

Supporting Information

Catalytic Boracarboxylation of Alkynes with Diborane and Carbon Dioxide by an N-Heterocyclic Carbene Copper Catalyst

Liang Zhang, Jianhua Cheng, Béatrice Carry, and Zhaomin Hou*

Organometallic Chemistry Laboratory and Advanced Catalyst Research Team, RIKEN Advanced
Science Institute, 2-1 Hirosawa, Wako, Saitama 351-0198, Japan

Table of Contents

General Information	S2
Synthetic Procedures	S2
Spectral Data	S3
X-ray Data for 1	S8
X-ray Data for 2	S18
X-ray Data for Complex 3	S28
X-ray Data for Complex 4k	S37
References	S63
Copies of NMR Spectra	S63

General Information

Unless otherwise noted, all manipulations were performed under a dry nitrogen atmosphere using Schlenk-line techniques or under a nitrogen atmosphere in an Mbraun glovebox. Nitrogen gas was purified by being passed through a Dryclean column (4Å molecular sieves, Nikka Seiko Co.) and a Gasclean GC-RX column (Nikka Seiko Co.).

The NMR spectra were recorded on a JEOL AL-300, JEOL AL-400 or JEOL ECS-400 spectrometer. Elemental analyses were conducted by a MICRO CORDER JM10 instrument.

Carbon dioxide and commercially available reagents were used without further purification unless otherwise stated. THF was dried by Mbraun SPS-800 solvent purification system and stored over fresh Na chip. Diaryl alkynes¹ and (3,3-dimethyl-1-butynyl)benzen² were prepared by literature methods. [(IPr)CuCl], [(IMes)CuCl], [(SIMes)CuCl],³ and [(IPr)Cu(OtBu)]⁴ were prepared according to literature.

Synthetic Procedures

Synthesis of Complex 1

A solution of [(IPr)Cu(OtBu)] (0.2855 mmol, 150 mg) and B₂pin₂ (0.287 mmol, 73 mg) in 2 mL hexane was stirred at room temperature for 10 min. After diphenylacetylene (0.314 mmol, 60 mg) was added, the solution was stirred at room temperature for additional 20 min. The reaction mixture was filtrated and washed with hexane to give complex **1** as a white powder (198 mg, 92%). Single crystals suitable for X-ray analysis were obtained by cooling a benzene/hexane solution at -30 °C.

Synthesis of Complex 2

Carbon dioxide (1 atm) was introduced to a solution of complex **1** (75.7 mg, 0.1 mmol) in benzene (2 mL). After the reaction mixture was stirred at room temperature for 1 h, the precipitate formed was filtrated and washed with hexane to give complex **2** as a white solid (76.5 mg, 95%). Single crystals suitable for X-ray analysis were obtained from slow evaporation of a THF/hexane solution at room temperature.

The Reaction of Complex 2 and LiOtBu

LiOtBu (0.125 mmol, 10 mg) was added into a THF (2 mL) solution of complex **2** (0.125 mmol, 100 mg) at room temperature. After it was stirred for 15 min, THF was evaporated. The reaction mixture was extracted with C₆D₆, and the residual solid was dissolved in THF-*d*₈. The formation of [(IPr)Cu(OtBu)] was confirmed by NMR analysis of the C₆D₆ solution and X-ray analysis of single crystals obtained from the same solution.⁴ The formation of **3** was confirmed by NMR analysis of the THF-*d*₈ solution. Single crystals of **3** suitable for X-ray analysis were obtained by cooling a THF/hexane solution at -30 °C.

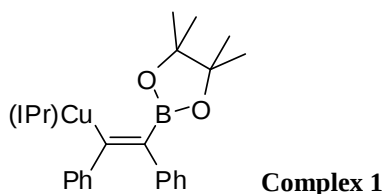
Typical Procedure for the Borylation/Carboxylation of Diphenylacetylene

In a glovebox, [(SImes)CuCl] (5 mol%, 10.0 mg), B₂pin₂ (0.5 mmol, 126.0 mg), diphenylacetylene (0.5 mmol, 90.0 mg), LiOtBu (1.1 equiv, 44.0 mg), and THF (3 mL) were added into a 30-mL Schlenk tube equipped with a magnetic stirring bar and a Teflon cap. After the solution was stirred at room temperature for 5 min, the sealed reaction tube was taken out of the glovebox. The reaction mixture was subjected to vacuum for a while, CO₂ (1 atm) was then introduced into the reaction tube. The sealed Schlenk tube was stirred in an oil bath at 80 °C for 14 h. After the reaction mixture was cooled to room temperature, it was diluted with 5 mL of THF and filtrated. The filtrate was concentrated to 1.5 mL under vacuum. And 0.5 mL of benzene was added, followed by 20 mL of hexane. The product **4a** was obtained as a white powder (144.7 mg, 81%) after recrystallization.

Suzuki-Miyaura Cross-Coupling of **4h**

Pd(OAc)₂ (5%, 6.7 mg), PtBu₃ (20%, 24.3 mg), Cs₂CO₃ (3.0 equiv, 586.5 mg, in 0.36 mL H₂O), iodobenzene (1.5 equiv, 183.6 mg), **4h** (0.6 mmol, 176.5 mg), and dioxane (2.0 mL) were added into a 30-mL Schlenk tube equipped with a magnetic stirring bar and a Teflon cap. After the reaction mixture was stirred at 100 °C for 6 h, EtOAc (10 mL) and 3 M HCl (5 mL) were added. The aqueous phase was extracted with EtOAc (10 mL×5). The organic phase was dried over Na₂SO₄. The product **5** was obtained in 45% yield (64.5 mg) after silica gel flash chromatography.⁵

Spectral Data

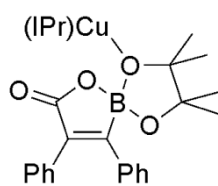


¹H NMR (300 MHz, C₆D₆) δ 0.93 (s, 12H), 1.06 (d, *J* = 6.9 Hz, 12H), 1.32 (d, *J* = 6.8 Hz, 12H), 2.63-2.77 (m, 4H), 6.26 (s, 2H), 6.46 (d, *J* = 7.0 Hz, 2H), 6.67-6.82 (m, 4H), 6.98 (t, *J* = 7.6 Hz, 2H), 7.06 (d, *J* = 7.7 Hz, 4H), 7.20-7.25 (m, 4H).

¹³C NMR (75 MHz, C₆D₆) δ 197.7, 186.4, 156.2, 146.9, 145.8, 136.3, 131.5, 130.7, 127.7, 127.1, 126.6, 124.8, 123.8, 123.1, 120.9, 82.1, 29.2, 25.1, 24.9, 24.1.

¹¹B NMR (128.3 MHz, C₆D₆) δ 30.4.

Anal. Calcd. for C₄₇H₅₈BCuN₂O₂: C, 74.54; H, 7.72; N, 3.70. Found: C, 74.76; H, 7.75; N, 3.92.



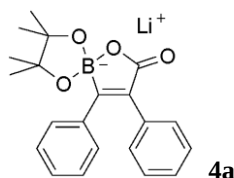
Complex 2

^1H NMR (400 MHz, CD_2Cl_2) δ 0.73 (s, 12H), 1.22 (d, J = 6.8 Hz, 12H), 1.28 (d, J = 6.9 Hz, 12H), 2.50-2.60 (m, 4H), 6.68 (d, J = 5.9 Hz, 2H), 7.02-7.15 (m, 8H), 7.25 (s, 2H), 7.32 (d, J = 7.8 Hz, 4H), 7.44 (t, J = 7.9 Hz, 2H).

^{13}C NMR (100 MHz, CD_2Cl_2) δ 178.8, 174.3, 146.2, 141.4, 136.4, 135.2, 135.0, 131.3, 131.0, 127.8, 127.4, 126.7, 125.8, 125.0, 124.5, 81.4, 29.3, 25.9, 25.0, 24.3.

^{11}B NMR (128.3 MHz, CD_2Cl_2) δ 7.5.

Anal. Calcd. for $\text{C}_{48}\text{H}_{58}\text{BCuN}_2\text{O}_4$: C, 71.94; H, 7.30; N, 3.50. Found: C, 71.50; H, 7.40; N, 3.87.



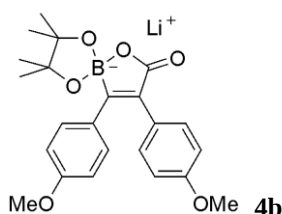
4a

^1H NMR (400 MHz, $\text{THF-}d_8$) δ 0.93 (s, 6H), 1.18 (s, 6H), 7.01-7.28 (m, 10H).

^{13}C NMR (100 MHz, $\text{THF-}d_8$) δ 179.0, 142.6, 136.5, 135.0, 131.3, 128.9, 128.1, 127.9, 126.8, 126.5, 79.9, 26.7, 26.5.

^{11}B NMR (128.3 MHz, $\text{THF-}d_8$) δ 9.3.

Anal. Calcd. for $\text{C}_{25}\text{H}_{30}\text{BLiO}_5$ (**4a**·THF): C, 70.11; H, 7.06. Found: C, 69.85; H, 6.70.



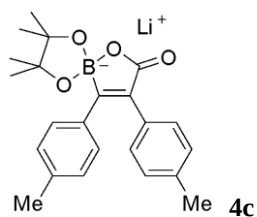
4b

^1H NMR (400 MHz, $\text{THF-}d_8$) δ 0.99 (s, 6H), 1.21 (s, 6H), 3.69 (d, J = 2.2 Hz, 3H), 3.71 (d, J = 2.2 Hz, 3H), 6.65 (dd, J = 8.8 Hz, 2.1 Hz, 2H), 6.71 (dd, J = 8.7 Hz, 2.0 Hz, 2H), 7.12 (dd, J = 8.7 Hz, 2.1 Hz, 2H), 7.26 (dd, J = 8.7 Hz, 2.0 Hz, 2H).

^{13}C NMR (100 MHz, $\text{THF-}d_8$) δ 179.5, 159.4, 159.3, 134.5, 134.0, 132.3, 130.4, 128.8, 113.7, 113.5, 80.0, 55.24, 55.17, 26.8, 26.5.

^{11}B NMR (128.3 MHz, $\text{THF-}d_8$) δ 9.4.

Anal. Calcd. for $\text{C}_{23}\text{H}_{26}\text{BLiO}_6$: C, 66.37; H, 6.30. Found: C, 66.52; H, 6.42.

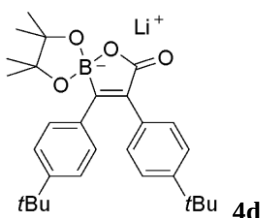


^1H NMR (400 MHz, THF- d_8) δ 0.95 (s, 6H), 1.18 (s, 6H), 2.22 (s, 3H), 2.24 (s, 3H), 6.88 (d, J = 8.0 Hz, 2H), 6.93 (d, J = 7.6 Hz, 2H), 7.07 (d, J = 7.6 Hz, 2H), 7.18 (d, J = 8.0 Hz, 2H).

^{13}C NMR (100 MHz, THF- d_8) δ 179.0, 139.3, 135.7, 135.5, 134.3, 133.4, 131.0, 128.7, 128.6, 128.4, 79.7, 26.6, 26.4, 21.4, 21.2.

^{11}B NMR (128.3 MHz, THF- d_8) δ 9.3.

Anal. Calcd. for $\text{C}_{27}\text{H}_{34}\text{BLiO}_5$ (**4c**·THF): C, 71.07; H, 7.51. Found: C, 70.92; H, 7.12.

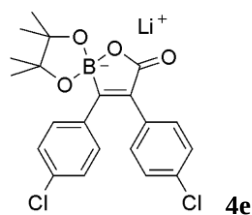


^1H NMR (400 MHz, THF- d_8) δ 0.94 (s, 6H), 1.19 (s, 6H), 1.26 (s, 9H), 1.27 (s, 9H), 7.14-7.19 (m, 6H), 7.25 (d, J = 8.8 Hz, 2H).

^{13}C NMR (100 MHz, THF- d_8) δ 179.1, 149.0, 148.9, 139.2, 134.1, 133.6, 130.7, 128.5, 124.6, 124.5, 79.7, 34.9(8), 34.9(6), 31.7(8), 31.7(3), 26.6, 26.4.

^{11}B NMR (128.3 MHz, THF- d_8) δ 9.4.

Anal. Calcd. for $\text{C}_{29}\text{H}_{38}\text{BLiO}_4$: C, 74.37; H, 8.18. Found: C, 74.04; H, 8.46.

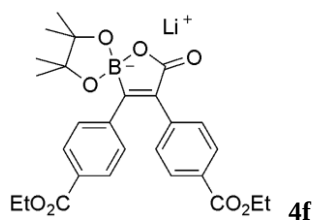


^1H NMR (400 MHz, THF- d_8) δ 0.91 (s, 6H), 1.13 (s, 6H), 7.09-7.16 (m, 6H), 7.21-7.24 (m, 2H).

^{13}C NMR (100 MHz, THF- d_8) δ 178.3, 140.8, 135.0, 133.7, 132.4, 132.1, 130.2, 128.3, 128.1, 79.4, 26.8, 26.4.

^{11}B NMR (128.3 MHz, THF- d_8) δ 9.3.

Anal. Calcd. for $\text{C}_{21}\text{H}_{20}\text{BCl}_2\text{LiO}_4$: C, 59.34; H, 4.74. Found: C, 59.70; H, 5.33.

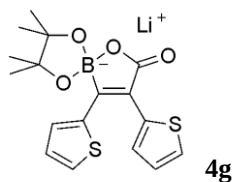


^1H NMR (400 MHz, THF- d_8) δ 0.90 (s, 6H), 1.17 (s, 6H), 1.29-1.35 (m, 6H), 4.23-4.31 (m, 4H), 7.25 (d, J = 8.0 Hz, 2H), 7.30 (d, J = 8.0 Hz, 2H), 7.75 (d, J = 8.0 Hz, 2H), 7.84 (d, J = 8.0 Hz, 2H).

^{13}C NMR (100 MHz, THF- d_8) δ 178.1, 166.9, 166.7, 147.7, 140.9, 135.0, 131.0, 129.7, 129.4, 129.3, 129.1, 128.4, 79.9, 61.2, 61.1, 26.7, 26.4, 14.8, 14.7.

^{11}B NMR (128.3 MHz, THF- d_8) δ 9.4.

Anal. Calcd. for $\text{C}_{27}\text{H}_{30}\text{BLiO}_8$: C, 64.82; H, 6.04. Found: C, 64.13; H, 6.29.

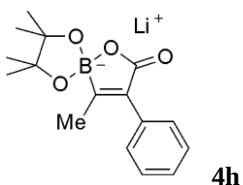


^1H NMR (400 MHz, THF- d_8) δ 1.01 (s, 6H), 1.17 (s, 6H), 6.83 (dd, J = 5.1, 3.6 Hz, 1H), 6.93 (dd, J = 5.1, 3.6 Hz, 1H), 7.05 (dd, J = 3.6, 1.1 Hz, 1H), 7.17-7.25 (m, 3H).

^{13}C NMR (100 MHz, THF- d_8) δ 178.1, 142.9, 137.5, 128.6, 128.2, 127.3, 127.2, 126.7, 126.3, 126.1, 80.0, 27.0, 26.9.

^{11}B NMR (128.3 MHz, THF- d_8) δ 9.0.

Anal. Calcd. for $\text{C}_{21}\text{H}_{26}\text{BLiO}_5\text{S}_2$ (**4g**·THF): C, 57.28; H, 5.95. Found: C, 57.21; H, 6.31.

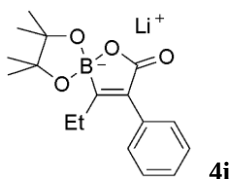


^1H NMR (400 MHz, THF- d_8) δ 1.14 (s, 12H), 1.91 (s, 3H), 7.10-7.14 (m, 1H), 7.21-7.29 (m, 4H).

^{13}C NMR (100 MHz, THF- d_8) δ 179.0, 136.7, 134.8, 130.6, 127.9, 126.5, 79.4, 26.5, 15.8.

^{11}B NMR (128.3 MHz, THF- d_8) δ 9.0.

Anal. Calcd. for $\text{C}_{16}\text{H}_{20}\text{BLiO}_4$: C, 65.35; H, 6.85. Found: C, 64.98; H, 6.89.



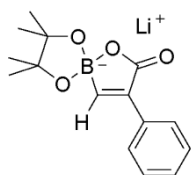
4i

^1H NMR (400 MHz, THF- d_8) δ 1.15 (s, 12H), 1.17 (t, J = 8.0 Hz, 3H), 2.31 (q, J = 7.6 Hz, 2H), 7.14 (t, J = 7.2 Hz, 1H), 7.22-7.28 (m, 4H).

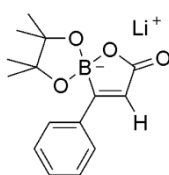
^{13}C NMR (100 MHz, THF- d_8) δ 179.1, 136.8, 134.7, 130.4, 127.9, 126.6, 79.6, 26.3, 23.5, 14.1.

^{11}B NMR (128.3 MHz, THF- d_8) δ 9.2.

Anal. Calcd. for $\text{C}_{17}\text{H}_{22}\text{BLiO}_4$: C, 66.27; H, 7.20. Found: C, 66.73; H, 7.41.



4k (major)



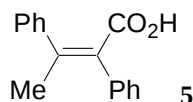
4k' (minor)

4k and **4k'** were isolated as a mixture. The ratio of these isomers (98:2) was determined by ^1H NMR analysis. For spectral data of major product **4k**:

^1H NMR (400 MHz, THF- d_8) δ 1.15(s, 12H), 7.08-7.16 (m, 3H), 7.24 (s, 1H), 7.69 (d, J = 7.2 Hz, 2H).

^{13}C NMR (100 MHz, THF- d_8) δ 178.7, 140.7, 137.1, 128.4, 127.3, 79.9, 25.8.

^{11}B NMR (128.3 MHz, THF- d_8) δ 8.6.



5

^1H NMR (400 MHz, CDCl_3) δ 2.01 (s, 3H), 7.27-7.42 (m, 10H).

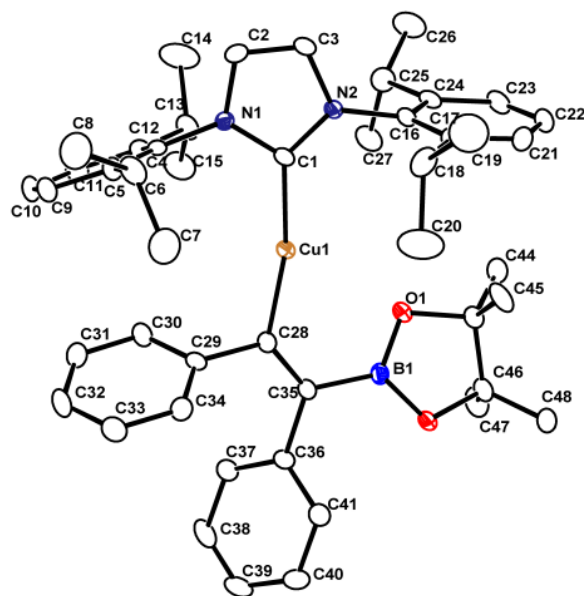
^{13}C NMR (100 MHz, CDCl_3) δ 172.9, 147.3, 142.5, 137.1, 131.3, 129.4, 128.4, 128.3, 127.7(4), 127.7(0), 126.8, 23.4.

