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Polymeric (diphenylphosphinato)tetrahydrofuranlithium

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.006 Å
R factor = 0.050
wR factor = 0.130
Data-to-parameter ratio = 13.9

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

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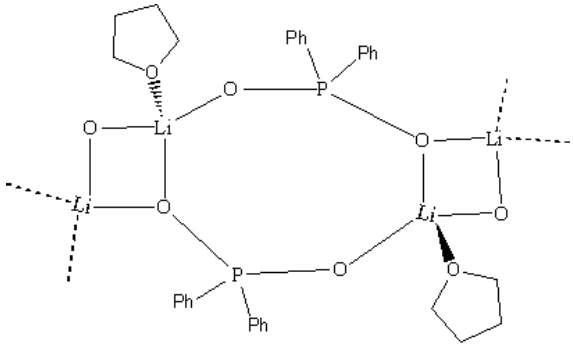
In the title compound, $[Li(C_{12}H_{10}O_2P)(C_4H_8O)]_n$, the O atoms of adjacent and bridging diphenylphosphinate ligands and that from a tetrahydrofuran (thf) molecule are arranged in a tetrahedral manner around the Li atoms, resulting in a one-dimensional array (parallel to the a axis) of alternate eight-membered and rectangular planar four-membered rings [the two Li—O distances are 1.962 (6) and 1.991 (6) Å, and the Li—O—Li and O—Li—O angles are 88.3 (2) and 91.7 (2)°, respectively]. The Li—O distances for the O atoms of the phosphinate ligand are 1.992 (6) (for the μ-O atom) and 1.897 (6) Å, and the distance from Li to the O atom of the thf ligand is 2.028 (6) Å.

Comment

There has been only one previously reported structure of a lithium diorganophosphinate complex, namely $Li[Mes_2PO_2]$ (Beswick *et al.*, 1997). This complex consists of discrete dimeric molecules with two bridging $Mes_2PO_2^-$ (dimesitylphosphinate) ligands attached to two Li^+ cations, forming eight-membered rings. Two thf molecules, attached to each Li^+ cation *via* lone pairs on the O atoms, complete the coordination geometry for these distinct dimers.



(I)

In contrast, the title compound, (I), has a linear polymeric arrangement with two types of rings, alternating with each other. As seen in Fig. 1, there is an eight-membered ring, previously observed with bridging dimesitylphosphinate ligands, and also rectangular planar arrays consisting of two Li and two O atoms from adjacent phosphinate ligands (as seen in the packing diagram, Fig. 2). The rectangular part is a result of the eight-membered ring dimers binding with each other. An O atom from a thf molecule completes the tetrahedral geometry around each Li^+ atom. This arrangement no doubt results as this $Ph_2PO_2^-$ ligand is less sterically hindered than the $Mes_2PO_2^-$ one. The linear arrangement appears to be very

stable, as the compound does not dissolve in common organic solvents.

Experimental

1,4-Dibromo-2,3-dinitro-benzene was reacted with 2 equivalents of *n*-butyllithium (anhydrous thf, 173 K), followed by the addition of 2.5 equivalents of diphenylchlorophosphine (anhydrous thf, 193 K). The subsequent work-up (filtration, solvent removal, washings with diethyl ether) yielded a pale brown powder as a mixture of reaction products. Crystals of the title compound were obtained by allowing diethyl ether diffusion into a thf solution of the product mixture.

Crystal data

$[Li(C_{12}H_{10}O_2P)(C_4H_8O)]$	D_x = 1.306 Mg m^{-3}
M_r = 296.21	Mo $K\alpha$ radiation
Monoclinic, $P2_1/n$	Cell parameters from 25 reflections
a = 5.790 (1) Å	θ = 10–15°
b = 16.655 (3) Å	μ = 0.19 mm^{-1}
c = 15.782 (3) Å	T = 293 (2) K
β = 98.07 (2)°	Prism, light yellow
V = 1506.9 (6) Å^3	0.35 × 0.15 × 0.10 mm
Z = 4	

Data collection

Enraf–Nonius TurboCAD-4 diffractometer	R_{int} = 0.023
Non-profiled $\omega/2\theta$ scans	θ_{max} = 25.0°
Absorption correction: ψ scan (North *et al.*, 1968)	$h = 0 \rightarrow 6$
T_{min} = 0.941, T_{max} = 0.981	$k = 0 \rightarrow 19$
2913 measured reflections	$l = -18 \rightarrow 18$
2635 independent reflections	3 standard reflections
1602 reflections with $I > 2\sigma(I)$	frequency: 166 min
	intensity decay: 2%

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0465P)^2 + 0.9374P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)]$ = 0.050	$(\Delta/\sigma)_{max}$ = 0.006
$wR(F^2)$ = 0.130	$\Delta\rho_{max}$ = 0.30 e Å^{-3}
S = 1.03	$\Delta\rho_{min}$ = −0.29 e Å^{-3}
2635 reflections	
190 parameters	
H-atom parameters constrained	

Table 1
Selected geometric parameters (Å, °).

