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Triethylammonium tetraphenylborate dichloromethane solvate

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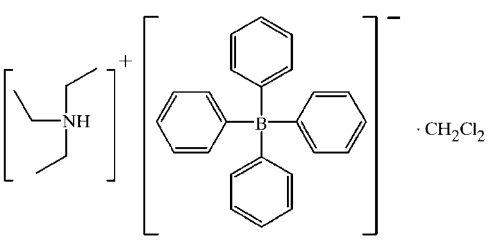
The crystal structure of the title compound, $C_6H_{16}N^+\cdot C_{24}H_{20}B^-\cdot CH_2Cl_2$, displays an N—H···π interaction between the triethylammonium cation and a phenyl ring of the tetraphenylborate anion. The distance between the ammonium H atom and the edge of this phenyl ring is 2.40 (3) Å. The ammonium group and the aryl moiety are nearly perpendicular, forming an intramolecular dihedral angle of 90.4°. A C—H···π interaction between the disordered dichloromethane solvate molecule and a phenyl ring of the tetraphenylborate anion is also present.

Comment

Structures with strong hydrogen-bonding donors lacking traditional acceptor groups can exhibit intermolecular interactions with π systems of aromatic rings as hydrogen-bond acceptors (Hanton *et al.*, 1992). Two unsolvated structures of the salt triethylammonium tetraphenylborate have been determined previously (King & Bryant, 1991; Bakshi *et al.*, 1994), both of which contain N—H···π interactions with phenyl rings. In each case, the closest N—H···π system contact is not with the center of the aromatic ring but is rather a clear interaction with one C atom of a phenyl ring. Malone *et al.* (1997) have investigated *X*—H···π interactions with phenyl rings in a variety of structures and established six possible categories of these interactions on the basis of geometry and distance considerations. We report here the structure of the title compound, (I) (Fig. 1), a dichloromethane solvate of triethylammonium tetraphenylborate.



(I)

Molecules of (I) show intermolecular N—H···π interactions between ammonium atom H1*A* and the edge of one phenyl ring of the tetraphenylborate anion (Table 1). Fig. 2 shows a view of this interaction. Atom H1*A* is directed at the center of the bond between atoms C10 and C11, the distance between atom H1*A* and the centroid (π_1) calculated from the atomic positions of atoms C10 and C11 being 2.40 (3) Å. The angle formed by atom N1, atom H1*A* and π_1 is 172 (3)°, and the angle formed by atom H1*A*, π_1 and the centroid of the entire phenyl ring (calculated from the positions of atoms C7–C12) is 90.4°. The approach geometry of the NH group to the π system of the phenyl ring is characterized as a type II interaction in the scheme of Malone *et al.* (1997).

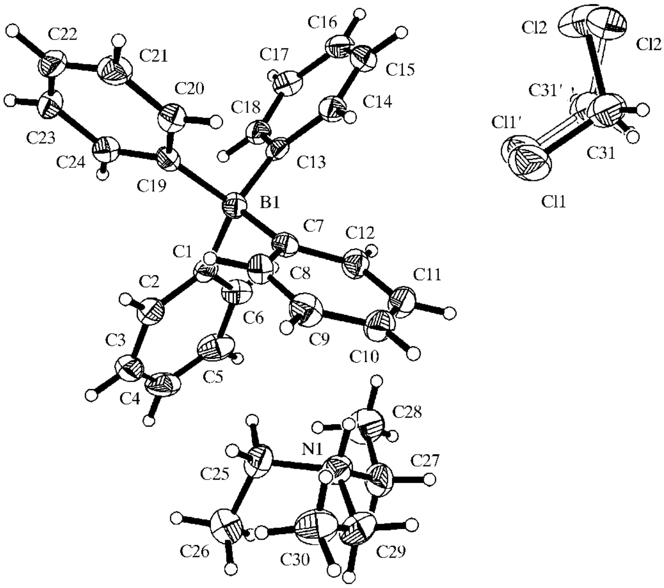


Figure 1
A view of (I), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.

