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catena-Poly[[(diethyl ether-κ*O*)lithium(I)]-μ-1,1,3,3-tetraisopropyltriphosphane-κP^2:κ$^2P^1$,P^3-lithium(I)-μ-1,1,3,3-tetraisopropyltriphosphane-κ$^2P^1$,P^3:κP^2]

Wioleta Domanska, Jaroslaw Chojnacki* and Jerzy Pikies

Department of Chemistry, Technical University of Gdańsk, 11/12 G. Narutowicz Street, 80952-PL, Gdańsk, Poland

Correspondence e-mail:
jarekch@chem.pg.gda.pl

The title compound, $[Li_2(C_{12}H_{28}P_3)_2(C_4H_{10}O)]_n$, is a by-product of the reaction of $[(Et_2PhP)_2PtCl_2]$ with $^iPr_2P$—$P(SiMe_3)Li$ in tetrahydrofuran. It is the second coordination polymer among known triphosphane–lithium salts. Of the two independent Li atoms, one is tetrahedrally coordinated, whereas the second has a trigonal environment.

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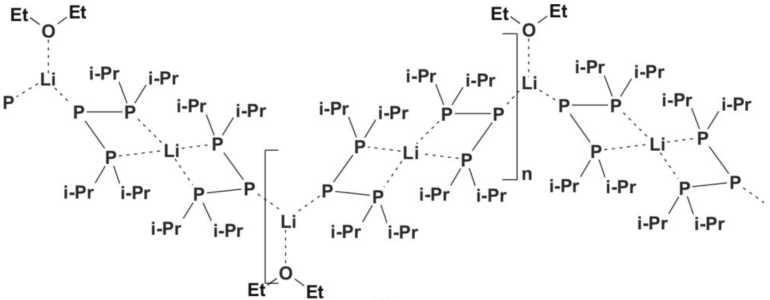
Key indicators

Single-crystal X-ray study
T = 100 K
Mean σ(C–C) = 0.004 Å
Disorder in main residue
R factor = 0.059
wR factor = 0.124
Data-to-parameter ratio = 31.6

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

Comment

We have a long-standing interest in the reaction of the lithiated diphosphanes, R_2P—$P(SiMe_3)Li$, with transition metal chlorides (Baum *et al.*, 2004; Pikies *et al.*, 2004; Domanska *et al.*, 2005). We have studied the reaction of $^iPr_2P$—$P(SiMe_3)Li$ with $[(R'_3P)_2PtCl_2]$ in order to obtain the phosphanophosphinidene complexes $[(R'_3P)_2Pt(\eta^2\text{-}P{=}PR_2)]$. In the case of $R'_3P = Et_3P$, the title compound, (I), forms in the reaction mixture in the early stages of the reaction. However, for $R'_3P = Et_2PhP$, compound (I) was not observed in the early stages of the reaction but only after prolonged crystallization. In both cases, the related phosphanophosphinidene complexes $[(R'_3P)_2Pt(\eta^2\text{-}P{=}PR_2)]$ were found in the reaction mixtures.



(I)

The structure of the monomeric unit of (I) is shown in Fig. 1. The polymeric structure contains a repeated spiro unit, $[(^iPr_2P)_2PLiP(^iPr_2P)_2]$, with atom Li1 in the central position. The planar Li1—P1—P2—P3 backbone is partially similar to the arrangement of the Li atom and the $^iPr_2P$—P—P^iPr_2 backbone in $[(LiC_{12}H_{28}P_3)(C_4H_8O)_2]$ (Kovacs *et al.*, 1996), with short P1—P2 and P2—P3 bonds of 2.163 (2) Å. The terminal P atoms are bonded to the central atom Li1 in a chelating manner, with relatively long P1—Li1 and P3—Li1 bonds of 2.575 (5) and 2.543 (5) Å, respectively. The second backbone, Li1—P4—P5—P6, displays similar features. These units are connected *via* an $Li(C_4H_{10}O)$ group, with an almost-planar trigonal coordination around atom Li2. The Li2—P5 [2.445 (5) Å] and Li2—P2′ [2.465 (5) Å] distances are shorter than in the case of the tetrahedrally coordinated Li atom in

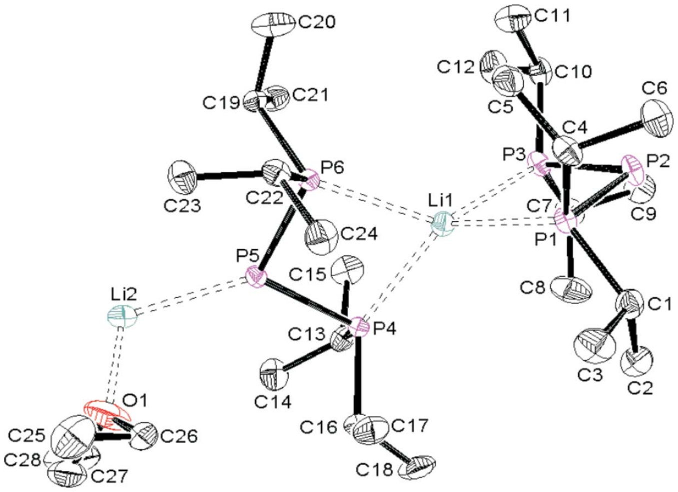


Figure 1
The molecular structure of the monomeric unit of (I), with the atomic numbering scheme. Displacement ellipsoids are drawn at the 50% probability level. All H atoms bonded to C atoms have been omitted.

