Monomeric Di-t-butylmethyleneaminoboranes †

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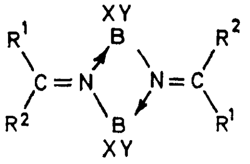
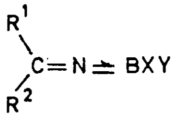
The following new di-t-butylmethyleneaminoboranes have been prepared (the reagents in parentheses): $\mathrm{Bu^t_2C{:}NBPh_2}$ (from $\mathrm{Ph_2BCl + Bu^t_2C{:}NLi}$); $\mathrm{Bu^t_2C{:}NBCl_2}$ ($\mathrm{BCl_3 + Bu^t_2C{:}NLi}$); and $\mathrm{Bu^t_2C{:}NBBu^n_2}$ ($\mathrm{Bu^t_2C{:}NBCl_2 + 2Bu^nLi}$ or $\mathrm{Bu^n_2BCl + Bu^t_2C{:}NLi}$). These di-t-butylmethyleneaminoboranes are monomeric in benzene solution, with i.r. azomethine stretching frequencies in the range 1812—1839 $\mathrm{cm^{-1}}$. Their $^{11}\mathrm{B}$ n.m.r. show resonances at 32 ± 4 p.p.m. relative to $\mathrm{BF_3,OEt_2}$ as is consistent with boron being in a three-co-ordinate environment. Their $^1\mathrm{H}$ n.m.r. spectra have only a singlet absorption attributable to the t-butyl protons, even at −60°, interpreted as indicating that they probably contain linear $\mathrm{C{=}N{\rightleftharpoons}B}$ groups. Features of their mass spectra, which confirm their monomeric state in the gas phase, are briefly discussed.

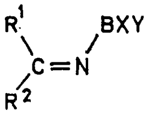
Alkylideneaminoboranes[2-10] $(\mathrm{R^1R^2C{:}NBXY})_n$ are normally associated [n generally = 2; structure (I)], except, apparently, when the substituents R, X, and Y are so bulky as to prevent association.[2,11-15] Considerable interest attaches to the structures of the monomers, for which a linear allene-type skeleton (II) would allow maximum $\mathrm{N{\rightleftharpoons}B}$ dative π-bonding. The only structural information available until recently has been the high (1780—1850 $\mathrm{cm^{-1}}$) azomethine stretching frequency of the monomers, appropriate [assigned $\nu(\mathrm{C{=}N{\rightleftharpoons}B})$] for (II) but difficult to reconcile with a bent structure (III), for which a frequency nearer that of the dimers[2-10] and of azomethines in general[16] (1590—1690 $\mathrm{cm^{-1}}$) might have been expected. The molecular structure of the crystalline monomeric $\mathrm{Ph_2C{:}NB(mesityl)_2}$ has established a linear geometry at the nitrogen atom.[17]



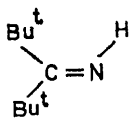
(I)



(II)



(III)



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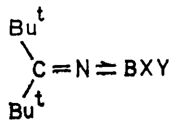

(V)

To obtain further structural information, we have prepared a new series of alkylideneaminoboranes containing the di-t-butylmethyleneamino group, $\mathrm{Bu^t_2C{:}N}$, chosen so that the bulk of the substituents should be sufficient to prevent association and so that their $^1\mathrm{H}$ n.m.r. spectra could be studied for evidence of linearity of the C:NB skeleton. Di-t-butylmethyleneamine itself, $\mathrm{Bu^t_2C{:}NH}$, which has the bent structure (IV), with magnetically nonequivalent t-butyl groups, gives separate resonances attributable to these.[1] A di-t-butylmethyleneaminoborane $\mathrm{Bu^t_2C{:}NBXY}$ with a linear skeleton (V) and magnetically equivalent t-butyl groups, should give only one signal. Spectra consistent with linear C:NB skeletons for the new monomeric di-t-butylmethyleneaminoboranes are described below.