X-ray Data for Crystallographic Analysis

A crystal was sealed in a thin-walled glass capillary under a microscope in the glove box. Data collections were performed at $-100\text{ }^\circ\text{C}$ on a Bruker SMART APEX diffractometer with a CCD area detector using graphite-monochromated Mo $\text{K}\alpha$ radiation ($\lambda = 0.71069\text{ \AA}$). The determination of crystal class and unit cell was carried out by SMART program package. The raw frame data were processed using SAINT and SADABS to yield the reflection data file. The structures were solved by using SHELXTL program. Refinements were performed on F^2 anisotropically for non-hydrogen atoms by the full-matrix least-squares method. The SQUEEZE routine of the program PLATON was implemented to remove the contributions of the disordered solvents (benzene in **1**, THF in **2** and **4k**) to the observed structure factors.

The analytical scattering factors for neutral atoms were used throughout the analysis. The hydrogen atoms were placed at the calculated positions and were included in the structure calculation without further refinement of the parameters. The residual electron densities were of no chemical significance. CCDC 879834 (**1**), CCDC 879835 (**2**), CCDC 879833 (**3**), and CCDC 882655 (**4k**) contain the supplementary crystallographic data for this paper. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif.

X-ray Data for Complex **1**



ORTEP drawing of **1**. Thermal ellipsoids set at 30% probability. Hydrogen atoms have been omitted for clarity.

Table 1. Crystal data and structure refinement for **1**.

Identification code	1
Empirical formula	C ₄₇ H ₅₈ B Cu N ₂ O ₂
Formula weight	757.30
Temperature	163(2) K
Wavelength	0.71073 Å
Crystal system, space group	Monoclinic, Cc
Unit cell dimensions	a = 23.443(5) Å alpha = 90 °. b = 12.578(3) Å beta = 116.908(3) °. c = 18.292(4) Å gamma = 90 °.
Volume	4809.7(17) Å ³
Z, Calculated density	4, 1.046 Mg/m ³
Absorption coefficient	0.488 mm ⁻¹

F(000)	1616
Crystal size	0.30 x 0.18 x 0.15 mm
Theta range for data collection	1.89 to 25.00 °.
Limiting indices	-26<=h<=27, -14<=k<=14, -21<=l<=13
Reflections collected / unique	12817 / 6051 [R(int) = 0.0431]
Completeness to theta = 25.00	99.9 %
Absorption correction	Empirical
Max. and min. transmission	0.9304 and 0.8674
Refinement method	Full-matrix least-squares on F ²
Data / restraints / parameters	6051 / 8 / 480
Goodness-of-fit on F ²	0.984
Final R indices [I>2sigma(I)]	R1 = 0.0411, wR2 = 0.0872
R indices (all data)	R1 = 0.0462, wR2 = 0.0895
Absolute structure parameter	0.036(14)
Largest diff. peak and hole	0.448 and -0.192 e.A ⁻³

Table 2. Atomic coordinates (x 10⁴) and equivalent isotropic displacement parameters (Å² x 10³) for **1**.

U(eq) is defined as one third of the trace of the orthogonalized Uij tensor.

	x	y	z	U _{iso} */U _{eq}	Occ. (<1)
C1	0.16385 (15)	0.7207 (2)	0.9834 (2)	0.0225 (8)	
C2	0.24457 (16)	0.7148 (3)	1.1140 (2)	0.0285 (8)	
H2	0.2694	0.6932	1.1692	0.034*	
C3	0.25787 (16)	0.7938 (3)	1.0758 (2)	0.0264 (8)	
H3	0.2942	0.8390	1.0986	0.032*	
C4	0.15458 (15)	0.5844 (3)	1.0771 (2)	0.0230 (8)	
C5	0.17682 (17)	0.4809 (3)	1.0797 (2)	0.0308 (9)	
C6	0.22955 (17)	0.4536 (3)	1.0570 (3)	0.0388 (10)	
H6	0.2507	0.5196	1.0508	0.047*	
C7	0.1961 (5)	0.4291 (13)	0.9651 (6)	0.0542 (15)	0.53 (2)
H7A	0.2281	0.4111	0.9466	0.081*	0.53 (2)
H7B	0.1718	0.4916	0.9351	0.081*	0.53 (2)
H7C	0.1669	0.3689	0.9548	0.081*	0.53 (2)
C8	0.2765 (6)	0.3671 (12)	1.1045 (9)	0.0542 (15)	0.53 (2)
H8A	0.3079	0.3586	1.0832	0.081*	0.53 (2)
H8B	0.2535	0.3001	1.0984	0.081*	0.53 (2)
H8C	0.2987	0.3867	1.1626	0.081*	0.53 (2)

C7'	0.2039 (6)	0.3868 (13)	0.9783 (8)	0.0542 (15)	0.47 (2)
H7'1	0.2389	0.3707	0.9647	0.081*	0.47 (2)
H7'2	0.1706	0.4268	0.9332	0.081*	0.47 (2)
H7'3	0.1859	0.3203	0.9867	0.081*	0.47 (2)
C8'	0.2773 (7)	0.3846 (13)	1.1267 (9)	0.0542 (15)	0.47 (2)
H8'1	0.3129	0.3651	1.1150	0.081*	0.47 (2)
H8'2	0.2559	0.3200	1.1315	0.081*	0.47 (2)
H8'3	0.2938	0.4244	1.1783	0.081*	0.47 (2)
C9	0.14742 (18)	0.4019 (3)	1.1036 (3)	0.0351 (9)	
H9	0.1612	0.3303	1.1066	0.042*	
C10	0.09820 (19)	0.4259 (3)	1.1232 (3)	0.0412 (11)	
H10	0.0791	0.3708	1.1402	0.049*	
C11	0.07681 (19)	0.5283 (3)	1.1183 (3)	0.0379 (10)	
H11	0.0428	0.5436	1.1313	0.045*	
C12	0.10444 (17)	0.6097 (3)	1.0944 (2)	0.0297 (8)	
C13	0.08113 (18)	0.7247 (3)	1.0908 (3)	0.0364 (10)	
H13	0.0910	0.7642	1.0504	0.044*	
C14	0.1170 (2)	0.7785 (4)	1.1730 (4)	0.0700 (16)	
H14A	0.1019	0.8518	1.1695	0.105*	
H14B	0.1629	0.7788	1.1885	0.105*	
H14C	0.1096	0.7398	1.2144	0.105*	
C15	0.0090 (2)	0.7321 (4)	1.0618 (3)	0.0578 (13)	
H15A	-0.0136	0.6978	1.0081	0.087*	
H15B	-0.0037	0.8069	1.0574	0.087*	
H15C	-0.0018	0.6961	1.1014	0.087*	
C16	0.20371 (16)	0.8840 (3)	0.9416 (2)	0.0262 (8)	
C17	0.23265 (15)	0.8746 (3)	0.8899 (2)	0.0270 (8)	
C18	0.26396 (18)	0.7713 (3)	0.8824 (3)	0.0399 (10)	
H18	0.2722	0.7266	0.9314	0.048*	
C19	0.3268 (2)	0.7874 (4)	0.8802 (4)	0.0680 (15)	
H19A	0.3441	0.7183	0.8754	0.102*	
H19B	0.3570	0.8226	0.9308	0.102*	
H19C	0.3201	0.8318	0.8330	0.102*	
C20	0.2174 (3)	0.7109 (4)	0.8064 (4)	0.0766 (17)	
H20A	0.2374	0.6446	0.8014	0.115*	
H20B	0.2066	0.7548	0.7576	0.115*	

H20C	0.1784	0.6946	0.8112	0.115*
C21	0.23072 (17)	0.9637 (3)	0.8432 (2)	0.0355 (9)
H21	0.2502	0.9604	0.8077	0.043*
C22	0.20131 (18)	1.0556 (3)	0.8477 (3)	0.0391 (10)
H22	0.2010	1.1153	0.8157	0.047*
C23	0.17223 (18)	1.0625 (3)	0.8978 (3)	0.0382 (10)
H23	0.1512	1.1265	0.8990	0.046*
C24	0.17295 (16)	0.9775 (3)	0.9470 (2)	0.0302 (9)
C25	0.14072 (18)	0.9849 (3)	1.0031 (3)	0.0360 (9)
H25	0.1598	0.9290	1.0464	0.043*
C26	0.1511 (2)	1.0921 (4)	1.0456 (3)	0.0550 (13)
H26A	0.1293	1.0931	1.0804	0.083*
H26B	0.1337	1.1485	1.0044	0.083*
H26C	0.1970	1.1039	1.0795	0.083*
C27	0.06963 (18)	0.9610 (3)	0.9538 (3)	0.0433 (11)
H27A	0.0488	0.9662	0.9896	0.065*
H27B	0.0641	0.8889	0.9312	0.065*
H27C	0.0503	1.0124	0.9089	0.065*
C28	0.01754 (15)	0.5963 (3)	0.8088 (2)	0.0244 (8)
C29	0.01632 (15)	0.4844 (3)	0.8371 (2)	0.0262 (8)
C30	0.00181 (18)	0.4626 (3)	0.9010 (3)	0.0342 (9)
H30	-0.0045	0.5201	0.9302	0.041*
C31	-0.00376 (19)	0.3598 (3)	0.9238 (3)	0.0412 (10)
H31	-0.0152	0.3472	0.9667	0.049*
C32	0.0074 (2)	0.2755 (3)	0.8838 (3)	0.0487 (12)
H32	0.0030	0.2046	0.8982	0.058*
C33	0.0249 (2)	0.2949 (3)	0.8230 (3)	0.0472 (11)
H33	0.0343	0.2368	0.7969	0.057*
C34	0.02924 (18)	0.3968 (3)	0.7991 (3)	0.0369 (10)
H34	0.0411	0.4084	0.7565	0.044*
C35	-0.02627 (16)	0.6252 (3)	0.7324 (2)	0.0253 (8)
C36	-0.07746 (16)	0.5499 (3)	0.6751 (2)	0.0294 (9)
C37	-0.11723 (17)	0.4964 (3)	0.7010 (3)	0.0367 (10)
H37	-0.1137	0.5100	0.7540	0.044*
C38	-0.16222 (19)	0.4231 (3)	0.6496 (3)	0.0539 (13)
H38	-0.1878	0.3851	0.6688	0.065*

C39	-0.1698 (2)	0.4054 (3)	0.5728 (3)	0.0534 (13)
H39	-0.2011	0.3563	0.5381	0.064*
C40	-0.1319 (2)	0.4588 (3)	0.5446 (3)	0.0480 (11)
H40	-0.1367	0.4460	0.4910	0.058*
C41	-0.08653 (17)	0.5317 (3)	0.5960 (2)	0.0337 (9)
H41	-0.0614	0.5697	0.5762	0.040*
B1	-0.02474 (19)	0.7377 (3)	0.6979 (3)	0.0270 (9)
C43	0.00465 (17)	0.9088 (3)	0.6911 (3)	0.0312 (9)
C44	-0.0035 (2)	1.0174 (3)	0.7216 (3)	0.0441 (11)
H44A	-0.0465	1.0231	0.7176	0.066*
H44B	0.0024	1.0729	0.6879	0.066*
H44C	0.0284	1.0264	0.7788	0.066*
C45	0.07086 (19)	0.8998 (3)	0.6965 (3)	0.0478 (12)
H45A	0.0759	0.8296	0.6770	0.072*
H45B	0.1031	0.9091	0.7536	0.072*
H45C	0.0764	0.9550	0.6624	0.072*
C46	-0.04952 (17)	0.8749 (3)	0.6072 (3)	0.0343 (9)
C47	-0.11457 (18)	0.9153 (3)	0.5922 (3)	0.0521 (13)
H47A	-0.1218	0.8987	0.6397	0.078*
H47B	-0.1476	0.8810	0.5433	0.078*
H47C	-0.1165	0.9925	0.5840	0.078*
C48	-0.0364 (2)	0.9001 (3)	0.5346 (3)	0.0497 (12)
H48A	0.0058	0.8725	0.5454	0.075*
H48B	-0.0373	0.9772	0.5268	0.075*
H48C	-0.0692	0.8667	0.4850	0.075*
N1	0.18756 (13)	0.6701 (2)	1.05757 (18)	0.0222 (7)
N2	0.20900 (12)	0.7978 (2)	0.99688 (18)	0.0225 (6)
O1	0.00169 (12)	0.82593 (18)	0.74593 (16)	0.0351 (6)
O2	-0.05071 (11)	0.76025 (18)	0.61592 (16)	0.0330 (6)
Cu1	0.08825 (2)	0.67481 (3)	0.89008 (3)	0.02421 (11)

Table 3. Bond lengths [Å] and angles [°] for **1**.

C1—N1	1.368 (5)	C22—C23	1.370 (6)
C1—N2	1.373 (4)	C22—H22	0.9500
C1—Cu1	1.910 (3)	C23—C24	1.393 (5)
C2—C3	1.330 (5)	C23—H23	0.9500

C2—N1	1.385 (4)	C24—C25	1.527 (5)
C2—H2	0.9500	C25—C26	1.521 (5)
C3—N2	1.379 (4)	C25—C27	1.523 (5)
C3—H3	0.9500	C25—H25	1.0000
C4—C12	1.385 (5)	C26—H26A	0.9800
C4—C5	1.395 (5)	C26—H26B	0.9800
C4—N1	1.461 (4)	C26—H26C	0.9800
C5—C9	1.389 (5)	C27—H27A	0.9800
C5—C6	1.512 (5)	C27—H27B	0.9800
C6—C8	1.511 (10)	C27—H27C	0.9800
C6—C7	1.530 (10)	C28—C35	1.356 (5)
C6—C8'	1.530 (11)	C28—C29	1.505 (5)
C6—C7'	1.535 (11)	C28—Cu1	1.924 (4)
C6—H6	1.0000	C29—C30	1.385 (5)
C7—H7A	0.9800	C29—C34	1.407 (5)
C7—H7B	0.9800	C30—C31	1.383 (5)
C7—H7C	0.9800	C30—H30	0.9500
C8—H8A	0.9800	C31—C32	1.378 (6)
C8—H8B	0.9800	C31—H31	0.9500
C8—H8C	0.9800	C32—C33	1.371 (6)
C7'—H7'1	0.9800	C32—H32	0.9500
C7'—H7'2	0.9800	C33—C34	1.373 (5)
C7'—H7'3	0.9800	C33—H33	0.9500
C8'—H8'1	0.9800	C34—H34	0.9500
C8'—H8'2	0.9800	C35—C36	1.516 (5)
C8'—H8'3	0.9800	C35—B1	1.557 (5)
C9—C10	1.387 (5)	C36—C41	1.383 (5)
C9—H9	0.9500	C36—C37	1.395 (5)
C10—C11	1.371 (6)	C37—C38	1.396 (6)
C10—H10	0.9500	C37—H37	0.9500
C11—C12	1.383 (5)	C38—C39	1.353 (7)
C11—H11	0.9500	C38—H38	0.9500
C12—C13	1.538 (5)	C39—C40	1.386 (6)
C13—C14	1.511 (6)	C39—H39	0.9500
C13—C15	1.528 (5)	C40—C41	1.396 (5)
C13—H13	1.0000	C40—H40	0.9500

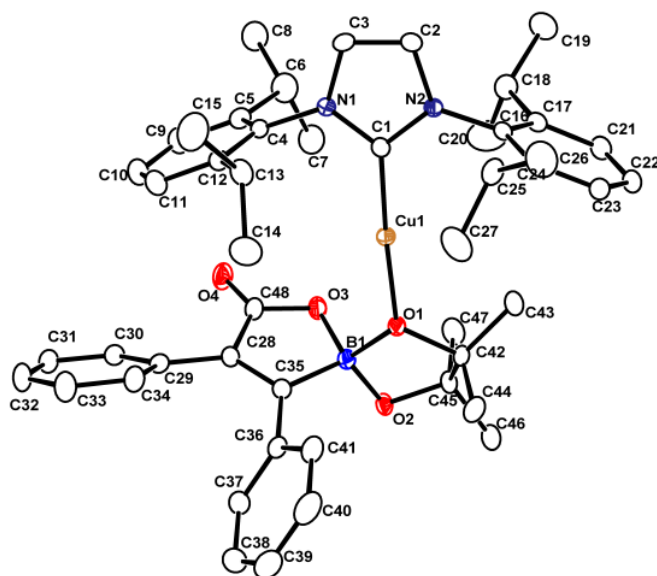
C14—H14A	0.9800	C41—H41	0.9500
C14—H14B	0.9800	B1—O2	1.369 (5)
C14—H14C	0.9800	B1—O1	1.376 (5)
C15—H15A	0.9800	C43—O1	1.470 (4)
C15—H15B	0.9800	C43—C45	1.514 (5)
C15—H15C	0.9800	C43—C44	1.520 (5)
C16—C17	1.397 (5)	C43—C46	1.544 (6)
C16—C24	1.406 (5)	C44—H44A	0.9800
C16—N2	1.449 (4)	C44—H44B	0.9800
C17—C21	1.396 (5)	C44—H44C	0.9800
C17—C18	1.528 (5)	C45—H45A	0.9800
C18—C19	1.505 (6)	C45—H45B	0.9800
C18—C20	1.526 (7)	C45—H45C	0.9800
C18—H18	1.0000	C46—O2	1.452 (4)
C19—H19A	0.9800	C46—C47	1.510 (5)
C19—H19B	0.9800	C46—C48	1.526 (6)
C19—H19C	0.9800	C47—H47A	0.9800
C20—H20A	0.9800	C47—H47B	0.9800
C20—H20B	0.9800	C47—H47C	0.9800
C20—H20C	0.9800	C48—H48A	0.9800
C21—C22	1.367 (5)	C48—H48B	0.9800
C21—H21	0.9500	C48—H48C	0.9800
N1—C1—N2	102.5 (3)	C22—C23—C24	121.3 (4)
N1—C1—Cu1	122.8 (2)	C22—C23—H23	119.4
N2—C1—Cu1	134.6 (3)	C24—C23—H23	119.4
C3—C2—N1	106.8 (3)	C23—C24—C16	116.9 (4)
C3—C2—H2	126.6	C23—C24—C25	121.5 (3)
N1—C2—H2	126.6	C16—C24—C25	121.6 (3)
C2—C3—N2	107.2 (3)	C26—C25—C27	110.9 (3)
C2—C3—H3	126.4	C26—C25—C24	112.6 (3)
N2—C3—H3	126.4	C27—C25—C24	109.4 (3)
C12—C4—C5	123.3 (3)	C26—C25—H25	107.9
C12—C4—N1	119.0 (3)	C27—C25—H25	107.9
C5—C4—N1	117.7 (3)	C24—C25—H25	107.9
C9—C5—C4	116.6 (3)	C25—C26—H26A	109.5

