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DIRECT SPECTROPHOTOMETRIC DETERMINATION OF GOLD (III) WITH 5-BROMO-2-HYDROXY-3-METHOXYBENZALDEHYDE-P-HYDROXYBENZOIC HYDRAZONE REDDY BV, SARITHA B, GIRI A, REDDY TS

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Abstract: A rapid, simple, sensitive and selective direct spectrophotometric method has been developed for the determination of gold (III) in various real samples. 5-Bromo-2-hydroxy-3-methoxy benzaldehyde-p-hydroxybenzoic hydrazone reacts with Au (III) forming ruby coloured soluble complex [Au (III)-5-BHMBHBH] in basic buffer solution. The maximum absorbance was observed in the pH range 4.0-7.0 and Au (III)-5-BHMBHBH shows at λ_{\max} at 400 nm. The method obeys Beer's law in the range 1.96 – 19.69 $\mu\text{g}/\text{ml}$. The molar absorptivity is $5.722 \times 10^3 \text{ L mol}^{-1} \text{ cm}^{-1}$ and Sandell's sensitivity is $0.0342 \text{ mg}/\text{cm}^2$. The standard deviation of the method for ten determinations of $7.87 \mu\text{g}/\text{ml}$ of Au (III) is 0.00199. The correlation coefficient (γ) of the calibration equation of the experimental data is 0.9999. The effect of various diverse ions is studied. The formula of the complex is 1:1 and its stability constant is 1.866×10^6 . The method developed has been used for the determination of Au (III) in alloy samples, water samples.

Keywords: Au (III), Spectrophotometry, 5-Bromo-2-hydroxy-3-methoxybenzaldehyde-p-hydroxybenzoic hydrazone (5-BHMBHBH). Alloys, Water samples



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INTRODUCTION

Gold (III) is one of the most important noble metals because it has wide application industry and economic activity. The beauty and rarity of gold has lead to its used in ornaments and as a standard for monitory systems throughout the world. It is often used a medicine. It is usually alloyed with copper or silver to give more strength, as gold is a soft metal. It a good conductor of heat and electricity. Gold is used in textile industry. Gold - silver or gold-platinum find dental applications.

Sophisticated techniques, such as inductively coupled plasma mass spectrometry(ICP-MS)[1], inductively coupled plasma atomic emission spectrometry (ICP-AES)[2], electrochemical[3], spectrophotometry[4], neutron activation analysis[5] and atomic absorption spectrophotometry [6](AAS) have widely been applied to the determination of gold in various samples. Some factors such as initial cost of instrument, technical know-how, consumable and costly maintenance of technique restrict the wider applicability of these techniques, particularly in laboratories with limited budget in developing countries and for field work. A wide variety of spectrophotometric methods for determination of gold have been reported, each chromogenic systems has its advantages and disadvantages with respect to sensitivity, selectivity and convenience. Many spectrophotometric methods[7-17] have been reported for the determination of gold (III). Rubina Soomro et al.[18] have reviewed several spectrophotometric procedures using Schiff's base reagents. Most of the spectrophotometric methods reported for the determination of gold(III) suffer from interference due to diverse ions or less sensitive or involve extraction or use surfactants[19]. Hence, there is a need for direct spectrophotometric methods for the determination of gold (III).

Potentiality of hydrazone derivatives have been critically reviewed by Singh et al [20]. Survey of literature reveal no direct spectrophotometric determination of gold (III) using parahydroxybenzoichydrazone. Present paper report a direct, simple spectrophotometric procedure for the determination of gold (III) using 5-Bromo-2-hydroxy-3-methoxybenzaldehyde-p-hydroxybenzoichydrazone (5-BHMBHBH).

MATERIALS AND METHODS:

The chromogenicreagent, 5-bromo-2-hydroxy-3-methoxybenzaldehyde-p-hydroxy benzoichydrazone was synthesized in the laboratory by condensing 5-Bromo-2- hydroxy-3-methoxybenzaldehyde and p-hydroxybenzoichydrazide. A 0.01M DMF solution of the reagent is used in the studies.

0.01M stock solution of Au (III) was prepared by dissolving requisite amount of gold(III)chloride in distilled water and standardized[21]. The working solutions were prepared by diluting the stock solutions with distilled water.

