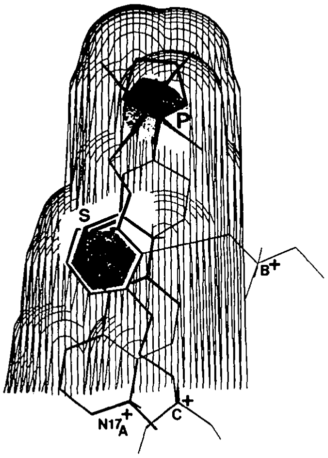

Fig. 6. *BILBO* graphics showing the docking of the gallamine *A*-chain quarternary nitrogen (N) into a proposed topographical receptor site formed by the van der Waals surface of the steroidal neuromuscular blocker chandonium iodide (HS310). The primary (inner) binding site (*P*) of the active centre is formed by the quaternary *A*-ring end of HS310 and the secondary (outer) esterophilic binding site (*S*) by the *B*-ring double bond of HS310 associated with enhanced neuromuscular blocking activity (Marshall, Paul & Singh, 1973).

molecular geometry of gallamine, determined in the present analysis, keys into the topography of the previously proposed active-site model (Palmer *et al.*, 1983). In this example the object molecule forming the active-site pocket is chandonium (Fig. 1*d*). The cationic head N_A^+ of gallamine is buried deeply within the pocket (*P*) and the phenyl group (φ) aligns with the C=C bond of chandonium, associated with enhanced potency, in the secondary outer binding site (*S*). This diagram also shows the close correspondence between the other cationic sites, $\mathrm{N}(17)_A^+$ in chandonium and N_C^+ of gallamine, when the two molecules are superposed in this way. As mentioned previously the $\mathrm{N}_A^+\cdots\mathrm{N}_C^+$ separation in gallamine conforms to the requirements of Pauling & Petcher's (1973) model [as does the $\mathrm{N}(31)^+\cdots\mathrm{N}(17)_A^+$ separation of chandonium]. The proposed acetylcholine receptor model does not preclude the existence of two or more similar binding sites in the actual receptor assembly and therefore leaves open the question why a biscationic design for neuromuscular blocking agents has traditionally been so successful. The third cationic centre of gallamine, N_B^+, is suitably oriented so as not to interfere with the proposed receptor surface, and presumably merely acts to stabilize the molecular conformation and to potentiate the initial attraction of the molecule into the active-site pocket.

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The Structure of Methylenetriphenylphosphorane(*C*–*B*)triborane(7) at 185 K

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Abstract. $C_{19}H_{24}B_3P$, $M_r = 315{\cdot}8$, monoclinic, $P2_1/n$, $a = 13{\cdot}854\ (3)$, $b = 8{\cdot}860\ (2)$, $c = 15{\cdot}667\ (2)$ Å, $\beta = 104{\cdot}66\ (1)°$, $U = 1860{\cdot}5$ Å^3, $Z = 4$, $D_x = 1{\cdot}127$ Mg m^{-3}, Mo $K\alpha$, $\lambda = 0{\cdot}71069$ Å, $\mu = 0{\cdot}138$ mm^{-1}, $F(000) = 672$, $T = 185$ K, $R = 0{\cdot}0695$ for 2159 observed reflections. The title compound is zwitterionic, with the P atom positively charged and the negative charge delocalized over the borane ring. The

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boron triangle has one fully bridging hydrogen atom opposite and one semi-bridging hydrogen atom adjacent to the substituted atom. The relative conformation of the phenyl groups is the same as that found in other coordinated triphenylphosphines.

Introduction. The stereochemistries of substituted triboranes are delineated by the number and nature of bridging hydrogen atoms. A single H bridge opposite the substituted atom is found in B_3H_7CO (Glore, Rathke & Schaeffer, 1973), whereas in $[B_3H_6Cl_2]^-$ (Arunchaiya, Morris, Andrews, Welch & Welch, 1984) and $[B_3H_8]^-$ itself (Peters & Nordman, 1960) there are two bridging hydrogen atoms. However, in $B_3H_7NH_3$ (Nordman & Reimann, 1959) and $[B_3H_7X]^-$ (X = NCS, NCSe; Andrews & Welch, 1984) an intermediate arrangement is observed with a semi-bridging H (more closely bound to the substituted atom) in addition to the (opposite) full hydrogen bridge. It has been suggested (Glore *et al.*, 1973; Brown & Lipscomb, 1977) that these results are a consequence of the σ-donor character of the substituent, but, except for the NH_3 derivative, an alternative explanation could be based on the substituent's π-acceptor ability.

