Azomethine Derivatives. Part XVI.[1] Some Diphenylmethyleneaminosilanes and Di-t-butylmethyleneaminosilanes

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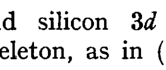
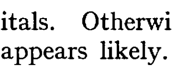
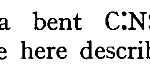
Reactions between the chlorosilanes, Me_nSiCl_{4-n} (n = 0—3), and di-t-butylmethyleneaminolithium, $Bu^t_2C:NLi$, afford the mono(alkylideneamino)silanes, $Bu^t_2C:NSiMe_nCl_{3-n}$, even when an excess of $Bu^t_2C:NLi$ is used. These alkylideneaminosilanes have i.r. and ^1H n.m.r. spectra consistent with linearity of their C:NSi skeletons, as appropriate for maximum N$\rightleftharpoons$Si ($p \longrightarrow d$) π-bonding. Analogous reactions between equimolar proportions of Me_nSiCl_{4-n} (n = 0—2) and $Ph_2C:NLi$ afford the di(alkylideneamino)silanes $(Ph_2C:N)_2SiMe_nCl_{2-n}$, apparently through disproportionation of the mono-substituted compounds $Ph_2C:NSiMe_nCl_{3-n}$ which could be isolated only when n = 2. The spectra of the diphenylmethyleneaminosilanes show that these may have bent C:NSi skeletons.

EARLIER studies[2,3] of alkylideneaminosilanes $(R^1R^2C:N)_nSiR_{4-n}$ raised, but did not resolve, the question of whether such derivatives contain appreciable N$\rightleftharpoons$Si ($p \longrightarrow d$) dative π-bonding. A linear C=N$\rightleftharpoons$Si molecular skeleton, as in (I), is to be expected if N$\rightleftharpoons$Si π-interactions are important, as this geometry ensures maximum overlap of the appropriate nitrogen $2p$ and silicon $3d$ orbitals. Otherwise, a bent C:NSi skeleton, as in (II), appears likely. We here describe


(I)


(II)


(III)

some new di-t-butylmethyleneaminosilanes $Bu^t_2C:NSiMe_nCl_{3-n}$ (n = 0—3) the ^1H n.m.r. and i.r. spectra of

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[1] Part XV, M. R. Collier, M. F. Lappert, R. Snaith, and K. Wade, *J.C.S. Dalton*, 1972, 370.

[2] L.-H. Chan and E. G. Rochow, *J. Organometallic Chem.*, 1967, **9**, 231.

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which are consistent with linear skeletons. We also report our attempts to synthesise some related diphenylmethyleneaminosilanes $Ph_2C{:}NSiMe_nCl_{3-n}$ ($n = 0$—2), which apparently differ markedly from their di-t-butyl analogues in two respects; in being unstable to disproportionation into bisalkylideneamino-derivatives $(Ph_2C{:}N)_2SiMe_nCl_{2-n}$, and possibly also in their skeletal shapes.

The di-t-butylmethyleneaminosilanes $Bu^t{}_2C{:}NSiMe_nCl_{3-n}$ were prepared by reactions between the appropriate methylchlorosilane Me_nSiCl_{4-n} and an equimolar proportion of di-t-butylmethyleneaminolithium in boiling hexane. The solution was normally kept boiling for about 20 h to ensure as near complete precipitation of lithium chloride as possible [equation (1)]:

$$Bu^t{}_2C{:}NLi + Me_nSiCl_{4-n} \longrightarrow Bu^t{}_2C{:}NSiMe_nCl_{3-n} + LiCl \qquad (1)$$

