

Anthranilic Acid as a Spectrophotometric Reagent for the Determination of Vanadium in Presence of 0-Phenanthroline Dilip Kumar

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Article Info	Introduction : A review of the photometric methods reveals that the
Volume 4 Issue 4	complexation reaction between vanadium (IV) and anthranilic acid in presence of
Page Number : 99-101	o-phenanthroline has not been exploited analytically. The complex of vanadiuin
	(iv) can be extracted into different solvents. Among the solvents tested, isoamyl
Publication Issue :	alcohol was found to be suitable extractant which extracts the complex
July-August-2021	conveniently from slightly acidic solutions. An analytical method is proposed for
	the spectrophotometric determination of vanadium after extracting
Article History	$vanadium (\mathrm{IV})\text{-}anthranilic\ acid-o-phenanthroline\ complex\ in\ isoamyl\ alcohol.\ The$
Accepted : 01 July 2021	advantages of the method are its simplicity, selectivity and rapidity of
Published : 18 July 2021	determination without the use of any catalyst.
	Keywords: Determination, Vanadium, Presence, O-Phenanthroline, Anthranilic,
	Reagent.

Results and Discussion: Vanadium (IV) obtained by reducing vanadium (V) with hydrazine sulphate forms a complex with anthranilic acid and in presence of o-phenanthroline and can be quantitatively extracted into isoamyl alcohol. The absorption spectrum of the yellowish brownV^{IV}-anthranilic acid-o-phenanthroline complex in isoamyl alcohol along with that of the reagent blank was studied. The blank absorbs very strongly at 375 nm and the absorption decreases sharply to a very low value as the wavelength reaches at 400 nm and beyond which it becomes negligibly small. The complex shows an absorption maximum at 460-465 nm which is chosen for absorbance measurements.

Hydrazinė sulphate is found to be a suitable reductant which rapidly reduces V^V to V^{IV} at pH \leq 4. Vanadium (IV) forms a complex with anthranilic acid but the sensitivity of the reaction is increased several times by the addition of a nitrogenous base of the bases tested, viz. pyridine, 2,2'- dipyridyl and o-phenanthroline, the increase in sensitivity was found to be maximum with the latter. The effect of pH on the extraction of the complex was studied. The complex is slightly extracted at pH \leq 3. The optimum pH range found for maximum extraction is 4.8-5.2. The optimum conditions found for maximum absorbance are 20-150 mg of hydrazine sulphate, 1 ml of anthranilic acid solution (40-120mg m 1⁻¹), 1 ml of o-phenanthroline solution (2.5 - 5mgml⁻¹) and 0.25-10 min of equilibration time.

Many solvents were tested for the best results. The complex is not extracted with benzene, carbon tetrachloride, ethyl acetate, amyl acetate and n-hexane. However, it is extracted easily in chloroform but the formation of a gel-type at the interface causes difficulty in the separation of phases. n-Butanol and isoamyl alcohol both extract the complex well but the absorbance in the latter solvent is higher, hence it is chosen as

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an extractant. Beer's law is obeyed in the range 10-90 μ g V/ml with a sensitivity of 0.057 μ g V/cm² The absorbance of the complex remains unchanged for at least 2 h. The ratio of vanadium to anthranilic acid and o-phenanthroline in the extracted species was studied by Jobs method of continous variation¹. On the basis of these studies, the composition of vanadium (IV): anthranilic acid o-phenanthroline in the extracted species may be formulated as 1:2:1.

Effect of diverse ions: The study was carried cut in solutions containing 50 μ g (V v / ml and in presence of the desired amount of foreign ions. Phosphate and fluoride lower the extraction considerably whereas EDTA, tartrate, citrate and oxalate interfere seriously. Al^{III}, Ti ^{iv}, Th ^{iv}, Bi ⁱⁱⁱ, Snⁱⁱ, Sb^v and Zr^{iv} get hydrolysed under the proposed conditions leading to emulsion formation, thus their tolerance limits are lowered appreciably The tolerance limits of different anions and cations in the determination of 50 μ g V^v /m l are as follows:50 mg each of chloride. sulphate, nitrate: 100 mg each of acetate, thiourea, 10 mg each of Hg["], Znⁱⁱ, Cdⁱⁱ, Mgⁱⁱ, Beⁱⁱ 8 mg each of Nⁱⁱ, Coⁱⁱ, 5 mg each of W^{vi} Cuⁱⁱ : 3 mg of Mo^{vi}, 0.5 mg of Alⁱⁱⁱ 0.1 mg each of Ti^{iv}, Th^{iv} 0.05 mg each of Biⁱⁱⁱ, Sbⁱⁱⁱ, Sn ⁱⁱ and Zr^{iv} Feⁱⁱⁱ, Agⁱ, U^{v1} Cr^{V1} give yellow to brown coloured extracts.

The proposed method is simple and takes hardly 15 min for a single estimation. The method is quite selective as most of the analytically inportant elements such as molybdenum, tungsten, aluminium, cobalt, nickel, copper and zinc do not interfere. The applicability of the method has been tested by analysing a few synthetic samples and a steel sample with satisfactory results. In the analysis of steel sample, firstly the excess of iron was removed by boiling the sample solution with 5% sodium hydroxide The precipitates thus obtained were filtered off and after neutralising the filtrate with 2 N sulphuric acid, the volume was made up to 70.10 ml with distilled water. Aliquots of the sample solution were analysed for vanadium con tents by the proposed method. Vanadium found in carbon steel is 0.068% (certified value, 0.08%) The result of the proposed method compares favourably with some known methods of vanadium determination.

Experimental : A stock solution of vanadium (1mg ml⁻¹) was prepared by dissolving ammonium metavanadate and standardised utrimetrically with ferrous sulphate³. A solution of 100 ug V/ml was prepared by proper dilution o-Phenanthroline solution (5 mg ml⁻¹) and anthranilic acid solution (40 mg ml⁻¹) were prepared in acetone. Solutions of other metai ions were prepared by dissolving the corresponding salts in disolved water or in dilute hydrochloric acid A Shimadzu UV-100-02 spectrophotometer was used for absorbance measurements using 1-cm glass cells and a Systronics 335 pH meter was used for pH-adjustment.

General procedure : To an aliquot of the sample solution, hydrazine sulphate (50 mg) was added after making it slightly acidic with 1N H₂*SO₄ The solution was heated to boiling and after cooling, anthranile acid solution (1 ml) and o-phenanthroline solution (1 ml) were added. The volume was made to 20 mi and pH of the solution was adjusted to 5.0. The complex was extracted isoamyl alcohol (2 x 10ml) shaking each time for 30 s. The two extracts were combined and traces of water were removed by anhydrous sodium sulphate. The volume of the extract was made to 25 ml with isoamyl alcohol, and the absorbance was measured at 460 nm against the reagent blank. The amount of vanadium was deduced from the standard calibration curve.

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