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PREPARATION AND CRYSTAL STRUCTURE OF [TRIS(TRIMETHYLSILYL)METHYL]DIPHENYLBORANE

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Summary

The compound $(Me_3Si)_3CBPh_2$ has been made by treatment of Ph_2BBr with [tris(trimethylsilyl)methyl]lithium in tetrahydrofuran/benzene, and its crystal structure has been determined. The coordination about the boron atom is strictly planar, but the $(Me_3Si)_3C$–B–C(Ph) angles (122.5(2) and 127.1(2)°) are substantially larger than the C(Ph)–B–C(Ph) angle (110.4(2)°); the planes of the phenyl rings make angles of 91 and 47°, respectively, with the BC_3 plane. The three B–C distances (mean 1.583(4) Å) are all the same within the limits of error, and similar to those in BPh_3 (mean 1.577 Å). The mean B–C–Si angle is 110.3(16)°. Within the $(Me_3Si)_3C$ group the C–$SiMe_3$ bonds (mean 1.934(3) Å) are significantly longer than the Si–Me bonds (mean 1.873(6) Å), and the C($(SiMe_3)_2$)–Si–C(Me) angles (mean 114(2)°) substantially larger than the C(Me)–Si–C(Me) angles (mean 105(2)°). The Me_3Si groups are twisted by 18(5)° from the fully staggered positions, but (in contrast to the situation in $(Me_3Si)_3CPH_2$ in the gas phase) the Me groups are fully staggered.

Introduction

There is considerable interest in the chemistry of compounds containing the very bulky tris(trimethylsilyl)methyl group (the "trisyl" group, denoted by Tsi), and diffraction methods have been used to determine the structure of several of them, viz. TsiH [1], $TsiSiMe_2Ph$ [2], $(Tsi)_2Hg$ [3], $[Li(THF)_4][Li(Tsi)_2]$ [4], $[Li(THF)_4][Cu(Tsi)_2]$ [5], $TsiBPh[O(CH_2)_4Tsi]$ [6], and $TsiPH_2$ [7]. We now present the results of a single crystal X-ray diffraction study of the boron compound $TsiBPh_2$ (I), which we obtained from the reaction of tris(trimethylsilyl)methyllithium with Ph_2BBr in tetrahydrofuran.

Experimental

Preparation of $TsiBPh_2$

A solution of MeLi in Et_2O (6 cm^3 of 1.5 *M* MeLi) was added to a solution of

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tris(trimethylsilyl)methane (2.0 g, 8.6 mmol) in THF (25 cm^3). The ether was distilled off and the remaining solution was refluxed for 2 h, after which TsiLi had been formed in 95% yield *. The solution was cooled to 0 °C and a solution of Ph_2BBr (2.0 g, 8.2 mmol) in benzene (25 cm^3) was added dropwise during 0.5 h. The mixture was subsequently refluxed for 3 h, then the solvent was evaporated off and the residue was extracted with pentane. The pentane solution was filtered and evaporated to leave a viscous residue to which MeOH was added. Upon overnight storage at ca. 5 °C a solid separated, and this was recrystallized from CH_2Cl_2/MeOH to give [tris(trimethylsilyl)methyl]diphenylborane (0.50 g, 14%), m.p. 107–109 °C; ^1H NMR $\delta(CCl_4)$ 0.19 (27H, s, $SiMe_3$), 7.09 (2H, t (J 7.2 Hz), p-H), 7.17 (4H, dd (J 8.0, 7.4 Hz), m-H) 7.31 ppm (4H, d (J 8.1 Hz), o-H); ^{11}B NMR $\delta(C_6H_6$, relative to $BF_3 \cdot OEt_2)$ 77.5 ppm; m/z (EI) 396 (10%, M^+), 381 (9, $[M-Me]^+$), 365 (1, $[M-Me-CH_4]^+$), 322 (60, $[M-Me_3SiH]^+$), 308 (55, $[M-Me_4Si]^+$), 307 (55, $[M-Me_3SiH-Me]^+$, 303 (70, $[M-Me-C_6H_6]^+$), 293 (80, $[M-Me-Me_4Si]^+$); 242 (70, $[M-Me-Ph_2]^+$), 231.1197 (70, $[M-Me-Me_3SiPh]^+$, calcd. 231.1197), 215 (30, $[M-Me-CH_4-Me_3SiPh]^+$), 169.1031 (80, $[(Me_3Si)_2C{=}B]^+$, calcd. 169.1040) 135 (90, $[Me_2PhSi]^+$), 73 (100, $[Me_3Si]^+$); m/z (CI, NH_3), 414 (100%, $[M+NH_4]^+$) (Found: C, 65.6; H, 9.3. $C_{22}H_{37}BSi_3$ calcd.: C, 66.6; H, 9.4%).

