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Spectrophotometric determination of gold(III) using alfuzosin hydrochloride (R,S)-N-[3-[(4-amino-6,7-dimethoxy-2-quinazolinyl) methylamino] propyl] tetrahydro-2-furancarboxamide hydrochloride. reagent

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ABSTRACT

A Alfuzosin hydrochloride (AFZ) is chemically (R,S)-N-[3-[(4-amino-6,7-dimethoxy-2-quinazolinyl) methylamino] propyl] tetrahydro-2-furancarboxamide hydrochloride. The empirical formula and molecular weight of alfuzosin hydrochloride are $C_{19}H_{27}N_5O_4HCl$ and 425.9 respectively, was employed for the sensitive spectrophotometric determination of gold(III). The gold(III) ion forms a red-coloured complex with AFZ in an aqueous solution at pH 4.0. The gold complex shows the absorption maximum at 540 nm, and Beer's law obeyed in the range of 4.92-78.76 μ g/ml. The molar absorptivity and sandell, s sensitivity were found to be $1.92 \times 10^3 \text{ mol}^{-1} \text{ cm}^{-1}$ and 0.0010 μ g/cm² respectively. The complex shows 1:1 [Au(III) : AFZ] stochiometry with a stability constant of 4.31 $\times 10^4$. The interference effect of various diverse ions has been studied. In addition to zero- order, first derivative spectrophotometric methods was also developed for the determination of gold(III) in trace amount which was more sensitive than the zero-order method. The developed method has been used for the determination of gold(III) in various kinds of alloys. The results was in excellent agreement with certified values.

INTRODUCTION

Gold is considered to be one of the most significant noble metals due to its wide application in industry and economic activity. It is the most interesting micro amount elements due to its significant role on biology. Gold occurs at very low natural contents, about $4ng g^{-1}$ In rocks, $1ngg^{-1}$ in soils, and 0.05 ng mL⁻¹ in sea water. Gold particles are present in the bodies of fish, aquatic invertebrates, and humans. It has been used in medicine, for example, to cure rheumatoid arthritis under treatment called "chrysoteraphy [sic]." It is prescribed when treatment with non steroid anti-inflammatory drugs is failed to give relief. Gold is a soft metal and so is usually alloyed to give it more strength. Alloy of gold with the other elements of I B group in the periodic table are most frequently used, and gold still has great significance in international business and banking. So, simple, sensitive and selective methods for determination of trace gold are always significant.

Among the several instrumental techniques spectrophotometric methods for the determination of metal ions have an attractive attention due to their simplicity and low operating costs. A variety of spectrophotometric method for the determination of gold have been reported includes with (R,S)-N-[3-[(4-amino-6,7-dimethoxy-2-quinazolinyl)methylamino]propyl]tetrahydro-2-furancarboxamide hydrochloride(AFZ). Reagent.

Alfuzosin hydrochloride is one of the important classes of reagents widely employed for the spectrophotometric determination of metal ions. Alfuzosin hydrochloride contain good chelating agents and form complexes with various metal ions by bonding

Structural of Alfuzosin hydrochloride is shown below:



In the literature survey the spectophotometric methods for the determination of gold(III) using Alfuzosin hydrochloride are very less. The present work report the simple, sensitive, selective and non extracrive spectrophotometric determination of gold(III) using (R,S)-N-[3-[(4-amino-6,7-dimethoxy-2-quinazolinyl) methylamino] propyl] tetrahydro-2-furancarboxamide hydrochloride Reagent. The developed method has been used for the determination of gold(III) in various kinds of alloys.

MATERIALS AND METHODS

2.1 APPARATUS

The absorbance and pH measurements were made on a shimadzu UV – visible spectrophotometer (model UV – 160) fitted with 1-cm quartz cells and Philips digital pH meter(model L1 613), respectively. The pH meter has temperature compensation arrangement and has reproducibility of measurements within ± 0.01 pH

2.2 REAGENTS AND CHEMICALS

The [(R,S) - N-[3-[(4-amino-6,7-dimethoxy-2-quinazolinyl) methylamino] propyl] tetrahydro-2-furancarboxamide hydrochloride(AFZ) was purchased from SD fine chemicals, India. The gold(III) chloride trihydrate (HAuCl4.3H2O) was obtained from Alchemy Laboratories, India. All chemicals and solvents used were of analytical reagent grade. Doubly distilled water was used for the preparation of all solutions and experiments.

