



SYNTHESIS AND SPECTRAL STUDIES OF 1-(2'-HYDROXY 5'METHYL PHENYL)-3-PHENYL PROPANE-1, 3-DIONE WITH FE (III) IN AQUO-ORGANIC MEDIA

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ABSTRACT:

In the spectral study, the complexes of 1-(2'-hydroxy 5'methyl Phenyl)-3-Phenyl Propane-1, 3- dione (β -diketone) with Fe (III) cations were synthesized and their visible spectrum were recorded. The maximum wavelength of absorption was derived from the recorded spectrum. The stability constants of these complexes were determined by UV-Vis spectrometry. In view of the biological, clinical, biochemical, application of 1,3-dione in the synthesis of drugs as reagent in quantitative analysis, few system were studied using spectrophotometric measurements. Spontaneous wine red colour formation by adding the chloro methyl or anisyl substituted β -diketone in Fe (III) ion solution even at pH less than 2.0, promoted to investigate the behaviour of these complexes in a wide range of pH, conc. μ and temperature. Absorption spectra of various complex solution with varying (M: L) composition and at various condition are recorded in range of 400nm to 800nm.

KEYWORDS:

Stability constant. , Slope Ratio method, Fe (III) Metal ion, K_{cond} etc.

INTRODUCTION

In view of the application of 1, 3-dione in the synthesis of drugs as areagent in quntitative analysis, few system were studied using spectrophotometric measurements. Spontaneous wine red colour formation by adding the chloro methyl or anisyl substituted β -diketone in Fe(III) ion solution even at pH less than 2.0,promoted to investigate the behaviour of these complex in a wide range of pH,conc., μ and temperature .Absorption spectra of various complex solution with varying (M:L) composition and at various condition are recorded in range of 400nm to 800nm.Some worker reporting some formation constant of FeIII some drugs¹. Spectrometric determination of Iron III with ligand and confermation of composition of its complex have studied²⁻³. Some data calculated of iron in water

sample⁴. Susaki ,Yoshiro and co worker study some characterization of Iron II with β -diketones⁵.Ronald Gene Rowland ,study on Chelation of transition metal ion with incotinyldoxamic acid⁶.Cuba M.R.,Ramirez studied on mixed ligand Iron complex⁷⁻⁹.

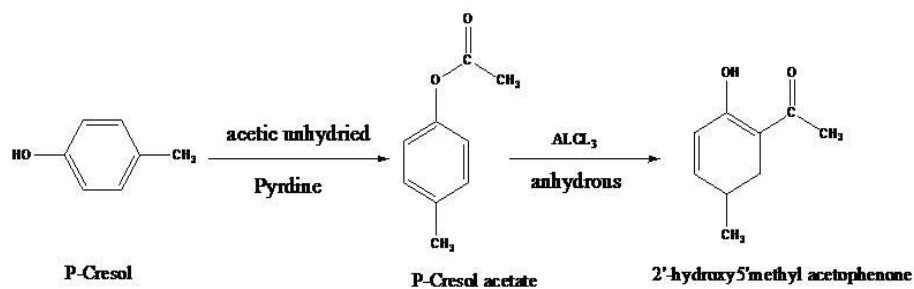
MATERIALS AND METHODS

β -diketone were synthesized by using Baker Venkatraman Transformation method. Synthesized compounds were checked by their M.P. coloration, IR,on silica gel by TLC.

2.1 Synthesis of 1-(2'-hydroxy Phenyl)-3-Phenyl Propane-1,3-dione (β -diketone):

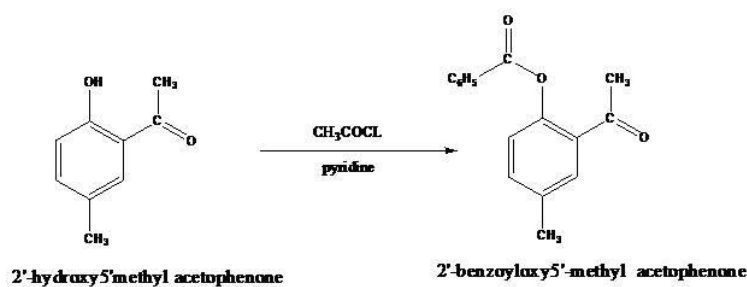
It is prepared From P-cresol. The necessary steps are given below.

