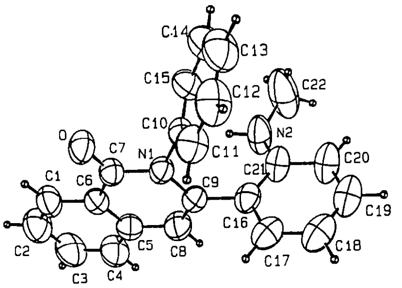

Fig. 1. A perspective view of the molecule (2).

Discussion. The *ORTEP* drawing (Johnson, 1976) of the molecule with atomic numbering scheme is shown in Fig. 1. The atomic parameters of the C, N, O atoms and the H(N) atom are listed in Table 1.* Bond lengths, bond angles and selected torsion angles are given in Table 2. The X-ray structural data reveal intermolecular hydrogen bonding between the NH and amide carbonyl groups, with NH, N···O, H···O distances and N—H···O angle of 0·89, 3·04, 2·15 Å and 150·3°, respectively.

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* Lists of structure factors, anisotropic thermal parameters and H-atom coordinates have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51515 (11 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 $[PhCH_2NMe_3]^+[B_{10}H_{13}]^-$

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Abstract. Benzyltrimethylammonium tridecahydro-*nido*-decaborate(1−), $[PhCH_2NMe_3]^+[B_{10}H_{13}]^-$, $M_r = 271{\cdot}5$, monoclinic, $P2_1/c$, $a = 8{\cdot}8759\,(13)$, $b = 18{\cdot}739\,(3)$, $c = 10{\cdot}8215\,(13)$ Å, $\beta = 96{\cdot}223\,(11)°$, $V = 1789{\cdot}3$ Å^3, $Z = 4$, $D_x = 1{\cdot}008$ Mg m^{-3}, Mo $K\alpha$, $\lambda = 0{\cdot}71069$ Å, $\mu = 0{\cdot}046$ mm^{-1}, $F(000) = 584$, room temperature, $R = 0{\cdot}0524$, based on 1581 unique observed reflections. The structural effects of deprotonation are shortenings of the B(9)—B(10) and B(5)—B(10) connectivities, and these changes are rationalized by the results of a molecular orbital calculation.

Introduction. We are currently investigating (Wynd, McLennan, Reed & Welch, 1987; Wynd, Parish & Welch, 1989) how the introduction of $\{AuPR_3\}$ (R = alkyl) fragments into $\{B_{10}\}$ cages affects the electronic and consequent geometric structure of the cage. The analysis of the metallaborane cage architecture relies mainly on comparison with the structures of analogous boranes. Whilst the literature contains a communication (Sneddon, Huffman, Schaeffer & Streib, 1972) reporting the structure of the $[B_{10}H_{13}]^-$ anion, as its $[NHEt_3]^+$ salt, no fractional coordinates were published or deposited, nor are any available from the authors. The structure of the isoelectronic, isovertex species, $B_{10}H_{14}$, is known to a high degree of accuracy (Brill, Dietrich & Dierks, 1971). However since the effects of deprotonation can substantially modify the internuclear distances in such species (Mitchell & Welch, 1987), we have determined the structure of $[PhCh_2NMe_3]^+$ $[B_{10}H_{13}]^-$ (1). This enables useful comparisons to be drawn against the structures of other boranes and metallaborane derivatives (this paper; Wynd, Parish & Welch, 1989). (1) was synthesized using a method based on literature preparations (Hawthorne, Pitochelli, Strahm & Miller, 1960).

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Table 1. *Fractional coordinates of refined atoms in* $[PhCH_2NMe_3]^+ [B_{10}H_{13}]^-$

