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# 1,3-Di-1-adamantylimidazolium (phthalocyaninato)lithium(I) acetone hemisolvate monohydrate

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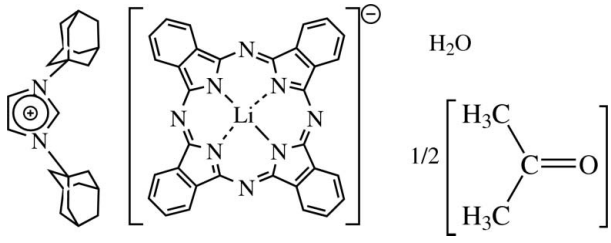
Key indicators: single-crystal X-ray study; $T$ = 173 K; mean $\sigma$(C–C) = 0.002 Å; disorder in solvent or counterion; $R$ factor = 0.052; $wR$ factor = 0.129; data-to-parameter ratio = 23.0.

The asymmetric unit of the title compound, $(C_{23}H_{33}N_2)$-$[Li(C_{32}H_{16}N_8)]\cdot 0.5C_3H_6O\cdot H_2O$, consists of two symmetry-unrelated lithium phthalocyanine ($LiPc^-$) half-anions, centered at (1,0,0) and (0,$\frac{1}{2}$,0), respectively, the bis(adamantyl)imidazolium cation ($BAI^+$), occupying a general site, an acetone molecule, disordered about the inversion centre at (0, $\frac{1}{2}$, $\frac{1}{2}$) and a water molecule at a general site. The $LiPc^-$ anions pack in a stepped pattern enclosing the bis(adamantyl)imidazolium cation. Attractions between the anion and cation are mediated by a water molecule which forms O—H···N hydrogen bonds. In addition, two C—H···O interactions are seen.

## Related literature

Similar compounds utilizing nitrogen-based cations have been reported by Homborg & Kalz (1978*a*,*b*). For related structures see: Grossie *et al.* (2006).



## Experimental

### Crystal data

$(C_{23}H_{33}N_2)[Li(C_{32}H_{16}N_8)]\cdot 0.5C_3H_6O\cdot H_2O$
$M_r$ = 904.04
Monoclinic, $P2_1/n$
$a$ = 15.799 (3) Å
$b$ = 17.165 (4) Å
$c$ = 17.831 (4) Å
$\beta$ = 108.374 (3)°
$V$ = 4588.9 (16) Å$^3$
$Z$ = 4
Mo $K\alpha$ radiation
$\mu$ = 0.08 mm$^{-1}$
$T$ = 173 (2) K
0.44 × 0.39 × 0.20 mm

### Data collection

Bruker Smart APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2003)
$T_{min}$ = 0.901, $T_{max}$ = 0.985
54676 measured reflections
14917 independent reflections
11038 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.039

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.052
$wR(F^2)$ = 0.129
$S$ = 1.03
14917 reflections
649 parameters
8 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.37 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.30 e Å$^{-3}$

**Table 1**
Hydrogen-bond geometry (Å, °).

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| O1—H1$A$···N6 | 0.861 (18) | 2.405 (17) | 3.1618 (17) | 147.1 (18) |
| O1—H1$B$···N3$^{i}$ | 0.889 (18) | 2.025 (19) | 2.8911 (17) | 164.3 (16) |
| C33—H33···O1 | 0.95 | 2.18 | 3.127 (2) | 171 |
| C37—H37$A$···O1 | 0.99 | 2.52 | 3.493 (2) | 166 |

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

Data collection: *SMART* (Bruker, 2002); cell refinement: *SAINT-Plus* (Bruker, 2003); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *Mercury* (Macrae *et al.*, 2006), *ORTEP-3 for Windows*, (Farrugia, 1997), *OSCAIL*, (McArdle, 1995); software used to prepare material for publication: *enCIFer* (Allen *et al.*, 2004) and *publCIF* (Westrip, 2009).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SJ2558).

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# supplementary materials

*Acta Cryst.* (2009). E**65**, m72 [ **doi:10.1107/S1600536808041135** ]

# 1,3-Di-1-adamantylimidazolium (phthalocyaninato)lithium(I) acetone hemisolvate monohydrate

**D. A. Grossie, W. A. Feld and J. Kelley**

## Comment

The asymmetric unit of the title compound consists of two symmetry-unrelated halves of lithium phthalocyanine anions ($LiPc^-$), centered at 1,0,0 and 0, 1/2,0, respectively; the bis(adamantyl)imidazolium cation ($BAI^+$), occupying a general site, an acetone molecule, at 0, 1/2,0.5, and a water molecule at a general site, Fig 1. In addition, the unit cell packing provides interesting detail of the arrangement of molecules within the crystal structure, as seen in Figure 2. Similar compounds utilizing nitrogen-based cations have been reported by Homborg & Kalz (1978a, 1978b).

Although they may appear to be in Figure 2, the symmetry-related $LiPc^-$ anions are not parallel, as seen in the angle (2.39°) between their mean planes. Additionally, their intermolecular distances are quite large (10.10 Å as measured between mesonitrogens) in comparison to those seen in $Li_2PC$ (3.06–3.38 Å)(Grossie, *et al.*, 2006) The large spacing between $LiPc^-$ molecules is easily attributed to the bulky adamantyl substituents of the $BAI^+$ molecules, in which one cation appears to be enclosed within four $LiPc^-$ anions forming ionic pockets. This is shown in Fig. 2, which presents the organized but unique packing of molecules. Rows of symmetry-related anions along the *b* axis are offset from each other, stacking in a stair-step manner. These molecules appear to be nearly orthogonal to the columnar anions, in which alternating columns have slightly different orientations. It is necessary, though, to view the packing from all angles to get a true understanding of ion arrangements.

Probably the most intriguing information obtained was the role of solvent molecules within the crystal structure. It was seen in the crystal structure of $Li_2PC$ that acetone and water ligated to lithium, forming dimers that were found in between $LiPc^-$ pairs. In the current structure it can be seen that water molecules are crucial to the crystallization of the complex ions. Here, it is noticed that water forms O—H···N and C—H···O hydrogen bonds, Table 1, with the two symmetry-unrelated $LiPc^-$ anions and one $BAI^+$ cation, acting as an intermediate to the three ions. Interatomic distances between hydrogen atoms and isoindoline and *meso* nitrogen of individual $LiPc^-$ ions were found to be 2.405 (17) Å and 2.025 (19) Å, respectively. The distance between the oxygen atom of water and the hydrogen on the 2- position of the $BAI^+$ cation was calculated to be about 2.18 Å.

## Experimental

The 1,3-bis(1-adamantyl)imidazolium tetrafluoroborate (0.884 g) was purified by dissolving it in 70 ml of acetone and filtering the insoluble impurities. The solution was evaporated to dryness to give 0.843 g (1.98 mmol) of the pure salt, which was redissolved in 10 ml of acetone and added to a solution of 0.991 g (1.98 mmol) of dilithium phthalocyanine in 100 ml of acetone. The solution was evaporated to approximately 20–30 ml under reduced pressure to the point of crystallization, sealed and crystallized at 5°C for 72 h. The resulting solid was redissolved in 125 ml of hot acetone with stirring (some undissolved solid remained). The volume was reduced and crystallized at 5°C. The dry product isolated by filtration gave

0.893 g (55.3%) of purple crystals. m.p. 349–351°C. Anal. Calc. for C55H49LiN10 (856.99): C, 77.08; H, 5.76; N, 16.34. Found: C, 76.89; H, 5.90; N, 15.94.

### Refinement

Hydrogen atoms of the water molecule and of the methyl group of the acetone molecule were located in a difference Fourier map and refined with appropriate distance restraints. All other H-atoms were positioned geometrically and refined using a riding model with d(C-H) = 0.95Å, $U_{iso}$=1.2$U_{eq}$ (C) for aromatic 1.00Å, $U_{iso}$ = 1.2$U_{eq}$ (C) for CH and 0.99Å, $U_{iso}$ = 1.2$U_{eq}$ (C) for $CH_2$ atoms

### Figures

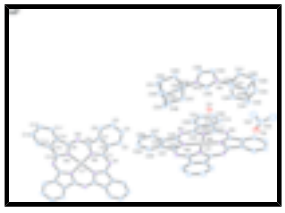

Fig. 1. The asymmetric unit of I. Labelled atoms are related to unlabelled atoms by the symmetry codes –x+2, -y, -z for the Li1 anion and -x, -y+1, -z for the Li2 anion. Hydrogen atoms are not shown.

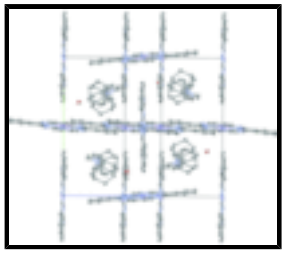

Fig. 2. Unit cell packing diagram, viewed along the *b* axis. Acetone molecules have been omitted for clarity.

