IUCrData

ISSN 2414-3146

Bis[μ-2-(phenylsulfanyl)anilido-κ^2N:N]bis[bis(tetrahydrofuran-κO)lithium]

Velabo Mdluli, James A. Golen and David R. Manke*

Department of Chemistry and Biochemistry, University of Massachusetts Dartmouth, 285 Old Westport Road, North Dartmouth, MA 02747, USA. *Correspondence e-mail: dmanke@umassd.edu

Received 19 January 2016
Accepted 20 January 2016

Edited by M. Weil, Vienna University of Technology, Austria

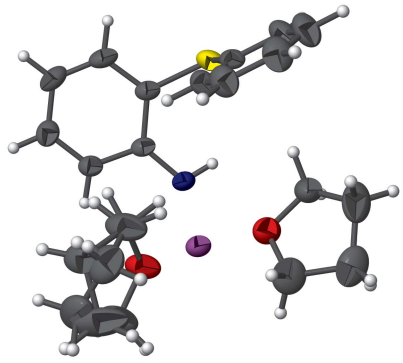
Keywords: crystal structure; lithium anilide; thioaniline.

CCDC reference: 1448763

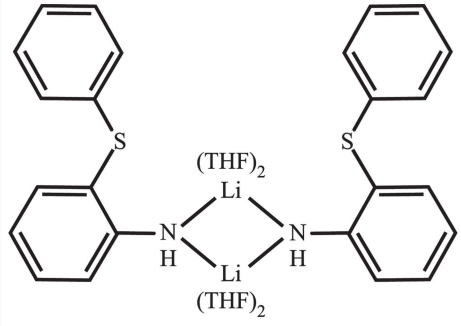
Structural data: full structural data are available from iucrdata.iucr.org

The title compound, $[Li_2(C_{12}H_{10}NS)_2(C_4H_8O)_4]$, exists as a dimer in the solid state, with a central four-membered Li_2N_2 ring that is planar by crystallographic inversion symmetry. The Li atoms are bridged by the N atoms of two anilide ligands, and each Li atom is coordinated by two O atoms from tetrahydrofuran ligands, resulting in a distorted tetrahedral N_2O_2 environment. One of the tetrahydrofuran rings is disordered over two sets of sites in a 0.665 (16):0.335 (6) ratio.

3D view



Chemical scheme



Structure description

Lithium amide complexes can lead to ladder-shaped conformations with solvation of the lithium atom often controlling the oligimerization/polymerization of the lithium complex (Clegg *et al.*, 1995). Herein, we report the crystal structure of the lithium 2-(phenylthio)aniline bis(tetrahydrofuran) dimer. In the crystal, the dimerization results in a centrosymmetric Li_2N_2 ring (Fig. 1), with Li—N—Li^i and N—Li—N^i [symmetry code: (i) $-x, -y+1, -z+1$] angles of 75.65 (10) and 104.35 (13)°, respectively, that are consistent with similar dimers (von Bülow *et al.*, 1996, 2004; Cole *et al.*, 2002). The dihedral angle between the least-squares planes of the Li_2N_2 ring and the aniline aromatic ring is 84.14 (12)°, and the dihedral angle between the two aromatic ring planes in the 2-(phenylthio)anilide is 79.52 (7)°, similar to that observed in the parent 2-(phenylthio)-aniline (Mdluli *et al.*, 2016). No π–π interactions are noted between the aromatic rings. The packing of the molecules in the title compound is shown in Fig. 2.