	x	y	z	U_{iso}(Å^2)
N	0·19902 (25)	0·59661 (12)	0·26319 (20)	0·0534 (15)
C(1)	0·15660	0·55862	0·03550	0·0474 (16)
C(2)	0·24947 (22)	0·50316 (9)	0·00303 (18)	0·0579 (19)
C(3)	0·30173	0·50239	−0·11397	0·0754 (23)
C(4)	0·26113	0·55709	−0·19851	0·080 (3)
C(5)	0·16826	0·61256	−0·16605	0·0760 (24)
C(6)	0·11600	0·61332	−0·04904	0·0632 (20)
C(7)	0·0989 (3)	0·55784 (15)	0·1608 (3)	0·0552 (18)
C(8)	0·3577 (3)	0·57068 (17)	0·2716 (3)	0·0623 (20)
C(9)	0·1382 (4)	0·58131 (21)	0·3841 (3)	0·089 (3)
C(10)	0·1980 (4)	0·67514 (16)	0·2394 (4)	0·0796 (24)
B(1)	0·6211 (4)	0·83986 (19)	0·2036 (3)	0·0584 (22)
B(2)	0·6020 (4)	0·74532 (18)	0·1913 (3)	0·0551 (21)
B(3)	0·7832 (4)	0·78474 (18)	0·1965 (3)	0·0535 (20)
B(4)	0·7783 (4)	0·87201 (18)	0·1372 (3)	0·0578 (22)
B(5)	0·4820 (4)	0·80292 (20)	0·0907 (3)	0·0608 (23)
B(6)	0·5408 (4)	0·71686 (21)	0·0446 (3)	0·0620 (23)
B(7)	0·7370 (4)	0·71450 (19)	0·0951 (3)	0·0582 (22)
B(8)	0·8491 (4)	0·80227 (19)	0·0521 (3)	0·0582 (22)
B(9)	0·7315 (5)	0·87047 (21)	−0·0227 (4)	0·0681 (25)
B(10)	0·5905 (4)	0·88447 (19)	0·0630 (3)	0·0588 (22)
H(1)	0·581 (3)	0·8610 (15)	0·288 (3)	0·0719 (24)
H(2)	0·564 (3)	0·7171 (15)	0·276 (3)	
H(3)	0·865 (3)	0·7689 (15)	0·279 (3)	
H(4)	0·846 (3)	0·9143 (16)	0·185 (3)	
H(5)	0·359 (4)	0·8095 (14)	0·0951 (24)	
H(6)	0·465 (3)	0·6713 (15)	0·016 (3)	
H(7)	0·799 (3)	0·6646 (16)	0·102 (3)	
H(8)	0·960 (4)	0·7909 (15)	0·0334 (25)	
H(9)	0·762 (3)	0·9069 (15)	−0·101 (3)	
H(10)	0·522 (3)	0·9359 (16)	0·0585 (24)	
H(5,6)	0·499 (3)	0·7691 (16)	−0·016 (3)	
H(6,7)	0·655 (3)	0·7123 (15)	−0·017 (3)	
H(8,9)	0·766 (3)	0·8039 (15)	−0·048 (3)	

Experimental. Yellow blocks, 0·04 × 0·04 × 0·03 cm, were grown by the diffusion of hexane into a CH_2Cl_2 solution; CAD-4 diffractometer, 25 centred reflections ($13 < \theta < 13·5°$), graphite-monochromated Mo $K\alpha$; for data collection: $\theta_{max} = 22°$, ω–2θ scans in 96 steps, ω-scan width $(0·8 + 0·34\tan\theta)°$. The data(h 0 to 9; k 0 to 19; l −11 to 11) were measured over a period of 46 hours with no detectable crystal movement or decay. 2359 reflections were measured (scan speeds 1·268–3·296° min^{-1}), yielding 2194 unique ($R_{merge} = 0·0103$) of which 1581 with $F > 2\sigma(F)$ were retained. Space group $P2_1/c$ from systematic absences ($h0l$: $l = 2n$; $0k0$: $k = 2n$).

Atoms located by automatic direct methods (*SHELX*86; Sheldrick, 1986) followed by difference-Fourier syntheses with full-matrix, least-squares refinement on F (*SHELX*76; Sheldrick, 1976), $w = [\sigma^2(F) + 0·002200(F)^2]^{-1}$, anisotropic thermal parameters for all non-H atoms. Phenyl ring constrained to be a regular planar hexagon (C—C = 1·395 Å). All organic H atoms set in calculated positions (C—H = 1·08 Å) and refined with a group thermal parameter [0·103 (3) Å^2 at convergence]. Cage H atoms freely refined with a group thermal parameter [0·0719 (24) Å^2 at convergence]. $R = 0·0524$, $wR = 0·0730$, $S = 1·079$, data:variable = 7:1, $(\Delta\rho)_{max} = 0·132$ e Å^{-3}, $(\Delta\rho)_{min} = -0·161$ e Å^{-3}. Max. Δ/σ in final cycle was 0·063.

