

## 3-Hydroxy-3-Ethyl-1-o-Carboxy Phenyl Triazene: A New Reagent for Spectrophotometric Determination of Palladium

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### ABSTRACT

The Reagent (in six fold molar excess) reacts with Palladium (II) and forms a yellow complex instantaneously at room temperature. The complex exhibits  $\lambda_{max}$  at 368 nm and all the estimation were made at this wave length. The pH range of constant maximum absorbance was found between 2.0 to 2.8. Using Job's method, Mole ratio method and slope ratio method the molar composition of palladium complex with the reagent is found as 1:1. The molar absorptivity and sandell's sensitivity at 368nm are 8000 Lit. mole<sup>-1</sup> cm<sup>-1</sup> and 13.30ng per cm<sup>2</sup>. The stability constant of the complex was determined by Harvey and Manning's method (using mole ratio curve) and Purohit's method<sup>2</sup> (using Job's curve). Using the above methods the value of log  $\beta$  were found as 6.748 and 6.551. Interference studies have also been made

**Key words:** 3- Hydroxy-3-Ethyl-1-o-Carboxy Phenyl Triazene, Palladium, Molar absorptivity

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### I. EXPERIMENTAL

#### Preparation of Reagent Solution

A stock solution of  $5 \times 10^{-4}$  M was prepared by dissolving the required amount of reagent in ethanol. By appropriate dilution of the stock solution with ethanol solution of desired lower concentration were prepared

#### Preparation of Pd(II) solution

$1 \times 10^{-2}$  M solution of palladium chloride was prepared by dissolving the required quantity of palladium chloride in 2 ml. Hot conc. HCl and diluting to 250 ml with distilled water.

#### Composition of the Complex

The composition of the complex was found to be methods 1:1 (Pd:R) with the help of job's method<sup>3</sup> mole ratio method of yoe and jones<sup>4</sup>, Zolotov's mole ratio method<sup>5</sup> and slop ratio method<sup>6</sup>. Further slope ratio method of course given same composition 1:1 but when metal is present in excess the absorbance in more then expected similiarly Zolotov's mole ratio method also constant maximum absorbance value is higher than expected value.

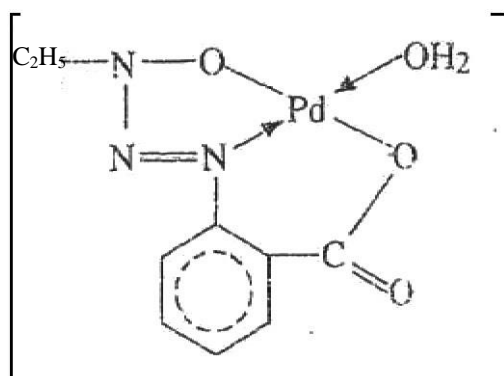
#### Preparation of Solid Complex

The molar proportion of palladium chloride dissolved in minimum of conc. HCl and reagent solution in alcohol at desired pH are mixed and solid complex was obtained. The yellowish orange complex has melting point 205°C and molecular formula Pd(C<sub>9</sub>H<sub>9</sub>N<sub>3</sub>O<sub>3</sub>)H<sub>2</sub>O as revealed by elemental analysis.

	%C	%H	%N	Pd
Theroretical	52.60	3.34	12.67	32.09
Experimental	32.84	3.28	12.60	32.45

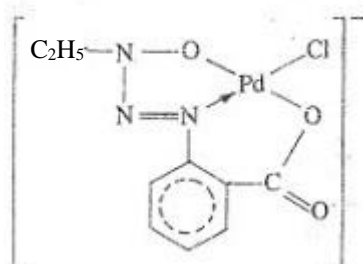
## II. Results & Discussion

The ligand has presence of carboxy group in ortho position on the 1 - Phenyl ring and would act as dibasic tridentate ligand. The 1:1 palladium complex may tentatively be assigned following square planar structure.



The presence of water molecule is indicated by elemental analysis in solid complex. In case of slope ratio method & zolotov's mole ratio method the absorbance is more than expected when metal is present in excess with increase in palladium conc. HCl conc. also increases. It is likely that H<sub>2</sub>O present is replaced partially or wholly by chloro group giving another species.

Might be this negatively changed species has higher absorbance as compared to aquo species.



$\lambda$  max working wave length and optimum pH for palladium complex

$\lambda$ max (nm)	Wave length (nm)	pH of constant absorbance
368	374	2.0 to 2.8

Stability constant of Pd complex with 3 - Hydroxy - 3- Ethyl - 1-0- Carboxy phenyl triazene

Composition of complex	Long $\beta$ from Harvey & manning method	Long $\beta$ from Job's curves
Pd : R 1:1	6.748	6.551

IR spectra of the reagent 3-hydroxy -3-Ethyl- 1-o-Carboxy phenyl Triazene shows presence of  $\nu$

$\nu_{\text{NH}}$ cm <sup>-1</sup>	$\delta_{\text{NOH}}$ cm <sup>-1</sup>	$\delta_{\text{NH}}$ cm <sup>-1</sup>	$\delta_{\text{OH}}$ cm <sup>-1</sup> -COOH	
3100w	1090 s	1500 s	3220 w	

On complexation both hydrogen ions on - N-OH and - COOH group are removed  $\nu_{\text{OH}}$  of - COOH should also disappear

New  $\nu_{\text{OH}}$  water band in complex appears at 3175 cm<sup>-1</sup>

$\nu_{\text{C}}=\text{O}$  in ligand - 1680 cm<sup>-1</sup> (s)

$\nu_{\text{C}}=\text{O}$  in ligand - 1625 cm<sup>-1</sup> (w)

**Reference**

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