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Lithium–tetrakis(pentafluorophenyl)borate–benzene (1/1/2)

Michael Bolte,[a] Ilya Ruderfer[b] and Thomas Müller[a]*

[a]Institut für Anorganische Chemie, J. W. Goethe-Universität Frankfurt, Marie-Curie-Strasse 11, 60439 Frankfurt/Main, Germany, and [b]Department of Chemistry and The Lise Meitner Minerva Center for Computational Quantum Chemistry, Technion-Israel Institute of Technology, Haifa 32000, Israel

Correspondence e-mail:
bolte@chemie.uni-frankfurt.de

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The title compound, $Li^+\cdot C_{24}BF_{20}^-\cdot 2C_6H_6$, crystallizes as centrosymmetric dimers, with two η^6-coordinated and two uncoordinated molecules of benzene per dimer. There are two crystallographically independent dimers.

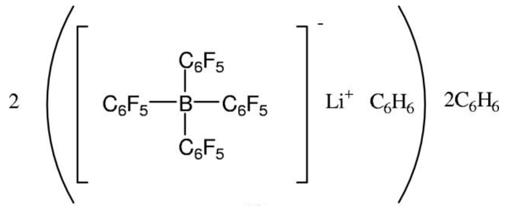
Key indicators

Single-crystal X-ray study
T = 173 K
Mean σ(C–C) = 0.009 Å
Disorder in solvent or counterion
R factor = 0.094
wR factor = 0.246
Data-to-parameter ratio = 11.9

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

Comment

In the course of our studies on weakly coordinating anions, colourless crystals of the title compound, (I), were obtained by recrystallization of the diethyl ether solvate of lithium tetrakispentafluoroborate (Chien *et al.*, 1991; Massey & Park, 1964) from benzene.



$$2\left(\left[B(C_6F_5)_4\right]^- Li^+\ C_6H_6\right) 2C_6H_6$$

(I)

A perspective view of (I) is shown in Fig. 1. The structure is composed of $[C_{24}BF_{20}]^-$ anions, Li^+ cations and benzene molecules. Each Li^+ cation links two $[C_{24}BF_{20}]^-$ anions *via* short Li···F contacts to form a centrosymmetric dimer. In addition, the Li^+ cation displays a short contact to an F atom of a neighbouring $[C_{24}BF_{20}]^-$ anion. Furthermore, each Li^+ cation is η^6-coordinated by a benzene ring. These distances are compiled in Table 1. The remaining holes in the structure are filled by disordered benzene molecules. It is interesting to note that the structure shows a pseudo-A centring. The reflections with $k + l$ uneven show a lower intensity than the others, but they are definitely observed.

Experimental

Crystals were obtained by recrystallization of the diethyl ether solvate of lithium tetrakispentafluoroborate from benzene in a glass ampoule after 1 d at room temperature. ^{19}F NMR [235.33 MHz, 303 K, CD_3CN/C_6D_6; $\delta(C_6F_6) = -162.9$]: δ −166.4 (b, 8F), −162.2 (t, 4F, $^3J_{FF}$ = 20.8 Hz), −131.7 (b, 8F).

Crystal data

$Li^+\cdot C_{24}BF_{20}^-\cdot 2C_6H_6$
M_r = 842.21
Triclinic, $P\overline{1}$
a = 10.1025 (6) Å
b = 17.3954 (11) Å
c = 20.3856 (13) Å
α = 102.267 (5)°
β = 103.752 (5)°
γ = 93.744 (5)°
V = 3375.0 (4) Å^3

Z = 4
D_x = 1.658 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 71332 reflections
θ = 3.6–25.5°
μ = 0.17 mm^{-1}
T = 173 (2) K
Block, colourless
0.39 × 0.37 × 0.34 mm

doi:10.1107/S1600536805022336

Data collection

Stoe IPDS-II two-circle diffractometer
ω scans
Absorption correction: none
71332 measured reflections
12200 independent reflections

8647 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.100$
$\theta_{\max} = 25.4°$
$h = -12 \rightarrow 12$
$k = -20 \rightarrow 20$
$l = -24 \rightarrow 24$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.094$
$wR(F^2) = 0.246$
$S = 1.08$
12200 reflections
1027 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0711P)^2 + 17.0328P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 1.10$ e Å^{-3}
$\Delta\rho_{\min} = -0.71$ e Å^{-3}

Table 1
Selected bond lengths (Å).

