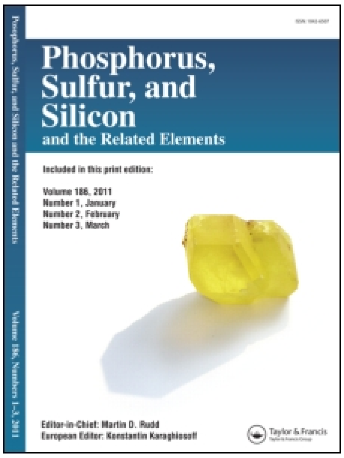

Phosphorus, Sulfur, and Silicon and the Related Elements
Publication details, including instructions for authors and subscription information:
http://www.informaworld.com/smpp/title~content=t713618290

COORDINATION BEHAVIOR OF BIOLOGICALLY ACTIVE SCHIFF BASES OF AMINO ACIDS TOWARDS SILICON(IV) ION

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To cite this Article Sharma, M. , Khungar, B. , Varshney, S. , Singh, H. L. , Tripaathi, U. D. and Varshney, A. K.(2001) 'COORDINATION BEHAVIOR OF BIOLOGICALLY ACTIVE SCHIFF BASES OF AMINO ACIDS TOWARDS SILICON(IV) ION', Phosphorus, Sulfur, and Silicon and the Related Elements, 174: 1, 239 — 246

To link to this Article: DOI: 10.1080/10426500108040248

URL: http://dx.doi.org/10.1080/10426500108040248

Phosphorus, Sulfur and Silicon, 2001, Vol. 174, pp. 239-246
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COORDINATION BEHAVIOR OF BIOLOGICALLY ACTIVE SCHIFF BASES OF AMINO ACIDS TOWARDS SILICON(IV) ION

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(Received May 23, 2000; In final form January 25, 2001)

Reactions of diethoxydimethylsilane with Schiff bases derived from the condensation of furfuraldehyde, indole-3-carbaldehyde with alanine, glycine, valine, isoleucine and tryptophan in a 1:2 molar ratio give a new series of $Me_2Si\ (\overset{\frown}{N\ O})_2$ type of organosilicon (IV) complexes. The complexes are monomeric and non-electrolytic in nature. The coordination behaviour of Schiff bases through organosilicon (IV) has been investigated by IR, ^1H, ^{13}C & ^{29}Si NMR spectral studies. Schiff bases and their silicon complexes have also been screened for their antifungal activity. Several of these complexes were found to be quite active in this respect.

INTRODUCTION

Schiff bases are an important class of ligands and have found wide applications in various fields[1–3]. Amino acids and their ligands with different metal ions also play an important role in biology, pharmacy, and industry[4–6]. Continuing our earlier research[7–10] on biologically active complexes, an attempt has now been made to synthesize amino acid Schiff base complexes of diethoxydimethylsilane. The Schiff bases used in this study are the following:

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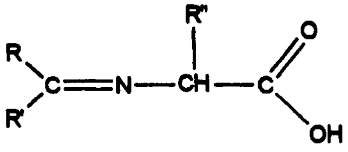
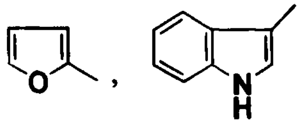
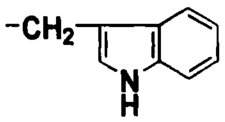


where R = H

R' =



R'' = H, CH_3, $-CH(CH_3)_2$, $-CH(CH_3)-CH_2-CH_3$,



RESULT AND DISCUSSION

The reactions of diethoxydimethylsilane with these ligands have been carried out in benzene medium as.

$$(CH_3)_2\,Si(OC_2H_5)_2 + 2\overset{\frown}{N\,OH} \longrightarrow (CH_3)_2\,Si(\overset{\frown}{N\,O})_2 + 2C_2H_5OH$$

Where NOH is represent donor set of ligands. On refluxing for about 6–7 hours the resulting complexes are obtained as coloured solids. Low values of molar conductance (10–15 $Ohm^{-1}cm^2mol^{-1}$) show that these complexes are non-electrolytes. The molecular weights of the complexes determined by the Rast method correspond to the formula weight indicating their monomeric nature.

IR Spectra

The IR spectra of these Schiff bases exhibit a sharp and strong band due to ν(C=N) vibrations at 1610–1625 cm^{-1}. It shifts to the lower frequency region in the silicon complexes and appears at ~1600 cm^{-1} in the 1:2 complexes, indicating the coordination of azomethine nitrogen to the silicon atom[11].

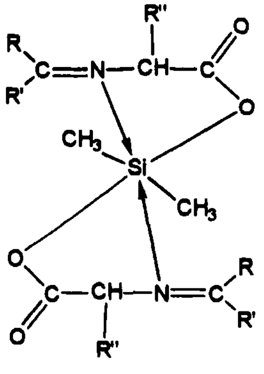
A new band observed in the complexes at ~570 cm^{-1} is due to ν(Si←N)[12] mode. Many new bands of strong to medium intensity in the spectra of the complexes at ~1265 may be due to the asymmetric deformation[13] mode of CH_3-Si.