- 1) Prepration of 2'-hydroxy5'methyl acetophenone



- 2) Preparation of 2'-benzoyloxy 5'-methyl acetophenone
Benzoyl chloride was added to a mixture of 2'-benzoyloxy 5'-methyl acetophenone and pyridine

added slowly with stirring . low temperature 20°C. The compound 2'-benzoyloxy5'-methyl acetophenone obtained .



- 3) Preparation of 1-(2'-hydroxy 5'-methyl Phenyl)-3-Phenyl Propane-1, 3-dione .(β-diketone)

The above ester was subjected to B.V.Tranceformation methods to gate appropriate reagent.

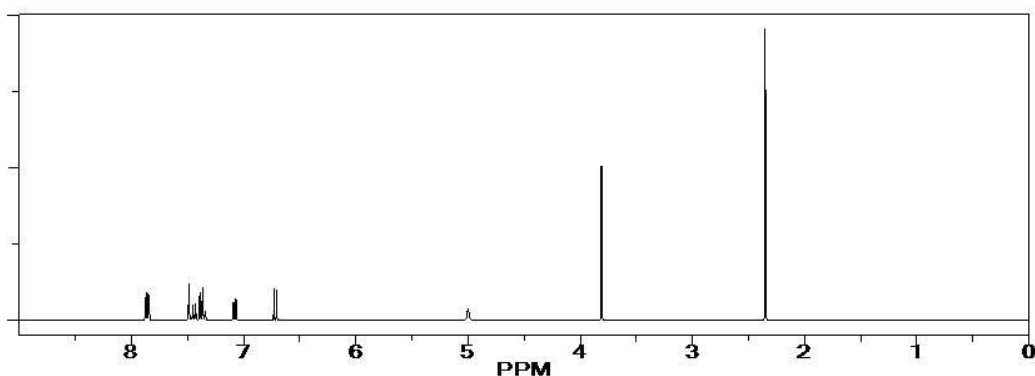
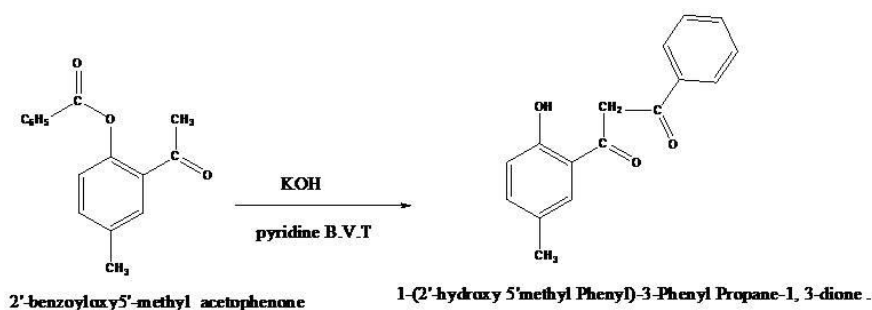


Fig 3: Standard H-1 NMR Prediction:

Spectrophotometric measurements were made by using UV-VIS1601A Shimadzu spectrophotometer using quartz cuvettes.. Selection of Wave length was made by recording the absorption spectra Fe(III) 1-(2'-hydroxy 5'methyl Phenyl)-3-Phenyl Propane-1, 3-dione complex at different range of 400 nm to 800 nm.The effect of pH ,temperature and composition on absorption and wavelength has studied in methanol water medium. Job methods of continuous variation, slope ratio method & Mole ratio method are used at 298 K and 0.1 M ionic strength. The graph of OD v/s composition gave a dome shape cure showing maximum at 1:1 composition.Equimolar solution 0.001M of iron(III) ion and respective ligand were prepared in 80-95% methanol- water medium. Complex solution was prepared by mixing 1 ml of 0.01M metal ion and 10 ml of 0.01 M ligand in a 25 ml standard flask. All chemicals are Anala R grade.

Determinations of the K_{cond} and Confirmation of Empirical formula.

