Spectrophotometric Determination of Tantalum(V) with 5-Iodo-8--hydroxyquinoline-7-sulphonic Acid

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Tantalum(V) reacts with 5-iodo-8-hydroxyquinoline-7-sulphonic acid to form a yellowish coloured complex having maximum absorbance at 405 nm in citric acid medium. The relatively slow reaction at room temperature is completed in 10 minutes at 40 °C. The colour is stable for at least 12 hr. Effects of heating temperature, time, pH, reagent concentration and other variables have been studied. The system confirms the Beer's law over the concentration range investigated. The optimum condition range for measurement in 10 mm cell is 1—7.5 μg of tantalum(V). The molar absorptivity is 5.753×10^4 mol^{-1} dm^2. The interference of foreign ions has been evaluated. The metal-ligand ratio of 1 : 2 was confirmed by the mole ratio method.

INTRODUCTION

5-Iodo-8-hydroxyquinoline-7-sulphonic acid like its parent compound 8-hydroxyquinoline acts as a chelating agent. The complex prepared by the reaction of tantalum(V) with 5-iodo-8-hydroxyquinoline-7-sulphonic acid (IHQSA) in presence of some additives was studied. The present work deals with the spectrophotometrical investigations of tantalum-5-iodo-8-hydroxyquinoline-7-sulphonic acid chelate regarding its composition, sensitivity and stability. Since tantalum(V) forms weak acids, it hydrolyses readily in the absence of complexing agents. Complex tantalates formed by replacing the oxygen atoms with radicals such as oxalate, tartarate and citrate are well known[1-3]. Citric acid is widely used to stabilise tantalum solution against hydrolysis. This stabilising effect results from the formation of fairly stable chelates, thereby suppressing the tendency to form colloidal solution due to hydroxo complex polymerisation.

EXPERIMENTAL

Materials and Method

Spectral measurements at a fixed wavelength were made with a Backmann model DU Quartz spectrophotometer. Matched Cells of 10.0 mm optical path were used with a fixed slitwidth of 0.08 mm. Measurements of pH were made with a pH meter having both standard and expanded scales and a miniature type of calomel-glass electrode system. All the chemical used were of analytical grade.

Standard Tantalum(V) Solution (1 mg/ml)

Pure tantalum pentoxide (0.138 g) was fused with 10 grams of potassium bisulphate in a silica crucible and after cooling, the warm melt was extracted with 10 ml of a 25% citric acid solution. The solution was transferred in a 100 ml standard flask and made up to the volume with distilled water. The pH was adjusted to 4.0 with dil. ammonia or dil. H_2SO_4[4]. The solution was standardised with tannin by precipitation and weighing of tantalum as oxide[5].

Recommended Procedure

A suitable aliquote of sample solution containing 5 ml of tantalum(V) of 1.0×10^{-3} mol/dm³ and about a three fold molar excess of 1.0×10^{-4} mol/dm³ of the reagent solution was adjusted to pH = 6.3 and made up to 20 ml. The absorbance was measured at 405 nm against the reagent blank prepared under indentical conditions; the amount of tantalum(V) was determined from the calibration curve.

RESULTS AND DISCUSSION

Spectral Characteristics

In order to observe the wavelength of maximum absorption of the complex, a solution containing a fixed amount of metal and an excess of the ligand was prepared and pH of the solution was adjusted to 6.3 making the total volume 25 ml. The absorption spectra were recorded against the corresponding reagent blank. Results indicate that the complex has a maximum absorbance at 405 nm. In order to observe the spectral changes on varying the pH, absorption curves for the tantalum complex at different pH values were prepared. The maximum absorbance of the tantalum complex is found at 405 nm at pH = 6.3, and shorter wavelength at a lower pH.

Effect of Hydrogen Ion Concentration

To study the effect of pH on the absorbance, a series of solutions containing a fixed amount of metal and ligand were prepared at different pH values making a total volume of 25 ml. The absorbance was measured at 405 nm. A curve was obtained by plotting the absorbance against the pH of the solution. Maximum absorbance was obtained in the pH range from 6.0 to 6.5 and therefore further studies were carried out at pH = 6.3 and measurements were made at 405 nm.