### 1,3-Bis(1-adamantyl)imidazolium lithium phthalocyanine monohydrate acetone hemisolvate.

*Crystal data*

| | |
|---|---|
| $(C_{23}H_{33}N_2)[Li(C_{32}H_{16}N_8)]\cdot 0.5C_3H_6O\cdot H_2O$ | $F_{000}$ = 1912 |
| $M_r$ = 904.04 | $D_x$ = 1.309 Mg m$^{-3}$ |
| Monoclinic, $P2_1/n$ | Melting point: 622 K |
| Hall symbol: -P 2yn | Mo $K\alpha$ radiation<br>λ = 0.71073 Å |
| $a$ = 15.799 (3) Å | Cell parameters from 64888 reflections |
| $b$ = 17.165 (4) Å | θ = 2.4–30.1° |
| $c$ = 17.831 (4) Å | μ = 0.08 mm$^{-1}$ |
| β = 108.374 (3)° | $T$ = 173 (2) K |
| $V$ = 4588.9 (16) Å$^3$ | Block, violet |
| $Z$ = 4 | 0.44 × 0.39 × 0.20 mm |

*Data collection*

| | |
|---|---|
| Bruker Smart APEXII CCD diffractometer | 14917 independent reflections |

| | |
|---|---|
| Radiation source: fine-focus sealed tube | 11038 reflections with $I > 2\sigma(I)$ |
| Monochromator: graphite | $R_{int} = 0.039$ |
| $T = 173(2)$ K | $\theta_{max} = 31.7^{o}$ |
| ω scans | $\theta_{min} = 1.7^{o}$ |
| Absorption correction: multi-scan (SADABS; Bruker, 2003) | $h = -22 \rightarrow 23$ |
| $T_{min} = 0.901$, $T_{max} = 0.985$ | $k = -25 \rightarrow 24$ |
| 54676 measured reflections | $l = -26 \rightarrow 26$ |

*Refinement*

| | |
|---|---|
| Refinement on $F^2$ | Secondary atom site location: difference Fourier map |
| Least-squares matrix: full | Hydrogen site location: inferred from neighbouring sites |
| $R[F^2 > 2\sigma(F^2)] = 0.052$ | H atoms treated by a mixture of independent and constrained refinement |
| $wR(F^2) = 0.129$ | $w = 1/[\sigma^2(F_o^2) + (0.0482P)^2 + 2.081P]$ where $P = (F_o^2 + 2F_c^2)/3$ |
| $S = 1.03$ | $(\Delta/\sigma)_{max} = 0.001$ |
| 14917 reflections | $\Delta\rho_{max} = 0.37$ e Å$^{-3}$ |
| 649 parameters | $\Delta\rho_{min} = -0.29$ e Å$^{-3}$ |
| 8 restraints | Extinction correction: none |
| Primary atom site location: structure-invariant direct methods | |

*Special details*

**Experimental**. 1H NMR (300 MHz, DMSO-d6) δ = 9.35–9.25 (m, 8H, Ar—H), 9.06 (s, 1H, Ar—H), 8.12–8.02 (m, 8H, Ar—H), 8.01 (d, 2H, 3 J = 1.1 Hz, Ar—H), 2.24–2.09 (m, 18H, Al—H), 1.82–1.62 (m, 12H, Al—H); 13 C-NMR (75 MHz, DMSO-d6) δ = 154.14, 140.05, 131.23, 127.51, 119.31, 59.35, 41.46, 34.81, 28.83; IR (KBr) cm$^{-1}$ = 3053 (Ar—H), 2912 (C—H), 1604 (C—C), 1583 (C—N), 1485 (C—N), 1092 (C—C), 1055 (C—N); UV/Vis (DMSO) λmax nm (log ε) = 665 (5.25), 636 (4.47), 601 (4.48), 380 (4.55), 327 (4.55), 255 (4.63);

**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.

**Refinement**. Refinement of $F^2$ against ALL reflections. The weighted *R*-factor *wR* and goodness of fit *S* are based on $F^2$, conventional *R*-factors *R* are based on *F*, with *F* set to zero for negative $F^2$. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on $F^2$ are statistically about twice as large as those based on *F*, and *R*- factors based on ALL data will be even larger.

*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å$^2$)*

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ | Occ. (<1) |
|---|---|---|---|---|---|
| Li1 | 1.0000 | 0.0000 | 0.0000 | 0.0221 (7) | |
| N1 | 0.84511 (7) | 0.01780 (6) | 0.09200 (6) | 0.0183 (2) | |
| N2 | 0.94090 (7) | 0.08811 (6) | 0.03257 (6) | 0.0173 (2) | |

| | | | | |
|---|---|---|---|---|
| N3 | 1.01005 (7) | 0.19613 (6) | −0.01323 (6) | 0.0182 (2) |
| N4 | 1.06695 (7) | 0.07380 (6) | −0.04398 (6) | 0.0178 (2) |
| C1 | 0.87993 (8) | 0.08236 (7) | 0.07194 (7) | 0.0167 (2) |
| C2 | 0.85314 (8) | 0.15984 (7) | 0.08970 (7) | 0.0173 (2) |
| C3 | 0.79734 (9) | 0.18458 (8) | 0.13159 (8) | 0.0213 (3) |
| H3 | 0.7654 | 0.1483 | 0.1526 | 0.026* |
| C4 | 0.78994 (10) | 0.26408 (8) | 0.14163 (8) | 0.0240 (3) |
| H4 | 0.7524 | 0.2828 | 0.1701 | 0.029* |
| C5 | 0.83698 (10) | 0.31696 (8) | 0.11036 (9) | 0.0256 (3) |
| H5 | 0.8310 | 0.3711 | 0.1183 | 0.031* |
| C6 | 0.89232 (9) | 0.29251 (8) | 0.06800 (8) | 0.0222 (3) |
| H6 | 0.9237 | 0.3290 | 0.0466 | 0.027* |
| C7 | 0.90028 (9) | 0.21275 (7) | 0.05796 (7) | 0.0179 (2) |
| C8 | 0.95462 (8) | 0.16503 (7) | 0.02226 (7) | 0.0173 (2) |
| C9 | 1.06334 (8) | 0.15280 (7) | −0.04204 (7) | 0.0172 (2) |
| C10 | 1.12886 (8) | 0.18684 (7) | −0.07575 (7) | 0.0174 (2) |
| C11 | 1.15259 (9) | 0.26268 (8) | −0.08836 (8) | 0.0201 (2) |
| H11 | 1.1230 | 0.3062 | −0.0750 | 0.024* |
| C12 | 1.22089 (9) | 0.27270 (8) | −0.12104 (8) | 0.0221 (3) |
| H12 | 1.2382 | 0.3240 | −0.1301 | 0.026* |
| C13 | 1.26472 (9) | 0.20899 (8) | −0.14097 (8) | 0.0228 (3) |
| H13 | 1.3115 | 0.2177 | −0.1629 | 0.027* |
| C14 | 1.24094 (9) | 0.13324 (8) | −0.12920 (8) | 0.0213 (3) |
| H14 | 1.2704 | 0.0898 | −0.1428 | 0.026* |
| C15 | 1.17233 (9) | 0.12325 (7) | −0.09671 (8) | 0.0181 (2) |
| C16 | 1.13064 (9) | 0.05364 (7) | −0.07705 (7) | 0.0177 (2) |
| Li2 | 0.0000 | 0.5000 | 0.0000 | 0.0223 (7) |
| N5 | −0.00519 (7) | 0.50482 (6) | 0.18826 (6) | 0.0193 (2) |
| N6 | 0.08896 (7) | 0.49153 (7) | 0.10472 (6) | 0.0192 (2) |
| N7 | 0.22215 (7) | 0.47223 (6) | 0.06714 (7) | 0.0195 (2) |
| N8 | 0.09266 (7) | 0.48310 (7) | −0.04998 (6) | 0.0187 (2) |
| C17 | 0.07282 (9) | 0.49409 (7) | 0.17554 (7) | 0.0183 (2) |
| C18 | 0.15585 (9) | 0.48247 (7) | 0.24040 (8) | 0.0187 (2) |
| C19 | 0.17516 (10) | 0.47967 (8) | 0.32180 (8) | 0.0229 (3) |
| H19 | 0.1297 | 0.4863 | 0.3457 | 0.027* |
| C20 | 0.26314 (10) | 0.46682 (9) | 0.36727 (8) | 0.0279 (3) |
| H20 | 0.2782 | 0.4649 | 0.4232 | 0.033* |
| C21 | 0.32991 (10) | 0.45674 (9) | 0.33197 (9) | 0.0286 (3) |
| H21 | 0.3895 | 0.4475 | 0.3643 | 0.034* |
| C22 | 0.31062 (9) | 0.45991 (8) | 0.25046 (8) | 0.0237 (3) |
| H22 | 0.3561 | 0.4534 | 0.2266 | 0.028* |
| C23 | 0.22251 (9) | 0.47299 (8) | 0.20492 (8) | 0.0196 (2) |
| C24 | 0.17785 (9) | 0.47881 (7) | 0.11946 (8) | 0.0187 (2) |
| C25 | 0.18188 (9) | 0.47596 (7) | −0.01094 (8) | 0.0183 (2) |
| C26 | 0.23144 (9) | 0.47344 (7) | −0.06777 (8) | 0.0183 (2) |
| C27 | 0.32210 (9) | 0.47045 (8) | −0.05927 (8) | 0.0213 (3) |
| H27 | 0.3657 | 0.4665 | −0.0086 | 0.026* |
| C28 | 0.34626 (10) | 0.47343 (8) | −0.12769 (9) | 0.0251 (3) |
| H28 | 0.4077 | 0.4719 | −0.1235 | 0.030* |