In order to determine the K_{cond} , the equimolar solution of Fe(III) Metal ion(0.01M) Substituted β -diketone i.e. 1-(2'-hydroxy 5'methyl Phenyl)-3-Phenyl Propane-1, 3-dione ,(0.01M) were mixed with varying proportion (1+9,2+8,3+7,.....,9+1.) and maintaining the Percentage of Methanol and Ionic strength ,OD of each solution was measured by fixing λ_{max} i.e.534nm.The plot was drawn between OD v/s composition . A parabolic nature of cure was obtained showing (1:1) complex formation .by selecting 2:8 and 3:7 M:L Composition the K_{cond} was evaluated .The Empirical formula observed by above procedure is confirmed by adopting slope ratio method i.e by fixing metal ion concentration (5ml of .01M) and β -diketone concentration (0.0005M to 0.0025M)in one set and concentration of β -diketone was fixed (0.0025M) and Fe(III) Metal ion from 0.0005M to 0.0025M and absorbance of both set are measured and plot were drawn , straight line was obtain in each plot Showing in Fig.

Table No 1: Lambert –Beer’s law Fe (III) Metal ion(0.01M)- Substituted β -diketone (Job Method) at 298 K in MeOH

Complex Solution ml	Solvent ml	Concentration of metal	Optical Density		
			pH		
			2.00	2.20	2.50
2	18	0.5	0.040	0.045	0.050
4	16	1.0	0.081	0.090	0.100
6	14	1.5	0.120	0.135	0.150
8	12	2.0	0.160	0.180	0.200
10	10	2.5	0.203	0.221	0.245

Table No 2: Spectrophotometric Data of Fe(III) Metal ion(0.01M)- Substituted β -diketone at 298 K in MeOH

Fe(III) Metal ion	β -diketone solution	Optical Density		
		pH		
		2.00	2.20	2.50
1.0	9.0	0.131	0.138	0.149
2.0	8.0	0.155	0.165	0.187
3.0	7.0	0.159	0.185	0.201
4.0	6.0	0.189	0.197	0.219

5.0	5.0	0.203	0.221	0.245
6.0	4.0	0.151	0.172	0.207
7.0	3.0	0.100	0.125	0.141
8.0	2.0	0.065	0.080	0.100
9.0	1.0	0.044	0.052	0.062
		$K_{\text{Cond.}}=3.94$	$K_{\text{Cond.}}=3.91$	$K_{\text{Cond.}}=3.88$

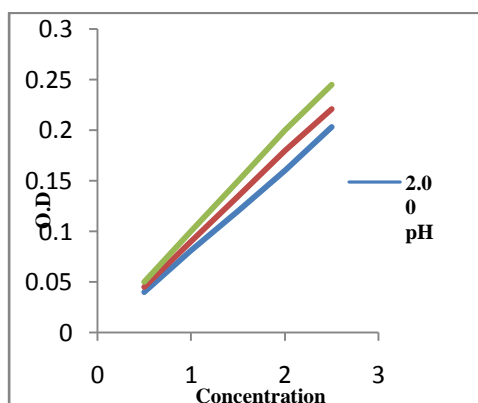


Fig.2. Plot of OD vs Concentration Fe (III) Metal ion (0.01M) - Substituted β -diketone at 298 K in MeOH

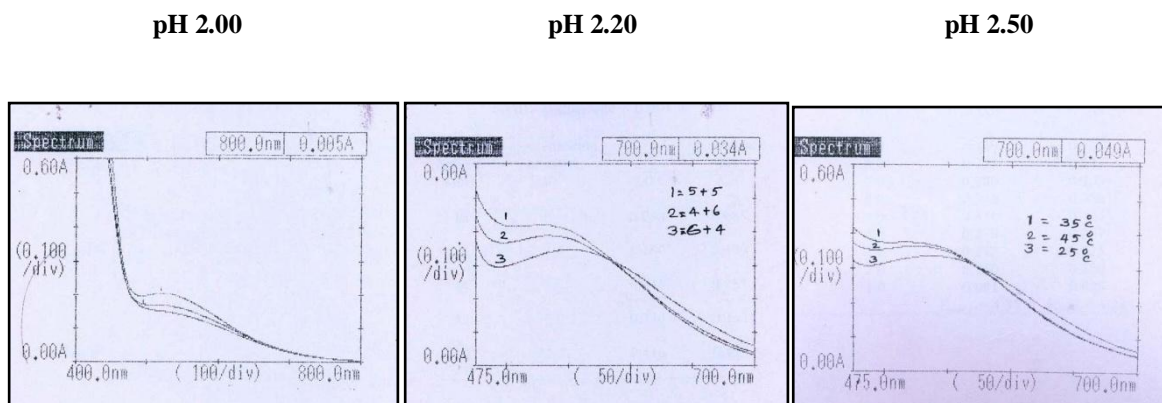
Table No 3: Spectrophotometric Data of Fe(III) Metal ion(0.01M)- Substituted β -diketone(Slope Ratio Method) at 298 K in MeOH