Structure determination

Crystal data. $C_{22}H_{37}BSi_3$, $M = 396.6$, triclinic, a 9.436(1), b 9.971(2), c 13.893(3) Å, α 89.33(2), β 76.70(2), γ 73.98(2)°, U 1220.8 Å^3 $Z = 2$, D_c 1.08 g cm^{-3}, $F(000) = 432$. Monochromated Mo-K_α radiation, λ 0.71069 Å, μ 2.0 cm^{-1}. Space group $P\bar{1}$ from successful structure refinement.

Data were measured on an Enraf-Nonius CAD4 diffractometer using a crystal of size ca. 0.30 × 0.20 × 0.15 mm. Preliminary cell dimensions were found using the SEARCH and INDEX routines of the CAD4 and final values were calculated from the setting angles for 25 reflections with $\theta \approx 15°$. Intensities for $h \pm k \pm l$ reflections with $2 < \theta < 22°$ were measured by a $\theta/2\theta$ scan with a scan width of $\Delta\theta = (0.8 + 0.35 \tan\theta)°$. The scan rate for each reflection was determined by a rapid pre-scan at 10° min^{-1} in θ, at which point any reflection with $I < \sigma(I)$ was coded as unobserved. The remaining reflections were rescanned at such a speed as to give a minimum value of $\sigma(I)/I$ of 0.05, subject to a maximum scan time of 60 seconds. Two standard reflections monitored every hour showed no significant variation. Data were corrected for Lp effects but not for absorption, and after averaging any equivalent reflections 2082 reflections with $|F^2| > \sigma(F^2)$ were used in the structure refinement. The values of $\sigma(F^2)$ were taken as $[\sigma^2(I) + (0.02I)^2]^{1/2}/Lp$.

The structure was solved by direct methods using the MULTAN program [9]. Refinement of non-hydrogen atoms with anisotropic temperature factors was by full matrix least squares. H atoms were located on a difference map and included in the refinement with isotropic temperature factors. Refinement converged at $R = 0.037$, $R' = 0.040$, when the maximum shift/error was 0.6 and the weighting scheme was $w = 1/\sigma^2(F)$. A final difference map was everywhere featureless.

(Continued on p. 5)

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* This procedure [8b], involving removal of ether, which reduces the reflux time, is a modification of the original one [8a].

TABLE 1

FRACTIONAL ATOMIC COORDINATES ($\times 10^4$ for Si, C, B, and $\times 10^3$ for H) WITH ESTIMATED STANDARD DEVIATIONS IN PARENTHESES

