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Lithium tetrakis(diphenylphosphino)borate diethyl ether solvate

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In the title compound, $Li^+\cdot C_{48}H_{40}BP_4^-\cdot C_4H_{10}O$, the Li^+ cation is coordinated by two P atoms, two aromatic C atoms and the ether O atom.

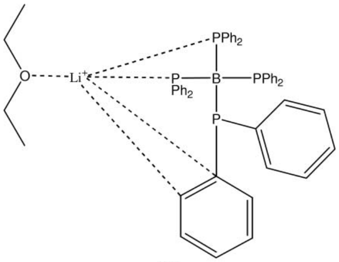
Key indicators

Single-crystal X-ray study
T = 173 K
Mean σ(C–C) = 0.003 Å
R factor = 0.038
wR factor = 0.097
Data-to-parameter ratio = 16.5

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Comment

In contrast to the well established tetraalkyl- and tetraarylborates, there are only two examples of tetraphosphinoborate anions, namely the parent compound $B(PH_2)_4^-$ (Baudler & Block, 1988, 1989) and the ethyl derivative $B(PEt_2)_4^-$ (Fritz & Pfannerer, 1970; Fritz & Sattler, 1975). To our knowledge, none of them has been structurally characterized. We report here the synthesis and structure of the title compound, (I) (Fig. 1), which is a lithium salt and diethylether solvate of the phenyl derivative, $B(PPh_2)_4^-$. The compound is stable at room temperature but the solution decomposes immediately upon contact with stainless steel canulas or catalytic amounts of zirconium oxide with formation of a colourless precipitate. The decomposition products were not identified.



(I)

The bond lengths and angles in (I) can be regarded as normal (Cambridge Structural Database; Version 5.27, November 2005 updated August 2006; Mogul Version 1.1; Allen, 2002). Compound (I) contains tetrakis(diphenylphosphino)borate anions, Li cations and diethyl ether solvent molecules. The Li cation is coordinated by two P atoms, two aromatic C atoms and the ether O atom (Table 1). The B—P distances of the Li-coordinating P atoms are longer than the other two B—P distances.

Experimental

Reactions and manipulations were carried out under an atmosphere of dry nitrogen using standard Schlenk techniques. Solvents were freshly distilled under argon from sodium/benzophenone (diethyl ether, C_6D_6) or sodium–lead alloy (pentane) prior to use. A solution of $BF_3\cdot OEt_2$ (79 mg, 0.56 mmol) in diethyl ether (5 ml) was added to a stirred solution of Ph_2PLi (0.433 g, 2.25 mmol) in diethyl ether (15 ml) at 195 K. An orange suspension formed. After the mixture had warmed to ambient temperature overnight, a colourless precipitate was filtered off. All volatiles were removed from the pale-yellow filtrate *in vacuo*. The yellow residue was washed with pentane (2 × 5 ml) and redissolved in a minimum amount of diethyl ether. X-ray quality crystals of (I) separated upon cooling to 278 K. Yield 176 mg (38%).

Crystal data

$Li^+\cdot C_{48}H_{40}BP_4^-\cdot C_4H_{10}O$
$M_r = 832.55$
Monoclinic, $P2_1$
$a = 11.7386$ (7) Å
$b = 13.2135$ (11) Å
$c = 14.4602$ (9) Å
$\beta = 91.216$ (5)°
$V = 2242.4$ (3) Å^3
$Z = 2$
$D_x = 1.233$ Mg m^{-3}
Mo $K\alpha$ radiation
$\mu = 0.21$ mm^{-1}
$T = 173$ (2) K
Block, yellow
0.50 × 0.49 × 0.47 mm

Data collection

Stoe IPDS-II two-circle diffractometer
ω scans
Absorption correction: multi-scan (*MULABS*; Spek, 2003; Blessing, 1995)
$T_{min} = 0.904$, $T_{max} = 0.909$
16919 measured reflections
8770 independent reflections
8251 reflections with $I > 2\sigma(I)$
$R_{int} = 0.038$
$\theta_{max} = 27.7°$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.038$
$wR(F^2) = 0.097$
$S = 1.05$
8770 reflections
533 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0682P)^2 + 0.0442P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.28$ e Å^{-3}
$\Delta\rho_{min} = -0.23$ e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.0061 (12)
Absolute structure: Flack (1983), 3383 Friedel pairs
Flack parameter: −0.04 (5)

Table 1
Selected bond lengths (Å).

B1—P2	2.013 (2)	Li1—P4	2.532 (4)
B1—P1	2.015 (2)	Li1—C31	2.543 (5)
B1—P3	2.032 (2)	Li1—C32	2.560 (5)
B1—P4	2.053 (2)	Li1—O1	1.881 (5)
Li1—P3	2.599 (5)		

The H atoms were positioned geometrically (C—H = 0.95–0.99 Å) and refined as riding with $U_{iso}(H) = 1.2U_{eq}(C)$ or $1.5U_{eq}$(methyl C).

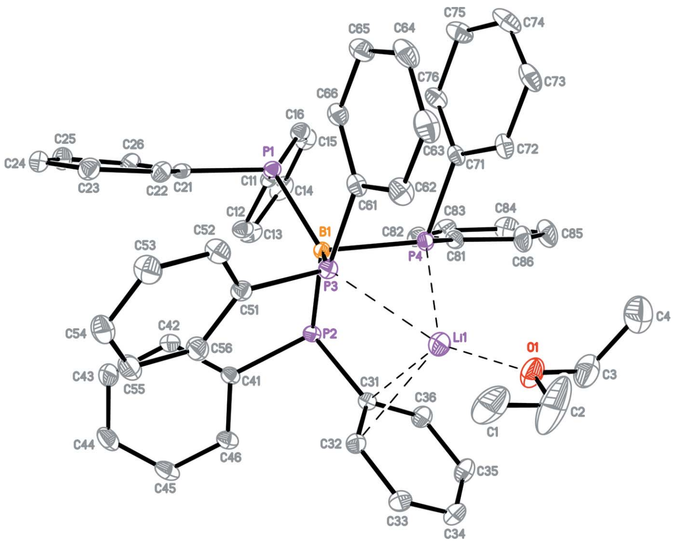


Figure 1
The asymmetric unit of (I) showing 50% displacement ellipsoids; H atoms have been omitted for clarity. The bonds to Li are shown as dashed lines.

Data collection: *X-AREA* (Stoe & Cie, 2001); cell refinement: *X-AREA*; data reduction: *X-AREA*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *XP* in *SHELXTL-Plus* (Sheldrick, 1991); software used to prepare material for publication: *SHELXL97*.

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