Fe(III) Metal ion	β - diketone solution	Optical Density		
		pH		
		2.00	2.20	2.50
Part I				
1.0	5.0	0.042	0.044	0.042
2.0	5.0	0.081	0.100	0.112
3.0	5.0	0.100	0.112	0.170
4.0	5.0	0.162	0.170	0.180
5.0	5.0	0.200	0.220	0.243
Part II				
5.0	1.0	0.040	0.049	0.060
5.0	2.0	0.082	0.090	0.110
5.0	3.0	0.110	0.115	0.121
5.0	4.0	0.172	0.181	0.189
5.0	5.0	0.203	0.222	0.245

Table No 4: Spectrophotometric Data of Fe(III) Metal ion(0.01M)- Substituted β -diketone at different Temp.in MeOH at pH 2.00

Fe(III) Metal ion	β -diketone solution	Optical Density		
		Temperature		
		25 ⁰ C	35 ⁰ C	45 ⁰ C
1.0	9.0	0.131	0.162	0.132
2.0	8.0	0.155	0.177	0.164
3.0	7.0	0.275	0.200	0.188
4.0	6.0	0.189	0.217	0.200
5.0	5.0	0.203	0.229	0.214
6.0	4.0	0.150	0.179	0.210
7.0	3.0	0.105	0.140	0.153
8.0	2.0	0.075	0.100	0.190
9.0	1.0	0.044	0.058	0.078
		K _{Cond.} = 3.94	K _{Cond.} = 3.95	K _{Cond.} = 3.94

Fig.1 : Absorption spectra of Fe(III) Metal ion(0.01M)- Substituted β -diketone at 298 K in MeOH composition at,2.20,2.50 of(4:6) ,(5:5) ,(6:4).



RESULT AND DISCUSSION

In view of application of 1,3-diones in the synthesis of drugs and as a reagent in quantitative analysis. The spontaneous wine red colour formation by adding Substituted β -diketone in Fe⁺⁺⁺ ion solution even pH less than 2.00, promote to investigate behavior of these complexes in the wide range of pH, Concentration and temperature. The absorption spectra of various complex solution with varying (M:L) composition and

at different condition are recorded in the range 400nm to 800nm and presented in Fig. 1

The result given in tubular form in Table 1-4. K_{Cond.} Values in 90% MeOH deferent pH given in Table 5

It could be seen from above result that the K cond. Valve were found to decrease in pH. Similarly it was also observed that formation constant initially increases with temperature, i.e. up to 35⁰C and than it decrease. Thus the 1:1 complex species in all the system study this part are stable at low pH 2.0.

Table No 5: Stability constant of K and 1:1Complex of Fe(III) Metal ion(0.01M)- Substituted β diketone in 90%MeOH at 0.05M ionic strength.

pH 298 K	2.00	2.20	2.50
Log K _{Cond}	3.94	3.92	3.88
Temp. 2.00 pH	298K	308K	318K
Log K _{Cond}	3.94	3.94	3.94

CONCLUSIONS

In this study, the complex of diketone with metal ion, the maximum wavelength of absorption was defined from recorded spectrum. The job method is verified and complex and stoichiometry were defined. The absorption spectra of complex solution with varying wavelength M:L and K cond. Value were found to be decrease in pH and also observed that the formation cont. initially increase with Temperature.

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