| | | | | |
|---|---|---|---|---|
| C29 | 0.28251 (10) | 0.47858 (9) | −0.20269 (9) | 0.0255 (3) |
| H29 | 0.3013 | 0.4799 | −0.2483 | 0.031* |
| C30 | 0.19217 (10) | 0.48184 (8) | −0.21123 (8) | 0.0224 (3) |
| H30 | 0.1487 | 0.4857 | −0.2619 | 0.027* |
| C31 | 0.16757 (9) | 0.47920 (7) | −0.14276 (8) | 0.0185 (2) |
| C32 | 0.08121 (9) | 0.48562 (7) | −0.12921 (8) | 0.0179 (2) |
| N9 | 0.21347 (8) | 0.75837 (7) | 0.32337 (7) | 0.0206 (2) |
| N10 | 0.31358 (8) | 0.73900 (7) | 0.26556 (7) | 0.0210 (2) |
| C33 | 0.22743 (9) | 0.73102 (8) | 0.25812 (8) | 0.0212 (3) |
| H33 | 0.1832 | 0.7094 | 0.2137 | 0.025* |
| C34 | 0.29366 (9) | 0.78481 (9) | 0.37394 (8) | 0.0238 (3) |
| H34 | 0.3033 | 0.8071 | 0.4247 | 0.029* |
| C35 | 0.35583 (9) | 0.77306 (9) | 0.33786 (8) | 0.0245 (3) |
| H35 | 0.4173 | 0.7859 | 0.3584 | 0.029* |
| C36 | 0.12688 (9) | 0.75260 (8) | 0.33990 (8) | 0.0191 (2) |
| C37 | 0.05180 (9) | 0.78827 (8) | 0.27195 (8) | 0.0205 (3) |
| H37A | 0.0471 | 0.7607 | 0.2220 | 0.025* |
| H37B | 0.0649 | 0.8438 | 0.2654 | 0.025* |
| C38 | −0.03623 (9) | 0.78110 (8) | 0.29063 (8) | 0.0222 (3) |
| H38 | −0.0855 | 0.8046 | 0.2466 | 0.027* |
| C39 | −0.02848 (10) | 0.82412 (9) | 0.36785 (9) | 0.0258 (3) |
| H39A | −0.0155 | 0.8799 | 0.3624 | 0.031* |
| H39B | −0.0856 | 0.8204 | 0.3796 | 0.031* |
| C40 | 0.04642 (10) | 0.78776 (9) | 0.43521 (8) | 0.0262 (3) |
| H40 | 0.0512 | 0.8156 | 0.4856 | 0.031* |
| C41 | 0.13491 (9) | 0.79498 (9) | 0.41737 (8) | 0.0245 (3) |
| H41A | 0.1491 | 0.8506 | 0.4127 | 0.029* |
| H41B | 0.1837 | 0.7717 | 0.4610 | 0.029* |
| C42 | −0.05610 (10) | 0.69499 (9) | 0.29889 (9) | 0.0252 (3) |
| H42A | −0.0612 | 0.6673 | 0.2489 | 0.030* |
| H42B | −0.1135 | 0.6896 | 0.3098 | 0.030* |
| C43 | 0.01881 (10) | 0.65890 (8) | 0.36645 (9) | 0.0263 (3) |
| H43 | 0.0055 | 0.6026 | 0.3719 | 0.032* |
| C44 | 0.02643 (11) | 0.70154 (10) | 0.44379 (9) | 0.0300 (3) |
| H44A | −0.0301 | 0.6963 | 0.4563 | 0.036* |
| H44B | 0.0748 | 0.6781 | 0.4876 | 0.036* |
| C45 | 0.10709 (10) | 0.66639 (8) | 0.34806 (9) | 0.0242 (3) |
| H45A | 0.1560 | 0.6426 | 0.3912 | 0.029* |
| H45B | 0.1027 | 0.6386 | 0.2983 | 0.029* |
| C46 | 0.35926 (9) | 0.71388 (8) | 0.20826 (7) | 0.0188 (2) |
| C47 | 0.29214 (9) | 0.67521 (8) | 0.13680 (8) | 0.0218 (3) |
| H47A | 0.2441 | 0.7124 | 0.1105 | 0.026* |
| H47B | 0.2648 | 0.6295 | 0.1540 | 0.026* |
| C48 | 0.34101 (9) | 0.64948 (9) | 0.07899 (8) | 0.0240 (3) |
| H48 | 0.2977 | 0.6241 | 0.0319 | 0.029* |
| C49 | 0.38222 (11) | 0.72049 (10) | 0.05245 (9) | 0.0301 (3) |
| H49A | 0.3349 | 0.7583 | 0.0262 | 0.036* |
| H49B | 0.4125 | 0.7044 | 0.0140 | 0.036* |
| C50 | 0.44974 (11) | 0.75862 (9) | 0.12455 (9) | 0.0294 (3) |

| | | | | | |
|---|---|---|---|---|---|
| H50 | 0.4773 | 0.8048 | 0.1071 | 0.035* | |
| C51 | 0.52255 (10) | 0.70054 (10) | 0.16613 (9) | 0.0302 (3) | |
| H51A | 0.5655 | 0.7255 | 0.2128 | 0.036* | |
| H51B | 0.5553 | 0.6842 | 0.1297 | 0.036* | |
| C52 | 0.48106 (9) | 0.62939 (9) | 0.19228 (8) | 0.0243 (3) | |
| H52 | 0.5289 | 0.5912 | 0.2189 | 0.029* | |
| C53 | 0.43204 (9) | 0.65519 (8) | 0.24972 (8) | 0.0221 (3) | |
| H53A | 0.4747 | 0.6795 | 0.2970 | 0.027* | |
| H53B | 0.4049 | 0.6094 | 0.2671 | 0.027* | |
| C54 | 0.41484 (9) | 0.59165 (9) | 0.11957 (9) | 0.0254 (3) | |
| H54A | 0.4463 | 0.5752 | 0.0822 | 0.030* | |
| H54B | 0.3884 | 0.5448 | 0.1357 | 0.030* | |
| C55 | 0.40064 (10) | 0.78504 (8) | 0.18204 (9) | 0.0266 (3) | |
| H55A | 0.4430 | 0.8107 | 0.2286 | 0.032* | |
| H55B | 0.3534 | 0.8230 | 0.1557 | 0.032* | |
| O1 | 0.06786 (8) | 0.67405 (6) | 0.11486 (7) | 0.0297 (2) | |
| H1A | 0.0548 (13) | 0.6284 (10) | 0.0945 (12) | 0.045* | |
| H1B | 0.0393 (13) | 0.7070 (11) | 0.0768 (10) | 0.045* | |
| O2 | −0.0403 (2) | 0.44760 (18) | 0.39840 (18) | 0.0555 (7) | 0.50 |
| C56 | −0.0162 (3) | 0.4789 (2) | 0.4612 (2) | 0.0451 (9) | 0.50 |
| C57 | 0.0801 (2) | 0.47921 (16) | 0.50835 (16) | 0.0646 (7) | |
| H57A | 0.1107 (14) | 0.4355 (14) | 0.4973 (17) | 0.097* | |
| H57B | 0.0942 (15) | 0.4617 (18) | 0.5603 (12) | 0.097* | |
| H57C | 0.1131 (14) | 0.5205 (14) | 0.4976 (16) | 0.097* | |

