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# Polymeric (diphenylphosphinato)tetrahydro-furanlithium

Natcharee Kongprakaiwoot

Rudy L. Luck

Eugenijus Urnezisus

University of Portland, urnezisus@up.edu

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Natcharee Kongprakaiwoot,  
Rudy L. Luck\* and Eugenijus  
UrneziusDepartment of Chemistry, Michigan  
Technological University, 1400 Townsend  
Drive, Houghton, MI 49931, USA

Correspondence e-mail: rluck@mtu.edu

## Key indicators

Single-crystal X-ray study

T = 293 K

Mean  $\sigma(\text{C}-\text{C}) = 0.006 \text{ \AA}$ 

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>.Polymeric (diphenylphosphinato)tetrahydro-  
furanlithium

In the title compound,  $[\text{Li}(\text{C}_{12}\text{H}_{10}\text{O}_2\text{P})(\text{C}_4\text{H}_8\text{O})]_n$ , 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  $\mu$ -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.

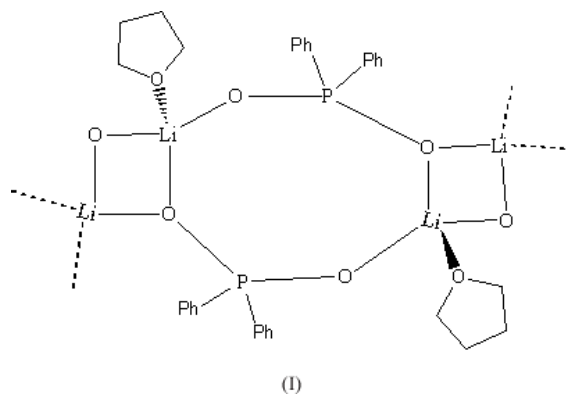
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## Comment

There has been only one previously reported structure of a lithium diorganophosphate complex, namely  $\text{Li}[\text{Mes}_2\text{PO}_2]$  (Beswick *et al.*, 1997). This complex consists of discrete dimeric molecules with two bridging  $\text{Mes}_2\text{PO}_2^-$  (dimesitylphosphinate) ligands attached to two  $\text{Li}^+$  cations, forming eight-membered rings. Two thf molecules, attached to each  $\text{Li}^+$  cation *via* lone pairs on the O atoms, complete the coordination geometry for these distinct dimers.



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  $\text{Li}^+$  atom. This arrangement no doubt results as this  $\text{Ph}_2\text{PO}_2^-$  ligand is less sterically hindered than the  $\text{Mes}_2\text{PO}_2^-$  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 <sub>12</sub> H <sub>10</sub> O <sub>2</sub> P)(C <sub>4</sub> H <sub>8</sub> O)]	$D_x = 1.306 \text{ Mg m}^{-3}$
$M_r = 296.21$	Mo $K\alpha$ radiation
Monoclinic, $P2_1/n$	Cell parameters from 25 reflections
$a = 5.790 (1) \text{ \AA}$	$\theta = 10\text{--}15^\circ$
$b = 16.655 (3) \text{ \AA}$	$\mu = 0.19 \text{ mm}^{-1}$
$c = 15.782 (3) \text{ \AA}$	$T = 293 (2) \text{ K}$
$\beta = 98.07 (2)^\circ$	Prism, light yellow
$V = 1506.9 (6) \text{ \AA}^3$	$0.35 \times 0.15 \times 0.10 \text{ mm}$
$Z = 4$	

#### Data collection

Enraf-Nonius TurboCAD-4 diffractometer	$R_{\text{int}} = 0.023$
Non-profiled $\omega/2\theta$ scans	$\theta_{\text{max}} = 25.0^\circ$
Absorption correction: $\psi$ scan (North <i>et al.</i> , 1968)	$h = 0 \rightarrow 6$
$T_{\text{min}} = 0.941$ , $T_{\text{max}} = 0.981$	$k = 0 \rightarrow 19$
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 $F^2$	$w = 1/[\sigma^2(F_o^2) + (0.0465P)^2 + 0.9374P]$
$R[F^2 > 2\sigma(F^2)] = 0.050$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.130$	$(\Delta/\sigma)_{\text{max}} = 0.006$
$S = 1.03$	$\Delta\rho_{\text{max}} = 0.30 \text{ e \AA}^{-3}$
2635 reflections	$\Delta\rho_{\text{min}} = -0.29 \text{ e \AA}^{-3}$
190 parameters	
H-atom parameters constrained	

**Table 1**

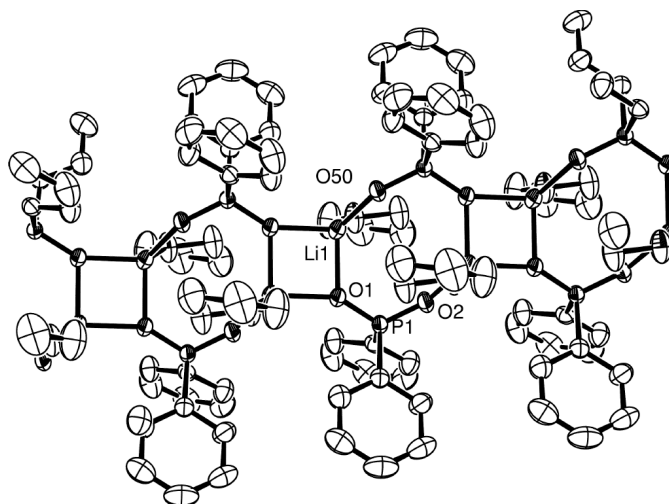
Selected geometric parameters ( $\text{\AA}$ ,  $^\circ$ ).

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

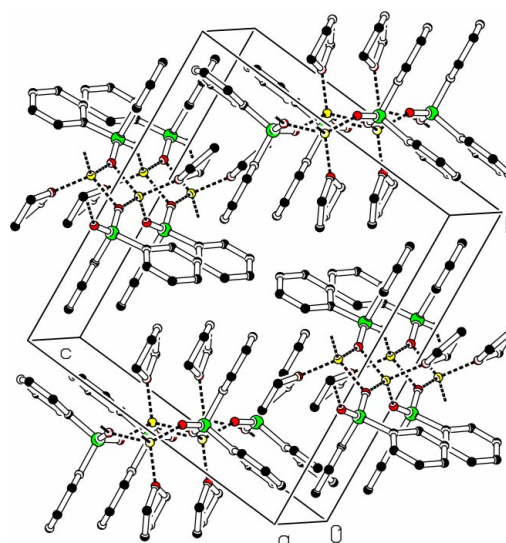
Symmetry codes: (i)  $1 - x, -y, 1 - z$ ; (ii)  $2 - x, -y, 1 - z$ .

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: *XCAD4* (Harms & Wocadlo, 1995); program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1999); program(s) used to refine structure: *SHELXL97* (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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