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catena-Poly[[(tetrahydrofuran-κO)lithium(**I**)]-μ-1,1,3,3-tetraphenyltriphosphane-$\kappa^2P^1,P^3{:}\kappa P^2$]

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The title compound, $[\mathrm{Li}(\mathrm{C}_{24}\mathrm{H}_{20}\mathrm{P}_3)(\mathrm{C}_4\mathrm{H}_8\mathrm{O})]_n$, was obtained by the reaction of bis(triethylphosphane)platinum(II) chloride with 1,1-diphenyl-2-(trimethylsilyl)-2-lithiadiphosphane, $(\mathrm{C}_6\mathrm{H}_5)_2\mathrm{P}-\mathrm{P}(\mathrm{SiMe}_3)\mathrm{Li}\cdot3\mathrm{C}_4\mathrm{H}_8\mathrm{O}$. The compound is formed in the latter stages of the reaction. It is the first coordination polymer among known triphosphane lithium salts.

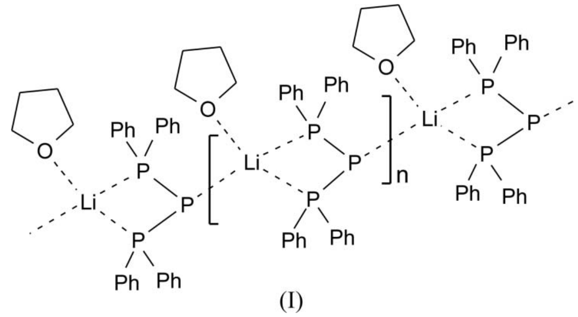
Key indicators

Single-crystal X-ray study
T = 120 K
Mean σ(C–C) = 0.003 Å
R factor = 0.064
wR factor = 0.139
Data-to-parameter ratio = 24.6

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 of the reactivity of lithium derivatives of silylated diphosphanes $R_2\mathrm{P}-\mathrm{P}(\mathrm{SiMe}_3)\mathrm{Li}$ towards compounds of transition metals (Baum *et al.*, 2004; Pikies *et al.*, 2004), we have studied the reaction of $(\mathrm{C}_6\mathrm{H}_5)_2\mathrm{P}-\mathrm{P}(\mathrm{SiMe}_3)$-$\mathrm{Li}\cdot3\mathrm{C}_4\mathrm{H}_8\mathrm{O}$ with $[(\mathrm{Et}_3\mathrm{P})_2\mathrm{PtCl}_2]$. The formation of the expected complex $[(\mathrm{Et}_3\mathrm{P})_2\mathrm{Pt}(\eta^2\text{-}\mathrm{Ph}_2\mathrm{P}{=}\mathrm{P})]$ was not observed. Instead, a cleavage of the P—P bond in the parent diphosphane takes place, together with the formation of phosphanes $\mathrm{Et}_3\mathrm{P}$ and $\mathrm{Ph}_2\mathrm{P}-\mathrm{PPh}_2$, the diphosphorus Pt^0 complex $[\{(\mathrm{Et}_3\mathrm{P})_2\mathrm{Pt}\}_2(\mathrm{P}_2)]$ and the complex described here, *viz*. $[(\mathrm{C}_6\mathrm{H}_5)_2\mathrm{P}]_2\mathrm{PLi}(\mathrm{C}_4\mathrm{H}_8\mathrm{O})$, (I).



(I)

