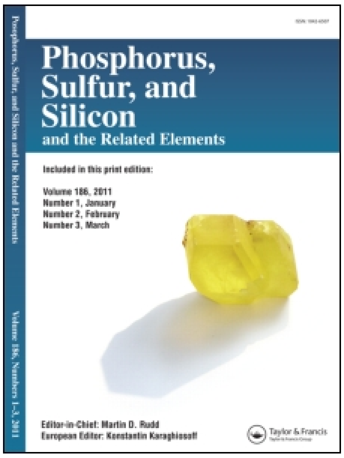

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SYNTHETIC AND STRUCTURAL ASPECTS OF CERTAIN DIORGANOSILICON(IV) CHELATES DERIVED FROM STERICALLY DEMANDING 4-(2'-MERCAPTO PHENYL IMINO ALKYL/AROYL) -2,4-DIHYDRO-5-METHYL-2-PHENYL-3H- PYRAZOL-3-ONES

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SYNTHETIC AND STRUCTURAL ASPECTS OF CERTAIN DIORGANOSILICON(IV) CHELATES DERIVED FROM STERICALLY DEMANDING 4-(2′-MERCAPTO PHENYL IMINO ALKYL/AROYL) - 2,4-DIHYDRO-5-METHYL-2-PHENYL-3H-PYRAZOL-3-ONES

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Diorganosilicon (IV) Schiff base chelates of the type $RR'Si[R''C(:o\text{-}NC_6H_4S)C:C(O)N(C_6H_5)N:CCH_3$, have been synthesised by the reaction of diorganodichlorosilanes, $RR'SiCl_2$where (i)R =R′ = $-CH_3$ (ii) R = $-CH_3$ and R′ = $-C_6H_5$ with the disodium salt of 4-(2′-mercapto phenyl imino alkyl/aroyl) – 2,4 – dihydro-5-methyl – 2-phenyl-3H-pyrazol-3-ones i.e. $R''C(:o\text{-}NC_6H_4SNa)C:C(ONa)N(C_6H_5)N:CCH_3$ (where R″ = $-CH_3$, $-C_2H_5$ – C_6H_5 and – p – Cl C_6H_4) in benzene solution. These chelates may alternatively be prepared by the reaction of Schiff bases with the corresponding diorganodichlorosilanes in presence of triethylamine in benzene solution in a 1:1:2 molar ratio. The compounds, thus obtained, were characterised on the basis of molecular weight measurements, IR and ^1H, ^{13}C, ^{29}Si NMR spectral studies.

Keywords: Diorganodichlorosilane; IR; NMR; 4-(2′-mercapto phenyl imino alkyl/aroyl)-2, 4-dihydro-5-methyl-2-phenyl-3H-pyrazol-3-ones

INTRODUCTION

Apart from industrial applications and utilisation in organic and inorganic syntheses[1–5], organosilicon complexes are shown to possess a variety of biological applications including antifungal, antibacterial antifertility[6–8]

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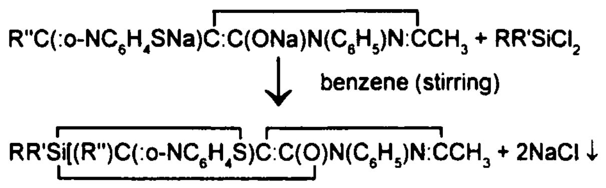
* Correspondence author.

and also antitumor activities[9,10]. Some organosilicon Schiff base complexes are reported to possess significant biological activity.[8] In continuation to our work on group (IV) complexes derived from 4-[2′-mercapto phenyl imino alkyl/aroyl) – 2,4-dihydro-5-methyl-2-phenyl-3H-pyrazol-3-ones[11], it has been considered worthwhile to synthesize their corresponding organosilicon (IV) complexes. These complexes were characterised on the basis of spectral studies (IR, ^1H, ^{13}C and ^{29}Si NMR) and osmometric molecular weight measurements.

