SIMULTANEOUS SECOND ORDER DERIVATIVE SPECTROPHOTOMETRIC DETERMINATION OF IRON AND BISMUTH IN ALLOYS AND ORES

MOHAN REDDY KPPR*,
*Department of Chemistry, Y.A.Govt.Degree College for women, CHIRALA, Prakasam (Dist).

ABSTRACT

The present paper describes a simple, sensitive and highly selective second order derivative spectrophotometric method for the simultaneous determination of iron (II) and bismuth (III) using 2-hydroxy-3-methoxy benzaldehyde isonicotinoyl hydrazone (HMBAINH) in presence of sodium dodecyl sulphate (SDS). The method does not require separation of the solutes and there is no need to solve the simultaneous equations. Fe (II) reacts with HMBAINH at pH 3.0 forming reddish brown colored solution. At the same pH, Bi (III) also reacts with the reagent forming yellow colored solution. The second order derivative spectrum of Fe(II)-HMBAINH shows maximum derivative amplitude at 433 nm while the Bi(III)-HMBAINH shows zero amplitude. Similarly the Bi(III)-HMBAINH colored solution exhibits maximum derivative amplitude at 500 nm where the Fe(II)-HMBAINH system shows zero amplitude. Therefore, using the zero cross method, Fe (II) and Bi(III) can be determined simultaneously by measuring second order derivative amplitudes at 433 nm and 500 nm respectively. The method is applied for the determination of iron and bismuth in cast iron and brass alloys and in molybdenum and Cu-Pb ores.

KEYWORDS: Derivative Spectrophotometric determination,

INTRODUCTION

Bismuth and iron are important components of number of alloys and ore materials. Bismuth and its alloys are often added to molten iron, steel and aluminium alloys to produce casting and forgings, which can be machined more easily. Good number of derivative spectrophotometric methods is proposed for the simultaneous determination of iron with other metals in binary mixtures is proposed in recent times. But some of these methods involve either extraction into organic solvents or ternary systems. Comparatively very few simultaneous methods for mixtures containing bismuth are available. Some of the direct spectrophotometric methods reported for bismuth are highly sensitive but less selective. Gumus *et.al* has reported a first order derivative spectrophotometric method for the simultaneous determination of Bi (III) and Zn (II) using dithiozone. The only simultaneous derivative method reported in literature for the determination of iron and bismuth was the one proposed by Bermejo-Barrera *et.al* using EDTA. We are now reporting a very simple and effective simultaneous derivative method for the determination of iron and bismuth in binary mixtures using 2-hydroxy-3-methoxy benzaldehyde isonicotinoyl hydrazone (HMBAINH) without solving simultaneous equations.

MATERIALS AND METHODS

Variable amounts of Fe (II) (1-14 µgs) or Bi(III) (4.18-42 µgs) were taken in different 10 ml volumetric flasks and treated with 0.3 ml of HMBAINH (1 x 10^{-2} M), 1.5 ml of SDS (1%) and 5 ml of buffer solution (pH 3.0). The contents were made upto the mark with distilled water and the second order derivative spectra were recorded in the wavelength region 350-600 nm against the reagent blank. The derivative amplitudes were measured at 433 nm for Fe(II) species and at 500 nm for Bi(III) species respectively and plotted against the amount of metal ion. The resultant plots were found to be straight lines with a slope and intercepts as $A_{433} = 0.0907\ C - 0.0016$ for Fe (II) and $A_{500} = 0.0121\ C + 0.0007$ for Bi(III).

RESULTS AND DISCUSSION

Effect of reaction parameters: In order to establish the optimum conditions for the simultaneous determination of Fe(II) and Bi(III), the effect of pH , reagent concentration, surfactant concentration were studied.

The colour intensities of reddish brown and yellow coloured solutions of Fe (II)-HMBAINH and Bi(III)-HMBAINH were found to be maximum and constant in the pH range 2.5 to 5.0. A 15 fold and 30 fold excess of reagent was found to be optimum to get maximum colour intensity with a given amount of Fe (II) and Bi (III) respectively. Among the various surfactants studied, 0.15 % of sodium dodecyl sulphate was found to give a clear experimental solution with maximum colour intensity.

The second order derivative spectra of Fe (II)-HMBAINH and Bi(III)-HMBAINH recorded for variable amounts of metal ions are shown in fig 1. It can be noticed from the figure that maximum derivative amplitudes is observed at 433 nm for Fe (II)-HMBAINH where the Bi (III)-HMBAINH system shows zero amplitude. In the same way the Bi (III)-HMBAINH species exhibits maximum amplitude at 500 nm where the Fe (II)-HMBAINH system has no amplitude. Further, Fe (II)-HMBAINH species obeys Beer's law at 433 nm in the range 0.14 - 1.40 µg mL^{-1} of Fe(II). The Bi (III)-HMBAINH species obeys Beer's law at 500 nm in the range 0.418 – 4.179 µg mL^{-1} of Bi (III). Hence Fe (II) and Bi (III) can be determined simultaneously by measuring the second derivative amplitudes at 433 nm and 500 nm respectively.

Applications: The proposed simultaneous derivative method was applied for the determination of iron and bismuth in cast iron and brass alloys, molybdenum ores and Cu-Pb ores.

Preparation of sample solutions: The cast iron (NBS 33 b) and brass (BCS 41) sample solutions were prepared as per the described procedure. The ore sample solutions were prepared as follows.