Atom	x	y	z
Si(1)	7105.7(10)	2095.4(9)	1176.0(7)
Si(2)	6329.3(10)	1702.7(9)	3431.4(7)
Si(3)	3716.7(10)	2337.5(10)	2257.6(7)
B	5267(4)	4347(3)	2641(2)
C(1)	5555(3)	2736(3)	2386(2)
C(2)	6364(5)	2786(4)	75(3)
C(3)	8932(4)	2560(4)	1040(3)
C(4)	7752(4)	150(4)	975(3)
C(5)	8420(4)	1399(4)	3293(3)
C(6)	6023(5)	−78(4)	3524(3)
C(7)	5453(4)	2615(3)	4680(2)
C(8)	4017(4)	668(4)	1517(3)
C(9)	2612(4)	3737(5)	1601(3)
C(10)	2401(4)	2120(4)	3449(3)
C(11)	6577(3)	5089(3)	2393(2)
C(12)	7385(3)	5216(3)	3090(2)
C(13)	8500(4)	5903(4)	2898(3)
C(14)	8828(4)	6501(3)	2008(3)
C(15)	8024(4)	6424(3)	1331(3)
C(16)	6922(3)	5735(3)	1514(2)
C(17)	3704(3)	5436(3)	3158(2)
C(18)	3298(4)	6747(3)	2776(2)
C(19)	1962(4)	7752(4)	3189(3)
C(20)	1014(4)	7513(4)	4027(3)
C(21)	1392(4)	6262(4)	4446(2)
C(22)	2688(3)	5240(3)	4012(2)
H(2A)	558(4)	243(3)	0(2)
H(2B)	728(4)	251(4)	−52(3)
H(2C)	599(4)	375(3)	16(3)
H(3A)	965(4)	194(4)	75(3)
H(3B)	917(5)	289(5)	160(3)
H(3C)	897(4)	312(4)	63(3)
H(4A)	706(4)	−33(4)	105(3)
H(4B)	835(4)	−1(4)	40(3)
H(4C)	835(4)	−28(4)	138(3)
H(5A)	901(1)	70(3)	271(2)
H(5B)	865(4)	90(4)	385(3)
H(5C)	865(4)	220(4)	326(2)
H(6A)	500(4)	−7(3)	366(2)
H(6B)	637(4)	−55(4)	300(3)
H(6C)	643(4)	−44(4)	405(3)
H(7A)	546(3)	359(3)	470(2)
H(7B)	443(3)	260(3)	493(2)
H(7C)	597(3)	219(3)	506(2)
H(8A)	446(4)	−19(4)	182(3)
H(8B)	467(4)	69(4)	90(2)
H(8C)	310(4)	66(3)	156(2)
H(9A)	201(5)	449(5)	200(4)
H(9B)	314(4)	413(4)	115(3)
H(9C)	192(5)	350(5)	144(3)
H(10A)	168(5)	295(5)	369(4)
H(10B)	182(4)	170(4)	334(3)

TABLE 1 (continued)

Atom	*x*	*y*	*z*
H(10C)	278(4)	194(4)	397(3)
H(12)	716(3)	483(3)	370(2)
H(13)	895(3)	596(3)	336(2)
H(14)	962(3)	695(3)	189(2)
H(15)	822(3)	682(3)	74(2)
H(16)	638(3)	566(3)	104(2)
H(18)	391(3)	695(2)	229(2)
H(19)	175(3)	857(3)	288(2)
H(20)	16(3)	813(3)	429(2)
H(21)	77(3)	606(3)	500(2)
H(22)	297(3)	436(3)	431(2)

TABLE 2

INTRAMOLECULAR DISTANCES (Å) AND ANGLES (°) WITH ESTIMATED STANDARD DEVIATIONS IN PARENTHESES

Si(1)–C(1)	1.937(2)	Si(1)–C(2)	1.873(4)
Si(1)–C(3)	1.873(4)	Si(1)–C(4)	1.869(4)
Si(2)–C(1)	1.934(3)	Si(2)–C(5)	1.876(4)
Si(2)–C(6)	1.874(4)	Si(2)–C(7)	1.866(4)
Si(3)–C(1)	1.930(3)	Si(3)–C(8)	1.886(4)
Si(3)–C(9)	1.863(5)	Si(3)–C(10)	1.877(5)
B–C(1)	1.584(4)	B–C(11)	1.580(4)
B–C(17)	1.586(4)	C(11)–C(12)	1.389(4)
C(11)–C(16)	1.388(4)	C(12)–C(13)	1.383(5)
C(13)–C(14)	1.374(5)	C(14)–C(15)	1.352(5)
C(15)–C(16)	1.373(5)	C(17)–C(18)	1.393(4)
C(17)–C(22)	1.393(4)	C(18)–C(19)	1.381(4)
C(19)–C(20)	1.358(5)	C(20)–C(21)	1.362(5)
C(21)–C(22)	1.374(4)	C–H(min)	0.79(4)
		C–H(max)	1.03(3)
C(1)–Si(1)–C(2)	110.4(2)	C(1)–Si(1)–C(3)	117.7(2)
C(1)–Si(1)–C(4)	113.1(2)	C(2)–Si(1)–C(3)	105.6(3)
C(2)–Si(1)–C(4)	106.3(2)	C(3)–Si(1)–C(4)	102.8(2)
C(1)–Si(2)–C(5)	114.0(2)	C(1)–Si(2)–C(6)	113.1(2)
C(1)–Si(2)–C(7)	112.9(2)	C(5)–Si(2)–C(6)	105.2(3)
C(5)–Si(2)–C(7)	104.2(2)	C(6)–Si(2)–C(7)	106.5(2)
C(1)–Si(3)–C(8)	114.8(2)	C(1)–Si(3)–C(9)	110.7(2)
C(1)–Si(3)–C(10)	115.6(2)	C(8)–Si(3)–C(9)	104.9(3)
C(8)–Si(3)–C(10)	102.5(2)	C(9)–Si(3)–C(10)	107.3(3)
C(1)–B–C(11)	122.5(2)	C(1)–B–C(17)	127.1(2)
C(11)–B–C(17)	110.4(2)	Si(1)–C(1)–Si(2)	106.2(1)
Si(1)–C(1)–Si(3)	109.0(1)	Si(1)–C(1)–B	111.6(2)
Si(2)–C(1)–Si(3)	110.7(1)	Si(2)–C(1)–B	108.0(2)
Si(3)–C(1)–B	111.2(2)	B–C(11)–C(12)	120.8(3)
B–C(11)–C(16)	123.1(3)	C(12)–C(11)–C(16)	115.9(3)
C(11)–C(12)–C(13)	121.6(4)	C(12)–C(13)–C(14)	120.6(4)
C(13)–C(14)–C(15)	118.7(4)	C(14)–C(15)–C(16)	121.0(4)
C(11)–C(16)–C(15)	122.2(4)	B–C(17)–C(18)	119.2(3)
B–C(17)–C(22)	126.0(2)	C(18)–C(17)–C(22)	114.7(3)
C(17)–C(18)–C(19)	122.5(3)	C(18)–C(19)–C(20)	120.4(4)
C(19)–C(20)–C(21)	119.0(3)	C(20)–C(21)–C(22)	120.6(3)
C(17)–C(22)–C(21)	122.6(3)		

