# metal-organic papers

## catena-Poly[[bis(tetrahydrofuran)lithium(I)]-μ-N-(2-isopropylphenyl)benzamidinato]

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**Key indicators**

Single-crystal X-ray study
$T$ = 203 K
Mean $\sigma$(C–C) = 0.004 Å
$R$ factor = 0.060
$wR$ factor = 0.148
Data-to-parameter ratio = 15.1

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

The title compound, $[Li(C_{16}H_{17}N_2)(C_4H_8O)_2]_n$, is an unsymmetrical benzamidinate exhibiting a one-dimensional chain made up of lithium-linked N—C—N units. Two tetrahydrofuran molecules complete the distorted $LiO_2N_2$ tetrahedral coordination.

## Comment

Amidinate ligands form complexes with a wide variety of metals from all parts of the periodic table and have been proposed as alternatives for cyclopentadienyl species (Barker & Kilner, 1994; Edelmann, 1994). Lithium amidinates can act as ligand-transfer reagents for the corresponding metal complexes. Here, we present the title compound, (I), which is a new lithium amidinate. It was prepared by treatment of lithiated 2-isopropylaniline with one equivalent of benzonitrile.



(I)
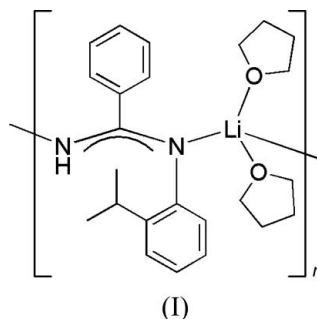
Compound (I) contains an unsymmetrical benzamidinate ligand (Fig. 1), with only one 2-isopropylphenyl group attached to the N atom. We note that the H atom originally attached to the anilido N atom is activated after the addition reaction and it can migrate to the other N atom, in order to form a conjugated amidinate backbone. This H atom does not participate in any hydrogen bonds. The bonding mode of the ligand and the Li$^I$ ion results in $N,N'$-$\mu$-bridges and a polymeric chain structure (Fig. 2) comprising repeating –N—C—N—Li– units propagating along [010]. The extended chain is thus generated by the $2_1$ screw axis, resulting in a screw pitch of 11.174 (3) Å.

For the conjugated N—C—N group, the C10—N1 and C10—N2 bond lengths are in the normal range (Guo *et al.*, 2004), at 1.336 (3) and 1.312 (3) Å, respectively. However, the N1—C10—N2 angle of 129.44 (19)° in (I) is larger than the corresponding angle of 119.1 (5)° in a chelate benzamidinate lithium compound, $Li_2(C_{14}H_{24}N_3Si)_2$ (Tong *et al.*, 2004). Both of the N atoms in the N—C—N conjugated anion in (I) are $sp^2$ hybridized; the sums of the angles around N1 and N2 are 359.4
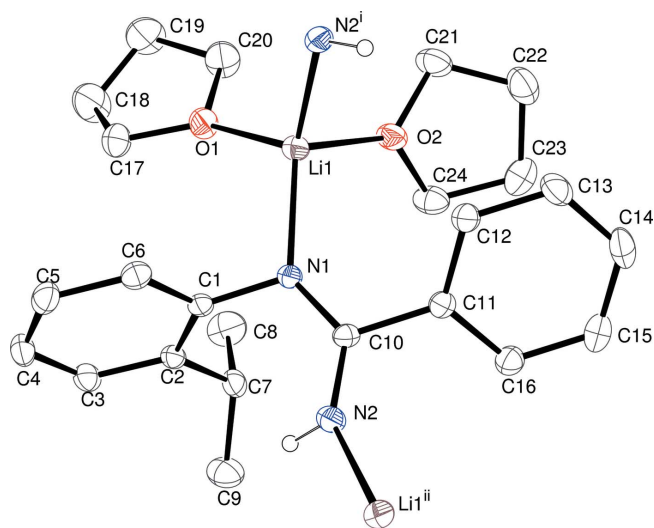
**Figure 1**
A view of (I), showing 30% probability displacement ellipsoids. All H atoms except H2 have been omitted for clarity. Symmetry codes as in Table 1.

and 360.0°, respectively. The mean plane of the benzene ring attached to C10 makes a dihedral angle of 44.76 (27)° with respect to the N—C—N amidinate plane. The dihedral angle between the mean plane of the benzene ring attached to N1 and the N—C—N plane is 82.65 (24)°.

