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Second order derivative spectrophotometry for simultaneous determination of Hg(II) and Ti(IV) using 3,4-dihydroxybenzaldehyde isonicotinoylhydrazone (DHBINH)

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A B S T R A C T

A second order derivative spectrophotometric method is developed for the simultaneous determination of mercury (II) and titanium (IV) using 3, 4-DHBINH without any prior separation. Beer's law is obeyed between 4.014 to 24.06 µg/ml of mercury (II) at 375 nm and 0.949 to 5.724 µg/ml of titanium (IV) at 360 nm. The metal ion interfere with the determination of each other in zero order as well as in first order spectrophotometry. Zero crossing wavelengths are 363 and 372 nm. The optimum condition for maximum color development and other analytical parameters were evaluated.

Keywords: 3, 4-DHBINH, Hg (II), Ti (IV), second order spectrophotometry, Mercury, Wavelength, & Beer's law

A R T I C L E I N F O

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1. Introduction

Simultaneous determination of metal ions using derivative spectrophotometry decreases the interference of foreign ions. This method is particularly advantageous in the case of metal ions having overlapping spectra. Number of reports 1-18 is available in recent years on derivative spectrophotometry for the simultaneous determination of metal ions. 2,4-dihydroxybenzaldehyde isonicotinoyl hydrazine 19 and Salicylaldehyde p-hydroxy benzoyl hydrazone20 in the presence of Triton X-100 are used for the simultaneous determination of Mo(VI) and Ti(IV).

Methods for the simultaneous determination of metal ions on derivative spectrophotometry using 3, 4-DHBINH is not available. A second order derivative spectrophotometric method has been developed for the simultaneous determination of mercury (II) and titanium (IV) using 3,4-DHBINH and results are presented in this paper.

2. Materials and Methods

U.V visible recording spectrophotometer (UV – 160A):

A Shimadzu UV-Visible Spectrophotometer (UV-160A) a equipped with 1.0 cm quartz cells was used for allteh spectral measurements. The most common instrumental parameters that usually affect the shape of the derivative spectra are the scan speed, slit width and the value of the Δλ. These parameters were optimized and the best results were obtained with a scan speed 145 nm/min, slit width of 1 nm and Δλ=2 nm for second order derivative mode in the wavelength range 350 – 500 nm. ELICO LI 160 digital pH meter were used for the absorbance and pH measurements respectively.

Reagents:

They were prepared by mixing 1M HCL and 1M sodium acetate (pH 1.0 – 3.0) and 0.2M acetic acid and 0.2M sodium acetate (pH 3.5 – 7.0) in various proportions. The pH of these solutions was adjusted to their appropriate values using pH meter. Stock solutions of 0.01M of Hg (II) and Ti (IV) were prepared by dissolving requisite amounts of analytical grade Hgcl2 and Na2WO4 in distilled water and standardized. Lower concentrations were prepared by appropriate dilution of the stock solutions. All other chemicals used were of analytical grade.

Synthesis of 3, 4-DHBINH:

This was prepared by taking equimolar solutions of 3,4-dihydroxybenzaldehyde and isonicotinic acid hydrazide in methanol and refluxing for 3 hrs. It was allowed to stand at room temperature until the yellow color crystals were formed. A 0.01M solution of the reagent in DMF was used in the studies.

General method:

2 ml of reagent (2 x 10-2M), 10 ml of buffer solution of pH 6.0, were taken in 25 ml volumetric flask, known aliquots of Hg(II) and Ti(IV) were added and made upto the mark with distilled water. Second derivative spectra of these solutions were recorded against reagent blank solution in the wavelength range 350 to 500 nm with scan speed 145 nm/min of the derivative peak height were measured by peak zero method at 362 nm and 374 nm for Hg(II) and Ti(IV). Calibration plots were drawn for the experimental data and they are shown in figures 3 and 4.

Analytical parameters and sensitivity:

The molar absorptivity and Sandell's sensitivity values of Ti(IV) -3,4-DHBINH and Hg(II)-3,4-DHBINH species calculated from Beer's law data were 0.2816 x 104 L.mol-1cm-1, 0.816 and 0.883 x 103 L.mol-1cm-1.