C9—C5—C6	120.5 (3)	C25—C26—H26B	109.5
C4—C5—C6	122.9 (3)	H26A—C26—H26B	109.5
C8—C6—C5	118.7 (6)	C25—C26—H26C	109.5
C8—C6—C7	111.4 (6)	H26A—C26—H26C	109.5
C5—C6—C7	105.7 (5)	H26B—C26—H26C	109.5
C8—C6—C8'	17.2 (7)	C25—C27—H27A	109.5
C5—C6—C8'	106.3 (7)	C25—C27—H27B	109.5
C7—C6—C8'	128.3 (7)	H27A—C27—H27B	109.5
C8—C6—C7'	91.1 (7)	C25—C27—H27C	109.5
C5—C6—C7'	111.2 (5)	H27A—C27—H27C	109.5
C7—C6—C7'	21.8 (5)	H27B—C27—H27C	109.5
C8'—C6—C7'	107.4 (7)	C35—C28—C29	118.8 (3)
C8—C6—H6	113.1	C35—C28—Cu1	130.2 (3)
C5—C6—H6	110.6	C29—C28—Cu1	110.9 (2)
C7—C6—H6	94.4	C30—C29—C34	116.9 (3)
C8'—C6—H6	110.6	C30—C29—C28	121.8 (3)
C7'—C6—H6	110.6	C34—C29—C28	121.3 (3)
C6—C7—H7A	109.5	C31—C30—C29	122.1 (4)
C6—C7—H7B	109.5	C31—C30—H30	118.9
H7A—C7—H7B	109.5	C29—C30—H30	118.9
C6—C7—H7C	109.5	C32—C31—C30	119.5 (4)
H7A—C7—H7C	109.5	C32—C31—H31	120.2
H7B—C7—H7C	109.5	C30—C31—H31	120.2
C6—C8—H8A	109.5	C33—C32—C31	119.5 (4)
C6—C8—H8B	109.5	C33—C32—H32	120.2
H8A—C8—H8B	109.5	C31—C32—H32	120.2
C6—C8—H8C	109.5	C32—C33—C34	121.1 (4)
H8A—C8—H8C	109.5	C32—C33—H33	119.5
H8B—C8—H8C	109.5	C34—C33—H33	119.5
C6—C7'—H7'1	109.5	C33—C34—C29	120.7 (4)
C6—C7'—H7'2	109.5	C33—C34—H34	119.6
H7'1—C7'—H7'2	109.5	C29—C34—H34	119.6
C6—C7'—H7'3	109.5	C28—C35—C36	122.6 (3)
H7'1—C7'—H7'3	109.5	C28—C35—B1	121.0 (3)
H7'2—C7'—H7'3	109.5	C36—C35—B1	116.4 (3)
C6—C8'—H8'1	109.5	C41—C36—C37	117.7 (3)

C6—C8'—H8'2	109.5	C41—C36—C35	121.7 (3)
H8'1—C8'—H8'2	109.5	C37—C36—C35	120.6 (3)
C6—C8'—H8'3	109.5	C36—C37—C38	120.5 (4)
H8'1—C8'—H8'3	109.5	C36—C37—H37	119.7
H8'2—C8'—H8'3	109.5	C38—C37—H37	119.7
C10—C9—C5	121.0 (3)	C39—C38—C37	120.7 (4)
C10—C9—H9	119.5	C39—C38—H38	119.6
C5—C9—H9	119.5	C37—C38—H38	119.6
C11—C10—C9	120.6 (3)	C38—C39—C40	120.1 (4)
C11—C10—H10	119.7	C38—C39—H39	119.9
C9—C10—H10	119.7	C40—C39—H39	119.9
C10—C11—C12	120.4 (4)	C39—C40—C41	119.3 (4)
C10—C11—H11	119.8	C39—C40—H40	120.4
C12—C11—H11	119.8	C41—C40—H40	120.4
C11—C12—C4	118.0 (3)	C36—C41—C40	121.5 (4)
C11—C12—C13	120.1 (3)	C36—C41—H41	119.3
C4—C12—C13	121.8 (3)	C40—C41—H41	119.3
C14—C13—C15	110.4 (4)	O2—B1—O1	112.4 (3)
C14—C13—C12	110.6 (4)	O2—B1—C35	123.5 (3)
C15—C13—C12	112.8 (3)	O1—B1—C35	124.1 (4)
C14—C13—H13	107.6	O1—C43—C45	105.4 (3)
C15—C13—H13	107.6	O1—C43—C44	109.6 (3)
C12—C13—H13	107.6	C45—C43—C44	109.8 (3)
C13—C14—H14A	109.5	O1—C43—C46	102.1 (3)
C13—C14—H14B	109.5	C45—C43—C46	113.7 (3)
H14A—C14—H14B	109.5	C44—C43—C46	115.4 (3)
C13—C14—H14C	109.5	C43—C44—H44A	109.5
H14A—C14—H14C	109.5	C43—C44—H44B	109.5
H14B—C14—H14C	109.5	H44A—C44—H44B	109.5
C13—C15—H15A	109.5	C43—C44—H44C	109.5
C13—C15—H15B	109.5	H44A—C44—H44C	109.5
H15A—C15—H15B	109.5	H44B—C44—H44C	109.5
C13—C15—H15C	109.5	C43—C45—H45A	109.5
H15A—C15—H15C	109.5	C43—C45—H45B	109.5
H15B—C15—H15C	109.5	H45A—C45—H45B	109.5
C17—C16—C24	122.7 (3)	C43—C45—H45C	109.5

C17—C16—N2	119.7 (3)	H45A—C45—H45C	109.5
C24—C16—N2	117.5 (3)	H45B—C45—H45C	109.5
C21—C17—C16	117.0 (3)	O2—C46—C47	106.7 (3)
C21—C17—C18	120.8 (3)	O2—C46—C48	108.8 (3)
C16—C17—C18	122.1 (3)	C47—C46—C48	110.9 (4)
C19—C18—C20	110.3 (4)	O2—C46—C43	102.3 (3)
C19—C18—C17	113.8 (4)	C47—C46—C43	113.3 (4)
C20—C18—C17	109.3 (3)	C48—C46—C43	114.1 (3)
C19—C18—H18	107.8	C46—C47—H47A	109.5
C20—C18—H18	107.8	C46—C47—H47B	109.5
C17—C18—H18	107.8	H47A—C47—H47B	109.5
C18—C19—H19A	109.5	C46—C47—H47C	109.5
C18—C19—H19B	109.5	H47A—C47—H47C	109.5
H19A—C19—H19B	109.5	H47B—C47—H47C	109.5
C18—C19—H19C	109.5	C46—C48—H48A	109.5
H19A—C19—H19C	109.5	C46—C48—H48B	109.5
H19B—C19—H19C	109.5	H48A—C48—H48B	109.5
C18—C20—H20A	109.5	C46—C48—H48C	109.5
C18—C20—H20B	109.5	H48A—C48—H48C	109.5
H20A—C20—H20B	109.5	H48B—C48—H48C	109.5
C18—C20—H20C	109.5	C1—N1—C2	111.8 (3)
H20A—C20—H20C	109.5	C1—N1—C4	124.3 (3)
H20B—C20—H20C	109.5	C2—N1—C4	123.8 (3)
C22—C21—C17	121.3 (4)	C1—N2—C3	111.7 (3)
C22—C21—H21	119.3	C1—N2—C16	126.0 (3)
C17—C21—H21	119.3	C3—N2—C16	121.7 (3)
C21—C22—C23	120.7 (4)	B1—O1—C43	106.5 (3)
C21—C22—H22	119.6	B1—O2—C46	107.7 (3)
C23—C22—H22	119.6	C1—Cu1—C28	166.39 (14)

X-ray Data for Complex 2



ORTEP drawing of **2**. Thermal ellipsoids set at 30% probability. Hydrogen atoms have been omitted for clarity.

Table 1. Crystal data and structure refinement for **2**.

Identification code	2
Empirical formula	C ₄₈ H ₅₈ B Cu N ₂ O ₄
Formula weight	801.31
Temperature	163(2) K
Wavelength	0.71073 Å
Crystal system, space group	Monoclinic, P2 ₁ /n
Unit cell dimensions	a = 10.384(2) Å alpha = 90 °. b = 24.651(6) Å beta = 97.555(4) °. c = 18.961(4) Å gamma = 90 °.
Volume	4811.4(19) Å ³
Z, Calculated density	4, 1.106 Mg/m ³
Absorption coefficient	0.494 mm ⁻¹
F(000)	1704
Crystal size	0.48 x 0.38 x 0.25 mm
Theta range for data collection	1.36 to 25.00 °.
Limiting indices	-12 ≤ h ≤ 12, -29 ≤ k ≤ 29, -22 ≤ l ≤ 15
Reflections collected / unique	25913 / 8458 [R(int) = 0.0539]
Completeness to theta = 25.00	99.7 %
Absorption correction	Empirical

Max. and min. transmission	0.8864 and 0.7974
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	8458 / 12 / 519
Goodness-of-fit on F^2	1.030
Final R indices [$I > 2\sigma(I)$]	$R1 = 0.0463$, $wR2 = 0.1028$
R indices (all data)	$R1 = 0.0673$, $wR2 = 0.1097$
Largest diff. peak and hole	0.417 and -0.343 e. $\text{\AA}^{-3}$

Table 2. Atomic coordinates ($\times 10^4$) and equivalent isotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for **2**.
 $U(\text{eq})$ is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$	Occ. (<1)
C1	0.3934 (2)	0.60763 (9)	0.24962 (13)	0.0225 (5)	
C2	0.1752 (2)	0.59619 (9)	0.24405 (13)	0.0279 (6)	
H2	0.0849	0.5978	0.2274	0.033*	
C3	0.2308 (2)	0.57387 (10)	0.30471 (13)	0.0281 (6)	
H3	0.1873	0.5563	0.3394	0.034*	
C4	0.4609 (2)	0.56370 (10)	0.36498 (13)	0.0257 (6)	
C5	0.5123 (2)	0.51156 (10)	0.36156 (14)	0.0323 (6)	
C6	0.4705 (3)	0.47401 (11)	0.29935 (17)	0.0501 (6)	
H6	0.4180	0.4952	0.2608	0.060*	
C7	0.5774 (5)	0.4719 (3)	0.2488 (3)	0.0501 (6)	0.616 (7)
H7A	0.5491	0.4479	0.2086	0.075*	0.616 (7)
H7B	0.6584	0.4581	0.2749	0.075*	0.616 (7)
H7C	0.5918	0.5085	0.2311	0.075*	0.616 (7)
C8	0.4357 (7)	0.4173 (2)	0.3199 (3)	0.0501 (6)	0.616 (7)
H8A	0.4099	0.3959	0.2769	0.075*	0.616 (7)
H8B	0.3634	0.4189	0.3483	0.075*	0.616 (7)
H8C	0.5111	0.4004	0.3480	0.075*	0.616 (7)
C7'	0.5877 (8)	0.4491 (4)	0.2696 (5)	0.0501 (6)	0.384 (7)
H7'1	0.5571	0.4254	0.2294	0.075*	0.384 (7)
H7'2	0.6393	0.4279	0.3068	0.075*	0.384 (7)
H7'3	0.6414	0.4781	0.2535	0.075*	0.384 (7)
C8'	0.3822 (10)	0.4310 (3)	0.3277 (5)	0.0501 (6)	0.384 (7)
H8'1	0.3519	0.4054	0.2896	0.075*	0.384 (7)
H8'2	0.3073	0.4489	0.3441	0.075*	0.384 (7)
H8'3	0.4314	0.4113	0.3674	0.075*	0.384 (7)

C9	0.6073 (3)	0.49669 (12)	0.41653 (15)	0.0415 (7)	
H9	0.6443	0.4615	0.4165	0.050*	
C10	0.6487 (3)	0.53170 (12)	0.47075 (16)	0.0445 (8)	
H10	0.7146	0.5206	0.5075	0.053*	
C11	0.5959 (3)	0.58268 (12)	0.47263 (15)	0.0402 (7)	
H11	0.6262	0.6065	0.5106	0.048*	
C12	0.4989 (2)	0.59990 (10)	0.41985 (13)	0.0295 (6)	
C13	0.4407 (3)	0.65633 (11)	0.42221 (15)	0.0365 (7)	
H13	0.3751	0.6605	0.3789	0.044*	
C14	0.5427 (3)	0.70018 (12)	0.41932 (19)	0.0609 (9)	
H14A	0.5021	0.7360	0.4209	0.091*	
H14B	0.5814	0.6966	0.3751	0.091*	
H14C	0.6106	0.6962	0.4601	0.091*	
C15	0.3691 (4)	0.66256 (14)	0.48718 (17)	0.0612 (9)	
H15A	0.3043	0.6337	0.4872	0.092*	
H15B	0.3257	0.6979	0.4855	0.092*	
H15C	0.4315	0.6601	0.5306	0.092*	
C16	0.2633 (2)	0.64260 (9)	0.14141 (12)	0.0234 (5)	
C17	0.2679 (2)	0.60992 (10)	0.08136 (13)	0.0282 (6)	
C18	0.2771 (3)	0.54886 (10)	0.08634 (15)	0.0363 (7)	
H18	0.2691	0.5348	0.1350	0.044*	
C19	0.1588 (17)	0.5206 (8)	0.0456 (13)	0.063 (4)	0.64 (6)
H19A	0.1685	0.4812	0.0510	0.095*	0.64 (6)
H19B	0.1521	0.5302	-0.0049	0.095*	0.64 (6)
H19C	0.0800	0.5323	0.0646	0.095*	0.64 (6)
C20	0.4141 (14)	0.5312 (9)	0.0764 (17)	0.076 (5)	0.64 (6)
H20A	0.4208	0.4916	0.0802	0.114*	0.64 (6)
H20B	0.4766	0.5479	0.1133	0.114*	0.64 (6)
H20C	0.4332	0.5427	0.0294	0.114*	0.64 (6)
C19'	0.176 (4)	0.5250 (15)	0.029 (2)	0.077 (11)	0.36 (6)
H19D	0.1791	0.4853	0.0317	0.115*	0.36 (6)
H19E	0.1945	0.5369	-0.0179	0.115*	0.36 (6)
H19F	0.0893	0.5376	0.0367	0.115*	0.36 (6)
C20'	0.391 (2)	0.5237 (13)	0.054 (2)	0.064 (6)	0.36 (6)
H20D	0.3892	0.4842	0.0598	0.096*	0.36 (6)
H20E	0.4728	0.5381	0.0784	0.096*	0.36 (6)

H20F	0.3832	0.5327	0.0033	0.096*	0.36 (6)
C21	0.2629 (2)	0.63613 (11)	0.01570 (14)	0.0336 (6)	
H21	0.2664	0.6154	-0.0262	0.040*	
C22	0.2530 (2)	0.69174 (11)	0.01121 (14)	0.0338 (6)	
H22	0.2520	0.7091	-0.0336	0.041*	
C23	0.2445 (2)	0.72250 (10)	0.07125 (13)	0.0293 (6)	
H23	0.2352	0.7607	0.0668	0.035*	
C24	0.2491 (2)	0.69894 (10)	0.13759 (13)	0.0260 (6)	
C25	0.2403 (3)	0.73290 (10)	0.20406 (14)	0.0326 (6)	
H25	0.2119	0.7083	0.2410	0.039*	
C26	0.1407 (3)	0.77788 (12)	0.19093 (17)	0.0520 (8)	
H26A	0.0564	0.7624	0.1718	0.078*	
H26B	0.1683	0.8038	0.1567	0.078*	
H26C	0.1330	0.7966	0.2358	0.078*	
C27	0.3714 (3)	0.75596 (13)	0.23356 (17)	0.0553 (9)	
H27A	0.4349	0.7265	0.2418	0.083*	
H27B	0.3646	0.7746	0.2786	0.083*	
H27C	0.3998	0.7818	0.1994	0.083*	
C28	0.9065 (2)	0.62029 (9)	0.35759 (13)	0.0254 (6)	
C29	0.9524 (2)	0.61760 (10)	0.43510 (13)	0.0280 (6)	
C30	1.0212 (2)	0.57297 (11)	0.46594 (14)	0.0328 (6)	
H30	1.0396	0.5434	0.4368	0.039*	
C31	1.0627 (3)	0.57122 (12)	0.53803 (15)	0.0403 (7)	
H31	1.1086	0.5403	0.5579	0.048*	
C32	1.0387 (3)	0.61372 (13)	0.58145 (16)	0.0488 (8)	
H32	1.0683	0.6125	0.6310	0.059*	
C33	0.9706 (3)	0.65834 (13)	0.55198 (15)	0.0472 (8)	
H33	0.9538	0.6880	0.5814	0.057*	
C34	0.9273 (3)	0.65991 (11)	0.48006 (14)	0.0357 (7)	
H34	0.8793	0.6905	0.4608	0.043*	
C35	0.8952 (2)	0.66311 (10)	0.31339 (13)	0.0257 (6)	
C36	0.9405 (2)	0.71858 (10)	0.33340 (13)	0.0284 (6)	
C37	1.0685 (3)	0.72792 (11)	0.36324 (14)	0.0368 (7)	
H37	1.1273	0.6983	0.3711	0.044*	
C38	1.1106 (3)	0.77982 (13)	0.38147 (15)	0.0504 (8)	
H38	1.1983	0.7857	0.4013	0.061*	

C39	1.0266 (4)	0.82299 (13)	0.37114 (16)	0.0563 (9)
H39	1.0561	0.8585	0.3842	0.068*
C40	0.9003 (4)	0.81469 (12)	0.34193 (16)	0.0511 (8)
H40	0.8421	0.8445	0.3350	0.061*
C41	0.8572 (3)	0.76289 (11)	0.32250 (14)	0.0366 (7)
H41	0.7699	0.7576	0.3015	0.044*
C42	0.7146 (2)	0.68678 (11)	0.13304 (13)	0.0301 (6)
C43	0.5888 (2)	0.67682 (13)	0.08473 (15)	0.0458 (8)
H43A	0.5741	0.6377	0.0793	0.069*
H43B	0.5942	0.6930	0.0380	0.069*
H43C	0.5166	0.6933	0.1056	0.069*
C44	0.7394 (3)	0.74660 (11)	0.14216 (15)	0.0409 (7)
H44A	0.8211	0.7523	0.1736	0.061*
H44B	0.6679	0.7634	0.1632	0.061*
H44C	0.7455	0.7631	0.0957	0.061*
C45	0.8331 (2)	0.65465 (11)	0.11226 (13)	0.0321 (6)
C46	0.9056 (3)	0.68357 (12)	0.05907 (14)	0.0423 (7)
H46A	0.9289	0.7202	0.0764	0.063*
H46B	0.8501	0.6859	0.0132	0.063*
H46C	0.9847	0.6633	0.0532	0.063*
C47	0.7968 (3)	0.59754 (12)	0.08507 (15)	0.0469 (8)
H47A	0.7497	0.5788	0.1194	0.070*
H47B	0.8760	0.5773	0.0793	0.070*
H47C	0.7415	0.5999	0.0391	0.070*
C48	0.8626 (3)	0.57000 (11)	0.31675 (14)	0.0322 (6)
B1	0.8359 (3)	0.64197 (12)	0.23497 (16)	0.0285 (7)
N1	0.36395 (18)	0.58111 (7)	0.30756 (10)	0.0228 (5)
N2	0.27595 (18)	0.61661 (7)	0.21021 (10)	0.0219 (4)
O1	0.70512 (15)	0.66399 (7)	0.20388 (9)	0.0289 (4)
O2	0.91534 (15)	0.65154 (7)	0.17925 (9)	0.0300 (4)
O3	0.81687 (17)	0.58274 (7)	0.24965 (9)	0.0344 (4)
O4	0.8669 (2)	0.52397 (7)	0.33841 (10)	0.0469 (5)
Cu1	0.55346 (3)	0.631820 (12)	0.227910 (16)	0.02587 (10)

Table 3. Bond lengths [Å] and angles [°] for 2.