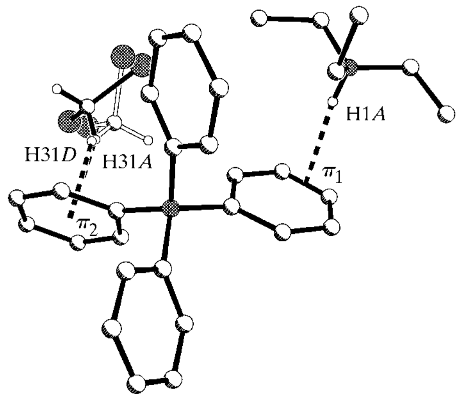


Figure 2
A view of the structure of (I), showing the intermolecular N—H···π_1 and C—H···π_2 interactions. H atoms bonded to C atoms have been omitted for clarity, except for those in the dichloromethane molecule. The atoms of the dichloromethane molecule shown are at the symmetry position $(1 - x, 1 - y, 1 - z)$ relative to the cation and anion moieties shown.

The dichloromethane solvent molecule is disordered and was refined anisotropically over two similar positions. The site occupancy of atoms C31, Cl1 and Cl2 is 0.80 (1), with sites C31′, Cl1′ and Cl2′ being occupied for the remainder. Atoms H31*A* and H31*D* of the dichloromethane molecule also show intermolecular C—H$\cdots\pi$ interactions with the centroid (π_2) of the C13–C18 phenyl ring of the tetraphenylborate anion. The angles of the dichloromethane C—H$\cdots\pi$ interactions deviate significantly from 180° (Table 1). Atoms H31*A* and H31*D* lie on the vector normal to the phenyl ring at π_2.

Experimental

The title compound was isolated from a reaction mixture containing NEt_3, $Na[BPh_4]$, $[Ru(p\text{-cymene})Cl_2]_2$ [*p*-cymene is 4-*i*-$CH(CH_3)_2$-1-CH_3-C_6H_4] and 2-phenylpyridine in dichloromethane. Crystals were grown by slow diffusion of hexane into a dichloromethane solution of the crude reaction mixture.

Crystal data

$C_6H_{16}N^+\cdot C_{24}H_{20}B^-\cdot CH_2Cl_2$
$M_r = 506.33$
Monoclinic, $P2_1/n$
$a = 9.9331\ (17)$ Å
$b = 18.258\ (3)$ Å
$c = 15.958\ (3)$ Å
$\beta = 104.168\ (3)°$
$V = 2806.1\ (9)$ Å^3
$Z = 4$
$D_x = 1.199$ Mg m^{-3}

Mo $K\alpha$ radiation
Cell parameters from 998 reflections
$\theta = 2.6$–$25.0°$
$\mu = 0.25$ mm^{-1}
$T = 173\ (2)$ K
Block, pale yellow
0.21 × 0.08 × 0.05 mm

Data collection

Siemens SMART PLATFORM CCD diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Blessing, 1995)
$T_{\min} = 0.936$, $T_{\max} = 0.99$
13 804 measured reflections

4946 independent reflections
3943 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.032$
$\theta_{\max} = 25.0°$
$h = -11 \rightarrow 11$
$k = -15 \rightarrow 21$
$l = -11 \rightarrow 19$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.069$
$wR(F^2) = 0.140$
$S = 1.03$
4946 reflections
335 parameters
H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0387P)^2 + 4P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 0.47$ e Å^{-3}
$\Delta\rho_{\min} = -0.23$ e Å^{-3}

Table 1
Hydrogen-bonding geometry (Å, °).

π_1 is the centroid calculated from the positions of atoms C10 and C11, and π_2 is the centroid of the C13–C18 phenyl ring.

D—H$\cdots$*A*	*D*—H	H$\cdots$*A*	*D*$\cdots$*A*	*D*—H$\cdots$*A*
N1—H1*A*$\cdots\pi_1$	0.94 (3)	2.40 (3)	3.338 (3)	172 (3)
C31—H31*A*$\cdots\pi_2^{\rm i}$	0.99	2.50	3.403 (5)	152
C31′—H31*D*$\cdots\pi_2^{\rm i}$	0.99	2.43	3.347 (19)	153

Symmetry code: (i) $1 - x, 1 - y, 1 - z$.

The coordinates of atom H1*A* were refined, but the displacement parameter was constrained [$U_{\rm iso}({\rm H1}A) = 1.2U_{\rm eq}({\rm N1})$]. All other H atoms were placed in idealized positions and constrained to ride on their parent atoms, with C—H distances in the range 0.95–0.99 Å and $U_{\rm iso}$(H) values of $1.2U_{\rm eq}$(C). Attempts to refine the coordinates of the H atoms on the disordered dichloromethane molecule were unsuccessful.

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1998); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1990); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1998); software used to prepare material for publication: *SHELXTL*.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: SQ1147). Services for accessing these data are described at the back of the journal.

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