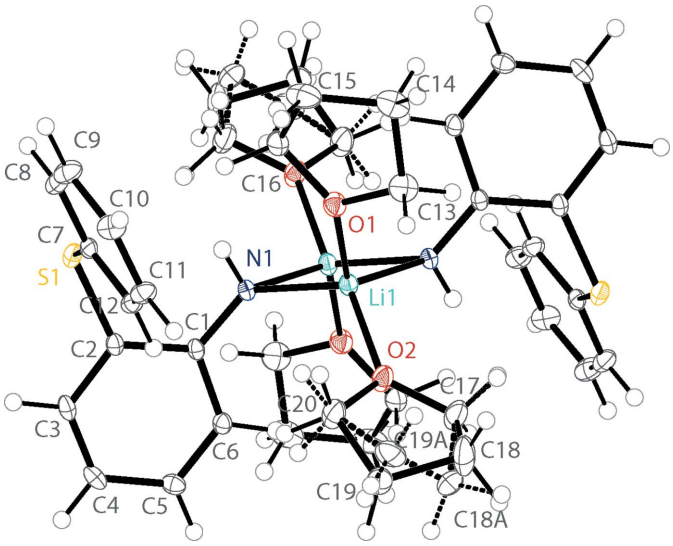


Figure 1
The molecular structure of the title compound, showing the atom-labeling scheme. Displacement ellipsoids are drawn at the 30% probability level; H atoms are drawn as spheres of arbitrary radius. Non-labelled atoms are generated by symmetry code ($-x$, $-y+1$, $-z+1$). Bonds in the minor disorder components of the disordered tetrahydrofuran molecules are drawn as dashed lines.

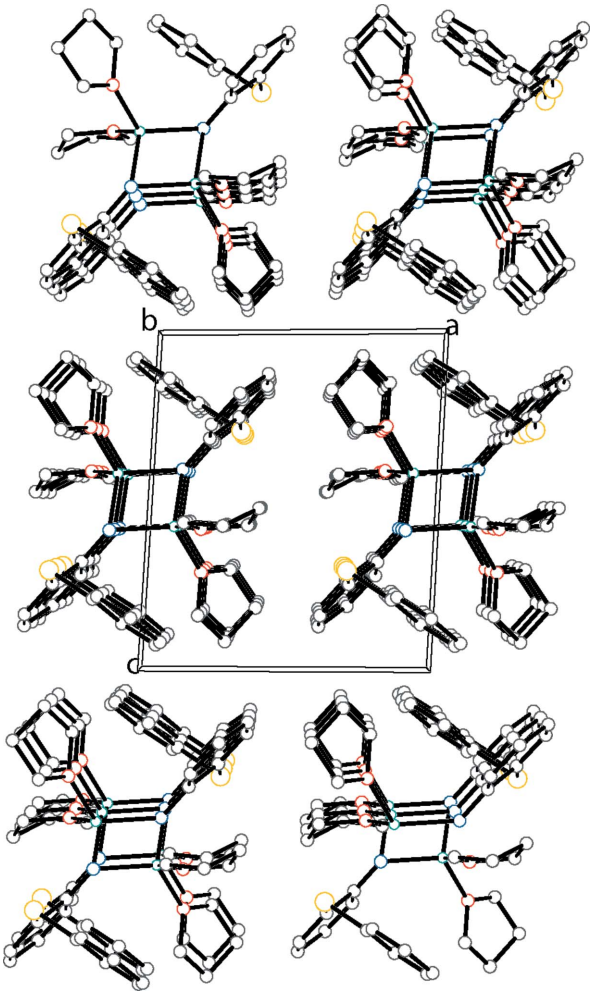


Figure 2
View of the molecular packing of the title compound along the *b* axis. Only the major component of the disordered tetrahydrofuran molecule is shown.

Table 1
Experimental details.

Crystal data	
Chemical formula	$[Li_2(C_{12}H_{10}NS)_2(C_4H_8O)_4]$
M_r	702.83
Crystal system, space group	Triclinic, $P\overline{1}$
Temperature (K)	200
a, b, c (Å)	9.6540 (7), 9.9211 (6), 11.2854 (8)
α, β, γ (°)	73.537 (3), 88.138 (3), 72.420 (3)
V (Å^3)	986.50 (12)
Z	1
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.18
Crystal size (mm)	0.5 × 0.4 × 0.3
Data collection	
Diffractometer	Bruker D8 Venture CMOS
Absorption correction	Multi-scan (*SADABS*; Bruker, 2014)
T_{min}, T_{max}	0.717, 0.745
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	28827, 3607, 3140
R_{int}	0.028
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.604
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.042, 0.111, 1.06
No. of reflections	3607
No. of parameters	249
No. of restraints	2
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.31, −0.29

Computer programs: *APEX2* (Bruker, 2014), *SAINT* (Bruker, 2014), *SHELXS97* (Sheldrick, 2008), *SHELXL2014* (Sheldrick, 2015), *OLEX2* (Dolomanov *et al.*, 2009), *publCIF* (Westrip, 2010).

