Research Paper

A Simple and Effective Spectrophotometric Method for the Determination of Iron(III) with Res-Acetophenone Guanylhydrazone (Rag)

Chemistry

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ABSTRACT

We report here a new simple effective spectrophotometric method for the determination of Iron(III) with Res-acetophenone guanylhydrazone [RAG]. A standard procedure of the adsorption spectra of the reagent and the complex is recommended. The absorbance measurements are carried out at 520 nm [Molar extinction co-efficient is 0.4033 x 104 lit. mole cm-1] at pH 7.6. Beer's law is valid up to 10.0 ppm. Sandell's sensitivity of the method is 0.1164 µg/cm2. The Job's variation and mole ratio method show that the composition of Fe(III) RAG complex is 1:2. The instability constant is 0.1289 x10-8. The method is applied for the determination of iron in cupranickel alloy and synthetic mixtures.

INTRODUCTION :

Numerous methods for the spectroscopic determination of Iron have been reported [1-6], but very few are in practice. Res-acetophenone guanylhydrazone (RAG) has been used as sensitive reagent for Fe(III). It forms an red colored complex with iron at pH 7.6, which leads to the development of a simple and rapid spectrophotometric method for the determination of iron at tracer level.

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 filteration and crystallized from absolute ethanol (_2.0g) (percentage = 65.48 % ee) M.P. = 94⁰± 1⁰C.

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:
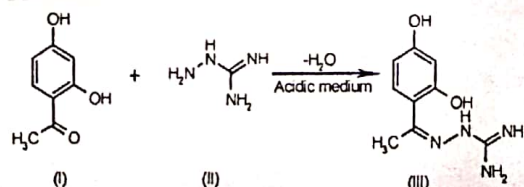
Res-acetophenone Aminoguanidine (RAG)
Res-acetophenone guanylhydrazone

Materials and methods :

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.

Procedure :

A suitable aliquot of the solution containing 50 µg of Fe (III) was taken in a 10ml volumetric flask. To this, was added 2.5 ml of reagent (RAG) solution of concentration (1.0×10^{-2} M) and diluted to 2/3 volume of the flask. Then pH of the solution was adjusted to 7.6 by adding buffer solution and diluted to volume with distilled water. The absorbance of the Fe(III)-RAG complex was measured at 520 nm against reagent blank. The concentration of 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 Fe(III) in the analysis of Cupranickel alloy and synthetic mixtures.

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

A 0.7g alloy sample is treated with 2.0 ml conc.HNO_3 when the reaction is almost over, the mixture is warmed on a hot plate to complete dissolution. 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. To oxidize Fe(II) to Fe(III), 2.0ml 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

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

Sample	Composition %	Iron(III) Found %	Iron(III) X %	Relative standard deviation %
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

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