$[Li(C_{24}H_{20}P_3)(C_4H_8O)]_n$ (Domanska *et al.*, 2005). The Li2—O1 distance of 1.902 (5) Å to the diethyl ether molecule is practically the same as in the case of tetrahedrally coordinated Li.

The polymeric chain of (I) is shown in Fig. 2. The most striking difference between (I) and the other known compounds of this type is the polymeric structure, with spiro units linked through a trigonally coordinated $Li(C_4H_{10}O)$ spacer.

Experimental

All experiments were carried out under an atmosphere of carefully purified argon, using standard Schlenk methods. To a suspension of $[(Et_2PhP)_2PtCl_2]$ (0.100 g, 0.199 mmol) in tetrahydrofuran (THF, 1.5 ml), a solution of $^iPr_2P—P(SiMe_3)Li·3THF$ (0.170 g, 0.404 mmol) in THF (1.5 ml) was added at 233 K. The colourless solution slowly turned yellow at 233 K and became orange at room temperature. The solution was studied using ^{31}P NMR. After standing for 1 d at room temperature, the mixture was dried under vacuum at 1 mTorr (1 Torr = 133.322 Pa) for 1 h, and the residue dissolved in diethyl ether (4 ml) and filtered. After one month at 277 K, the solution yielded small pale-yellow crystals of (I). Spectroscopic analysis: $^{31}P\{^1H\}$ NMR (162 MHz, external standard 85% H_3PO_4, solvent THF, C_6D_6, δ, p.p.m.): 33.2 (*d*), −179.8 [*t*, $^1J(P—P) = −312.6$ Hz].

Crystal data

$[Li_2(C_{12}H_{28}P_3)_2(C_4H_{10}O)]$
$M_r = 618.51$
Orthorhombic, $P2_12_12_1$
$a = 10.4017$ (8) Å
$b = 18.1543$ (14) Å
$c = 20.9916$ (14) Å
$V = 3964.0$ (5) Å^3
$Z = 4$
$D_x = 1.036$ Mg m^{-3}

Mo $K\alpha$ radiation
Cell parameters from 3951 reflections
$\theta = 5–30°$
$\mu = 0.29$ mm^{-1}
$T = 100$ (2) K
Prism, pale yellow
0.42 × 0.26 × 0.1 mm

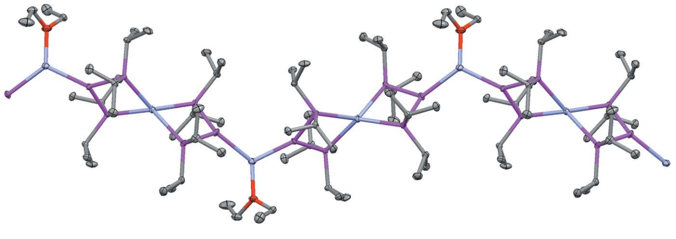


Figure 2
The polymeric chain formed by (I) in the solid state. H atoms have been omitted.

Data collection

Kuma KM-4 diffractometer with Sapphire 2 CCD area-detector
ω scans
Absorption correction: none
28850 measured reflections
11464 independent reflections

10078 reflections with $I > 2\sigma(I)$
$R_{int} = 0.063$
$\theta_{max} = 30°$
$h = −13 \rightarrow 14$
$k = −25 \rightarrow 22$
$l = −29 \rightarrow 29$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.059$
$wR(F^2) = 0.124$
$S = 1.07$
11464 reflections
363 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0515P)^2 + 1.6056P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.51$ e Å^{-3}
$\Delta\rho_{min} = −0.33$ e Å^{-3}
Absolute structure: Flack (1983), with 3434 Friedel pairs
Flack parameter: 0.02 (9)

All H atoms (methyl, methylene and methine) were refined as riding, with C—H distances of 0.98–0.99 Å and with $U_{iso}(H)$ constrained to be $1.2U_{eq}$(parent atom). One ethyl group (atoms C25–C26) was refined as disordered over two positions, with occupancy factors of 0.698 (9) and 0.302 (9).

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2005); cell refinement: *CrysAlis RED* (Oxford Diffraction, 2005); data reduction: *CrysAlis RED*; 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: *WinGX* (Farrugia, 1999).

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