Synthesis and crystallization

A solution of 2-phenylthioaniline (668 mg, 3.34 mmol) in 3 ml of dry tetrahydrofuran was frozen. Upon melting, 1.41 ml of a 2.5 *M* *n*-butyllithium solution in hexanes (3.53 mmol) was added dropwise. After stirring for 30 minutes at room temperature, 7 ml of dry pentane was added and the solution was stirred for additional 1.5 h. The resulting precipitate was isolated *via* vacuum filtration as a white powder (589 mg, 84% yield). ^{1}H NMR (400 MHz, C_6D_6): δ 7.60 (*d*, *J* = 7.2 Hz, 1 H, Ar-*H*), 7.24 (*d*, *J* = 7.6 Hz, 2 H, Ar-*H*), 7.17 (*dt*, *J* = 7.6 Hz, 1.6 Hz, 1 H, Ar-*H*), 7.14 (*t*, *J* = 7.6 Hz, 2 H, Ar-*H*), 6.77 (*m*, 2H, Ar-*H*), 6.38 (*t*, *J* = 7.2 Hz, 1 H, Ar-*H*), 3.68 (*br s*, 1 H, N*H*), 3.33 (*m*, 8 H, OCH_2), 1.25 (*m*, 8 H, CH_2); ^{13}C NMR (100 MHz, C_6D_6): δ 165.8, 139.9, 138.2, 131.1, 128.9, 127.9, 125.3, 123.9, 117.7, 110.1, 67.5, 24.9. A sample suitable for single-crystal X-ray analysis was grown from pentane/tetrahydrofuran layering.

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 1. One of the tetrahydrofuran ligands (C17–C20) is disordered over two sets of sites with a refined occupancy ratio of 0.665 (6):0.335 (6).

Acknowledgements

We greatly acknowledge support from the National Science Foundation (CHE-1429086).

References

Bruker (2014). *APEX2*, *SAINT* and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
Bülow, R. von, Deuerlein, S., Stey, T., Herbst-Irmer, R., Gornitzka, H. & Stalke, D. (2004). *Z. Naturforsch. Teil B*, **59**, 1471–1479.
Bülow, R. von, Gornitzka, H., Kottke, T. & Stalke, D. (1996). *Chem. Commun.* pp. 1639–1640.
Clegg, W., Horsburgh, L., Mackenzie, F. M. & Mulvey, R. E. (1995). *J. Chem. Soc. Chem. Commun.* pp. 2011–2012.
Cole, M. L., Jones, C. & Junk, P. C. (2002). *New J. Chem.* **26**, 89–93.
Dolomanov, O. V., Bourhis, L. J., Gildea, R. J., Howard, J. A. K. & Puschmann, H. (2009). *J. Appl. Cryst.* **42**, 339–341.
Mdluli, V., Golen, J. A. & Manke, D. R. (2016). *IUCrData*, **1**, x152489.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Sheldrick, G. M. (2015). *Acta Cryst.* C**71**, 3–8.
Westrip, S. P. (2010). *J. Appl. Cryst.* **43**, 920–925.

full crystallographic data

IUCrData (2016). **1**, x160130 [doi:10.1107/S2414314616001309]

Bis[μ-2-(phenylsulfanyl)anilido-κ^2N:N]bis[bis(tetrahydrofuran-κO)lithium]

Velabo Mdluli, James A. Golen and David R. Manke

Bis[μ-2-(phenylsulfanyl)anilido-κ^2N:N]bis[bis(tetrahydrofuran-κO)lithium]

