Available online www.jocpr.com

Journal of Chemical and Pharmaceutical Research, 2016, 8(8):360-363

Research Article

ISSN : 0975-7384
CODEN(USA) : JCPRC5

Simultaneous Derivative Spectrophotometric Determination of Indium (III) and Iron (II)

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ABSTRACT

A simultaneous second order derivative spectrophotometric method was developed for the determination of indium (III) and iron (II) using 2-hydroxy-3-methoxy benzaldehyde thiosemicarbazone (HMBATSC) as a chromophoric reagent. The reagent reacts with indium (III) and iron (II) at pH 6.0. In (III) and Fe (II) present in the mixture were simultaneously determined without solving the simultaneous equations by measuring the second order derivative amplitudes at 428 nm and 407 nm respectively. The derivative amplitudes obey Beer's law in the range 0.131-4.592 μgml^{-1} of In (III) and 0.109-3.965 μgml^{-1} of Fe (II). Large number of foreign ions does not interfere in the present method. The present simultaneous method was used for the determination of micro amounts of indium (III) and iron (II) in synthetic mixtures.

Keywords: In (III) and Fe (II), Spectrophotometry, 2-hydroxy-3-methoxy benzaldehyde thiosemicarbazone (HMBATSC)

INTRODUCTION

Indium is a soft, silvery, white metal which looks like zinc, but it is chemically similar to aluminum and gallium. Pure indium in metal form is considered to be toxic. Indium in very small amounts is used in aluminum alloys which act as sacrificial anodes to prevent passivation of aluminum. It is used in fusible alloys, solders and also as a dopant for semiconductors.

Iron (II) is a necessary trace element used by almost all living organisms. The only exceptions are organisms that live in iron poor environments and have evolved to use different elements in their metabolic process, such as manganese instead of iron for catalysis instead of hemoglobin. Iron containing enzymes usually containing hemoprosthetic groups; participate in the catalysis of oxidation reactions in biology and in transport of a number of soluble gases. Iron is of most importance when mixed with certain other metals and with carbon to form steels. There are many types of steels, all with different properties, and an understanding of the properties of the allotropes of iron is the key to the manufacture of good quality steels.

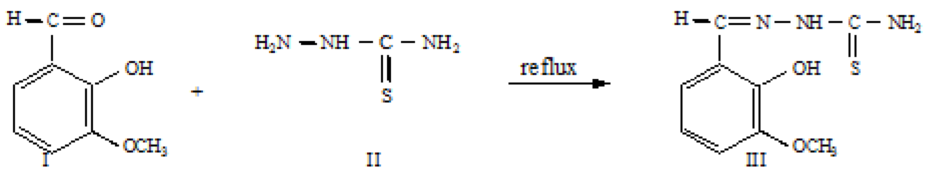
Literature search suggests several techniques such as X-ray fluorescence, atomic absorption spectrometric, atomic fluorescence spectrometric, electrochemical, chromatographic, etc. have been published for the individual determinations of indium and iron in different samples. However, very rare methods are available for the simultaneous determination of In (III) and Fe (II). UV-Vis spectrophotometric analytical procedures are most widely used for the simultaneous determination of metals. The obvious reasons being experimental simplicity, rapidity, and the wide applicability of these procedures. However, in many cases traditional spectrophotometric techniques are not suitable for simultaneous determination, because the absorption spectra overlap and are not suitable for simultaneous quantitative analysis. In particular derivative spectrophotometry has been extremely useful analytical technique for the simultaneous determination of binary mixtures. In this work, it was aimed to develop a simple and sensitive determination of indium (III) and iron (II) by second order derivative spectrophotometery [1-13].

The present work describes 2-hydroxy-3-methoxybenzaldehyde thiosemicarbazone (HMBATSC) as a chromophoric reagent for a simple, sensitive and selective determination of indium (III) and iron (II) by second order derivative spectrophotometry. The reagent HMBATSC has been used to develop determination of metal ions individually and also simultaneously. The simplicity and low operating costs of spectrophotometric methods made spectrophotometry as an attractive alternative technique for the determination of metal ions in different matrices.