The molecular structure of the monomeric unit of (I) is shown in Fig. 1. To the best of our knowledge, only four similar compounds are known, *viz*. $\mathrm{Li(THF)}_2[\eta^2\text{-}({}^t\mathrm{Bu}_2\mathrm{P})_2\mathrm{P}]$, (II), $\mathrm{Li(TMEDA)}[\eta^2\text{-}({}^t\mathrm{Bu}_2\mathrm{P})_2\mathrm{P}]$, (III), $\mathrm{Li(THF)}_2[\eta^2\text{-}({}^i\mathrm{Pr}_2\mathrm{P})_2\mathrm{P}]$, (IV), and $\mathrm{Li(THF)}_2[\eta^2\text{-}{}^t\mathrm{Bu}_2\mathrm{P}-\mathrm{P}-\mathrm{P}(\mathrm{NEt}_2)_2]$, (V) (Kovacs *et al.*, 1996), where THF is tetrahydrofuran and TMEDA is *N,N,N′,N′*-tetramethylethylenediamine. The geometry of the Li/P1/P3/P2 unit in (I) is similar to the arrangement of the Li atom and the triphosphane backbone in (II), (III) and (IV). In all these structures, the Li atom is bonded to the two terminal P atoms in a chelating manner, rather than to the central P atom. In (I), however, the Li atom forms an additional bond to the central P atom of a neighbouring triphosphane unit. In spite of the small steric repulsion of the $\mathrm{Ph}_2\mathrm{P}$ groups, the distances P1—P3 and P2—P3 in (I) are larger than the related distance of 2.1627 (8) Å in (IV). The Li—O(THF) bond length is in the typical range for a tetrahedrally coordinated Li atom

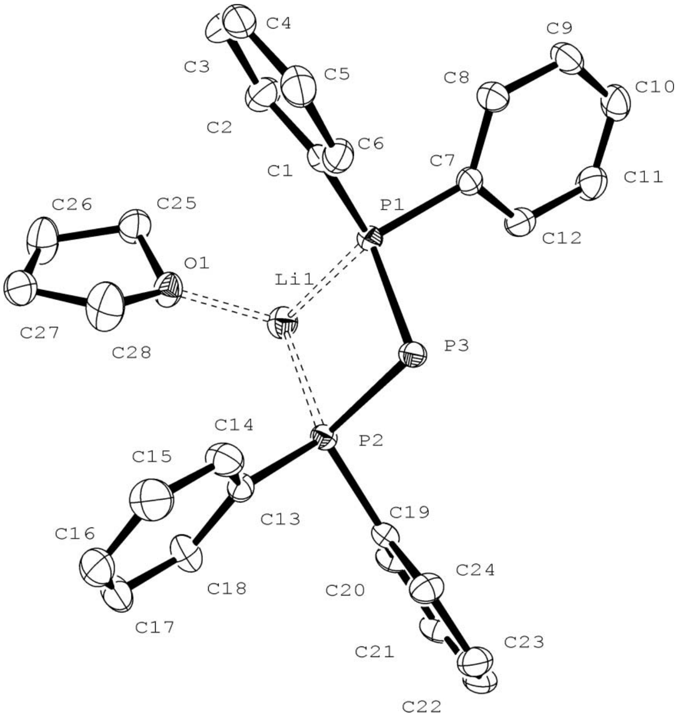


Figure 1
The molecular structure of the monomeric unit of (I), showing the atomic numbering scheme. Displacement ellipsoids are drawn at the 50% probability level. All H atoms have been omitted.

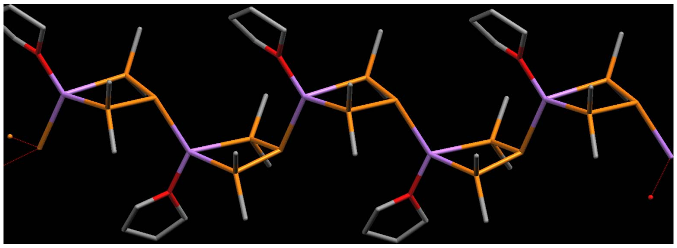


Figure 2
The polymeric chain formed by (I) in the solid state. Phenyl rings are represented only by the first C atom. H atoms have been omitted. Key: P orange, Li violet, O red and C gray.

with two THF molecules. The small steric repulsion of Ph_2P groups manifests itself in a small P1—P3—P2 angle of 92.51 (4)°, compared with 96.74 (7)° in (II) and 94.21 (4)° in (IV). Nonetheless, the most striking difference between (II), (III), (IV) and (I) is the polymeric structure of (I). The tetrahedral arrangement around the Li atom is created by two PPh_2 groups, a THF molecule and the central P3 atom of a neighbouring triphosphane molecule.

