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Lithiation of the diaminopyridine protio-ligand MeC(2-C_5H_4N){$CH_2N(H)Mes$}$_2$ (Mes = 2,4,6-$C_6H_2Me_3$)

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Key indicators

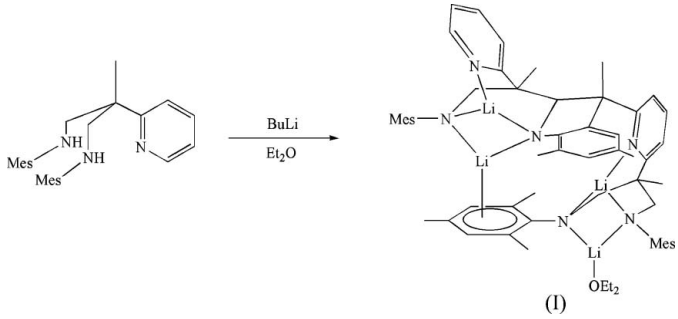
Single-crystal X-ray study
T = 173 K
Mean σ(C–C) = 0.005 Å
Disorder in main residue
R factor = 0.051
wR factor = 0.054
Data-to-parameter ratio = 7.3

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

Reaction of the diamidopyridine protio-ligand MeC(2-C_5H_4N){$CH_2N(H)Mes$}$_2$ (Mes = 2,4,6-$C_6H_2Me_3$) with butyllithium in diethyl ether affords the compound (diethyl ether)(μ_4-2-{6-[1,3-dimethyl-3-(2-pyridyl)-1,4-bis(2,4,6-trimethylanilinio)butyl]-2-pyridyl}-2-methyl-1,3-bis(2,4,6-trimethylanilinio)propane)tetralithium(I), [$Li_4(C_{55}H_{64}N_6)(C_4H_{10}O)$] or Li_4[MeC(2-C_5H_4N){$CH_2N(Mes)$}CHN(2,4,6-$C_6H_2Me_2$C(Me)(2,2′-C_5H_3N){C(Me)}($CH_2NMes)_2$](OEt$_2$), which shows the methylation of the pyridyl *ortho*-position with a methyl group of one of the mesityl groups. The complex contains four Li atoms, each of which is chemically distinct. The tetraanionic ligand contains two protio-ligand units which are fused together into a single entity. The structure contains two disordered molecules of diethyl ether, one of which is coordinated to one of the Li atoms.

Comment

Diamidopyridine ligands of the general formula [MeC(C_5H_4N)($CH_2NR)_2$]$^{2-}$ (R = silyl or aryl) have found a variety of uses in early transition metal chemistry and catalysis over the last decade (Gade & Mountford, 2001; Mehrkhodavandi *et al.*, 2000). Whereas the silylated derivatives (R = $SiMe_3$ or $SiMe_2{}^tBu$) are known as their dilithium salts (Friedrich *et al.*, 1997), the corresponding *N*-arylated derivatives (R = 3,5-$C_6H_3Cl_2$, 4-C_6H_4Me or 2,4,6-$C_6H_2Me_3$) have remained elusive. In attempting to prepare such complexes in our laboratories, we have consistently observed degradation products, and we report here the structural characterization of a product, (I), arising from the reaction of the mesityl protio-ligand (Mehrkhodavandi *et al.*, 2000) with butyllithium in diethyl ether.



(I)

The tetralithium complex, (I), crystallizes in space group $P\overline{1}$, and contains four Li environments which each occupy chemically different sites within the molecule. The Li atoms are arranged in two pairs, each occupying a bridging position between two amide N atoms. Each pair of Li atoms is capped

at one end by a pyridyl group. At the other end, one pair of Li atoms is capped by one of the mesityl groups bonding in an η^3 mode. The second pair is capped by a molecule of diethyl ether coordinated to one of the Li centres; one of the ethyl groups of this ligand was found to be disordered over two sites. The disorder was modelled by using an occupancy of 0.5 for each atom within the ethyl group for each of the two sites.

The structure clearly does not represent a stoichiometric dimerization of the dilithium compound, since it contains an 'extra' methyl group (C29). The presence of this methyl group suggests that the structure arises from the rearrangement of three diamidopyridine units. The mechanistic details behind this rearrangement are not clear, and no other well defined product could be obtained from the reaction mixture. The structure also contains a disordered non-coordinated molecule of diethyl ether.

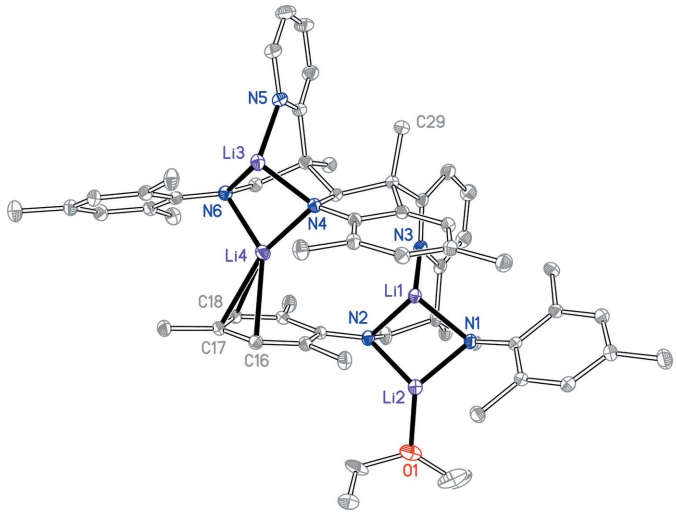


Figure 1
A view of the molecular structure of (I). Displacement ellipsoids are drawn at the 25% probability level and H atoms have been omitted for clarity. The solvent of crystallization has also been omitted for clarity and only a single orientation of the disordered coordinated diethyl ether molecule is shown.

Experimental

The diaminopyridine protio-ligand MeC(2-C_5H_4N){$CH_2N(H)Mes$}$_2$ (0.50 g, 1.25 mmol) was dissolved in diethyl ether (20 ml) and cooled to 195 K, followed by the dropwise addition of butyllithium (1.56 ml of a 1.6 *M* solution in hexanes, 2.50 mmol, 2 equivalents). The colourless solution immediately turned bright yellow, and the reaction was allowed to warm slowly to ambient temperature and stirred for 1 h. The reaction mixture was concentrated to 5 ml, and crystals of (I) suitable for X-ray diffraction were formed on allowing the mixture to stand overnight.