*Atomic displacement parameters (Å$^2$)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| Li1 | 0.0250 (17) | 0.0152 (14) | 0.0268 (17) | −0.0003 (12) | 0.0094 (14) | 0.0006 (12) |
| N1 | 0.0193 (5) | 0.0163 (5) | 0.0190 (5) | −0.0001 (4) | 0.0058 (4) | 0.0004 (4) |
| N2 | 0.0191 (5) | 0.0153 (5) | 0.0171 (5) | −0.0011 (4) | 0.0052 (4) | 0.0003 (4) |
| N3 | 0.0199 (5) | 0.0168 (5) | 0.0177 (5) | 0.0000 (4) | 0.0056 (4) | 0.0007 (4) |
| N4 | 0.0191 (5) | 0.0151 (5) | 0.0194 (5) | −0.0001 (4) | 0.0064 (4) | 0.0010 (4) |
| C1 | 0.0175 (6) | 0.0162 (5) | 0.0154 (5) | 0.0004 (4) | 0.0038 (4) | −0.0011 (4) |
| C2 | 0.0184 (6) | 0.0161 (5) | 0.0162 (6) | 0.0010 (4) | 0.0036 (5) | −0.0009 (4) |
| C3 | 0.0215 (6) | 0.0214 (6) | 0.0214 (6) | 0.0017 (5) | 0.0073 (5) | 0.0006 (5) |
| C4 | 0.0251 (7) | 0.0235 (6) | 0.0239 (7) | 0.0061 (5) | 0.0085 (5) | −0.0022 (5) |
| C5 | 0.0304 (7) | 0.0180 (6) | 0.0272 (7) | 0.0051 (5) | 0.0075 (6) | −0.0026 (5) |
| C6 | 0.0248 (7) | 0.0162 (6) | 0.0245 (7) | 0.0011 (5) | 0.0060 (5) | −0.0001 (5) |
| C7 | 0.0191 (6) | 0.0163 (5) | 0.0168 (6) | 0.0019 (4) | 0.0035 (5) | −0.0001 (4) |
| C8 | 0.0182 (6) | 0.0156 (5) | 0.0165 (6) | 0.0004 (4) | 0.0033 (5) | −0.0001 (4) |
| C9 | 0.0183 (6) | 0.0152 (5) | 0.0166 (6) | −0.0016 (4) | 0.0034 (5) | 0.0001 (4) |
| C10 | 0.0185 (6) | 0.0168 (5) | 0.0156 (5) | −0.0022 (4) | 0.0039 (4) | 0.0005 (4) |
| C11 | 0.0231 (6) | 0.0170 (6) | 0.0189 (6) | −0.0031 (5) | 0.0049 (5) | −0.0008 (5) |
| C12 | 0.0242 (7) | 0.0197 (6) | 0.0213 (6) | −0.0064 (5) | 0.0056 (5) | 0.0008 (5) |
| C13 | 0.0215 (6) | 0.0247 (6) | 0.0231 (6) | −0.0061 (5) | 0.0083 (5) | 0.0003 (5) |
| C14 | 0.0210 (6) | 0.0210 (6) | 0.0225 (6) | −0.0013 (5) | 0.0075 (5) | −0.0006 (5) |
| C15 | 0.0192 (6) | 0.0172 (5) | 0.0175 (6) | −0.0016 (5) | 0.0054 (5) | 0.0010 (4) |

| | | | | | | |
|---|---|---|---|---|---|---|
| C16 | 0.0187 (6) | 0.0165 (5) | 0.0171 (6) | −0.0011 (4) | 0.0046 (5) | 0.0001 (4) |
| Li2 | 0.0212 (16) | 0.0281 (17) | 0.0162 (15) | 0.0012 (13) | 0.0040 (12) | −0.0005 (13) |
| N5 | 0.0207 (5) | 0.0195 (5) | 0.0172 (5) | 0.0008 (4) | 0.0052 (4) | 0.0001 (4) |
| N6 | 0.0185 (5) | 0.0233 (5) | 0.0155 (5) | 0.0016 (4) | 0.0047 (4) | −0.0006 (4) |
| N7 | 0.0193 (5) | 0.0208 (5) | 0.0176 (5) | 0.0020 (4) | 0.0046 (4) | −0.0005 (4) |
| N8 | 0.0179 (5) | 0.0215 (5) | 0.0160 (5) | 0.0010 (4) | 0.0043 (4) | 0.0004 (4) |
| C17 | 0.0192 (6) | 0.0184 (6) | 0.0164 (6) | 0.0002 (5) | 0.0042 (5) | −0.0002 (5) |
| C18 | 0.0202 (6) | 0.0168 (5) | 0.0175 (6) | 0.0008 (5) | 0.0036 (5) | 0.0003 (5) |
| C19 | 0.0276 (7) | 0.0225 (6) | 0.0169 (6) | 0.0025 (5) | 0.0047 (5) | −0.0004 (5) |
| C20 | 0.0320 (8) | 0.0305 (7) | 0.0163 (6) | 0.0040 (6) | 0.0004 (6) | 0.0013 (5) |
| C21 | 0.0241 (7) | 0.0334 (8) | 0.0218 (7) | 0.0057 (6) | −0.0022 (5) | 0.0019 (6) |
| C22 | 0.0205 (6) | 0.0263 (7) | 0.0217 (7) | 0.0042 (5) | 0.0028 (5) | 0.0007 (5) |
| C23 | 0.0213 (6) | 0.0183 (6) | 0.0171 (6) | 0.0021 (5) | 0.0027 (5) | −0.0007 (5) |
| C24 | 0.0184 (6) | 0.0190 (6) | 0.0174 (6) | 0.0009 (5) | 0.0038 (5) | −0.0001 (5) |
| C25 | 0.0180 (6) | 0.0181 (6) | 0.0185 (6) | 0.0016 (5) | 0.0052 (5) | −0.0010 (5) |
| C26 | 0.0204 (6) | 0.0155 (5) | 0.0194 (6) | 0.0007 (5) | 0.0068 (5) | −0.0015 (4) |
| C27 | 0.0199 (6) | 0.0213 (6) | 0.0224 (6) | 0.0007 (5) | 0.0063 (5) | −0.0025 (5) |
| C28 | 0.0218 (7) | 0.0266 (7) | 0.0296 (7) | 0.0014 (5) | 0.0122 (6) | −0.0024 (6) |
| C29 | 0.0284 (7) | 0.0276 (7) | 0.0248 (7) | 0.0010 (6) | 0.0143 (6) | −0.0002 (5) |
| C30 | 0.0262 (7) | 0.0221 (6) | 0.0201 (6) | 0.0012 (5) | 0.0089 (5) | −0.0007 (5) |
| C31 | 0.0205 (6) | 0.0154 (5) | 0.0203 (6) | 0.0012 (5) | 0.0074 (5) | −0.0010 (5) |
| C32 | 0.0198 (6) | 0.0169 (5) | 0.0175 (6) | 0.0002 (5) | 0.0065 (5) | −0.0010 (4) |
| N9 | 0.0190 (5) | 0.0252 (6) | 0.0181 (5) | 0.0013 (4) | 0.0064 (4) | −0.0031 (4) |
| N10 | 0.0189 (5) | 0.0261 (6) | 0.0180 (5) | 0.0018 (4) | 0.0059 (4) | −0.0028 (4) |
| C33 | 0.0185 (6) | 0.0275 (7) | 0.0173 (6) | 0.0018 (5) | 0.0054 (5) | −0.0022 (5) |
| C34 | 0.0203 (6) | 0.0303 (7) | 0.0196 (6) | 0.0003 (5) | 0.0048 (5) | −0.0059 (5) |
| C35 | 0.0198 (6) | 0.0318 (7) | 0.0206 (6) | −0.0002 (5) | 0.0047 (5) | −0.0063 (5) |
| C36 | 0.0190 (6) | 0.0223 (6) | 0.0175 (6) | 0.0013 (5) | 0.0078 (5) | −0.0012 (5) |
| C37 | 0.0211 (6) | 0.0226 (6) | 0.0187 (6) | −0.0001 (5) | 0.0073 (5) | 0.0011 (5) |
| C38 | 0.0200 (6) | 0.0246 (6) | 0.0223 (6) | 0.0011 (5) | 0.0071 (5) | 0.0009 (5) |
| C39 | 0.0246 (7) | 0.0260 (7) | 0.0305 (7) | 0.0011 (5) | 0.0140 (6) | −0.0044 (6) |
| C40 | 0.0267 (7) | 0.0349 (8) | 0.0202 (6) | −0.0023 (6) | 0.0120 (5) | −0.0080 (6) |
| C41 | 0.0225 (7) | 0.0336 (7) | 0.0189 (6) | −0.0012 (6) | 0.0085 (5) | −0.0069 (5) |
| C42 | 0.0247 (7) | 0.0276 (7) | 0.0256 (7) | −0.0065 (5) | 0.0113 (6) | −0.0046 (5) |
| C43 | 0.0315 (8) | 0.0223 (6) | 0.0286 (7) | −0.0010 (6) | 0.0146 (6) | 0.0030 (5) |
| C44 | 0.0327 (8) | 0.0385 (8) | 0.0226 (7) | 0.0008 (6) | 0.0142 (6) | 0.0050 (6) |
| C45 | 0.0283 (7) | 0.0219 (6) | 0.0243 (7) | 0.0036 (5) | 0.0108 (6) | 0.0020 (5) |
| C46 | 0.0176 (6) | 0.0239 (6) | 0.0161 (6) | 0.0005 (5) | 0.0072 (5) | −0.0011 (5) |
| C47 | 0.0164 (6) | 0.0301 (7) | 0.0189 (6) | 0.0002 (5) | 0.0057 (5) | −0.0037 (5) |
| C48 | 0.0199 (6) | 0.0342 (7) | 0.0177 (6) | −0.0007 (5) | 0.0057 (5) | −0.0044 (5) |
| C49 | 0.0334 (8) | 0.0388 (8) | 0.0206 (7) | 0.0006 (6) | 0.0121 (6) | 0.0029 (6) |
| C50 | 0.0348 (8) | 0.0307 (7) | 0.0284 (7) | −0.0084 (6) | 0.0180 (6) | −0.0012 (6) |
| C51 | 0.0200 (7) | 0.0452 (9) | 0.0278 (7) | −0.0074 (6) | 0.0112 (6) | −0.0105 (7) |
| C52 | 0.0182 (6) | 0.0320 (7) | 0.0226 (7) | 0.0049 (5) | 0.0061 (5) | −0.0035 (5) |
| C53 | 0.0194 (6) | 0.0274 (7) | 0.0194 (6) | 0.0027 (5) | 0.0058 (5) | −0.0003 (5) |
| C54 | 0.0227 (7) | 0.0293 (7) | 0.0254 (7) | 0.0001 (5) | 0.0092 (5) | −0.0068 (6) |
| C55 | 0.0313 (8) | 0.0233 (7) | 0.0271 (7) | −0.0022 (6) | 0.0121 (6) | −0.0006 (5) |
| O1 | 0.0350 (6) | 0.0229 (5) | 0.0269 (6) | 0.0015 (4) | 0.0035 (5) | 0.0014 (4) |
| O2 | 0.071 (2) | 0.0482 (17) | 0.0459 (17) | −0.0024 (15) | 0.0170 (15) | 0.0017 (13) |

| | | | | | | |
|---|---|---|---|---|---|---|
| C56 | 0.065 (3) | 0.0350 (18) | 0.0354 (19) | −0.0166 (17) | 0.0160 (18) | 0.0093 (15) |
| C57 | 0.0824 (19) | 0.0574 (15) | 0.0572 (14) | −0.0113 (13) | 0.0265 (14) | 0.0118 (12) |