Experimental

All experiments were conducted under an atmosphere of carefully purified argon using standard Schlenk methods. To a suspension of $(Et_3P)_2PtCl_2$ (0.086 g, 0.175 mmol) in tetrahydrofuran (THF; 2 ml), a solution of $Ph_2P—P(SiMe_3)Li·3THF$ (0.176 g, 0.35 mmol) in THF (2 ml) was added at 233 K. The colourless solution slowly turned yellow at 233 K and red at room temperature. The solution was studied using ^{31}P NMR (see below). After standing for one month at room temperature the mixture was evacuated at 1 mTorr (15 min) and the residue dissolved in diethyl ether (4 ml) and filtered. After another month at 277 K, the solution yielded a small amount of orange crystals of (I). ^{31}P{^{1}H} NMR (162 MHz, external standard 85% H_3PO_4) of the reaction mixture (THF, C_6D_6), only for main products: $P(SiMe_3)_2Li$ δ −303.5 (*s*); $P(SiMe_3)_3$ δ −252.9 (*s*); $Ph_2P—P(SiMe_3)Li$ δ −206.8 (*d*) and −2.0 (*d*), ^{2}J (P—P) 288.0 Hz; Et_3P δ −19.2 (*s*); $Ph_2P—PPh_2$ δ −16.2 (*s*); $[(Et_3P)_2Pt]_2P_2$ δ 10.5 (*t*), ^{1}J (Pt—P) 3486 Hz and 46,2 (quintet), ^{2}J (P—P) 23.9 Hz (Schäfer & Binder, 1988).

Crystal data

$[Li(C_{24}H_{20}P_3)(C_4H_8O)]$
M_r = 480.35
Monoclinic, $P2_1/n$
a = 11.332 (5) Å
b = 9.519 (5) Å
c = 23.514 (8) Å
β = 94.735 (3)°
V = 2527.8 (19) Å^3
Z = 4

D_x = 1.262 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 11 491 reflections
θ = 2–25°
μ = 0.25 mm^{-1}
T = 120 (2) K
Plate, orange
0.31 × 0.27 × 0.10 mm

Data collection

Kuma KM-4 diffractometer with Oxford Diffraction Sapphire 2 CCD Camera
ω scans
Absorption correction: none
22 768 measured reflections
7335 independent reflections

6563 reflections with $I > 2\sigma(I)$
$R_{\rm int}$ = 0.044
$\theta_{\rm max}$ = 30.0°
h = −15 → 15
k = −9 → 13
l = −32 → 33

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.064
$wR(F^2)$ = 0.139
S = 1.14
7335 reflections
298 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0491P)^2 + 2.4715P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max}$ = 0.001
$\Delta\rho_{\rm max}$ = 0.54 e Å^{-3}
$\Delta\rho_{\rm min}$ = −0.29 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Li1—O1	1.909 (4)	Li1—P1	2.650 (4)
Li1—P3^{i}	2.569 (4)	P1—P3	2.1751 (10)
Li1—P2	2.623 (4)	P2—P3	2.1745 (10)
O1—Li1—P3^{i}	124.24 (18)	P3^{i}—Li1—P1	113.30 (14)
O1—Li1—P2	111.77 (17)	P2—Li1—P1	73.14 (11)
P3^{i}—Li1—P2	115.14 (14)	P2—P3—P1	92.51 (4)
O1—Li1—P1	107.66 (16)		

Symmetry code: (i) $\frac{1}{2} - x, \frac{1}{2} + y, \frac{1}{2} - z$.

All H atoms were positioned geometrically (C—H = 0.95–0.99 Å) and refined as riding, with $U_{\rm iso} = 1.2U_{\rm eq}$(parent atom).

Data collection: *CrysAlisCCD* (Oxford Diffraction, 2003); cell refinement: *CrysAlisRED* (Oxford Diffraction, 2003); data reduction: *CrysAlisRED*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

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