Crystal data

$[Li_4(C_{55}H_{64}N_6)(C_4H_{10}O)]$
M_r = 911.04
Triclinic, $P\overline{1}$
a = 11.607 (2) Å
b = 14.302 (3) Å
c = 17.477 (4) Å
α = 78.18 (3)°
β = 72.68 (3)°
γ = 80.31 (3)°
V = 2693.0 (11) Å^3
Z = 2
D_x = 1.123 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 22654 reflections
θ = 5–28°
μ = 0.07 mm^{-1}
T = 173 K
Block, yellow
0.20 × 0.20 × 0.15 mm

Data collection

Nonius KappaCCD area-detector diffractometer
ω scans
Absorption correction: multi-scan *DENZO/SCALEPACK* (Otwinowski & Minor, 1997)
T_{min} = 0.99, T_{max} = 0.99
22654 measured reflections
12141 independent reflections
4760 reflections with $I > 3\sigma(I)$
R_{int} = 0.026
θ_{max} = 27.5°
$h = -14 \rightarrow 15$
$k = -17 \rightarrow 18$
$l = 0 \rightarrow 22$

Refinement

Refinement on F
$R[F^2 > 2\sigma(F^2)]$ = 0.051
$wR(F^2)$ = 0.054
S = 1.09
4760 reflections
649 parameters
H-atom parameters constrained
$w = [1 - (F_o - F_c)^2/36\sigma^2(F)]^2/[0.516T_o(x) + 0.342T_1(x) + 0.252T_2(x)]$, where T_i are the Chebychev polynomials and $x = F_c/F_{max}$ (Prince, 1982; Watkin, 1994)
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.33 e Å^{-3}
$\Delta\rho_{min}$ = −0.23 e Å^{-3}

The crystal was a weak diffractor. Although sufficient data were collected, the number of data with $I > 3\sigma(I)$ was low. It is therefore inappropriate to compare bond lengths and angles with structures of higher precision, although the connectivity is thought to be reliable. The *Comment* has been written to take this into account.

The structure contains a coordinated molecule of diethyl ether, of which one ethyl group was disordered over two sites. The disorder was modelled with each C atom given 0.5 occupancy.

The asymmetric unit also contains a non-coordinated molecule of diethyl ether lying close to a centre of symmetry. Attempts were made to model this using disordered ether molecules. One model consisted of a molecule of ether interpenetrating its image in a mirror plane perpendicular to the medial axis of the molecule, *i.e.* the central region of the difference electron density phased on all the non-ether atoms was occupied by a 'split' O atom. This model needed geometric restraints and led to atoms falling on regions of relatively low electron density. Refinement of the unrestrained isotropic displacement parameters led to unacceptable values. A second model displaced the ether molecule sideways along its longest axis, so that the central region of the difference-density map now contained a disordered O and C atom. This model also needed restraints, gave a similar R factor to the previous model, and also gave a poor (but different) fit to the difference density. An unrestrained model gave a good fit to the density, but unacceptable distances and angles. Based on this evidence, it was felt that an atomic model was unsuitable. The disordered region was modelled using *SQUEEZE* (van der Sluis & Spek, 1990) in its advanced mode, in which the *A* and *B* parts of the structure factor are passed back to *CRYSTALS* (Betteridge *et al.*, 2003) for inclusion in F_c (rather than the term being subtracted from F_o).

H atoms associated with the aryl-methyl groups were found in a difference Fourier map. All other H atoms were placed geometrically after each cycle of refinement, with C—H distances in the range 0.96–1.1 Å. All H atoms were treated with the riding model during the refinement, with $U_{iso}(H) = 1.3U_{eq}(C)$.

Data collection: *COLLECT* (Nonius, 2000); cell refinement: *DENZO/SCALEPACK* (Otwinowski & Minor, 1997); data reduc-

tion: *DENZO*/*SCALEPACK*; program(s) used to solve structure: *SHELXS86* (Sheldrick, 1985); program(s) used to refine structure: *CRYSTALS* (Betteridge *et al.*, 2003); molecular graphics: *CAMERON* (Watkin *et al.*, 1996); software used to prepare material for publication: *CRYSTALS*.

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(diethyl ether)(μ_4-2-{6-[1,3-dimethyl-3-(2-pyridyl)-1,4-bis(2,4,6- trimethylanilinio)butyl]-2-pyridyl}-2-methyl-1,3-bis(2,4,6- trimethylanilinio)propane)tetralithium(I) diethyl ether solvate

Crystal data

$[Li_4(C_{55}H_{64}N_6)(C_4H_{10}O)]$
M_r = 911.04
Triclinic, $P\overline{1}$
Hall symbol: -P 1
a = 11.607 (2) Å
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α = 78.18 (3)°
β = 72.68 (3)°
γ = 80.31 (3)°
V = 2693.0 (11) Å^3
Z = 2
$F(000)$ = 980
D_x = 1.123 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 22654 reflections
θ = 5–28°
μ = 0.07 mm^{-1}
T = 173 K
Block, yellow
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Data collection

Nonius KappaCCD area-detector diffractometer
Graphite monochromator
CCD scans
Absorption correction: multi-scan DENZO/SCALEPACK (Otwinowski & Minor, 1997)
T_{min} = 0.99, T_{max} = 0.99
22654 measured reflections
12141 independent reflections
4760 reflections with $I > 3\sigma(I)$
R_{int} = 0.026
θ_{max} = 27.5°, θ_{min} = 5.1°
h = −14→15
k = −17→18
l = 0→22

Refinement

Refinement on F
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.051
$wR(F^2)$ = 0.054
S = 1.09
4760 reflections
649 parameters
36 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = [1-(F_o-F_c)^2/36\sigma^2(F)]^2/[0.516T_o(x) + 0.342T_1(x) + 0.252T_2(x)]$, where T_i are the Chebychev polynomials and x = F_c/F_{max} (*P*rince, 1982; Watkin, 1994)
$(\Delta/\sigma)_{max}$ = 0.000236
$\Delta\rho_{max}$ = 0.33 e Å^{-3}
$\Delta\rho_{min}$ = −0.23 e Å^{-3}

Special details

Refinement. Loose vibration and thermal similarity restraints were applied to the coordinated molecule of diethyl ether.