A 100mg of Alfuzosin hydrochloride is weighed accurately and transferred in to a 100ml standard flask dissolved made up to the mark with methanol this solution is diluted as required.

Stock solution of 0.1 gold(III) was prepared by dissolving precise amount of HAuCl₄.3H₂O in 1M hydrochloric acid and standardized using standard procedure.

The buffer solution were prepared by mixing in 0.2M sodium acetate + 1M hydrochloric acid (pH 2.5 - 3.5) and 0.2M sodium acetate + 0.2M acetic acid (pH 3.0 - 6.0). The pH of these solutions was checked with above mentioned pH meter.

The SDS surfactant was prepared by dissolving 2gm of Sodium Dodecyl Sulphate(SRL Chemicals) in 100ml of double distilled water.

The working solutions were prepared daily by diluting the stock solution to an appropriate volume. The solutions of the studied interfering ions of suitable concentrations were prepared using analytical grade reagents.

2.3 GENERAL PROCEDURE 2.3.1 DIRECT SPECTROPHOTOMETRY

In each of a set of different 10ml standard flasks, 5ml buffer solution (pH 4.0), varying volumes of 5.0×10^{-3} M Au(III) solution, and 2ml of AFZ (2.35 X 10^{-3} M) solution were taken, then add 2% SDS surfactant solution and the volume made up to the mark with doubly distilled water this solution heated 60minutes at 65° C for red colure complex formation, after cooling it to room temperature. The absorbance was measured at 570nm against the reagent blank. The calibration curve was constructed by plotting the absorbance against the amount of Au(III). The calibration graph follows the straight line equation , where was the concentration of solution, was measured absorbance or peak height, and "" were constants.

2.3.2 DERIVATIVE METHODS

For the above solutions, first derivative spectra were recorded with a scan speed of fast (nearly 2400nm min⁻¹) slit width of 1nm with one degree of freedom. The derivative peak height was measured by the peak-zero method at respective wavelengths. The peak height was plotted against the amount of Au(III) to obtain the calibration.

RESULTS AND DISCUSSION

3.1. ZERO-ORDER METHOD

The gold(III) reacts with AFZ (Alfuzosin hydrochloride) forming a red – colored soluble complex in the acidic buffer solution in the presence 1.5ml of 2% SDS surfactant. The absorbance spectra of the AFZ and its gold(III) complex under optimum conditions were presented in Figure 1.The gold (III) complex has an absorption maximum at 540nm, and at this wavelength the reagent has very small absorbance, Hence, further analytical studies were carried out at 540nm.



 $[Au(III)] = 5.0 \times 10^{-3}; [AFZ] = 2.35 \times 10^{-3}M$

Preliminary syudies have indicated that AFZ reacts with Au(III) in aqueous acidic medium to form red colour species after heating the experimental solution at 65° C for 60 minutes and cooling it to room temperature. The absorbance of the complex was found to be constant for more than 85 hours. The effect of pH on the intensity of colour formation was studied to optimize the pH of the gold complex. The results indicate that absorbance was maximum and constant in the pH range of 3.5 - 4.5. Hence pH 4.0 was chosen for further studies.

A 10-fold molar excess of AFZ was necessary for comlex and constant colour development. Excess of the reagent has no effect on the sensitivity and absorbance of the complex. To determine the amount of Au(III) at micro levels, beer's law was verified for [Au(III)-AFZ] complex by measuring the absorbance of the solutions containing different amounts of Au(III). A linear plot between the absorbance and the amount of Au(III) gives the straight line which obeys the equation (Figure 2). The correlation coeffcient (γ) of the calibration curve for experimental data was 0.99996. From the calibration plot, it is observed that Beer's law was obeyed in the range of 4.92-78.76 µg/ml. The molar absorptivity and Sandall's sensitivity were 1.92 x 10³ mol⁻¹ cm⁻¹ and 0.0010 µg/cm² respectively. The relative standard deviation at a concentration level of 19.69 µg/ml of Au(III) was found to be 0.0034 (10 determinations).