*Geometric parameters (Å, °)*

| | | | |
|---|---|---|---|
| Li1—N2 | 1.9592 (11) | N9—C33 | 1.3358 (17) |
| Li1—N2$^{i}$ | 1.9592 (11) | N9—C34 | 1.3798 (18) |
| Li1—N4 | 1.9650 (11) | N9—C36 | 1.4906 (17) |
| Li1—N4$^{i}$ | 1.9650 (11) | N10—C33 | 1.3323 (17) |
| N1—C1 | 1.3352 (16) | N10—C35 | 1.3808 (17) |
| N1—C16$^{i}$ | 1.3365 (16) | N10—C46 | 1.4885 (16) |
| N2—C8 | 1.3597 (16) | C33—H33 | 0.9500 |
| N2—C1 | 1.3625 (16) | C34—C35 | 1.3481 (19) |
| N3—C9 | 1.3411 (16) | C34—H34 | 0.9500 |
| N3—C8 | 1.3424 (16) | C35—H35 | 0.9500 |
| N4—C9 | 1.3581 (16) | C36—C45 | 1.529 (2) |
| N4—C16 | 1.3617 (16) | C36—C41 | 1.5307 (18) |
| C1—C2 | 1.4603 (17) | C36—C37 | 1.5309 (19) |
| C2—C3 | 1.3895 (18) | C37—C38 | 1.5337 (19) |
| C2—C7 | 1.4020 (18) | C37—H37A | 0.9900 |
| C3—C4 | 1.3859 (19) | C37—H37B | 0.9900 |
| C3—H3 | 0.9500 | C38—C42 | 1.528 (2) |
| C4—C5 | 1.396 (2) | C38—C39 | 1.533 (2) |
| C4—H4 | 0.9500 | C38—H38 | 1.0000 |
| C5—C6 | 1.388 (2) | C39—C40 | 1.528 (2) |
| C5—H5 | 0.9500 | C39—H39A | 0.9900 |
| C6—C7 | 1.3914 (18) | C39—H39B | 0.9900 |
| C6—H6 | 0.9500 | C40—C44 | 1.531 (2) |
| C7—C8 | 1.4688 (18) | C40—C41 | 1.5335 (19) |
| C9—C10 | 1.4720 (18) | C40—H40 | 1.0000 |
| C10—C11 | 1.3923 (18) | C41—H41A | 0.9900 |
| C10—C15 | 1.4016 (18) | C41—H41B | 0.9900 |
| C11—C12 | 1.3892 (19) | C42—C43 | 1.529 (2) |
| C11—H11 | 0.9500 | C42—H42A | 0.9900 |
| C12—C13 | 1.399 (2) | C42—H42B | 0.9900 |
| C12—H12 | 0.9500 | C43—C44 | 1.532 (2) |
| C13—C14 | 1.3875 (19) | C43—C45 | 1.535 (2) |
| C13—H13 | 0.9500 | C43—H43 | 1.0000 |
| C14—C15 | 1.3913 (18) | C44—H44A | 0.9900 |
| C14—H14 | 0.9500 | C44—H44B | 0.9900 |
| C15—C16 | 1.4593 (18) | C45—H45A | 0.9900 |
| C16—N1$^{i}$ | 1.3365 (16) | C45—H45B | 0.9900 |
| Li2—N6 | 1.9567 (11) | C46—C55 | 1.5267 (19) |
| Li2—N6$^{ii}$ | 1.9567 (11) | C46—C47 | 1.5287 (18) |
| Li2—N8 | 1.9606 (11) | C46—C53 | 1.5322 (19) |
| Li2—N8$^{ii}$ | 1.9606 (11) | C47—C48 | 1.5347 (19) |
| N5—C17 | 1.3343 (17) | C47—H47A | 0.9900 |
| N5—C32$^{ii}$ | 1.3345 (17) | C47—H47B | 0.9900 |

| | | | |
|---|---|---|---|
| N6—C24 | 1.3625 (17) | C48—C49 | 1.526 (2) |
| N6—C17 | 1.3656 (17) | C48—C54 | 1.529 (2) |
| N7—C24 | 1.3357 (17) | C48—H48 | 1.0000 |
| N7—C25 | 1.3374 (17) | C49—C50 | 1.534 (2) |
| N8—C25 | 1.3667 (17) | C49—H49A | 0.9900 |
| N8—C32 | 1.3672 (17) | C49—H49B | 0.9900 |
| C17—C18 | 1.4634 (18) | C50—C51 | 1.526 (2) |
| C18—C19 | 1.3867 (18) | C50—C55 | 1.536 (2) |
| C18—C23 | 1.3984 (19) | C50—H50 | 1.0000 |
| C19—C20 | 1.388 (2) | C51—C52 | 1.527 (2) |
| C19—H19 | 0.9500 | C51—H51A | 0.9900 |
| C20—C21 | 1.399 (2) | C51—H51B | 0.9900 |
| C20—H20 | 0.9500 | C52—C54 | 1.530 (2) |
| C21—C22 | 1.389 (2) | C52—C53 | 1.5315 (19) |
| C21—H21 | 0.9500 | C52—H52 | 1.0000 |
| C22—C23 | 1.3910 (19) | C53—H53A | 0.9900 |
| C22—H22 | 0.9500 | C53—H53B | 0.9900 |
| C23—C24 | 1.4671 (18) | C54—H54A | 0.9900 |
| C25—C26 | 1.4636 (18) | C54—H54B | 0.9900 |
| C26—C27 | 1.3930 (19) | C55—H55A | 0.9900 |
| C26—C31 | 1.4019 (19) | C55—H55B | 0.9900 |
| C27—C28 | 1.3894 (19) | O1—H1A | 0.862 (15) |
| C27—H27 | 0.9500 | O1—H1B | 0.890 (15) |
| C28—C29 | 1.400 (2) | O2—C56 | 1.191 (5) |
| C28—H28 | 0.9500 | C56—C57$^{iii}$ | 1.476 (5) |
| C29—C30 | 1.388 (2) | C56—C57 | 1.487 (5) |
| C29—H29 | 0.9500 | C56—C56$^{iii}$ | 1.503 (8) |
| C30—C31 | 1.3939 (18) | C57—C56$^{iii}$ | 1.476 (5) |
| C30—H30 | 0.9500 | C57—H57A | 0.948 (18) |
| C31—C32 | 1.4628 (18) | C57—H57B | 0.931 (18) |
| C32—N5$^{ii}$ | 1.3345 (17) | C57—H57C | 0.935 (18) |
| N2—Li1—N2$^{i}$ | 180.00 (8) | N10—C35—H35 | 126.3 |
| N2—Li1—N4 | 89.28 (5) | N9—C36—C45 | 108.13 (11) |
| N2$^{i}$—Li1—N4 | 90.72 (5) | N9—C36—C41 | 109.07 (11) |
| N2—Li1—N4$^{i}$ | 90.72 (5) | C45—C36—C41 | 109.59 (11) |
| N2$^{i}$—Li1—N4$^{i}$ | 89.28 (5) | N9—C36—C37 | 110.33 (10) |
| N4—Li1—N4$^{i}$ | 180.00 (8) | C45—C36—C37 | 109.37 (11) |
| C1—N1—C16$^{i}$ | 122.68 (11) | C41—C36—C37 | 110.32 (11) |
| C8—N2—C1 | 107.98 (10) | C36—C37—C38 | 109.00 (11) |
| C8—N2—Li1 | 126.75 (9) | C36—C37—H37A | 109.9 |
| C1—N2—Li1 | 125.25 (8) | C38—C37—H37A | 109.9 |
| C9—N3—C8 | 122.79 (11) | C36—C37—H37B | 109.9 |
| C9—N4—C16 | 107.90 (11) | C38—C37—H37B | 109.9 |
| C9—N4—Li1 | 126.97 (9) | H37A—C37—H37B | 108.3 |
| C16—N4—Li1 | 124.98 (9) | C42—C38—C39 | 109.88 (12) |
| N1—C1—N2 | 128.03 (11) | C42—C38—C37 | 109.06 (11) |
| N1—C1—C2 | 121.71 (11) | C39—C38—C37 | 109.59 (11) |

