RESEARCH ARTICLE

A Spectrophotometric Simultaneous Determination of Cobalt (II) and Iron (III) With Res-Acetophenone Guanylhydrazone (RAG)

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ABSTRACT

We report here a new simple effective spectrophotometric simultaneous determination of Cobalt (II) and Iron (III) with Res-acetophenone guanylhydrazone [RAG]. A standard procedure of the absorption spectra of the reagent and the complex is recommended. The absorbance measurements of Co(II) are carried out at 415 nm [Molar extinction co-efficient is 0.4426×10^4 lit. mole cm^{-1}] and at 520nm [Molar extinction co-efficient is 0.1230×10^4 lit. mole cm^{-1}] while of Fe(III) at 415 nm [Molar extinction co-efficient is 0.1223×10^4 lit. mole cm^{-1}] and at 520nm [Molar extinction co-efficient is 0.2016×10^4 lit. mole cm^{-1}] at pH 7.6. The method is applied for the determination of Cobalt and iron in steel alloy and synthetic mixtures.

KEYWORDS

RAG, Cobalt(II), Iron(III) , Spectrophotometry

INTRODUCTION

Separate estimation of cobalt and iron in excess of other metal radicals is well developed and various reagents have been used for spectroscopic simultaneous determinations of individual element. Numerous methods for the spectroscopic simultaneous determination of cobalt and Iron have been reported[1-5]. Res-acetophenone guanylhydrazone (RAG) has been used as sensitive reagent for spectroscopic simultaneous determinations of Co (II) and Fe (III). It forms a yellow colored complex with cobalt and red colored complex with Iron at pH 7.6, which leads to the development of a simple and rapid simultaneous spectrophotometric determination of cobalt and iron at tracer level.

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MATERIALS AND METHOD

A standard solution of Co (III) (1 mg/ml i.e. 1.6×10^{-3} M) was prepared by dissolving 0.477 g of A.R. grade Cobalt sulphate heptahydrate in 100 ml distilled water. The solution was standardized with EDTA volumetrically.

A standard solution of Fe (III) (1 mg/ml i.e. 1.7910×10^{-2} M) was prepared by dissolving 0.308 g of A.R. grade Ferric ammonium sulphate monohydrate in 100 ml distilled water containing a 2-3 drops of sulphuric acid. The solution was standardized with Volhard's method volumetrically.[7]

A stock solution of 0.5×10^{-2} M reagent (RAG) was prepared by dissolving 0.104 g of it in 100ml ethyl alcohol. The buffer solutions were prepared by dissolving appropriate amounts of boric acid and sodium hydroxide.

Impact Factor = 1.0285

Procedure

A suitable aliquot of the solution containing 50 μg of Co (II) and 50 μg of Fe (III) was taken in a 10ml volumetric flask. To this, was added 1.5 ml of reagent (RAG) solution of concentration $(5.0 \times 10^{-3} \text{ M})$ and diluted to 2/3 volume of the flask. Then pH of the solution was adjusted to 7.4 by adding buffer solution and diluted to volume with distilled water. The absorbance of the Co (II) and Fe (III) complexes with RAG were measured at 415nm and 520 nm respectively. The concentration of Co (II) and Fe (III) in an unknown solution was determined from a calibration curve obtained under identical conditions.

Applications

The method was applied in the determination of Co (II) and Fe (III) in the steel alloy and synthetic mixtures.

Determination of Co(II) and Iron(III) in Steel Alloy

A 0.1g steel alloy sample is treated with 15.0 ml conc. HNO_3 until the brisk reaction ceased. Aqua-regia 10.0ml was added in it and then solution was evaporated to near dryness to expel nitrogen oxides. The residue was dissolved in 50.0 ml slightly acidified water by heating. The residue was dissolved.

After addition of 1.0ml conc. H_2SO_4, the mixture is evaporated until white fumes appear, then cooled and diluted to about 100ml with 0.3ml HCl. The solution is heated almost to boiling and H_2S is passed through it to precipitate all the copper. The Copper sulphide is filtered off and washed.

The filtrate is boiled to remove hydrogen sulphide then evaporated to about 50ml.

Table 1: Determination of Iron (III) in Cupronickel Alloy

Sample	Composition %	Iron(III)		Relative standard deviation %
		Found %	X %	
Cupronickel Alloy	Cu(II) = 67.0 Fe(III) = 0.83 Mn(II) = 0.80 Ni(II) = 31.2	0.827 0.835 0.841 0.838	0.835	0.98

Table 2: Determination of Iron (III) in synthetic mixtures

Iron (III) = 17.5 μg
Each ion added = 125.0 μg

Ions added, μg	Iron (III), μg	
	Found	X
Mg(II), Zn(II), Ce(IV)	17.2, 17.7, 17.3	17.40
Bi(III), Sb(III), Al(III)	17.4, 17.5, 17.6	17.50
Ag(I), Th(IV), Pb(II)	17.7, 17.4, 17.2	17.43
Mo(VI), Cr(III), Pt(IV)	17.0, 17.6, 17.7	17.43
Hg(II), Cd(II), Ni(II)	17.8, 17.3, 17.5	27.53

To oxidize Fe (II) to Fe (III), 2.0 ml of 30% hydrogen peroxide solution is added and the solution is boiled to decompose the excess of peroxide. The solution is then made up accurately to 100 ml with distilled water.

