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

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Key indicators

Single-crystal X-ray study
T = 173 K
Mean σ(C–C) = 0.004 Å
Disorder in main residue
R factor = 0.053
wR factor = 0.154
Data-to-parameter ratio = 13.5

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

The molecular structure of the tetrahydrofuran (THF) adduct of the sodium salt of the primary phosphane DnpPH$_2$ (Dnp is 2,6-di-1-naphthylphenyl), of composition [Na(THF){P(H)-Dnp}(THF)]$_2$ or [Na$_2$(C$_{26}$H$_{18}$P)$_2$(C$_4$H$_8$O)$_2$], is reported. The dimeric and centrosymmetric complex features a three-coordinate Na atom in a slightly pyramidal coordination environment, with a sum of angles around sodium of 348.9°, and a P atom in a highly distorted tetrahedral coordination environment.

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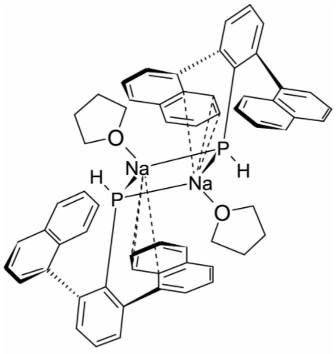
Comment

A number of secondary and a couple of primary lithium phosphanide compounds have been structurally characterized since the early 1980s (Izod, 2000; Rabe *et al.*, 2001, 2004). From the early 1990s, a number of reports of structure determinations of phosphanide compounds of the heavier alkali metals followed (Izod, 2000). To date, however, compared with the ubiquitous lithium compounds, there is still a dearth of structural data on sodium phosphanide compounds, most of which exhibit a dimeric arrangement in the solid state (Andrianarison *et al.*, 1990; Aspinall & Tillotson, 1996; Driess *et al.*, 1997; Koutsantonis *et al.*, 1995; Kühl *et al.*, 2000; Rabe *et al.*, 2002). We here report the molecular structure of the tetrahydrofuran solvated sodium salt, (I), of the novel terphenyl-based phosphane DnpPH$_2$ (Dnp is 2,6-di-1-naphthylphenyl).



(I)

Atom Na1 is close to atoms C18*A*, C19*A* and C20*A* (Fig. 1), with distances of 3.088 (2), 2.917 (3) and 3.003 (3) Å, respectively. This configuration may indicate the presence of a weak allyl-like electrostatic interaction between the Na atom and these atoms. The naphthyl ring systems are tilted with respect to the central benzene ring, with dihedral angles of 76.40 (7)° for the C7–C16 ring and 74.17 (8)° for the C17–C26 ring.

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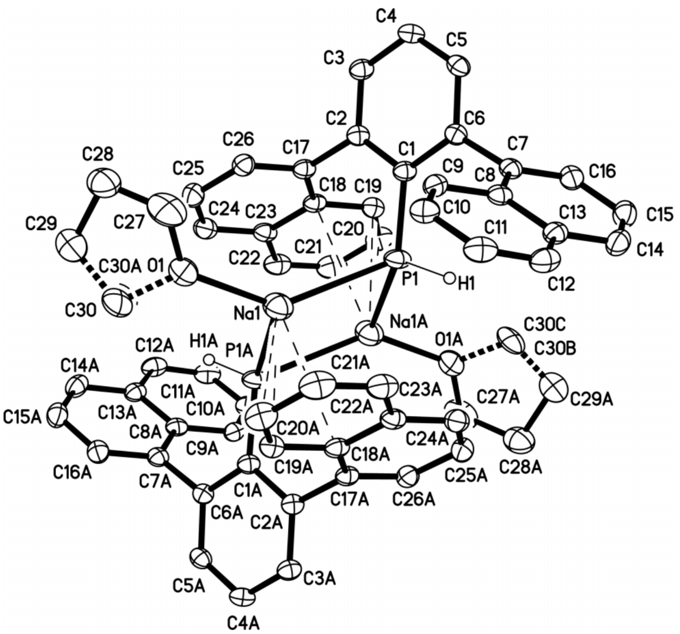

Figure 1
Molecular structure of the title compound, showing the atom-labeling scheme. Displacement ellipsoids are drawn at the 30% probability level. H atoms, except the P-bound H atoms, have been omitted for clarity. Heavy dashed lines are used to highlight the disorder with respect to C30, while a light dashed line illustrates the weak allyl-like interaction with Na1. Except for the disorder of C30, all atoms labeled as A were generated using the symmetry operator $(1 - x, 1 - y, 1 - z)$.

Compound (I) was found to be isostructural with its lithium analog (Rabe *et al*.,, 2004).