Crystal data

$[Li_2(C_{12}H_{10}NS)_2(C_4H_8O)_4]$
M_r = 702.83
Triclinic, $P\overline{1}$
Hall symbol: -P 1
a = 9.6540 (7) Å
b = 9.9211 (6) Å
c = 11.2854 (8) Å
α = 73.537 (3)°
β = 88.138 (3)°
γ = 72.420 (3)°
V = 986.50 (12) Å^3
Z = 1
$F(000)$ = 376
D_x = 1.183 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 9896 reflections
θ = 2.8–25.4°
μ = 0.18 mm^{-1}
T = 200 K
Block, colourless
0.5 × 0.4 × 0.3 mm

Data collection

Bruker D8 Venture CMOS diffractometer
Radiation source: Mo
TRIUMPH monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2014)
T_{min} = 0.717, T_{max} = 0.745
28827 measured reflections
3607 independent reflections
3140 reflections with $I > 2\sigma(I)$
R_{int} = 0.028
θ_{max} = 25.4°, θ_{min} = 2.8°
h = −11→11
k = −11→11
l = −13→13

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.042
$wR(F^2)$ = 0.111
S = 1.06
3607 reflections
249 parameters
2 restraints
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0453P)^2 + 0.5014P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.31 e Å^{-3}
$\Delta\rho_{min}$ = −0.29 e Å^{-3}

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
S1	−0.30805 (5)	0.81860 (5)	0.70119 (4)	0.04242 (15)	
O2	0.20072 (17)	0.30404 (15)	0.70998 (12)	0.0599 (4)	
O1	0.19444 (14)	0.62852 (15)	0.58373 (13)	0.0495 (3)	
N1	−0.11758 (15)	0.57436 (16)	0.58744 (13)	0.0363 (3)	
C5	−0.2987 (2)	0.3401 (2)	0.80627 (15)	0.0417 (4)	
H5	−0.2982	0.2402	0.8287	0.050*	
C4	−0.3909 (2)	0.4374 (2)	0.86382 (16)	0.0475 (5)	
H4	−0.4536	0.4053	0.9244	0.057*	
C3	−0.38852 (19)	0.5810 (2)	0.83053 (15)	0.0428 (4)	
H3	−0.4499	0.6482	0.8697	0.051*	
C2	−0.29859 (17)	0.63076 (18)	0.74082 (14)	0.0343 (4)	
C7	−0.15499 (19)	0.81895 (18)	0.78363 (14)	0.0364 (4)	
C8	−0.1379 (2)	0.9553 (2)	0.77518 (17)	0.0486 (5)	
H8	−0.2073	1.0430	0.7273	0.058*	
C9	−0.0207 (3)	0.9644 (2)	0.83585 (19)	0.0584 (5)	
H9	−0.0101	1.0583	0.8296	0.070*	
C10	0.0814 (3)	0.8377 (2)	0.90577 (18)	0.0559 (5)	
H10	0.1620	0.8442	0.9474	0.067*	
C6	−0.20865 (18)	0.38618 (18)	0.71778 (14)	0.0358 (4)	
H6	−0.1463	0.3161	0.6818	0.043*	
C1	−0.20482 (16)	0.53452 (17)	0.67779 (13)	0.0305 (3)	
C11	0.0650 (2)	0.7019 (2)	0.91449 (17)	0.0496 (5)	
H11	0.1351	0.6147	0.9622	0.060*	
C12	−0.0523 (2)	0.69133 (19)	0.85455 (15)	0.0409 (4)	
H12	−0.0630	0.5973	0.8617	0.049*	
C13	0.3512 (2)	0.5869 (3)	0.5733 (2)	0.0645 (6)	
H13A	0.3806	0.5253	0.5159	0.077*	
H13B	0.4024	0.5306	0.6552	0.077*	
C16	0.1363 (3)	0.7835 (2)	0.5654 (2)	0.0651 (6)	
H16A	0.0683	0.8051	0.6299	0.078*	
H16B	0.0841	0.8331	0.4831	0.078*	
C15	0.2689 (4)	0.8345 (3)	0.5745 (2)	0.0821 (8)	
H15A	0.2522	0.9379	0.5237	0.098*	
H15B	0.2940	0.8255	0.6613	0.098*	
C14	0.3859 (3)	0.7291 (3)	0.5241 (2)	0.0685 (7)	
H14A	0.3803	0.7608	0.4325	0.082*	
H14B	0.4840	0.7204	0.5548	0.082*	
C19A	0.339 (2)	0.1959 (19)	0.8969 (15)	0.072 (4)	0.335 (16)
H19A	0.4223	0.2352	0.8796	0.086*	0.335 (16)
H19B	0.3322	0.1589	0.9874	0.086*	0.335 (16)
C19	0.2946 (10)	0.1622 (9)	0.9113 (7)	0.0678 (19)	0.665 (16)
H19C	0.2378	0.0942	0.9484	0.081*	0.665 (16)
H19D	0.3514	0.1730	0.9776	0.081*	0.665 (16)
C18A	0.3465 (19)	0.0797 (15)	0.8334 (7)	0.085 (5)	0.335 (16)
H18A	0.4470	0.0124	0.8387	0.102*	0.335 (16)

