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Monoorganosilicon(IV) Complexes of 2-Aminocyclopentene-1-carbodithioic Acid and its *N*-/*S*-Alkyl Derivatives: Synthesis and Characterization

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2-Aminocyclopentene-1-carbodithioic acid (ACDA) and its *N*-/*S*-alkyl derivatives of methylsilicon(IV) chloride have been synthesized by the reactions of $MeSiCl_3$ with sodium salt of ACDA and its *N*-/*S*-alkyl derivatives in 1:1 and 1:2 molar ratios. All these complexes have been characterized by elemental analysis, molecular weight measurements and spectroscopic (IR, ^{1}H and ^{13}C NMR) studies. The central silicon atom is coordinated to two sulphur atoms or one sulphur atom and one nitrogen atom of the ligand moiety in all of these derivatives, resulting in five and six coordination.

Keywords: Methylsilicon(IV) chloride, 2-Aminocyclopentene-1-carbodithioic acid (ACDA), *N*-/*S*-Alkyl derivatives.

INTRODUCTION

In an extensive search for a suitable "single source" precursor for sulphides, number of complexes are known with organotin(IV) [1,2], organogermanium(IV) [3], bismuth(III) [4] as well as some transition metals such as Ni(II) [5], Cu(II) [6], Co(II) [7], Cd(II) [8] with 2-aminocyclopentene-1-carbodithioic acid (ACDA) ligand. Recently, these metal complexes have been used to prepare nanomaterials for different applications. Bismuth sulphide (Bi_2S_3) nanoparticles [4] exhibit high electrocatalytic activity toward reduction of H_2O_2 and is used for amperometric sensing and estimation of H_2O_2. CdS nanoparticles [8] have been used in the degradation of aqueous Rose Bengal solution and nano- structured Ni(II)/ACDA film [5] deposited on a gold nano-particles-cysteine-gold electrode which is used in the oxidation of tryptophan.

2-Aminocyclopentene-1-carbodithioic acid (ACDA) ligand and its N-/S-alkyl derivatives constitute interesting class of nitrogen and sulphur containing bidentate ligands owing to their biological activity [9-11] and exhibit remarkable diversity in coordination patterns [12-16]. Coordination of ligand to metal is either through S-, S- or through N-, S- mode. X-ray crystal structure of some complexes are reported with Bi(III), diorganotin(IV), La(III) [17] and Co(II) [7] where coordination is through S-, S- whereas in other cases DFT study [17] and *ab initio* calculations [12] were carried out to study the coordination properties of some transition metal complexes.

In continuation of our previous synthesis of di- and tri-organosilicon [18,19] derivatives of ACDA, the structural versatility of the ligand and the preparation of nanoparticles motivate us to prepare monoorganosilicon(IV) derivatives. In this work, the synthesis and structural characterization (elemental analysis, molecular weight measurement and ^{1}H, ^{13}C, IR spectroscopic studies) of dichloromethylsilicon(IV) derivatives of ACDA and chloromethylsilicon(IV) *bis*(ACDA) derivatives and their *N*-/*S*-alkyl derivatives are reported.

EXPERIMENTAL

All the manipulations were carried out under anhydrous conditions and the chemicals used were of reagent grade. Cyclopentanone (b.p. 130 ºC) and $MeSiCl_3$ (b.p. 66 ºC) were distilled before use. All the solvents were made anhydrous by standard methods. The ligand, 2-aminocyclopentene-1-carbodithioic acid (ACDA) and its *N*-/*S*-substituted derivatives were synthesized by the literature method [6]. Elements (Si, N and S) were analyzed according to reported method [20]. Molecular weight measurements were carried out on a Knauer

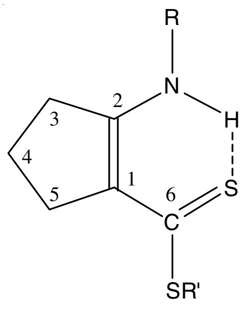
Vapour Pressure Osmometer in chloroform at 45 °C. IR spectra were recorded in Nujal mull in the range 4000-200 cm^{-1} on a Perkin-Elmer 577 spectrophotometer. The ^{1}H & ^{13}C NMR spectra were recorded in $CDCl_3$ and $CHCl_3$ solutions, respectively on a JEOL FX 90Q spectrometer using TMS as an internal reference.

Since all these silicon complexes have been synthesized by a similar route, the synthesis of one representative complex is described below and the synthetic details and analytical data of the analogous complexes are summarized in Table-1.