The products were moisture-sensitive pale yellow or colourless liquids which could be purified by vacuum distillation below 100 °C; their volatilities increase as n increases (see Experimental section for details). Attempts to prepare bis-, tris- and tetrakis-(di-t-butylmethyleneamino)silanes, $(Bu^t{}_2C{:}N)S_xiMe_nCl_{4-n-x}$ ($x = 2$, 3, or 4), by use of correspondingly greater proportions of di-t-butylmethyleneaminolithium [equation (2)] and by use of more vigorous reaction conditions, *e.g.* heating the reactants together in boiling toluene for a day, were unsuccessful, the mono(alkylideneamino)silane $Bu^t{}_2C{:}NSiMe_nCl_{3-n}$ being the only alkylideneaminosilane isolated in each case:

$$xBu^t{}_2C{:}NLi + Me_nSiCl_{4-n} \not\longrightarrow (Bu^t{}_2C{:}N)_xSiMe_nCl_{4-n-x} + xLiCl \qquad (2)$$
$$(x = 2,\ 3,\ \text{or}\ 4)$$

The azomethine stretching absorption, ν(C=N), in the i.r. spectra of the di-t-butylmethyleneaminosilanes $Bu^t{}_2C{:}NSiMe_nCl_{3-n}$ occurs at *ca.* 1730 cm^{-1} (see Table 1), some 120 cm^{-1} higher in frequency than ν(C=N) for the parent imine, $Bu^t{}_2C{:}NH$. A marked increase in ν(C=N) on replacing the imino hydrogen of imino-compounds $R^1R^2C{:}NH$ by a group MX_n has been taken elsewhere to indicate probable linearity of the C:NM skeleton of alkylideneamino-derivatives of such elements as boron,[1,3-7] aluminium,[8] and beryllium[9] [justifiably in the case of the compounds $mesityl_2BN{:}CPh_2$[10] and $LiAl(N{:}CBu^t{}_2)_4$,[11] in which essentially linear C:NM units have been found by *X*-ray crystallography]. It therefore appears likely that the present di-t-butylmethyleneaminosilanes $Bu^t{}_2C{:}NSiMe_nCl_{3-n}$ have linear $C{=}N{\rightleftharpoons}Si$ skeletons as in (I). The frequencies of the bands assignable to the Si–N stretching vibrations (*ca.* 960 cm^{-1}), which too are listed in Table 1, are also consistent with linearity of the C:NSi skeletons.

Support for structure (I) for the di-t-butylmethyleneaminosilanes is provided by their ^{1}H n.m.r. spectra, which were recorded at various temperatures using *ca.* 20 wt. % solutions in toluene. In each of these spectra the t-butyl protons give rise to a single sharp absorption at +33 °C which does not change significantly in shape or chemical shift when the solution is cooled to −60 °C (for details see Table 2). By contrast, the ^{1}H n.m.r. spectrum of di-t-butylmethyleneamine, $Bu^t{}_2C{:}NH$, in toluene at −60 °C has two peaks of equal intensity attributable to the magnetically distinct t-butyl groups in its bent structure (III). We conclude that the t-butyl groups of our di-t-butylmethyleneaminosilanes are probably magnetically equivalent, as in structure (I); a less likely alternative is that a bent structure (II), containing two types of t-butyl group, is inverting at nitrogen so rapidly at −60° as to render all the t-butyl protons apparently equivalent. Our findings may be compared with those of Martin and his co-workers,[13] who were unable to distinguish geometrical isomers of the compound $PhOC(NEt_2){:}NSiMe_3$, using ^{1}H n.m.r. spectra of solutions at −97°.

TABLE 1

Skeletal stretching frequencies (cm^{-1}) of some methyleneaminosilanes

Compound	ν(C=N)	ν(Si–N)	Compound	ν(C=N)	ν(Si–N)
$Bu^t{}_2C{:}NSiMe_3$	1735	959	$Ph_2C{:}NSiMe_3$	1642	907
$Bu^t{}_2C{:}NSiMe_2Cl$	1736	965	$Ph_2C{:}NSiMe_2Cl$	1660	907
$Bu^t{}_2C{:}NSiMeCl_2$	1736	966	$(Ph_2C{:}N)_2$-SiMeCl	1645, 1630	907
$Bu^t{}_2C{:}NSiCl_3$	1729	967	$(Ph_2C{:}N)_2SiCl_2$	1656	913
			$(Ph_2C{:}N)_2SiMe_2$	1634	905