Buffer solution of pH 5.0 was prepared by mixing 0.2M sodium acetate and 0.1M acetic acid in suitable proportion and the pH was adjusted by a pH meter.

The absorbance and pH measurements were made on a Perkin Elmer (LAMDA 25) UV-Visible spectrophotometer (Model UV-160A) controlled by a computer fitted with 1cm path length quartz cells and an ELICO digital pH meter of (Model LI 613) respectively.

PROCEDURE:

To 5 ml of buffer solution (pH 5.0), 0.5 ml of 5-BHMBHBH (1×10^{-2} M) in DMF, 1.5 ml of DMF taken in each of a set of 10 ml volumetric flasks, varying amounts of Au(III) were added and diluted to 10 ml in a volumetric flask with distilled water. The absorbance of these solutions was measured at 400 nm. A straight line is obtained which corresponded to the equation $A_{400} = 0.01908C + 0.0005$. (C is the amount of gold in $\mu\text{g/ml}$).

RESULTS AND DISCUSSIONS:

The reagent 5-bromo-2-hydroxy-3-methoxy benzaldehyde-4-hydroxy benzoichydrazone (5-BHMBHBH) was used for the spectrophotometric method for determination of gold(III). Gold(III) reacts with 5-BHMBHBH to form ruby coloured complex in the pH range 4.0-7.0. The absorption spectra of 5-BHMBHBH and its gold (III) complex under the optimum conditions were shown in Figure1. The gold (III)-5-BHMBHBH complex shows maximum absorbance at 400 nm, where the reagent has negligible absorbance . The colour formation is instantaneous. It is stable for 48 hours. The analytical measurements were made at pH 5.0. A five fold molar excess of the reagent is sufficient to produce maximum absorbance. The formula of the complex was ascertained from jobs and molar ratio methods as 1:1. The stability constant of the complex is 1.866×10^6 . The analytical characteristics of the complex are given in table-1.

Effect of foreign ions:

The effect of foreign ions and cations on the determination of Au(III) under optimal conditions were studied table-2. Indicate that many metal ions and anions do not interfere in the determination of Au(III). 10 fold excess of Cu(II) is masked with thiosulphate and 30 fold excess of Fe(III) , Ti(IV) and Al(III) are masked with fluoride.

Applications

The developed spectrophotometric method for the determination of gold (III) was applied successfully for its determination in alloy and water samples.

a) Determination of Au(III) in Alloy sample:

The alloy sample was brought into solution by the procedure. An aliquot of the sample was analysed by the following procedure.

1gm of the alloy sample was dissolved in a mixture of 2ml of concentrated hydrochloric acid and 10ml of concentrated nitric acid. The solution was evaporated to a small volume. 5ml of 1:1 sulfuric acid was added and the solution was evaporated to dryness. The residue left over was extracted with 15ml of water and the solution was diluted to 100ml with double distilled water. This serves as the stock solution. The stock solution was appropriately diluted and analyzed by the general procedure. The results are given in table-3.

b) Détermination of Au(III) in Water samples:

The water samples were collected from different parts of ananthapuramu district. (A.P. India) and treated as follows.

1 liter of the water sample was taken in a 2 liter beaker and evaporated slowly 225 ml. 5 ml of H₂O₂ were added and evaporated to dryness in a china dish. filtered. It was then dissolved in 20 ml of water and transferred quantitatively into a 100 ml volumetric flask and made up to the mark with distilled water. An aliquot of the sample was analysed by the following procedure. The results are given in table-4.

CONCLUSION:

The proposed method is simple, accurate, sensitive, do not involve extraction or heating and do not use surfactants. The method can be easily applied for the determination of gold (III) in alloy and water samples.

Table 1. Analytical characteristics of [Au(III) – 5-BHMBHBH]

Parameter	Direct method (400 nm)
Beer's law range ($\mu\text{g/ml}^{-1}$)	1.96 – 19.69 $\mu\text{g/ml}$
Molar absorptivity ($\text{L mol}^{-1}\text{cm}^{-1}$)	$5.722 \times 10^3 \text{ lit mol}^{-1} \text{ cm}^{-1}$
Sandell's sensitivity ($\mu\text{g/cm}^2$)	0.0342
Correlation coefficient (γ)	0.9999
Standard deviation	0.00199
Y- intercept(b)	0.0005
Detection limit($\mu\text{g/ml}^{-1}$)	0.2085
Composition (Metal :Ligand)	1:1
Stability constant(β)	1.866×10^6