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

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
Li1	0.7451 (4)	0.2096 (3)	0.7821 (3)	0.0324	
Li2	0.6433 (5)	0.0889 (3)	0.7436 (3)	0.0391	
Li3	0.6008 (5)	0.6041 (3)	0.8068 (3)	0.0353	
Li4	0.5436 (5)	0.4873 (3)	0.7342 (3)	0.0360	
N1	0.7888 (2)	0.06762 (15)	0.78459 (15)	0.0304	
N2	0.6711 (2)	0.22286 (16)	0.68750 (15)	0.0306	
N3	0.8956 (2)	0.26499 (15)	0.71481 (15)	0.0310	
N4	0.6483 (2)	0.46919 (15)	0.81021 (14)	0.0298	
N5	0.7514 (2)	0.65913 (16)	0.79263 (16)	0.0350	
N6	0.5895 (2)	0.62457 (16)	0.69496 (15)	0.0310	
O1	0.5815 (2)	0.00686 (18)	0.69246 (15)	0.0554	
C1	0.9679 (3)	0.1061 (2)	0.5593 (2)	0.0429	
C2	0.8928 (3)	0.1430 (2)	0.63835 (19)	0.0347	
C3	0.8734 (3)	0.05210 (19)	0.70595 (18)	0.0326	
C4	0.8107 (3)	0.00375 (18)	0.85267 (18)	0.0295	
C5	0.7140 (3)	−0.04321 (19)	0.90868 (18)	0.0303	
C6	0.7307 (3)	−0.1085 (2)	0.97646 (19)	0.0343	
C7	0.8424 (3)	−0.1300 (2)	0.99362 (19)	0.0364	
C8	0.9358 (3)	−0.0813 (2)	0.9414 (2)	0.0369	
C9	0.9243 (3)	−0.01446 (19)	0.87250 (19)	0.0331	
C10	0.5890 (3)	−0.0244 (2)	0.8962 (2)	0.0404	
C11	0.8586 (3)	−0.2023 (3)	1.0674 (2)	0.0517	
C12	1.0298 (3)	0.0404 (2)	0.8250 (2)	0.0408	
C13	0.7737 (3)	0.1982 (2)	0.61890 (19)	0.0389	
C14	0.5879 (2)	0.30074 (18)	0.67972 (17)	0.0261	
C15	0.4828 (3)	0.30696 (19)	0.74838 (18)	0.0308	
C16	0.3866 (3)	0.37940 (19)	0.74953 (19)	0.0315	
C17	0.3853 (2)	0.45402 (19)	0.68461 (18)	0.0294	
C18	0.4878 (3)	0.45240 (19)	0.61887 (18)	0.0305	
C19	0.5878 (3)	0.3806 (2)	0.61350 (17)	0.0295	
C20	0.4723 (3)	0.2288 (2)	0.82212 (19)	0.0388	
C21	0.2785 (3)	0.5309 (2)	0.6854 (2)	0.0379	
C22	0.6898 (3)	0.3941 (2)	0.5356 (2)	0.0408	
C23	0.9626 (3)	0.20880 (19)	0.66114 (19)	0.0333	
C24	1.0893 (3)	0.2105 (2)	0.6328 (2)	0.0423	
C25	1.1430 (3)	0.2677 (2)	0.6633 (2)	0.0464	
C26	1.0729 (3)	0.3261 (2)	0.7180 (2)	0.0412	
C27	0.9478 (3)	0.32595 (19)	0.74035 (19)	0.0325	
C28	0.8583 (3)	0.39146 (19)	0.79394 (19)	0.0313	
C29	0.9203 (3)	0.4466 (2)	0.8343 (2)	0.0383	
C30	0.7659 (3)	0.33536 (19)	0.86184 (18)	0.0298	

C31	0.7846 (3)	0.2525 (2)	0.91553 (19)	0.0333	
C32	0.6893 (3)	0.2212 (2)	0.98112 (19)	0.0345	
C33	0.5763 (3)	0.2774 (2)	0.99052 (19)	0.0342	
C34	0.5541 (3)	0.3617 (2)	0.93692 (19)	0.0336	
C35	0.6509 (3)	0.39003 (19)	0.87025 (18)	0.0289	
C36	0.7091 (3)	0.1318 (2)	1.0415 (2)	0.0418	
C37	0.4317 (3)	0.4200 (2)	0.9504 (2)	0.0406	
C38	0.7665 (3)	0.45829 (18)	0.74766 (18)	0.0294	
C39	0.8086 (3)	0.55284 (19)	0.68906 (18)	0.0311	
C40	0.8384 (3)	0.62864 (19)	0.72886 (19)	0.0329	
C41	0.9478 (3)	0.6686 (2)	0.6995 (2)	0.0428	
C42	0.9702 (3)	0.7369 (2)	0.7376 (3)	0.0536	
C43	0.8824 (3)	0.7654 (2)	0.8041 (2)	0.0528	
C44	0.7749 (3)	0.7260 (2)	0.8286 (2)	0.0429	
C45	0.9204 (3)	0.5227 (2)	0.62126 (19)	0.0372	
C46	0.7103 (3)	0.6012 (2)	0.64285 (18)	0.0323	
C47	0.5060 (3)	0.69239 (19)	0.66532 (18)	0.0307	
C48	0.4053 (3)	0.72876 (19)	0.72614 (19)	0.0328	
C49	0.3128 (3)	0.7954 (2)	0.7067 (2)	0.0366	
C50	0.3128 (3)	0.8325 (2)	0.6266 (2)	0.0386	
C51	0.4093 (3)	0.7973 (2)	0.5678 (2)	0.0403	
C52	0.5054 (3)	0.7288 (2)	0.58290 (19)	0.0331	
C53	0.4016 (3)	0.6961 (2)	0.8141 (2)	0.0441	
C54	0.2146 (3)	0.9079 (3)	0.6061 (3)	0.0564	
C55	0.5987 (3)	0.6981 (2)	0.50957 (19)	0.0419	
C56	0.7084 (6)	−0.1091 (4)	0.6073 (4)	0.1115	
C57	0.6320 (6)	−0.0917 (3)	0.6879 (3)	0.1025	
C58	0.4671 (12)	0.0682 (10)	0.6657 (6)	0.0516	0.5000
C59	0.3535 (8)	0.0419 (6)	0.7316 (7)	0.0633	0.5000
C62	0.4962 (9)	0.0113 (9)	0.6502 (7)	0.0654	0.5000
C63	0.4024 (17)	0.0866 (12)	0.6824 (13)	0.0971	0.5000
O2	1.0339	0.4814	−0.0072	0.0500*	0.0000
C60	1.0944	0.5482	−0.0728	0.0500*	0.0000
C61	1.0743	0.6558	−0.0664	0.0500*	0.0000
C64	0.9068	0.4760	0.0202	0.0500*	0.0000
C65	0.8849	0.3908	0.0907	0.0500*	0.0000
H2	0.7784	0.3979	0.5445	0.0500*	
H3	0.1393	0.9136	0.6524	0.0500*	
H4	0.6230	0.6246	0.5197	0.0500*	
H5	0.5641	0.7150	0.4590	0.0500*	
H6	0.6738	0.7308	0.4965	0.0500*	
H7	0.6550	0.4579	0.5033	0.0500*	
H8	0.2348	0.5183	0.6531	0.0500*	
H9	0.3037	0.5916	0.6578	0.0500*	
H11	0.9210	0.0627	0.5451	0.0536*	
H12	1.0462	0.0697	0.5671	0.0536*	
H13	0.9853	0.1620	0.5144	0.0536*	
H14	0.3726	0.3858	0.9984	0.0500*	