Synthesis of dichloromethylsilicon(IV)(2-aminocyclopentene-1-carbodithioic acid)-MeSi(ACDA)Cl_2: A weighed amount of sodium (0.12 g, 5.21 mmol) was reacted with anhydrous methanol (~ 10 mL) then ACDA (0.83 g, 5.21 mmol) was added. The reaction mixture was refluxed for ~4 h. To this solution, a benzene solution of $MeSiCl_3$ (0.78 g, 5.22 mmol) was added dropwise with constant stirring. The stirring was continued for ~ 4 h and the finally refluxed for ~ 3 h to ensure the completion of the reaction. The precipitate was filtered off and solvent removed under the reduced pressure from the filtrate to yield a brown sticky solid compound. The product was purified by dissolving in chloroform followed by the addition of *n*-hexane. Yield and elemental analysis are reported in Table-1.

RESULTS AND DISCUSSION

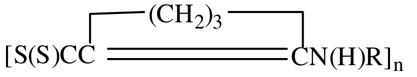
The following tautomer (Fig. 1) appears to predominate in the solution among the several probable tautomeric forms of 2-aminocyclopentene-1-carbodithioic acid and its *N*-/*S*-derivatives.



α

Fig. 1. Tautomer of ACDA; where R′ = H and R = H (HACDA), CH_3 (HMeACDA), C_2H_5 (HEtACDA), n-C_4H_9 (HBuACDA); R = H and R′ = CH_3(HACDE)

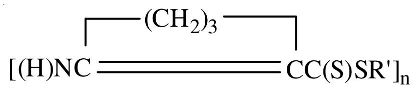
Methylsilicon((IV) complexes of the types $MeSiCl_{3-n}$



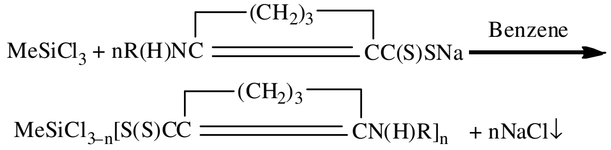
$[S(S)C\overset{(CH_2)_3}{C=C}N(H)R]_n$ and $MeSiCl_{3-n}$

TABLE-1
PHYSICO-ANALYTICAL DATA OF METHYLSILICON(IV) COMPLEXES OF
2-AMINOCYCLOPENTENE-1-CARBODITHIOIC ACID AND ITS *N*-/*S*-ALKYL DERIVATIVES

Reactants in g (mmol)			Product m.f. [compound number, % yield]	NaCl Found (calcd.)	Elemental analysis (%): Found (calcd.)				m.w. Found (calcd.)
Sodium	Ligand	Organosilicon chlorides			Si	N	S	Cl	
0.12 (5.21)	ACDA 0.83 (5.21)	$MeSiCl_3$ 0.78 (5.22)	$C_7H_{11}NS_2Cl_2Si$ [(**1**), 72%]	0.39 (0.30)	10.12 (10.31)	5.02 (5.14)	23.24 (23.55)	25.92 (26.04)	270 (272.29)
0.11 (4.78)	MeACDA 0.82 (4.73)	$MeSiCl_3$ 0.71 (4.75)	$C_8H_{13}NS_2Cl_2Si$ [(**2**), 74%]	0.26 (0.38)	9.70 (9.81)	4.62 (4.89)	22.14 (22.39)	24.58 (24.76)	284 (286.32)
0.14 (6.08)	EtACDA 1.14 (5.95)	$MeSiCl_3$ 0.90 (6.02)	$C_9H_{15}NS_2Cl_2Si$ [(**3**), 75%]	0.33 (0.35)	9.18 (9.35)	4.44 (4.66)	21.16 (21.35)	23.44 (23.61)	296 (300.35)
0.10 (4.35)	BuACDA 0.94 (4.36)	$MeSiCl_3$ 0.65 (4.35)	$C_{11}H_{19}NS_2Cl_2Si$ [(**4**), 70%]	0.23 (0.25)	8.36 (8.55)	4.14 (4.26)	19.28 (19.53)	21.34 (21.59)	285 (286.32)
0.12 (5.21)	ACDE 0.90 (5.19)	$MeSiCl_3$ 0.78 (5.22)	$C_8H_{13}NS_2Cl_2Si$ [(**9**), 74%]	0.27 (0.30)	9.64 (9.81)	4.66 (4.89)	22.12 (22.39)	24.58 (24.76)	323 (286.32)
0.14 (6.08)	ACDA 0.97 (6.09)	$MeSiCl_3$ 0.45 (3.01)	$C_{13}H_{19}N_2S_4ClSi$ [(**5**), 70%]	0.32 (0.35)	7.02 (7.11)	6.98 (7.09)	32.28 (32.46)	8.68 (8.97)	392 (395.11)
0.15 (6.52)	MeACDA 1.13 (6.42)	$MeSiCl_3$ 0.48 (3.21)	$C_{15}H_{23}N_2S_4ClSi$ [(**6**), 74%]	0.36 (0.38)	6.46 (6.63)	6.44 (6.62)	30.14 (30.30)	8.12 (8.37)	421 (423.16)
0.13 (5.65)	EtACDA 1.05 (5.60)	$MeSiCl_3$ 0.42 (2.81)	$C_{17}H_{27}N_2S_4ClSi$ [(**7**), 72%]	0.30 (0.33)	6.02 (6.22)	6.03 (6.20)	28.26 (28.42)	7.86 (7.85)	449 (451.22)
0.14 (6.08)	BuACDA 1.31 (6.08)	$MeSiCl_3$ 0.45 (3.01)	$C_{21}H_{35}N_2S_4ClSi$ [(**8**), 75%]	0.32 (0.35)	5.32 (5.54)	5.02 (5.52)	25.02 (25.28)	6.72 (6.99)	505 (507.32)
0.12 (5.21)	ACDE 0.90 (5.19)	$MeSiCl_3$ 0.39 (2.61)	$C_{15}H_{23}N_2S_4ClSi$ [(**10**), 74%]	0.29 (0.30)	6.46 (6.63)	6.46 (6.62)	30.14 (30.30)	8.14 (8.37)	425 (423.16)

