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A Comparative Analysis of 5-(5'-P-Methoxyphenylazo Salicylidine)-Rhodanine and (2-Hydroxy Naphthalidine) Rhodanine With Respect To Their Synthesis, Characterization and Antimicrobial Studies

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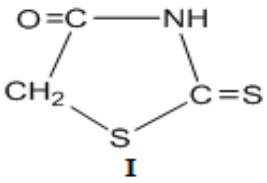
ABSTRAT: *Rhodanine derivatives having benzylidine linkage were prepared by Condensation of Rhodanine (I), and 2-Hydroxo naphthaldehyde and p-methyl phenyl azo-salicylaldehyde separately to get 5-substituted Rhodanine derivatives (II- III). The resulting product was found to be solid which have been characterized by chemical and spectral data. The derivatives (II-III) shown distinctive antimicrobial and antitubercular activity; and their insecticidal activity has been determined with respect to mortality of mosquito larvae.*

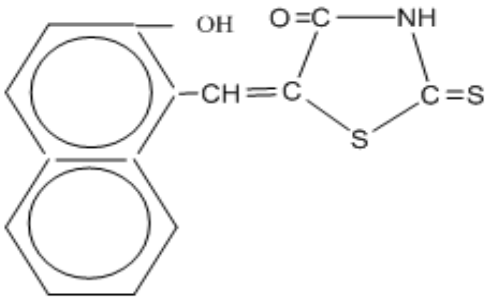
Keywords: Rhodanine, 2-Hydroxo naphthaldehyde, p-methyl phenyl azo-salicylaldehyde, benzylidine, antimicrobial, antitubercular, mosquito larvae.

I. INTRODUCTION

5-substituted Rhodanine derivatives are important class of mixed ligands find extensive application in different fields[1]. They had known for their multiplex analytical and pharmaceutical properties such as fungicides, herbicides, bactericides, aldose reductase, anti-inflammatory, anti-viral and anti-cancerous. Keeping this in view the present paper describes a systematic study of synthesis, characterization and application of some 5-substituted Rhodanine derivatives (II&III).



I



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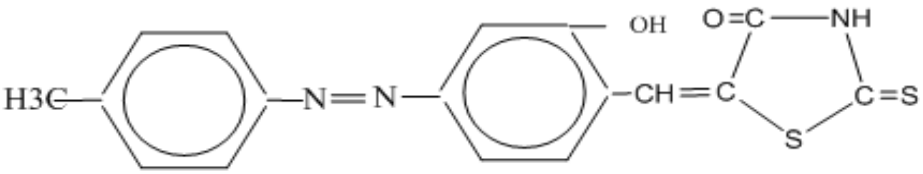

III

II. EXPERIMENTAL

All chemicals used were of AR grade. The Rhodanine (I) was prepared as per reported litrature[2]. Melting point were recorded by using Gallen kamp melting point apparatus are uncorrected. The IR (KBr) spectra were recorded on Perkin-Elmer1800FTIR; Electronic spectra (DMF+DMSO) were obtained using Shimadzu UV-160 spectrophotometer at 25°C.^{1}H NMR ($CDCl_3$+DMSO-d_6/TMS) spectra were measured by Varian EM-360L spectrophotometer. Elemental analyses C, H, N were performed using a Carlo Erba 1108 elemental analyzer. Sulphur was estimated by Mesenger's method and Molecular weights were determined by Rast method.

2.1. Preparation of mixed ligands of 2-Hydroxy naphthaldehyde and p-methyl phenyl azo salicylaldehyde with Rhodanine; (II & III):

A mixture of 2-hydroxy naphthaldehyde (1.72 g) and Rhodanine (1.33 g) in glacial acetic acid (15 mL) was refluxed on a water bath for 4 h. then cooled. The resulting reddish yellow crystals of (II) were found; (1.8 g,59.01%) .The procedure adopted for the preparation of (III)
was similar to that of II with the difference that p-methyl phenyl azo salicylaldehyde (2.40 g) was used and the refluxing time required was 2 h. to get brown colored crystals. The analytical and physical data of mixed ligands are given in table-1.