H15	0.8173	−0.1822	1.1252	0.0500*
H16	1.0859	0.0451	0.8621	0.0500*
H17	0.3840	0.4291	0.9027	0.0500*
H18	1.0834	0.0026	0.7819	0.0500*
H19	1.0012	0.1168	0.8026	0.0500*
H20	0.6970	0.3352	0.5056	0.0500*
H21	0.5360	−0.0650	0.9467	0.0500*
H22	0.2270	0.5283	0.7460	0.0500*
H23	0.9468	−0.2157	1.0649	0.0500*
H24	0.4415	0.4958	0.9506	0.0500*
H25	0.6307	0.1233	1.0888	0.0500*
H27	0.8426	−0.2703	1.0604	0.0500*
H28	0.5585	0.0456	0.8936	0.0500*
H29	0.5907	−0.0419	0.8392	0.0500*
H30	0.7438	0.0698	1.0140	0.0500*
H31	0.9541	0.0251	0.7156	0.0406*
H32	0.8423	0.0041	0.6851	0.0406*
H33	0.4032	0.2499	0.8727	0.0500*
H34	0.4821	0.7152	0.8279	0.0500*
H35	0.4066	0.6265	0.8286	0.0500*
H36	0.4587	0.1634	0.8022	0.0500*
H37	0.5468	0.2199	0.8409	0.0500*
H38	0.3246	0.7150	0.8502	0.0500*
H61	0.6601	−0.1414	1.0136	0.0449*
H81	1.0167	−0.0948	0.9532	0.0460*
H131	0.7949	0.2596	0.5815	0.0489*
H132	0.7446	0.1568	0.5905	0.0489*
H161	0.3154	0.3778	0.7989	0.0422*
H181	0.4909	0.5057	0.5715	0.0431*
H241	1.1391	0.1708	0.5910	0.0527*
H251	1.2333	0.2673	0.6451	0.0562*
H261	1.1113	0.3671	0.7412	0.0508*
H291	0.8572	0.4889	0.8686	0.0517*
H292	0.9658	0.4000	0.8691	0.0517*
H293	0.9781	0.4870	0.7914	0.0517*
H311	0.8672	0.2153	0.9075	0.0452*
H331	0.5075	0.2565	1.0380	0.0456*
H361	0.7762	0.1443	1.0670	0.0499*
H381	0.7529	0.4177	0.7116	0.0368*
H411	1.0098	0.6482	0.6505	0.0570*
H421	1.0492	0.7649	0.7174	0.0701*
H431	0.8972	0.8133	0.8335	0.0671*
H441	0.7103	0.7476	0.8760	0.0630*
H451	0.8997	0.4733	0.5959	0.0473*
H452	0.9898	0.4948	0.6448	0.0473*
H453	0.9438	0.5800	0.5790	0.0473*
H461	0.7389	0.6618	0.6075	0.0432*
H462	0.7042	0.5556	0.6082	0.0432*

H491	0.2438	0.8175	0.7517	0.0476*	
H511	0.4117	0.8228	0.5097	0.0507*	
H541	0.2496	0.9752	0.5874	0.0673*	
H542	0.1897	0.8903	0.5557	0.0673*	
H561	0.7410	−0.1786	0.6082	0.1308*	
H562	0.7775	−0.0692	0.5905	0.1308*	
H563	0.6584	−0.0896	0.5676	0.1308*	
H571	0.6828	−0.1120	0.7270	0.1343*	
H572	0.5637	−0.1324	0.7041	0.1343*	
H581	0.4735	0.1387	0.6566	0.0638*	0.5000
H582	0.4593	0.0502	0.6153	0.0638*	0.5000
H591	0.2766	0.0772	0.7205	0.0995*	0.5000
H592	0.3638	0.0596	0.7814	0.0995*	0.5000
H593	0.3496	−0.0289	0.7402	0.0995*	0.5000
H621	0.4681	−0.0527	0.6555	0.0938*	0.5000
H622	0.5282	0.0379	0.5914	0.0938*	0.5000
H631	0.3333	0.0974	0.6573	0.1803*	0.5000
H632	0.3737	0.0587	0.7412	0.1803*	0.5000
H633	0.4338	0.1493	0.6771	0.1803*	0.5000