Seeking further structural data on substituted triboranes where π interaction between the borane and substituent is not possible, we have studied the title compound in which a CH_2 function separates the B_3H_7 and PPh_3 fragments. The compound is prepared by reaction between the ylide Ph_3PCH_2 (prepared *in situ*) and $B_3H_7.H_4$furan, and was recrystallized from dichloromethane (Choi & Morris, 1984).

Experimental. White block, 0·05 × 0·04 × 0·04 cm, mounted under N_2 in Lindemann tube; space group and preliminary cell dimensions from oscillation and Weissenberg photography; accurate parameters using CAD-4 diffractometer, 185 K (ULT-1 apparatus), 25 reflections ($13 < \theta < 15°$) centred, graphite-monochromated Mo $K\alpha$; for data collection $\theta_{\rm max} = 25°$, ω–2θ scans in 96 steps, ω-scan width $(0{\cdot}8 + 0{\cdot}35\tan\theta)°$, rapid prescan after which reflections with $I \geq 1{\cdot}0\sigma(I)$ remeasured such that final net intensity had $I > 50\sigma(I)$ subject to max. measuring time of 90 s; 3271 unique data measured over 180 X-ray hours with no detectable decay or movement; for structure solution and refinement 2159 amplitudes $[F \geq 2{\cdot}0\sigma(F)]$; automatic centrosymmetric direct methods (P) and iterative refinement/ΔF syntheses (C,B,H), full-matrix least squares (F), $w^{-1} = [\sigma^2(F) + 0{\cdot}000955(F)^2]$; anisotropic thermal parameters for all non-H atoms, phenyl groups idealized (C—C 1·395, C—H 1·08 Å), methylene H atoms idealized (C—H 1·08 Å), borane H atoms positionally refined ($U_{\rm H}^* = 0{\cdot}05$ Å^2); R 0·0695, wR 0·0858; data: variable ratio 11:1; max. Δ/σ in final cycle <0·01; max. peak and min. trough in final ΔF synthesis 0·34 and −0·31 e Å^{-3}; neutral atom scattering factors for C, B and P (Cromer & Liberman, 1970) and H (Stewart, Davidson & Simpson, 1965); computer programs: *SHELX*76 (Sheldrick, 1976), *XANADU* (Roberts & Sheldrick, 1976), *CALC* (Gould & Taylor, 1984), and *ORTEP*II (Johnson, 1976).

Table 1. *Fractional coordinates of refined atoms*

$$U_{\rm eq} = \tfrac{1}{3}\textstyle\sum_i\sum_j U_{ij}a^*_ia^*_j\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$U_{\rm eq}$(Å^2)
P(1)	0·98452 (8)	0·00774 (14)	0·25135 (7)	0·0279
C(12)	1·11892 (18)	−0·1116 (3)	0·16390 (18)	0·0361
C(13)	1·14531	−0·2055	0·10197	0·0477
C(14)	1·07260	−0·2917	0·04472	0·0451
C(15)	0·97350	−0·2842	0·04940	0·0444
C(16)	0·94711	−0·1903	0·11133	0·0370
C(11)	1·01981	−0·1040	0·16858	0·0300
C(22)	0·78675 (20)	−0·0271 (4)	0·25580 (17)	0·0403
C(23)	0·68414	−0·0108	0·22112	0·0508
C(24)	0·64786	0·0710	0·14355	0·0452
C(25)	0·71420	0·1366	0·10067	0·0447
C(26)	0·81681	0·1203	0·13536	0·0400
C(21)	0·85308	0·0384	0·21292	0·0304
C(32)	1·07653 (23)	0·2502 (3)	0·19021 (16)	0·0383
C(33)	1·11497	0·3964	0·19649	0·0476
C(34)	1·12070	0·4815	0·27255	0·0504
C(35)	1·08798	0·4202	0·34234	0·0465
C(36)	1·04953	0·2740	0·33606	0·0409
C(31)	1·04330	0·1889	0·26000	0·0304
C(1)	1·0168 (3)	−0·0881 (6)	0·3536 (3)	0·0357
B(1)	1·1360 (4)	−0·1146 (7)	0·3952 (4)	0·0343
B(2)	1·1576 (5)	−0·2358 (7)	0·4916 (4)	0·0461
B(3)	1·1901 (5)	−0·0428 (7)	0·5037 (4)	0·0424
HB(11)	1·167 (4)	−0·002 (6)	0·401 (3)	
HB(12)	1·170 (4)	−0·166 (5)	0·348 (3)	
HB(21)	1·222 (3)	−0·325 (6)	0·491 (3)	
HB(22)	1·078 (4)	−0·287 (5)	0·489 (3)	
HB(31)	1·269 (4)	−0·017 (5)	0·530 (3)	
HB(32)	1·133 (4)	0·044 (5)	0·521 (3)	
H('23)	1·182 (3)	−0·155 (6)	0·561 (3)	

