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Bis(μ-*N*-*n*-propyl-*N*-trimethylsilylbenzamidinato)bis[(diethyl ether-*O*)lithium]

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Key indicators

Single-crystal X-ray study
T = 150 K
Mean σ(C–C) = 0.003 Å
R factor = 0.046
wR factor = 0.042
Data-to-parameter ratio = 20.7

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

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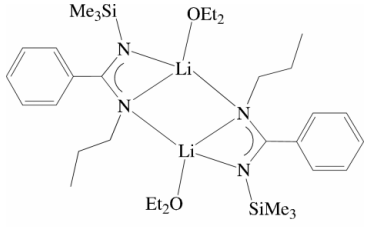
The title compound, $[Li_2(C_{13}H_{22}N_2Si)_2(C_4H_{10}O)_2]$, possesses a dinuclear structure featuring four-coordinate Li atoms, each of which shows a further, weak, contact to the central C atom of the NCN linkage of the amidinate ligand. The molecules possess crystallographically imposed inversion symmetry.

Comment

In recent years, there has been much interest in *N*-silylated benzamidinates (Edelmann, 1994). New types of amidinate ligands with pendant amine or pyridine functionalities have recently been reported (Brandsma *et al.*, 1998; Doyle *et al.*, 2000; Kincaid *et al.*, 1999). We have been interested in developing the chemistry of pendant-arm-functionalized amidinates as supporting ligands for group 4 imido chemistry (Boyd *et al.*, 2002). As part of these studies, we were interested in using an *N*-propyl-substituted benzamidinate ligand, namely $Me_3SiNC(Ph)NCH_2CH_2CH_3$, for comparison with the pendant alkylamine-substituted amidinates $Me_3SiNC(Ph)NCH_2CH_2NMe_2$ and $Me_3SiNC(Ph)NCH_2CH_2CH_2NMe_2$ (Brandsma *et al.*, 1998; Doyle *et al.*, 2000). We describe here the structure of the diethyl ether adduct of the lithium salt of this ligand, (I).



(I)

Molecules of (I) adopt a dinuclear structure in the solid state, possessing crystallographically imposed inversion symmetry. Each Li atom, and each propyl-substituted benzamidinate N atom, is four-coordinate, with Li deviating the most from an ideal tetrahedral geometry due to the restricted bite angle of the amidinate group. The central motif of the structure is a four-membered ring of alternating Li and N atoms, which is planar by symmetry. To either end of this ring is fused another, slightly puckered, four-membered ring. This puckering sees atoms C1 and Li lying out of the Li1/N1/C1/N2 least-squares plane of the ring by *ca* 0.19 and 0.07 Å, respectively. This creates a close Li1···C1 contact of 2.334 (3) Å, which is possibly electrostatic in nature. This distance is at the lower end of Li···CN_2 distances observed for other lithiated amidinates, which are in the range *ca* 2.30–2.42 Å (Fletcher *et al.*, 1996). The Li—N distances and N—Li—N angles in (I)

span the typical ranges reported for lithiated amides (Fletcher *et al.*, 1996; Allen & Kennard, 1993).

The structural motif in (I) is closely related to that of the Li salt of the pendant *N*-propylamine-substituted ligand, namely $Li_2\{Me_3SiNC(Ph)NCH_2CH_2CH_2NMe_2\}_2$, synthesized by Lappert and co-workers (Doyle *et al.*, 2000). In this case, there is no Et_2O coordinated to Li and the remaining site is occupied by the chelating pendant amine N-donor.

Experimental

The title compound was prepared according to previously described procedures (Boyd *et al.*, 2002). Crystallization of the crude product from a mixture of hexane and Et_2O afforded the title compound as air-sensitive colourless blocks.

Crystal data

$[Li_2(C_{13}H_{22}N_2Si)_2(C_4H_{10}O)_2]$
$M_r = 628.94$
Triclinic, $P\overline{1}$
$a = 8.4111$ (17) Å
$b = 10.486$ (2) Å
$c = 11.774$ (2) Å
$\alpha = 106.59$ (3)°
$\beta = 96.05$ (3)°
$\gamma = 99.38$ (3)°
$V = 969.2$ (4) Å^3
$Z = 1$
$D_x = 1.078$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 3991 reflections
$\theta = 5$–27°
$\mu = 0.12$ mm^{-1}
$T = 150$ K
Block, colourless
0.32 × 0.28 × 0.28 mm