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Li1	0.032 (3)	0.027 (2)	0.038 (3)	−0.0029 (19)	−0.009 (2)	−0.006 (2)
Li2	0.041 (3)	0.028 (2)	0.051 (3)	0.001 (2)	−0.022 (3)	−0.005 (2)
Li3	0.036 (3)	0.037 (3)	0.034 (3)	−0.003 (2)	−0.011 (2)	−0.008 (2)
Li4	0.037 (3)	0.033 (2)	0.039 (3)	−0.006 (2)	−0.011 (2)	−0.005 (2)
N1	0.0310 (14)	0.0261 (12)	0.0344 (14)	−0.0009 (10)	−0.0110 (11)	−0.0045 (10)
N2	0.0271 (13)	0.0293 (12)	0.0337 (14)	0.0035 (10)	−0.0085 (11)	−0.0066 (10)
N3	0.0288 (13)	0.0233 (11)	0.0394 (15)	−0.0019 (10)	−0.0083 (11)	−0.0045 (10)
N4	0.0287 (13)	0.0277 (12)	0.0314 (14)	−0.0026 (10)	−0.0084 (11)	−0.0018 (10)
N5	0.0372 (15)	0.0293 (12)	0.0420 (16)	−0.0036 (10)	−0.0147 (13)	−0.0082 (11)
N6	0.0296 (14)	0.0301 (12)	0.0312 (14)	−0.0012 (10)	−0.0065 (11)	−0.0047 (10)
O1	0.0483 (15)	0.0715 (16)	0.0548 (15)	−0.0224 (12)	−0.0179 (12)	−0.0106 (12)
C1	0.0414 (19)	0.0418 (18)	0.0404 (19)	0.0116 (14)	−0.0080 (15)	−0.0138 (15)
C2	0.0333 (17)	0.0318 (15)	0.0331 (17)	0.0064 (12)	−0.0036 (14)	−0.0085 (13)
C3	0.0353 (17)	0.0281 (14)	0.0353 (17)	0.0014 (12)	−0.0109 (14)	−0.0095 (12)
C4	0.0344 (17)	0.0192 (13)	0.0368 (17)	−0.0005 (11)	−0.0132 (14)	−0.0061 (12)
C5	0.0296 (16)	0.0282 (14)	0.0348 (17)	−0.0026 (12)	−0.0130 (13)	−0.0034 (13)
C6	0.0330 (17)	0.0324 (15)	0.0381 (18)	−0.0073 (12)	−0.0091 (14)	−0.0048 (13)
C7	0.0394 (19)	0.0337 (16)	0.0384 (18)	−0.0050 (13)	−0.0177 (15)	0.0004 (13)
C8	0.0328 (17)	0.0354 (16)	0.046 (2)	0.0019 (13)	−0.0200 (15)	−0.0066 (14)
C9	0.0334 (17)	0.0266 (14)	0.0402 (18)	−0.0013 (12)	−0.0114 (14)	−0.0075 (13)
C10	0.0349 (18)	0.0439 (18)	0.0414 (19)	−0.0086 (14)	−0.0158 (15)	0.0066 (14)
C11	0.041 (2)	0.054 (2)	0.059 (2)	−0.0085 (16)	−0.0257 (18)	0.0129 (17)
C12	0.0335 (18)	0.0427 (17)	0.049 (2)	−0.0081 (14)	−0.0136 (15)	−0.0076 (15)
C13	0.0383 (18)	0.0425 (17)	0.0333 (18)	0.0077 (14)	−0.0114 (15)	−0.0079 (14)
C14	0.0244 (15)	0.0296 (14)	0.0271 (15)	−0.0028 (11)	−0.0091 (13)	−0.0079 (12)

C15	0.0278 (16)	0.0297 (14)	0.0352 (17)	−0.0028 (12)	−0.0112 (13)	−0.0027 (12)
C16	0.0234 (15)	0.0339 (15)	0.0350 (17)	−0.0051 (12)	−0.0035 (13)	−0.0063 (13)
C17	0.0258 (16)	0.0299 (14)	0.0351 (17)	−0.0009 (11)	−0.0130 (13)	−0.0061 (13)
C18	0.0332 (17)	0.0302 (14)	0.0317 (17)	−0.0024 (12)	−0.0156 (14)	−0.0042 (12)
C19	0.0266 (15)	0.0341 (15)	0.0297 (17)	−0.0053 (12)	−0.0069 (13)	−0.0090 (12)
C20	0.0326 (17)	0.0383 (16)	0.0370 (18)	0.0008 (13)	−0.0040 (14)	0.0008 (14)
C21	0.0319 (17)	0.0364 (16)	0.0452 (19)	−0.0013 (13)	−0.0137 (15)	−0.0040 (14)
C22	0.0410 (19)	0.0354 (16)	0.0403 (19)	0.0015 (13)	−0.0070 (15)	−0.0042 (14)
C23	0.0320 (17)	0.0266 (14)	0.0343 (17)	0.0003 (12)	−0.0020 (13)	−0.0024 (12)
C24	0.0308 (18)	0.0443 (18)	0.045 (2)	0.0000 (14)	−0.0007 (15)	−0.0099 (15)
C25	0.0266 (17)	0.0456 (19)	0.059 (2)	−0.0045 (14)	−0.0026 (16)	−0.0038 (16)
C26	0.0297 (18)	0.0354 (16)	0.054 (2)	−0.0060 (13)	−0.0084 (15)	−0.0003 (15)
C27	0.0263 (16)	0.0259 (14)	0.0416 (18)	−0.0018 (11)	−0.0069 (13)	−0.0015 (12)
C28	0.0271 (16)	0.0297 (15)	0.0396 (18)	−0.0029 (11)	−0.0123 (13)	−0.0071 (12)
C29	0.0381 (18)	0.0344 (16)	0.050 (2)	−0.0056 (13)	−0.0214 (15)	−0.0078 (14)
C30	0.0330 (17)	0.0286 (14)	0.0300 (16)	−0.0047 (12)	−0.0105 (13)	−0.0060 (12)
C31	0.0325 (17)	0.0314 (15)	0.0398 (18)	0.0006 (12)	−0.0162 (14)	−0.0088 (13)
C32	0.0400 (18)	0.0309 (15)	0.0343 (18)	−0.0066 (13)	−0.0135 (14)	−0.0023 (13)
C33	0.0326 (17)	0.0389 (16)	0.0305 (17)	−0.0032 (13)	−0.0078 (14)	−0.0060 (13)
C34	0.0292 (16)	0.0345 (16)	0.0373 (18)	−0.0029 (12)	−0.0104 (14)	−0.0050 (13)
C35	0.0288 (16)	0.0291 (14)	0.0315 (16)	−0.0020 (12)	−0.0118 (13)	−0.0069 (12)
C36	0.047 (2)	0.0354 (16)	0.0431 (19)	−0.0047 (14)	−0.0140 (16)	−0.0038 (14)
C37	0.0318 (17)	0.0447 (17)	0.0382 (19)	0.0004 (13)	−0.0059 (15)	0.0001 (14)
C38	0.0301 (16)	0.0250 (14)	0.0354 (17)	−0.0021 (11)	−0.0107 (13)	−0.0083 (12)
C39	0.0272 (16)	0.0314 (15)	0.0341 (17)	−0.0045 (12)	−0.0065 (13)	−0.0064 (12)
C40	0.0323 (17)	0.0269 (14)	0.0395 (18)	−0.0039 (12)	−0.0107 (15)	−0.0037 (13)
C41	0.0385 (19)	0.0363 (17)	0.052 (2)	−0.0099 (14)	−0.0085 (16)	−0.0059 (15)
C42	0.051 (2)	0.0421 (18)	0.075 (3)	−0.0195 (16)	−0.019 (2)	−0.0118 (18)
C43	0.061 (2)	0.0404 (19)	0.067 (3)	−0.0123 (16)	−0.021 (2)	−0.0205 (17)
C44	0.052 (2)	0.0334 (16)	0.050 (2)	−0.0039 (14)	−0.0206 (17)	−0.0133 (14)
C45	0.0362 (18)	0.0362 (16)	0.0382 (18)	−0.0042 (13)	−0.0079 (14)	−0.0070 (13)
C46	0.0360 (17)	0.0303 (15)	0.0309 (16)	−0.0041 (12)	−0.0092 (14)	−0.0057 (12)
C47	0.0340 (17)	0.0254 (14)	0.0353 (17)	−0.0077 (12)	−0.0120 (14)	−0.0037 (12)
C48	0.0338 (17)	0.0289 (14)	0.0360 (18)	−0.0036 (12)	−0.0106 (14)	−0.0047 (12)
C49	0.0308 (17)	0.0328 (15)	0.044 (2)	−0.0011 (12)	−0.0072 (14)	−0.0072 (14)
C50	0.0370 (18)	0.0355 (16)	0.043 (2)	−0.0035 (13)	−0.0144 (16)	−0.0021 (14)
C51	0.046 (2)	0.0411 (17)	0.0356 (19)	−0.0091 (14)	−0.0176 (16)	0.0027 (14)
C52	0.0317 (17)	0.0326 (15)	0.0347 (18)	−0.0049 (12)	−0.0111 (14)	−0.0011 (13)
C53	0.0426 (19)	0.0457 (18)	0.0372 (19)	0.0100 (14)	−0.0078 (15)	−0.0088 (14)
C54	0.049 (2)	0.057 (2)	0.058 (2)	0.0109 (17)	−0.0210 (19)	−0.0021 (18)
C55	0.0450 (19)	0.0490 (19)	0.0307 (18)	−0.0052 (15)	−0.0106 (15)	−0.0036 (14)
C56	0.135 (5)	0.091 (4)	0.108 (4)	−0.057 (3)	0.010 (4)	−0.045 (3)
C57	0.165 (5)	0.056 (3)	0.077 (3)	−0.061 (3)	0.017 (3)	−0.027 (2)
C58	0.065 (7)	0.051 (7)	0.044 (5)	−0.013 (6)	−0.018 (5)	−0.009 (4)
C59	0.042 (5)	0.055 (5)	0.092 (7)	−0.021 (4)	−0.021 (5)	0.005 (5)
C62	0.067 (6)	0.062 (5)	0.083 (6)	−0.011 (5)	−0.051 (5)	0.002 (5)
C63	0.093 (12)	0.076 (9)	0.134 (14)	0.023 (9)	−0.053 (11)	−0.038 (8)

