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THE SYNTHESIS AND STRUCTURE OF *nido*-$Me_3NCB_{10}H_{10}PR$ DERIVATIVES

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Summary

Reaction of $B_{10}H_{12}CNMe_3$ with triethylamine and then $RPCl_2$ (R = Me, Et or Ph) forms *nido*-$Me_3NCB_{10}H_{10}PR$ in good yield. A low-temperature single crystal X-ray structure determination of $Me_3NCB_{10}H_{10}PPh$ shows that the "PPh" unit bridges two boron atoms in the open face of the $B_{10}H_{10}CNMe_3$ icosahedral fragment.

Introduction

Twelve-atom *nido*-carboranes such as $B_{10}C_2H_{12}^{2-}$ [1,2], $B_{10}C_2H_{13}^-$ [3] and $B_{10}H_{11}(CPh)_2^-$ [1] have been known for several years. A large class of related twelve-atom *nido*-heteroatom boranes should in principle exist and recently a few examples have been reported. Reaction of $RAsCl_2$ (R = Ph or Me) with 1,2-$B_9C_2H_{11}^{2-}$ gave *nido*-$B_9C_2H_{11}AsR$ [4,5]. Treatment of $B_9C_2H_{13}$ with trimethylaluminum or trimethylgallium generated $B_9C_2H_{12}M(CH_3)_2$ (M = Al or Ga) [6]. Reaction of $B_9H_{12}S^-$ with $AsCl_3$ and $(C_2H_5)_3N$ produced the unexpected compound $B_8H_8As_2S$ which is also a member of this class of molecules [4].

We report here the synthesis, characterization and X-ray structure determination of a new series of twelve-atom *nido*-heteroatom borane molecules with the general formula $Me_3NCB_{10}H_{10}PR$.

Experimental

General comments

Tetrahydrofuran was distilled from a deep blue-purple solution of benzophenone ketyl under nitrogen prior to use [7]. Triethylamine was dried over solid potassium hydroxide and distilled under nitrogen prior to use. $B_{10}H_{12}CNMe_3$ was prepared according to the literature method [8].

Boron (^{11}B NMR) spectra were obtained at 70.6 MHz with a Varian HR-220 spectrometer and were externally referenced to $BF_3 \cdot O(C_2H_5)_2$. Positive ^{11}B

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* Contribution No. 3055.

shielding values are downfield from the standard which is the convention agreed to at the Third International Meeting on Boron Chemistry, Munich, West Germany, July, 1976. Proton NMR spectra were also measured on the HR-220 instrument. Infrared spectra were taken in KBr disks and recorded on a Perkin—Elmer 467 grating instrument. Elemental analyses were performed by Schwarzkopf Microanalytical Laboratories, Woodside, New York.

$Me_3NCB_{10}H_{10}PR$ (I, R = Ph; II, R = CH_3; III, R = C_2H_5)

All three compounds were prepared in the same manner, only the preparation of $Me_3NCB_{10}H_{10}PC_6H_5$ which was used for the crystal structure determination will be described in detail. One gram (5.2 mmol) of $B_{10}H_{12}CNMe_3$ was dissolved by stirring with a bar magnet in 75 ml of tetrahydrofuran in a nitrogen flushed 250 ml 3-neck round-bottom flask equipped with condenser and two pressure equalizing dropping funnels. To one dropping funnel was added an excess of triethylamine (1.8 ml); to the other was added 0.7 ml $C_6H_5PCl_2$ dissolved in 15—20 ml of tetrahydrofuran. The triethylamine was added dropwise over a period of three to five minutes. The $C_6H_5PCl_2$ in tetrahydrofuran was then added dropwise over a period of 40 minutes. During this time there was a change from a clear colorless solution to a cloudy light yellow mixture. After all the $C_6H_5PCl_2$ had been added, the solution was gently refluxed for 24 h, then allowed to return to room temperature and stirred under these conditions for 16 h, all volatiles were removed from the flask in vacuo leaving a light yellow solid coating the flask walls. Acetone (100 ml) was added to the flask and stirred for 15 minutes. Filtration through a medium-porosity frit gave a white solid on the frit (identified as $(Et_3NH)Cl$ by infrared and ^1H NMR) and a clear yellowish filtrate. The volume of filtrate was reduced, neutral alumina was added to the solution and the solid slurry was placed on top of a neutral alumina column (30 × 2.5 cm). The compound was eluted as a fast moving band with acetone. The desired compound was further purified by recrystallization from either acetone, acetone/methanol or acetonitrile to give $Me_3NCB_{10}H_{10}PC_6H_5$ (I) 1.29 g (83% yield), m.p. 295—296°C; By this same procedure $Me_3NCB_{10}H_{10}PCH_3$ (II) was obtained in 85% yield, and $Me_3NCB_{10}H_{10}PC_2H_5$ (III) was obtained in 88% yield.