Reagent Concentration

A series of solutions containing a fixed amount of tantalum(V) and varying amounts of reagent (IHQSA) were prepared. The pH was adjusted to 6.3 and total volume raised to 25 ml. A plot of absorbance versus reagent concentration shows that about three moles of the reagent per mole of tantalum(V) are required for full colour development. A three fold molar excess of the ligand was maintained in subsequent studies to ensure full colour development.

Citric Acid Concentration

Citric acid[3] is added to prevent the hydrolysis of tantalum salt. Tests showed that the minimum amount of citric acid necessary to prevent this hydrolysis is about 2 grams, a reasonable safety margin is provided in the presence of 10 ml of a 25% solution of the citric acid. Increase in the amount of citric acid by 30—35% caused a decrease in absorbance.

and 10 ml of the ligand solution were mixed with 2 ml of tantalum solution. The total volume was made to 25 ml. The experiment was also repeated at another concentration (1.0×10^{-4} mol/dm³) for both metal and ligand. The absorbance of the solutions was measured at 405 nm. A plot of absorbance versus ratio of ligand to metal concentration was drawn. A sharp break on the curve occurs at 1 : 2 composition of tantalum and the reagent (IHQSA). The observations, therefore, confirm the 1 : 2 stoichiometry of the complex.

DISCUSSION

Tantalum has been found to form complexes with carboxylic and hydroxy acids viz. citric[3], tartaric[2] and oxalic acid[1]. In the present study the citrato complex has been taken as an auxiliary complex. Owing to the insolubility of tantalum pentoxide, it was preferred to fuse it with bisulphate of potassium and then leach the melt with citric acid solution to give the citrato complex of tantalum. Aqueous citric acid solution dissolves the precipitated hydrated tantalum pentoxide and is frequently used as a stabilising agent for tantalum solutions. The formation of chelate complexes between tantalum and citric acid greatly retards the polymerisation and hydrolysis.

The citric acid may be replaced by another α-hydroxy carboxylic acid such as tartaric acid. Tartaric acid is commonly used to stabilize tantalum solutions against hydrolysis[2]. The tantalum stabilizing power of this acid is greater than that of oxalic or citric acid. In the presence of tartarate in acid medium tantalum can be extracted by many polyamino carboxylic acids[10-12]:

Since alkali-tantalate solutions are hydrolizing spontaneadisly, it was necessary to start with solution containing tantalum in the form of these complexes. On the other hand, the acid tantalates are fairly stable for a long time if stored properly at suitable a pH. Further the fusion method for the preparation of a tantalum(V) solution was preferred because other salts of tantalum like halids can not be used in aqueous medium because an immediate hydrolysis would result in the formation of insoluble earth acids.

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SAŽETAK

Spektrofotometrijsko određivanje tantala(V) s 5-jodo-8-hidroksikinolin-7-sulfonskom kiselinom

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Tantal(V) reagira s 5-jodo-8-hidroksikinolin-7-sulfonskom kiselinom tvoreći žućkasto obojeni kompleks koji u otopinama citronske kiseline pokazuje maksimum apsorpcije pri 405 nm. Pri sobnoj temperaturi reakcija nastajanja kompleksa je spora. Pri 40 °C maksimalni doseg reakcija postiže u roku od 10 min. Boja nastalog kompleksa stabilna je kroz najmanje 12 sati. U radu su ispitani utjecaji temperature, vremena, pH i sastava otopine na značajke kompleksa. Utvrđeno je da sistem slijedi Beer-ov zakon u ispitanom području koncentracija. Optimalni uvjeti mjerenja kiveti debljine 10 mm postižu se u području sadržaja tantala od 1 do 7,5 µg. Molarni koeficijent apsorpcije kompleksa iznosi $5.75 \cdot 10^4$ mol^{-1} dm^2. Ispitan je utjecaj drugih iona i utvrđen odnos metal : ligand u iznosu od 1 : 2, metodom molnih udjela.

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