C1—N1	1.348 (3)	C21—C22	1.376 (4)
C1—N2	1.362 (3)	C21—H21	0.9500
C1—Cu1	1.862 (2)	C22—C23	1.380 (4)
C2—C3	1.336 (3)	C22—H22	0.9500
C2—N2	1.392 (3)	C23—C24	1.381 (3)
C2—H2	0.9500	C23—H23	0.9500
C3—N1	1.388 (3)	C24—C25	1.526 (3)
C3—H3	0.9500	C25—C27	1.514 (4)
C4—C12	1.388 (3)	C25—C26	1.514 (4)
C4—C5	1.396 (3)	C25—H25	1.0000
C4—N1	1.448 (3)	C26—H26A	0.9800
C5—C9	1.387 (4)	C26—H26B	0.9800
C5—C6	1.517 (4)	C26—H26C	0.9800
C6—C8	1.508 (5)	C27—H27A	0.9800
C6—C7'	1.535 (8)	C27—H27B	0.9800
C6—C8'	1.544 (8)	C27—H27C	0.9800
C6—C7	1.560 (6)	C28—C35	1.343 (3)
C6—H6	1.0000	C28—C29	1.486 (3)
C7—H7A	0.9800	C28—C48	1.501 (3)
C7—H7B	0.9800	C29—C34	1.393 (4)
C7—H7C	0.9800	C29—C30	1.397 (3)
C8—H8A	0.9800	C30—C31	1.379 (4)
C8—H8B	0.9800	C30—H30	0.9500
C8—H8C	0.9800	C31—C32	1.376 (4)
C7'—H7'1	0.9800	C31—H31	0.9500
C7'—H7'2	0.9800	C32—C33	1.385 (4)
C7'—H7'3	0.9800	C32—H32	0.9500
C8'—H8'1	0.9800	C33—C34	1.379 (4)
C8'—H8'2	0.9800	C33—H33	0.9500
C8'—H8'3	0.9800	C34—H34	0.9500
C9—C10	1.368 (4)	C35—C36	1.479 (3)
C9—H9	0.9500	C35—B1	1.619 (4)
C10—C11	1.374 (4)	C36—C41	1.392 (3)
C10—H10	0.9500	C36—C37	1.393 (4)
C11—C12	1.390 (3)	C37—C38	1.381 (4)

C11—H11	0.9500	C37—H37	0.9500
C12—C13	1.520 (4)	C38—C39	1.373 (5)
C13—C14	1.520 (4)	C38—H38	0.9500
C13—C15	1.528 (4)	C39—C40	1.370 (5)
C13—H13	1.0000	C39—H39	0.9500
C14—H14A	0.9800	C40—C41	1.387 (4)
C14—H14B	0.9800	C40—H40	0.9500
C14—H14C	0.9800	C41—H41	0.9500
C15—H15A	0.9800	C42—O1	1.471 (3)
C15—H15B	0.9800	C42—C44	1.503 (4)
C15—H15C	0.9800	C42—C43	1.514 (3)
C16—C24	1.398 (3)	C42—C45	1.557 (4)
C16—C17	1.401 (3)	C43—H43A	0.9800
C16—N2	1.444 (3)	C43—H43B	0.9800
C17—C21	1.397 (4)	C43—H43C	0.9800
C17—C18	1.511 (4)	C44—H44A	0.9800
C18—C20	1.523 (9)	C44—H44B	0.9800
C18—C19'	1.526 (13)	C44—H44C	0.9800
C18—C19	1.530 (9)	C45—O2	1.437 (3)
C18—C20'	1.531 (13)	C45—C46	1.514 (4)
C18—H18	1.0000	C45—C47	1.529 (4)
C19—H19A	0.9800	C46—H46A	0.9800
C19—H19B	0.9800	C46—H46B	0.9800
C19—H19C	0.9800	C46—H46C	0.9800
C20—H20A	0.9800	C47—H47A	0.9800
C20—H20B	0.9800	C47—H47B	0.9800
C20—H20C	0.9800	C47—H47C	0.9800
C19'—H19D	0.9800	C48—O4	1.206 (3)
C19'—H19E	0.9800	C48—O3	1.336 (3)
C19'—H19F	0.9800	B1—O2	1.443 (3)
C20'—H20D	0.9800	B1—O3	1.504 (3)
C20'—H20E	0.9800	B1—O1	1.509 (3)
C20'—H20F	0.9800	O1—Cu1	1.8723 (16)
N1—C1—N2	104.08 (19)	C22—C21—C17	120.6 (2)
N1—C1—Cu1	129.82 (17)	C22—C21—H21	119.7

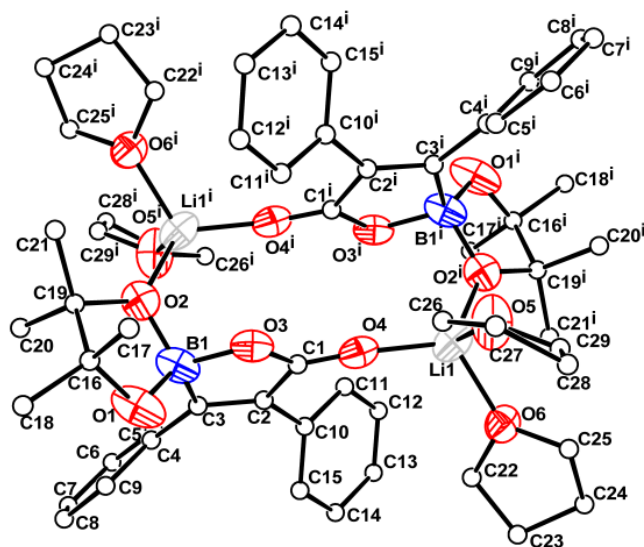
N2—C1—Cu1	125.98 (18)	C17—C21—H21	119.7
C3—C2—N2	106.3 (2)	C21—C22—C23	120.6 (2)
C3—C2—H2	126.9	C21—C22—H22	119.7
N2—C2—H2	126.9	C23—C22—H22	119.7
C2—C3—N1	107.2 (2)	C22—C23—C24	121.5 (2)
C2—C3—H3	126.4	C22—C23—H23	119.3
N1—C3—H3	126.4	C24—C23—H23	119.3
C12—C4—C5	123.7 (2)	C23—C24—C16	117.1 (2)
C12—C4—N1	118.3 (2)	C23—C24—C25	121.5 (2)
C5—C4—N1	118.0 (2)	C16—C24—C25	121.3 (2)
C9—C5—C4	116.5 (2)	C27—C25—C26	110.3 (2)
C9—C5—C6	121.7 (2)	C27—C25—C24	111.2 (2)
C4—C5—C6	121.7 (2)	C26—C25—C24	112.3 (2)
C8—C6—C5	114.7 (3)	C27—C25—H25	107.6
C8—C6—C7'	87.3 (4)	C26—C25—H25	107.6
C5—C6—C7'	111.7 (4)	C24—C25—H25	107.6
C8—C6—C8'	25.8 (3)	C25—C26—H26A	109.5
C5—C6—C8'	105.7 (4)	C25—C26—H26B	109.5
C7'—C6—C8'	112.9 (5)	H26A—C26—H26B	109.5
C8—C6—C7	110.1 (3)	C25—C26—H26C	109.5
C5—C6—C7	110.1 (3)	H26A—C26—H26C	109.5
C7'—C6—C7	25.7 (3)	H26B—C26—H26C	109.5
C8'—C6—C7	134.0 (4)	C25—C27—H27A	109.5
C8—C6—H6	123.2	C25—C27—H27B	109.5
C5—C6—H6	108.8	H27A—C27—H27B	109.5
C7'—C6—H6	108.8	C25—C27—H27C	109.5
C8'—C6—H6	108.8	H27A—C27—H27C	109.5
C7—C6—H6	86.0	H27B—C27—H27C	109.5
C6—C7—H7A	109.5	C35—C28—C29	130.0 (2)
C6—C7—H7B	109.5	C35—C28—C48	109.4 (2)
H7A—C7—H7B	109.5	C29—C28—C48	120.7 (2)
C6—C7—H7C	109.5	C34—C29—C30	117.4 (2)
H7A—C7—H7C	109.5	C34—C29—C28	120.7 (2)
H7B—C7—H7C	109.5	C30—C29—C28	121.9 (2)
C6—C8—H8A	109.5	C31—C30—C29	121.0 (3)
C6—C8—H8B	109.5	C31—C30—H30	119.5

H8A—C8—H8B	109.5	C29—C30—H30	119.5
C6—C8—H8C	109.5	C32—C31—C30	120.8 (3)
H8A—C8—H8C	109.5	C32—C31—H31	119.6
H8B—C8—H8C	109.5	C30—C31—H31	119.6
C6—C7'—H7'1	109.5	C31—C32—C33	119.1 (3)
C6—C7'—H7'2	109.5	C31—C32—H32	120.4
H7'1—C7'—H7'2	109.5	C33—C32—H32	120.4
C6—C7'—H7'3	109.5	C34—C33—C32	120.3 (3)
H7'1—C7'—H7'3	109.5	C34—C33—H33	119.9
H7'2—C7'—H7'3	109.5	C32—C33—H33	119.9
C6—C8'—H8'1	109.5	C33—C34—C29	121.4 (3)
C6—C8'—H8'2	109.5	C33—C34—H34	119.3
H8'1—C8'—H8'2	109.5	C29—C34—H34	119.3
C6—C8'—H8'3	109.5	C28—C35—C36	124.6 (2)
H8'1—C8'—H8'3	109.5	C28—C35—B1	108.2 (2)
H8'2—C8'—H8'3	109.5	C36—C35—B1	127.1 (2)
C10—C9—C5	121.4 (3)	C41—C36—C37	118.1 (2)
C10—C9—H9	119.3	C41—C36—C35	121.1 (2)
C5—C9—H9	119.3	C37—C36—C35	120.7 (2)
C9—C10—C11	120.7 (3)	C38—C37—C36	120.5 (3)
C9—C10—H10	119.7	C38—C37—H37	119.7
C11—C10—H10	119.7	C36—C37—H37	119.7
C10—C11—C12	121.0 (3)	C39—C38—C37	120.5 (3)
C10—C11—H11	119.5	C39—C38—H38	119.7
C12—C11—H11	119.5	C37—C38—H38	119.7
C4—C12—C11	116.8 (2)	C40—C39—C38	119.8 (3)
C4—C12—C13	122.4 (2)	C40—C39—H39	120.1
C11—C12—C13	120.8 (2)	C38—C39—H39	120.1
C12—C13—C14	111.6 (2)	C39—C40—C41	120.2 (3)
C12—C13—C15	110.7 (2)	C39—C40—H40	119.9
C14—C13—C15	111.9 (3)	C41—C40—H40	119.9
C12—C13—H13	107.5	C40—C41—C36	120.7 (3)
C14—C13—H13	107.5	C40—C41—H41	119.6
C15—C13—H13	107.5	C36—C41—H41	119.6
C13—C14—H14A	109.5	O1—C42—C44	107.5 (2)
C13—C14—H14B	109.5	O1—C42—C43	109.6 (2)

H14A—C14—H14B	109.5	C44—C42—C43	110.5 (2)
C13—C14—H14C	109.5	O1—C42—C45	100.76 (18)
H14A—C14—H14C	109.5	C44—C42—C45	113.5 (2)
H14B—C14—H14C	109.5	C43—C42—C45	114.3 (2)
C13—C15—H15A	109.5	C42—C43—H43A	109.5
C13—C15—H15B	109.5	C42—C43—H43B	109.5
H15A—C15—H15B	109.5	H43A—C43—H43B	109.5
C13—C15—H15C	109.5	C42—C43—H43C	109.5
H15A—C15—H15C	109.5	H43A—C43—H43C	109.5
H15B—C15—H15C	109.5	H43B—C43—H43C	109.5
C24—C16—C17	123.0 (2)	C42—C44—H44A	109.5
C24—C16—N2	119.0 (2)	C42—C44—H44B	109.5
C17—C16—N2	118.1 (2)	H44A—C44—H44B	109.5
C21—C17—C16	117.2 (2)	C42—C44—H44C	109.5
C21—C17—C18	120.7 (2)	H44A—C44—H44C	109.5
C16—C17—C18	122.1 (2)	H44B—C44—H44C	109.5
C17—C18—C20	109.2 (8)	O2—C45—C46	108.9 (2)
C17—C18—C19'	107.9 (15)	O2—C45—C47	109.9 (2)
C20—C18—C19'	112 (3)	C46—C45—C47	109.3 (2)
C17—C18—C19	112.5 (8)	O2—C45—C42	101.61 (19)
C20—C18—C19	120.6 (17)	C46—C45—C42	114.1 (2)
C19'—C18—C19	15 (2)	C47—C45—C42	112.7 (2)
C17—C18—C20'	115.1 (13)	C45—C46—H46A	109.5
C20—C18—C20'	18.7 (14)	C45—C46—H46B	109.5
C19'—C18—C20'	93 (3)	H46A—C46—H46B	109.5
C19—C18—C20'	102.9 (19)	C45—C46—H46C	109.5
C17—C18—H18	113.0	H46A—C46—H46C	109.5
C20—C18—H18	102.0	H46B—C46—H46C	109.5
C19'—C18—H18	113.0	C45—C47—H47A	109.5
C19—C18—H18	98.6	C45—C47—H47B	109.5
C20'—C18—H18	113.0	H47A—C47—H47B	109.5
C18—C19—H19A	109.5	C45—C47—H47C	109.5
C18—C19—H19B	109.5	H47A—C47—H47C	109.5
H19A—C19—H19B	109.5	H47B—C47—H47C	109.5
C18—C19—H19C	109.5	O4—C48—O3	122.6 (2)
H19A—C19—H19C	109.5	O4—C48—C28	127.4 (2)

H19B—C19—H19C	109.5	O3—C48—C28	110.1 (2)
C18—C20—H20A	109.5	O2—B1—O3	113.3 (2)
C18—C20—H20B	109.5	O2—B1—O1	103.12 (19)
H20A—C20—H20B	109.5	O3—B1—O1	106.74 (19)
C18—C20—H20C	109.5	O2—B1—C35	115.8 (2)
H20A—C20—H20C	109.5	O3—B1—C35	100.71 (19)
H20B—C20—H20C	109.5	O1—B1—C35	117.2 (2)
C18—C19'—H19D	109.5	C1—N1—C3	111.24 (19)
C18—C19'—H19E	109.5	C1—N1—C4	123.15 (19)
H19D—C19'—H19E	109.5	C3—N1—C4	125.6 (2)
C18—C19'—H19F	109.5	C1—N2—C2	111.2 (2)
H19D—C19'—H19F	109.5	C1—N2—C16	122.46 (19)
H19E—C19'—H19F	109.5	C2—N2—C16	126.32 (19)
C18—C20'—H20D	109.5	C42—O1—B1	109.24 (18)
C18—C20'—H20E	109.5	C42—O1—Cu1	122.91 (13)
H20D—C20'—H20E	109.5	B1—O1—Cu1	119.69 (15)
C18—C20'—H20F	109.5	C45—O2—B1	109.00 (18)
H20D—C20'—H20F	109.5	C48—O3—B1	111.43 (19)
H20E—C20'—H20F	109.5	C1—Cu1—O1	173.33 (9)

X-ray Data for 3



ORTEP drawing of **3**. Thermal ellipsoids set at 30% probability. Hydrogen atoms have been omitted for clarity.

Table 1. Crystal data and structure refinement for **3**.

Identification code	3
Empirical formula	C ₅₈ H ₇₆ B ₂ Li ₂ O ₁₂
Formula weight	1000.69
Temperature	163(2) K
Wavelength	0.71073 Å
Crystal system, space group	Monoclinic, P2 ₁ /c
Unit cell dimensions	a = 13.943(7) Å alpha = 90 °. b = 20.195(10) Å beta = 99.507(11) °. c = 10.094(5) Å gamma = 90 °.
Volume	2803(2) Å ³
Z, Calculated density	2, 1.186 Mg/m ³
Absorption coefficient	0.080 mm ⁻¹
F(000)	1072
Crystal size	0.31 x 0.28 x 0.25 mm
Theta range for data collection	1.48 to 25.00 °.
Limiting indices	-14<= <i>h</i> <=16, -22<= <i>k</i> <=24, -9<= <i>l</i> <=12
Reflections collected / unique	14853 / 4872 [R(int) = 0.0509]
Completeness to theta = 25.00	99.0 %
Absorption correction	Empirical
Max. and min. transmission	0.9803 and 0.9756
Refinement method	Full-matrix least-squares on F ²
Data / restraints / parameters	4872 / 40 / 375
Goodness-of-fit on F ²	1.043
Final R indices [I>2sigma(I)]	R1 = 0.0779, wR2 = 0.1989
R indices (all data)	R1 = 0.1442, wR2 = 0.2427
Largest diff. peak and hole	0.292 and -0.168 e.Å ⁻³

Table 2. Atomic coordinates (x 10⁴) and equivalent isotropic displacement parameters (Å² x 10³) for **3**.