Scattering factors for C, H, B and N inlaid in *SHELX*76. Other computer programs used were *CADABS* (Gould & Smith, 1986), *CALC* (Gould & Taylor, 1988), and *EASYORTEP* (Mallinson, 1984; see also Johnson, 1976).

Discussion. Table 1 lists the fractional coordinates of the refined atoms,* and Fig. 1 is a perspective view of one ion-pair, showing the atomic numbering scheme. There are no significant intermolecular contacts, and the bond lengths and angles in the cation are unexceptional.

Table 2 lists the boron—boron internuclear distances and interatomic angles. The B—H and B—(μ-H) distances lie in the range expected. Deprotonation of $B_{10}H_{14}$ removes one μ-H atom [nominally H(9,10)] and there is no structural rearrangement accompanying this loss. Moreover, in the present crystallographic analysis, there is no evidence for disorder of the deprotonated connectivity. As previously reported (Sneddon, Huffman, Schaeffer & Streib, 1972) there are, however, several significant bond-length variations as a consequence of deprotonation.

The B(9)—B(10) connectivity is shortened from 1·788 (4) Å (average) in $B_{10}H_{14}$ (Brill, Dietrich & Dierks, 1971) to 1·657 (5) Å in (1); the B(5)—B(10) connectivity is similarly shortened, 1·987 (3) to 1·848 (5) Å. Using Edinburgh software (Gould & Taylor, 1988) we can compare two crystallographically determined fragments, and calculate their r.m.s. misfit. For $B_{10}H_{14}$/(1) this is 0·077 Å, with the largest difference involving the atomic positions of B(5) (0·147 Å) and B(9) (0·158 Å). This is presumably due to the two bond-length changes discussed above.

The reason for the shorter B(5)—B(6) and B(5)—B(10) distances in (1) compared to $B_{10}H_{14}$ may be

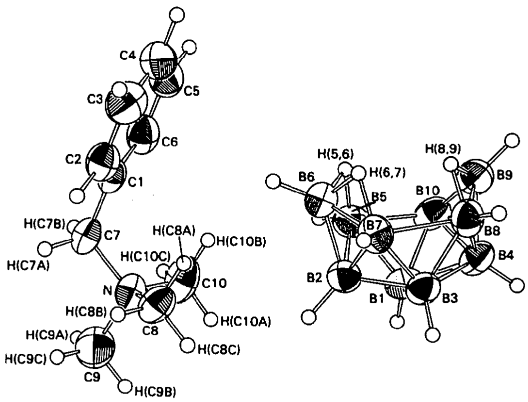

Fig. 1. A perspective view of one ion-pair. All atoms are drawn at the 50% probability level, except for H atoms, which have been given artificial radii of 0·1 Å, for clarity.

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* Lists of structure factors, anisotropic thermal parameters, H-atom parameters and additional bond lengths and angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51529 (14 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 2. *Bond lengths* (Å) *and angles* (°) *in the* $[B_{10}H_{13}]^-$ *anion*

B(1)—B(2)	1·783 (5)	B(3)—B(8)	1·758 (5)
B(1)—B(3)	1·780 (5)	B(4)—B(8)	1·754 (5)
B(1)—B(4)	1·745 (5)	B(4)—B(9)	1·736 (5)
B(1)—B(5)	1·780 (5)	B(4)—B(10)	1·786 (5)
B(1)—B(10)	1·731 (5)	B(5)—B(6)	1·783 (5)
B(2)—B(3)	1·765 (5)	B(5)—B(10)	1·848 (5)
B(2)—B(5)	1·798 (5)	B(6)—B(7)	1·768 (5)
B(2)—B(6)	1·707 (5)	B(7)—B(8)	2·003 (5)
B(2)—B(7)	1·767 (5)	B(8)—B(9)	1·788 (5)
B(3)—B(4)	1·756 (5)	B(9)—B(10)	1·657 (5)
B(3)—B(7)	1·734 (5)		