2.2. Spectral Studies

Electronic Spectra of mixed ligands II & III

The spectrum of ligands II shows band at 302,462 & 564 nm which is attributed to n π*.the spectrum of III show absorption bands at 271, 348 nm respectively due to π π* transition.

IR Spectra of mixed ligands II & III

Characteristic ν_{max} values of mixed ligand II exhibited weak bands at 4000-3480,1425 and1250 cm^{-1},this could be attributed to ν(O-H). The band found at 3040,1340 cm^{-1} belongs to ν (NH).While other bands found at1740(C=O);1480(mercaptamide);1180,780 (C=S) and1100,1020,880,820 $cm^{-1}$for(Ar). In the spectrum of ligand III it was noticed that the weak with slight hump in between4000-3580 band at 3213 cm-1 which could be attributed to ν (OH) with shoulder at 3074 cm-1 due to ν(C-H) aromatic. The other strong bands are appeared at 3040,1340,1290;1577 cm-1, 1500 cm-1 and 1284 cm-1 which attributed to ν(C=N), ν(C=C) and ν(C-O)respectively (Nakamoto, 1986).

^{1}HNMR Spectra of mixed ligands II & III

The mixed ligand II shows δ value for (OH) at 1.89, 5.80 (naphthyl), 7.0-7.70 (Ar, H, C=CH), and 7.95 for (NH).Characteristics δ values shown in the spectrum of III 1.25(CH_3), 2.0 (OH),7.05- 7.30 (Ar-H and C=CH), 7.7 0-7.80 and 7.98 (NH). All spectral results were collected in Table -2.

Table2 - Spectral Data of mixed ligands II & III

S. No.	Mixed ligand	Spectral Data								
		UV λ max	IR (vmax)					¹HNMR (δ)		
			OH	NH	C=O	C=S	Ar	OH	NH	(Ar),H,C=CH,(Naphthyl*)
1	5-(2-hydroxynaphthalidene) Rhodanine, (II)	302, 462, 564.	4000-3480, 1425, 1250,	3040, 1340.	1740	1180,780.	1100, 1020, 880, 820.	(1.89	7.95	7.0-7.70 5.80*.
2	5-(5'-p-methyl phenylazo-salicylidene) Rhodanine, (III)	271, 348.	4000-3360,1445,1240.	3050-3040,1335.	1720-1700	1180-1170,780.	1460 Corresponding to-CH_3.	2.0	7.98	7.05-7.80.and1.25 corresponding toCH_3.

2.3. Biological Screening

The anti-microbial activity of II- III was evaluated in vitro by using some selected bacterial and fungal species. The inhibitory zone by "Agar Diffusion Method", Laben (1950) was used to determine the potentiality of II-III. To inhibit the growth of selected test organism the solution
(DMF) of II-III werw employed at 30,50 and 100 µg mL^{-1} concentrations. For bacterial species the incubation period demanded 24 h (37^oC) but for fungal species it required 8 days (27^oC). All the experiments were repeated in triplicate and the average results are being recorded in Table-3.

Table-3 Antimicrobial Activity of mixed ligand II-III

Test Species	DP	(II)		(III)	
		Z	Res	Z	Res
Bacterial					
E. Aeruginosa					
	30	**10**		**18**	
	50	**15**	**S**	**21**	**S**
P. Putida	**100**	**18**		**24**	
	30	**02**		**20**	
	50	**10**	**PS**	**24**	**VS**
Rhz. Sp.	**100**	**12**		**26**	
	30	**09**		**21**	
	50	**13**	**S**	**23**	**VS**
S. Faecalis	**100**	**18**		**28**	
	30	**20**		**12**	
	50	**28**	**VS**	**14**	**S**
S. Aureus	**100**	**36**		**18**	
	30	**23**		**15**	
	50	**29**	**VS**	**18**	**S**
	100	**37**		**21**	
Fungal					
S. Schencki	**30**	**08**		**20**	
	50	**20**	**S**	**26**	**VS**
A. Fumigates	**1000**	**22**		**30**	
	30	**14**		**18**	
	50	**25**	**VS**	**24**	**VS**
	100	**32**		**32**	

DP = Disc Potency (µg), Z = Zone of inhibition (mm), Res = Result, S = Sensitive, PS =Partially Sensitive.

