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Bis[μ-bis(2,6-di-1-naphthylphenyl)phosphanido]-bis[(tetrahydrofuran)lithium(I)]

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Key indicators

Single-crystal X-ray study
T = 173 K
Mean σ(C–C) = 0.009 Å
Disorder in main residue
R factor = 0.083
wR factor = 0.262
Data-to-parameter ratio = 13.2

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

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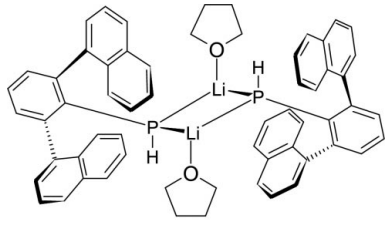
The molecular structure of the tetrahydrofuran adduct of the lithium salt of the primary phosphane $DnpPH_2$ (Dnp = 2,6-di-1-naphthylphenyl) of composition $[Li(THF)\{P(H)Dnp\}(THF)]_2$ is reported. The dimeric and centrosymmetric complex, $[Li_2(C_4H_8O)_2(C_{26}H_{22}OP)_2]$, features a three-coordinate Li atom in a slightly pyramidal coordination environment, with the sum of angles around it equal to 352.0° and a P atom in a highly distorted tetrahedral coordination environment.

Comment

Lithium phosphanide compounds have been known for decades and have been used extensively for the synthesis of a variety of metal complexes (Izod, 2000; Rabe *et al.*, 2001). Thus nowadays both primary and secondary lithium phosphanides are a well investigated class of compounds and several examples have been structurally characterized. We here report the first example of an alkali metal complex of the novel chiral (racemic) terphenyl-based phosphane $DnpH_2$ ($DnpPH_2$ = 2,6-di-1-naphthylphenyl). The terphenyl-like moiety adopts the chiral D,L form.



(I)

The central four-membered ring is planar, with Li···Li and P···P distances of 3.24 (2) and 3.778 (3) Å, and angles at P1 and Li1 of 81.2 (4) and 98.8 (4)°, respectively. Atom Li1 was found to be close to atoms $C18^i$, $C19^i$ and $C20^i$ with distances of 3.04 (1), 2.81 (1) and 3.07 (1) Å, respectively [symmetry code: (i) $1-x, 1-y, 1-z$]. This may indicate the presence of weak allyl-like electrostatic interactions between the Li atom and these atoms. The naphthyl ring systems are tilted with respect to the central benzene ring, with dihedral angles of 78.2 (2)° for C7–C16 and 75.5 (2)° for C17–C26. Compound (I) was found to be isostructural with its sodium analogue (Rabe, 2004).

Experimental

$DnpPH_2$ (Rabe, 2004) was prepared following the published procedures for $^tBu_3C_6H_2PH_2$ (Cowley *et al.*, 1990) and $DmpPH_2$ (Urnezius & Protasiewicz, 1996). Li(THF)P(H)Dnp was prepared by reaction of equimolar amounts of $DnpPH_2$ and *n*-BuLi in tetrahydrofuran at

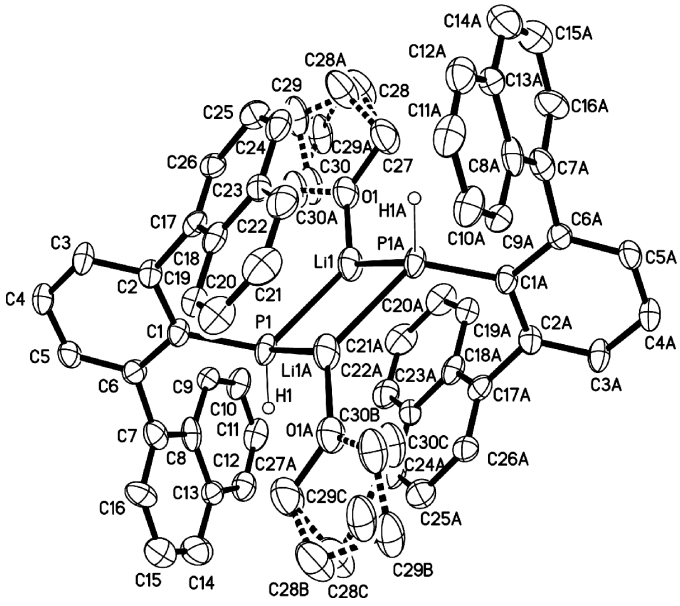


Figure 1
The molecular structure of the title compound, showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level. H atoms, except for those of the the P—H groups, have been omitted for clarity. Dashed lines are used to highlight the disorder with respect to C28, C29 and C30. Except for THF atoms, all atoms labelled as *A* were generated using the symmetry code $(1-x, 1-y, 1-z)$.