TABLE 2

^{1}H N.m.r. spectroscopic results for the di-t-butylmethyleneaminosilanes $Bu^t{}_2C{:}NSiMe_nCl_{3-n}$ (as *ca.* 20 wt. % solutions in toluene at +33 °C)

Compound	τ values (relative intensities in parentheses)
$Bu^t{}_2C{:}NSiMe_3$	8·88(2), 9·79(1)
$Bu^t{}_2C{:}NSiMe_2Cl$	8·84(3), 9·52(1)
$Bu^t{}_2C{:}NSiMeCl_2$	8·88(6), 9·40(1)
$Bu^t{}_2C{:}NSiCl_3$	8·86

It is interesting that neither the chemical shift of the t-butyl resonance in the ^{1}H n.m.r. spectra, nor the frequency of the azomethine stretching absorption in the i.r. spectra of the di-t-butylmethyleneaminosilanes $Bu^t{}_2C{:}NSiMe_nCl_{3-n}$, varies appreciably with n.

The mass spectra of all the di-t-butylmethyleneaminosilanes were recorded. Details for the compound $Bu^t{}_2C{:}NSiMeCl_2$ which illustrate the type of fragmenta-

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tion processes shown by all these compounds, are in Table 3. In no case was a parent peak attributable to the ion $[\mathrm{Bu^t{}_2C{:}NSiMe}_n\mathrm{Cl}_{3-n}]^+$ observed. Initial fragmentation apparently involved loss of a methyl group or a chlorine atom. The imino-residue tended to lose a butyl group initially, and subsequently C_4H_8 (probably isobutene), affording ions $[\mathrm{BuCNSiMe}_n\mathrm{Cl}_{3-n}]^+$ and $[\mathrm{HCNSiMe}_n\mathrm{Cl}_{3-n}]^+$ which in turn afforded Si–N

TABLE 3

Mass spectroscopic results for $\mathrm{Bu^t{}_2C{:}NSiMeCl_2}$

m/e	Relative intensity	Assignment
238	1	$\mathrm{Bu_2CNSiCl_2}$
218	5	$\mathrm{Bu_2CNSiMeCl}$
196	64	$\mathrm{BuCNSiMeCl_2}$
140	60	$\mathrm{HCNSiMeCl_2}$
113	27	$\mathrm{MeSiCl_2}$
84	38	BuCNH
63	8	SiCl
57	100	$\mathrm{Bu^t}$
41	88	MeCN
36	5	HCl
27	18	HCN
15	4	Me

In both this Table and Table 4, *m/e* values for chlorine-containing ions relate to ^{35}Cl isotopes, and for silicon-containing ions to ^{28}Si isotopes.

obtained as an oil from the reaction between $\mathrm{Ph_2C{:}NLi}$ and $\mathrm{Me_2SiCl_2}$ below room temperature. It disproportionated during several days at 15—20°, and much more rapidly above about 50°, depositing $\mathrm{(Ph_2C{:}N)_2SiMe_2}$ as a yellow solid.

The stability of the compounds $\mathrm{Ph_2C{:}NSiMe}_n\mathrm{Cl}_{3-n}$ to disproportionation apparently decreases as n decreases; it was not found possible to isolate samples of either $\mathrm{Ph_2C{:}NSiMeCl_2}$ or $\mathrm{Ph_2C{:}NSiCl_3}$ even from reaction mixtures which had not been allowed to warm above 15 °C; the products obtained were the new bis(diphenylmethyleneamino)silanes $\mathrm{(Ph_2C{:}N)_2SiMeCl}$ and $\mathrm{(Ph_2C{:}N)_2SiCl_2}$.