The Li$^I$ ion links two benzamidinate ligands and is also coordinated by two tetrahydrofuran molecules, in a somewhat distorted tetrahedral coordination, with N—Li—N [123.91 (18)°] and O—Li—O [96.34 (16)°] being notably obtuse and acute, respectively.
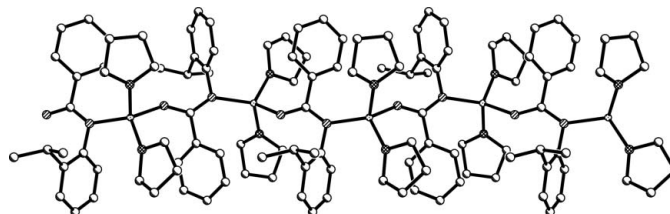
## Experimental

All reactions were carried out under a nitrogen atmosphere in flamed Schlenk-type glassware on a dual-manifold Schlenk line. To a solution of [(2-$^i$PrC$_6$H$_4$)NH$_2$] (0.48 g, 3.6 mmol) in a mixture of Et$_2$O (20 ml) and tetrahydrofuran (THF, 5 ml) was slowly added a solution of LiBu$^n$ (2.93 $M$, 1.2 ml, 3.6 mmol) in hexane at 273 K. The reaction mixture was allowed to warm to room temperature and stirred continuously for 2 h. Benzonitrile (0.37 ml, 3.6 mmol) was injected by syringe at 273 K. The mixture was stirred at room temperature for 8 h and a white compound was precipitated. After removing the solvent by filtration, compound (I) was recrystallized from THF (yield 1.31 g, 94%; m.p. 379–381 K). Spectroscopic analysis: $^1$H NMR (300 MHz, C$_6$D$_6$, $\delta$, p.p.m.): 7.88–6.81 ($m$, 9H, phenyls), 4.64, 4.08 ($d$, 1H, NH), 3.79 ($m$, 1H, CH), 3.62 ($s$, 8H, OCH$_2$ of THF), 1.45 ($s$, 8H, 3,4-2CH$_2$ of THF), 1.30 ($dd$, $J_{HH}$ = 10.5 and 6.6 Hz, 6H, methyls).

### Crystal data

| | |
|---|---|
| [Li(C$_{16}$H$_{17}$N$_2$)(C$_4$H$_8$O)$_2$] | $D_x = 1.150$ Mg m$^{-3}$ |
| $M_r = 388.46$ | Mo $K\alpha$ radiation |
| Monoclinic, $P2_1/n$ | Cell parameters from 2066 |
| $a = 8.964$ (2) Å | reflections |
| $b = 11.174$ (3) Å | $\theta = 2.3–21.6°$ |
| $c = 22.689$ (6) Å | $\mu = 0.07$ mm$^{-1}$ |
| $\beta = 99.142$ (4)° | $T = 203$ (2) K |
| $V = 2243.7$ (10) Å$^3$ | Prism, colourless |
| $Z = 4$ | $0.30 \times 0.30 \times 0.20$ mm |



**Figure 2**
The chain structure of (I).

### Data collection

| | |
|---|---|
| Bruker SMART CCD area-detector diffractometer | 3948 independent reflections |
| | 3074 reflections with $I > 2\sigma(I)$ |
| $\omega$ scans | $R_{int} = 0.034$ |
| Absorption correction: multi-scan (SADABS; Bruker, 2000) | $\theta_{max} = 25.0°$ |
| | $h = -10 \rightarrow 10$ |
| $T_{min} = 0.818$, $T_{max} = 0.986$ | $k = -13 \rightarrow 12$ |
| 9335 measured reflections | $l = -26 \rightarrow 24$ |

### Refinement

| | |
|---|---|
| Refinement on $F^2$ | $w = 1/[\sigma^2(F_o^2) + (0.0504P)^2$ |
| $R[F^2 > 2\sigma(F^2)] = 0.060$ | $+ 1.379P]$ |
| $wR(F^2) = 0.148$ | where $P = (F_o^2 + 2F_c^2)/3$ |
| $S = 1.05$ | $(\Delta/\sigma)_{max} < 0.001$ |
| 3948 reflections | $\Delta\rho_{max} = 0.44$ e Å$^{-3}$ |
| 262 parameters | $\Delta\rho_{min} = -0.30$ e Å$^{-3}$ |
| H-atom parameters constrained | |

**Table 1**
Selected geometric parameters (Å, °).

| | | | |
|---|---|---|---|
| Li1—O1 | 2.040 (4) | Li1—N1 | 2.059 (4) |
| Li1—O2 | 2.032 (4) | Li1—N2$^i$ | 2.028 (4) |
| C10—N1—Li1 | 135.78 (17) | C10—N2—Li1$^{ii}$ | 138.55 (18) |

Symmetry codes: (i) $-x + \frac{3}{2}, y + \frac{1}{2}, -z + \frac{1}{2}$; (ii) $-x + \frac{3}{2}, y - \frac{1}{2}, -z + \frac{1}{2}$.

H atoms were placed in idealized positions, with C—H = 0.93–0.97 Å and N—H = 0.86 Å, and refined as riding, with $U_{iso}$(H) = 1.2$U_{eq}$(carrier) or $U_{iso}$(H) = 1.5$U_{eq}$(methyl carrier).

Data collection: SMART (Bruker, 2000); cell refinement: SAINT (Bruker, 2000); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 1997); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: SHELXTL/PC (Sheldrick, 1999); software used to prepare material for publication: SHELXL97.

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