H18B	0.2806	0.0212	0.8692	0.102*	0.335 (16)
C18	0.3925 (10)	0.1098 (14)	0.8142 (6)	0.106 (4)	0.665 (16)
H18C	0.4769	0.1483	0.8042	0.128*	0.665 (16)
H18D	0.4282	0.0007	0.8367	0.128*	0.665 (16)
C17	0.2979 (3)	0.1703 (3)	0.7028 (2)	0.0780 (8)	
H17A	0.3555	0.1867	0.6289	0.094*	0.665 (16)
H17B	0.2443	0.1015	0.6969	0.094*	0.665 (16)
H17C	0.3821	0.1873	0.6553	0.094*	0.335 (16)
H17D	0.2493	0.1196	0.6616	0.094*	0.335 (16)
C20	0.1985 (3)	0.3084 (3)	0.83512 (19)	0.0763 (8)	
H20A	0.0981	0.3262	0.8631	0.092*	0.665 (16)
H20B	0.2351	0.3888	0.8430	0.092*	0.665 (16)
H20C	0.1136	0.2826	0.8751	0.092*	0.335 (16)
H20D	0.1941	0.4079	0.8394	0.092*	0.335 (16)
Li1	0.1010 (3)	0.4805 (3)	0.5771 (3)	0.0373 (6)	
H1	−0.127 (2)	0.666 (2)	0.5744 (18)	0.046 (5)*	