The i.r. spectra of these diphenylmethyleneaminosilanes differ from those of the di-t-butylmethyleneaminosilanes in two important respects (see Table 1 for details). The absorptions attributable to both the C=N and the Si–N skeletal stretching vibrations occur at significantly lower frequency than the corresponding absorptions of the di-t-butylmethyleneaminosilanes. The parent imines, $\mathrm{Bu^t{}_2C{:}NH}$ and $\mathrm{Ph_2C{:}NH}$, have very similar azomethine stretching frequencies (1610 and 1607 cm^{-1} respectively [14]), so it appears possible that the two sets of imino-derivatives differ in geometry, the diphenylmethyleneamino compounds adopting nonlinear

TABLE 4

Mass spectroscopic results for $\mathrm{(Ph_2C{:}N)_2SiMeCl}$

m/e	Relative intensity	Assignment
438	2	$\mathrm{(Ph_2CN)_2SiMeCl}$
403	<1	$\mathrm{(Ph_2CN)_2SiMe}$
388	<1	$\mathrm{(Ph_2CN)_2Si}$
361	2	$\mathrm{(Ph_2CN)(PhCN)SiMeCl}$
293	48	$\mathrm{Ph_2CNSiMeCl_2}$
278	4	$\mathrm{Ph_2CNSiCl_2}$
258	12	$\mathrm{Ph_2CNSiMeCl}$
216	100	$\mathrm{PhCNSiMeCl_2}$
181	23	$\mathrm{PhCNSiMeCl}$
180	50	$\mathrm{Ph_2CN}$
175	7	$\mathrm{PhSiCl_2}$
155	22	$\mathrm{PhSiMeCl}$
154	3	$\mathrm{Ph_2}$
113	95	$\mathrm{SiMeCl_2}$
103	11	PhCN
98	3	$\mathrm{SiCl_2}$
77	50	Ph
63	28	SiCl
43	1	SiMe

of the group $\mathrm{Ph_2C{:}N}$, involving essentially successive loss of phenyl groups, is simpler than fragmentation of $\mathrm{Bu^t_2C{:}N}$, in which elimination of olefin is important.

EXPERIMENTAL

Materials and Techniques.—Chlorosilanes were distilled before use. Diphenylmethyleneamine was prepared by methanolysis of the product of reaction between phenyl cyanide and phenylmagnesium bromide.[15] Its *N*-lithio-derivative was prepared in solution from equimolar proportions of diphenylmethyleneamine in ether and n-butyl-lithium in hexane. Di-t-butylmethyleneamino-lithium was prepared in solution as required from t-butyl-lithium and t-butyl cyanide in pentane.[14] Manipulations were carried out using a dry nitrogen-filled glove box or nitrogen-filled apparatus as appropriate.

I.r. spectra (4000—400 cm^{-1}) were recorded on a Grubb–Parsons prism-grating spectrometer or Spectromaster. ^{1}H N.m.r. spectra were recorded at 60 MHz on a Perkin-Elmer R10 spectrometer. Mass spectra were recorded on an A.E.I. M.S.9 instrument at 70 eV and an accelerating potential of 8 kV, with a source temperature of 150—250°, direct insertion, and electromagnetic scanning.

Combustion techniques were used for C, H, and N analyses, and a sodium fusion method for Cl analyses.[16] Molecular weights were determined cryoscopically in benzene. Analytical results are in Table 5.

TABLE 5

Analytical data for the new di-t-butylmethyleneamino-silanes and diphenylmethyleneaminosilanes

Compound	C	H	Cl	N	*M*
$\mathrm{Bu^t_2C{:}NSiMe_3}$	67·6	11·1	—	6·5	215
$\mathrm{C_{12}H_{27}NSi}$ requires	67·6	12·7	—	6·6	213
$\mathrm{Bu^t_2C{:}NSiMe_2Cl}$	56·7	9·6	15·6	5·7	214
$\mathrm{C_{11}H_{24}ClNSi}$ requires	56·7	10·1	15·2	6·0	233
$\mathrm{Bu^t_2C{:}NSiMeCl_2}$	47·9	8·9	27·3	5·3	241
$\mathrm{C_{10}H_{21}Cl_2NSi}$ requires	47·2	8·3	28·0	5·5	254
$\mathrm{Bu^t_2C{:}NSiCl_3}$	40·3	6·2	37·6	4·8	286
$\mathrm{C_9H_{18}Cl_3NSi}$ requires	39·5	6·6	38·6	5·1	274
$\mathrm{Ph_2C{:}NSiMe_2Cl}$	65·5	6·0	12·3	5·0	260
$\mathrm{C_{15}H_{16}ClNSi}$ requires	65·8	5·9	13·0	5·1	273
$\mathrm{(Ph_2C{:}N)_2SiMeCl}$	73·9	5·1	8·1	6·4	436
$\mathrm{C_{27}H_{23}ClN_2Si}$ requires	73·9	5·2	8·1	6·4	438
$\mathrm{(Ph_2C{:}N)_2SiCl_2}$	67·8	4·3	15·4	5·9	445
$\mathrm{C_{26}H_{20}Cl_2N_2Si}$ requires	68·0	4·4	15·4	6·1	459