| | | | |
|---|---|---|---|
| N2—C1—C2 | 110.25 (11) | C42—C38—H38 | 109.4 |
| C3—C2—C7 | 121.79 (12) | C39—C38—H38 | 109.4 |
| C3—C2—C1 | 132.17 (12) | C37—C38—H38 | 109.4 |
| C7—C2—C1 | 105.98 (11) | C40—C39—C38 | 109.39 (11) |
| C4—C3—C2 | 117.65 (13) | C40—C39—H39A | 109.8 |
| C4—C3—H3 | 121.2 | C38—C39—H39A | 109.8 |
| C2—C3—H3 | 121.2 | C40—C39—H39B | 109.8 |
| C3—C4—C5 | 120.76 (13) | C38—C39—H39B | 109.8 |
| C3—C4—H4 | 119.6 | H39A—C39—H39B | 108.2 |
| C5—C4—H4 | 119.6 | C39—C40—C44 | 109.89 (12) |
| C6—C5—C4 | 121.77 (13) | C39—C40—C41 | 109.54 (12) |
| C6—C5—H5 | 119.1 | C44—C40—C41 | 109.21 (12) |
| C4—C5—H5 | 119.1 | C39—C40—H40 | 109.4 |
| C5—C6—C7 | 117.74 (13) | C44—C40—H40 | 109.4 |
| C5—C6—H6 | 121.1 | C41—C40—H40 | 109.4 |
| C7—C6—H6 | 121.1 | C36—C41—C40 | 109.10 (11) |
| C6—C7—C2 | 120.29 (12) | C36—C41—H41A | 109.9 |
| C6—C7—C8 | 133.90 (12) | C40—C41—H41A | 109.9 |
| C2—C7—C8 | 105.72 (11) | C36—C41—H41B | 109.9 |
| N3—C8—N2 | 127.26 (12) | C40—C41—H41B | 109.9 |
| N3—C8—C7 | 122.66 (11) | H41A—C41—H41B | 108.3 |
| N2—C8—C7 | 110.07 (11) | C38—C42—C43 | 109.65 (12) |
| N3—C9—N4 | 126.87 (12) | C38—C42—H42A | 109.7 |
| N3—C9—C10 | 122.93 (11) | C43—C42—H42A | 109.7 |
| N4—C9—C10 | 110.20 (11) | C38—C42—H42B | 109.7 |
| C11—C10—C15 | 120.37 (12) | C43—C42—H42B | 109.7 |
| C11—C10—C9 | 134.17 (12) | H42A—C42—H42B | 108.2 |
| C15—C10—C9 | 105.46 (11) | C42—C43—C44 | 109.72 (12) |
| C12—C11—C10 | 117.89 (12) | C42—C43—C45 | 109.12 (11) |
| C12—C11—H11 | 121.1 | C44—C43—C45 | 109.47 (12) |
| C10—C11—H11 | 121.1 | C42—C43—H43 | 109.5 |
| C11—C12—C13 | 121.46 (12) | C44—C43—H43 | 109.5 |
| C11—C12—H12 | 119.3 | C45—C43—H43 | 109.5 |
| C13—C12—H12 | 119.3 | C40—C44—C43 | 109.39 (12) |
| C14—C13—C12 | 120.97 (13) | C40—C44—H44A | 109.8 |
| C14—C13—H13 | 119.5 | C43—C44—H44A | 109.8 |
| C12—C13—H13 | 119.5 | C40—C44—H44B | 109.8 |
| C13—C14—C15 | 117.54 (13) | C43—C44—H44B | 109.8 |
| C13—C14—H14 | 121.2 | H44A—C44—H44B | 108.2 |
| C15—C14—H14 | 121.2 | C36—C45—C43 | 109.16 (11) |
| C14—C15—C10 | 121.77 (12) | C36—C45—H45A | 109.8 |
| C14—C15—C16 | 132.13 (12) | C43—C45—H45A | 109.8 |
| C10—C15—C16 | 106.10 (11) | C36—C45—H45B | 109.8 |
| $N1^{i}$—C16—N4 | 128.09 (12) | C43—C45—H45B | 109.8 |
| $N1^{i}$—C16—C15 | 121.59 (12) | H45A—C45—H45B | 108.3 |
| N4—C16—C15 | 110.31 (11) | N10—C46—C55 | 109.00 (11) |
| N6—Li2—$N6^{ii}$ | 180.00 (7) | N10—C46—C47 | 109.81 (10) |
| N6—Li2—N8 | 90.47 (5) | C55—C46—C47 | 110.10 (11) |

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| N6$^{ii}$—Li2—N8 | 89.53 (5) | N10—C46—C53 | 108.16 (10) |
| N6—Li2—N8$^{ii}$ | 89.53 (5) | C55—C46—C53 | 110.19 (11) |
| N6$^{ii}$—Li2—N8$^{ii}$ | 90.47 (5) | C47—C46—C53 | 109.55 (11) |
| N8—Li2—N8$^{ii}$ | 180.00 (3) | C46—C47—C48 | 108.73 (11) |
| C17—N5—C32$^{ii}$ | 122.18 (11) | C46—C47—H47A | 109.9 |
| C24—N6—C17 | 107.97 (11) | C48—C47—H47A | 109.9 |
| C24—N6—Li2 | 125.63 (9) | C46—C47—H47B | 109.9 |
| C17—N6—Li2 | 126.35 (9) | C48—C47—H47B | 109.9 |
| C24—N7—C25 | 122.74 (12) | H47A—C47—H47B | 108.3 |
| C25—N8—C32 | 107.97 (11) | C49—C48—C54 | 109.21 (12) |
| C25—N8—Li2 | 125.43 (9) | C49—C48—C47 | 109.43 (12) |
| C32—N8—Li2 | 126.19 (9) | C54—C48—C47 | 109.83 (11) |
| N5—C17—N6 | 127.84 (12) | C49—C48—H48 | 109.5 |
| N5—C17—C18 | 122.04 (12) | C54—C48—H48 | 109.5 |
| N6—C17—C18 | 110.13 (11) | C47—C48—H48 | 109.5 |
| C19—C18—C23 | 121.39 (12) | C48—C49—C50 | 109.44 (12) |
| C19—C18—C17 | 132.72 (13) | C48—C49—H49A | 109.8 |
| C23—C18—C17 | 105.89 (11) | C50—C49—H49A | 109.8 |
| C18—C19—C20 | 117.78 (13) | C48—C49—H49B | 109.8 |
| C18—C19—H19 | 121.1 | C50—C49—H49B | 109.8 |
| C20—C19—H19 | 121.1 | H49A—C49—H49B | 108.2 |
| C19—C20—C21 | 120.99 (13) | C51—C50—C49 | 110.30 (13) |
| C19—C20—H20 | 119.5 | C51—C50—C55 | 109.30 (12) |
| C21—C20—H20 | 119.5 | C49—C50—C55 | 108.73 (13) |
| C22—C21—C20 | 121.20 (13) | C51—C50—H50 | 109.5 |
| C22—C21—H21 | 119.4 | C49—C50—H50 | 109.5 |
| C20—C21—H21 | 119.4 | C55—C50—H50 | 109.5 |
| C21—C22—C23 | 117.79 (13) | C50—C51—C52 | 109.91 (12) |
| C21—C22—H22 | 121.1 | C50—C51—H51A | 109.7 |
| C23—C22—H22 | 121.1 | C52—C51—H51A | 109.7 |
| C22—C23—C18 | 120.83 (12) | C50—C51—H51B | 109.7 |
| C22—C23—C24 | 133.03 (13) | C52—C51—H51B | 109.7 |
| C18—C23—C24 | 106.13 (11) | H51A—C51—H51B | 108.2 |
| N7—C24—N6 | 127.88 (12) | C51—C52—C54 | 108.96 (12) |
| N7—C24—C23 | 122.24 (12) | C51—C52—C53 | 109.23 (12) |
| N6—C24—C23 | 109.88 (11) | C54—C52—C53 | 109.53 (11) |
| N7—C25—N8 | 127.62 (12) | C51—C52—H52 | 109.7 |
| N7—C25—C26 | 122.45 (12) | C54—C52—H52 | 109.7 |
| N8—C25—C26 | 109.93 (11) | C53—C52—H52 | 109.7 |
| C27—C26—C31 | 120.84 (12) | C52—C53—C46 | 109.16 (11) |
| C27—C26—C25 | 132.99 (12) | C52—C53—H53A | 109.8 |
| C31—C26—C25 | 106.09 (11) | C46—C53—H53A | 109.8 |
| C28—C27—C26 | 117.44 (13) | C52—C53—H53B | 109.8 |
| C28—C27—H27 | 121.3 | C46—C53—H53B | 109.8 |
| C26—C27—H27 | 121.3 | H53A—C53—H53B | 108.3 |
| C27—C28—C29 | 121.79 (13) | C48—C54—C52 | 110.00 (12) |
| C27—C28—H28 | 119.1 | C48—C54—H54A | 109.7 |
| C29—C28—H28 | 119.1 | C52—C54—H54A | 109.7 |

