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Bis(μ-*N*-*tert*-butyl-2-{*N*-[2-(dimethylamino)ethyl]-*N*-methylamino}ethanaminato)dilithium(I)

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The title compound $[Li_2(C_{11}H_{26}N_3)_2]$, is a dimer chelate complex and features a planar Li_2N_2 ring with an Li$\cdots$Li distance of 2.449 (3) Å and a bicyclic ring system. The coordination geometry of both Li atoms is distorted tetrahedral, each Li unit being surrounded by two amide and two amine N atoms.

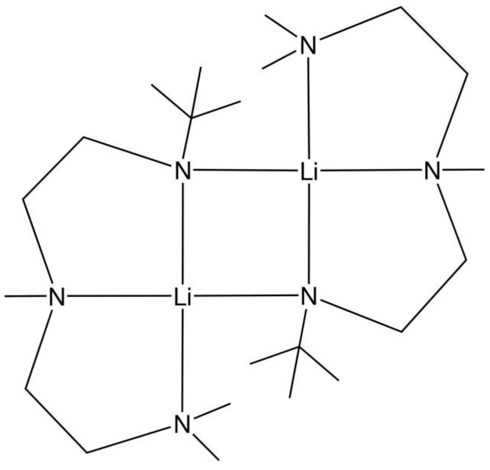
Key indicators

Single-crystal X-ray study
T = 150 K
Mean σ(C–C) = 0.002 Å
Disorder in main residue
R factor = 0.051
wR factor = 0.146
Data-to-parameter ratio = 16.6

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

Comment

Lithium amides (LiNR_2) are important reagents for the generation of enolates and their analogues from carbonyl compounds and related carbon-based acids. In this context, we have studied the crystal structure of the title compound, (I).



(I)

Compound (I) is a dimer chelate complex and consists of two Li[TBTMEDA] (TBTMEDA = *N*-*tert*-butyl-2-{*N*-[2-(dimethylamino)ethyl]-*N*-methylamino}ethanaminate) units (Fig. 1). In the center of the molecule is a planar Li_2N_2 ring, in which the Li atoms are linked by amide atoms N1 and N4. The Li$\cdots$Li distance is 2.449 (3) Å, which agrees with 2.43 (5) Å found by Trepanier & Wang (1993) in $[Li(TEDTA)]_2$. The 5,8-N atoms of the TBTMEDA ligand are attached to the Li atom in a bicyclic ring system.

Each Li atom is surrounded in a distorted tetrahedral arrangement by four N atoms, two amide (N1 and N4) and two amine (N2 and N3 for Li1, and N5 and N6 for Li2). The bond lengths Li1—N1 and Li2—N4 (Table 1) are rather short and agree with amide N—Li distances [1.995 (3)–2.047 (2) Å] in $[Li(TACN\text{-}^{i}Pr_2)]_2$ reported by Qian *et al.* (2000). The bond lengths Li1—N4 and Li2—N1 are longer and agree with amide N—Li distances (2.049–2.162 Å) in $[(Me_3B_3N_3H_2Li(TMTA)]_2$ and $[(Me_3B_3N_3H_2Li(TMTA)]_2$ (Nöth *et al.*, 2005).

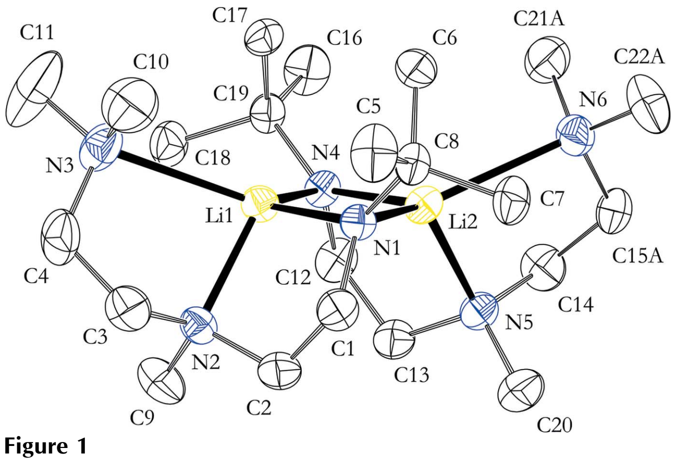


Figure 1
The molecular structure of (I), showing the atomic numbering scheme. Displacement ellipsoids are drawn at the 50% probability level. H atoms have been omitted for clarity. Only the major disorder component is shown.