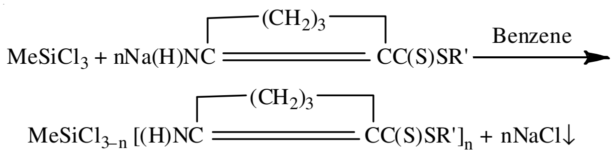


$[(H)N\overbrace{C=C}^{(CH_2)_3}C(S)SR']_n$ have been synthesized by the reaction of $MeSiCl_3$ with the sodium salt of ligands in different stoichiometric ratios (1:1 and 1:2 molar ratios) in refluxing benzene solution shown below by eqns. 1 and 2:



$$MeSiCl_3 + nR(H)N\overbrace{C=C}^{(CH_2)_3}C(S)SNa \xrightarrow{Benzene} MeSiCl_{3-n}[S(S)C\overbrace{C=C}^{(CH_2)_3}N(H)R]_n + nNaCl\downarrow \quad (1)$$

R= -H; n =1 (**1**) and n = 2 (**5**); R = $-CH_3$; n =1 (**2**) and n = 2 (**6**); R= $-C_2H_5$; n =1 (**3**) and n = 2 (**7**); R = $-nC_4H_9$; n =1 (**4**) and n = 2 (**8**).



$$MeSiCl_3 + nNa(H)N\overbrace{C=C}^{(CH_2)_3}C(S)SR' \xrightarrow{Benzene} MeSiCl_{3-n}[(H)N\overbrace{C=C}^{(CH_2)_3}C(S)SR']_n + nNaCl\downarrow \quad (2)$$

(**9**) $R' = CH_3$; n = 1; (**10**) $R' = CH_3$; n = 2

These complexes (**1-10**) were found to be brown sticky solids. They have low solubility in benzene, chloroform, THF *etc*. and monomeric in chloroform at 45 ºC.

Broad bands found at 2550 and 3400 cm^{-1} in the IR spectra of ACDA and its derivatives can be assigned to SH and NH/NH_2, respectively. The absence of the former and the preservation of the latter in methylsilicon (IV) complexes of ACDA and its *N*-alkyl derivatives (**1-8**) suggests that the SH group [21] is deprotonated. A new IR band appeared in the region 575-530 cm^{-1} (Table-2) could be attributed to the presence of silicon-sulphur bond [22,23]. The appearance of only one band in the region 950-920 cm^{-1} attributed to asymmetrical mode of vibrations of CSS shows that both sulphur atoms are involved in these complexes. The combination of NH_2 + C=C, CH_2 + C=C, C=N + C=S and C=S + C=N bonds can be attributed to the strong IR bands found in the region 1610-1600 cm^{-1}, 1505-1470 cm^{-1}, 1360-1340 cm^{-1} and 1280-1275 cm^{-1}, respectively. Even after complexation, the positions of all of these bands remain unchanged. A symmetrical CSS or C-S bond has been attributed to the band in the region 615-610 cm^{-1} in the ligands and associated organosilicon(IV) complexes. IR spectral data of all the derivatives (**1-10**) is reported in Table-2.