This paper arises from independent investigations at Durham and Sussex, both of which clearly follow from earlier studies.[1,2] The three compounds here described were prepared and characterised in both laboratories. Apart from the problems outlined above, the Durham work forms part of a programme to examine *inter alia* the series $(\mathrm{Bu^t_2C{:}N})_n\mathrm{BX}_{3-n}$ while the Sussex results relate to the series $\mathrm{Bu^t_2C{:}NBX_2}$ (X = F, Cl, Br, or I) and $\mathrm{Bu^tHC{:}NBX_2}$.[2]

Table 1

Skeletal stretching frequencies $\nu(\mathrm{C{=}N{\rightleftharpoons}B})$

Compound	$\nu(\mathrm{C{=}N{\rightleftharpoons}B})$ ($\mathrm{cm^{-1}}$)
$\mathrm{Bu^t_2C{:}NBCl_2}$	1839 (liquid film)
$\mathrm{Bu^t_2C{:}NBPh_2}$	1820 (Nujol mull)
$\mathrm{Bu^t_2C{:}NBBu^n_2}$	1821 (liquid film)

The di-t-butylmethyleneaminoboranes prepared are listed in Table 1, together with their azomethine stretching frequencies. The compounds, except $\mathrm{Bu^t_2C{:}NBPh_2}$,

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† Part XV of the series 'Azomethine Derivatives,' for Part XIV, see ref. 1; also Part II of the series 'Imido Derivatives of Metals and Metalloids,' ref. 2 to be regarded as Part I.

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are readily hydrolysed in the atmosphere. The compounds $Bu^t_2C{:}NBCl_2$, $Bu^t_2C{:}NBBu^n_2$, and $Bu^t_2C{:}NBPh_2$ were prepared from equimolar proportions of $Bu^t_2C{:}NLi$ (prepared *in situ* from Bu^tCN and Bu^tLi) and BCl_3, Bu^n_2BCl, and Ph_2BCl respectively [equation (1)]. In the procedure found most convenient (see Experimental section for details) the reagents in the form of solutions in hydrocarbons were mixed at low temperature. Lithium chloride normally separated as the mixture warmed up, and reaction was found to be complete after a few hours of stirring at room temperature.

$$Bu^tC{:}N \xrightarrow{Bu^tLi} Bu^t_2C{:}NLi \xrightarrow{R_nBCl_{3-n}} Bu^t_2C{:}NBR_nCl_{2-n} + LiCl \quad (1)$$

An alternative route (2) to di-t-butylmethyleneamino-di-n-butylborane was also used.

$$Bu^t_2C{:}NBCl_2 + 2Bu^nLi \longrightarrow Bu^t_2C{:}NBBu^n_2 + 2LiCl \quad (2)$$

Reaction (2) reveals the low susceptibility to reduction of the di-t-butylmethyleneamino-group, in that in reaction with n-butyl-lithium, the chlorine atoms of $Bu^t_2C{:}NBCl_2$ are replaced by butyl groups while the azomethine group remains unaffected.

State of Association and Probable Structures of the Di-t-butylmethyleneaminoboranes.—All the di-t-butylmethyleneaminoboranes in Table 1 were monomeric (by cryoscopy) in benzene solution and (by mass spectrometry) in the vapour. Of the known related diphenylmethyleneaminoboranes $Ph_2C{:}NBXY$, the compounds $(Ph_2C{:}NBCl_2)_2$ [10] and $(Ph_2C{:}NBPhCl)_n$ [14,15] had been found to be associated, and $Ph_2C{:}NBPh_2$ [13-15] and $(Ph_2C{:}N)_2BPh$ [14] monomeric. Of the known t-butylmethyleneaminoboranes $Bu^tHC{:}NBXY$, the di-n-butyl compound ($X = Bu^n = Y$) is monomeric in the vapour at *ca.* 110°, but is dimeric in benzene solution, while the dihalides ($X = Cl$ or $Br = Y$) are invariably dimeric.[2]

Experiments with scale models suggest that association of the di-t-butylmethyleneaminoboranes is prevented by the bulk of the alkylideneamino groups, the t-butyl substituents being better than phenyl at preventing association.

The high values of the azomethine stretching frequencies of the di-t-butylmethyleneaminoboranes with $\nu(C{:}N)$ 1812—1839 cm^{-1} (see Table 1) are consistent with these absorptions being attributable to the unsymmetrical stretching vibrations of a linear $C{=}N{\rightleftharpoons}B$ skeleton.