All four other neutral amine N atoms are bound to the central Li atoms, two to each Li atom. The bond lengths Li1—N2 and Li—N4 compare well with the Li—N distances in [Li(DMEDA]$_2$ and [Li(TMEDA]$_2$ [2.082 (2) Å] reported by van Vliet *et al.* (1999). Li1—N3 and Li2—N6 are very long. A search for the tetrahedral LiN$_4$ fragment in the Cambridge Structural Database (Version 5.28, November 2006; Allen, 2002) revealed an Li—N distance range of 1.938–2.483 Å, with a mean of 2.155 (4) Å. There is an outlier in this search of 2.627 Å, which is perpendicular to a pyridine ring and is not a regular coordinate bond but ascribed to a weak π-interaction (Clegg *et al.*, 2000). The tetrahedral geometry of N3 and N6 suggests that the free electron pairs of both N atoms are pointing in the directions of the central Li1 (for N3) and Li2 (for N6) atoms. Therefore, we assume a bonding interaction, despite the unusually long distances for Li1—N3 and Li2—N2.

The coordination environments of both Li atoms are similar, the largest angle deviation being 4.05° for N2—Li1—N4/N1—Li2—N6. The bond distances of the Li2 unit are about 0.03 Å longer than the corresponding distances in the Li1 unit, except for the Li2—N6 bond, which is 0.110 Å shorter than Li1—N4.

Experimental

To a solution of TBTMEDA in pentane was added 1.0 equivalent BuLi at 195 K. After stirring for 15 min at room temperature, the reaction mixture was evaporated to dryness. Brown crystals suitable for data collection were obtained after recrystallization from pentane. Synthesis and crystallization were performed under argon in oven-dried glassware.

Crystal data

[Li$_2$(C$_{11}$H$_{26}$N$_3$)$_2$]
M_r = 414.58
Monoclinic, $P2_1/c$
a = 15.3016 (2) Å
b = 9.6296 (1) Å
c = 20.2624 (2) Å
β = 112.5155 (8)°
V = 2758.05 (6) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.06 mm^{-1}
T = 150 (2) K
0.50 × 0.30 × 0.25 mm

Data collection

Nonius KappaCCD diffractometer
Absorption correction: none
41056 measured reflections
5184 independent reflections
4396 reflections with $I > 2\sigma(I)$
$R_{\rm int}$ = 0.091

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.051
$wR(F^2)$ = 0.146
S = 1.06
5184 reflections
312 parameters
H-atom parameters constrained
$\Delta\rho_{\rm max}$ = 0.43 e Å^{-3}
$\Delta\rho_{\rm min}$ = −0.26 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Li1—N1	2.001 (2)	Li2—N4	2.031 (3)
Li1—N2	2.064 (2)	Li2—N5	2.098 (3)
Li1—N4	2.099 (2)	Li2—N1	2.124 (2)
Li1—N3	2.628 (3)	Li2—N6	2.518 (3)
Li1—Li2	2.449 (3)		
N1—Li1—N2	90.88 (10)	C11—N3—C10	109.7 (2)
N1—Li1—N4	108.29 (11)	C11—N3—C4	109.55 (19)
N2—Li1—N4	121.11 (12)	C10—N3—C4	109.99 (16)
N1—Li1—N3	116.34 (10)	C11—N3—Li1	128.73 (13)
N2—Li1—N3	78.07 (8)	C10—N3—Li1	99.67 (13)
N4—Li1—N3	131.16 (11)	C4—N3—Li1	97.92 (11)
N4—Li2—N5	89.61 (10)	C21*A*—N6—C15*A*	112.27 (16)
N4—Li2—N1	106.20 (11)	C21*A*—N6—C22*A*	109.16 (18)
N5—Li2—N1	125.16 (12)	C15*A*—N6—C22*A*	106.77 (15)
N4—Li2—N6	115.45 (10)	C21*A*—N6—Li2	101.57 (13)
N5—Li2—N6	79.67 (8)	C15*A*—N6—Li2	99.75 (10)
N1—Li2—N6	131.73 (11)	C22*A*—N6—Li2	126.95 (12)

All H atoms were introduced in geometrically idealized positions (C—H = 0.98–0.99 Å) and refined with a riding model, with $U_{\rm iso}$(H) = 1.2$U_{\rm eq}$(C) for H atoms of CH$_2$ groups and with $U_{\rm iso}$(H) = 1.5$U_{\rm eq}$(C) for methyl H atoms. The three C atoms bonded to N6 are conformationally disordered: C15*A*/C15*B*, C21*A*/C21*B* and C22*A*/C22*B*. The occupancy factors for these positions were fixed at an *A*:*B* ratio of 0.85:0.15 and their anisotropic displacement parameters were restrained to approximately isotropic behaviour. The C—N and C—C bonds involving the disordered atoms were refined with distance and angle restraints.

Data collection: *COLLECT* (Nonius, 1999); cell refinement: *DENZO* (Version 1.11.0; Otwinowski & Minor, 1997); data reduction: *DENZO* and *SORTAV* (Blessing, 1987); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97*.

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