Elemental analyses, infrared, ^1H and ^{11}B NMR data are given in Tables 1—3.

Single crystal X-ray study of $Me_3NCB_{10}H_{10}PPh$

A well formed crystal (0.35 × 0.30 × 0.40 mm) of I was cooled to —160

TABLE 1

CHEMICAL ANALYSES

Compound	Analysis (Found (Calcd.) (%))	
	C	H
$Me_3NCB_{10}H_{10}PC_6H_5$ (I)	40.16 (40.38)	8.14 (8.13)
$Me_3NCB_{10}H_{10}PCH_3$ (II)	25.82 (25.52)	9.40 (9.42)
$Me_3NCB_{10}H_{10}PC_2H_5$ (III)	28.93 (28.90)	9.59 (9.70)

TABLE 2

INFRARED SPECTRA

Compound	Wavenumber (cm^{-1})
I	3050m, 2570s, 2520s, 1480s, 1458s, 1430s, 1410m, 1310m, 1275w, 1258w, 1240w, 1188w, 1160w, 1128w, 1114w, 1092w, 1068m, 1036m, 998m, 970w, 906s, 878w, 862m, 792m, 742s, 725w
II	2962w, 2894w, 2585s, 2535s, 1482m, 1458m, 1408m, 1260m, 1238w, 1190w, 1104m, 1028m, 996w, 945w, 910m, 888w, 860m, 792m, 742w, 725w
III	2965w, 2580m, 2530s, 1478m, 1454m, 1405m, 1260m, 1210w, 1095w, 1024m, 905m, 859w, 800m, 724w, 738w

± 5°C on a locally constructed diffractomer system [9] equipped with a gaseous nitrogen cooling system. A systematic search of a limited region of reciprocal space located sufficient reflections to unambiguously assign the space group *Pbca* (No. 61, D_{2h}^{15}). A least squares fit of angular settings from 16 reflections centered in $\pm 2\theta$ gave final cell parameters (at −160°C) of: a 21.572(11), b 8.797(5), and c 17.949(10) Å, V 3406.2 Å^3 and D 1.160 g/cm^3 for $Z = 8$. The linear absorption coefficient (1.425 cm^{-1}) for the graphite monochromatized Mo-K_2 radiation (λ 0.71069 Å) was sufficiently small that no absorption correction was attempted.

A total of 2240 unique reflections were collected using $\theta - 2\theta$ scan techniques (angular rate of 3.0°/min over a range symmetrically disposed 1° on either side of the calculated K_{α_1} and K_{α_2} positions; 10 second backgrounds) for the region $5° < 2\theta < 50°$. Of the 2240 unique reflections 2061 were non-zero and were used in all subsequent calculations. The data were corrected for Lorentz and polarization terms in the usual manner [10], using an ignorance factor of 0.06.

TABLE 3

^{11}B AND ^1H NMR DATA

Compound	^{11}B chemical shift (ppm) ($J(BH)(Hz)$)^a	^1H chemical shift (ppm)	Assignment^a
I	1.5	7.4, multiplet	phenyl group
	−4.08(142)	3.3, singlet	NMe_3 group
	−8.42(159)		
	−13.42(142)		
	−20.18(144)		
II	0.94(146)	3.3, singlet	NMe_3 group
	−3.92(139)	1.4, $J(^{31}P{-}H)$ 10 Hz	PMe group
	−8.18(154)		
	−11.90(142)		
	−21.86(146)		
III	0.81(149)	3.3, singlet	NMe_3 group
	−3.92(134)	1.55, multiplet	$P{-}\underline{CH_2}{-}CH_3$
	−8.13(147)	1.08, doublet of triplets	$P{-}CH_2{-}\underline{CH_3}$
	−13.14(142)		
	−22.34(142)		

^{a} Acetone-d_6 solvent.