EXPERIMENTAL SECTION

A shimadzu UV-visible spectrophotometer (model UV-160A) fitted with 1cm quartz cells and slit width of 2mm was used to measure absorbance of all analytical species. All spectral measurements were performed using the blank solution as a reference measurement of pH was carried on a Phillips digital pH meter (model LI 613).

The reagent (HMBATSC) is prepared by sah and daniels [14] procedure. 11.25gms of 2-hydroxy-3-methoxy benzaldehyde (I) and 4.55gms of thiosemicarbazide (II) are dissolved in sufficient volume of methanol and the mixture refluxed for 60minutes. The contents are allowed to cool and the product separated by filtration. A crude sample (yield 80%) is obtained ($C_9H_{11}O_2SN_3$). The resultant product is recrystallized twice from hot methanol. Pure light yellowish green crystals of 2-hydroxy-3-methoxy benzaldehyde thiosemicarbazone (III) with melting point 220-225^{0}C are obtained.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A 0.01 M solution of HMBATSC in dimethylformamide (DMF) was employed in the present studies. Stock solution (0.01M) of indium (III) was prepared by dissolving 0.5178gms of indium Sulphate in 2ml of 2M H_2SO_4 and diluting to 100ml with distilled water. This solution was standardized by using xelenol orange as indicator [15]. Stock solution of ferrous ammonium sulphate was dissolved in distilled water containing few drops of concentrated H_2SO_4 and made up to the mark in a 250ml volumetric flask to get 0.1M solution. The resulting solution was standardized [16] .Working solutions were prepared by diluting appropriate volume of this stock solution with distilled water.

Procedure

Various known aliquots of solutions containing of 0.131-4.592 µgml^{-1} of In (III) and 0.109-3.965 µgml^{-1} of Fe (II) were taken in various 10ml volumetric flasks each containing 5ml of buffer solution of the selected pH. The contents of each flask were made up to the mark with distilled water and the derivative spectra of these solutions were recorded against reagent blank in the wavelength 380-520nm with scan speed fast and with suitable degrees of freedom. Derivative amplitudes were measured at 428nm of In (III) and 407nm of Fe (II) against the concentrations of metal ions. The slope and intercept of the plots and Beer's law range were evaluated.

RESULTS AND DISCUSSION

The second order derivative spectra of In (III)-HMBATSC and Fe (II)-HMBATSC species with different amounts of metal ions are shown in Fig 1. It can be noticed in the figure that [In (III)-HMBATSC] show considerably large derivative amplitude at 428nm and zero amplitude at 407nm and [Fe(II)-HMBATSC] complex gives sufficient amplitude at 407nm and zero amplitude at 428nm. Hence In (III) and Fe (II) were simultaneously measured by second derivative amplitudes at 428nm and 407nm respectively.

The calibration plots drawn between the amount of indium with the derivative amplitudes at 428nm and the amount of iron with the derivative amplitudes measured at 407nm .The plots indicate that Beer's law is obeyed in the range 0.131-4.592 µgml^{-1} of In (III) and 0.109-3.965 µgml^{-1} of Fe (II) . The high values of correlation coefficients and closeness of intercepts to zero indicate the good linearity of the calibration plots and conformity to Beer's law. The amounts of indium and iron present in the mixtures were computed from the measured amplitudes with the help of predetermined calibration graph and the results are given in table 1. The analytical characteristics derived both for In(III) and Fe(II) in their simultaneous determination are compared and tabulated in table 2.

