

# International Journal of Chemistry and Pharmaceutical Sciences



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# **RESEARCH ARTICLE**

# Second order derivative spectrophotometry for simultaneous determination of Pd(II) and W(VI) using 3,4-dihydroxybenzaldehyde isonicotinoyl hydrazone (DHBINH)

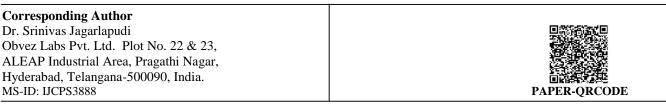
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## ABSTRACT

A second order derivative spectrophotometric method is developed for the simultaneous determination of palladium (II) and tungsten (VI) using 3,4-DHBINH without any prior separation. Beer's law is obeyed between 0.2128 to 3.192  $\mu$ g/ml of palladium (II) at 362 nm and 0.3676 to 5.514  $\mu$ g/ml of tungsten (VI) at 374 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 366 and 373 nm. The optimum condition for maximum color development and other analytical parameters were evaluated. **Keywords:** *3,4-DHBINH, Pd(II), W(VI), second order spectrophotometry &Wavelength etc.* 

# ARTICLE INFO



A R T I C L E H I S T O R Y: Received 11 January 2019, Accepted 25 February 2019, Available Online 27 March 2019

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**Citation:** Srinivas Jagarlapudi. Second order derivative spectrophotometry for simultaneous determination of Pd(II) and W(VI) using 3,4-dihydroxybenzaldehyde isonicotinoyl hydrazone (DHBINH). Int. J. Chem, Pharm, Sci., 2019, 7(3): 64-66.

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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. Numbers of reports<sup>1-18</sup> are available in recent years on derivative spectrophotometry for the simultaneous determination of metal ions. 2,4-dihydroxybenzaldehyde isonicotinoyl International Journal of Chemistry and Pharmaceutical Sciences

hydrazone<sup>19</sup> and Salicylaldehyde p-hydroxybenzoyl hydrazone<sup>20</sup> 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 are not available. A second order derivative spectrophotometric method has been developed for the simultaneous determination of palladium (II) and tungsten(VI) using 3,4-DHBINH and results are presented in this paper.

#### 2. Experimental

#### Apparatus

#### U.V visible recording spectrophotometer (UV - 160A)

A Shimadzu UV-Visible Spectrophotometer (UV-160A) and ELICO LI 160 digital pH meter were used for the absorbance and pH measurements respectively.

#### Reagents

#### 3,4- di hydroxy benzaldehyde isonicotinoyl hydrazone

This was prepared by taking equimolar solutions of 3,4dihydroxybenzaldehyde and iso nicotinic 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.

#### **Buffer solutions**

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.

#### Palladium (II) and tungsten (VI) solution

Stock solutions of 0.01M of Pd(II) and W(VI) were prepared by dissolving requisite amounts of analytical grade PdCl<sub>2</sub>,  $Na_2WO_4$  in distilled water and standardized. Lower concentrations were prepared by appropriate dilution of the stock solutions. All other chemicals used were of analytical grade.

#### Procedure

#### Simultaneous determination of Pd(II) and W(VI)

2 ml of reagent ( $2 \times 10^{-2}$ M), 10 ml of buffer solution of pH 5.5, were taken in 25 ml volumetric flask, known aliquots of Pd(II) and W(VI) 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 of the derivative peak height were measured by peak zero method at 362 nm and 374 nm for Pd(II) and W(VI). Calibration plots were drawn for the experimental data and they are shown in figures 3 and 4.

#### 3. Results and Discussions

Zero order spectra of W(VI) and Pd(II) are recorded in a solution of pH 5.5 at 390 and 395 nm respectively. When a simultaneous zero order spectrum is recorded only a single peak is noticed at 400 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 362 nm corresponds to palladium (II) and the other at 374 nm corresponds to tungsten (VI). The peak at 362 nm and the valley at 369 nm corresponds to Pd(II). Similarly the peak at 374 nm and the valley at 381 nm corresponds to W(VI). Typical second order derivative

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#### CODEN (USA): IJCPNH | ISSN: 2321-3132

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 Pd(II) and W(VI) respectively.

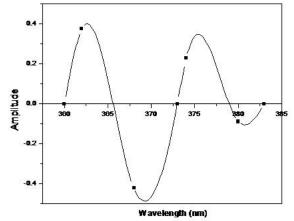


Fig. 1.Typical second order derivative spectrum of Pd (II) + W (VI) in presence of 3,4-DHBINH  $[3,4-DHBINH] = 2 \times 10^{-4}$ M; [Pd (II)] = [W (VI)] = 1 x 10<sup>-5</sup>M; pH = 5.5

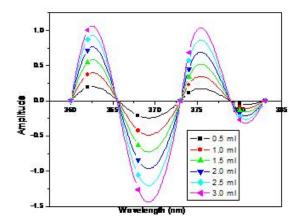
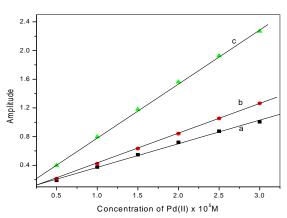
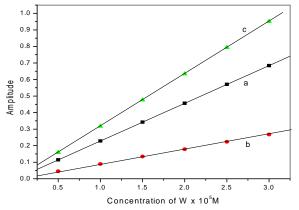


Fig. 2. Second order spectra of Pd (II) + W (VI) in presence of 3,4-DHBINH  $[3, 4-DHBINH] = 2 \times 10^{-4}M;$  $[Pd (II)] = [W (VI)] = 1 \times 10^{-5}M; pH = 5.5$ 



**Fig. 3.** Second derivative amplitude Vs Concentration of Pd (II) Wavelength = 362; pH = 5.5 a = Peak ; b = Valley ; c = Peak + Valley



**Fig. 4.**Second derivative amplitude Vs Concentration of W (VI) Wavelength = 374; pH = 5.5; a = Peak; b = Valley; c = Peak + Valley

#### 4. Conclusion

Several reagents are used as photometric reagents for the simultaneous determination of Pd(II) and W(VI) using 3,4-DHBINH without any prior separation. Beer's law is obeyed between 0.2128 to  $3.192 \mu g/ml$  of palladium (II) at 362 nm and 0.3676 to  $5.514 \mu g/ml$  of tungsten (VI) at 374 nm. 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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