TABLE 4

FRACTIONAL COORDINATES OF ATOMS

Atom	$10^4 X$	$10^4 Y$	$10^4 Z$	$^{10}B_{iso}$
B(1)	3395(3)	1673(6)	775(3)	27
B(2)	3321(3)	3003(6)	1497(3)	23
B(3)	3395(2)	3631(6)	565(3)	22
B(4)	3930(2)	2404(6)	106(3)	22
B(5)	4146(2)	915(6)	738(3)	24
B(6)	3803(2)	1422(6)	1623(3)	22
C(7)	3833(2)	4395(4)	1252(2)	18
B(8)	4228(2)	4128(6)	478(3)	20
B(9)	4688(2)	2346(6)	495(3)	20
B(10)	4599(2)	1709(6)	1464(3)	22
B(11)	4100(2)	3221(6)	1885(3)	22
P(12)	5066(1)	3661(1)	1280(1)	21
N(13)	3706(2)	6058(4)	1484(2)	21
C(14)	4310(3)	6813(6)	1697(3)	30
C(15)	3420(2)	6972(6)	868(3)	26
C(16)	3283(3)	6151(7)	2141(3)	31
C(17)	5874(2)	2986(5)	1216(2)	20
C(18)	6311(2)	4091(5)	1396(3)	23
C(19)	6939(2)	3799(6)	1323(3)	31
C(20)	7138(2)	2402(6)	1059(3)	29
C(21)	6714(2)	1289(6)	890(2)	27
C(22)	6084(2)	1561(5)	977(2)	25
H(1)	3025(18)	947(41)	591(20)	31(10)
H(2)	2879(18)	3319(43)	1820(21)	39(10)
H(3)	3029(17)	4217(38)	278(18)	23(9)
H(4)	3891(15)	2203(37)	−532(21)	24(8)
H(5)	4229(15)	−215(37)	568(19)	17(8)
H(6)	3702(17)	592(43)	2022(21)	31(10)
H(7)	4339(15)	5078(37)	129(18)	14(8)
H(8)	5013(18)	2068(37)	98(20)	22(8)
H(9)	4911(14)	1009(35)	1761(17)	9(7)
H(10)	4132(15)	3613(39)	2492(20)	24(8)
H(11)	4596(18)	6817(46)	1281(23)	32(11)
H(12)	4216(18)	7819(46)	1873(2)	30(11)
H(13)	4486(21)	6258(50)	2157(25)	50(13)
H(14)	3417(15)	8074(42)	1021(19)	14(8)
H(15)	3680(16)	6950(38)	411(21)	13(9)
H(16)	3007(19)	6608(43)	752(20)	26(10)
H(17)	3246(19)	7165(47)	2307(22)	31(11)
H(18)	3463(20)	5540(51)	2526(23)	42(13)
H(19)	2869(17)	5828(38)	1966(18)	12(9)
H(20)	6170(16)	5102(43)	1566(19)	21(9)
H(21)	7228(16)	4484(43)	1491(20)	25(10)
H(22)	7574(19)	2213(43)	1000(20)	30(10)
H(23)	6844(23)	183(57)	762(26)	69(15)
H(24)	5795(17)	792(40)	873(19)	19(10)

All non-hydrogen atoms were readily located by direct methods and Fourier techniques *, and after two cycles of isotropic refinement all hydrogen atoms were readily located in a difference Fourier synthesis. The structure was then refined by full-matrix least squares in which hydrogen atoms were assigned iso-

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* All computations were performed on a CDC6600-CYBER172 system using the IUMSC interactive XTEL program library. The latter is based in part on A.C. Larson LASL code and J.A. Ibers Northwestern University Program Library.