Table 1: Simultaneous second order derivative spectrophotometric determination of In (III) and Fe (II)

Amount taken* (μgml^{-1})		Amount found* (μgml^{-1})		Relative error (%)	
In (III)	Fe(II)	In(III)	Fe (II)	In (III)	Fe (II)
0.574	0.279	0.576	0.278	-0.35	+0.40
0.574	0.558	0.572	0.560	+0.35	-0.35
0.574	0.837	0.573	0.835	+0.23	+0.23
0.574	0.279	0.575	0.280	-0.35	-0.35
1.148	0.279	1.143	0.277	+0.43	+0.71
2.296	0.279	2.294	0.281	+0.09	-0.72

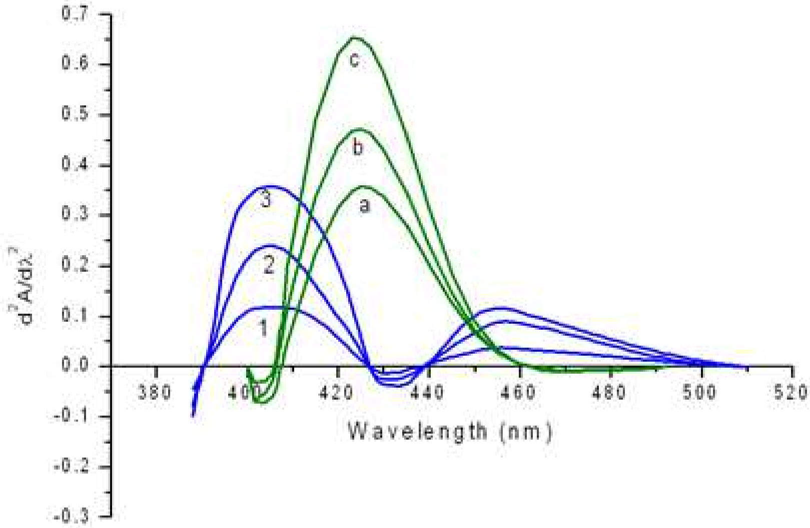

Figure 1: Second Derivative Spectra of In (III) – HMBATSC (Green lines) and Fe (II) – HMBATSC (Blue lines) at Different Concentrations

a) [In (III) ($\mu g\ ml^{-1}$) : 1) 0.574 2) 1.148 3) 2.296; b) [Fe (II) ($\mu g\ ml^{-1}$) : 1) 0.279 2) 0.558 3) 0.837

Applications

A solution of synthetic mixture containing 2mg of indium (III) and iron (II) were taken and different known aliquots of other metal ions were prepared. Determination of indium (III) and iron (II) in the solution was carried out simultaneously by using the proposal method. The results obtained were in good agreement with the amounts added. The results obtained are presented in table 3

Table 2: Analytical characteristics of In(III) and Fe(II)

Parameter	In(III)	Fe(II)
Zero cross wavelength(nm)	407	428
Analytical wavelength measured	428	407
Beer's law range ($\mu g\ ml^{-1}$)	0.131-4.592	0.109-3.965
Angular coefficient	0.3057	0.3903
Y-intercept	0.0013	0.0056
Correlation coefficient (r)	0.9997	0.9995
Standard deviation (s)	0.0113	0.0068

Table 3: Determination of Indium and Iron in synthetic mixtures

Synthetic Mixture (μgml^{-1})	Amounts taken (μgml^{-1})		Amount found (μgml^{-1})		Recovery (%)		Relative error (%)	
	In(III)	Fe(II)	In(III)	Fe(II)	In(III)	Fe(II)	In(III)	Fe(II)
In(III) (2.0) Sn(IV) (2.0) Cd(II) (2.0) Fe(II) (2.0)	2.0	2.0	1.98	1.97	99.0	98.5	-1.0	-1.5
In(III) (2.0) Sn(IV) (2.0) Pb (II) (2.0) Fe(II) (2.0)	2.0	2.0	1.98	1.98	99.0	99.0	-1.0	-1.0
In(III) (2.0) Sn(IV) (2.0) Sb (IV) (2.0) Fe(II) (2.0)	2.0	2.0	1.97	1.96	98.5	98.0	-1.5	-2.0

CONCLUSION

The present method is simple, sensitive, and highly selective for the simultaneous determination of In (III) and Fe (II) in admixtures without separation and without solving simultaneous equations. The developed method was applied for determination of In (III) and Fe (II) in synthetic mixtures.

Acknowledgement
The authors thank the department of Chemistry of S.K. University Anantapuramu and KSRM College of Engineering, Kadapa for providing necessary facilities.

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