

Spectrophotometric Determination of Micro Amounts of Uranium(VI) Using Resacetophenone

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Uranium(VI) forms an orange yellow soluble complex with resacetophenone (RP) in acid and alkaline media. λ_{\max} of the complex is at 410 nm in the visible range (410-700 nm). 1 ml of the buffer ($\text{CH}_3\text{COOH}-\text{CH}_3\text{COONa}$) solution of pH 5.85 is found optimum. A 50-fold excess of the reagent is essential for complete complexation. The complex is stable for 24 hrs. Beer's law is obeyed upto 47ppm of U(VI). The molar absorptivity and Sandell's sensitivity with respect to U(VI) are 1.18×10^3 l. mol⁻¹. cm⁻¹, and 0.2017 $\mu\text{g. cm}^{-2}$ respectively. Standard deviation evaluated is 0.2ppm. The composition of the complex is established by Job's method and Slope ratio method as 1 : 2 [U(VI) : RP]. The stability constant value evaluated from Job's curve is 1.508×10^7 . The effect of various diverse ions is also studied.

INTRODUCTION

Neelakantam and Urs¹ have reported a method for the determination of U(VI) using resacetophenone oxime in 1952. Recently Huq and Rao² have successfully employed the colour reaction of the complex for the indirect determination of anions. The observation that the parent ketone, resacetophenone (RP) (2,4-dihydroxy acetophenone) itself forms an orange yellow soluble complex in both acid and alkaline media and the fact that one extra step of preparation of its oxime can be avoided prompted the present authors to study the use and versatility of resacetophenone as an analytical reagent for the determination of U(VI).

EXPERIMENTAL

Absorbance measurements are made on Systronics Spectro-Colorimeter-103 and pHs are read on Elico-Digital pH meter model LI-120.

Resacetophenone is prepared by the standard procedure³. Stock solutions of 0.1M RP in 50% aqueous ethanol and 0.01M U(VI) prepared by dissolving requisite amount of $\text{UO}_2(\text{CH}_3\text{COO})_2 \cdot 2\text{H}_2\text{O}$ are used for the studies. All dilutions are made using double distilled water. All other chemicals used are of AR/GR grade.

RESULTS AND DISCUSSION

The colour reaction is tested in both acid and alkaline media using buffer systems, $\text{HCl}-\text{CH}_3\text{COONa}$ (pH 1.22-4.97), $\text{CH}_3\text{COOH}-\text{CH}_3\text{COONa}$ (pH 3.35-5.85) and $\text{NH}_4\text{OH}-\text{NH}_4\text{Cl}$ (pH 6.25-8.68) and found to form a

soluble orange yellow complex at all pHs. From the various preliminary studies made of the complex in the entire range of pHs studied, $\text{CH}_3\text{COOH}-\text{CH}_3\text{COONa}$ buffer of pH 5.85 is chosen as optimum buffer solution for further studies based on its colour and stability.

1ml of 0.01M U(VI) solution 1ml of buffer solution of pH 5.85 and 5 ml of 0.1M RP in a total volume of 25 ml are taken in all measurements excepting in studies where the concentration of the respective species is to be varied. The solvent blank used in each case contains all species except the reagent in the same concentrations as that of the solution.

Absorption Spectra

The spectrum of the complex solution is recorded over the wavelength range 410-700 nm against solvent blank. The complex has maximum absorption at 410 nm. Hence all other studies are made at 410 nm.

Effect of Reagent

The study is made by measuring absorbance at 410 nm of various solutions containing different amounts of the reagent. The study reveals that a minimum of 50-fold excess of reagent is essential for complete complexation. Hence 50-fold excess of reagent is maintained in further studies of the complex.

Effect of Buffer Concentration

The absorbances of various solutions containing different volumes of buffer solution of pH 5.85 are measured at 410 nm against solvent blank and found that the absorbance is maximum with 1 ml of buffer. Hence 1 ml of buffer is chosen as optimum volume for further studies. The effect of time reveals that the complex is stable for 24 hrs.

Beer's Law Obedience

The absorbances of various solutions containing different amounts of U(VI) are measured at 410nm against solvent blank. A linear increase in the absorbance values indicates that Beer's law is obeyed upto $2 \times 10^{-4}\text{M}$ U(VI) [47.6ppm of U(VI)].

The molar absorptivity and Sandell's sensitivity⁴ with respect to U(VI) are $1.18 \times 10^3 \text{ l. mol}^{-1} \cdot \text{cm}^{-1}$. and $0.2017 \mu\text{g. cm}^{-2}$ respectively at 410nm. The standard deviation in this method is found to be 0.2ppm.

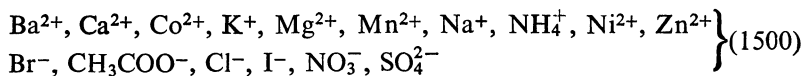
Composition of the Complex and Stability Constant

The composition of the complex is established as 1.2 [U(VI):RP] by Job's method by studying the absorbances of the various solutions containing varying volumes of equimolar (0.01M) solutions of U(VI) and RP at 410nm against solvent blank. The stability constant evaluated from Job's curve is 1.508×10^7 . The composition of the complex is verified by

slope-ratio method and found to be confirming to the same composition of metal and ligand as that obtained from Job's method.

Effect of Diverse Ions

The interference studies of various cations and anions keeping the concentration of U(VI) at 2×10^{-4} M (47ppm) is carried out. The tolerance limit is set at 0.005 scale unit at 410nm. The tolerance limit evaluated for each of the various ions studied is as indicated against the ion in parenthesis in ppm.



Al^{3+} (50), Cu^{2+} (100), Zr^{4+} (50), Fe^{3+} (6), Oxalate(10), Citrate(10), Fluoride (20), Phosphate(6), Tartrate(5), CO_3^{2-} (300), HCO_3^- (100), Tetraborate(500) and EDTA(60).

The present method of determination of uranium(VI) is simple, rapid and sensitive one, allowing determination of micro quantities of U(VI) and hence offers an advantage over the U(VI)-RP oximate method (vide ref. 1) where in the range of determination is 0.3 mg to 5 mg.

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