Geometric parameters (Å, º)

N1—C3	1.466 (4)	C30—C35	1.412 (4)
N1—C4	1.404 (4)	C31—C32	1.392 (4)
N2—C13	1.470 (4)	C31—H311	1.000
N2—C14	1.359 (3)	C32—C33	1.401 (4)
N3—C23	1.339 (4)	C32—C36	1.514 (4)
N3—C27	1.351 (4)	C33—C34	1.406 (4)
N4—C35	1.380 (4)	C33—H331	1.000
N4—C38	1.485 (4)	C34—C35	1.402 (4)
N5—C40	1.354 (4)	C34—C37	1.500 (4)
N5—C44	1.350 (4)	C36—H25	1.037
N6—C46	1.453 (4)	C36—H30	1.058
N6—C47	1.388 (4)	C36—H361	1.060
O1—C57	1.441 (5)	C37—H14	1.009
O1—C58	1.600 (14)	C37—H17	1.107
O1—C62	1.388 (9)	C37—H24	1.109
C1—C2	1.541 (4)	C38—C39	1.573 (4)
C1—H11	1.000	C38—H381	1.000
C1—H12	1.000	C39—C40	1.535 (4)
C1—H13	1.000	C39—C45	1.542 (4)
C2—C3	1.564 (4)	C39—C46	1.577 (4)
C2—C13	1.565 (4)	C40—C41	1.393 (4)
C2—C23	1.518 (4)	C41—C42	1.386 (5)
C3—H31	1.000	C41—H411	1.000
C3—H32	1.000	C42—C43	1.380 (6)
C4—C5	1.414 (4)	C42—H421	1.000
C4—C9	1.430 (4)	C43—C44	1.370 (5)
C5—C6	1.391 (4)	C43—H431	1.000
C5—C10	1.503 (4)	C44—H441	1.000
C6—C7	1.388 (4)	C45—H451	1.000
C6—H61	1.000	C45—H452	1.000
C7—C8	1.377 (4)	C45—H453	1.000
C7—C11	1.517 (5)	C46—H461	1.000
C8—C9	1.401 (4)	C46—H462	1.000
C8—H81	1.000	C47—C48	1.431 (4)
C9—C12	1.502 (4)	C47—C52	1.430 (4)
C10—H21	1.041	C48—C49	1.385 (4)
C10—H28	1.000	C48—C53	1.503 (5)
C10—H29	1.070	C49—C50	1.392 (5)
C11—H15	1.057	C49—H491	1.000
C11—H23	0.998	C50—C51	1.379 (5)
C11—H27	1.057	C50—C54	1.504 (5)
C12—H16	1.065	C51—C52	1.406 (4)
C12—H18	1.010	C51—H511	1.000
C12—H19	1.112	C52—C55	1.501 (5)
C13—H131	1.000	C53—H34	1.119
C13—H132	1.000	C53—H35	0.972