Li1—F33	2.015 (12)	Li2—F63	2.046 (9)
Li1—F22^{i}	2.071 (13)	Li2—F86^{iii}	2.047 (9)
Li1—F23^{i}	2.232 (15)	Li2—F74^{iv}	2.213 (10)
Li1—F44^{ii}	2.238 (14)	Li2—F85^{iii}	2.231 (8)
Li1—C95	2.533 (16)	Li2—C106	2.501 (10)
Li1—C94	2.559 (15)	Li2—C104	2.568 (10)
Li1—C96	2.602 (17)	Li2—C105	2.591 (9)
Li1—C93	2.674 (15)	Li2—C102	2.674 (11)
Li1—C91	2.700 (16)	Li2—C101	2.716 (10)
Li1—C92	2.729 (16)	Li2—C103	2.751 (11)

Symmetry codes: (i) $-x, -y+2, -z+1$; (ii) $x-1, y, z$; (iii) $-x+2, -y+1, -z$; (iv) $x+1, y, z$.

H atoms were refined with fixed individual displacement parameters [$U_{\rm iso}({\rm H}) = 1.2U_{\rm eq}({\rm C})$] using a riding model, with C—H = 0.95 Å. The C atoms of the solvent benzene molecules which are heavily disordered were refined isotropically. For the refinement of these atoms, distance restraints of 1.40 (1) and 2.44 (1) Å for 1–2 and 1–3 atoms, respectively, were used. The site-occupation factors for the disordered benzene molecules refined to 0.567 (9)/0.433 (9) and 0.598 (10)/0.402 (10). In addition, these rings were restrained to be planar. The highest peak in the final difference electron-density map is located 0.24 Å from atom H12′.

Data collection: *X-AREA* (Stoe & Cie, 2001); cell refinement: *X-AREA*; data reduction: *X-AREA*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990); 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* and *PLATON* (Spek, 2003).

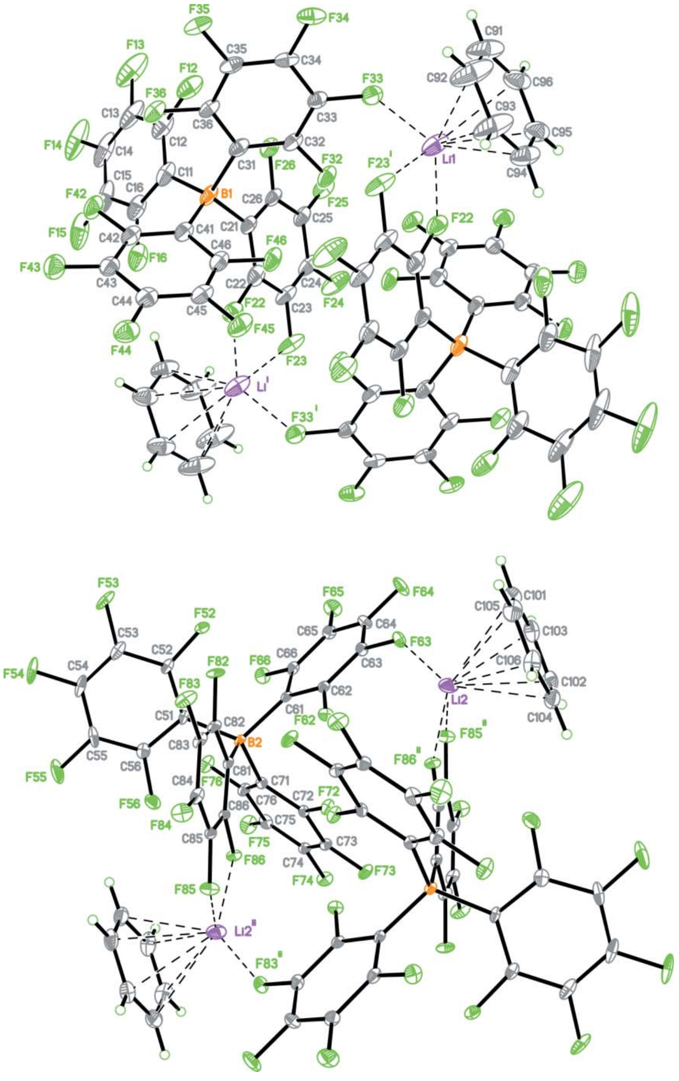


Figure 1
Perspective view of the two independent dimers of the title compound, showing the atom numbering. Displacement ellipsoids are drawn at the 50% probability level and the disordered benzene molecules have been omitted for clarity. [Symmetry codes: (i) $-x, -y+2, -z+1$; (ii) $-x+2, -y+1, -z$.]

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