B(2)—B(1)—B(3)	59·39 (19)	B(8)—B(4)—B(9)	61·63 (21)
B(2)—B(1)—B(5)	60·61 (20)	B(9)—B(4)—B(10)	56·13 (20)
B(3)—B(1)—B(4)	59·74 (19)	B(1)—B(5)—B(2)	59·78 (20)
B(4)—B(1)—B(10)	61·81 (20)	B(1)—B(5)—B(10)	56·98 (19)
B(5)—B(1)—B(10)	63·48 (21)	B(2)—B(5)—B(6)	56·93 (20)
B(1)—B(2)—B(3)	60·20 (19)	B(2)—B(6)—B(5)	61·99 (21)
B(1)—B(2)—B(5)	59·61 (20)	B(2)—B(6)—B(7)	61·10 (21)
B(3)—B(2)—B(7)	58·80 (19)	B(2)—B(7)—B(3)	60·54 (20)
B(5)—B(2)—B(6)	61·08 (21)	B(2)—B(7)—B(6)	57·74 (20)
B(6)—B(2)—B(7)	61·16 (21)	B(3)—B(7)—B(8)	55·57 (19)
B(1)—B(3)—B(2)	60·41 (19)	B(3)—B(8)—B(4)	59·98 (20)
B(1)—B(3)—B(4)	59·16 (19)	B(3)—B(8)—B(7)	54·43 (18)
B(2)—B(3)—B(7)	60·66 (20)	B(4)—B(8)—B(9)	58·68 (21)
B(4)—B(3)—B(8)	59·89 (20)	B(4)—B(9)—B(8)	59·69 (21)
B(7)—B(3)—B(8)	70·01 (21)	B(4)—B(9)—B(10)	63·46 (22)
B(1)—B(4)—B(3)	61·10 (20)	B(1)—B(10)—B(4)	59·48 (20)
B(1)—B(4)—B(10)	58·71 (20)	B(1)—B(10)—B(5)	59·55 (20)
B(3)—B(4)—B(8)	60·13 (20)	B(4)—B(10)—B(9)	60·40 (21)

readily understood *via* an extended Huckel molecular orbital calculation (Howell, Rossi, Wallace, Haraki & Hoffmann, 1977) on a model of $[B_{10}H_{13}]^-$ derived from the structure of $B_{10}H_{14}$ by μ-H(9,10) deprotonation. The highest occupied molecular orbital of this model is localized on, and bonding between, B(5) and B(6). A minor contribution on B(10) is in phase with that on B(5). Protonation causes partial deoccupation of this orbital (Mitchell & Welch, 1987) thereby lengthening both B(5)—B(6) and B(5)—B(10).

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Structure of *cis*-Bis(pentafluorophenyl)vinylene Bis(pentafluorobenzoate)

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Abstract. $C_{28}F_{20}O_4$, $M_r = 780{\cdot}31$, triclinic, $P\bar{1}$, $a = 8{\cdot}200\,(1)$, $b = 9{\cdot}899\,(2)$, $c = 18{\cdot}160\,(3)$ Å, $\alpha = 75{\cdot}75\,(1)$, $\beta = 81{\cdot}40\,(1)$, $\gamma = 73{\cdot}47\,(1)°$, $V = 1363{\cdot}5\,(4)$ Å^3, $Z = 2$, $D_x = 1{\cdot}90$ g cm^{-3}, $\lambda(\text{Cu } K\alpha) = 1{\cdot}5418$ Å, $\mu = 19{\cdot}9$ cm^{-1}, $F(000) = 760$, $T = 300$ K, final $R = 0{\cdot}0485$, $wR = 0{\cdot}0621$ for 3699 independent reflections. The compound contains no H atoms. The benzoate phenyl rings of the title compound are rotated 36·2 (14) and 36·4 (5)° relative to the carboxyl group whereas in the non-perfluorinated parent compound these rings are coplanar with the carboxyl groups [torsion angles 6·7 (8) and 0·3 (2)°]. The average torsion angle about the double bond is also larger in the title compound [11·4 (23) *vs* 6·6 (30)°].

Introduction. In our investigations of the electrochemistry of fluorinated compounds, we have discovered that the reduction of decafluorobenzil in the presence of pentafluorobenzoyl chloride produces a mixture of *cis*- and *trans*-bis(pentafluorophenyl)vinylene bis(pentafluorobenzoate). Similar behavior is observed in the parent diphenylvinylene dibenzoate

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