2.4. Insecticidal Activity

Mosquito (anopheles and culex sp.) eggs were collected, identified and kept separately in proper environment for hatching. The ethanolic-DMF (5:1) solutions (10 mL), with different concentrations (10-90 µg mL^{-1}), of mixed ligands (II-III) were tested for insecticidal activity against twenty mosquito larvae, at subsequent developing stages, in water (100mL). The results have been recorded in Table-4

Table – 4 Insecticidal Activity of Mixed Ligand II-III

Larva in subsequent Stages	Time (min) for 100 % mortality; 0.1 % solution; 25^0c		Control
	II	III	
Anopheles Sp.			
Wriggler	**8.5**	**9.8**	**80**
3-dasy old	**8.5**	**10.0**	**95**
6-days old	**11.0**	**11.3**	**110.0**
Full grown	**14.0**	**15.2**	**125.0**
Culex Sp.			
Wriggler	**8.2**	**9.4**	**79.0**
3-dasy old	**9.5**	**9.8**	**98.0**
6-days old	**10.9**	**11.0**	**110.0**
Full grown	**13.9**	**14.2**	**124.8**

III. RESULTS AND DISCUSSION

The recorded analytical and physical data support the proposed structures of the synthesized mixed ligands (II-III). The IR[3] spectra showed no indication of a -SH band in the region2600 – 2500 cm^{-1} but exhibited a strong absorption at 1480 cm^{-1} pertaining to mixed vibration of mercaptomide band. Along with frequent absorption of aromatic rings the II-III showed bands at 4000-3480, 1420, and 1250- 1240; 3040, 1340 and 1280; 1740-1700,1180-1175 and 780 cm^{-1} corresponding to ν(OH), ν(NH), ν(C=O) and ν(C=S) respectively. Thus the association of Rhodanine moiety with respective aldehyde is evident in **II-III**.

The electronic spectra[3] of II-III exhibited characteristic maxima for π-π* transitions, corresponding to conjugated aromatic rings containing a thio-keto structure, in the range 269-302 nm. The other absorption bands observed at 393 (II) and 462 (III) nm may be ascribed to π-π* transitions related to thio-keto structure, and lengthening of conjugated system. An additional band at 564 nm found in II may however be attributed to existence of a naphthyl system.

The ^{1}H NMR spectra[3] of II-III have shown sharp to medium bands at δ 1.89, 2.85, 5.80 in (III), 6.80-7.80, and7.95 corresponding to the resonated at –OH protons, aromatic and C=CH protons and –NH protons respectively. In II the –OH proton has shown a medium intensity band at δ 1.89 in comparison to sharp band at δ 2.85 in II.and the -CH proton has exhibited an additional brad sharp band at δ 5.80. These differences may be attributed to different electronic environment due to the presence of hydroxy naphthyl system in II.

In biological screening, Table-3, it was found that the solution of II very sensitive against *S. Faecalis and S. aureus*, and *III* is very sensitive against *P.putida and Rhizobium sps*. II &III are very sensitive against the fungal sps. *S.schencki* and *A. fumigates.* It is also apparent from the results Table-2,that the sensitivity of II is distinctively greater than of III against all test sps. .

Insecticidal activity result, Table – 4, clearly indicate that the toxicity effect for 100% mortality of mosquito (anopheles and culex sp.) larvae is maximum at 90 µg mL^{-1} concentration with respect to control. It is also evident that II is more effective than III in respect of both sps. And II – III are slightly more efficacious for culex species.

IV. CONCLUSION

The comparative Study of synthesis and characterization and application of Mixed ligand showed good antibacterial, antifungal and insecticidal activity. The II appeared more active than III. The activity of III reduced due to -CH_3 group in the terminal benzene ring. The results were shown that the mixed ligand II was more effective due to the presence of naphthyl ring.

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