U(eq) is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U _{iso} */U _{eq}	Occ. (<1)
C1	0.4470 (4)	-0.00287 (18)	0.8428 (3)	0.0698 (11)	
C2	0.3775 (3)	0.05343 (16)	0.8093 (3)	0.0685 (11)	
C3	0.2856 (4)	0.03264 (17)	0.8043 (4)	0.0784 (12)	
C4	0.1969 (3)	0.07293 (18)	0.7668 (5)	0.0954 (15)	

C5	0.1879 (3)	0.13563 (19)	0.8179 (5)	0.1041 (16)
H5	0.2399	0.1540	0.8796	0.125*
C6	0.1032 (4)	0.1720 (2)	0.7797 (7)	0.125 (2)
H6	0.0969	0.2143	0.8190	0.150*
C7	0.0288 (5)	0.1482 (3)	0.6867 (8)	0.156 (3)
H7	-0.0284	0.1736	0.6603	0.187*
C8	0.0385 (5)	0.0864 (3)	0.6319 (8)	0.165 (3)
H8	-0.0121	0.0695	0.5659	0.198*
C9	0.1216 (4)	0.0487 (2)	0.6721 (6)	0.138 (3)
H9	0.1268	0.0060	0.6345	0.165*
C10	0.4159 (3)	0.11996 (16)	0.7784 (4)	0.0701 (11)
C11	0.4860 (3)	0.15116 (17)	0.8683 (4)	0.0707 (11)
H11	0.5115	0.1302	0.9509	0.085*
C12	0.5198 (3)	0.21322 (18)	0.8391 (4)	0.0800 (12)
H12	0.5680	0.2349	0.9018	0.096*
C13	0.4834 (4)	0.2433 (2)	0.7191 (5)	0.0920 (14)
H13	0.5067	0.2857	0.6986	0.110*
C14	0.4136 (5)	0.2122 (2)	0.6291 (5)	0.1070 (17)
H14	0.3891	0.2328	0.5458	0.128*
C15	0.3790 (4)	0.15130 (19)	0.6592 (4)	0.0944 (15)
H15	0.3293	0.1305	0.5975	0.113*
C16	0.1957 (4)	-0.1411 (2)	0.8237 (7)	0.121 (2)
C17	0.2721 (4)	-0.19653 (19)	0.8411 (6)	0.1161 (18)
H17A	0.3323	-0.1802	0.8952	0.174*
H17B	0.2850	-0.2104	0.7527	0.174*
H17C	0.2476	-0.2343	0.8865	0.174*
C18	0.1014 (4)	-0.1670 (2)	0.7390 (8)	0.179 (4)
H18A	0.0523	-0.1318	0.7280	0.268*
H18B	0.0774	-0.2049	0.7845	0.268*
H18C	0.1144	-0.1808	0.6506	0.268*
C19	0.1840 (3)	-0.1085 (2)	0.9597 (6)	0.1069 (18)
C20	0.0887 (4)	-0.0692 (2)	0.9514 (8)	0.139 (2)
H20A	0.0816	-0.0394	0.8738	0.208*
H20B	0.0902	-0.0432	1.0337	0.208*
H20C	0.0336	-0.1000	0.9414	0.208*
C21	0.1943 (4)	-0.1551 (2)	1.0790 (6)	0.120 (2)

H21A	0.1856	-0.1302	1.1596	0.181*	
H21B	0.2591	-0.1752	1.0927	0.181*	
H21C	0.1448	-0.1899	1.0619	0.181*	
O6	0.7018 (6)	0.0554 (6)	0.6997 (11)	0.085 (3)	0.512 (5)
C22	0.6359 (13)	0.0988 (12)	0.6193 (17)	0.178 (12)	0.512 (5)
H22A	0.6261	0.1392	0.6709	0.213*	0.512 (5)
H22B	0.5721	0.0767	0.5935	0.213*	0.512 (5)
C23	0.6757 (7)	0.1164 (5)	0.4998 (11)	0.093 (3)	0.512 (5)
H23A	0.6614	0.1631	0.4744	0.111*	0.512 (5)
H23B	0.6491	0.0875	0.4232	0.111*	0.512 (5)
C24	0.7807 (7)	0.1057 (5)	0.5404 (9)	0.101 (3)	0.512 (5)
H24A	0.8125	0.1445	0.5891	0.121*	0.512 (5)
H24B	0.8127	0.0959	0.4622	0.121*	0.512 (5)
C25	0.7815 (6)	0.0475 (4)	0.6299 (10)	0.093 (3)	0.512 (5)
H25A	0.7745	0.0061	0.5766	0.112*	0.512 (5)
H25B	0.8434	0.0456	0.6940	0.112*	0.512 (5)
O6'	0.6558 (9)	0.0528 (5)	0.6543 (9)	0.078 (3)	0.488 (5)
C22'	0.6428 (11)	0.1159 (5)	0.5933 (14)	0.080 (4)	0.488 (5)
H22C	0.7050	0.1310	0.5685	0.096*	0.488 (5)
H22D	0.6238	0.1481	0.6583	0.096*	0.488 (5)
C23'	0.5674 (8)	0.1141 (4)	0.4722 (9)	0.097 (3)	0.488 (5)
H23C	0.5963	0.1170	0.3892	0.116*	0.488 (5)
H23D	0.5195	0.1503	0.4728	0.116*	0.488 (5)
C24'	0.5224 (7)	0.0483 (4)	0.4853 (9)	0.100 (3)	0.488 (5)
H24C	0.4691	0.0510	0.5389	0.120*	0.488 (5)
H24D	0.4969	0.0290	0.3963	0.120*	0.488 (5)
C25'	0.6052 (7)	0.0094 (4)	0.5559 (8)	0.089 (3)	0.488 (5)
H25C	0.5818	-0.0302	0.5990	0.107*	0.488 (5)
H25D	0.6480	-0.0052	0.4924	0.107*	0.488 (5)
Li1	0.6673 (6)	0.0195 (3)	0.8593 (7)	0.084 (2)	
B1	0.2891 (4)	-0.0453 (2)	0.8477 (5)	0.0805 (15)	
O3	0.3999 (2)	-0.05818 (11)	0.8595 (2)	0.0731 (8)	
O4	0.5355 (2)	-0.00081 (12)	0.8520 (3)	0.0766 (8)	
O1	0.2317 (3)	-0.08802 (13)	0.7520 (3)	0.1121 (12)	
O2	0.26342 (19)	-0.06147 (11)	0.9788 (3)	0.0811 (8)	
O5	0.7292 (2)	-0.06785 (13)	0.8381 (4)	0.1110 (12)	

C26	0.6674 (10)	-0.1284 (7)	0.8174 (18)	0.1055 (16)	0.472 (5)
H26A	0.6080	-0.1204	0.7508	0.127*	0.472 (5)
H26B	0.6485	-0.1435	0.9028	0.127*	0.472 (5)
C27	0.7326 (9)	-0.1779 (6)	0.7660 (17)	0.1055 (16)	0.472 (5)
H27A	0.6943	-0.2073	0.6987	0.127*	0.472 (5)
H27B	0.7662	-0.2054	0.8407	0.127*	0.472 (5)
C28	0.8080 (8)	-0.1376 (4)	0.7006 (12)	0.1055 (16)	0.472 (5)
H28A	0.8732	-0.1582	0.7191	0.127*	0.472 (5)
H28B	0.7874	-0.1335	0.6023	0.127*	0.472 (5)
C29	0.8070 (8)	-0.0732 (4)	0.7674 (14)	0.1055 (16)	0.472 (5)
H29A	0.8688	-0.0673	0.8304	0.127*	0.472 (5)
H29B	0.8023	-0.0376	0.6994	0.127*	0.472 (5)
C26'	0.6803 (9)	-0.1209 (6)	0.7760 (16)	0.1055 (16)	0.528 (5)
H26C	0.6313	-0.1055	0.6999	0.127*	0.528 (5)
H26D	0.6459	-0.1446	0.8401	0.127*	0.528 (5)
C27'	0.7495 (8)	-0.1664 (5)	0.7260 (14)	0.1055 (16)	0.528 (5)
H27C	0.7630	-0.1524	0.6369	0.127*	0.528 (5)
H27D	0.7250	-0.2125	0.7203	0.127*	0.528 (5)
C28'	0.8390 (6)	-0.1592 (4)	0.8339 (11)	0.1055 (16)	0.528 (5)
H28C	0.8345	-0.1884	0.9114	0.127*	0.528 (5)
H28D	0.8988	-0.1702	0.7978	0.127*	0.528 (5)
C29'	0.8389 (7)	-0.0883 (4)	0.8741 (13)	0.1055 (16)	0.528 (5)
H29C	0.8798	-0.0615	0.8231	0.127*	0.528 (5)
H29D	0.8630	-0.0830	0.9714	0.127*	0.528 (5)

Table 3. Bond lengths [Å] and angles [°] for **3**.

C1—O4	1.222 (5)	C23—H23A	0.9900
C1—O3	1.320 (5)	C23—H23B	0.9900
C1—C2	1.496 (5)	C24—C25	1.482 (10)
C2—C3	1.342 (6)	C24—H24A	0.9900
C2—C10	1.498 (5)	C24—H24B	0.9900
C3—C4	1.476 (6)	C25—H25A	0.9900
C3—B1	1.633 (6)	C25—H25B	0.9900
C4—C5	1.381 (6)	O6'—C22'	1.414 (12)
C4—C9	1.387 (6)	O6'—C25'	1.422 (10)
C5—C6	1.390 (6)	O6'—Li1	2.157 (12)

C5—H5	0.9500	C22'—C23'	1.476 (12)
C6—C7	1.367 (7)	C22'—H22C	0.9900
C6—H6	0.9500	C22'—H22D	0.9900
C7—C8	1.380 (9)	C23'—C24'	1.485 (11)
C7—H7	0.9500	C23'—H23C	0.9900
C8—C9	1.389 (7)	C23'—H23D	0.9900
C8—H8	0.9500	C24'—C25'	1.479 (11)
C9—H9	0.9500	C24'—H24C	0.9900
C10—C11	1.372 (5)	C24'—H24D	0.9900
C10—C15	1.381 (5)	C25'—H25C	0.9900
C11—C12	1.388 (5)	C25'—H25D	0.9900
C11—H11	0.9500	Li1—O4	1.873 (8)
C12—C13	1.375 (6)	Li1—O2 ⁱ	1.949 (8)
C12—H12	0.9500	Li1—O5	1.991 (7)
C13—C14	1.369 (6)	B1—O1	1.435 (5)
C13—H13	0.9500	B1—O2	1.464 (6)
C14—C15	1.374 (6)	B1—O3	1.552 (6)
C14—H14	0.9500	O2—Li1 ⁱ	1.949 (8)
C15—H15	0.9500	O5—C26'	1.366 (13)
C16—O1	1.429 (6)	O5—C29	1.398 (11)
C16—C17	1.535 (6)	O5—C26	1.491 (14)
C16—C18	1.537 (6)	O5—C29'	1.569 (9)
C16—C19	1.555 (8)	C26—C27	1.499 (11)
C17—H17A	0.9800	C26—H26A	0.9900
C17—H17B	0.9800	C26—H26B	0.9900
C17—H17C	0.9800	C27—C28	1.559 (13)
C18—H18A	0.9800	C27—H27A	0.9900
C18—H18B	0.9800	C27—H27B	0.9900
C18—H18C	0.9800	C28—C29	1.465 (10)
C19—O2	1.448 (5)	C28—H28A	0.9900
C19—C21	1.516 (7)	C28—H28B	0.9900
C19—C20	1.538 (6)	C29—H29A	0.9900
C20—H20A	0.9800	C29—H29B	0.9900
C20—H20B	0.9800	C26'—C27'	1.479 (10)
C20—H20C	0.9800	C26'—H26C	0.9900
C21—H21A	0.9800	C26'—H26D	0.9900

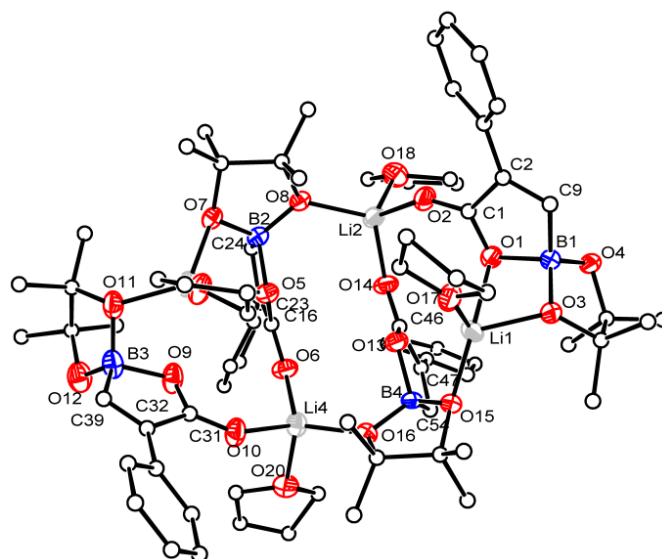
C21—H21B	0.9800	C27'—C28'	1.522 (13)
C21—H21C	0.9800	C27'—H27C	0.9900
O6—C25	1.420 (11)	C27'—H27D	0.9900
O6—C22	1.423 (12)	C28'—C29'	1.487 (9)
O6—Li1	1.899 (13)	C28'—H28C	0.9900
C22—C23	1.453 (14)	C28'—H28D	0.9900
C22—H22A	0.9900	C29'—H29C	0.9900
C22—H22B	0.9900	C29'—H29D	0.9900
C23—C24	1.469 (11)		
O4—C1—O3	122.4 (3)	C22'—O6'—C25'	103.8 (7)
O4—C1—C2	126.8 (4)	C22'—O6'—Li1	133.0 (8)
O3—C1—C2	110.8 (4)	C25'—O6'—Li1	115.3 (7)
C3—C2—C1	110.5 (4)	O6'—C22'—C23'	111.1 (8)
C3—C2—C10	130.2 (3)	O6'—C22'—H22C	109.4
C1—C2—C10	119.2 (4)	C23'—C22'—H22C	109.4
C2—C3—C4	126.3 (4)	O6'—C22'—H22D	109.4
C2—C3—B1	107.8 (3)	C23'—C22'—H22D	109.4
C4—C3—B1	125.9 (4)	H22C—C22'—H22D	108.0
C5—C4—C9	118.3 (4)	C22'—C23'—C24'	101.7 (7)
C5—C4—C3	122.3 (4)	C22'—C23'—H23C	111.4
C9—C4—C3	119.3 (4)	C24'—C23'—H23C	111.4
C4—C5—C6	120.5 (4)	C22'—C23'—H23D	111.4
C4—C5—H5	119.8	C24'—C23'—H23D	111.4
C6—C5—H5	119.8	H23C—C23'—H23D	109.3
C7—C6—C5	121.1 (5)	C25'—C24'—C23'	102.2 (8)
C7—C6—H6	119.4	C25'—C24'—H24C	111.3
C5—C6—H6	119.4	C23'—C24'—H24C	111.3
C6—C7—C8	118.8 (5)	C25'—C24'—H24D	111.3
C6—C7—H7	120.6	C23'—C24'—H24D	111.3
C8—C7—H7	120.6	H24C—C24'—H24D	109.2
C7—C8—C9	120.7 (5)	O6'—C25'—C24'	105.5 (7)
C7—C8—H8	119.7	O6'—C25'—H25C	110.6
C9—C8—H8	119.7	C24'—C25'—H25C	110.6
C4—C9—C8	120.6 (5)	O6'—C25'—H25D	110.6
C4—C9—H9	119.7	C24'—C25'—H25D	110.6

C8—C9—H9	119.7	H25C—C25'—H25D	108.8
C11—C10—C15	119.2 (4)	O4—Li1—O6	115.8 (5)
C11—C10—C2	120.9 (3)	O4—Li1—O2 ⁱ	118.4 (4)
C15—C10—C2	119.9 (4)	O6—Li1—O2 ⁱ	112.7 (5)
C10—C11—C12	120.3 (4)	O4—Li1—O5	104.0 (3)
C10—C11—H11	119.9	O6—Li1—O5	94.1 (4)
C12—C11—H11	119.9	O2 ⁱ —Li1—O5	108.2 (4)
C13—C12—C11	119.8 (4)	O4—Li1—O6'	96.5 (5)
C13—C12—H12	120.1	O6—Li1—O6'	19.3 (3)
C11—C12—H12	120.1	O2 ⁱ —Li1—O6'	128.1 (4)
C14—C13—C12	120.0 (4)	O5—Li1—O6'	97.9 (4)
C14—C13—H13	120.0	O1—B1—O2	106.4 (4)
C12—C13—H13	120.0	O1—B1—O3	112.9 (4)
C13—C14—C15	120.1 (4)	O2—B1—O3	106.4 (3)
C13—C14—H14	120.0	O1—B1—C3	114.2 (3)
C15—C14—H14	120.0	O2—B1—C3	117.2 (3)
C14—C15—C10	120.6 (5)	O3—B1—C3	99.6 (4)
C14—C15—H15	119.7	C1—O3—B1	111.0 (3)
C10—C15—H15	119.7	C1—O4—Li1	169.1 (3)
O1—C16—C17	108.0 (5)	C16—O1—B1	108.3 (4)
O1—C16—C18	108.3 (5)	C19—O2—B1	108.7 (4)
C17—C16—C18	109.2 (4)	C19—O2—Li1 ⁱ	131.3 (4)
O1—C16—C19	103.1 (3)	B1—O2—Li1 ⁱ	120.0 (3)
C17—C16—C19	112.3 (4)	C26'—O5—C29	94.3 (6)
C18—C16—C19	115.5 (5)	C26'—O5—C26	19.7 (10)
C16—C17—H17A	109.5	C29—O5—C26	110.1 (6)
C16—C17—H17B	109.5	C26'—O5—C29'	107.0 (6)
H17A—C17—H17B	109.5	C29—O5—C29'	44.6 (6)
C16—C17—H17C	109.5	C26—O5—C29'	109.6 (7)
H17A—C17—H17C	109.5	C26'—O5—Li1	123.6 (6)
H17B—C17—H17C	109.5	C29—O5—Li1	120.7 (5)
C16—C18—H18A	109.5	C26—O5—Li1	119.5 (6)
C16—C18—H18B	109.5	C29'—O5—Li1	129.2 (4)
H18A—C18—H18B	109.5	O5—C26—C27	103.1 (8)
C16—C18—H18C	109.5	O5—C26—H26A	111.2
H18A—C18—H18C	109.5	C27—C26—H26A	111.2

H18B—C18—H18C	109.5	O5—C26—H26B	111.2
O2—C19—C21	109.3 (4)	C27—C26—H26B	111.2
O2—C19—C20	107.7 (3)	H26A—C26—H26B	109.1
C21—C19—C20	109.3 (5)	C26—C27—C28	106.8 (8)
O2—C19—C16	101.9 (4)	C26—C27—H27A	110.4
C21—C19—C16	115.4 (4)	C28—C27—H27A	110.4
C20—C19—C16	112.7 (5)	C26—C27—H27B	110.4
C19—C20—H20A	109.5	C28—C27—H27B	110.4
C19—C20—H20B	109.5	H27A—C27—H27B	108.6
H20A—C20—H20B	109.5	C29—C28—C27	102.1 (8)
C19—C20—H20C	109.5	C29—C28—H28A	111.3
H20A—C20—H20C	109.5	C27—C28—H28A	111.3
H20B—C20—H20C	109.5	C29—C28—H28B	111.3
C19—C21—H21A	109.5	C27—C28—H28B	111.3
C19—C21—H21B	109.5	H28A—C28—H28B	109.2
H21A—C21—H21B	109.5	O5—C29—C28	111.8 (8)
C19—C21—H21C	109.5	O5—C29—H29A	109.3
H21A—C21—H21C	109.5	C28—C29—H29A	109.3
H21B—C21—H21C	109.5	O5—C29—H29B	109.3
C25—O6—C22	105.7 (10)	C28—C29—H29B	109.3
C25—O6—Li1	135.0 (7)	H29A—C29—H29B	107.9
C22—O6—Li1	119.2 (9)	O5—C26'—C27'	109.9 (8)
O6—C22—C23	108.9 (11)	O5—C26'—H26C	109.7
O6—C22—H22A	109.9	C27'—C26'—H26C	109.7
C23—C22—H22A	109.9	O5—C26'—H26D	109.7
O6—C22—H22B	109.9	C27'—C26'—H26D	109.7
C23—C22—H22B	109.9	H26C—C26'—H26D	108.2
H22A—C22—H22B	108.3	C26'—C27'—C28'	101.6 (9)
C22—C23—C24	103.4 (9)	C26'—C27'—H27C	111.5
C22—C23—H23A	111.1	C28'—C27'—H27C	111.5
C24—C23—H23A	111.1	C26'—C27'—H27D	111.5
C22—C23—H23B	111.1	C28'—C27'—H27D	111.5
C24—C23—H23B	111.1	H27C—C27'—H27D	109.3
H23A—C23—H23B	109.0	C29'—C28'—C27'	104.5 (7)
C23—C24—C25	101.1 (7)	C29'—C28'—H28C	110.9
C23—C24—H24A	111.5	C27'—C28'—H28C	110.9

C25—C24—H24A	111.5	C29'—C28'—H28D	110.9
C23—C24—H24B	111.5	C27'—C28'—H28D	110.9
C25—C24—H24B	111.5	H28C—C28'—H28D	108.9
H24A—C24—H24B	109.4	C28'—C29'—O5	103.6 (6)
O6—C25—C24	106.6 (7)	C28'—C29'—H29C	111.0
O6—C25—H25A	110.4	O5—C29'—H29C	111.0
C24—C25—H25A	110.4	C28'—C29'—H29D	111.0
O6—C25—H25B	110.4	O5—C29'—H29D	111.0
C24—C25—H25B	110.4	H29C—C29'—H29D	109.0
H25A—C25—H25B	108.6		

X-ray Data for **4k**



ORTEP drawing of **4k**. Thermal ellipsoids set at 30% probability. Hydrogen atoms have been omitted for clarity.