Data collection

Enraf–Nonius KappaCCD diffractometer
φ and ω scans
Absorption correction: multi-scan (*DENZO*; Otwinowski & Minor, 1997)
$T_{min} = 0.97$, $T_{max} = 0.97$
7923 measured reflections
4372 independent reflections
2907 reflections with $I > 2\sigma(I)$
$R_{int} = 0.02$
$\theta_{max} = 27.5°$
$h = -10 \rightarrow 10$
$k = -13 \rightarrow 13$
$l = -14 \rightarrow 15$

Refinement

Refinement on F
$R = 0.046$
$wR = 0.042$
$S = 1.05$
4117 reflections
199 parameters
H-atom parameters constrained
Weighting scheme: see text
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.39$ e Å^{-3}
$\Delta\rho_{min} = -0.47$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Li1—N1	2.023 (3)	N1—Si1	1.7083 (15)
Li1—N2	2.145 (3)	N1—C1	1.330 (2)
Li1—N2^{i}	2.073 (3)	N2—C1	1.332 (2)
Li1—O1	1.965 (3)	N2—C8	1.470 (2)
Li1—C1	2.334 (3)	C1—C2	1.512 (2)
N1—Li1—N2	66.61 (11)	Li1^{i}—N2—C1	127.44 (15)
N1—Li1—N2^{i}	121.29 (16)	Li1—N2—C8	141.30 (14)
N2—Li1—N2^{i}	107.22 (14)	Li1^{i}—N2—C8	108.94 (14)
N1—Li1—O1	123.08 (16)	C1—N2—C8	119.81 (15)
N2—Li1—O1	111.49 (16)	Li1—O1—C15	134.78 (15)
N2^{i}—Li1—O1	113.47 (15)	Li1—O1—C16	109.67 (14)
Li1—N1—Si1	140.05 (12)	C15—O1—C16	112.43 (15)
Li1—N1—C1	85.59 (14)	N1—C1—N2	118.71 (16)
Si1—N1—C1	131.63 (13)	N1—C1—C2	119.68 (15)
Li1—N2—Li1^{i}	72.78 (14)	N2—C1—C2	121.55 (15)
Li1—N2—C1	80.64 (13)		

Symmetry code: (i) $1-x, 1-y, 2-z$.

The weighting scheme used in the refinement was a Prince modified Chebychev polynomial (Watkin, 1994), in which $W = [\text{weight polynomial}][1 - (\Delta F/6\sigma F)^2]^2$, with coefficients −0.679, −2.35, −1.48, −0.984 and −0.429. All H atoms were placed geometrically and refined with a riding model.

Data collection: *COLLECT* (Nonius, 2000); cell refinement: *DENZO* (Otwinowski & Minor, 1997); data reduction: *DENZO*; program(s) used to solve structure: *SIR*97 (Altomare *et al.*, 1999); program(s) used to refine structure: *CRYSTALS* (Watkin *et al.*, 2001); molecular graphics: *CAMERON* (Watkin *et al.*, 2001); software used to prepare material for publication: *CRYSTALS*.

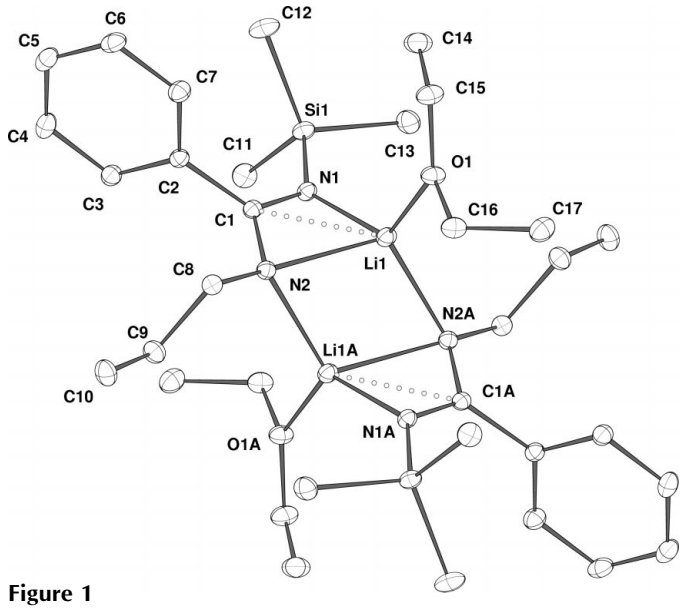


Figure 1
View of the molecular structure of (I). Displacement parameters are drawn at the 20% probability level and H atoms have been omitted for clarity. Atoms carrying the suffix A are related to their counterparts by the symmetry code $(1-x, 1-y, 2-z)$.

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