Table 2. Tolerance limit of foreign ions. Amount of Au(III) = 7.87 $\mu\text{g/ml}$, pH = 5.0

Ion	Tolerance limit ($\mu\text{g/ml}$)	Ion	Tolerance limit ($\mu\text{g/ml}$)
Fluoride	999	Se (IV)	80
Chloride	710	Pb (II)	84
Bromide	400	Mg (II)	100
Iodide	127	Zr (IV)	66
Carbonate	130	Cr (VI)	60
Nitrate	480	Hg (II)	24
Sulphate	620	Th (IV)	40
Phosphate	195	Ru(III)	42
Thiosulphate	240	Co (II)	21
Oxalate	44	Te (IV)	25
Thiocyanate	290	W (VI)	55
Tartarate	1480	Ce (IV)	42
Citrate	1360	Pd(II)	interferes
Ascorbate	1305	Al (III)*	40
Thiourea	960	Mn (II)	66
EDTA	interferes	Cd (II)	340
Fe(III)*	156	Zn (II)	330
Ti(IV)*	50	Ca(II)	400
Cu(II)#	31	Ni (II)	8
V(V)	51		

* masked with fluoride

masked with thiosulphate

Table 3. Determination of Au (III) in alloy samples.

Sample	Composition certified	(%)	Composition found*	(%)	Relative error (%)
1*18 Carat gold*	75		74.6		- 0.53
2*	87.66		88.01		0.40
3*	74.92		74.88		-0.05
4*	55.15		55.07		-0.14

* Average of seven determinations.

Composition of sample (%)

1. Au, 75 ; Cu, 2.2 ; Zn, 5.5 ; Ni, 17.3 ;
2. Au, 87.66; Cu, 6.81;Ag, 6.12;
3. Au, 74.92 ; Cu, 12.53; Ag, 12.48;
4. Au, 55.15 ; Cu, 20.82 ; Ag, 20.82;

Table 4. Determination of Au (III) in water samples.

Sample	Gold (III) ($\mu\text{g/l}$)		Recovery(%)
	Added	Found*	
Jewels waste water ^a	0	112.0	99.0
	50	161.5	
Drain water ^b	0	27.0	99.6
	50	76.8	

* Average of seven determinations.

a. Water collected from Jewellery Shops in Ananthapuramu town.

b. Water collected from Jewellery shops area in Ananthapuramu town.

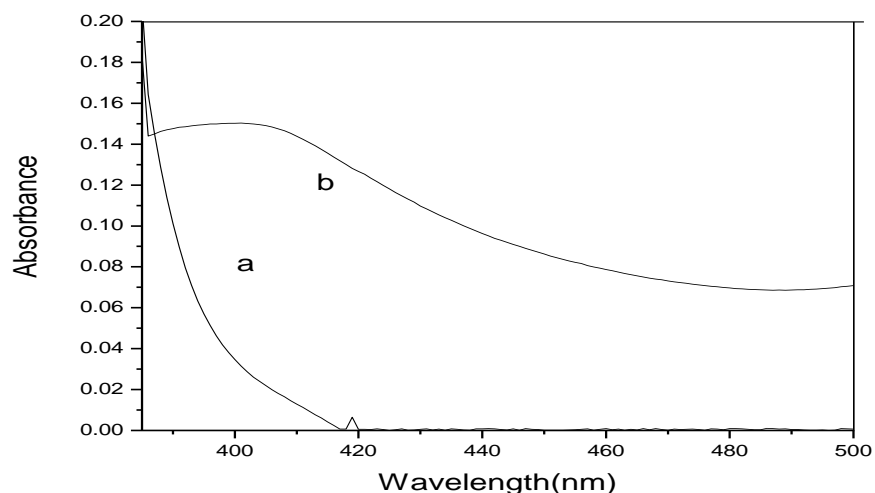


Fig.4.3.1. Absorption spectra of

a) 5-BHMBHBH Vs buffer blank

b) [Au (III)] – 5-BHMBHBH Vs reagent blank

[Au(III)] = 2.0×10^{-5} M; [5-BHMBHBH] = 1.0×10^{-2} M; pH = 5.0

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