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| C30—C29—C28 | 120.83 (13) | C48—C54—H54B | 109.7 |
| C30—C29—H29 | 119.6 | C52—C54—H54B | 109.7 |
| C28—C29—H29 | 119.6 | H54A—C54—H54B | 108.2 |
| C29—C30—C31 | 117.62 (13) | C46—C55—C50 | 108.86 (12) |
| C29—C30—H30 | 121.2 | C46—C55—H55A | 109.9 |
| C31—C30—H30 | 121.2 | C50—C55—H55A | 109.9 |
| C30—C31—C26 | 121.48 (12) | C46—C55—H55B | 109.9 |
| C30—C31—C32 | 132.41 (13) | C50—C55—H55B | 109.9 |
| C26—C31—C32 | 106.03 (11) | H55A—C55—H55B | 108.3 |
| N5$^{ii}$—C32—N8 | 127.75 (12) | H1A—O1—H1B | 105.0 (18) |
| N5$^{ii}$—C32—C31 | 122.23 (12) | O2—C56—C57$^{iii}$ | 120.8 (4) |
| N8—C32—C31 | 109.99 (11) | O2—C56—C57 | 120.0 (4) |
| C33—N9—C34 | 108.36 (11) | C57$^{iii}$—C56—C57 | 119.1 (3) |
| C33—N9—C36 | 124.24 (11) | O2—C56—C56$^{iii}$ | 177.4 (5) |
| C34—N9—C36 | 127.12 (11) | C57$^{iii}$—C56—C56$^{iii}$ | 59.9 (3) |
| C33—N10—C35 | 108.33 (11) | C57—C56—C56$^{iii}$ | 59.2 (3) |
| C33—N10—C46 | 127.08 (11) | C56$^{iii}$—C57—C56 | 60.9 (3) |
| C35—N10—C46 | 124.55 (11) | C56$^{iii}$—C57—H57A | 156.5 (19) |
| N10—C33—N9 | 108.73 (12) | C56—C57—H57A | 112.2 (15) |
| N10—C33—H33 | 125.6 | C56$^{iii}$—C57—H57B | 76.6 (18) |
| N9—C33—H33 | 125.6 | C56—C57—H57B | 116.3 (15) |
| C35—C34—N9 | 107.22 (12) | H57A—C57—H57B | 88 (2) |
| C35—C34—H34 | 126.4 | C56$^{iii}$—C57—H57C | 101.4 (18) |
| N9—C34—H34 | 126.4 | C56—C57—H57C | 115.1 (15) |
| C34—C35—N10 | 107.36 (12) | H57A—C57—H57C | 102 (3) |
| C34—C35—H35 | 126.3 | H57B—C57—H57C | 118 (3) |
| N4—Li1—N2—C8 | −0.79 (11) | C24—N7—C25—N8 | −3.2 (2) |
| N4$^{i}$—Li1—N2—C8 | 179.21 (11) | C24—N7—C25—C26 | 176.20 (12) |
| N4—Li1—N2—C1 | −178.85 (10) | C32—N8—C25—N7 | 179.25 (13) |
| N4$^{i}$—Li1—N2—C1 | 1.15 (10) | Li2—N8—C25—N7 | 6.3 (2) |
| N2—Li1—N4—C9 | 1.91 (11) | C32—N8—C25—C26 | −0.18 (14) |
| N2$^{i}$—Li1—N4—C9 | −178.09 (11) | Li2—N8—C25—C26 | −173.14 (8) |
| N2—Li1—N4—C16 | 176.80 (11) | N7—C25—C26—C27 | −3.0 (2) |
| N2$^{i}$—Li1—N4—C16 | −3.20 (11) | N8—C25—C26—C27 | 176.45 (14) |
| C16$^{i}$—N1—C1—N2 | 2.2 (2) | N7—C25—C26—C31 | −179.63 (12) |
| C16$^{i}$—N1—C1—C2 | −179.29 (12) | N8—C25—C26—C31 | −0.17 (14) |
| C8—N2—C1—N1 | 177.65 (12) | C31—C26—C27—C28 | 0.03 (19) |
| Li1—N2—C1—N1 | −3.99 (19) | C25—C26—C27—C28 | −176.19 (13) |
| C8—N2—C1—C2 | −1.00 (14) | C26—C27—C28—C29 | −0.5 (2) |
| Li1—N2—C1—C2 | 177.36 (8) | C27—C28—C29—C30 | 0.8 (2) |
| N1—C1—C2—C3 | 5.0 (2) | C28—C29—C30—C31 | −0.5 (2) |
| N2—C1—C2—C3 | −176.21 (13) | C29—C30—C31—C26 | 0.0 (2) |
| N1—C1—C2—C7 | −177.94 (12) | C29—C30—C31—C32 | 176.01 (14) |
| N2—C1—C2—C7 | 0.80 (14) | C27—C26—C31—C30 | 0.24 (19) |
| C7—C2—C3—C4 | −0.38 (19) | C25—C26—C31—C30 | 177.36 (12) |

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| C1—C2—C3—C4 | 176.23 (13) | C27—C26—C31—C32 | −176.71 (12) |
| C2—C3—C4—C5 | 0.2 (2) | C25—C26—C31—C32 | 0.41 (13) |
| C3—C4—C5—C6 | 0.3 (2) | C25—N8—C32—N5$^{ii}$ | −177.29 (13) |
| C4—C5—C6—C7 | −0.5 (2) | Li2—N8—C32—N5$^{ii}$ | −4.40 (19) |
| C5—C6—C7—C2 | 0.3 (2) | C25—N8—C32—C31 | 0.45 (14) |
| C5—C6—C7—C8 | −175.63 (14) | Li2—N8—C32—C31 | 173.34 (8) |
| C3—C2—C7—C6 | 0.2 (2) | C30—C31—C32—N5$^{ii}$ | 0.9 (2) |
| C1—C2—C7—C6 | −177.23 (12) | C26—C31—C32—N5$^{ii}$ | 177.34 (12) |
| C3—C2—C7—C8 | 177.10 (12) | C30—C31—C32—N8 | −177.02 (14) |
| C1—C2—C7—C8 | −0.29 (13) | C26—C31—C32—N8 | −0.55 (14) |
| C9—N3—C8—N2 | −2.3 (2) | C35—N10—C33—N9 | −0.38 (16) |
| C9—N3—C8—C7 | 176.25 (12) | C46—N10—C33—N9 | 177.78 (12) |
| C1—N2—C8—N3 | 179.48 (12) | C34—N9—C33—N10 | 0.13 (16) |
| Li1—N2—C8—N3 | 1.15 (19) | C36—N9—C33—N10 | −174.13 (12) |
| C1—N2—C8—C7 | 0.81 (14) | C33—N9—C34—C35 | 0.17 (17) |
| Li1—N2—C8—C7 | −177.52 (8) | C36—N9—C34—C35 | 174.22 (13) |
| C6—C7—C8—N3 | −2.7 (2) | N9—C34—C35—N10 | −0.40 (17) |
| C2—C7—C8—N3 | −179.05 (12) | C33—N10—C35—C34 | 0.48 (17) |
| C6—C7—C8—N2 | 176.03 (14) | C46—N10—C35—C34 | −177.73 (13) |
| C2—C7—C8—N2 | −0.30 (14) | C33—N9—C36—C45 | 64.57 (16) |
| C8—N3—C9—N4 | 3.6 (2) | C34—N9—C36—C45 | −108.59 (15) |
| C8—N3—C9—C10 | −175.40 (12) | C33—N9—C36—C41 | −176.32 (13) |
| C16—N4—C9—N3 | −179.22 (12) | C34—N9—C36—C41 | 10.52 (19) |
| Li1—N4—C9—N3 | −3.61 (19) | C33—N9—C36—C37 | −55.00 (17) |
| C16—N4—C9—C10 | −0.14 (14) | C34—N9—C36—C37 | 131.84 (14) |
| Li1—N4—C9—C10 | 175.46 (8) | N9—C36—C37—C38 | 179.77 (11) |
| N3—C9—C10—C11 | −1.2 (2) | C45—C36—C37—C38 | 60.96 (14) |
| N4—C9—C10—C11 | 179.65 (14) | C41—C36—C37—C38 | −59.65 (14) |
| N3—C9—C10—C15 | 178.30 (12) | C36—C37—C38—C42 | −60.66 (14) |
| N4—C9—C10—C15 | −0.82 (14) | C36—C37—C38—C39 | 59.67 (14) |
| C15—C10—C11—C12 | −0.87 (19) | C42—C38—C39—C40 | 59.37 (15) |
| C9—C10—C11—C12 | 178.61 (13) | C37—C38—C39—C40 | −60.45 (15) |
| C10—C11—C12—C13 | 0.1 (2) | C38—C39—C40—C44 | −59.55 (15) |
| C11—C12—C13—C14 | 0.4 (2) | C38—C39—C40—C41 | 60.44 (15) |
| C12—C13—C14—C15 | −0.2 (2) | N9—C36—C41—C40 | −178.95 (11) |
| C13—C14—C15—C10 | −0.6 (2) | C45—C36—C41—C40 | −60.75 (15) |
| C13—C14—C15—C16 | 179.63 (13) | C37—C36—C41—C40 | 59.72 (15) |
| C11—C10—C15—C14 | 1.1 (2) | C39—C40—C41—C36 | −59.79 (15) |
| C9—C10—C15—C14 | −178.49 (12) | C44—C40—C41—C36 | 60.61 (15) |
| C11—C10—C15—C16 | −179.02 (12) | C39—C38—C42—C43 | −59.47 (15) |
| C9—C10—C15—C16 | 1.36 (13) | C37—C38—C42—C43 | 60.67 (14) |
| C9—N4—C16—N1$^{i}$ | −178.37 (13) | C38—C42—C43—C44 | 59.57 (15) |
| Li1—N4—C16—N1$^{i}$ | 5.92 (19) | C38—C42—C43—C45 | −60.37 (15) |
| C9—N4—C16—C15 | 1.03 (14) | C39—C40—C44—C43 | 59.74 (15) |
| Li1—N4—C16—C15 | −174.68 (8) | C41—C40—C44—C43 | −60.45 (16) |
| C14—C15—C16—N1$^{i}$ | −2.3 (2) | C42—C43—C44—C40 | −59.58 (16) |
| C10—C15—C16—N1$^{i}$ | 177.90 (12) | C45—C43—C44—C40 | 60.15 (16) |

