchlorate, alkali and alkaline earth metals do not interfere in the estimation while U(VI), Mn(II), Zn(II), Fe(III), Al(III) in large amounts, Se(IV) (4 mg), Mo(VI) (0.6 mg), Cr(III) (0.4 mg), Cu(II) (0.1 mg) (in the presence of thiosulphate); and Co(II), Ni(II), W(VI), Ti(IV), Zr(IV) (50 µg each) are tolerated. Chloride (4 mg), citrate, tartrate, oxalate (0.4 mg) are also tolerated.

The precision of the method was determined by taking 10 measurements each containing 5 ppm of metal. The relative standard deviation was found to be 1.53%.

The composition of the complex was determined by Job's method of continuous variation⁷ and the mole ratio method⁸ and was found to be 1:1 (metal: ligand). The stability constant of the V-SAT complex was determined to be 1.8×10^5 .

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