C14—C15	1.439 (4)	C53—H38	0.961
C14—C19	1.450 (4)	C54—H3	1.003
C15—C16	1.388 (4)	C54—H541	1.060
C15—C20	1.513 (4)	C54—H542	1.090
C16—C17	1.391 (4)	C55—H4	1.034
C16—H161	1.000	C55—H5	1.045
C17—C18	1.387 (4)	C55—H6	1.004
C17—C21	1.512 (4)	C56—C57	1.470 (7)
C18—C19	1.408 (4)	C56—H561	1.000
C18—H181	1.000	C56—H562	1.000
C19—C22	1.518 (4)	C56—H563	1.000
C20—H33	1.059	C57—H571	1.000
C20—H36	1.112	C57—H572	1.000
C20—H37	0.993	C58—C59	1.514 (15)
C21—H8	0.923	C58—H581	1.000
C21—H9	0.950	C58—H582	1.000
C21—H22	1.046	C59—H591	1.000
C22—H2	1.096	C59—H592	1.000
C22—H7	1.056	C59—H593	1.000
C22—H20	1.060	C62—C63	1.467 (16)
C23—C24	1.408 (4)	C62—H621	1.000
C24—C25	1.373 (5)	C62—H622	1.000
C24—H241	1.000	C63—H631	1.000
C25—C26	1.382 (5)	C63—H632	1.000
C25—H251	1.000	C63—H633	1.000
C26—C27	1.387 (4)	O2—C60^{i}	1.7553 (7)
C26—H261	1.000	O2—C64^{i}	0.9421 (3)
C27—C28	1.512 (4)	O2—O2^{i}	0.8722 (3)
C28—C29	1.536 (4)	O2—C60	1.4205 (6)
C28—C30	1.531 (4)	O2—C64	1.4197 (3)
C28—C38	1.599 (4)	C60—C65^{i}	0.9122 (3)
C29—H291	1.000	C60—C64^{i}	0.9092 (3)
C29—H292	1.000	C60—C61	1.5398 (3)
C29—H293	1.000	C61—C65^{i}	0.8594 (3)
C30—C31	1.383 (4)	C64—C65	1.5396 (6)

C3—N1—C4	115.8 (2)	H25—C36—H30	113.529
C13—N2—C14	122.3 (2)	C32—C36—H361	106.537
C23—N3—C27	120.7 (3)	H25—C36—H361	106.592
C35—N4—C38	105.6 (2)	H30—C36—H361	106.592
C40—N5—C44	118.6 (3)	C34—C37—H14	109.350
C46—N6—C47	120.6 (2)	C34—C37—H17	117.537
C57—O1—C58	130.7 (6)	H14—C37—H17	101.091
C57—O1—C62	97.6 (6)	C34—C37—H24	109.678
C2—C1—H11	109.446	H14—C37—H24	118.077
C2—C1—H12	109.615	H17—C37—H24	101.108
H11—C1—H12	109.476	N4—C38—C28	105.9 (2)
C2—C1—H13	109.339	N4—C38—C39	116.2 (2)

H11—C1—H13	109.476	C28—C38—C39	119.0 (2)
H12—C1—H13	109.476	N4—C38—H381	104.737
C1—C2—C3	106.2 (2)	C28—C38—H381	104.926
C1—C2—C13	104.9 (3)	C39—C38—H381	104.631
C3—C2—C13	114.8 (3)	C38—C39—C40	115.8 (2)
C1—C2—C23	110.3 (3)	C38—C39—C45	107.2 (2)
C3—C2—C23	108.9 (2)	C40—C39—C45	110.0 (2)
C13—C2—C23	111.5 (2)	C38—C39—C46	110.3 (2)
C2—C3—N1	116.2 (2)	C40—C39—C46	108.7 (2)
C2—C3—H31	107.856	C45—C39—C46	104.1 (2)
N1—C3—H31	107.572	C39—C40—N5	117.4 (2)
C2—C3—H32	107.739	C39—C40—C41	122.3 (3)
N1—C3—H32	107.888	N5—C40—C41	120.2 (3)
H31—C3—H32	109.467	C40—C41—C42	120.1 (3)
N1—C4—C5	119.2 (2)	C40—C41—H411	119.683
N1—C4—C9	124.4 (3)	C42—C41—H411	120.255
C5—C4—C9	116.3 (3)	C41—C42—C43	119.3 (3)
C4—C5—C6	121.8 (3)	C41—C42—H421	120.416
C4—C5—C10	120.6 (2)	C43—C42—H421	120.315
C6—C5—C10	117.7 (3)	C42—C43—C44	118.1 (3)
C5—C6—C7	121.9 (3)	C42—C43—H431	120.785
C5—C6—H61	118.912	C44—C43—H431	121.118
C7—C6—H61	119.160	C43—C44—N5	123.6 (3)
C6—C7—C8	116.7 (3)	C43—C44—H441	118.411
C6—C7—C11	120.9 (3)	N5—C44—H441	117.944
C8—C7—C11	122.3 (3)	C39—C45—H451	109.124
C7—C8—C9	123.8 (3)	C39—C45—H452	109.650
C7—C8—H81	118.020	H451—C45—H452	109.475
C9—C8—H81	118.135	C39—C45—H453	109.625
C4—C9—C8	119.3 (3)	H451—C45—H453	109.477
C4—C9—C12	122.2 (3)	H452—C45—H453	109.475
C8—C9—C12	118.4 (3)	C39—C46—N6	115.0 (2)
C5—C10—H21	104.472	C39—C46—H461	108.127
C5—C10—H28	110.553	N6—C46—H461	108.328
H21—C10—H28	110.838	C39—C46—H462	107.732
C5—C10—H29	110.748	N6—C46—H462	108.089
H21—C10—H29	114.436	H461—C46—H462	109.467
H28—C10—H29	105.877	N6—C47—C48	115.0 (3)
C7—C11—H15	117.562	N6—C47—C52	129.0 (3)
C7—C11—H23	109.217	C48—C47—C52	116.0 (3)
H15—C11—H23	103.878	C47—C48—C49	122.3 (3)
C7—C11—H27	108.389	C47—C48—C53	119.0 (3)
H15—C11—H27	116.128	C49—C48—C53	118.7 (3)
H23—C11—H27	99.862	C48—C49—C50	122.0 (3)
C9—C12—H16	111.273	C48—C49—H491	118.971
C9—C12—H18	108.312	C50—C49—H491	119.020
H16—C12—H18	105.104	C49—C50—C51	115.9 (3)
C9—C12—H19	112.757	C49—C50—C54	121.7 (3)