TABLE 5

ANISOTROPIC THERMAL PARAMETERS

Atom	$10^4 b_{11}$	$10^4 b_{22}$	$10^4 b_{33}$	$10^4 b_{12}$	$10^4 b_{13}$	$10^4 b_{23}$
B(1)	9(1)	78(9)	30(2)	−4(3)	−0(1)	−2(4)
B(2)	13(1)	64(8)	19(2)	−2(3)	3(1)	−5(3)
B(3)	10(1)	61(7)	22(2)	−0(3)	0(1)	−8(4)
B(4)	8(1)	91(8)	19(2)	2(3)	−2(1)	−7(3)
B(5)	13(1)	71(9)	21(2)	2(3)	0(1)	−1(3)
B(6)	15(1)	56(8)	18(2)	−0(3)	5(1)	8(3)
C(7)	11(1)	53(6)	15(2)	1(2)	1(1)	−1(3)
B(8)	11(1)	58(8)	16(2)	4(3)	−2(1)	7(3)
B(9)	9(1)	68(8)	16(2)	2(3)	−0(1)	−0(3)
B(10)	13(1)	58(8)	19(2)	3(3)	0(1)	4(3)
B(11)	14(1)	64(8)	17(2)	−2(2)	3(1)	−4(3)
P(12)	11(0)	66(2)	18(0)	3(1)	−1(0)	0(1)
N(13)	12(1)	59(5)	17(1)	2(2)	0(1)	−5(2)
C(14)	16(1)	84(9)	28(2)	1(3)	−2(1)	−19(4)
C(15)	15(1)	59(7)	26(2)	−0(3)	−3(1)	4(3)
C(16)	19(2)	94(9)	24(2)	11(3)	4(1)	−16(4)
C(17)	12(1)	79(7)	10(2)	−1(2)	−1(1)	6(3)
C(18)	13(1)	57(7)	21(2)	3(2)	−1(1)	4(3)
C(19)	13(1)	111(8)	26(2)	−9(3)	−2(1)	10(4)
C(20)	10(1)	121(9)	24(2)	12(3)	5(1)	17(3)
C(21)	15(1)	81(7)	22(2)	9(3)	−2(1)	2(3)
C(22)	13(1)	69(7)	23(2)	0(3)	−3(1)	−0(3)

Form of the thermal parameter is: $\exp[-(h^2b_{11} + k^2b_{22} + l^2b_{33} + 2\,hkb_{12} + 2\,hlb_{13} + 2klb_{23})]$

tropic thermal parameters and non-hydrogen atoms were assigned anisotropic thermal parameters. The goodness of fit of the last cycle was 0.74, and the maximum shift/error was 0.02. An isotropic extinction parameter did not differ significantly from zero and was excluded in the last cycles. Final residuals for the non-zero data are $R(F) = 0.094$ and $Rw(F) = 0.042$, where $R(F) = \Sigma||F_o| - |F_c||/\Sigma|F_o|$, $Rw(F) = [\Sigma w(|F_o| - |F_c|)^2/\Sigma w F_o^2]^{1/2}$ and $w = 1/\sigma(F_o)$. Final positional and thermal parameters are listed in Tables 4 and 5, and observed and calculated structure amplitudes are available *. Bond distances and angles are given in Tables 6 and 7.

Results and discussion

In the first attempt to prepare these compounds, $Me_3NCB_{10}H_{12}$ was deprotonated with sodium hydride in the THF solvent followed by reaction with $RPCl_2$. This did not lead to the expected $Me_3NCB_{10}H_{10}PR$ in our hands. A material which was isolated appeared to be an isomer of the starting carboranes in which the NMe_3 group had migrated from carbon to boron [11]. Use of trimethylamine as the deprotonation reagent and subsequent reaction with $RPCl_2$ (R = Ph, Me or Et) gave the desired twelve-atom *nido*-heteroatom boranes in good yield. These compounds are high melting solids which are stable in air for several weeks

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* A complete summary of crystallographic data for $Me_3NCB_{10}H_{10}PPh$ is available in microfiche form only for $ 2.50 from the Chemistry Library, Indiana University, Bloomington, Indiana 47401. Request Molecular Structure Center Report No. 7735.