Table 1. Crystal data and structure refinement for **4k**.

Identification code	4k
Empirical formula	C ₇₆ H ₁₀₄ B ₄ Li ₄ O ₂₀
Formula weight	1408.59
Temperature	163(2) K
Wavelength	0.71073 Å
Crystal system, space group	Monoclinic, C2/c
Unit cell dimensions	a = 47.510(7) Å alpha = 90 °. b = 17.784(3) Å beta = 92.437(3) °. c = 19.386(3) Å gamma = 90 °.
Volume	16365(5) Å ³
Z, Calculated density	8, 1.143 Mg/m ³
Absorption coefficient	0.079 mm ⁻¹
F(000)	6016
Crystal size	0.28 x 0.22 x 0.20 mm
Theta range for data collection	1.22 to 25.00 °.
Limiting indices	-56<=h<=51, -21<=k<=14, -23<=l<=21
Reflections collected / unique	42079 / 14249 [R(int) = 0.0745]
Completeness to theta = 25.00	98.9 %
Absorption correction	Empirical
Max. and min. transmission	0.9843 and 0.9781
Refinement method	Full-matrix least-squares on F ²
Data / restraints / parameters	14249 / 38 / 950
Goodness-of-fit on F ²	1.098
Final R indices [I>2sigma(I)]	R1 = 0.0762, wR2 = 0.1534
R indices (all data)	R1 = 0.1608, wR2 = 0.1742
Largest diff. peak and hole	0.588 and -0.339 e.Å ⁻³

Table 2. Atomic coordinates (x 10⁴) and equivalent isotropic displacement parameters (Å² x 10³) for **4k**.
U(eq) is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U _{iso} */U _{eq}	Occ. (<1)
C1	0.12958 (8)	0.0621 (2)	0.56361 (18)	0.0485 (10)	
C2	0.14155 (7)	-0.0134 (2)	0.54832 (17)	0.0426 (9)	
C3	0.17066 (7)	-0.0224 (2)	0.52379 (18)	0.0455 (9)	
C4	0.18235 (8)	0.0286 (2)	0.4789 (2)	0.0602 (11)	

H4	0.1716	0.0704	0.4624	0.072*
C5	0.20959 (9)	0.0184 (3)	0.4583 (2)	0.0704 (12)
H5	0.2174	0.0533	0.4275	0.085*
C6	0.22527 (9)	-0.0411 (3)	0.4816 (2)	0.0701 (13)
H6	0.2440	-0.0473	0.4674	0.084*
C7	0.21396 (9)	-0.0922 (3)	0.5257 (2)	0.0724 (13)
H7	0.2247	-0.1341	0.5419	0.087*
C8	0.18677 (8)	-0.0819 (2)	0.5462 (2)	0.0597 (11)
H8	0.1790	-0.1173	0.5767	0.072*
C9	0.12242 (8)	-0.0658 (2)	0.56173 (18)	0.0458 (10)
C10	0.05839 (8)	-0.0785 (2)	0.6535 (2)	0.0572 (11)
C11	0.04225 (9)	-0.0607 (3)	0.7171 (2)	0.0813 (15)
H11A	0.0389	-0.0064	0.7195	0.122*
H11B	0.0532	-0.0770	0.7584	0.122*
H11C	0.0242	-0.0872	0.7146	0.122*
C12	0.06369 (9)	-0.1618 (2)	0.6473 (2)	0.0754 (13)
H12A	0.0721	-0.1808	0.6909	0.113*
H12B	0.0766	-0.1710	0.6101	0.113*
H12C	0.0458	-0.1878	0.6369	0.113*
C13	0.04558 (8)	-0.0440 (3)	0.5868 (2)	0.0604 (11)
C14	0.03459 (8)	0.0367 (3)	0.5959 (2)	0.0810 (14)
H14A	0.0491	0.0671	0.6201	0.121*
H14B	0.0176	0.0356	0.6228	0.121*
H14C	0.0301	0.0588	0.5504	0.121*
C15	0.02297 (8)	-0.0922 (3)	0.5503 (2)	0.0871 (16)
H15A	0.0157	-0.0661	0.5087	0.131*
H15B	0.0076	-0.1008	0.5814	0.131*
H15C	0.0311	-0.1405	0.5374	0.131*

C16	0.12479 (8)	0.3854 (2)	0.68961 (19)	0.0468 (10)
C17	0.12416 (8)	0.4403 (2)	0.6325 (2)	0.0533 (10)
C18	0.10274 (9)	0.5027 (3)	0.6221 (2)	0.0777 (15)
C19	0.09370 (11)	0.5418 (3)	0.6784 (3)	0.0999 (17)
H19	0.1002	0.5284	0.7237	0.120*
C20	0.07450 (13)	0.6026 (4)	0.6676 (4)	0.124 (2)
H20	0.0679	0.6297	0.7060	0.149*
C21	0.06585 (12)	0.6213 (4)	0.6037 (4)	0.108 (2)
H21	0.0525	0.6608	0.5973	0.130*
C22	0.07497 (11)	0.5875 (3)	0.5496 (4)	0.1011 (18)
H22	0.0687	0.6024	0.5044	0.121*
C23	0.09444 (10)	0.5280 (3)	0.5595 (3)	0.0904 (15)
H23	0.1020	0.5050	0.5201	0.108*
C24	0.14634 (8)	0.4290 (2)	0.5936 (2)	0.0463 (10)
C25	0.21246 (7)	0.3420 (2)	0.60508 (18)	0.0485 (10)
C26	0.24043 (7)	0.3274 (3)	0.6425 (2)	0.0666 (12)
H26A	0.2378	0.2921	0.6806	0.100*
H26B	0.2481	0.3748	0.6610	0.100*
H26C	0.2536	0.3057	0.6104	0.100*
C27	0.21633 (8)	0.3971 (2)	0.54578 (19)	0.0631 (11)
H27A	0.1981	0.4059	0.5214	0.095*
H27B	0.2296	0.3760	0.5137	0.095*
H27C	0.2237	0.4448	0.5642	0.095*
C28	0.19612 (7)	0.2705 (2)	0.58130 (19)	0.0482 (10)
C29	0.19965 (8)	0.2059 (2)	0.6316 (2)	0.0678 (12)
H29A	0.1956	0.2232	0.6781	0.102*
H29B	0.2191	0.1872	0.6314	0.102*
H29C	0.1866	0.1653	0.6179	0.102*

C30	0.20215 (8)	0.2434 (3)	0.5091 (2)	0.0725 (13)
H30A	0.1999	0.2853	0.4765	0.109*
H30B	0.1890	0.2031	0.4956	0.109*
H30C	0.2215	0.2243	0.5085	0.109*
C31	0.15033 (9)	0.3875 (2)	0.8887 (2)	0.0549 (10)
C32	0.17054 (8)	0.3910 (2)	0.94996 (18)	0.0477 (10)
C33	0.16792 (8)	0.3442 (2)	1.01182 (19)	0.0506 (10)
C34	0.14161 (9)	0.3231 (2)	1.0351 (2)	0.0598 (11)
H34	0.1250	0.3382	1.0100	0.072*
C35	0.13961 (10)	0.2805 (2)	1.0942 (2)	0.0687 (12)
H35	0.1216	0.2667	1.1095	0.082*
C36	0.16350 (13)	0.2578 (3)	1.1313 (2)	0.0793 (14)
H36	0.1621	0.2286	1.1720	0.095*
C37	0.18948 (11)	0.2780 (3)	1.1085 (2)	0.0753 (13)
H37	0.2061	0.2621	1.1333	0.090*
C38	0.19151 (8)	0.3207 (2)	1.0505 (2)	0.0594 (11)
H38	0.2096	0.3347	1.0361	0.071*
C39	0.19006 (9)	0.4403 (3)	0.9347 (2)	0.0606 (12)
C40	0.18988 (12)	0.5895 (3)	0.8006 (3)	0.0850 (15)
C41	0.19725 (13)	0.6719 (3)	0.8140 (3)	0.117 (2)
H41A	0.2125	0.6751	0.8497	0.175*
H41B	0.1806	0.6984	0.8297	0.175*
H41C	0.2034	0.6950	0.7713	0.175*
C42	0.16587 (12)	0.5852 (3)	0.7441 (3)	0.115 (2)
H42A	0.1607	0.5325	0.7358	0.172*
H42B	0.1723	0.6075	0.7013	0.172*
H42C	0.1494	0.6129	0.7594	0.172*
C43	0.21451 (11)	0.5378 (3)	0.7862 (2)	0.0773 (14)

C44	0.24059 (10)	0.5546 (3)	0.8322 (3)	0.1077 (18)
H44A	0.2354	0.5564	0.8806	0.162*
H44B	0.2485	0.6031	0.8192	0.162*
H44C	0.2546	0.5150	0.8265	0.162*
C45	0.22289 (12)	0.5359 (3)	0.7114 (2)	0.113 (2)
H45A	0.2062	0.5257	0.6813	0.169*
H45B	0.2369	0.4962	0.7055	0.169*
H45C	0.2309	0.5846	0.6991	0.169*
C46	0.08759 (8)	0.2331 (2)	0.6424 (2)	0.0539 (11)
C47	0.05692 (7)	0.2468 (3)	0.64956 (19)	0.0593 (11)
C48	0.04006 (8)	0.2906 (2)	0.5955 (2)	0.0812 (15)
C49	0.04845 (8)	0.3613 (2)	0.5737 (3)	0.1056 (19)
H49	0.0653	0.3831	0.5925	0.127*
C50	0.03214 (11)	0.4002 (3)	0.5243 (3)	0.119 (2)
H50	0.0385	0.4474	0.5083	0.143*
C51	0.00685 (11)	0.3716 (3)	0.4979 (3)	0.131 (2)
H51	-0.0046	0.3990	0.4653	0.157*
C52	-0.00104 (9)	0.3011 (3)	0.5211 (3)	0.138 (3)
H52	-0.0169	0.2763	0.5002	0.166*
C53	0.01431 (10)	0.2668 (3)	0.5751 (3)	0.122 (2)
H53	0.0063	0.2253	0.5981	0.146*
C54	0.04953 (8)	0.2172 (2)	0.7105 (2)	0.0580 (12)
C55	0.09038 (7)	0.1687 (2)	0.87015 (18)	0.0488 (10)
C56	0.12211 (8)	0.1559 (2)	0.8766 (2)	0.0611 (11)
H56A	0.1287	0.1361	0.8330	0.092*
H56B	0.1264	0.1198	0.9137	0.092*
H56C	0.1316	0.2037	0.8873	0.092*
C57	0.07991 (8)	0.2001 (2)	0.93673 (17)	0.0561 (10)

H57A	0.0595	0.2080	0.9320	0.084*	
H57B	0.0893	0.2482	0.9470	0.084*	
H57C	0.0842	0.1646	0.9744	0.084*	
C58	0.07435 (8)	0.0977 (2)	0.84315 (19)	0.0547 (10)	
C59	0.04286 (8)	0.1013 (3)	0.8564 (2)	0.0773 (14)	
H59A	0.0351	0.1491	0.8392	0.116*	
H59B	0.0401	0.0975	0.9061	0.116*	
H59C	0.0331	0.0596	0.8325	0.116*	
C60	0.08668 (9)	0.0236 (2)	0.8692 (2)	0.0740 (13)	
H60A	0.0755	-0.0181	0.8498	0.111*	
H60B	0.0864	0.0222	0.9197	0.111*	
H60C	0.1061	0.0190	0.8549	0.111*	
C61	0.14228 (10)	-0.0657 (3)	0.7465 (3)	0.0835 (14)	
H61A	0.1352	-0.0980	0.7080	0.100*	
H61B	0.1329	-0.0806	0.7890	0.100*	
C62	0.1670 (4)	-0.0718 (9)	0.7770 (11)	0.113 (3)	0.477 (14)
H62A	0.1748	-0.1227	0.7700	0.136*	0.477 (14)
H62B	0.1659	-0.0626	0.8272	0.136*	0.477 (14)
C63	0.1862 (3)	-0.0095 (11)	0.7423 (10)	0.113 (3)	0.477 (14)
H63A	0.2018	0.0079	0.7740	0.136*	0.477 (14)
H63B	0.1939	-0.0270	0.6984	0.136*	0.477 (14)
C62'	0.1774 (3)	-0.0726 (9)	0.7575 (12)	0.113 (3)	0.523 (14)
H62C	0.1834	-0.1071	0.7955	0.136*	0.523 (14)
H62D	0.1867	-0.0864	0.7146	0.136*	0.523 (14)
C63'	0.1813 (3)	0.0130 (6)	0.7772 (11)	0.113 (3)	0.523 (14)
H63C	0.1759	0.0224	0.8252	0.136*	0.523 (14)
H63D	0.2011	0.0291	0.7723	0.136*	0.523 (14)
C64	0.16373 (10)	0.0493 (3)	0.7312 (3)	0.0948 (16)	

H64A	0.1714	0.0478	0.6845	0.114*	
H64B	0.1616	0.1026	0.7449	0.114*	
C65	0.1064 (3)	0.1931 (6)	0.4287 (7)	0.127 (3)	0.517 (10)
H65A	0.1157	0.1532	0.4022	0.152*	0.517 (10)
H65B	0.0965	0.1694	0.4671	0.152*	0.517 (10)
C66	0.0867 (3)	0.2353 (6)	0.3843 (7)	0.127 (3)	0.517 (10)
H66A	0.0865	0.2150	0.3368	0.152*	0.517 (10)
H66B	0.0674	0.2312	0.4016	0.152*	0.517 (10)
C67	0.0956 (3)	0.3133 (6)	0.3845 (7)	0.127 (3)	0.517 (10)
H67A	0.0800	0.3457	0.3992	0.152*	0.517 (10)
H67B	0.1004	0.3284	0.3373	0.152*	0.517 (10)
C68	0.1208 (5)	0.3238 (6)	0.4328 (10)	0.127 (3)	0.517 (10)
H68A	0.1367	0.3461	0.4087	0.152*	0.517 (10)
H68B	0.1162	0.3563	0.4723	0.152*	0.517 (10)
C65'	0.1258 (4)	0.2084 (7)	0.3937 (5)	0.141 (4)	0.483 (10)
H65C	0.1143	0.1625	0.3988	0.170*	0.483 (10)
H65D	0.1450	0.1931	0.3815	0.170*	0.483 (10)
C66'	0.1129 (4)	0.2570 (7)	0.3381 (6)	0.141 (4)	0.483 (10)
H66C	0.1247	0.2559	0.2973	0.170*	0.483 (10)
H66D	0.0940	0.2379	0.3241	0.170*	0.483 (10)
C67'	0.1107 (4)	0.3355 (7)	0.3638 (5)	0.141 (4)	0.483 (10)
H67C	0.0910	0.3536	0.3615	0.170*	0.483 (10)
H67D	0.1226	0.3705	0.3379	0.170*	0.483 (10)
C68'	0.1216 (6)	0.3255 (6)	0.4366 (8)	0.141 (4)	0.483 (10)
H68C	0.1393	0.3546	0.4434	0.170*	0.483 (10)
H68D	0.1077	0.3465	0.4680	0.170*	0.483 (10)
C69	0.18706 (9)	0.2318 (3)	0.8222 (2)	0.0732 (13)	
H69A	0.1668	0.2443	0.8235	0.088*	

H69B	0.1893	0.1927	0.7865	0.088*	
C70	0.2079 (2)	0.1781 (7)	0.8422 (6)	0.0734 (16)	0.414 (6)
H70A	0.2136	0.1481	0.8021	0.088*	0.414 (6)
H70B	0.2010	0.1437	0.8779	0.088*	0.414 (6)
C71	0.2316 (3)	0.2254 (10)	0.8702 (6)	0.0734 (16)	0.414 (6)
H71A	0.2313	0.2269	0.9212	0.088*	0.414 (6)
H71B	0.2497	0.2035	0.8571	0.088*	0.414 (6)
C70'	0.19656 (16)	0.2046 (5)	0.8860 (4)	0.0734 (16)	0.586 (6)
H70C	0.1954	0.1490	0.8867	0.088*	0.586 (6)
H70D	0.1849	0.2247	0.9228	0.088*	0.586 (6)
C71'	0.2264 (2)	0.2292 (7)	0.8976 (4)	0.0734 (16)	0.586 (6)
H71C	0.2396	0.1880	0.8877	0.088*	0.586 (6)
H71D	0.2300	0.2468	0.9456	0.088*	0.586 (6)
C72	0.22891 (8)	0.2957 (3)	0.8444 (2)	0.0746 (13)	
H72A	0.2321	0.3440	0.8689	0.090*	
H72B	0.2448	0.2868	0.8139	0.090*	
O20	0.0688 (8)	0.3919 (15)	0.853 (5)	0.0706 (13)	0.44 (2)
C73	0.0771 (4)	0.4646 (14)	0.8719 (17)	0.158 (6)	0.44 (2)
H73A	0.0905	0.4850	0.8389	0.190*	0.44 (2)
H73B	0.0864	0.4641	0.9185	0.190*	0.44 (2)
C74	0.0511 (3)	0.5118 (9)	0.8716 (16)	0.158 (6)	0.44 (2)
H74A	0.0486	0.5334	0.9180	0.190*	0.44 (2)
H74B	0.0525	0.5536	0.8382	0.190*	0.44 (2)
C75	0.0293 (3)	0.4676 (10)	0.8536 (15)	0.158 (6)	0.44 (2)
H75A	0.0159	0.4942	0.8216	0.190*	0.44 (2)
H75B	0.0192	0.4515	0.8948	0.190*	0.44 (2)
C76	0.0426 (4)	0.3989 (13)	0.8174 (15)	0.158 (6)	0.44 (2)
H76A	0.0310	0.3531	0.8227	0.190*	0.44 (2)