The copper-lead and molybdenum ore sample solutions were prepared by decomposing a known amount of ore with hydrochloric acid. 10 ml of 20% (v/v) bromine-CCl_4 solution were added followed by 15 ml of concentrated nitric acid. The solution was allowed to stand for fifteen minutes and then heated gently to remove bromine and CCl_4. 20 ml of 50% H_2SO_4 were added, heated gently until the evolution of nitrogen oxides seizes and then added 5 ml of concentrated hydrochloric acid. The solution was evaporated to fumes of sulfur trioxide and slowly almost to dryness. The residue was redissolved in distilled water and made upto 50 ml with distilled water.

The amount of iron and bismuth present in these alloy and ore samples were determined by the proposed simultaneous derivative method with the help of pre-determined calibration plots and compared with the certified values. The results are presented in table.1.

Table.1.Determination of iron and bismuth in alloys and ores

Sample and composition (%)	Amount of iron (%)		Amount of bismuth (%)	
	Certified	Found*	Certified	Found*
NBS 33b cast iron: C,2.25; Si,2.00; O,0.011; S,0.03; Mn,0.064; Ni,2.25; Cr,0.61; Mo,0.40; Fe,91.7; Bi,0.502	91.7	91.45±0.012	0.502	0.499±0.008
Brass (BCS41): Pb, 2.33; Zn,40.45; Cu,56.57; Fe,0.009; Bi,0.497	0.009	0.0088±0.007	0.497	0.498±0.0017
Mp-1 Zn-Sn-Cu-Pb ore: Zn,16.3; Sn,2.5; Cu,2.2; Pb,1.9; Fe,5.7; As,0.8; Si,19.4; Ca,3.4,Bi,0.025	5.7	5.67±0.009	0.025	0.024±0.006
PR-1 Molybdenum ore: Si,39.2; Fe,1.3; Mo,0.6; Ca,1.4; K,2.0; Bi,0.111	1.3	1.26±0.011	0.111	0.099±0.004

*average of four determinations ± SD

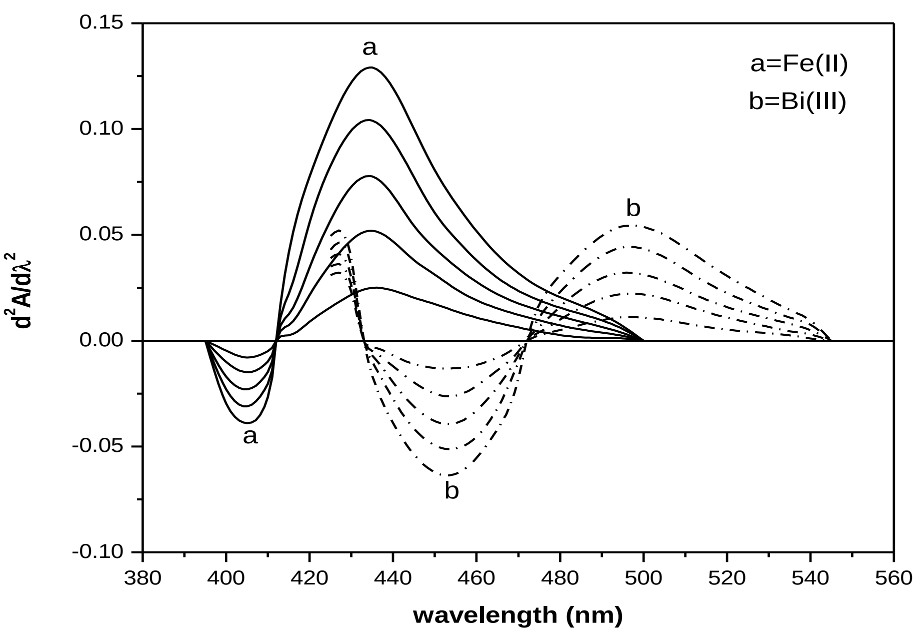

Figure.1. Second order derivative spectra of (a) Fe (II)-HMBAINH and(b) Bi(III)-HMBAINH

Fe(II) (μg mL^{-1}) (Solid line): 0.279; 0.558; 0.838; 1.117; 1.396
Bi(III) (μg mL^{-1}) (Dotted line): 0.836; 1.672; 2.507; 3.343; 4.179

CONCLUSION

The proposed simultaneous derivative method is simple and more effective in the determination of iron and bismuth in complex materials. The method requires neither extraction for the separation of solutes nor needs to solve the simultaneous equations. The zero cross method gives accurate results with good recovery of the metal ions from the alloys and ore samples. Except Bermejo-Barrera *et.al* method, no other simultaneous method is proposed in literature for the simultaneous determination of iron and bismuth till now. In this respect, the present method gains a major importance in the analysis of binary mixtures.

REFERENCES

Bermejo-Barrera, A. Pilar Bermejo-Barrera, Ma. Manuela Guisasola Escudero, Ma. and Francisco Bermajo-Martinez, Analyst, 1987, 112, 481-483.

El-Sayed, A.Y. Fresenius, Journal of Analytical Chemistry, 1996, 355(1), 29-33.

Gumus, G. Filik, H. and Demirata, B. Anal.Chim.Acta, 547(1), 2005, 138-143.

M.I. Toral MI and P. Richter, Simultaneous determination of copper and iron by solvent-extraction and first derivative spectrophotometry, Analytical letters, 28(6), 1995, 1083-1094.

Rauf MA, Ikram M and Ahmed M, spectrophotometric studies of ternary complexes of Vanadyl, Manganese, and Iron with *o*-Phenanthroline and Eosin, Journal of trace and Microbe Techniques, 2002, 20(2), 171-180.

Reddy VK, Reddy SM, Chennaiah A. Raveendra Reddy P and Reddy, TS, Journal of Analytical Chemistry, 2003, 58(5), 442-446.