TABLE 6

IMPORTANT DISTANCES IN THE MOLECULE, IN Å

P(12)—C(17)	1.845(4)	C(14)—H(11)	0.97(4)
P(12)—B(8)	2.347(5)	C(14)—H(12)	0.96(4)
P(12)—B(9)	1.998(5)	C(14)—H(13)	1.03(5)
P(12)—B(10)	2.018(5)	C(15)—H(14)	1.01(4)
P(12)—B(11)	2.381(5)	C(15)—H(15)	0.99(4)
N(13)—C(7)	1.545(5)	C(15)—H(16)	0.97(4)
N(13)—C(14)	1.512(6)	C(16)—H(17)	0.94(4)
N(13)—C(15)	1.500(5)	C(16)—H(18)	0.96(4)
N(13)—C(16)	1.493(6)	C(16)—H(19)	0.99(3)
C(7)—B(2)	1.706(6)	C(18)—H(20)	0.99(4)
C(7)—B(3)	1.692(6)	C(19)—H(21)	0.92(4)
C(7)—B(8)	1.647(6)	C(20)—H(22)	0.96(4)
C(7)—B(11)	1.640(6)	C(21)—H(23)	1.04(5)
C(17)—C(18)	1.392(5)	C(22)—H(24)	0.94(3)
C(17)—C(22)	1.400(6)	B(1)—H(1)	1.07(4)
C(18)—C(19)	1.384(6)	B(2)—H(2)	1.15(4)
C(19)—C(20)	1.384(7)	B(3)—H(3)	1.08(3)
C(20)—C(21)	1.374(6)	B(4)—H(4)	1.16(4)
C(21)—C(22)	1.389(6)	B(5)—H(5)	1.06(3)
B(1)—B(2)	1.752(7)	B(6)—H(6)	1.05(4)
B(1)—B(3)	1.764(7)	B(8)—H(7)	1.07(3)
B(1)—B(4)	1.787(7)	B(9)—H(8)	1.03(4)
B(1)—B(5)	1.755(7)	B(10)—H(9)	1.06(3)
B(1)—B(6)	1.773(8)	B(11)—H(10)	1.15(3)
B(2)—B(3)	1.768(7)		
B(2)—B(6)	1.752(7)		
B(2)—B(11)	1.830(7)		
B(3)—B(4)	1.782(7)		
B(3)—B(8)	1.855(7)		
B(4)—B(5)	1.795(7)		
B(4)—B(8)	1.778(7)		
B(4)—B(9)	1.778(6)		
B(5)—B(6)	1.808(7)		
B(5)—B(9)	1.772(7)		
B(5)—B(10)	1.772(7)		
B(6)—B(10)	1.759(7)		
B(6)—B(11)	1.770(7)		
B(8)—B(9)	1.855(7)		
B(9)—B(10)	1.839(7)		
B(10)—B(11)	1.870(7)		

but decompose in about one day in a solution exposed to the atmosphere. The ^{1}H NMR spectra of compounds II and III show the presence of ^{31}P—^{1}H coupling on signals in the 1 to 1.5 ppm region which strongly suggested that the P-alkyl group had been incorporated into the molecule. The 70.6 MHz ^{11}B NMR spectra of these derivatives consists of five doublets reading upfield with the area ratios 3/1/2/2/2. The simple ^{11}B NMR spectra suggest that these molecules may either have a mirror plane of symmetry or are fluxional at room temperature. The symmetrical nature of these compounds was confirmed by a single-crystal X-ray structure determination of I which is illustrated in Fig. 1. The "P—Ph" unit bridges B(9) and B(10) on the open face of the $B_{10}H_{10}CNMe_3$ icosahedral fragment. This structure is very similar to that previously reported for the twelve-atom *nido*-carborane $PhCHB_{10}H_{10}CPh^-$ [12]. If the "PPh" and "$CNMe_3$" units of I are replaced with the isoelectronic "PhCH" and "CPh" units respectively the two structures are seen to be similar.