H16—C12—H19	103.679	C51—C50—C54	122.4 (3)
H18—C12—H19	115.387	C50—C51—C52	125.2 (3)
C2—C13—N2	117.8 (3)	C50—C51—H511	117.676
C2—C13—H131	107.901	C52—C51—H511	117.154
N2—C13—H131	107.427	C47—C52—C51	118.6 (3)
C2—C13—H132	107.130	C47—C52—C55	125.2 (3)
N2—C13—H132	106.950	C51—C52—C55	116.2 (3)
H131—C13—H132	109.467	C48—C53—H34	111.414
N2—C14—C15	115.7 (2)	C48—C53—H35	111.663
N2—C14—C19	130.6 (3)	H34—C53—H35	104.529
C15—C14—C19	113.7 (2)	C48—C53—H38	112.697
C14—C15—C16	123.3 (3)	H34—C53—H38	114.636
C14—C15—C20	119.1 (2)	H35—C53—H38	101.133
C16—C15—C20	117.5 (3)	C50—C54—H3	113.934
C15—C16—C17	122.5 (3)	C50—C54—H541	108.541
C15—C16—H161	118.754	H3—C54—H541	108.679
C17—C16—H161	118.765	C50—C54—H542	108.587
C16—C17—C18	115.6 (3)	H3—C54—H542	108.312
C16—C17—C21	122.1 (3)	H541—C54—H542	108.679
C18—C17—C21	122.3 (3)	C52—C55—H4	110.286
C17—C18—C19	124.8 (3)	C52—C55—H5	110.507
C17—C18—H181	117.857	H4—C55—H5	108.207
C19—C18—H181	117.376	C52—C55—H6	111.175
C14—C19—C18	120.0 (3)	H4—C55—H6	108.320
C14—C19—C22	124.6 (2)	H5—C55—H6	108.254
C18—C19—C22	115.4 (3)	C57—C56—H561	111.023
C15—C20—H33	111.311	C57—C56—H562	108.617
C15—C20—H36	105.509	H561—C56—H562	109.476
H33—C20—H36	114.811	C57—C56—H563	108.749
C15—C20—H37	109.936	H561—C56—H563	109.476
H33—C20—H37	102.833	H562—C56—H563	109.476
H36—C20—H37	112.531	C56—C57—O1	114.9 (4)
C17—C21—H8	108.560	C56—C57—H571	107.950
C17—C21—H9	111.728	O1—C57—H571	107.987
H8—C21—H9	101.456	C56—C57—H572	107.818
C17—C21—H22	106.811	O1—C57—H572	108.603
H8—C21—H22	111.941	H571—C57—H572	109.467
H9—C21—H22	116.180	O1—C58—C59	107.9 (8)
C19—C22—H2	114.440	O1—C58—H581	111.049
C19—C22—H7	101.608	C59—C58—H581	111.346
H2—C22—H7	113.884	O1—C58—H582	111.158
C19—C22—H20	107.382	C59—C58—H582	105.834
H2—C22—H20	108.913	H581—C58—H582	109.467
H7—C22—H20	110.282	C58—C59—H591	113.835
C2—C23—N3	115.3 (2)	C58—C59—H592	104.478
C2—C23—C24	124.7 (3)	H591—C59—H592	109.476
N3—C23—C24	120.0 (3)	C58—C59—H593	109.947
C23—C24—C25	119.1 (3)	H591—C59—H593	109.476

C23—C24—H241	120.173	H592—C59—H593	109.476
C25—C24—H241	120.773	O1—C62—C63	102.9 (10)
C24—C25—C26	120.4 (3)	O1—C62—H621	112.365
C24—C25—H251	119.507	C63—C62—H621	115.945
C26—C25—H251	120.074	O1—C62—H622	111.383
C25—C26—C27	118.2 (3)	C63—C62—H622	104.364
C25—C26—H261	121.011	H621—C62—H622	109.467
C27—C26—H261	120.744	C62—C63—H631	111.868
C26—C27—N3	121.3 (3)	C62—C63—H632	102.419
C26—C27—C28	124.7 (3)	H631—C63—H632	109.476
N3—C27—C28	114.0 (2)	C62—C63—H633	113.860
C27—C28—C29	112.7 (2)	H631—C63—H633	109.476
C27—C28—C30	111.6 (2)	H632—C63—H633	109.476
C29—C28—C30	107.4 (2)	C60^{i}—O2—C64^{i}	137.69 (2)
C27—C28—C38	111.6 (2)	C60^{i}—O2—O2^{i}	53.44 (3)
C29—C28—C38	114.0 (2)	C64^{i}—O2—O2^{i}	102.91 (3)
C30—C28—C38	98.6 (2)	C60^{i}—O2—C60	150.449 (18)
C28—C29—H291	109.328	C64^{i}—O2—C60	39.05 (2)
C28—C29—H292	109.616	O2^{i}—O2—C60	97.01 (4)
H291—C29—H292	109.475	C60^{i}—O2—C64	31.049 (14)
C28—C29—H293	109.456	C64^{i}—O2—C64	143.213 (15)
H291—C29—H293	109.476	O2^{i}—O2—C64	40.300 (13)
H292—C29—H293	109.476	C60—O2—C64	126.18 (3)
C28—C30—C31	129.7 (3)	O2—C60—O2^{i}	29.551 (17)
C28—C30—C35	107.9 (2)	O2—C60—C65^{i}	147.255 (17)
C31—C30—C35	121.9 (3)	O2^{i}—C60—C65^{i}	123.59 (3)
C30—C31—C32	120.3 (3)	O2—C60—C64^{i}	40.746 (16)
C30—C31—H311	119.736	O2^{i}—C60—C64^{i}	53.643 (19)
C32—C31—H311	119.946	C65^{i}—C60—C64^{i}	115.40 (3)
C31—C32—C33	117.5 (3)	O2—C60—C61	119.78 (3)
C31—C32—C36	120.9 (3)	O2^{i}—C60—C61	94.97 (4)
C33—C32—C36	121.6 (3)	C65^{i}—C60—C61	28.684 (13)
C32—C33—C34	123.7 (3)	C64^{i}—C60—C61	98.22 (3)
C32—C33—H331	118.299	C60—C61—C65^{i}	30.627 (18)
C34—C33—H331	117.999	O2—C64—O2^{i}	36.79 (2)
C33—C34—C35	117.5 (3)	O2—C64—C60^{i}	95.31 (3)
C33—C34—C37	121.6 (3)	O2^{i}—C64—C60^{i}	100.21 (3)
C35—C34—C37	120.9 (3)	O2—C64—C65	107.28 (4)
C30—C35—C34	119.0 (3)	O2^{i}—C64—C65	127.64 (2)
C30—C35—N4	114.1 (2)	C60^{i}—C64—C65	32.357 (16)
C34—C35—N4	126.9 (2)	C64—C65—C60^{i}	32.240 (13)
C32—C36—H25	110.027	C64—C65—C61^{i}	100.65 (4)
C32—C36—H30	113.047	C60^{i}—C65—C61^{i}	120.69 (3)

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