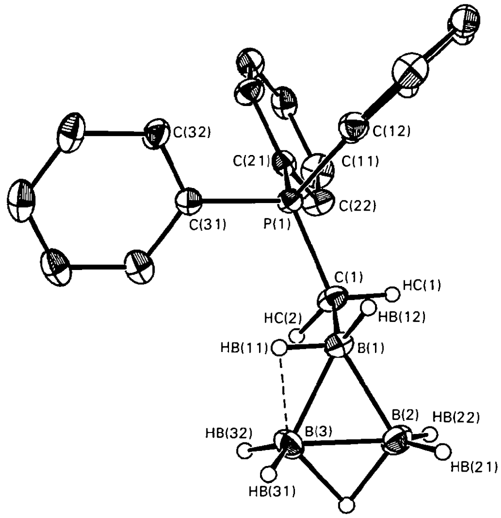

Fig. 1. View of the molecule $Ph_3PCH_2B_3H_7$. Atoms in phenyl rings numbered cyclically, C(ij), j = 1–6. Phenyl hydrogens bear the same number as their respective carbon atom.

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* The isotropic thermal parameter defined as $\exp[-8\pi^2U(\sin^2\theta)/\lambda^2]$.

Discussion. Table 1 lists final fractional coordinates.* A view of the molecule normal to the plane of the B_3 triangle is given in Fig. 1, from which phenyl H atoms have been removed and in which other H atoms are given an artificial radius of 0·1 Å for clarity. Table 2 lists derived molecular parameters, uncorrected for thermal effects.

Although possessing overall electroneutrality, the compound is zwitterionic in character, with a positive charge localized on the phosphorus atom and a negative charge delocalized over the borane fragment. The structural analysis clearly shows that the arrangement of μ-H atoms in 1-Ph_3PCH_2–B_3H_7 (1) is similar to that in $B_3H_7NH_3$ (2), $[B_3H_7NCS]^-$ (3), and $[B_3H_7NCSe]^-$ (4), having one H atom, H('23), fully bridging the B(2)—B(3) connectivity, and another, HB(11), in a semi-bridging mode, being strongly bound to B(1), 1·08 (5) Å, but only weakly linked to B(3), 1·60 (5) Å. By comparison the HB(12)···B(2) separation is non-bonding at 2·38 (5) Å. The B—H('23) distances is also longer to B(3), although not significantly so.

The B—B connectivity lengths measured in (1) are inversely proportional to the degree of H bridging they experience, specifically B(2)—B(3)<B(1)—B(3)<B(1)—B(2). Similar variation in distance with bridging has previously been measured, but not noted, in structural studies on (2)–(4).

In (1) the stereochemistry of the $B(1)H_2$ unit along the B—C bond is staggered, and the relative conformation of the three phenyl groups is closely similar to that found in a number of coordinated triphenylphosphine species in the solid state (Albano, Bellon & Sansoni, 1971; Kelly, Welch & Woodward, 1977). Thus the plane of C(21)–C(26) essentially contains the P(1)—C(1) vector [twist angle 8·2 (3)°], whilst the plane of C(11)–C(16) is almost perpendicular to this bond [77·4 (3)°], and that of C(31)–C(36) is intermediate [34·9 (3)°]. The limiting van der Waals contacts between H(22) and the methylene hydrogen atoms (2·42 and 2·51 Å in the observed structure) result in a relatively wide C(1)—P(1)—C(21) angle.