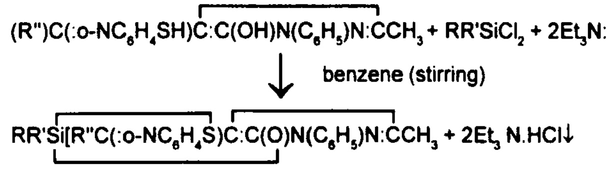
RESULTS AND DISCUSSION

The disodium salt of the Schiff bases react with Me_2SiCl_2 and $PhMeSiCl_2$ in 1:1 molar ratio in benzene solution to yield their corresponding chelates.where (i) R = R′ = Me (ii) R = Me and R′ = Ph and R" = $-CH_3$,$-C_2H_5$, $-C_6H_5$ and $-p-ClC_6H_4$



$$R''C(:o\text{-}NC_6H_4SNa)C{:}C(ONa)N(C_6H_5)N{:}CCH_3 + RR'SiCl_2 \xrightarrow{\text{benzene (stirring)}} RR'Si[(R'')C(:o\text{-}NC_6H_4S)C{:}C(O)N(C_6H_5)N{:}CCH_3 + 2NaCl\downarrow$$

Alternatively, these complexes may be prepared by the reaction of the Schiff bases with corresponding organosilicon (IV) chlorides in presence of triethylamine in benzene solution in 1:1:2 molar ratio, where (i) R = R′ = $-CH_3$ (ii) R = $-CH_3$, R′ = $-C_6H_5$ and R" = $-CH_3$, $-C_2H_5$, $-C_6H_5$ & $-p-ClC_6H_4$



$$(R'')C(:o\text{-}NC_6H_4SH)C{:}C(OH)N(C_6H_5)N{:}CCH_3 + RR'SiCl_2 + 2Et_3N{:} \xrightarrow{\text{benzene (stirring)}} RR'Si[R''C(:o\text{-}NC_6H_4S)C{:}C(O)N(C_6H_5)N{:}CCH_3 + 2Et_3\,N.HCl\downarrow$$

The precipitated NaCl and/or $Et_3N.HCl$ is filtered off. After stripping off the volatile fraction, green coloured solid products were obtained which were found to be soluble in common organic solvents like chloroform, benzene, THF etc. and insoluble in pet. ether, hexane etc. These complexes were recrystallised from a $CHCl_3$/pet. ether mixture. The osmometric molecular weight measurements in $CHCl_3$ solution at 45°C reveal their monomeric nature.

I R SPECTRA

The I.R. spectra of the above complexes were recorded as KBr pellets in 4000–400 cm^{-1} range. A broad band observed in the range 3440–3302cm^{-1} and assigned to ν-OH and ν >NH in the Schiff bases was found to be absent in the spectra of the corresponding organosilicon (IV) chelates showing the deprotonation of ν >NH/-OH protons. The band due to ν >C=N-[12] appears in the range 1633–1617 cm^{-1}. The bands observed in the region 540–515 cm^{-1} and 640–615 cm^{-1} may be assigned to ν Si-S[12] and ν Si-O[12]. Appearance of these bands indicates the bonding of both sulphur and oxygen atoms of the Schiff bases with the central silicon atom. Another band observed in the region 560–529 cm^{-1} may be assigned to ν Si<···· N[13]. Thus, IR spectral studies show the bifunctional tridentate behaviour of the schiff bases in organosilicon chelates.

^{1}H NMR SPECTRA

The ^{1}HNMR spectra were recorded in $CDCl_3$ solution using TMS as an internal reference. The observed values of ^{1}HNMR chemical shifts have been summarized in table I. The broad singlets observed in the region δ4.96–6.23 ppm and assigned to >NH/-SH protons in the ligands, were found to be absent in the corresponding organosilicon complexes supporting deprotonation of >NH/-SH protons. The ring methyl protons are observed in the region δ2.08 – 2.47 ppm. The terminal methyl protons in the chelate No.1 were observed at δ2.25 ppm as a singlet. In chelate No. 3 & 4 the terminal protons are observed at δ1.20 ppm as a triplet due to $-CH_3$ protons and at δ2.83 ppm as a quartet due to the $-CH_2$ protons, respectively. The ring phenyl/terminal phenyl & thiophenol proton signals are merged together and are observed as a complex pattern in the region δ6.61–8.13 ppm. The protons of methyl groups attached to silicon atom are observed in the region δ0.068–0.36 ppm.