The use of ^{11}B n.m.r. to establish the state of aggregation of organic boron compounds is well established [18] and has been employed to distinguish the monomeric form of $Bu^tHC{:}NBBu^n_2$ (chemical shift $-38{\cdot}8 \pm 0{\cdot}5$ p.p.m. relative to BF_3,OEt_2) from its dimer (chemical shift $-7{\cdot}4 \pm 0{\cdot}4$ p.p.m. on the same scale).[2] The dimer, with four-co-ordinate boron, has the boron atoms more magnetically shielded and hence the signal is at higher field. Our data of Table 2 are consistent with the formulation of the di-t-butylmethyleneaminoboranes as monomers, and also shown are values for compounds BX_3 and Me_2NBX_2 ($X = Ph$, Bu^n, or Cl) for comparison. The ^{11}B data for the di-t-butylmethyleneaminoboranes show that in these compounds the boron atom is more shielded than in the monomeric $Bu^tHC{:}NBBu^n_2$ or in the related amido-compounds Me_2NBX_2. These observations reflect the decreasing electron-release in the series $Bu^t_2C{:}N^- > Bu^tHC{:}N^- > Me_2N^-$, when these groups are attached to boron. The effect of varying the ligand X is similar in $Bu^t_2C{:}NBX_2$ as known for BX_3. A digital memory oscilloscope was required for the present data, especially for $Bu^t_2C{:}NBPh_2$ and less so for $Bu^t_2C{:}NBBu^n_2$, because of broadness of the ^{11}B signal. This is due to the adjacent ^{14}N quadrupolar nucleus and to coupling with Ph or Bu^n protons.

TABLE 2

^{11}B N.m.r. spectroscopic data

Compound	^{11}B Shift [a]	Compound	^{11}B Shift [a,b]
$Bu^t_2C{:}NBCl_2$	$-28{\cdot}7 \pm 0{\cdot}2$	Me_2NBCl_2	$-30{\cdot}7$
		Me_2NBPh_2	$-41{\cdot}8$
$Bu^t_2C{:}NBPh_2$	-31 ± 2	$Me_2NBBu^n_2$	$-45{\cdot}5$
$Bu^t_2C{:}NBBu^n_2$	-35 ± 1	BCl_3	$-47{\cdot}5$
		BPh_3	$-60{\cdot}0$
		BBu^n_3	$-86{\cdot}5$

[a] Shifts are relative to $BF_3{\cdot}OEt_2$; our data are for solutions in C_6H_6. [b] See ref. 18.

The ^1H n.m.r. spectra of the di-t-butylmethyleneaminoboranes indicate the linearity of the $C{:}NB$ skeletons. Spectra were recorded using *ca.* 20 wt.% toluene solutions at +33° and at temperatures down to −60 °C. Details of the +33° spectra are in Table 3, with data for CS_2 solutions. In all cases, the t-butyl absorption was a sharp singlet which did not change significantly in shape or chemical shift when the solutions were cooled. In contrast, the spectrum of di-t-butylmethyleneamine, $Bu^t_2C{:}NH$ (IV) in toluene, whilst having only a single absorption attributable to the t-butyl groups at +33° (presumably due to rapid exchange of positions between the *N*-hydrogen and the lone pair on nitrogen) has two peaks of equal intensity at −60° at τ 8·73 and 8·90 as inversion at nitrogen is then slow enough for the t-butyl groups to be seen to be

TABLE 3

^1H N.m.r. spectroscopic results; *ca.* 20 wt.% solutions in toluene at +33°; results for CS_2 solutions in brackets [ ] *

Compound	τ Values †	
$Bu^t_2C{:}NBCl_2$		8·92s [8·72]
$Bu^t_2C{:}NBPh_2$	2·3c (6), 2·5c (4); [2·4—2·9]	8·86s (18) [8·72]
$Bu^t_2C{:}NBBu^n_2$	8·4—9·2c; [8·6—9·4]	8·85s [8·82]

* Measured at 60 MHz; singlet Bu^t resonances were also observed for CS_2 solutions at +33° when measured at 100 MHz. † $\tau(Me_4Si) = 10{\cdot}00$; s = singlet, c = complex; relative intensities in parentheses.

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magnetically inequivalent.[1] The chemical shift difference of 0·17 p.p.m.[15] between the two types of proton, which are four bonds distant from the azomethine nitrogen, may be compared with a separation of 0·16—0·37 p.p.m. for the two types of methyl proton in isopropylideneamines $Me_2C{:}NR$[19] and of 0·42 p.p.m. for the methylene protons of $H_2C{:}NMe$,[20] at temperatures well above room temperature in the case of the *N*-alkylated or -arylated compounds. The sensitivity of the hydrogen resonance to *syn–anti* isomerism decreases progressively as the number of bonds separating the hydrogens from the azomethine nitrogen increases, but is still large enough for compounds $Bu^t_2C{:}NBX_2$ to be expected, if bent at nitrogen, to give rise to two readily distinguished resonances at temperatures low enough for inversion at nitrogen to be slow. The ^1H n.m.r. spectra of our di-t-butylmethyleneaminoboranes accordingly indicate either that their structures involve linear $C{:}NB$ units, or else that bent $C{:}NB$ skeletons invert at nitrogen with a rapidity at −60° implying an unexpectedly low activation energy for the process. Near-linear $C{:}NAl$ units have been found, by *X*-ray crystallography,[21] in the aluminium compound $LiAl(N{:}CBu^t_2)_4$.

