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SIMULTANEOUS DERIVATIVE SPECTROPHOTOMETRIC DETERMINATION OF CHROMIUM (VI) AND INDIUM (III)

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Abstract

A simultaneous second order derivative spectrophotometric method was developed for the determination of chromium (VI) and indium (III) using 2-hydroxy-3-methoxy benzaldehyde thiosemicarbazone (HMBATSC) as a chromophoric reagent. The reagent reacts with chromium(VI) and indium (III) forming intense green coloured solution at pH 6.0. Cr (VI) and In(III) present in the mixture were simultaneously determined without solving the simultaneous equations by measuring the second order derivative amplitudes at 407nm and 430nm respectively. The derivative amplitudes obey Beer's law in the range 0.143-3.44 μgml^{-1} of Cr (VI) and 0.131-3.413 μgml^{-1} of In (III). 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 chromium (VI) and indium (III) in synthetic mixtures.

Keywords: HMBATSC;Simultaneous determination;Cr(VI) and In(III);Derivative spectrophotometry

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1. Introduction

Hexavalent chromium compounds are geotaxis carcinogens. According to some researchers the damage is caused by hydroxyl radicals, produced during reoxidation of pentavalent chromium by hydrogen peroxide molecules present in the cell. Soluble compounds like chromic acid are much weaker carcinogens. Chromium (VI) compounds are much less toxic than chromium (VI) compounds. Low doses of Cr (VI) combined with vitamin 'C' produce up to 15 times as many chromosomal breaks and up to 10 times more mutations, compared with cells lacking vitamin C. Outside the cells, vitamin C actually protects against the cellular damage caused by hexavalent chromium.

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.

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 chromium and indium in different samples. However, very rare methods are available for the simultaneous determination of Cr(VI) and In(III). 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 analyses. 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 chromium (VI) and indium (III) by second order derivative spectrophotometry [1-13].

The present work describes 2-hydroxy-3-methoxybenzaldehyde thiosemicarbazone (HMBATSC) as a chromophoric reagent for a simple, sensitive and selective determination of

chromium (VI) and indium (III) 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.

2. Research Method

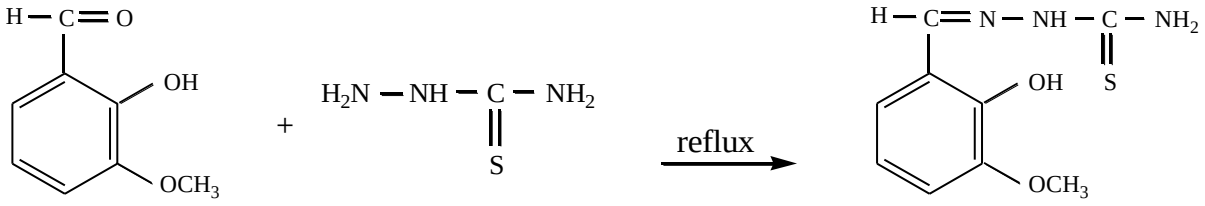
2.1 Apparatus

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 mete r(model LI 613)

2.2 Chemicals and solutions

Preparation of 2-hydroxy-3-methoxy benzaldehyde thiosemicarbazone (HMBATSC)

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.



I II III

II III

A 0.01 M solution of HMBATSC in dimethylformamide (DMF) was employed in the present studies.

Stock solution (0.01M) of chromium (VI) was prepared by dissolving 0.194gms of potassium Chromate in 100ml of distilled water and standardized [15].

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 [16].

Working solutions were prepared by diluting appropriate volume of this stock solution with Distilled water.

2.3 Procedure

Various known aliquots solutions containing of 0.143-3.44 μgml^{-1} of Cr (VI) and 0.131-3.413 μgml^{-1} of In(III) 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 350-600nm with scan speed fast and with suitable degrees of freedom. Derivative amplitudes were measured at 407nm of Cr (VI) and 430nm of In(III) against the concentrations of metal ions. The slope and intercept of the plots and Beer's law range were evaluated.

3. Results and Analysis

The second order derivative spectra of Cr (VI)-HMBATSC and In(III)-HMBATSC species with different amounts of metal ions are shown in figure 1. It can be noticed in the figure that [Cr(VI)-HMBATSC] show considerably large derivative amplitude at 407nm and zero amplitude at 430nm and [In(III)-HMBATSC] complex gives sufficient amplitude at 430nm and zero amplitude at 407nm.Hence Cr(VI) and In(III) were simultaneously measured by second derivative amplitudes at 407nm and 430nm respectively.

The calibration plots drawn between the amount of chromium with the derivative amplitudes at 407nm and the amount of indium with the derivative amplitudes measured at 430nm. The plots indicate that Beer's la is obeyed in the range 0.143-3.44 μgml^{-1} of Cr (VI) and 0.131-3.413 μgml^{-1} of In(III). 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 results obtained are shown in table 1.

Table 1 : **Simultaneous second order derivative spectrophotometric determination of Cr(VI) and In(III).**

Amount taken* (μgml^{-1})		**Amount found* (μgml^{-1})**		**Relative error (%)**	
Cr(VI)	In(III)	Cr(VI)	In(III)	Cr(VI)	In(III)
0.574	0.650	0.572	0.653	+0.34	-0.46
0.574	1.300	0.572	1.304	-0.34	-0.30
0.574	1.950	0.575	1.952	-0.17	-0.10
0.574	0.650	0.573	0.647	+0.17	+0.46
1.148	0.650	1.142	0.652	+0.52	-0.30
1.956	0.650	1.954	0.648	+0.10	+0.30

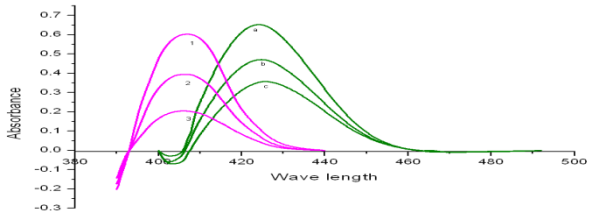


Figure 1: Second Derivative Spectra of Cr(IV) – HMBATSC (Pink lines) And In (III) – HMBATSC (Green lines) at Different Concentrations

a) [Cr (VI)] ($\mu g\ ml^{-1}$): 1) 0.65 2) 1.30 3) 1.95

b) [In (III) ($\mu g\ ml^{-1}$): 1) 0.574 2) 1.148 3) 2.296

3.1 Applications

A solution of synthetic mixture containing 20µg of chromium (VI) and indium(III) were taken and different known aliquots of other metal ions was prepared. Determination of chromium (VI) and indium(III) 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 2

Table 2: Determination of chromium and Indium in synthetic mixtures

Synthetic Mixture (μgml^{-1})	**Amounts taken (μgml^{-1})**		**Amount found (μgml^{-1})**		**Recovery (%)**		**Relative error(%)**	
	Cr(VI)	**In(III)**	**Cr(VI)**	**In(III)**	**Cr(VI)**	**In(III)**	**Cr(VI)**	**In(III)**
Cr(VI)-20 Pb(II)-25 Ga(III)-10 In(III)-20 Sn(IV)-25	20	20	19.85	19.91	99.25	99.25	-0.75	-0.45

4. Conclusion

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

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