^{13}C NMR SPECTRA

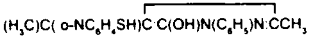
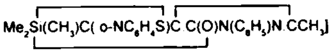
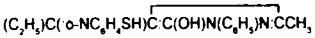
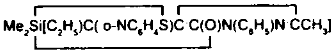
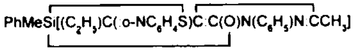
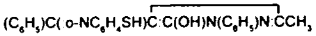
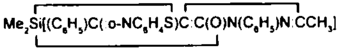
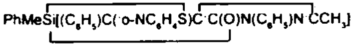
The ^{13}C NMR spectra were recorded in $CHCl_3$ solution using TMS as standard reference. The observed chemical shift values are summarised in table (II). On comparing the ^{13}C NMR spectra of the ligand with the corresponding organosilicon complexes, a slight upfield shift in the position of C_6 carbon signal has been observed. In Schiff bases, the C_6 carbon signal is observed in the range δ 190.85 – 198.00 ppm, however, in the complexes this carbon signal is observed in the range δ190.69 – 196.56 ppm. The C_3 and C_4 carbon signals are observed slightly downfield in comparison to their position in the ligands. The ring methyl carbon signal is observed at δ15.50 ppm. The terminal methyl carbon in the chelate No. 1 is observed at δ26.49 ppm and in chelate No.4, two signals are observed at δ8.12 ppm and δ32.29 ppm and may be assigned to the $-CH_3$ and $-CH_2$carbon atoms, respectively. The chemical shift values for the carbon signals of ring phenyl/terminal C_6H_5/C_6H_4Cl and thiophenol ring are observed in the range δ 115.07 – 148.42 ppm. The signal for the carbon atom of the methyl group attached to silicon atom are observed in the region δ0.93 – 0.98 ppm. Thus, ^{1}H NMR as well as ^{13}C NMR data are supportive of the fact that benzothiazoline form of the ligand rearranges to give the azomethine form, the disodium salt of which finally reacts to give their respective organosilicon chelate.

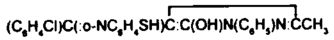
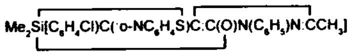
^{29}SI NMR

The ^{29}Si NMR spectra of these complexes have been recorded in $CHCl_3$ solution using TMS as standard reference The ^{29}Si NMR[14–18] offers an important method for determining the coordination environment around central silicon atom in silicon complexes. The ^{29}Si NMR chemical shifts values observed in some of these complexes are shown as follows:

Chelates	^{29}Si NMR (Chemical shifts in δ ppm)
Chelate No.1	−19.4
Chelate No.5	−19.75
Chelate No.6	−19.47

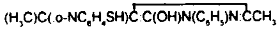
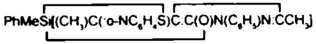
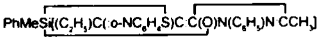
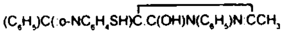
TABLE I The ^{1}HNMR spectral data of organosilicon (IV) chelates derived from 4-(2′-Mercapto phenyl imino alkyl/aroyl)-2,4-dihydro-5-methyl-2-phenyl-3H-pyrazol-3-ones