H76B	0.0448	0.4088	0.7677	0.190*	0.44 (2)
O20'	0.0689 (6)	0.3879 (11)	0.851 (4)	0.0706 (13)	0.56 (2)
C73'	0.0721 (4)	0.4663 (11)	0.8554 (13)	0.153 (4)	0.56 (2)
H73C	0.0888	0.4833	0.8307	0.184*	0.56 (2)
H73D	0.0743	0.4824	0.9043	0.184*	0.56 (2)
C74'	0.0458 (3)	0.4971 (8)	0.8225 (12)	0.153 (4)	0.56 (2)
H74C	0.0385	0.5380	0.8516	0.184*	0.56 (2)
H74D	0.0498	0.5186	0.7769	0.184*	0.56 (2)
C75'	0.0263 (3)	0.4431 (9)	0.8147 (11)	0.153 (4)	0.56 (2)
H75C	0.0168	0.4460	0.7683	0.184*	0.56 (2)
H75D	0.0119	0.4480	0.8499	0.184*	0.56 (2)
C76'	0.0418 (4)	0.3710 (8)	0.8233 (12)	0.153 (4)	0.56 (2)
H76C	0.0320	0.3373	0.8550	0.184*	0.56 (2)
H76D	0.0431	0.3454	0.7782	0.184*	0.56 (2)
Li1	0.09988 (14)	0.0526 (4)	0.7025 (3)	0.0564 (17)	
Li2	0.13564 (12)	0.2288 (3)	0.5541 (3)	0.0521 (16)	
Li3	0.18786 (13)	0.3820 (4)	0.7520 (3)	0.0559 (17)	
Li4	0.09924 (12)	0.3259 (4)	0.8167 (3)	0.0545 (16)	
B1	0.09429 (9)	-0.0284 (3)	0.5869 (2)	0.0496 (12)	
B2	0.16479 (8)	0.3603 (2)	0.6260 (2)	0.0436 (11)	
B3	0.18414 (11)	0.4791 (3)	0.8622 (3)	0.0625 (14)	
B4	0.07687 (9)	0.1827 (3)	0.7516 (2)	0.0499 (12)	
O1	0.10428 (5)	0.05659 (13)	0.58835 (12)	0.0515 (7)	
O2	0.14165 (5)	0.12191 (15)	0.55624 (14)	0.0633 (7)	
O3	0.08576 (5)	-0.04153 (14)	0.65886 (12)	0.0539 (7)	
O4	0.06965 (5)	-0.04038 (14)	0.54341 (12)	0.0525 (7)	
O5	0.14812 (5)	0.34399 (13)	0.69127 (11)	0.0450 (6)	
O6	0.10636 (5)	0.37628 (16)	0.73105 (13)	0.0596 (7)	

O7	0.19344 (4)	0.37594 (13)	0.65223 (11)	0.0466 (6)
O8	0.16716 (4)	0.29485 (13)	0.58103 (11)	0.0440 (6)
O9	0.15751 (5)	0.43260 (15)	0.83941 (14)	0.0649 (8)
O10	0.12956 (6)	0.34626 (16)	0.88419 (13)	0.0657 (8)
O11	0.20377 (6)	0.46553 (15)	0.80567 (13)	0.0656 (8)
O12	0.17954 (6)	0.55836 (16)	0.86341 (15)	0.0770 (9)
O13	0.09871 (5)	0.19408 (14)	0.69629 (12)	0.0519 (7)
O14	0.10124 (5)	0.25380 (15)	0.59482 (12)	0.0563 (7)
O15	0.07772 (5)	0.10306 (15)	0.76893 (12)	0.0537 (7)
O16	0.08458 (5)	0.22301 (14)	0.81537 (12)	0.0499 (7)
O17	0.13735 (5)	0.01202 (16)	0.73112 (14)	0.0647 (8)
O18	0.12708 (6)	0.24809 (19)	0.45514 (14)	0.0762 (9)
O19	0.20265 (5)	0.29737 (15)	0.80491 (13)	0.0634 (8)
H24	0.1511 (6)	0.4609 (19)	0.5560 (17)	0.050*
H9	0.1266 (7)	-0.115 (2)	0.5597 (17)	0.050*
H39	0.2032 (7)	0.451 (2)	0.9593 (18)	0.050*
H54	0.0295 (7)	0.2236 (18)	0.7225 (16)	0.050*

Table 3. Bond lengths [Å] and angles [°] for **4k**.

C1—O2	1.219 (4)	C57—H57C	0.9800
C1—O1	1.316 (4)	C58—O15	1.457 (4)
C1—C2	1.493 (5)	C58—C60	1.520 (5)
C2—C9	1.336 (5)	C58—C59	1.530 (5)
C2—C3	1.491 (5)	C59—H59A	0.9800
C3—C8	1.365 (5)	C59—H59B	0.9800
C3—C4	1.388 (5)	C59—H59C	0.9800
C4—C5	1.383 (5)	C60—H60A	0.9800
C4—H4	0.9500	C60—H60B	0.9800
C5—C6	1.359 (6)	C60—H60C	0.9800

C5—H5	0.9500	C61—C62	1.295 (18)
C6—C7	1.373 (6)	C61—O17	1.431 (5)
C6—H6	0.9500	C61—C62'	1.678 (14)
C7—C8	1.380 (5)	C61—H61A	0.9900
C7—H7	0.9500	C61—H61B	0.9900
C8—H8	0.9500	C62—C63	1.603 (16)
C9—B1	1.588 (6)	C62—H62A	0.9900
C9—H9	0.91 (3)	C62—H62B	0.9900
C10—O3	1.457 (4)	C63—C64	1.504 (18)
C10—C12	1.508 (5)	C63—H63A	0.9900
C10—C11	1.513 (5)	C63—H63B	0.9900
C10—C13	1.533 (6)	C62'—C63'	1.578 (14)
C11—H11A	0.9800	C62'—H62C	0.9900
C11—H11B	0.9800	C62'—H62D	0.9900
C11—H11C	0.9800	C63'—C64	1.360 (14)
C12—H12A	0.9800	C63'—H63C	0.9900
C12—H12B	0.9800	C63'—H63D	0.9900
C12—H12C	0.9800	C64—O17	1.418 (5)
C13—O4	1.450 (4)	C64—H64A	0.9900
C13—C15	1.524 (5)	C64—H64B	0.9900
C13—C14	1.541 (6)	C65—C66	1.453 (8)
C14—H14A	0.9800	C65—O18	1.464 (8)
C14—H14B	0.9800	C65—H65A	0.9900
C14—H14C	0.9800	C65—H65B	0.9900
C15—H15A	0.9800	C66—C67	1.450 (8)
C15—H15B	0.9800	C66—H66A	0.9900
C15—H15C	0.9800	C66—H66B	0.9900
C16—O6	1.224 (4)	C67—C68	1.499 (9)

C16—O5	1.330 (4)	C67—H67A	0.9900
C16—C17	1.477 (5)	C67—H67B	0.9900
C17—C24	1.336 (5)	C68—O18	1.442 (8)
C17—C18	1.513 (6)	C68—H68A	0.9900
C18—C23	1.339 (6)	C68—H68B	0.9900
C18—C19	1.378 (6)	C65'—O18	1.383 (8)
C19—C20	1.424 (7)	C65'—C66'	1.492 (8)
C19—H19	0.9500	C65'—H65C	0.9900
C20—C21	1.332 (8)	C65'—H65D	0.9900
C20—H20	0.9500	C66'—C67'	1.487 (9)
C21—C22	1.299 (7)	C66'—H66C	0.9900
C21—H21	0.9500	C66'—H66D	0.9900
C22—C23	1.413 (6)	C67'—C68'	1.494 (9)
C22—H22	0.9500	C67'—H67C	0.9900
C23—H23	0.9500	C67'—H67D	0.9900
C24—B2	1.615 (5)	C68'—O18	1.444 (9)
C24—H24	0.96 (3)	C68'—H68C	0.9900
C25—O7	1.444 (4)	C68'—H68D	0.9900
C25—C26	1.510 (5)	C69—C70'	1.385 (7)
C25—C27	1.528 (5)	C69—C70	1.416 (11)
C25—C28	1.550 (5)	C69—O19	1.429 (4)
C26—H26A	0.9800	C69—H69A	0.9900
C26—H26B	0.9800	C69—H69B	0.9900
C26—H26C	0.9800	C70—C71	1.489 (12)
C27—H27A	0.9800	C70—H70A	0.9900
C27—H27B	0.9800	C70—H70B	0.9900
C27—H27C	0.9800	C71—C72	1.350 (17)
C28—O8	1.443 (4)	C71—H71A	0.9900

C28—C29	1.511 (5)	C71—H71B	0.9900
C28—C30	1.519 (5)	C70'—C71'	1.491 (9)
C29—H29A	0.9800	C70'—H70C	0.9900
C29—H29B	0.9800	C70'—H70D	0.9900
C29—H29C	0.9800	C71'—C72	1.577 (11)
C30—H30A	0.9800	C71'—H71C	0.9900
C30—H30B	0.9800	C71'—H71D	0.9900
C30—H30C	0.9800	C72—O19	1.436 (4)
C31—O10	1.229 (4)	C72—H72A	0.9900
C31—O9	1.305 (4)	C72—H72B	0.9900
C31—C32	1.496 (5)	O20—C73	1.399 (13)
C32—C39	1.319 (5)	O20—C76	1.401 (14)
C32—C33	1.469 (5)	O20—Li4	2.01 (5)
C33—C38	1.386 (5)	C73—C74	1.493 (14)
C33—C34	1.398 (5)	C73—H73A	0.9900
C34—C35	1.380 (5)	C73—H73B	0.9900
C34—H34	0.9500	C74—C75	1.336 (15)
C35—C36	1.378 (6)	C74—H74A	0.9900
C35—H35	0.9500	C74—H74B	0.9900
C36—C37	1.376 (6)	C75—C76	1.556 (14)
C36—H36	0.9500	C75—H75A	0.9900
C37—C38	1.364 (5)	C75—H75B	0.9900
C37—H37	0.9500	C76—H76A	0.9900
C38—H38	0.9500	C76—H76B	0.9900
C39—B3	1.581 (6)	O20'—C76'	1.403 (12)
C39—H39	0.79 (3)	O20'—C73'	1.406 (12)
C40—O12	1.444 (5)	O20'—Li4	1.95 (4)
C40—C43	1.523 (7)	C73'—C74'	1.484 (12)

C40—C41	1.526 (6)	C73'—H73C	0.9900
C40—C42	1.549 (6)	C73'—H73D	0.9900
C41—H41A	0.9800	C74'—C75'	1.339 (13)
C41—H41B	0.9800	C74'—H74C	0.9900
C41—H41C	0.9800	C74'—H74D	0.9900
C42—H42A	0.9800	C75'—C76'	1.486 (13)
C42—H42B	0.9800	C75'—H75C	0.9900
C42—H42C	0.9800	C75'—H75D	0.9900
C43—O11	1.438 (5)	C76'—H76C	0.9900
C43—C45	1.521 (6)	C76'—H76D	0.9900
C43—C44	1.525 (6)	Li1—O15	1.920 (6)
C44—H44A	0.9800	Li1—O17	1.979 (7)
C44—H44B	0.9800	Li1—O3	1.980 (7)
C44—H44C	0.9800	Li1—O1	2.233 (6)
C45—H45A	0.9800	Li1—O13	2.519 (7)
C45—H45B	0.9800	Li1—B1	2.669 (7)
C45—H45C	0.9800	Li1—B4	2.746 (8)
C46—O14	1.208 (4)	Li2—O14	1.898 (6)
C46—O13	1.343 (4)	Li2—O2	1.922 (7)
C46—C47	1.489 (5)	Li2—O8	1.957 (6)
C47—C54	1.353 (5)	Li2—O18	1.974 (7)
C47—C48	1.509 (5)	Li3—O19	1.936 (7)
C48—C49	1.3900	Li3—O11	1.948 (7)
C48—C53	1.338 (5)	Li3—O7	1.967 (7)
C49—C50	1.3900	Li3—O5	2.285 (6)
C49—H49	0.9500	Li3—O9	2.443 (7)
C50—C51	1.382 (6)	Li3—B2	2.663 (7)
C50—H50	0.9500	Li3—B3	2.758 (8)

C51—C52	1.3900	Li4—O6	1.930 (7)
C51—H51	0.9500	Li4—O10	1.938 (6)
C52—C53	1.3900	Li4—O16	1.957 (7)
C52—H52	0.9500	B1—O4	1.429 (5)
C53—H53	0.9500	B1—O3	1.488 (5)
C54—B4	1.616 (6)	B1—O1	1.584 (5)
C54—H54	1.00 (3)	B2—O7	1.459 (4)
C55—O16	1.453 (4)	B2—O8	1.462 (5)
C55—C57	1.510 (5)	B2—O5	1.549 (4)
C55—C56	1.525 (5)	B3—O12	1.427 (5)
C55—C58	1.554 (5)	B3—O11	1.488 (6)
C56—H56A	0.9800	B3—O9	1.559 (5)
C56—H56B	0.9800	B4—O15	1.456 (5)
C56—H56C	0.9800	B4—O16	1.462 (5)
C57—H57A	0.9800	B4—O13	1.536 (4)
C57—H57B	0.9800		

O2—C1—O1	123.3 (4)	C66—C65—O18	105.5 (8)
O2—C1—C2	125.2 (3)	C66—C65—H65A	110.6
O1—C1—C2	111.5 (3)	O18—C65—H65A	110.6
C9—C2—C3	129.4 (3)	C66—C65—H65B	110.6
C9—C2—C1	108.8 (3)	O18—C65—H65B	110.6
C3—C2—C1	121.8 (3)	H65A—C65—H65B	108.8
C8—C3—C4	118.1 (4)	C67—C66—C65	108.2 (9)
C8—C3—C2	119.8 (3)	C67—C66—H66A	110.1
C4—C3—C2	122.0 (4)	C65—C66—H66A	110.1
C5—C4—C3	120.0 (4)	C67—C66—H66B	110.1
C5—C4—H4	120.0	C65—C66—H66B	110.1

C3—C4—H4	120.0	H66A—C66—H66B	108.4
C6—C5—C4	120.9 (4)	C66—C67—C68	110.3 (10)
C6—C5—H5	119.6	C66—C67—H67A	109.6
C4—C5—H5	119.6	C68—C67—H67A	109.6
C5—C6—C7	119.8 (4)	C66—C67—H67B	109.6
C5—C6—H6	120.1	C68—C67—H67B	109.6
C7—C6—H6	120.1	H67A—C67—H67B	108.1
C6—C7—C8	119.2 (4)	O18—C68—C67	102.7 (9)
C6—C7—H7	120.4	O18—C68—H68A	111.2
C8—C7—H7	120.4	C67—C68—H68A	111.2
C3—C8—C7	122.0 (4)	O18—C68—H68B	111.2
C3—C8—H8	119.0	C67—C68—H68B	111.2
C7—C8—H8	119.0	H68A—C68—H68B	109.1
C2—C9—B1	110.8 (3)	O18—C65'—C66'	109.2 (8)
C2—C9—H9	121 (2)	O18—C65'—H65C	109.8
B1—C9—H9	128 (2)	C66'—C65'—H65C	109.8
O3—C10—C12	107.3 (3)	O18—C65'—H65D	109.8
O3—C10—C11	109.2 (3)	C66'—C65'—H65D	109.8
C12—C10—C11	111.2 (4)	H65C—C65'—H65D	108.3
O3—C10—C13	101.6 (3)	C67'—C66'—C65'	109.6 (9)
C12—C10—C13	112.8 (3)	C67'—C66'—H66C	109.8
C11—C10—C13	114.0 (4)	C65'—C66'—H66C	109.8
C10—C11—H11A	109.5	C67'—C66'—H66D	109.8
C10—C11—H11B	109.5	C65'—C66'—H66D	109.8
H11A—C11—H11B	109.5	H66C—C66'—H66D	108.2
C10—C11—H11C	109.5	C66'—C67'—C68'	100.2 (10)
H11A—C11—H11C	109.5	C66'—C67'—H67C	111.7
H11B—C11—H11C	109.5	C68'—C67'—H67C	111.7

C10—C12—H12A	109.5	C66'—C67'—H67D	111.7
C10—C12—H12B	109.5	C68'—C67'—H67D	111.7
H12A—C12—H12B	109.5	H67C—C67'—H67D	109.5
C10—C12—H12C	109.5	O18—C68'—C67'	113.6 (10)
H12A—C12—H12C	109.5	O18—C68'—H68C	108.8
H12B—C12—H12C	109.5	C67'—C68'—H68C	108.8
O4—C13—C15	108.3 (3)	O18—C68'—H68D	108.8
O4—C13—C10	102.3 (3)	C67'—C68'—H68D	108.8
C15—C13—C10	114.4 (4)	H68C—C68'—H68D	107.7
O4—C13—C14	107.7 (3)	C70'—C69—C70	47.4 (5)
C15—C13—C14	109.9 (4)	C70'—C69—O19	110.0 (5)
C10—C13—C14	113.6 (3)	C70—C69—O19	104.6 (5)
C13—C14—H14A	109.5	C70'—C69—H69A	109.7
C13—C14—H14B	109.5	C70—C69—H69A	144.4
H14A—C14—H14B	109.5	O19—C69—H69A	109.7
C13—C14—H14C	109.5	C70'—C69—H69B	109.7
H14A—C14—H14C	109.5	C70—C69—H69B	67.8
H14B—C14—H14C	109.5	O19—C69—H69B	109.7
C13—C15—H15A	109.5	H69A—C69—H69B	108.2
C13—C15—H15B	109.5	C69—C70—C71	103.2 (10)
H15A—C15—H15B	109.5	C69—C70—H70A	111.1
C13—C15—H15C	109.5	C71—C70—H70A	111.1
H15A—C15—H15C	109.5	C69—C70—H70B	111.1
H15B—C15—H15C	109.5	C71—C70—H70B	111.1
O6—C16—O5	122.0 (3)	H70A—C70—H70B	109.1
O6—C16—C17	126.0 (4)	C72—C71—C70	109.3 (11)
O5—C16—C17	112.0 (3)	C72—C71—H71A	109.8
C24—C17—C16	109.4 (3)	C70—C71—H71A	109.8