^{1}H NMR Spectra

In the ^{1}H NMR spectra of the above ligands the broad signal due to the NH proton at δ~10.15 ppm disappears in the silicon (IV) complexes, indicating the coordination of nitrogen through a covalent bond formation by the nitrogen with a silicon atom. New signals at δ ~1.60 ppm in the complexes are due to the methyl protons of Me_2Si group.

^{29}Si NMR spectra

The ^{29}Si NMR spectrum of the silicon complexes gives a sharp signal at δ ~40.98 ppm clearly indicating the six coordinated environment around the silicon atom. On the basis of the above spectral studies the following octahedral geometry is suggested for the silicon(IV) complexes.



Antimicrobial activity

The Antibacterial and antifungal tests were carried out using the disc-diffusion method[14,15] for their activities at a concentration of 100 ppm. Streptomycin and mycostatin were used as reference compounds for antibacterial and antifungal activities, respectively.

E. coli, P. mirabilis, S. aureus, P. syringae (bacteria) or *Fusarium, R. phaseoli, A. niger, P. crysogenous* (fungi) were used as the test organisms. Results have been recorded in the form of inhibition zone (diameter mm) and activity index in Table III.

Results show that the silicon complexes are more potent in their inhibition properties than the free ligands. Among all the complexes $Me_2Si(L^3)_2$and $Me_2Si(L^{10})_2$ are more active against *P. crysogenous, R. phaseoli, P. syringal* and *P. mirabilis.*

EXPERIMENTAL

All the chemicals used in this work of AR grade and solvents were dried by standard method and all reactions were carried out under an anhydrous and oxygen free atmosphere. The infrared spectra were obtained on a Perkin-Elmer 577 grating spectrophotometer in the region 4000–200 cm^{-1} using KBr optics. ^1H NMR spectra were recorded on a Perkin-Elmer RB-12 Spectrometer in DMSO-d_6 using TMS as an internal standard at 90 MHz. ^{29}Si NMR spectra were recorded on a Jeol FX90 Q spectrometer in dry DMSO using TMS as an internal standard at 17.75 MHz. The Schiff bases have been synthesized by the condensation of furfuraldehyde and indole-3-carbaldehyde with amino acids viz. glycine, L-alanine, L-valine, L-isoleucine and L-tryptophan in 1:1 molar ratio using ethanol as the reaction medium and then refluxing for 5 hours. After which the solvent was removed *in vacuo.* The solids so obtained were recrystallised from ethanol (Table I).

Synthesis of Si (IV) Complexes

To a weighed amount of diethoxydimethylsilane in dry benzene was added the calculated amount of the appropriate ligand in a 1:2 molar ratio. The reactants were refluxed for about 6–7 hours to complete the reaction. The ethanol generated in the reaction was removed azeotropically with benzene, the excess of the solvent was removed under reduced pressure and the complexes were subsequently dried for 2–3 hours. These were then repeatedly washed with dry cyclohexane so as to ensure their purity and again dried under reduced pressure. The synthetic and analytical data of the resulting complexes are recorded in Table II.

TABLE I Physical Properties and Analytical Data of Ligands

S.N.	*Ligands*	*Colour*	*M.P.* °*C*	*Analysis* *C Found* *(Calc.)*	*H Found* *(Calc.)*	*N Found* *(Calc.)*	*Mol. Wt. Found* *(Calc.)*
1.	Alanine furfuraldehyde $C_8H_9NO_3$	Silvery brown solid	180	57.23 (57.42)	5.21 (5.38)	8.11 (8.37)	165.0 (167.18)
2.	Valine furfuraldehyde $C_{10}H_{13}NO_3$	silvery play solid	193	61.23 (61.46)	6.32 (6.66)	7.12 (7.17)	193.72 (195.24)
3.	Glycine furfuraldehyde $C_7H_7NO_3$	Reddish brown solid	200	54.11 (54.14)	4.43 (4.51)	9.01 (9.02)	152.99 (155.14)
4.	Tryptophan furfuraldehyde $C_{16}H_{14}N_2O_3$	Greenish brown solid	210	67.88 (68.01)	4.73 (4.96)	9.79 (9.92)	280.93 (282.32)
5.	Isoleucine furfuraldehyde $C_{11}H_{15}NO_3$	Silvery gray solid	200	63.00 (63.08)	7.12 (7.17)	6.52 (6.69)	206.19 (209.27)
6.	Alanine Indole 3-carbaldehyde $C_{12}H_{12}N_2O_2$	Pinkish brown solid	136	66.42 (66.59)	5.49 (5.55)	12.73 (12.95)	214.32 (216 25)
7.	Valine Indole 3- carbaldehyde $C_{14}H_{16}N_2O_2$	Pinkish brown solid	160	68.66 (68.77)	6.32 (6.55)	11.31 (11.46)	232.12 (244.31)
8.	Glycine Indole 3- carbaldehyde $C_{11}H_{10}N_2O_2$	Pinkish brown solid	132	64.51 (64.64)	4.71 (4.89)	13.61 (13.71)	200.12 (204.21)
9.	Tryptophan Indole 3- carbaldehyde $C_{20}H_{17}N_3O_2$	Pinkish brown solid	152	72.31 (72.42)	5.01 (5.13)	12.51 (12.67)	323.64 (331.39)
10.	Isoleucine Indole 3- carbaldehyde $C_{15}H_{18}N_2O_2$	Pinkish brown solid	142	69.50 (69.68)	6.72 (6.97)	10.73 (10.84)	253.30 (258 34)