A shift towards lower wave number of ~70 cm^{-1} (Table-2) for NH_2 group in *S*-alkyl-2-aminocyclopentene-1-carbodithioic acid (ACDE) derivatives demonstrates the participation of the amino nitrogen of the ligand moiety in the bonding. This is further supported by the formation of a new band at 580-560 cm^{-1} that could be attributed to ν(Si-N), as well as a downfield shift (of ~10 cm^{-1}) in the position of the ν(C=S + C=N) absorption bands. The presence of two bands for asymmetric CSS at 960-950 cm^{-1} and 925-920 cm^{-1} implies that one of the sulphur atoms is involved in the chelation process. The development of a new band at 560-540 cm^{-1} supports ν(Si-S) bonding.

Disappearance of a strong singlet for -SH proton [21] in the ^1H NMR spectra of ACDA and its *N*-alkyl derivatives indicate the removal of -SH proton on complexation and formation of silicon-sulphur bonds (Table-3). The non-equivalence nature of the amino group protons is demonstrated by two broad signals assigned at δ 11.2 and 7.13 ppm in the ^1H NMR spectrum of ACDA ligand. In the ^1H NMR spectra of the corresponding complexes, these signals shift downfield and present in the region δ 8.69-8.98 ppm and δ 7.69-7.72 ppm, respectively. This could be because the hydrogen bond [24] (N-H···S=) weakens during complexation. The protons attached to the ring carbon atoms [$-CH_2(4)$] and [$-CH_2(3\&5)$] (Fig. 1) in the complexes appear as quintet and multiplet in the regions δ 1.63-2.06 ppm and δ 2.50-2.82 ppm, respectively. The alkyl group's protons bound to nitrogen appear in their normal places.

The ^1H NMR signal observed at δ11.2 ppm for proton attached to nitrogen found in the free ligand was absent in the methylsilicon(IV) complexes of *S*-alkyl derivatives of ACDA (ACDE) (**9-10**). The free -NH signal in the ligand at δ 5.81 ppm is shifted in the region δ 6.17-6.18 ppm in complexes, indicate the absence of one of the proton from the amino group on complex formation. The participation of the -C=S group in bonding is further supported by a downfield shift in the position of the $S-CH_3$ signal (δ 2.64 ppm in free ligand). The methyl protons bonded to the core silicon atom were observed as a singlet at δ 0.17-0.19 ppm.

The ^{13}C NMR signal for C_6 carbon (Table-4) occurs at δ 189.07-196.12 ppm in free ligand [21], shifts downfield and

TABLE-2
IR SPECTRAL DATA OF METHYLSILICON(IV) COMPLEXES OF 2-AMINOCYCLOPENTENE-1- CARBODITHIOIC ACID AND ITS *N*-/*S*-ALKYL DERIVATIVES

Compound No.	$\nu NH/NH_2$	νNH_2 + C=C	νCH_2 + C=C	νC=N + C=S	νC=S + C=N	$\nu_{asym}CSS$	$\nu_{sym}CSS$	ν(Si-Cl)	ν(Si-S)/ and ν(Si-N)
1	3280b	1610s	1470s	1340s	1280s	930m	610b	500s	530s
2	3440b	1610s	1500s	1350s	1280s	940m	610b	515s	545s
3	3445b	1600s	1505s	1360s	1275s	935m	615b	525s	560s
4	3440b	1600s	1500s	1345s	1280s	950m	610b	520s	575s
9	3190b	1605s	1470s	1250s	1140s	960m, 920m	620b	540s	560s and 580s
5	3280b	1600s	1475s	1360s	1280s	920m	615b	500s	530s
6	3435b	1610s	1490s	1350s	1275s	930m	610b	510s	540s
7	3440b	1610s	1500s	1360s	1280s	925m	610b	510s	535s
8	3440b	1600s	1500s	1360s	1280s	920m	615b	515s	540s
10	3190b	1610s	1474s	1350s	1150s	950m, 925m	620b	530s	540s/ and 560s