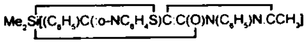
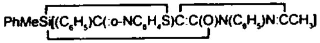
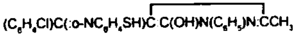
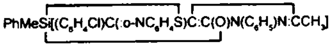
S No.	*Compound*	*>NH/ -SH*	*Ring Methyl*	*Ring Phenyl/Terminal C_6H_5/C_6H_4Cl/Thiophenol Ring*	*Terminal Protons*		*Si-CH$_3$*
					>CH$_2$		*-CH$_3$*
	 $\mathrm{(H_3C)C(\ o\text{-}NC_6H_4SH)C\cdot C(OH)N(C_6H_5)N{:}CCH_3}$	4.96 bs	2.50 d	6.52–7.95m	–	2.50 d	–
1.	 $\mathrm{Me_2Si(CH_3)C(\ o\text{-}NC_6H_4S)C\cdot C(O)N(C_6H_5)N\cdot CCH_3]}$	–	2.40s	6.61 – 8.05m	–	2.25s	0.069s
	 $\mathrm{(C_2H_5)C(\ o\text{-}NC_6H_4SH)C{:}C(OH)N(C_6H_5)N{:}CCH_3}$	5.54 bs	2.50 s	6.52 – 8.04m	2.01 q	1.25 t	–
2.	 $\mathrm{Me_2Si[C_2H_5)C(\ o\text{-}NC_6H_4S)C\cdot C(O)N(C_6H_5)N\cdot CCH_3]}$	–	2.47s	7.04 – 7.96m	2.86q	1.20t	–
3.	 $\mathrm{PhMeSi[(C_2H_5)C(\ o\text{-}NC_6H_4S)C{:}C(O)N(C_6H_5)N{:}CCH_3]}$	–	2.47s	6.59 – 8.12m	2.86q	1.20t	–
	 $\mathrm{(C_6H_5)C(\ o\text{-}NC_6H_4SH)C{:}C(OH)N(C_6H_5)N{:}CCH_3}$	6.23 bs	2.10 s	6.48 – 8.04m	–	–	–
4.	 $\mathrm{Me_2Si[(C_6H_5)C(\ o\text{-}NC_6H_4S)C{:}C(O)N(C_6H_5)N{:}CCH_3]}$	–	2.09s	6.59 – 8.13m	–	–	0.32s
5.	 $\mathrm{PhMeSi[(C_6H_5)C(\ o\text{-}NC_6H_4S)C\cdot C(O)N(C_6H_5)N\cdot CCH_3]}$	–	2.14s	7.33 – 8.12m	–	–	0.36s

S No.	*Compound*	*>NH/ -SH*	*Ring Methyl*	*Ring Phenyl/Terminal C_6H_5/C_6H_4Cl/Thiophenol Ring*	*Terminal Protons*		*Si-CH_3*
					>CH_2		*-CH_3*
	 $(C_6H_4Cl)C(:o\text{-}NC_6H_4SH)C:C(OH)N(C_6H_5)N:CCH_3$	5.14 bs	2.14 s	6.57 – 8.09m	–	–	–
6.	 $Me_2Si[(C_6H_4Cl)C(:o\text{-}NC_6H_4S)C:C(O)N(C_6H_5)N:CCH_3]$	–	2.09 s	6.89 – 7.98m	–	–	0.073s

Where s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, bs = broad singlet

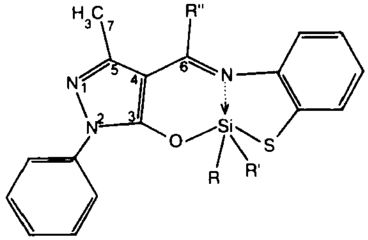
TABLE II The ^{13}C NMR spectral data of organosilicon (IV) chelates derived from 4-(2′-Mercapto phenyl imino alkyl/aroyl)-2,4-dihydro-5-methyl-2-phenyl-3H-pyrazol-3-ones[11]