P1—O2	1.494 (2)	O1—Li1^{i}	1.962 (6)
P1—O1	1.505 (2)	O1—Li1	1.991 (6)
P1—C1	1.810 (3)	Li1—O2^{ii}	1.897 (6)
P1—C7	1.811 (3)	Li1—O50	2.028 (6)
P1—O1—Li1^{i}	148.6 (2)	O1^{i}—Li1—O1	91.7 (2)
P1—O1—Li1	120.7 (2)	O2^{ii}—Li1—O50	104.4 (3)
Li1^{i}—O1—Li1	88.3 (2)	O1^{i}—Li1—O50	109.6 (3)
O2^{ii}—Li1—O1^{i}	122.6 (3)	O1—Li1—O50	107.3 (3)
O2^{ii}—Li1—O1	120.2 (3)		

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

H atoms were positioned geometrically and allowed to ride on their respective parent atoms.

Data collection: *CAD*-4 *EXPRESS* (Enraf–Nonius, 1994); cell refinement: *CAD*-4 *EXPRESS*; data reduction: *XCAD*4 (Harms & Wocadlo, 1995); program(s) used to solve structure: *SIR*97 (Altomare *et al.*, 1999); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*-3 (Farrugia, 1997) and *PLATON* (Spek, 1990); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

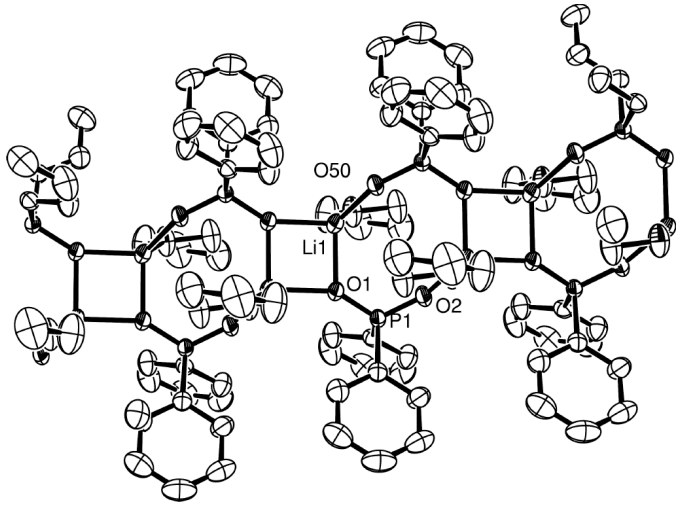


Figure 1
ORTEP-3 (Farrugia, 1997) view of (I), shown with 50% probability displacement ellipsoids. H atoms have been omitted.

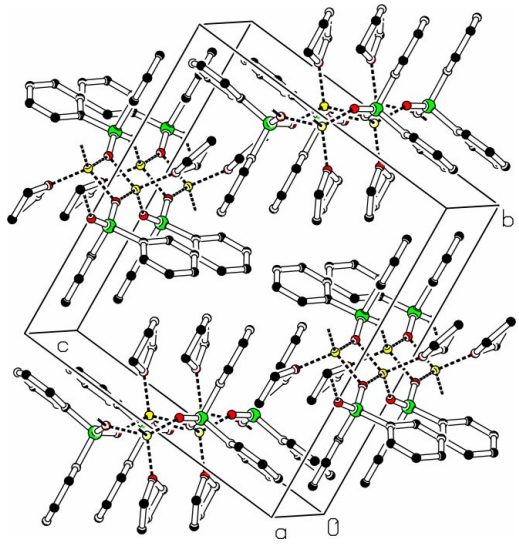


Figure 2
PLATON (Spek, 1990) diagram of the crystal packing. Color code: green P, yellow Li, red O and black C.

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