Preparation of Di-t-butylmethyleneaminosilanes.—(a) *Di-t-butylmethyleneamino(trimethyl)silane*, $\mathrm{Bu^t_2C{:}NSiMe_3}$. Chlorotrimethylsilane (4·34 g, 40 mmol) was added by syringe to a solution of di-t-butylmethyleneaminolithium (40 mmol) in hexane (40 ml). The mixture was held at 70 °C for 2 days, during which lithium chloride was precipitated and a yellow colour developed. The lithium chloride was removed by filtration and the solvent by distillation; the *product*, a greenish yellow liquid, was purified by distillation at 58—60 °C at 0·2 mm: $\nu_{\max.}$ (liquid film) 2994sh, 2963s, 2915sh, 2874m, 1735vs, 1704w, 1610w, 1486m, 1479m,sh, 1468wsh, 1393m, 1368m, 1321w, 1261m, 1252ms, 1230w, 1205w, 1042m, 959m, 930w, 899s, 834vs, 761w, 745w, 732w, 687w, 667w, 624w, and 532w cm^{-1}.

(b) *Di-t-butylmethyleneaminochloro(dimethyl)silane*, $\mathrm{Bu^t_2C{:}NSiMe_2Cl}$. Lithium chloride precipitated within a few minutes of heating to 80 °C a hexane (30 ml) solution containing 20 mmoles each of $\mathrm{Bu^t_2C{:}NLi}$ and $\mathrm{Me_2SiCl_2}$. The *product* was an extremely moisture-sensitive pale yellow liquid, b.p. *ca* 70° at 0·2 mm; $\nu_{\max.}$ (liquid film) 2959vs, 2933sh, 2874ms, 1736vs, 1610vw, 1486ms, 1468sh, 1395m, 1370m, 1325vw, 1297vw, 1258s, 1233m, 1205w, 1172w, 1045ms, 965ms, 940m, 910s, 831vs, 820vs, 794vs, 664m, 578wm, 542w, and 480ms cm^{-1}. This same product was obtained, together with unchanged $\mathrm{Bu^t_2C{:}NLi}$, from mixtures of $\mathrm{1Me_2SiCl_2 + 2Bu^t_2C{:}NLi}$ which had been held at 80 °C in hexane for 20 h, or at 120 °C in toluene for several days.

(c) *Di-t-butylmethyleneaminodichloro(methyl)silane*, $\mathrm{Bu^t_2C{:}NSiMeCl_2}$, prepared similarly from $\mathrm{MeSiCl_3}$ (20 mmoles) and $\mathrm{Bu^t_2C{:}NLi}$ (20 mmoles) in light petroleum at 70°, was a pale yellow liquid, b.p. *ca.* 80° at 0·8 mm: $\nu_{\max.}$ (liquid film) 2950s, 2915s, 2857s, 1762sh, 1736vs, 1672m, 1531w, 1484ms, 1468sh, 1404sh, 1393m, 1368ms, 1292w, 1263ms, 1232w, 1202w, 1149m, 1078ms, 1044s, 1005m, 966ms, 942w, 914ms, 815s, 797s, 741w, 733w, 642w, 614w, 581w, 541m, 525m, and 500m cm^{-1}.