The mass spectra of the butylmethyleneaminoboranes were recorded. The same fragments were found as in the mass spectrum[22] of $Bu^t_2C{:}NH$; there were certain other common features. In all cases a feeble monomeric parent peak was observed as the fragment with highest *m/e* value. Initial fragmentation invariably involved loss of a butyl group as the source of the most abundant fragments; and the groups attached to boron were cleaved in the order $R > Cl > N{:}CBu^t_2$, expected from the relative B–X bond energy terms.[23] The n-butyl group on boron in $Bu^t_2C{:}Bu^n_2$ lost C_4H_8, apparently to form B–H residues, but the t-butyl groups of the alkylideneamino-group much less readily eliminated C_4H_8 to leave C–H residues. Some of these points are illustrated by the data in Table 4.

TABLE 4

Boron-containing fragments in the mass spectrum of $Bu^t_2C{:}NBCl_2$

m/e *	Relative intensity	Assignment
221	0·3	$Bu_2CNBCl_2^+$
186	0·7	Bu_2CNBCl^+
164	62·0	$BuCNBCl_2^+$
149	0·6	$Me_2CCNBCl_2^+$

* *m/e* Values relate to ^{35}Cl and ^{11}B isotopes.

EXPERIMENTAL

t-Butyl cyanide was purified by distillation from phosphorus(V) oxide under dry nitrogen. t-Butyl-lithium (*ca.* 2M in hexane) (Alfa Inorganics, Inc.) was standardized by diluting 5 ml of the solution with 20 ml of dry pentane and titrating this solution with a 1M solution of s-butyl alcohol in dry xylene, with 1,10-phenanthroline as indicator.[24] The organoboron compounds[14,25] and di-t-butylmethyleneamine[26] were prepared and purified as described earlier. Materials throughout were handled under dry nitrogen, argon, or *in vacuo*.

Spectra.—I.r. spectra were recorded on a Grubb–Parsons Spectromaster (Durham) and on a Perkin-Elmer 457 instrument (Sussex). Mass spectra were recorded on an A.E.I. MS9 instrument at 70 eV and an accelerating potential of 8 kV, with a source temperature of 150—250° (Durham) or 90° (Sussex) and with electromagnetic scanning. Samples were inserted directly into the ion source (Durham) or by using break-seals (Sussex). ^1H N.m.r. spectra were recorded at 60 MHz on a Perkin-Elmer R10 (Durham) or Varian A60 (Sussex) spectrometers, or at 100 MHz on a Varian HA 100 instrument. ^{11}B N.m.r. data were obtained on a Perkin-Elmer R10 spectrometer, operating at 19·3 MHz and 33·5° with a Northern digital memory oscilloscope.

Analysis.—Carbon, hydrogen, boron, and nitrogen were determined by combustion techniques[8] at Durham, or (Sussex) by A. Bernhardt, and for B, Cl, and N as described elsewhere.[27] Chlorine was determined at Durham by the sodium fusion method.[28] Molecular weights were determined in benzene using solutions of concentration *ca.* 1 wt.%, cryoscopically.

Preparation of Dichloro(di-t-butylmethyleneamino)borane.—t-Butyl cyanide (21·84 ml, 200 mmol) was added dropwise, during 15 min, to a stirred solution of t-butyl-lithium (200 mmol) in n-pentane (100 ml) at −78°. The mixture was allowed to warm to 20° and was stirred for 60 min. Boron trichloride was condensed onto the frozen (−196°) solution of di-t-butylmethyleneaminolithium. The mixture was allowed to warm up, whereupon a vigorously exothermic reaction occurred and a heavy white precipitate (LiCl) was formed. The mixture was stirred at 20° for 2 h, and then filtered; the residue was extracted with n-pentane (2 × 100 ml). The combined solutions were freed from volatile materials *in vacuo* giving a pale yellow-green liquid. Distillation at reduced pressure using a 10 cm Vigreux column gave, as a colourless liquid, *dichloro(di-t-butylmethyleneamino)borane* (34·2 g, 77%), b.p. 63—67°, 2 mmHg (Found: C, 48·9; H, 8·3; B, 4·7; Cl, 31·7; N, 6·5%; *M*, 277. $C_9H_{18}BCl_2N$ requires C, 48·7; H, 8·15; B, 4·85; Cl, 31·9; N, 6·3%; *M*, 222), $\nu_{max.}$ (liquid film, cm^{-1}): 3325w, 3117w, 2965s, 2942s,sh, 2907s,sh, 2869s, 2562w, 2022w, 1839vs, 1772m,sh, 1484s, 1465s, 1396s, 1373s, 1317s,sh, 1307s, 1291s, 1247m,sh, 1201s, 1049s, 978s, 937s,sh, 915s, 878vs, 837s, 797vw, 739w, 736w, 656s, 557m, 478w,sh, 466m, 429w, and 377vw. This compound was readily hydrolysed and fumed strongly in moist air.