TABLE 3

DEVIATIONS (Å) OF ATOMS FROM VARIOUS MEAN PLANES[a] (Atoms marked with an asterisk were not used in the calculation of the plane)

(a) B 0.00, C(1) 0.00, C(11) 0.00, C(17) 0.00; Si(1)* 1.00, Si(2)* −1.82, Si(3)* 0.74

(b) C(11) 0.01, C(12) −0.01, C(13) 0.00, C(14) 0.01, C(15) −0.01, C(16) −0.01; B* −0.05

(c) C(17) 0.01, C(18) −0.02, C(19) 0.01, C(20) 0.01, C(21) −0.02, C(22) 0.01; B* −0.04

(d) B, C(1), Si(2)

[a] Angles between planes a–b 91, a–c 47, b–c 79, a–d 83°.

The structure solution and refinement were done on a PDP 11/34 computer using the Enraf-Nonius Structure Determination Package. Scattering factors for neutral atoms were taken from ref. 10. Final atom coordinates are listed in Table 1, and bond lengths and angles in Table 2, and deviations from mean planes in Table 3. Tables of temperature factors and final structure factors are available from the authors.

Results and discussion

Compound I crystallizes as discrete molecules (Fig. 1), with no significantly short intermolecular contacts.

The coordination about the boron atom is strictly planar, but the bulk of the Tsi

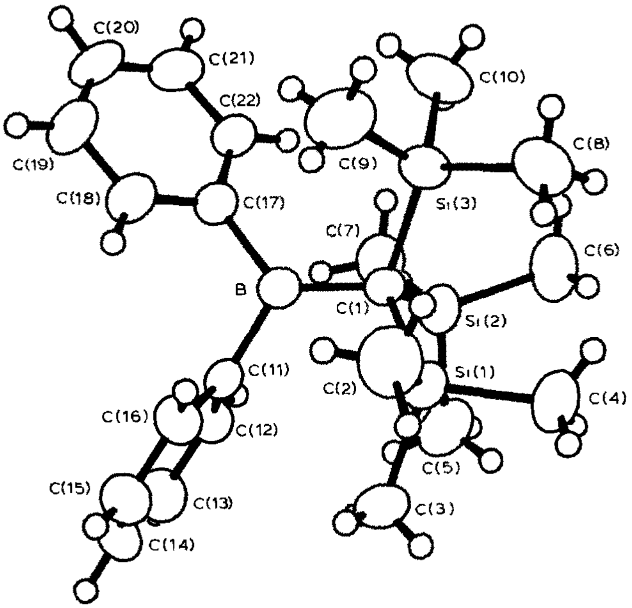

Fig. 1. An ORTEP [20] drawing showing the molecular structure of $TsiBPh_2$ and the atom numbering scheme. Atoms are drawn as 50% thermal vibration ellipsoids except for hydrogen atoms, which are drawn as spheres of arbitrary and equal size.