3. Results and discussion

Simultaneous determination of Ti(IV) and Hg(II):

Zero order spectra of Ti(IV) and Hg(II) are recorded in a solution of pH 6.0 at 420 and 440 nm respectively. When a simultaneous zero order spectrum is recorded only a single peak is noticed at 390 nm. Hence, zero order is not useful for simultaneous determination of metal ions. The first order derivative spectra are not useful because they are broader in shape and resolution is not possible. The second order spectrum recorded for experimental solution against the respective blank solution is presented in fig. 1. An examination of figure reveals that there are two peaks. The peak at 375 nm corresponds to mercury (II) and the other at 360 nm corresponds to titanium (IV). The peak at 375 nm and the valley at 380 nm corresponds to Hg (II). Similarly the peak at 360 nm and the valley at 367 nm corresponds to Ti (IV). Typical second order derivative spectrum is shown in fig. 2. To know the sensitivity of the method for simultaneous determination graphs are drawn between the concentration of metal ion and peak amplitude, valley amplitude and sum of peak and valley amplitudes are shown in Fig 3 and 4 for Hg(II) and Ti(IV) respectively.

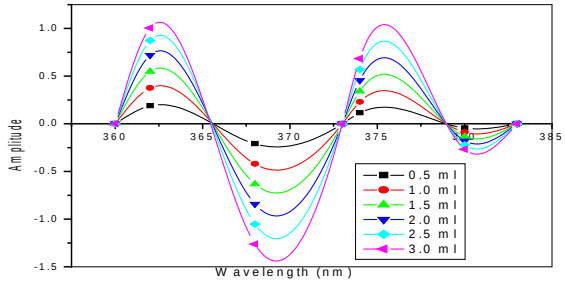

Fig 2: Second order spectra of Pd (II) + W (VI) in presence of 3,4-DHBINH [3,4-DHBINH] = 2 x 10-4M; [Pd (II)] = [W (VI)] = 1 x 10-5M; pH = 6.0

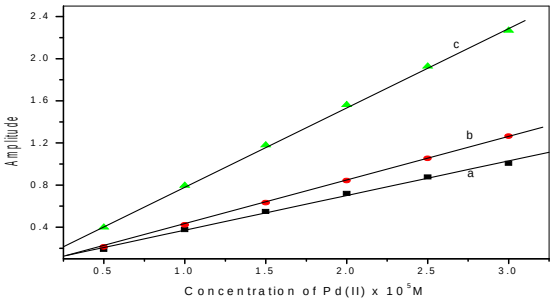

Fig 3: Second derivative amplitude Vs Concentration of Pd (II) Wavelength = 362; pH = 6.0 a = Peak; b = Valley; c = Peak + Valley

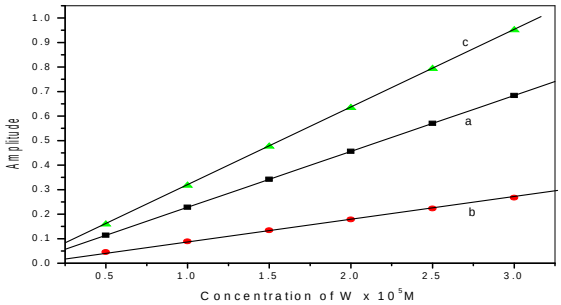

Fig 4: Second derivative amplitude Vs Concentration of W (VI) Wavelength = 374; pH = 6.0 a = Peak; b = Valley; c = Peak + Valley

4. Conclusion

Several reagents are used as photometric reagents for the simultaneous determination of mercury (II) and titanium (IV) using 3, 4-DHBINH without any prior separation. The metal ion interferes with the determination of each other in zero order as well as in first order spectrophotometry. The present method does not need solving of simultaneous equations. The present method is rapid sensitive and highly selective. Hence, it can be applied successfully to their analysis in steel samples.

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