(d) *Di-t-butylmethyleneaminotrichlorosilane*, $\mathrm{Bu^t_2C{:}NSiCl_3}$, from $\mathrm{Bu^t_2C{:}NLi}$ and $\mathrm{SiCl_4}$ in hexane at 80°, had b.p. 96—98° at 0·005 mm: $\nu_{\max.}$ (liquid film) 2959vs, 2924sh, 2874s, 1754sh, 1742sh, 1729vs, 1668m, 1537w, 1486s, 1468sh, 1395ms, 1370s, 1261w, 1235ms, 1202w, 1152sh, 1087m, 1045s, 1005m, 965s, 949ms, 923s, 890m, 840w, 814vs, 803s, 738w, 641w, 590vs, 562vs, 540vs, 522vs, and 489m cm^{-1}.

Preparation of Diphenylmethyleneaminosilanes.—(a) *Diphenylmethyleneaminochloro(dimethyl)silane*, $\mathrm{Ph_2C{:}NSiMe_2Cl}$. When $\mathrm{Me_2SiCl_2}$ (2·58 g, 20 mmol) and $\mathrm{Ph_2C{:}NLi}$ (20 mmol) in ether (60 ml) were mixed at −196° and then stirred at 20 °C for 3 h, lithium chloride was precipitated. The ether was removed under vacuum, toluene was added, and the solution was filtered. Removal of the toluene under reduced pressure left a pale yellow viscous oil which was identified as the *derivative* $\mathrm{Ph_2C{:}NSiMe_2Cl}$; $\nu_{\max.}$ (liquid film) 1660vs, 1653vs, 1600m, 1582m, 1488vw, 1447m, 1400w, 1377w, 1362w, 1312m, 1271sh, 1255s, 1193w, 1176w, 1149w, 1075m, 1028m, 1000w, 971vw, 935m, 907s, 864vs, 825s, 790vs, 757m, 696vs, 660m, 627m, 621m, 568w, 535m, and 470 cm^{-1}. After samples of this compound had been kept at about 15 °C for a week or held a few minutes at 50 °C, they deposited solid bis(diphenylmethyleneamino)dimethylsilane, $\mathrm{(Ph_2C{:}N)_2SiMe_2}$, m.p.

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126—128°, identified by comparison of its i.r. spectrum with that of an authentic sample prepared from Me_2SiCl_2 + $2Ph_2C{:}NLi$.

(b) *Bis(diphenylmethyleneamino)chloro(methyl)silane*, $(Ph_2C{:}N)_2SiMeCl$. Reaction of equimolar proportions of Me_2SiCl_2 and $Ph_2C{:}NLi$ using the procedure described above for the synthesis of $Ph_2C{:}NSiMe_2Cl$ afforded a yellow oil which during 1 h at 20 °C deposited a pale yellow *solid*, identified as $(Ph_2C{:}N)_2SiMeCl$; $\nu_{max.}$ (Nujol mull) 1661sh, 1655sh, 1645s, 1630vs, 1596s, 1578s, 1314m, 1272s, 1261sh, 1193vw, 1179w, 1156w, 1096m, 1074m, 1026m, 1000w, 973vw, 937s, 907s, 859vs, 838s, 787s, 766m, 758m, 727s, 700sh, 695vs, 676sh, 627s, 618sh, 568w, 545s, 500s, 490sh, 451w, and 431m cm^{-1}.

(c) *Bis(diphenylmethyleneamino)dichlorosilane*, $(Ph_2C{:}N)_2$-$SiCl_2$. An excess of silicon tetrachloride (5·1 g, 30 mmol) was added to a solution of diphenylmethyleneaminolithium (20 mmol) in ether (60 ml) at −196 °C. The mixture was stirred at 15 °C for 16 h; ether was removed under reduced pressure, and the residue was extracted with cold pentane, from which the pale yellow *product* $(Ph_2C{:}N)_2$-$SiCl_2$, crystallised, m.p. 103—105°; $\nu_{max.}$ (Nujol mull) 1663sh, 1656vs, 1602s, 1582m, 1316m, 1279s, 1247sh, 1178w, 1160w, 1105vw, 1074w, 1028w, 1000w, 973w, 939m, 932m, 913s, 885s, 855m, 789m, 760m, 724w, 702vs, 692vs, 633s, 618vw, 613vw, 567s, 556s, 536s, 474m, and 449w cm^{-1}.

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