group forces the Ph groups together, so that the $C(SiMe_3)_3$–B–C(Ph) angles (122.5(2) and 127.1(2)°) are significantly larger than the C(Ph)–B–C(Ph) angle (110.4(2)°). The two phenyl rings are oriented at 91 and 47°, respectively, with respect to the BC_3 plane (with an angle of 79° between the planes of the two rings), and the B–C(1)–Si(2) plane is at 83° to the BC_3 plane; in BPh_3 the rings are tilted by about 30° out of the BC_3 plane [11]. The B–C distances are all equivalent (mean 1.583(4) Å) and similar to those in BPh_3 (mean 1.577(8) Å) [1], and not significantly different from those in TsiBPh[$O(CH_2)_4$Tsi] (in which B–C(Tsi) is 1.567(11) and B–C(Ph) is 1.540(11) Å) [6].

The pattern of bond lengths and angles within the trisyl group is similar to that generally observed in neutral molecules; thus the C(1)–Si bonds (mean 1.934(3) Å) are significantly longer than the Si–C(Me) bonds (mean 1.873(6) Å), and the C(1)–Si–C(Me) angles (mean 114(2)°) are larger and the C(Me)–Si–C(Me) angles (mean 105(2)°) smaller than the tetrahedral value *. The Si–C–Si angles are close to the tetrahedral (mean 108.6(2)°).

The phenyl groups show significant deviation from exact D_{6h} symmetry, the bond lengths and angles averaged over equivalent parts of both rings being as shown in Fig. 2. This pattern is observed in compounds with an electropositive atom directly attached to the ring [12], e.g. in TsiB(Ph)[$O(CH_2)_4$Tsi] [6], $TsiSiMe_2Ph$ [2], EPh_3 (E = B [11], Ga or In [13]), and Al_2Ph_6 [14].

There is an interesting point of detail concerning the arrangement of the Me groups within the trisyl system. Electron diffraction studies and potential energy calculations on both TsiH [1] and $TsiPH_2$ [7] have indicated that the average X–C–Si–C(Me) (X = H or P) and C–Si–C(Me)–H torsion angles are not those expected for fully staggered arrangements. (There is also some deviation from staggered positions in $(Me_3Si)_2CH_2$ [15], as noted below.) The X-ray data for $TsiBPh_2$ are accurate enough to show whether there are similar effects for this compound (in the crystalline state), since the hydrogen atoms were located and refined successfully. (The large number of Me groups should make up for the relatively low precision for individual groups arising from the small scattering

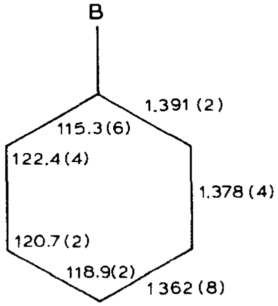

Fig. 2. Bond lengths (Å) and angles (°) for the phenyl groups in $TsiBPh_2$ averaged over equivalent parts for both rings.

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* The bond lengths in the Tsi groups in anions, e.g. $[Li(THF)_4][M(Tsi)_2]$, (M = Li, Cu, Ag) and $[Li(TMEDA)_2][Li(Tsi)_2]$ (TMEDA = *N,N,N′,N′*-tetramethylethylenediamine), may not conform to this pattern [5].

TABLE 4

VALUES OF THE TORSION ANGLES (°) SHOWING THE ORIENTATION OF THE Me_3Si AND CH_3 GROUPS

B–C(1)–Si(1)–C	−72.0	49.1	168.8
B–C(1)–Si(2)–C	−82.2	36.6	157.6
B–C(1)–Si(3)–C	−82.4	39.9	158.4
C(1)–Si(1)–C(2)–H	−65	55	173
C(1)–Si(1)–C(3)*–H	−110	15	144
C(1)–Si(1)–C(4)–H	−73	48	174
C(1)–Si(2)–C(5)–H	−70	57	179
C(1)–Si(2)–C(6)–H	−61	55	181
C(1)–Si(2)–C(7)–H	−49	73	191
C(1)–Si(3)–C(8)–H	−54	66	178
C(1)–Si(3)–C(9)*–H	−84	33	169
C(1)–Si(3)–C(10)*–H	−19	93	199