S.No.	Compound	C_3	C_4	C_5	C_6	C_7	$-CH_2$	$-CH_3$	$C_6H_5{}^a$ / C_6H_4	Ring Phenyl[a]	Thiophenol Ring	$Si\text{-}CH_3$
	 $(H_3C)C(\cdot o\text{-}NC_6H_4SH)C\cdot C(OH)N(C_6H_5)N\cdot CCH_3$	160.46	104.23	137.27	194.60	15.49	–	26.43	–	137.27,131.53 129.04,126.49	115.17,118.15 118.69,120.59 136.79,147.62	–
1.	 $PhMeSi[(CH_3)C(\cdot o\text{-}NC_6H_4S)C\cdot C(O)N(C_6H_5)N\cdot CCH_3]$	160.66	104.28	137.42	194.11	15.39	–	26.49	–	147.62,128.99 120.59,126.49	115.16,118.15 118.59,120.59 136.62,148.15	0.98
	 $(C_2H_5)C(\cdot o\text{-}NC_6H_4SH)C\cdot C(OH)N(C_6H_5)N\cdot CCH_3$	160.39	103.50	137.51	198.00	15.66	32.34	8.18	–	136.73,131.81 129.32,126.84	115.12,118.10 118.94,120.59 136.73,147.30	–
2.	 $PhMeSi[(C_2H_5)C(\cdot o\text{-}NC_6H_4S)C\cdot C(O)N(C_6H_5)N\cdot CCH_3]$	160.89	103.67	137.08	196.56	15.31	32.29	8.12	–	147.92,129.75 120.66,126.39	115.12,118.10 118.24,120.65 136.82,148.02	0.93
	 $(C_6H_5)C(\cdot o\text{-}NC_6H_4SH)C\cdot C(OH)N(C_6H_5)N\cdot CCH_3$	161.44	130.52	137.44	191.56	15.71		–	131.69,128.01 128.23,127.79	148.54,128.93 120.53,126.44	115.07,117.89 118.20,120.53 136.62,147.78	–

S.No.	*Compound*	C_3	C_4	C_5	C_6	C_7	$-CH_2$	$-CH_3$	$C_6H_5^a$ / C_6H_4	*Ring Phenyl*[a]	*Thiophenol Ring*	$Si\text{-}CH_3$
3.		161.49	103.69	137.06	191.51	15.55	–	–	137.55,128.17	147.84,128.82	115.07,117.89	
	 $Me_2Si[(C_6H_5)C(:o\text{-}NC_6H_4S)C:C(O)N(C_6H_5)N.CCH_3]$								128.82,127.74	120.49,126.28	118.24,120.48	0.93
											136.57,148.26	
4.		161.54	103.75	137.80	191.48	15.66	–	–	138.42,128.17	147.80,129.09	115.07,117.90	
	 $PhMeSi[(C_6H_5)C(:o\text{-}NC_6H_4S)C:C(O)N(C_6H_5)N:CCH_3]$								128.39,127.85	120.75,126.66	118.21,120.75	0.98
											136.55,148.42	
		161.17	103.47	138.09	190.85	15.82	–	–	138.09,129.04	148.60,129.31	115.12,118.15	
	 $(C_6H_4Cl)C(:o\text{-}NC_6H_4SH)C:C(OH)N(C_6H_5)N:CCH_3$								126.71,136.63	120.75,128.71	118.75,120.75	–
											136.08,147.57	
5.		161.06	103.47	137.00	190.69	15.71	–	–	137.98,129.26	147.51,129.58	115.23,118.21	
	 $PhMeSi[(C_6H_4Cl)C(:o\text{-}NC_6H_4S)C:C(O)N(C_6H_5)N:CCH_3]$								128.99,128.61	120.64,126.60	118.80,120.64	0.93
											136.46,148.11	

a. Values are given in the order of i, o, m and p, respectively.

In view of the monomeric nature of these diorganosilicon (IV) chelates (as shown by molecular weight measurements) the bifunctional tridentate behaviour of the ligands, as shown by IR, ^{1}H and ^{13}C NMR spectral evidences and the appearance of ^{29}Si NMR chemical shift values in the region δ (-)19 to (-)20 ppm indicates a penta-coordination around central silicon atom[19–22]. On the basis of above evidences the following structure may be assigned to these diorganosilicon (IV) chelates.