TABLE-3
^{1}H NMR SPECTRAL DATA OF METHYLSILICON(IV) DERIVATIVES OF
2-AMINOCYLOPENTENE-I-CARBODITHIOIC ACID AND ITS *N*-/*S*-ALKY DERIVATIVES (δ) ppm

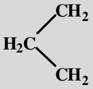
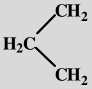
Compound No.			>NH (free)	>NH (chelated)	>N-R	-SCH$_3$	Si-R
1	1.72-2.01 (q) (2H) - CH$_2$ (4)	2.59-2.85 (m) (4H) -CH$_2$ (3&5)	7.69 (bs) (1H)	8.98 (bs) (1H)	–	–	0.19 (s)
2	1.70-1.99 (2H) -CH$_2$ (4)	2.61-2.90 (m) (4H) -CH$_2$ (3&5)	–	9.42 (bs) (1H)	3.24 (s) (3H)-CH$_3$ (7)	–	0.19 (s)
3	1.74-2.02 (q) (2H)-CH$_2$ (4)	2.68-2.99 (m) (4H) -CH$_2$ (3&5)	–	9.60 (1H)	3.40-3.74 (m) (2H)-CH$_3$ (7) 1.24-1.46 (t) (3H)-CH$_3$ (8)	–	0.17 (s)
4	1.68-2.01 (q) (2H) -CH$_2$ (4)	2.58-2.99 (m) (2H) -CH$_2$ (3&5),	–	10.88 (bs) (1H)	3.29-3.59 (m) (2H)-CH$_2$ (7) 1.28-1.68 (m) (4H)-CH$_2$ (8&9)	–	0.17 (s)
9	1.63-2.04 (q) (2H) -CH$_2$ (4)	2.50-2.82 (m) (4H) -CH$_2$ (3&5)	6.18 (bs) (1H)	–	–	2.69 (s) (3H) - CH$_3$ (α)	0.17 (s)
5	1.71-1.96 (q) (2H) -CH$_2$ (4)	2.56-2.83 (m) (4H) -CH$_2$ (3&5)	7.72 (bs) (1H)	8.69 (bs) (1H)	–	–	0.17 (s)
6	1.72-1.98 (q) (2H) -CH$_2$ (4)	2.59-2.89 (m) (4H) -CH$_2$ (3&5)	–	9.38 (bs) (1H)	3.26 (s) (3H)-CH$_3$ (7)	–	0.17 (s)
7	1.74-1.98 (q) (2H) -CH$_2$ (4)	2.63-2/85 (m) (4H) -CH$_2$ (3&5)	–	9.89 (bs) (1H)	3.34-3.61 (m) (2H)-CH$_2$ (7) 1.26-1.44 (t) (3H)-CH$_3$ (8)	–	0.17 (s)
8	1.70-1.96 (q) (2H) -CH$_2$ (4)	2.61-2.87 (m) (4H) -CH$_2$ (3&5)	–	10.78 (bs) (1H)	3.25-3.58 (m) (2H)-CH$_2$ (7) 1.27-1.70 (m) (4H)-CH$_2$ (8&9) 0.89-1.02 (t) (3H) -CH$_3$ (10)	--	0.17 (s)
10	1.67-2.06 (q) (2H) -CH$_2$ (4)	2.52-2.86 (m) (4H) -CH$_2$ (3&5)	6.17 (bs) (1H)	–	–	2.68 (s) (3H) - CH$_3$ (α)	0.17 (s)

TABLE-4
^{13}C NMR DATA OF METHYLSILICON(IV) DERVATIVES OF 2-AMINOCYCLOPENTENE-1-CARBODITHIOIC ACID AND ITS *N*-/*S*-ALKY DERIVATIVES (δ) ppm

Compd. No.	C_1	C_2	C_3	C_4	C_5	C_6	C_7	C_8	C_9	C_{10}	C_α	Me-Si
1	110.86	166.93	36.13	19.86	31.54	201.97	–	–	–	–	–	(-)5.03
2	117.52	170.04	35.87	19.82	33.62	198.99	32.66	–	–	–	–	(-)5.04
3	118.61	170.30	34.58	19.88	33.84	198.80	42.32	14.70	–	–	–	(-)5.04
4	119.14	169.87	35.68	20.12	33.03	199.81	44.89	31.42	20.01	13.72	–	(-)5.04
9	118.12	167.18	36.30	20.48	32.68	206.88	–	–	–	–	18.14	(-)4.98
5	112.92	170.15	36.13	19.98	31.54	202.51	–	–	–	–	–	(-)5.09
6	117.58	170.66	35.84	19.86	33.59	199.89	32.58	–	–	–	–	(-)5.14
7	118.49	170.71	33.82	20.04	31.31	200.04	45.84	15.27	–	–	–	(-)5.14
8	119.58	170.98	35.62	19.99	32.86	201.18	44.96	31.48	20.14	13.54	–	(-)5.14
10	118.14	167.16	36.32	20.46	32.68	207.92	–	–	–	–	18.16	(-)4.73