Preparation of Di-t-butylmethyleneamino(diphenyl)borane.

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—A similar reaction between di-t-butylmethyleneamino-lithium (45·6 mmol) and chloro(diphenyl)borane (45·6 mmol) in pentane (60 ml) afforded a solution from which were obtained white crystals of *di-t-butylmethyleneamino-(diphenyl)borane* (10·96 g, 79%), m.p. 63—66° (n-pentane) (Found: C, 82·5; H, 9·1; B, 3·05; N, 4·45%; *M*, 324. $C_{21}H_{28}BN$ requires C, 82·6; H, 9·25; B, 3·55; N, 4·6%; *M*, 305), $\nu_{max.}$ (Nujol mull, cm^{-1}): 3065m, 3047s, 2023vw, 1966vw,sh, 1951w, 1902vw, 1883vw, 1820vs, 1766m, 1663vw, 1593w, 1233vs, 1208m,sh, 1188w,sh, 1183m, 1154w, 1103w, 1067m, 1047s, 1033m, 1024m, 1001w, 974s, 952vw,sh, 932w, 919w, 903m, 885s, 858vw, 840m, 797vw, 764s, 748s, 725w, 708s,sh, 701vs, 699s,sh, 636w,sh, 632m, 627m, 621m, 582w, 551m, 469vw, 440w, 423vw, 395vw, 387vw, 364vw, and 353vw,sh.

Preparation of Di-n-butyl(di-t-butylmethyleneamino)borane.—(*a*) The reaction between di-t-butylmethyleneamino-lithium (44·7 mmol) and chloro(di-n-butyl)borane (44·7 mmol) in pentane (60 ml) afforded the colourless liquid *di-n-butyl(di-t-butylmethyleneamino)borane* (7·42 g, 63%), b.p. 85—90°, 0·1 mmHg (Found: C, 76·9; H, 13·5; B, 3·95; N, 5·4%; *M*, 277. $C_{17}H_{36}BN$ requires C, 77·0; H, 13·7; B, 4·1; N, 5·3%; *M*, 265), $\nu_{max.}$ (liquid film, cm^{-1}): 3586vw, 2996s,sh, 2949vs, 2914s, 2864s, 2711w, 2646vw, 2556vw, 2486vw, 2016vw, 1812vs, 1768m,sh, 1482s, 1465s, 1409w, 1391s, 1376m,sh, 1366s, 1340m, 1306s,sh, 1294s, 1269m,sh, 1233m, 1221m,sh, 1205m, 1186m,sh, 1158vw, 1104m, 1046s, 1029m,sh, 998vw, 975s, 935m, 900w, 891w,sh, 840m, 796w,sh, 782w, 768w,sh, 746w,sh, 734w, 720vw, 593w, 556w, 434w, and 424w,sh.

(*b*) n-Butyl-lithium (10 ml of 1·4M solution in hexane) was added by syringe to a frozen (−196°) solution of $\mathrm{Bu^t{}_2C{:}NBCl_2}$ (1·44 g, 6·6 mmol) in hexane (20 ml). A strongly exothermic reaction occurred as the mixture was allowed to warm to 15°, and lithium chloride was precipitated. The mixture was stirred at 15° for two days, solvent was removed *in vacuo*, and the residue was extracted with warm toluene, from which the liquid di-t-butylmethyleneamino(di-n-butyl)borane, $\mathrm{Bu^t{}_2C{:}NBBu^n{}_2}$, b.p. 85—90°, 0·1 mmHg was recovered on distillation (Found: C, 76·6; H, 13·3; N, 5·6%; *M*, 277).

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