Where $R = R' = -CH_3$ & $R'' = -CH_3$ (chelate No.1)
$R = -CH_3$, $R' = -C_6H_5$ & $R'' = -CH_3$ (chelate No.2)
$R = R' = -CH_3$ & $R'' = -C_2H_5$ (chelate No.3)
$R = -CH_3$, $R' = -C_6H_5$ & $R'' = -C_2H_5$ (chelate No.4)
$R = R' = -CH_3$ & $R'' = -C_6H_5$ (chelate No.5)
$R = -CH_3$, $R' = -C_6H_5$ & $R'' = -C_6H_5$ (chelate No. 6)
$R = R' = -CH_3$ & $R'' = -ClC_6H_4$ (chelate No.7)
$R = -CH_3$, $R' = -C_6H_5$ & $R'' = -ClC_6H_4$ (chelate No.8)

In the above structure, central silicon atom acquires five coordination by utilising O, S and N atoms of the ligand moiety.

EXPERIMENTAL

The reactions were carried out under anhydrous conditons. All the solvents were dried by standard methods. Dimethyldichlorosilane and methylphenyldichlorosilane were distilled before use. Sulphur was estimated by Messengers method[23]. Molecular weights were determined osmometrically on knaur vapour pressure osmometer in chloroform solution at 45°C. IR spectra were recorded as KBr pellets in the range 4000–400 cm^{-1} on a Nicolet Magna 550 FTIR spectrometer. ^{1}H, ^{13}C & ^{29}Si NMR spectra were recorded on a Jeol FX-90Q spectrometer in $CDCl_3$and $CHCl_3$ solution using TMS as standard reference, respectively. 4-(2'-mercapto phenyl imino alkyl/aroyl)-2,4-dihydro-5-methyl-2- phenyl-3H-pyrazol-3-ones were prepared by the method reported earlier[11]. Since the synthesis of organosilicon chelates derived from the above mentioned molecules follows the same procedure, only one complex will be described in detail.

Synthesis of [4-(2′- mercapto phenyl iminobenzyl)-2,4 – dihydro-5-methyl-2-phenyl-3H-pyrazol-3-onato) dimethylsilicon (IV)

The above mentioned complex was synthesized by two different reaction routes and therefore both the synthetic routes have been described in detail.

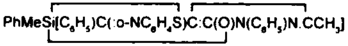
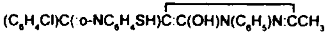
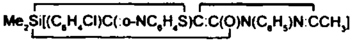
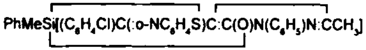
a) A weighed amount of sodium (0.138 gm, 0.60 m mol) was dissolved in dry methanol (~5 ml) and to this a benzene solution of 4-(2′-mercaptophenyl iminobenzyl)-2,4-dihydro-5-methyl-2-phenyl-3H-pyrazol-3-one (1.16 gm, 0.30 m mol) was added dropwise with constant stirring. The reaction mixture was refluxed for ~ 4 hours and then cooled. To this, a benzene solution of Me_2SiCl_2 (0.39 gm, 0.30 m mol) was added dropwise with constant stirring and after the addition was complete, the reaction mixture was refluxed for ~ 4 hours. After filtering off the precipitated NaCl, the volatile fraction was stripped off under reduced pressure and a dark green coloured solid product was obtained. This was recrystallised from $CHCl_3$/pet. ether mixture in 70% yield.
m.p = 63°C, %S found (Calcd) = 7.25 (7.26).

b) A benzene solution of 4-(2′-mercapto phenyl imino benzyl)-2,4-dihydro-5-methyl-2-phenyl-3H-pyrazol-3-one (1.17 gm, 0.30 m mol) and triethylamine (0.62 gm, 0.60 m mol) was added dropwise through a dropping funnel into the benzene solution of Me_2SiCl_2 (0.39 gm, 0.30 m mol) contained in a round bottom flask with constant stirring. The reaction mixture was refluxed for ~4 hours. The precipitated $Et_3N.HCl$ was filtered off and the filterate was dried under reduced pressure. A dark green coloured solid product was obtained which was recrystallised from $CHCl_3$/pet. ether mixture in 68% yield.