TABLE II Synthesis and Analysis of Si(IV) Complexes

S.N.	*Ractants*		*Molar ratio*	*Products colour & state*	*M.P °C*	*Analysis %*				*Mol. Wt. Found (Calcd)*
	Silicon compound	*Ligand*				*C Found (Calcd)*	*H Found (Calcd)*	*N Found Calcd)*	*Si Found (Calcd)*	
1.	$(C_2H_5O)_2Si(CH_3)_2$	$C_8H_9NO_3$	1:2	Brown shining crystals	258°C	55.01 (55.33)	5.21 (5.64)	7.0 (7.17)	7.05 (7.17)	388.01 (390.36)
2.	$(C_2H_5O)_2Si(CH_3)_2$	$C_{10}H_{13}NO_3$	1:2	Brown shining flakes	179(d)	59.03 (59.13)	6.56 (6.72)	6.11 (6.27)	6.10 (6.27)	443.92 (446.48)
3.	$(C_2H_5O)_2Si\ (CH_3)_2$	$C_7H_7NO_3$	1:2	Blackish Brown solid	232°C	52.20 (52.42)	4.73 (4.91)	7.09 (7.64)	7.18 (7.64)	362.79 (366.28)
4.	$(C_2H_5O)_2Si\ (CH_3)_2$	$C_{16}H_{14}N_2O_3$	1:2	Light shining solid	150–152(d)	65.32 (65.74)	5.00 (5.16)	8.69 (9.02)	4.44 (4.51)	617.36 (620.64)
5.	$(C_2H_5O)_2Si(CH_3)_2$	$C_{11}H_{15}NO_3$	1:2	Light shining flakes	173(d)	60 33 (60.69)	7.02 (7.16)	5.62 (5.90)	5.73 (5.90)	472.00 (474.54)
6.	$(C_2H_5O)_2Si(CH_3)_2$	$C_{12}H_{12}N_2O_2$	1:2	Light shining solid	126°C	63.72 (63.87)	5.67 (5.73)	11.22 (11.46)	5.52 (5.73)	485.72 (488.05)
7.	$(C_2H_5O)_2Si(CH_3)_2$	$C_{14}H_{16}N_2O_2$	1:2	Brown solid crystals	137(d)	66.00 (66.10)	6.51 (6.61)	10.11 (10.28)	5.01 (5.14)	540.33 (544.62)
8.	$(C_2H_5O)_2Si\ (CH_3)_2$	$C_{11}H_{10}N_2O_2$	1:2	Brown solid crystals	156	62.00 (62.01)	5.11 (5.17)	12.00 (12.06)	6.00 (6.03)	462.7 (464.42)
9.	$(C_2H_5O)_2Si(CH_3)_2$	$C_{20}H_{17}N_3O_2$	1:2	Brownish orange	258°C	70.03 (70.12)	5.03 (5.29)	11.63 (11.69)	3.62 (3.89)	713.2 (718.78)
10.	$(C_2H_5O)_2Si(CH_3)_2$	$C_{15}H_{18}N_2O_2$	1:2	Brown solid crystals	118	67.00 (67.05)	6.73 (6.98)	9.63 (9.78)	4.72 (4.89)	570.0 (572.68)

TABLE III Antimicrobial Activity of ligands and their Silicon (IV) Complexes

	Microorganisms		*Compounds*			
			L^3	$Me_2Si(L^3)_2$	L^{10}	$Me_2Si(L^{10})_2$
(1)	*E. coli*	IZ	13.2	13.7	11.0	13
		AI	1.46	1.52	1.22	1.44
(2)	*P. mirabilis*	IZ	13.6	14.5	12.2	15.2
		AI	1.23	1.31	1.10	1.38
(3)	*S. aureus*	IZ	14.9	16.4	15.2	17.8
		AI	1.06	1.17	1.08	1.27
(4)	*P. syringae*	IZ	11.2	12.9	10.6	14
		AI	1.4	1.61	1.32	1.75
(5)	*Fusarium*	IZ	14.9	16.2	16	17.9
		AI	0.76	1.15	1.14	1.27
(6)	*R. phaseoli*	IZ	12	14	12	13
		AI	1.33	1.55	1.33	1.44
(7)	*A. niger*	IZ	12.8	14	14	15.2
		AI	1.06	1.16	1.16	1.26
(8)	*P. crysogenous*	IZ	13	10	13.7	13.9
		AI	1.42	1.85	1.42	1.38

IZ=Inhibition zone (in mm), AI=Activity Index = Inhibition zone of test compounds/Inhibition zone of standard.

Acknowledgements

The authors express their sincere thanks to Prof. J.P. Tandon former Head, Department of Chemistry, University of Rajasthan, Jaipur, for his helpful suggestions.

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