TABLE 7

IMPORTANT ANGLES IN THE MOLECULE IN DEGREES

C(17)—P(12)—B(9)	99.0(2)
C(17)—P(12)—B(10)	102.0(2)
B(9)—P(12)—B(10)	54.5(2)
C(7)—N(13)—C(14)	109.3(3)
C(7)—N(13)—C(15)	112.5(3)
C(7)—N(13)—C(16)	111.9(4)
C(14)—N(13)—C(15)	107.9(4)
C(14)—N(13)—C(16)	107.6(4)
C(15)—N(13)—C(16)	107.5(4)
N(13)—C(7)—B(2)	119.7(3)
N(13)—C(7)—B(3)	118.2(3)
N(13)—C(7)—B(8)	117.0(3)
N(13)—C(7)—B(11)	118.2(3)
B(2)—C(7)—B(3)	62.7(3)
B(2)—C(7)—B(8)	116.7(3)
B(2)—C(7)—B(11)	66.3(3)
B(3)—C(7)—B(8)	67.5(3)
B(3)—C(7)—B(11)	116.8(4)
B(8)—C(7)—B(11)	108.2(3)
P(12)—C(17)—C(18)	113.7(3)
P(12)—C(17)—C(22)	127.7(3)
C(18)—C(17)—C(22)	118.5(4)
C(17)—C(18)—C(19)	120.7(4)
C(18)—C(19)—C(20)	120.0(5)
C(19)—C(20)—C(21)	120.2(5)
C(20)—C(21)—C(22)	120.2(5)
C(17)—C(22)—C(21)	120.4(4)
B(2)—B(1)—B(3)	60.4(3)
B(2)—B(1)—B(4)	108.4(4)
B(2)—B(1)—B(5)	111.5(4)
B(2)—B(1)—B(6)	59.6(3)
B(3)—B(1)—B(4)	60.3(3)
B(3)—B(1)—B(5)	111.2(4)
B(3)—B(1)—B(6)	107.7(4)
B(4)—B(1)—B(5)	60.9(3)
B(4)—B(1)—B(6)	107.5(4)
B(5)—B(1)—B(6)	61.7(3)
C(7)—B(2)—B(1)	103.3(4)
C(7)—B(2)—B(3)	58.2(3)
C(7)—B(2)—B(6)	102.6(4)
C(7)—B(2)—B(11)	55.1(3)
B(1)—B(2)—B(3)	60.1(3)
B(1)—B(2)—B(6)	60.8(3)
B(1)—B(2)—B(11)	105.5(4)
B(3)—B(2)—B(6)	108.5(4)
B(3)—B(2)—B(11)	104.1(4)
B(6)—B(2)—B(11)	59.2(3)
C(7)—B(3)—B(1)	103.4(4)
C(7)—B(3)—B(2)	59.1(3)
C(7)—B(3)—B(4)	102.5(3)
C(7)—B(3)—B(8)	55.1(3)
B(1)—B(3)—B(2)	59.5(3)
B(1)—B(3)—B(4)	60.5(3)
B(1)—B(3)—B(8)	104.4(4)
B(2)—B(3)—B(4)	107.9(4)
B(2)—B(3)—B(8)	104.0(4)
B(4)—B(3)—B(8)	58.5(3)
B(1)—B(4)—B(3)	59.2(3)
B(1)—B(4)—B(5)	58.7(3)
B(1)—B(4)—B(8)	106.7(4)
B(1)—B(4)—B(9)	108.6(4)
B(3)—B(4)—B(5)	108.5(4)
B(3)—B(4)—B(8)	62.8(3)
B(3)—B(4)—B(9)	115.5(4)
B(5)—B(4)—B(8)	106.9(3)
B(5)—B(4)—B(9)	59.4(3)
B(8)—B(4)—B(9)	62.9(3)
B(1)—B(5)—B(4)	60.4(3)
B(1)—B(5)—B(6)	59.7(3)
B(1)—B(5)—B(9)	110.4(4)
B(1)—B(5)—C(10)	109.4(4)
B(4)—B(5)—B(6)	105.6(4)
B(4)—B(5)—B(9)	59.8(3)
B(4)—B(5)—B(10)	108.7(4)
B(6)—B(5)—B(9)	108.1(4)
B(6)—B(5)—B(10)	58.8(3)
B(9)—B(5)—B(10)	62.5(3)
B(1)—B(6)—B(2)	59.6(3)
B(1)—B(6)—B(5)	58.7(3)
B(1)—B(6)—B(10)	109.2(4)
B(1)—B(6)—B(11)	107.2(4)
B(2)—B(6)—B(5)	109.0(4)
B(2)—B(6)—B(10)	116.4(4)
B(2)—B(6)—B(11)	62.6(3)
B(5)—B(6)—B(10)	59.6(3)
B(5)—B(6)—B(11)	107.8(4)
B(10)—B(6)—B(11)	64.0(3)
C(7)—B(8)—B(3)	57.4(3)
C(7)—B(8)—B(4)	104.6(4)
C(7)—B(8)—B(9)	112.6(4)
B(3)—B(8)—B(4)	58.7(3)
B(3)—B(8)—B(9)	108.5(3)
B(4)—B(8)—B(9)	58.6(3)
P(12)—B(9)—B(4)	129.5(3)
P(12)—B(9)—B(5)	120.4(3)
P(12)—B(9)—B(8)	75.0(2)
P(12)—B(9)—B(10)	63.3(2)
B(4)—B(9)—B(5)	60.8(3)
B(4)—B(9)—B(8)	58.5(3)
B(4)—B(9)—B(10)	106.6(4)