Experimental

DnpPH$_2$ (Rabe, 2004) was prepared following the published procedures for ^{t}Bu$_3$C$_6$H$_2$PH$_2$ (Cowley *et al*., 1990) and DmpPH$_2$ (Urnezius & Protasiewicz, 1996). Na(THF)P(H)Dnp was prepared by reaction of DnpPH$_2$ with excess sodium metal in THF at ambient temperature. The reaction mixture was centrifuged, evaporated to dryness, washed with hexanes and toluene, and dried under vacuum. Single crystals of the title compound were obtained from a toluene/THF (20:1) solution inside a glove-box at room temperature.

Crystal data

$[Na_2(C_{26}H_{18}P)_2(C_4H_8O)_2]$
$M_r = 912.94$
Triclinic, $P\bar{1}$
$a = 10.5408\ (9)$ Å
$b = 11.5054\ (10)$ Å
$c = 11.6538\ (10)$ Å
$\alpha = 61.951\ (1)^\circ$
$\beta = 75.180\ (1)^\circ$
$\gamma = 87.642\ (1)^\circ$
$V = 1200.64\ (18)$ Å^3
$Z = 1$
$D_x = 1.263$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 750 reflections
$\theta = 2.0$–25.0°
$\mu = 0.15$ mm^{-1}
$T = 173\ (2)$ K
Block, red
$0.40 \times 0.30 \times 0.20$ mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2001)
$T_{min} = 0.913$, $T_{max} = 0.970$
11 571 measured reflections
4213 independent reflections
3354 reflections with $I > 2\sigma(I)$
$R_{int} = 0.030$
$\theta_{max} = 25.0^\circ$
$h = -12 \rightarrow 12$
$k = -13 \rightarrow 13$
$l = -13 \rightarrow 13$

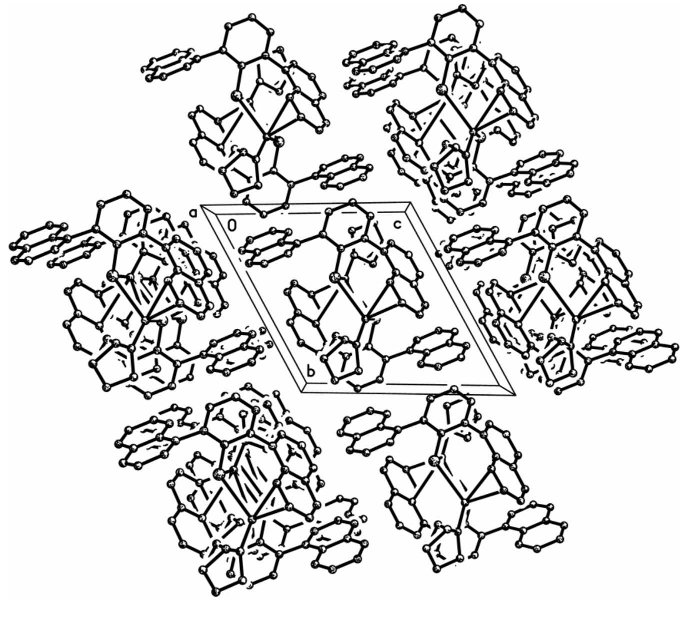

Figure 2
The crystal packing, viewed along the *a* axis. All H atoms have been omitted for clarity.

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.053$
$wR(F^2) = 0.154$
$S = 1.02$
4213 reflections
312 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0886P)^2 + 0.5175P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.51$ e Å^{-3}
$\Delta\rho_{min} = -0.35$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

P1—C1	1.810 (2)	P1—P1^i	4.153 (1)
P1—Na1	2.7769 (14)	Na1—O1	2.218 (3)
P1—Na1^i	2.8468 (13)	Na1—Na1^i	3.793 (2)
P1—H1	1.32 (3)		
Na1—P1—Na1^i	84.81 (4)	O1—Na1—P1^i	113.25 (8)
O1—Na1—P1	140.49 (7)	P1—Na1—P1^i	95.19 (4)

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

Atom C30 in the THF ring was found to be disordered over two positions with roughly 68 (3)/32 (3)% occupancy. The phosphorus H atom was found from a Fourier difference map and was allowed to refine isotropically. 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. Refinement in space group $P1$ gave higher standard uncertainties in bonding parameters and additional disorder with respect to C atoms of the THF rings.

Data collection: *SMART* (Bruker, 2001); cell refinement: *SMART*; data reduction: *SAINT* and *SHELXTL* (Bruker, 2001); program(s) used to solve structure: *SHELXS*97 (Cowley *et al*., 1990); 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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