195 K and worked up following standard laboratory procedures. Single crystalline material of the title compound was obtained from a toluene solution at 248 K.

Crystal data

$[Li_2(C_4H_8O)_2(C_{26}H_{22}OP)_2]$	$Z = 1$
$M_r = 880.84$	$D_x = 1.247$ Mg m^{-3}
Triclinic, $P\overline{1}$	Mo $K\alpha$ radiation
$a = 10.4586$ (19) Å	Cell parameters from 700 reflections
$b = 11.3387$ (19) Å	$\theta = 2.0$–$25.0°$
$c = 11.5813$ (19) Å	$\mu = 0.14$ mm^{-1}
$\alpha = 62.710$ (10)°	$T = 173$ (2) K
$\beta = 74.360$ (11)°	Block, red
$\gamma = 86.041$ (11)°	0.30 × 0.15 × 0.15 mm
$V = 1172.9$ (4) Å^3	

Data collection

Bruker SMART CCD area-detector diffractometer	4117 independent reflections
	2126 reflections with $I > 2\sigma(I)$
φ and ω scans	$R_{int} = 0.067$
Absorption correction: multi-scan (*SADABS*; Bruker, 2001)	$\theta_{max} = 25.0°$
	$h = -12 \rightarrow 12$
$T_{min} = 0.940$, $T_{max} = 0.980$	$k = -13 \rightarrow 13$
9754 measured reflections	$l = -12 \rightarrow 13$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.1225P)^2 + 0.6192P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)] = 0.083$	
$wR(F^2) = 0.262$	
$S = 1.06$	$(\Delta/\sigma)_{max} < 0.001$
4117 reflections	$\Delta\rho_{max} = 0.67$ e Å^{-3}
312 parameters	$\Delta\rho_{min} = -0.26$ e Å^{-3}
H atoms treated by a mixture of independent and constrained refinement	

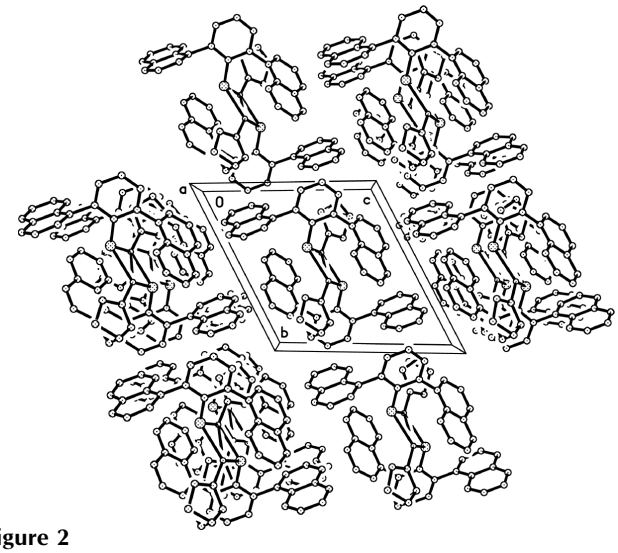


Figure 2
The contents of the unit cell, viewed along the *a* axis. All H atoms have been omitted for clarity.

Table 1
Selected geometric parameters (Å, °).

Li1—O1	1.867 (11)	P1—P1^i	3.778 (3)
Li1—P1	2.483 (11)	P1—C1	1.795 (5)
Li1—P1^i	2.493 (9)	P1—H1	1.34 (6)
Li1···Li1^i	3.24 (2)		
O1—Li1—P1	131.0 (4)	P1—Li1—P1^i	98.8 (4)
O1—Li1—P1^i	122.2 (5)	Li1—P1—Li1^i	81.2 (4)

Symmetry code: (i) $1-x, 1-y, 1-z$.

The high values of the residuals *R* and *wR* can be attributed to the poor quality of the crystal that was used. Atoms C28, C29 and C30 of the THF ring were found to be disordered over two positions with occupancy 0.62 (2)/0.38 (3). The C—C distances in the THF ring were restrained and the displacement parameters for each disordered pair (C28/C28A, C29/C29*A* and C30/C30*A*) were set equal to each other. The phosphorus H atom was found in a Fourier difference map and was refined freely. All other H atoms were placed in calculated positions (C—H = 0.95 and 0.99 Å), with isotropic displacement parameters fixed at 1.2 or 1.5 times U_{eq} of the parent atom and were refined as riding atoms.

Data collection: *SMART* (Bruker, 2001); cell refinement: *SMART*; data reduction: *SHELXTL* (Bruker, 2001); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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