B(5)—B(9)—B(8)	104.6(3)
B(5)—B(9)—B(10)	58.8(3)
B(8)—B(9)—B(10)	102.5(4)
P(12)—B(10)—B(5)	119.3(3)
P(12)—B(10)—B(6)	129.5(3)
P(12)—B(10—B(9)	62.2(2)
P(12)—B(10)—B(11)	75.4(2)
B(5)—B(10)—B(6)	61.6(3)
B(5)—B(10)—B(9)	58.7(3)
B(5)—B(10)—B(11)	105.1(4)
B(6)—B(10)—B(9)	107.4(4)
B(6)—B(10)—B(11)	58.3(3)
B(9)—B(10)—B(11)	103.0(3)
C(7)—B(11)—B(2)	58.6(3)
C(7)—B(11)—B(6)	104.6(4)
C(7)—B(11)—B(10)	111.8(4)
B(2)—B(11)—B(6)	58.2(3)
B(2)—B(11)—B(10)	107.5(3)
B(6)—B(11)—B(10)	57.7(3)
N(13)—C(14)—H(11)	110.8(24)
N(13)—C(14)—H(13)	107.8(25)
N(13)—C(14)—H(13)	108.2(26)
H(11)—C(14)—H(12)	112.6(35)
H(11)—C(14)—H(13)	112.7(35)
H(12)—C(14)—H(13)	104.5(34)
N(13)—C(15)—H(14)	108.5(20)
N(13)—C(15)—H(15)	111.4(20)
N(13)—C(15)—H(16)	111.1(23)
H(14)—C(15)—H(15)	104.3(28)
H(14)—C(15)—H(16)	111.8(31)
H(15)—C(15)—H(16)	109.6(31)
N(13)—C(16)—H(17)	110.7(25)
N(13)—C(16)—H(18)	107.0(26)
N(13)—C(16)—H(19)	106.6(20)
H(17)—C(16)—H(18)	109.7(36)
H(17)—C(16)—H(19)	107.2(32)
H(18)—C(16)—H(19)	115.7(34)
C(17)—C(18)—H(20)	119.4(21)
C(19)—C(18)—H(20)	119.9(21)
C(18)—C(19)—H(21)	120.8(24)
C(20)—C(19)—H(21)	119.0(24)
C(19)—C(20)—H(22)	119.6(24)
C(21)—C(20)—H(22)	120.2(24)
C(20)—C(21)—H(23)	122.6(28)
C(22)—C(21)—H(23)	116.8(28)
C(17)—C(22)—H(24)	119.5(23)
C(21)—C(22)—H(24)	120.2(23)
B(2)—B(1)—H(1)	123.9(21)
B(3)—B(1)—H(1)	121.1(20)
B(4)—B(1)—H(1)	119.1(21)
B(4)—B(1)—H(1)	116.6(21)
B(6)—B(1)—H(1)	124.0(20)
C(7)—B(2)—H(2)	119.5(20)
B(1)—B(2)—H(2)	127.6(20)
B(3)—B(2)—H(2)	118.5(20)
B(6)—B(2)—H(2)	128.2(20)
B(11)—B(2)—H(2)	123.0(19)
C(7)—B(3)—H(3)	124.7(19)
B(1)—B(3)—H(3)	124.7(19)
B(2)—B(3)—H(3)	122.4(19)
B(4)—B(3)—H(3)	123.0(18)
B(8)—B(3)—H(3)	124.0(19)
B(1)—B(4)—H(4)	120.1(17)
B(3)—B(4)—H(4)	120.1(17)
B(3)—B(4)—H(4)	120.1(17)
B(5)—B(4)—H(4)	122.1(17)
B(8)—B(4)—H(4)	121.8(17)
B(9)—B(4)—H(4)	116.7(17)
B(1)—B(5)—H(5)	121.7(19)
B(6)—B(5)—H(5)	123.2(20)
B(6)—B(5)—H(5)	123.8(19)
B(9)—B(5)—H(5)	119.1(19)
B(10)—B(5)—H(5)	119.5(19)
B(1)—B(6)—H(6)	124.8(21)
B(2)—B(6)—H(6)	121.3(21)
B(5)—B(6)—H(6)	121.0(21)
B(10)—B(6)—H(6)	114.5(21)
B(11)—B(6)—H(6)	121.2(21)
C(7)—B(8)—H(7)	119.8(18)
B(3)—B(8)—H(7)	116.7(18)
B(4)—B(8)—H(7)	121.8(18)
B(9)—B(8)—H(7)	123.3(18)
P(12)—B(9)—H(8)	110.3(21)
B(4)—B(9)—H(8)	111.3(21)
B(5)—B(9)—H(8)	116.9(20)
B(8)—B(9)—H(8)	123.7(20)
B(10)—B(9)—H(8)	130.8(20)
P(12)—B(10)—H(9)	105.1(17)
B(5)—B(10)—H(9)	119.4(17)
B(6)—B(10)—H(9)	117.2(17)
B(9)—B(10)—H(9)	126.0(17)
B(11)—B(10)—H(9)	125.4(17)
C(7)—B(11)—H(10)	119.4(18)
B(2)—B(11)—B(11)	116.7(17)
B(6)—B(11)—H(10)	122.9(18)
B(10)—B(11)—H(10)	124.3(18)