Fig. 2 views the contents of one unit cell along the *b* crystallographic axis. There are no close contacts between molecules.

We thank the SERC and Borax Research Ltd for support, and Dr J. H. Morris (Strathclyde) for preparing the crystalline sample.

Table 2. *Molecular dimensions* (Å, °)

P(1)–C(11)	1·795 (3)	B(1)–HB(12)	1·07 (5)
P(1)–C(21)	1·788 (3)	B(2)–B(3)	1·766 (9)
P(1)–C(31)	1·793 (3)	B(2)–HB(21)	1·20 (5)
P(1)–C(1)	1·768 (5)	B(2)–HB(22)	1·18 (5)
C(1)–B(1)	1·632 (8)	B(2)–H('23)	1·27 (5)
B(1)–B(2)	1·816 (9)	B(3)–HB(31)	1·10 (5)
B(1)–B(3)	1·793 (9)	B(3)–HB(32)	1·18 (5)
B(1)–HB(11)	1·08 (5)	B(3)–H('23)	1·36 (5)
		B(3)–HB(11)	1·60 (5)
C(11)–P(1)–C(21)	106·32 (14)	B(1)–B(2)–HB(22)	103·8 (24)
C(11)–P(1)–C(31)	110·20 (14)	B(1)–B(2)–H('23)	109·1 (22)
C(11)–P(1)–C(1)	109·72 (19)	B(3)–B(2)–HB(21)	118·2 (24)
C(21)–P(1)–C(31)	107·29 (14)	B(3)–B(2)–HB(22)	125·8 (24)
C(21)–P(1)–C(1)	112·37 (19)	B(3)–B(2)–H('23)	50·0 (22)
C(31)–P(1)–C(1)	110·83 (19)	HB(21)–B(2)–HB(22)	115·9 (33)
P(1)–C(1)–B(1)	115·4 (3)	HB(21)–B(2)–H('23)	110·3 (32)
C(1)–B(1)–B(2)	110·7 (4)	HB(22)–B(2)–H('23)	106·7 (32)
C(1)–B(1)–B(3)	117·7 (4)	B(1)–B(3)–B(2)	61·4 (4)
C(1)–B(1)–HB(11)	104·0 (27)	B(1)–B(3)–HB(31)	124·5 (26)
C(1)–B(1)–HB(12)	111·4 (26)	B(1)–B(3)–HB(32)	107·7 (24)
B(2)–B(1)–B(3)	58·6 (3)	B(1)–B(3)–H('23)	106·3 (20)
B(2)–B(1)–HB(11)	119·8 (27)	B(2)–B(3)–HB(31)	116·5 (26)
B(2)–B(1)–HB(12)	108·3 (26)	B(2)–B(3)–HB(32)	119·3 (24)
B(3)–B(1)–HB(11)	62·0 (26)	B(2)–B(3)–H('23)	45·8 (20)
B(3)–B(1)–HB(12)	130·7 (26)	HB(31)–B(3)–HB(32)	116·4 (35)
HB(11)–B(1)–HB(12)	102·4 (37)	HB(31)–B(3)–H('23)	98·8 (32)
B(1)–B(2)–B(3)	60·1 (3)	HB(32)–B(3)–H('23)	98·7 (31)
B(1)–B(2)–HB(21)	110·6 (24)	B(2)–H('23)–B(3)	84·1 (28)
P(1)–C(11)–C(12)	121·1 (3)		
P(1)–C(11)–C(16)	118·8 (3)		
P(1)–C(21)–C(22)	120·9 (3)		
P(1)–C(21)–C(26)	118·9 (3)		
P(1)–C(31)–C(32)	121·5 (3)		
P(1)–C(31)–C(36)	118·3 (3)		

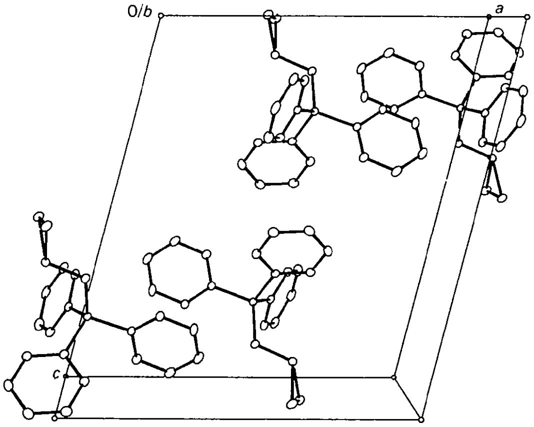

Fig. 2. The crystal packing of $Ph_3PCH_2B_3H_7$.

