# Crystal and Molecular Structure of $[Me_4N]^+[PhCHB_{10}H_{10}CPh]^-$: Relation to the $C_2B_{10}H_{13}^-$ Ion

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*Summary* A single-crystal *X*-ray diffraction study of $[Me_4N]^+[PhCHB_{10}H_{10}CPh]^-$ shows a unique bridging C atom of the PhCH group on the face of an icosahedral $(B_{10}C)$ fragment; there are no bridge hydrogens.

REDUCTION of *ortho*-diphenylcarborane under argon in THF,[1] followed by precipitation[2] with $Me_4N^+$ in $H_2O$ free of dissolved $O_2$, yields the compound $[Me_4N]^+[PhCHB_{10}H_{10}CPh]^-$ which has crystal data: $M$ 371·58, orthorhombic, $a = 15{\cdot}984(10)$, $b = 11{\cdot}158(5)$, $c = 12{\cdot}941(3)$ Å, $U = 2307{\cdot}8$ Å$^3$, $D_c = 1{\cdot}069$, $D_m = 1{\cdot}060$ g cm$^{-3}$, space group $P2_12_12_1$ ($D_2^4$, No. 19). Intensities ($2\theta < 115°$, Cu-$K_\alpha$), collected on an automated Picker diffractometer, yielded the structure upon phasing with a method[3] based upon the tangent formula. Refinement by Fourier and least-squares method has converged to $R = 0{\cdot}093$. All hydrogen atoms of the

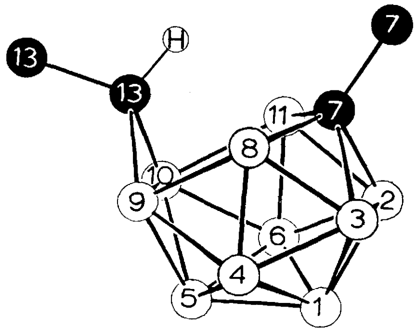

FIGURE. *Anion structure as determined by X-ray structural analysis. Ten terminal B–H hydrogens are omitted, and only the bonding carbon of each phenyl ring is shown.*

completely ordered anion were found in difference electron-density maps. Except for the orientations of phenyl groups (not shown in the Figure), the anion has $C_s$ symmetry, and has C(7) in the open face of an icosahedral fragment while C(13) bridges B(9) and B(10). Also C(13) has a terminal hydrogen (Figure). Non-hydrogen bond distances have standard deviations of 0·01, and include C(13)–B(9) = 1·66 Å.

The 32·1 MHz $^{11}B$ n.m.r. spectrum resembles that of a similarly prepared $B_{10}C_2H_{13}^-$ anion; while our crystal structure supports the opened icosahedral arrangement, our C atom positions of the $B_{10}C_2$ fragment are not the same as those deduced earlier[4] from the n.m.r. results. We note also that polyhedral opening by a proton yields a different structure from that found upon opening by a liganded transition metal.[5]

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[1] M. M. Fein, J. Bobinski, N. Mayes, N. Schwartz, and M. S. Cohen, *Inorg. Chem.*, 1963, **2**, 1111; L. I. Zakharkin and V. N. Kalinin, *Izvest. Akad. Nauk S.S.S.R., Ser. khim.*, 1967, **10**, 2310.

[2] L. I. Zakharkin, V. N. Kalinin, B. A. Kvasov, and A. P. Synakin, *Zhur. obshchei Khim.*, 1971, **41**, 1726.

[3] P. Main, M. M. Wolfson, and G. Germain, 1971, MULTAN, A Computer Program for the automatic Solution of Crystal Structures.

[4] G. B. Dunks, R. J. Wiersema, and M. F. Hawthorne, *J.C.S. Chem. Comm.*, 1972, 899.

[5] M. R. Churchill and B. G. DeBoer, *J.C.S. Chem. Comm.*, 1972, 1326.