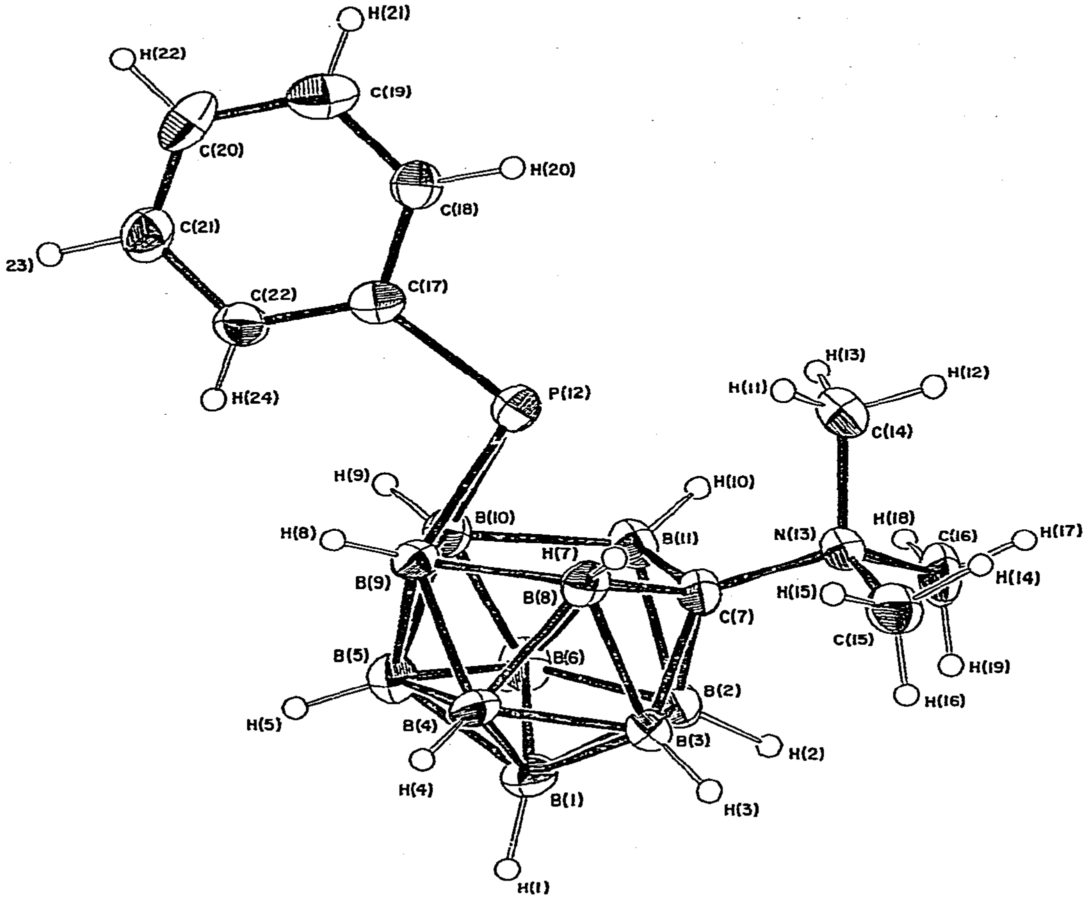

Fig. 1. ORTEP drawing of $Me_3NCB_{10}H_{10}PPh$. All thermal ellipsoids are drawn at the 50% probability level, and hydrogens have been assigned an artificial value of $B_{iso} = 0.5$ Å^2 for artistic purposes.

Preliminary studies suggest that heating I at 475°C in a sealed tube formed the *closo* compound $B_{10}H_{10}C(NMe_2)P$.

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