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* Lists of structure factors, anisotropic thermal parameters and phenyl and methylene H-atom parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 42338 (16 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Structure of 1-(2-Hydroxyethyl)uracil

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Abstract. $C_6H_8N_2O_3$, $M_r = 156{\cdot}14$, orthorhombic, *Pccn*, $a = 10{\cdot}993$ (1), $b = 19{\cdot}032$ (1), $c = 6{\cdot}625$ (1) Å, $V = 1386{\cdot}1$ (2) Å^3, $Z = 8$, $D_m = 1{\cdot}498$, $D_x = 1{\cdot}496$ g cm^{-3}, Cu $K\alpha$, $\lambda = 1{\cdot}54184$ Å, $\mu = 10{\cdot}5$ cm^{-1}, $F(000) = 656$, room temperature, $R = 0{\cdot}068$ for 962 reflexions. The uracil moieties form a pair through N(3)—H···O(2) hydrogen bonds around a twofold axis. The hydroxyl groups are hydrogen-bonded with O(4) atoms of the stacked pairs, constructing the columns along the *c* axis. There are no unusual bond distances or angles.

Introduction. As part of a series of studies on hydrogen bonds between hydroxyl groups and nucleic acid bases (Shibata, Takenaka, Sasada & Ohki, 1985*a,b*), the present paper deals with the structure of 1-(2-hydroxyethyl)uracil.

Experimental. 1-(2-Hydroxyethyl)uracil was synthesized by the reaction of uracil, its sodium salt and ethylene carbonate in dry dimethylformamide at 403 K (Prystaš & Gut, 1962). Purification was by fractional crystallization of a methanol solution, a small amount of acetone being added. Recrystallization from an ethanol solution gave colourless columnar crystals. D_m by flotation in a mixture of chloroform and cyclohexane. Rigaku four-circle diffractometer; graphite-monochromated Cu $K\alpha$ radiation; crystal $0{\cdot}6 \times 0{\cdot}3 \times 0{\cdot}2$ mm; unit-cell dimensions determined with 30 high-angle reflexions; intensities measured for $3 < 2\theta < 125°$; h 0–12, k 0–21, l 0–7; ω-scan mode; scan rate 8° (ω) min^{-1}; scan width 2° (ω); five reference reflexions showed no significant intensity deterioration; corrections for Lorentz and polarization factors, but not for absorption or extinction; 1109 independent reflexions; 126 zero reflexions; standard deviations $\sigma^2(F_o) = \sigma_p^2(F_o) + qF_o^2$, where $\sigma_p(F_o)$ was evaluated by counting statistics and q was estimated to be $5{\cdot}0 \times 10^{-5}$. Structure solved by the direct method; full-matrix least-squares refinement; all H atoms found on difference map; $\sum w(|F_o| - |F_c|)^2$ minimized, where $w = 1/\sigma^2(F_o)$; zero reflexions with $|F_c| > F_{\rm lim}$ ($F_{\rm lim} = 1{\cdot}719$) included in the refinement with the assumption $F_o = F_{\rm lim}$ and $w = w(F_{\rm lim})$; final $R = 0{\cdot}068$ for 962 reflexions with $F_o > 3\sigma$ ($wR = 0{\cdot}078$, $S = 3{\cdot}883$); $(\Delta/\sigma)_{\rm max} = 0{\cdot}1$ for non-H atoms; $\Delta\rho$ peak $0{\cdot}41$ e Å^{-3}; atomic scattering factors from *International Tables for X-ray Crystallography* (1974); program *MULTAN*78 (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978), *LSAP*80 (Takenaka & Sasada, 1980), *DCMS*82 (Takenaka & Sasada, 1982) and *LISTUP* (Takenaka & Sasada, 1983).

Discussion. Final atomic parameters are given in Table 1.* Fig. 1 shows the bond distances and angles. The dimensions of the uracil moiety are in good agreement with those of 1-methyluracil (Green, Mathews & Rich,

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* Lists of structure factors, anisotropic thermal parameters and atomic parameters for H atoms have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 42304 (8 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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