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0422 (3)	0.0367 (2)	0.0421 (3)	0.00113 (18)	−0.00049 (18)	−0.01541 (18)
O2	0.0736 (10)	0.0496 (8)	0.0369 (7)	0.0096 (7)	0.0010 (6)	−0.0123 (6)
O1	0.0451 (7)	0.0522 (8)	0.0577 (8)	−0.0190 (6)	0.0086 (6)	−0.0220 (6)
N1	0.0379 (8)	0.0371 (8)	0.0343 (7)	−0.0095 (6)	0.0115 (6)	−0.0136 (6)
C5	0.0497 (10)	0.0467 (10)	0.0314 (8)	−0.0207 (8)	0.0013 (7)	−0.0087 (7)
C4	0.0455 (10)	0.0687 (13)	0.0324 (9)	−0.0245 (9)	0.0137 (8)	−0.0148 (8)
C3	0.0375 (9)	0.0577 (11)	0.0330 (9)	−0.0083 (8)	0.0100 (7)	−0.0201 (8)
C2	0.0317 (8)	0.0409 (9)	0.0294 (8)	−0.0058 (7)	0.0028 (6)	−0.0143 (7)
C7	0.0462 (9)	0.0362 (8)	0.0245 (7)	−0.0061 (7)	0.0070 (7)	−0.0127 (6)
C8	0.0678 (13)	0.0328 (9)	0.0402 (10)	−0.0062 (8)	−0.0025 (9)	−0.0119 (7)
C9	0.0858 (16)	0.0410 (10)	0.0524 (12)	−0.0225 (10)	−0.0062 (11)	−0.0153 (9)
C10	0.0712 (14)	0.0565 (12)	0.0444 (11)	−0.0254 (10)	−0.0089 (10)	−0.0134 (9)
C6	0.0387 (9)	0.0396 (9)	0.0300 (8)	−0.0090 (7)	0.0026 (7)	−0.0142 (7)
C1	0.0270 (7)	0.0389 (8)	0.0243 (7)	−0.0054 (6)	0.0004 (6)	−0.0119 (6)
C11	0.0600 (12)	0.0427 (10)	0.0393 (10)	−0.0115 (9)	−0.0085 (8)	−0.0045 (8)
C12	0.0525 (10)	0.0334 (8)	0.0340 (9)	−0.0107 (8)	0.0007 (7)	−0.0080 (7)
C13	0.0451 (11)	0.0752 (15)	0.0701 (14)	−0.0195 (11)	−0.0112 (10)	−0.0131 (12)
C16	0.0808 (16)	0.0516 (12)	0.0715 (14)	−0.0256 (11)	0.0367 (12)	−0.0287 (11)
C15	0.139 (3)	0.0798 (17)	0.0572 (14)	−0.0705 (18)	0.0168 (15)	−0.0269 (13)
C14	0.0582 (13)	0.0924 (18)	0.0600 (13)	−0.0404 (13)	−0.0098 (11)	−0.0094 (12)
C19A	0.081 (9)	0.084 (8)	0.042 (5)	−0.026 (5)	0.002 (5)	−0.003 (5)
C19	0.073 (4)	0.066 (4)	0.040 (3)	0.004 (3)	0.000 (2)	−0.006 (3)
C18A	0.076 (9)	0.047 (5)	0.091 (8)	0.011 (5)	0.016 (6)	0.009 (5)
C18	0.074 (5)	0.126 (7)	0.068 (3)	0.043 (4)	0.000 (3)	−0.028 (3)
C17	0.105 (2)	0.0522 (13)	0.0573 (14)	0.0086 (13)	0.0074 (13)	−0.0215 (11)
C20	0.102 (2)	0.0644 (14)	0.0389 (11)	0.0107 (13)	0.0029 (12)	−0.0180 (10)
Li1	0.0353 (14)	0.0407 (15)	0.0350 (14)	−0.0082 (12)	0.0055 (11)	−0.0136 (11)

Geometric parameters (Å, º)

S1—C2	1.7631 (17)	C13—H13B	0.9900
S1—C7	1.7730 (18)	C13—C14	1.497 (3)
O2—C17	1.396 (2)	C16—H16A	0.9900
O2—C20	1.424 (2)	C16—H16B	0.9900
O2—Li1	1.957 (3)	C16—C15	1.527 (4)
O1—C13	1.453 (2)	C15—H15A	0.9900
O1—C16	1.424 (2)	C15—H15B	0.9900
O1—Li1	1.960 (3)	C15—C14	1.508 (4)
N1—C1	1.350 (2)	C14—H14A	0.9900
N1—Li1	2.047 (3)	C14—H14B	0.9900
N1—Li1^{i}	2.067 (3)	C19A—H19A	0.9900
N1—H1	0.85 (2)	C19A—H19B	0.9900
C5—H5	0.9500	C19A—C18A	1.503 (5)
C5—C4	1.394 (3)	C19A—C20	1.506 (18)
C5—C6	1.374 (2)	C19—H19C	0.9900
C4—H4	0.9500	C19—H19D	0.9900
C4—C3	1.373 (3)	C19—C18	1.523 (9)
C3—H3	0.9500	C19—C20	1.495 (8)
C3—C2	1.393 (2)	C18A—H18A	0.9900
C2—C1	1.435 (2)	C18A—H18B	0.9900
C7—C8	1.389 (2)	C18A—C17	1.496 (5)
C7—C12	1.395 (2)	C18—H18C	0.9900
C8—H8	0.9500	C18—H18D	0.9900
C8—C9	1.379 (3)	C18—C17	1.446 (7)
C9—H9	0.9500	C17—H17A	0.9900
C9—C10	1.383 (3)	C17—H17B	0.9900
C10—H10	0.9500	C17—H17C	0.9900
C10—C11	1.379 (3)	C17—H17D	0.9900
C6—H6	0.9500	C20—H20A	0.9900
C6—C1	1.423 (2)	C20—H20B	0.9900
C11—H11	0.9500	C20—H20C	0.9900
C11—C12	1.384 (3)	C20—H20D	0.9900
C12—H12	0.9500	Li1—N1^{i}	2.067 (3)
C13—H13A	0.9900	Li1—Li1^{i}	2.523 (6)
C2—S1—C7	103.95 (8)	C14—C15—H15B	111.3
C17—O2—C20	109.36 (16)	C13—C14—C15	102.65 (19)
C17—O2—Li1	129.63 (15)	C13—C14—H14A	111.2
C20—O2—Li1	120.28 (14)	C13—C14—H14B	111.2
C13—O1—Li1	116.03 (15)	C15—C14—H14A	111.2
C16—O1—C13	110.43 (16)	C15—C14—H14B	111.2
C16—O1—Li1	131.38 (16)	H14A—C14—H14B	109.1
C1—N1—Li1^{i}	124.66 (14)	H19A—C19A—H19B	109.6
C1—N1—Li1	128.65 (14)	C18A—C19A—H19A	111.9
C1—N1—H1	108.9 (14)	C18A—C19A—H19B	111.9
Li1—N1—Li1^{i}	75.65 (13)	C18A—C19A—C20	99.3 (12)