C24—C17—C18	125.3 (4)	C72—C71—H71B	109.8
C16—C17—C18	125.2 (4)	C70—C71—H71B	109.8
C23—C18—C19	117.3 (5)	H71A—C71—H71B	108.3
C23—C18—C17	122.5 (4)	C69—C70'—C71'	107.7 (6)
C19—C18—C17	119.8 (4)	C69—C70'—H70C	110.2
C18—C19—C20	119.2 (6)	C71'—C70'—H70C	110.2
C18—C19—H19	120.4	C69—C70'—H70D	110.2
C20—C19—H19	120.4	C71'—C70'—H70D	110.2
C21—C20—C19	119.7 (6)	H70C—C70'—H70D	108.5
C21—C20—H20	120.2	C70'—C71'—C72	102.7 (6)
C19—C20—H20	120.2	C70'—C71'—H71C	111.2
C22—C21—C20	122.4 (7)	C72—C71'—H71C	111.2
C22—C21—H21	118.8	C70'—C71'—H71D	111.2
C20—C21—H21	118.8	C72—C71'—H71D	111.2
C21—C22—C23	118.4 (6)	H71C—C71'—H71D	109.1
C21—C22—H22	120.8	C71—C72—O19	106.4 (7)
C23—C22—H22	120.8	C71—C72—C71'	21.9 (8)
C18—C23—C22	122.8 (6)	O19—C72—C71'	106.0 (4)
C18—C23—H23	118.6	C71—C72—H72A	127.9
C22—C23—H23	118.6	O19—C72—H72A	110.5
C17—C24—B2	108.8 (3)	C71'—C72—H72A	110.5
C17—C24—H24	124 (2)	C71—C72—H72B	90.6
B2—C24—H24	127.0 (19)	O19—C72—H72B	110.5
O7—C25—C26	109.2 (3)	C71'—C72—H72B	110.5
O7—C25—C27	107.9 (3)	H72A—C72—H72B	108.7
C26—C25—C27	109.9 (3)	C73—O20—C76	106.4 (13)
O7—C25—C28	102.3 (3)	C73—O20—Li4	116 (3)
C26—C25—C28	114.9 (3)	C76—O20—Li4	121 (4)

C27—C25—C28	112.2 (3)	O20—C73—C74	107.2 (15)
C25—C26—H26A	109.5	O20—C73—H73A	110.3
C25—C26—H26B	109.5	C74—C73—H73A	110.3
H26A—C26—H26B	109.5	O20—C73—H73B	110.3
C25—C26—H26C	109.5	C74—C73—H73B	110.3
H26A—C26—H26C	109.5	H73A—C73—H73B	108.5
H26B—C26—H26C	109.5	C75—C74—C73	107.7 (11)
C25—C27—H27A	109.5	C75—C74—H74A	110.2
C25—C27—H27B	109.5	C73—C74—H74A	110.2
H27A—C27—H27B	109.5	C75—C74—H74B	110.2
C25—C27—H27C	109.5	C73—C74—H74B	110.2
H27A—C27—H27C	109.5	H74A—C74—H74B	108.5
H27B—C27—H27C	109.5	C74—C75—C76	104.7 (10)
O8—C28—C29	108.1 (3)	C74—C75—H75A	110.8
O8—C28—C30	108.0 (3)	C76—C75—H75A	110.8
C29—C28—C30	109.5 (3)	C74—C75—H75B	110.8
O8—C28—C25	102.7 (3)	C76—C75—H75B	110.8
C29—C28—C25	113.0 (3)	H75A—C75—H75B	108.9
C30—C28—C25	115.0 (3)	O20—C76—C75	103 (2)
C28—C29—H29A	109.5	O20—C76—H76A	111.2
C28—C29—H29B	109.5	C75—C76—H76A	111.2
H29A—C29—H29B	109.5	O20—C76—H76B	111.2
C28—C29—H29C	109.5	C75—C76—H76B	111.2
H29A—C29—H29C	109.5	H76A—C76—H76B	109.1
H29B—C29—H29C	109.5	C76'—O20'—C73'	109.4 (11)
C28—C30—H30A	109.5	C76'—O20'—Li4	115 (2)
C28—C30—H30B	109.5	C73'—O20'—Li4	120 (3)
H30A—C30—H30B	109.5	O20'—C73'—C74'	104.6 (11)

C28—C30—H30C	109.5	O20'—C73'—H73C	110.8
H30A—C30—H30C	109.5	C74'—C73'—H73C	110.8
H30B—C30—H30C	109.5	O20'—C73'—H73D	110.8
O10—C31—O9	123.3 (4)	C74'—C73'—H73D	110.8
O10—C31—C32	124.6 (4)	H73C—C73'—H73D	108.9
O9—C31—C32	112.2 (4)	C75'—C74'—C73'	110.5 (10)
C39—C32—C33	130.1 (3)	C75'—C74'—H74C	109.5
C39—C32—C31	106.4 (4)	C73'—C74'—H74C	109.5
C33—C32—C31	123.4 (4)	C75'—C74'—H74D	109.5
C38—C33—C34	117.2 (4)	C73'—C74'—H74D	109.5
C38—C33—C32	121.2 (4)	H74C—C74'—H74D	108.1
C34—C33—C32	121.6 (4)	C74'—C75'—C76'	105.5 (10)
C35—C34—C33	120.7 (4)	C74'—C75'—H75C	110.6
C35—C34—H34	119.7	C76'—C75'—H75C	110.6
C33—C34—H34	119.7	C74'—C75'—H75D	110.6
C36—C35—C34	120.6 (4)	C76'—C75'—H75D	110.6
C36—C35—H35	119.7	H75C—C75'—H75D	108.8
C34—C35—H35	119.7	O20'—C76'—C75'	107.6 (10)
C37—C36—C35	119.1 (4)	O20'—C76'—H76C	110.2
C37—C36—H36	120.4	C75'—C76'—H76C	110.2
C35—C36—H36	120.4	O20'—C76'—H76D	110.2
C38—C37—C36	120.4 (4)	C75'—C76'—H76D	110.2
C38—C37—H37	119.8	H76C—C76'—H76D	108.5
C36—C37—H37	119.8	O15—Li1—O17	119.7 (3)
C37—C38—C33	122.0 (4)	O15—Li1—O3	119.7 (3)
C37—C38—H38	119.0	O17—Li1—O3	95.5 (3)
C33—C38—H38	119.0	O15—Li1—O1	136.7 (4)
C32—C39—B3	112.9 (4)	O17—Li1—O1	99.7 (3)

C32—C39—H39	124 (3)	O3—Li1—O1	69.4 (2)
B3—C39—H39	123 (3)	O15—Li1—O13	63.5 (2)
O12—C40—C43	102.3 (4)	O17—Li1—O13	113.3 (3)
O12—C40—C41	107.9 (4)	O3—Li1—O13	144.8 (3)
C43—C40—C41	116.0 (4)	O1—Li1—O13	85.6 (2)
O12—C40—C42	108.1 (4)	O15—Li1—B1	141.1 (3)
C43—C40—C42	112.9 (4)	O17—Li1—B1	95.4 (3)
C41—C40—C42	109.0 (4)	O3—Li1—B1	33.35 (15)
C40—C41—H41A	109.5	O1—Li1—B1	36.36 (14)
C40—C41—H41B	109.5	O13—Li1—B1	119.9 (3)
H41A—C41—H41B	109.5	O15—Li1—B4	30.27 (15)
C40—C41—H41C	109.5	O17—Li1—B4	125.2 (3)
H41A—C41—H41C	109.5	O3—Li1—B4	136.7 (3)
H41B—C41—H41C	109.5	O1—Li1—B4	111.9 (3)
C40—C42—H42A	109.5	O13—Li1—B4	33.59 (13)
C40—C42—H42B	109.5	B1—Li1—B4	136.0 (3)
H42A—C42—H42B	109.5	O14—Li2—O2	110.7 (3)
C40—C42—H42C	109.5	O14—Li2—O8	114.3 (3)
H42A—C42—H42C	109.5	O2—Li2—O8	118.5 (3)
H42B—C42—H42C	109.5	O14—Li2—O18	102.6 (3)
O11—C43—C45	109.9 (4)	O2—Li2—O18	102.6 (3)
O11—C43—C40	102.0 (4)	O8—Li2—O18	106.2 (3)
C45—C43—C40	114.9 (5)	O19—Li3—O11	100.7 (3)
O11—C43—C44	108.1 (4)	O19—Li3—O7	114.7 (3)
C45—C43—C44	108.8 (4)	O11—Li3—O7	120.1 (3)
C40—C43—C44	112.7 (4)	O19—Li3—O5	108.5 (3)
C43—C44—H44A	109.5	O11—Li3—O5	142.6 (3)
C43—C44—H44B	109.5	O7—Li3—O5	67.7 (2)

H44A—C44—H44B	109.5	O19—Li3—O9	97.5 (3)
C43—C44—H44C	109.5	O11—Li3—O9	64.9 (2)
H44A—C44—H44C	109.5	O7—Li3—O9	144.1 (3)
H44B—C44—H44C	109.5	O5—Li3—O9	88.1 (2)
C43—C45—H45A	109.5	O19—Li3—B2	120.0 (3)
C43—C45—H45B	109.5	O11—Li3—B2	137.3 (3)
H45A—C45—H45B	109.5	O7—Li3—B2	32.55 (14)
C43—C45—H45C	109.5	O5—Li3—B2	35.46 (13)
H45A—C45—H45C	109.5	O9—Li3—B2	117.2 (3)
H45B—C45—H45C	109.5	O19—Li3—B3	96.4 (3)
O14—C46—O13	123.0 (3)	O11—Li3—B3	31.25 (16)
O14—C46—C47	125.5 (4)	O7—Li3—B3	144.2 (3)
O13—C46—C47	111.5 (3)	O5—Li3—B3	120.4 (3)
C54—C47—C46	108.0 (4)	O9—Li3—B3	34.22 (15)
C54—C47—C48	131.1 (3)	B2—Li3—B3	139.8 (3)
C46—C47—C48	120.8 (3)	O6—Li4—O10	110.0 (3)
C49—C48—C53	117.9 (3)	O6—Li4—O16	119.9 (3)
C49—C48—C47	121.8 (3)	O10—Li4—O16	116.0 (3)
C53—C48—C47	119.5 (4)	O6—Li4—O20'	101 (2)
C50—C49—C48	120.0	O10—Li4—O20'	101.8 (12)
C50—C49—H49	120.0	O16—Li4—O20'	105.4 (11)
C48—C49—H49	120.0	O6—Li4—O20	100 (2)
C51—C50—C49	121.5 (3)	O10—Li4—O20	100.5 (14)
C51—C50—H50	119.2	O16—Li4—O20	106.8 (14)
C49—C50—H50	119.2	O20'—Li4—O20	1.6 (14)
C50—C51—C52	117.0 (3)	O4—B1—O3	106.3 (3)
C50—C51—H51	121.5	O4—B1—O1	113.0 (3)
C52—C51—H51	121.5	O3—B1—O1	103.1 (3)

C51—C52—C53	120.0	O4—B1—C9	115.8 (3)
C51—C52—H52	120.0	O3—B1—C9	119.2 (3)
C53—C52—H52	120.0	O1—B1—C9	98.6 (3)
C52—C53—C48	121.8 (3)	O4—B1—Li1	128.7 (3)
C52—C53—H53	119.1	O3—B1—Li1	47.0 (2)
C48—C53—H53	119.1	O1—B1—Li1	56.7 (2)
C47—C54—B4	110.2 (3)	C9—B1—Li1	115.4 (3)
C47—C54—H54	116.4 (18)	O7—B2—O8	105.4 (3)
B4—C54—H54	133.2 (18)	O7—B2—O5	104.6 (3)
O16—C55—C57	108.7 (3)	O8—B2—O5	113.1 (3)
O16—C55—C56	108.4 (3)	O7—B2—C24	118.4 (3)
C57—C55—C56	110.2 (3)	O8—B2—C24	115.1 (3)
O16—C55—C58	102.6 (3)	O5—B2—C24	99.9 (3)
C57—C55—C58	114.4 (3)	O7—B2—Li3	46.5 (2)
C56—C55—C58	112.0 (3)	O8—B2—Li3	128.5 (3)
C55—C56—H56A	109.5	O5—B2—Li3	58.8 (2)
C55—C56—H56B	109.5	C24—B2—Li3	116.3 (3)
H56A—C56—H56B	109.5	O12—B3—O11	105.9 (4)
C55—C56—H56C	109.5	O12—B3—O9	113.9 (4)
H56A—C56—H56C	109.5	O11—B3—O9	103.4 (3)
H56B—C56—H56C	109.5	O12—B3—C39	116.0 (4)
C55—C57—H57A	109.5	O11—B3—C39	119.4 (4)
C55—C57—H57B	109.5	O9—B3—C39	97.6 (3)
H57A—C57—H57B	109.5	O12—B3—Li3	130.3 (3)
C55—C57—H57C	109.5	O11—B3—Li3	42.8 (2)
H57A—C57—H57C	109.5	O9—B3—Li3	61.8 (2)
H57B—C57—H57C	109.5	C39—B3—Li3	113.6 (3)
O15—C58—C60	109.1 (3)	O15—B4—O16	106.1 (3)

O15—C58—C59	108.2 (3)	O15—B4—O13	106.0 (3)
C60—C58—C59	110.2 (3)	O16—B4—O13	111.9 (3)
O15—C58—C55	101.9 (3)	O15—B4—C54	119.8 (3)
C60—C58—C55	114.6 (3)	O16—B4—C54	113.5 (3)
C59—C58—C55	112.2 (3)	O13—B4—C54	99.1 (3)
C58—C59—H59A	109.5	O15—B4—Li1	41.7 (2)
C58—C59—H59B	109.5	O16—B4—Li1	128.1 (3)
H59A—C59—H59B	109.5	O13—B4—Li1	65.1 (2)
C58—C59—H59C	109.5	C54—B4—Li1	118.2 (3)
H59A—C59—H59C	109.5	C1—O1—B1	110.0 (3)
H59B—C59—H59C	109.5	C1—O1—Li1	119.3 (3)
C58—C60—H60A	109.5	B1—O1—Li1	87.0 (2)
C58—C60—H60B	109.5	C1—O2—Li2	142.6 (3)
H60A—C60—H60B	109.5	C10—O3—B1	106.4 (3)
C58—C60—H60C	109.5	C10—O3—Li1	134.2 (3)
H60A—C60—H60C	109.5	B1—O3—Li1	99.6 (3)
H60B—C60—H60C	109.5	B1—O4—C13	108.1 (3)
C62—C61—O17	108.2 (8)	C16—O5—B2	109.3 (3)
C62—C61—C62'	19.9 (13)	C16—O5—Li3	121.2 (3)
O17—C61—C62'	104.5 (6)	B2—O5—Li3	85.7 (2)
C62—C61—H61A	124.5	C16—O6—Li4	141.5 (3)
O17—C61—H61A	110.9	C25—O7—B2	107.4 (3)
C62'—C61—H61A	110.9	C25—O7—Li3	139.4 (3)
C62—C61—H61B	91.7	B2—O7—Li3	101.0 (3)
O17—C61—H61B	110.9	C28—O8—B2	109.6 (3)
C62'—C61—H61B	110.9	C28—O8—Li2	122.6 (3)
H61A—C61—H61B	108.9	B2—O8—Li2	124.1 (3)
C61—C62—C63	105.8 (14)	C31—O9—B3	110.7 (3)

C61—C62—H62A	110.6	C31—O9—Li3	117.5 (3)
C63—C62—H62A	110.6	B3—O9—Li3	84.0 (3)
C61—C62—H62B	110.6	C31—O10—Li4	137.0 (3)
C63—C62—H62B	110.6	C43—O11—B3	106.8 (3)
H62A—C62—H62B	108.7	C43—O11—Li3	132.5 (3)
C64—C63—C62	97.2 (13)	B3—O11—Li3	106.0 (3)
C64—C63—H63A	112.3	B3—O12—C40	107.9 (4)
C62—C63—H63A	112.3	C46—O13—B4	110.8 (3)
C64—C63—H63B	112.3	C46—O13—Li1	124.1 (3)
C62—C63—H63B	112.3	B4—O13—Li1	81.3 (3)
H63A—C63—H63B	109.9	C46—O14—Li2	138.3 (3)
C63'—C62'—C61	93.8 (10)	B4—O15—C58	106.8 (3)
C63'—C62'—H62C	113.0	B4—O15—Li1	108.1 (3)
C61—C62'—H62C	113.0	C58—O15—Li1	135.9 (3)
C63'—C62'—H62D	113.0	C55—O16—B4	109.0 (3)
C61—C62'—H62D	113.0	C55—O16—Li4	123.7 (3)
H62C—C62'—H62D	110.4	B4—O16—Li4	123.1 (3)
C64—C63'—C62'	103.7 (13)	C64—O17—C61	108.4 (3)
C64—C63'—H63C	111.0	C64—O17—Li1	127.9 (3)
C62'—C63'—H63C	111.0	C61—O17—Li1	123.2 (3)
C64—C63'—H63D	111.0	C65'—O18—C68	102.5 (9)
C62'—C63'—H63D	111.0	C65'—O18—C68'	105.7 (8)
H63C—C63'—H63D	109.0	C68—O18—C68'	3.5 (10)
C63'—C64—O17	107.3 (7)	C65'—O18—C65	49.7 (7)
C63'—C64—C63	33.1 (7)	C68—O18—C65	113.0 (7)
O17—C64—C63	107.3 (7)	C68'—O18—C65	116.1 (10)
C63'—C64—H64A	110.3	C65'—O18—Li2	138.4 (5)
O17—C64—H64A	110.3	C68—O18—Li2	119.0 (8)

C63—C64—H64A	79.9	C68'—O18—Li2	115.8 (6)
C63'—C64—H64B	110.3	C65—O18—Li2	109.4 (6)
O17—C64—H64B	110.3	C69—O19—C72	107.8 (3)
C63—C64—H64B	135.0	C69—O19—Li3	125.3 (3)
H64A—C64—H64B	108.5	C72—O19—Li3	125.9 (3)

References

1. Mio, M. J.; Kopel, L. C.; Braun, J. B.; Gadzikwa, T. L.; Hull, K. L.; Brisbois, R. G.; Markworth, C. J.; Grieco, P. A. *Org. Lett.* **2002**, 4, 3199.
2. Kropp, P. J.; Crawford, S. D. *J. Org. Chem.* **1994**, 59, 3102.
3. Citadelle, C. A.; Nouy, E. L.; Bisaro, F.; Slawin, A. M. Z.; Cazin, C. S. J. *Dalton Trans.* **2010**, 39, 4489.
4. Mankad, N. P.; Laitar, D. S.; Sadighi, J. P. *Organometallics* **2004**, 23, 3369.
5. Matsumoto, K.; Shindo, M. *Adv. Synth. Catal.* **2012**, 354, 642.

Copies of NMR Spectra