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| C14—C15—C16—N4 | 178.29 (14) | N9—C36—C45—C43 | 179.11 (11) |
| C10—C15—C16—N4 | −1.54 (14) | C41—C36—C45—C43 | 60.33 (15) |
| N8—Li2—N6—C24 | 0.27 (11) | C37—C36—C45—C43 | −60.72 (14) |
| N8$^{ii}$—Li2—N6—C24 | −179.73 (11) | C42—C43—C45—C36 | 60.18 (15) |
| N8—Li2—N6—C17 | −176.88 (11) | C44—C43—C45—C36 | −59.92 (15) |
| N8$^{ii}$—Li2—N6—C17 | 3.12 (11) | C33—N10—C46—C55 | 120.09 (15) |
| N6—Li2—N8—C25 | −4.10 (11) | C35—N10—C46—C55 | −62.03 (17) |
| N6$^{ii}$—Li2—N8—C25 | 175.90 (11) | C33—N10—C46—C47 | −0.60 (19) |
| N6—Li2—N8—C32 | −175.80 (11) | C35—N10—C46—C47 | 177.28 (13) |
| N6$^{ii}$—Li2—N8—C32 | 4.20 (11) | C33—N10—C46—C53 | −120.10 (14) |
| C32$^{ii}$—N5—C17—N6 | 0.5 (2) | C35—N10—C46—C53 | 57.77 (17) |
| C32$^{ii}$—N5—C17—C18 | −179.08 (12) | N10—C46—C47—C48 | −179.67 (11) |
| C24—N6—C17—N5 | −179.52 (13) | C55—C46—C47—C48 | 60.31 (15) |
| Li2—N6—C17—N5 | −2.0 (2) | C53—C46—C47—C48 | −61.02 (14) |
| C24—N6—C17—C18 | 0.07 (15) | C46—C47—C48—C49 | −59.97 (15) |
| Li2—N6—C17—C18 | 177.63 (9) | C46—C47—C48—C54 | 59.90 (15) |
| N5—C17—C18—C19 | 0.0 (2) | C54—C48—C49—C50 | −59.32 (16) |
| N6—C17—C18—C19 | −179.67 (14) | C47—C48—C49—C50 | 60.93 (16) |
| N5—C17—C18—C23 | 179.73 (12) | C48—C49—C50—C51 | 58.81 (16) |
| N6—C17—C18—C23 | 0.11 (15) | C48—C49—C50—C55 | −61.02 (16) |
| C23—C18—C19—C20 | −0.2 (2) | C49—C50—C51—C52 | −58.90 (15) |
| C17—C18—C19—C20 | 179.54 (14) | C55—C50—C51—C52 | 60.58 (15) |
| C18—C19—C20—C21 | −0.3 (2) | C50—C51—C52—C54 | 59.22 (15) |
| C19—C20—C21—C22 | 0.6 (2) | C50—C51—C52—C53 | −60.39 (15) |
| C20—C21—C22—C23 | −0.4 (2) | C51—C52—C53—C46 | 59.54 (15) |
| C21—C22—C23—C18 | −0.1 (2) | C54—C52—C53—C46 | −59.72 (15) |
| C21—C22—C23—C24 | −179.03 (14) | N10—C46—C53—C52 | −179.12 (11) |
| C19—C18—C23—C22 | 0.4 (2) | C55—C46—C53—C52 | −60.06 (14) |
| C17—C18—C23—C22 | −179.42 (12) | C47—C46—C53—C52 | 61.21 (14) |
| C19—C18—C23—C24 | 179.58 (12) | C49—C48—C54—C52 | 60.81 (15) |
| C17—C18—C23—C24 | −0.23 (14) | C47—C48—C54—C52 | −59.20 (15) |
| C25—N7—C24—N6 | −1.5 (2) | C51—C52—C54—C48 | −60.47 (15) |
| C25—N7—C24—C23 | 178.69 (12) | C53—C52—C54—C48 | 58.95 (16) |
| C17—N6—C24—N7 | 179.97 (13) | N10—C46—C55—C50 | 178.57 (11) |
| Li2—N6—C24—N7 | 2.4 (2) | C47—C46—C55—C50 | −60.91 (15) |
| C17—N6—C24—C23 | −0.22 (15) | C53—C46—C55—C50 | 60.03 (15) |
| Li2—N6—C24—C23 | −177.80 (9) | C51—C50—C55—C46 | −59.87 (16) |
| C22—C23—C24—N7 | −0.8 (2) | C49—C50—C55—C46 | 60.58 (16) |
| C18—C23—C24—N7 | −179.89 (12) | O2—C56—C57—C56$^{iii}$ | 177.2 (5) |
| C22—C23—C24—N6 | 179.34 (14) | C57$^{iii}$—C56—C57—C56$^{iii}$ | 0.000 (1) |
| C18—C23—C24—N6 | 0.28 (15) | | |

Symmetry codes: (i) −*x*+2, −*y*, −*z*; (ii) −*x*, −*y*+1, −*z*; (iii) −*x*, −*y*+1, −*z*+1.

*Hydrogen-bond geometry (Å, °)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| O1—H1A···N6 | 0.861 (18) | 2.405 (17) | 3.1618 (17) | 147.1 (18) |

| | | | | |
|---|---|---|---|---|
| O1—H1B···N3$^{iv}$ | 0.889 (18) | 2.025 (19) | 2.8911 (17) | 164.3 (16) |
| C33—H33···O1 | 0.95 | 2.18 | 3.127 (2) | 171 |
| C37—H37A···O1 | 0.99 | 2.52 | 3.493 (2) | 166 |

Symmetry codes: (iv) −*x*+1, −*y*+1, −*z*.

**Fig. 1**

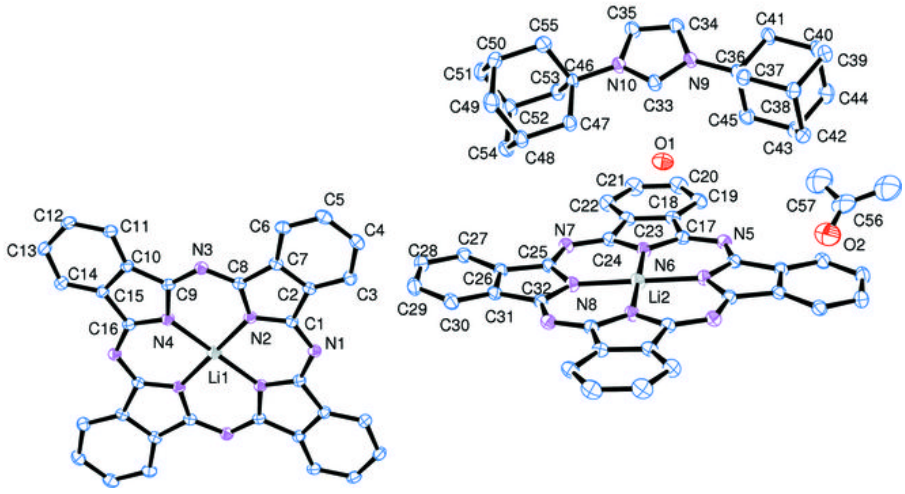
C35
C55
C34
C41
C50
C40
C46
C36
C39
C51
C53
N10
N9
C37
C49
C33
C44
C52
C47
C45
C38
C54
C48
C43
C42
O1
C21
C20
C57
C22
C18
C19
C56
C12
C5
C11
C6
C4
C17
N5
C13
C10
N3
C8
C7
C23
C28
C27
N7
C25
C24
O2
C14
C15
C9
C2
C3
C26
N6
C16
C1
C32
C29
N4
N2
N1
N8
Li2
C30
C31
Li1

**Fig. 2**