The aqueous solution was used for determination of iron (III) after elimination of Mn and Ni, as per recommended procedure. The results are given in table.1.

Determination of Iron(III) in synthetic mixtures

The method has been tested on a number of synthetic mixture and the results are shown in table.2

Experimental

The spectral measurements were done on ELICO (CL–27) visible digital spectrophotometer, equipped with 10.01 mm matched pair of glass cuvettes. The pH measurements were done on ELICO (L1-120) digital pH meter, using glass calomel combination electrode. For standardization of pH meter, potassium hydrogen phthalate (pH = 4.01) and borax (pH = 9.18) buffers were used.

Synthesis of RAG

For the synthesis of Res-acetophenone guanylhydrazone (RAG), aminoguanidine bicarbonate was used instead of amino guanidine dihydrochloride, which simplified and gave much better yield.

A solution of amino guanidine bicarbonate, 1.0g in 50% nitric acid and Res- acetophenone, 2.054g in 10ml absolute ethanol were mixed together. The mixture was kept in ice cold water for half an hour. The pale yellow colored product was separated by filtration and crystallized from absolute ethanol ($\sim$2.0g) (percentage = 65.48 % ee) M.P. = $94^0 \pm 1^0$C.

The molecular formula was confirmed on the basis of micro-elemental analysis as $C_9H_{12}N_4O_2$. Calculated percentage of elements was C = 51.92%, H = 5.76% N = 26.92% and O = 15.38%. Experimental percentage of elements was found to be C = 51.37%, H = 5.59% and N = 27.13%. The reagent RAG is soluble in ethyl alcohol, methyl alcohol and acetone but insoluble in benzene, chloroform and carbon tetrachloride.

Scheme

Res-acetophenone   Aminoguanidine   Res-acetophenone guanylhydrazone (RAG)
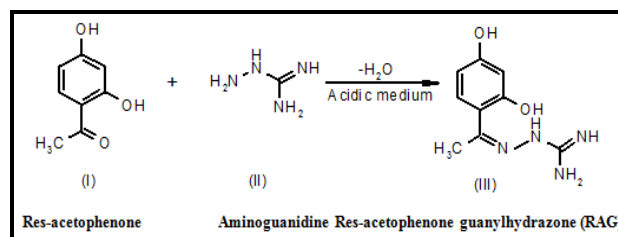
RESULTS AND DISCUSSION

The absorption spectrum of Fe (III)-RAG complex recorded at pH 7.6 shows a peak at 520 nm. At this wavelength, the molar extinction coefficient of the complex is 0.4033 x 10^4 lit. mole^{-1}. cm^{-1} and of the reagent 0.0080 x10^4 lit. mole^{-1}cm^{-1}. About five-fold molar excess of the reagent concentration was sufficient for full color development of the complex. The Beer's law is valid up to 10.0 ppm of Fe (III). Sandell's sensitivity[8] of the method is 0.1164 $\mu g/cm^2$. The composition of the complex was determined by Job's continuous variation method[9] and mole ratio method.[10] These methods indicate the formation of a single complex having the composition 1:2 [Fe (III) : RAG]. The degree of dissociation was obtained by mole ratio method and was found to be 0.1049. The apparent instability constant[11] was found to be 0.1289 x 10^{-8}. To study the effect of diverse ions, Fe (III) was determined in presence of various cations and anions. It is evident that Fe (III), like ions such as Ba (II), Au (III), Ag (I), Bi (III), Co (II), EDTA^{-4} and citrate ions interfere seriously and can be tolerated in presence of appropriate masking agents.[12]

REFERENCES

1. IIlys, S. Q. R. And Joshi, A. P. (1983). *Mikrochim. Acta*. 3, 271.

2. Mishra, V. and Jain, M. C. (1988). *J. Indian Chem. Soc.*, 65, 883.

3. Reddy N. S. R. and Reddy D. V. (1982). *Acta Ciencia Indica, 86*, 66.

4. Goel, D. P., Sengupta, S. and Banerjee, N. R. (1988). *J. Indian Chem. Soc., 65*, 607.

5. Vijaykumar, D., Hussain, R. C. and Appala Raju, N. (1990). *J. Indian Chem. Soc., 67(9),* 786.

6. Deshmukh, M. B. Ambore, D. P. and Joshi, A. P. (1985*). J. Indian Chem. Soc., 62,* 313.

7. Morgan, J. J. and Stumm. W. J. (1965). *Am. Wat. Wks Ass., 57,* p.107.

8. Vogel, A. I. (1968). *A text Book of Quantitative Inorganic Analysis*, 3rd Ed., Longman; p. 264.

9. Sandell, E. B. (1965). *Colorimetric Determination of Traces of metals*, 3rd Edn. Interscience, New York. p.84.

10. Job, P., Compt. Rend., 180, 928 (1925), *Ann.Chem. (Paris), 9,* (1928) p.113.

11. Yoe, J. H. and Jones, A.L. (1944). *Ind. Eng. Chem. Ann. Edd. 16,* p.111.

12. Trikha, K. C., Katyal, M. and Singh, R.P. (1967). *Talanta, 14,* 977.