Analogous organosilicon (IV) complexes of Schiff bases derived from substituted pyrazolones have been prepared by following one of the above routes and therefore for brevity, the synthetic and analytical details of these complexes have been summarised in table III.

TABLE III The synthetic and analytical data of organosilicon (IV) chelates derived from 4-(2′-Mercapto phenyl imino alkyl/aroyl)-2,4-dihydro-5-methyl-2-phenyl-3H-pyrazol-3-ones

S.No.	*Compound*	*Colour/m.p.(°C)/% Yield*	*Molecular weight found (calcd.)*	*NaCl found (calcd.)*	*% S found (calcd.)*
	$\mathrm{(CH_3)C(:o\text{-}NC_6H_4SH)C:C(OH)N(C_6H_5)N:CCH_3}$	Light Yellow/42/72	330.00 (323.43)	– –	9.88 (9.92)
1.	$\mathrm{Me_2Si[(CH_3)C(:o\text{-}NC_6H_4S)C:C(O)N(C_6H_5)N:CCH_3]}$	Dark Green/150/75	380.00 (379.55)	0.30 (0.30)	8.44 (8.45)
2.	$\mathrm{PhMeSi[(CH_3)C(:o\text{-}NC_6H_4S)C:C(O)N(C_6H_5)N:CCH_3]}$	Dark Green/80/75	440.00 (441.62)	0.22 (0.23)	8.12 (8.15)
	$\mathrm{(C_2H_5)C(:o\text{-}NC_6H_4SH)C:C(OH)N(C_6H_5)N:CCH_3}$	Yellow/85/68	340.00 (337.44)	– –	9.50 (9.50)
3.	$\mathrm{Me_2Si[(C_2H_5)C(:o\text{-}NC_6H_4S)C:C(O)N(C_6H_5)N:CCH_3]}$	Dark Green/78/70	400.00 (393.58)	0.32 (0.35)	7.25 (7.26)
4.	$\mathrm{PhMeSi[(C_2H_5)C(:o\text{-}NC_6H_4S)C:C(O)N(C_6H_5)N:CCH_3]}$	Dark Green/85/75	450.00 (455.65)	0.40 (0.40)	6.82 (6.86)
	$\mathrm{(C_6H_5)C(:o\text{-}NC_6H_4SH)C:C(OH)N(C_6H_5)N:CCH_3}$	Dark Yellow/80/74	380.00 (385.48)	– –	8.30 (8.32)
5.	$\mathrm{Me_2Si[(C_6H_5)C(:o\text{-}NC_6H_4S)C:C(O)N(C_6H_5)N:CCH_3]}$	Light Green/63/65	440.00 (441.62)	0.25 (0.25)	7.26 (7.26)

S.No.	*Compound*	*Colour/m.p.(°C)/% Yield*	*Molecular weight found (calcd.)*	*NaCl found (calcd.)*	*% S found (calcd.)*
6.	 $PhMeSi[C_6H_5)C(:o\text{-}NC_6H_4S)C:C(O)N(C_6H_5)N.CCH_3]$	Green Solid/90/70	500.00 (503.69)	0.22 (0.23)	8.12 (8.15)
	 $(C_6H_4Cl)C(:o\text{-}NC_6H_4SH)C:C(OH)N(C_6H_5)N:CCH_3$	Dark Yellow/70/78	420.00 (419.93)	– –	7.62 (7.64)
7.	 $Me_2Si[(C_6H_4Cl)C(:o\text{-}NC_6H_4S)C:C(O)N(C_6H_5)N:CCH_3]$	Dark Green/68/65	480.00 (476.07)	0.40 (0.43)	6.30 (6.37)
8.	 $PhMeSi[(C_6H_4Cl)C(:o\text{-}NC_6H_4S)C:C(O)N(C_6H_5)N:CCH_3]$	Light Green/85/70	540.00 (538.14)	0.30 (0.31)	5.90 (5.96)

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