amplitude for hydrogen.) Torsion angles showing the orientations of the Me_3Si and CH_3 groups, arranged in equivalent order, are shown in Table 4. The B–C–Si–C torsion angles are grouped fairly closely around −79(5), 42(5) and 162(5)° (average values with standard deviations), i.e. the Me_3Si groups are each rotated by about 18° in the same sense from staggered positions (torsion angles −60, +60 and 180°). This Me_3Si twist allows the C–Si–C–H torsion angles, except those involving C(3), C(9) and C(10) indicated with an asterisk in Table 4, to be close to those for a staggered arrangement. It can be seen from Fig. 1 that the C(3), C(9) and C(10) Me groups are those most likely to be affected by intramolecular contacts with the Ph groups, and these contacts may be responsible for the anomalous torsion angles.

The response of the Tsi group to the internal steric interactions is thus similar in $TsiBPh_2$ and in $TsiPH_2$ (Table 5). However, in $(Me_3Si)_2CH_2$ the distortion is different. In the position of minimum energy the Me_3Si groups are twisted only slightly and the Me groups twisted significantly (about 25°) from staggered positions. The Si–C bond lengths in $(Me_3Si)_2CH_2$ are similar to those in the two Tsi compounds, but the wider SiCSi angle (123°) in $(Me_3Si)_2CH_2$ allows the Me_3Si groups to adopt less strained conformations; intramolecular steric interaction is still

TABLE 5

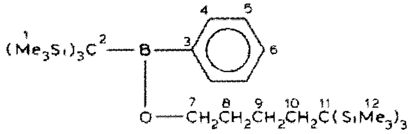
COMPARISON OF STRUCTURAL DATA FOR $(Me_3Si)_3CBPh_2$, $(Me_3Si)_3CPH_2$ AND $(Me_3Si)_2CH_2$

	$TsiBPh_2$	$TsiPH_2$	$(Me_3Si)_2CH_2$
Me_3Si–C (Å)	1.934(3)	1.941(5)	1.889
Me–Si (Å)	1.873(6)	1.883(2)	1.874
C(Me)–Si–C(Me) (°)	105(2)	104.3(4)	
(P or B)–C–Si (°)	110.3(16)	111.9(13)	
tilt[a] (°)	none	−7.3	
twist ($SiMe_3$) (°)	18(5)	21.2(4)	3(2.5)
twist (CH_3) (°)	1(8)	5.0 (fixed)	1(8)

[a] The angle between the three-fold axis of the SiC_3 group and the Si–C(1) bond, defined to be positive when the SiC_3 groups move closer together.

TABLE 6

^{13}C NMR (ppm from TMS)



(a) $TsiBPh_2$: C(1) 7.0, *J*(SiC) 44 Hz, C(2) 30.2, C(3) 150.7, C(4) 126.9, C(5) 128.7, C(6) 127.1

(b) $TsiB(Ph)O(CH_2)_4Tsi$: C(1) 5.3, C(2) 16.8, C(3) 142.7, C(4) 130.1, C(5) 129.6, C(6) 129.1, C(7) 69.4, C(8) 35.0, C(9) 33.1 C(10) 30.1, C(11) 5.9, C(12) 3.1

evident, however, from the twist of the Me groups.

The crystal structures of $TsiBPh_2$ and $TsiB(Ph)[O(CH_2)_4Tsi]$ [6] show that there can be little mesomeric interaction between the phenyl group and boron. This is confirmed by the ^{13}C chemical shifts for the *para* carbon atoms (C(6) in Table 6) which, as expected [16], are close to that in benzene (128.5 ppm). As in a number of other boron compounds [17], resonances from carbon atoms directly bound to boron are difficult to detect, and it is necessary to record the spectra at −60 °C to get sharp signals; those from quaternary carbon atoms may be picked out by spin echo experiments. There is some evidence [18] that the downfield chemical shifts of these *ipso* carbon atoms (C(3)) in phenylboron compounds $PhBX_2$ increase with increasing XBX angle. The shifts in our compounds, where steric factors make the XBX angle large, are in accord with this trend.

The mass spectrum of $TsiBPh_2$ shows the expected breakdown pattern, with fragmentation of the $(Me_3Si)_3C$ more prominent than that of the Ph_2B group. As in other TsiB compounds [19] a strong peak is observed at *m/z* 169, suggesting that $[(Me_3Si)_2C{=}B]^+$ is a reasonably stable ion in breakdown under electron impact.

Acknowledgments

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