Li1^{i}—N1—H1	110.8 (14)	C20—C19A—H19A	111.9
Li1—N1—H1	103.8 (14)	C20—C19A—H19B	111.9
C4—C5—H5	119.4	H19C—C19—H19D	109.3
C6—C5—H5	119.4	C18—C19—H19C	111.5
C6—C5—C4	121.22 (17)	C18—C19—H19D	111.5
C5—C4—H4	120.9	C20—C19—H19C	111.5
C3—C4—C5	118.21 (16)	C20—C19—H19D	111.5
C3—C4—H4	120.9	C20—C19—C18	101.3 (6)
C4—C3—H3	119.1	C19A—C18A—H18A	111.3
C4—C3—C2	121.79 (16)	C19A—C18A—H18B	111.3
C2—C3—H3	119.1	H18A—C18A—H18B	109.2
C3—C2—S1	118.23 (13)	C17—C18A—C19A	102.2 (10)
C3—C2—C1	121.48 (16)	C17—C18A—H18A	111.3
C1—C2—S1	120.23 (12)	C17—C18A—H18B	111.3
C8—C7—S1	117.10 (13)	C19—C18—H18C	111.1
C8—C7—C12	119.08 (17)	C19—C18—H18D	111.1
C12—C7—S1	123.82 (13)	H18C—C18—H18D	109.1
C7—C8—H8	119.8	C17—C18—C19	103.4 (6)
C9—C8—C7	120.44 (17)	C17—C18—H18C	111.1
C9—C8—H8	119.8	C17—C18—H18D	111.1
C8—C9—H9	119.8	O2—C17—C18A	106.2 (7)
C8—C9—C10	120.43 (18)	O2—C17—C18	107.1 (4)
C10—C9—H9	119.8	O2—C17—H17A	110.3
C9—C10—H10	120.3	O2—C17—H17B	110.3
C11—C10—C9	119.42 (19)	O2—C17—H17C	110.5
C11—C10—H10	120.3	O2—C17—H17D	110.5
C5—C6—H6	118.6	C18A—C17—H17C	110.5
C5—C6—C1	122.75 (15)	C18A—C17—H17D	110.5
C1—C6—H6	118.6	C18—C17—H17A	110.3
N1—C1—C2	125.42 (15)	C18—C17—H17B	110.3
N1—C1—C6	120.06 (14)	H17A—C17—H17B	108.6
C6—C1—C2	114.52 (14)	H17C—C17—H17D	108.7
C10—C11—H11	119.6	O2—C20—C19A	105.0 (6)
C10—C11—C12	120.72 (18)	O2—C20—C19	107.0 (3)
C12—C11—H11	119.6	O2—C20—H20A	110.3
C7—C12—H12	120.1	O2—C20—H20B	110.3
C11—C12—C7	119.90 (16)	O2—C20—H20C	110.7
C11—C12—H12	120.1	O2—C20—H20D	110.7
O1—C13—H13A	110.6	C19A—C20—H20C	110.7
O1—C13—H13B	110.6	C19A—C20—H20D	110.7
O1—C13—C14	105.51 (18)	C19—C20—H20A	110.3
H13A—C13—H13B	108.8	C19—C20—H20B	110.3
C14—C13—H13A	110.6	H20A—C20—H20B	108.6
C14—C13—H13B	110.6	H20C—C20—H20D	108.8
O1—C16—H16A	110.8	O2—Li1—O1	105.23 (14)
O1—C16—H16B	110.8	O2—Li1—N1	117.69 (14)
O1—C16—C15	104.7 (2)	O2—Li1—N1^{i}	108.46 (14)
H16A—C16—H16B	108.9	O2—Li1—Li1^{i}	129.5 (2)