Figure 2: Absorbance Vs amount of Au(III) (μ g/ml) [AFZ]= 2.35 x 10⁻³M ; pH = 4.0 ; λ = 540 nm

The composition of complex was determined by job's (Figure 3(a)) and molar ratio methods (Figure 3(b)). Both methods showed that a molar ratio of Au(III) to LMG was 1:1. The stability constant was determined by job's method as 4.31×10^4 .



Figure 3(a) : Job's Curve [Au(III)] = [AFZ] = 5.0 x 10^{-3} M ; pH = 4.0 ; λ = 540 nm



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The effect of various foreign ions that were generally associated with gold ion on its determination under optimum conditions was investigated by the determination $20 \,\mu g \text{ ml}^{-1}$ of Au(III), and the results are presented in table 1. The tolerance limit was set as the amount of foreign ion that caused an error in the absorbance by $\pm 4\%$. The effect of various cations and anions invesigated. It was found that all the ions did not interfer in the zero-order determination of Au(III).

Ion	Tolerance limit (µg/ml)	Ion	Tolerance limit (µg/ml)	
Iodide	28	Mo(VI)	159	
Bromide	87	Ir(III)	316	
Chloride	582	Co(II)	96	
Fluoride	416	Ba(II)	226	
Carbonate	659	Zn(II)	143	
Sulphate	1587	Ce(IV)	231	
Nitrate	1363	W(VI)	253	
Phosphate	1565	Mn(II)	91	
Oxalate	1157	V(V)	57	
Thiocyanate	318	Ag(I)	119	
EDTA	2425	U(VI)	391	
Tartrate	1499	Zr(VI)	151	
Citrate	472	Se(IV)	173	
Acetate	811	Th(IV)	382	
Ascarbate	494	Te(IV)	139	
Na(I)	312	Pb(II)	226	
K(I)	858	Ga(III)	152	
Mg(II)	527	In(III)	253	
Ca(II)	879	Y(III)	293	
Ru(III)	167	La(III)	609	
Cr(VI)	86	Ti(IV)	211	
Fe(III)	92	Ni(II)	257	
Cu(II)	104	Al(III)	119	
Cd(II)	186	NH4(I)	395	
Ho(II)	330	Pd(II)	108	

Table 1. Tolerance limit of foreign ions [gold(III)] = $20\mu g/ml$; pH = 4.0





3.2 derivative methods

the fist derivative spectra of experimental solutions containing different amounts of Au(III) were recorded in the wavelength region. The first derivative spectra (Figure 4) showed maximum amplitude at 570nm. The derivitive amlitudes at at 570nm were found to be proportional to the concentrations of Au(III).

The effect of various cations and anions on the derivative methods was also investigated. It was noticed that all the ions that did not interfere in the zero-order determinations of Au(III) (cf. Table 1) also did not interfere in all the derivative spectrophotometric methods.

Applications

The proposed method was applied for the determination of gold(III) in Egyptian gold alloy samples and certified reference materials. the results of the determinations are given in Table 2.

Egyptian Gold Alloy	Certified Composition (%)	Amount of gold(III) (µg/ml)		Error (%)		
(sample Nos)		Taken	Found *			
1.	Au (87.66); Cu (6.81); Ag (6.12)	87.66	87.99	0.37		
2.	Au (74.92); Cu (12.53); Ag (12.48)	74.92	74.16	-1.01		
3.	Au (55.15); Cu (20.82); Ag (20.82)	55.15	55.01	-0.25		
* Average of Seven determinations						

Determination of gold(III) in Egyptian Gold Alloy samples

CONCLUSION

The present work gives a rapid, simple, sensitive, and selective method for the non extractive spectrophotometric determination of gold(III). The developed method require the use of a surfactant. Further, derivative spectrophotometric methods also developed and are more sensitive than zero-order method. The most foreign ions do not interfere with the determination. The developed method was used for the determination of gold(III) in Egyptian gold alloy samples and certified reference materials.

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