is observed in the region of δ 198.80-202.51 ppm in methylsilicon (IV) complexes of ACDA and its *N*-alkyl derivatives (**1-8**). The deprotonation of one thiol group and coordination through the other sulphur atom may be accountable for the deshielding of the carbon atom. The complexation has no effect on the other ligand carbon atoms, as their positions in the spectra of the complexes remain unchanged. At (-)4.75 – (-)5.14 ppm, the methyl carbon bonded to the silicon atom can be seen.

Signals recorded at δ 166.15, 204.82 and 17.01 ppm for C_2, C_6 and C_α carbon atoms move downfield in the complexes (**9-10**) and appear in the region δ167.16-167.18, 206.88-207.92 and 18.14-1816 ppm, respectively, in the ^{13}C spectra of ACDE. These findings support the inclusion of nitrogen, sulphur and silicon atoms in a six-membered ring.

The poor solubilities of these complexes (**1-10**) prevented the recording of ^{29}Si NMR spectra. It is impossible to ascribe the conclusive structures to them in the absence of X-ray crystallographic evidence due to their low solubilities. The following structures (Figs. 2-5) for methyldichlorosilicon(IV)

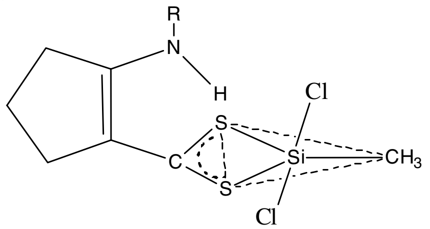


Fig. 2. Suggested structure for the derivatives (**1-4**); (R = -H, -CH$_3$, -C$_2$H$_5$, -nC$_4$H$_9$)

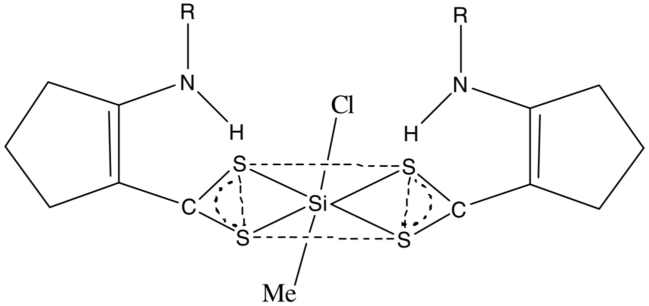


Fig. 3. Suggested Structure for the derivatives (**5-8**); (R = -H, -CH$_3$, -C$_2$H$_5$, -nC$_4$H$_9$)

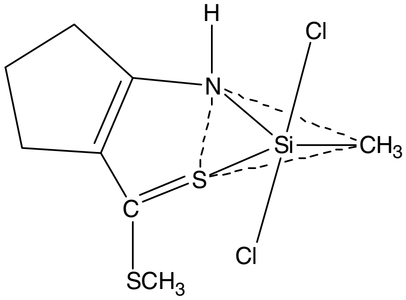


Fig. 4. Structural assignment of the derivative (**9**)

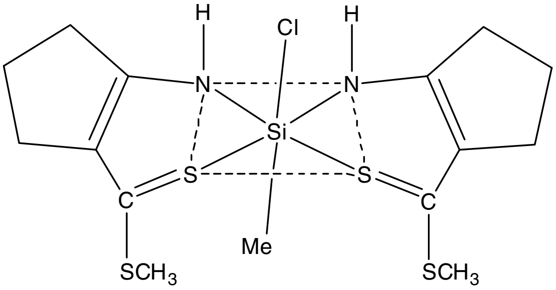


Fig. 5. Structural assignment of the derivative (**10**)

and methylchlorosilicon(IV) complexes of ACDA and its *N*-/*S*-alkyl derivatives may be proposed based on yield of the products and sodium chloride, molecular weight measurements, IR and NMR spectra.

CONFLICT OF INTEREST

The authors declare that there is no conflict of interests regarding the publication of this article.

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