C15—C16—H16A	110.8	O1—Li1—N1^{i}	114.96 (14)
C15—C16—H16B	110.8	O1—Li1—N1	106.56 (14)
C16—C15—H15A	111.3	O1—Li1—Li1^{i}	125.23 (19)
C16—C15—H15B	111.3	N1—Li1—N1^{i}	104.35 (13)
H15A—C15—H15B	109.2	N1—Li1—Li1^{i}	52.53 (10)
C14—C15—C16	102.42 (17)	N1^{i}—Li1—Li1^{i}	51.82 (10)
C14—C15—H15A	111.3		

S1—C2—C1—N1	0.7 (2)	C12—C7—C8—C9	0.2 (3)
S1—C2—C1—C6	−179.37 (11)	C13—O1—C16—C15	−13.8 (2)
S1—C7—C8—C9	−179.42 (16)	C16—O1—C13—C14	−10.1 (2)
S1—C7—C12—C11	179.10 (14)	C16—C15—C14—C13	−37.5 (2)
O1—C13—C14—C15	29.9 (2)	C19A—C18A—C17—O2	−30.5 (14)
O1—C16—C15—C14	31.9 (2)	C19—C18—C17—O2	32.4 (12)
C5—C4—C3—C2	0.8 (3)	C18A—C19A—C20—O2	−37.9 (13)
C5—C6—C1—N1	−177.75 (15)	C18—C19—C20—O2	22.8 (11)
C5—C6—C1—C2	2.3 (2)	C17—O2—C20—C19A	20.4 (7)
C4—C5—C6—C1	−1.0 (3)	C17—O2—C20—C19	−3.8 (5)
C4—C3—C2—S1	177.98 (14)	C20—O2—C17—C18A	6.4 (7)
C4—C3—C2—C1	0.6 (3)	C20—O2—C17—C18	−18.4 (7)
C3—C2—C1—N1	177.97 (15)	C20—C19A—C18A—C17	40.7 (16)
C3—C2—C1—C6	−2.1 (2)	C20—C19—C18—C17	−33.1 (13)
C2—S1—C7—C8	−176.80 (13)	Li1—O2—C17—C18A	176.5 (7)
C2—S1—C7—C12	3.65 (16)	Li1—O2—C17—C18	151.6 (6)
C7—S1—C2—C3	100.61 (14)	Li1—O2—C20—C19A	−150.7 (7)
C7—S1—C2—C1	−82.00 (14)	Li1—O2—C20—C19	−175.0 (5)
C7—C8—C9—C10	0.1 (3)	Li1—O1—C13—C14	155.11 (17)
C8—C7—C12—C11	−0.4 (3)	Li1—O1—C16—C15	−176.02 (17)
C8—C9—C10—C11	−0.1 (3)	Li1^{i}—N1—C1—C2	−133.47 (17)
C9—C10—C11—C12	−0.2 (3)	Li1—N1—C1—C2	126.91 (18)
C10—C11—C12—C7	0.5 (3)	Li1^{i}—N1—C1—C6	46.6 (2)
C6—C5—C4—C3	−0.6 (3)	Li1—N1—C1—C6	−53.1 (2)

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