Electronic Supplementary Information for

Indenyl-Functionalised Triethylborane Adduct of *N*-Heterocyclic Carbene: Stepwise Coordination of Indenyl and NHC Ligands toward Molybdenum Fragment

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Contents

S1. Experimental

S1-1. General procedures

All manipulations involving air- and moisture-sensitive organometallic compounds were carried out under an atmosphere of dry argon or nitrogen, which was purified by SICAPENT (Merck Co., Inc.), by using a standard Schlenk tube or high vacuum techniques. All solvents were distilled over appropriate drying agents prior to use. β-Bromoethylindene,[1] 1-isopropylimidazole,[2] and molybdenum complexes **3**[3] and **6**[4] were prepared according to literature methods. Another reagents employed in this research were commercially available and used without further purification.

IR spectra were recorded on a HORIBA FT-730 spectrometer. ^{1}H, ^{13}C{^{1}H} and ^{11}B{^{1}H} NMR spectra were recorded on JEOL EX-270, AL-400, and BRUKER DRX-300, DRX-500, and AVANCE III 600 spectrometers at ambient temperature, unless otherwise mentioned. ^{1}H and ^{13}C{^{1}H} NMR chemical shifts were recorded in ppm relative to internal Me_4Si. ^{11}B{^{1}H} NMR chemical shifts were recorded in ppm relative to external $BF_3{\cdot}OEt_2$. All coupling constants were recorded in Hz. Multiplicity is indicated by s (singlet), d (doublet), t (triplet), q (quartet), sept (septet), and m (multiplet). Elemental analyses were performed on a Perkin-Elmer 240C. High-resolution mass spectra (HRMS) were obtained using the electrospray ionization (ESI) method with HITACHI NanoFrontier LD.

S1-2. Preparation of 1a and 1b

1-Methylimidazole (391 mg, 4.76 mmol) and β-bromoethylindene (1036 mg, 4.64 mmol) were put in a Schlenk tube. After stirring at room temperature for ca. 48 h, the product was washed with ether (10 mL x 4) and then dried in vacuo to give compound **1a** as a white solid (863 mg, 2.83 mmol, 61%). ^{1}H NMR (in $CDCl_3$): δ 3.21 (t, *J* = 7.2 Hz, 2H, indenyl-C*H*$_2$CH$_2$-N), 3.32 (s, 2H, C*H*$_2$ in indenyl), 4.01 (s, 3H, N-C*H*$_3$), 4.67 (t, *J* = 7.2 Hz, 2H, N-C*H*$_2$CH$_2$-indenyl), 6.37 (s, 1H, C*H* in indenyl), 7.19 (t, *J* = 7.3 Hz, 1H, C*H* in indenyl), 7.26 (t, *J* = 7.3 Hz, 1H, C*H* in indenyl), 7.36 (d, *J* = 7.5 Hz, 1H, C*H* in imidazole), 7.43 (d, *J* = 7.3 Hz, 1H, C*H* in imidazole), 7.46 (broad-s, 1H, C*H* in indenyl), 7.54 (broad-s, 1H, C*H* in indenyl), 10.26 (s, 1H, N-C*H*-N). ^{13}C{^{1}H} NMR (in $CDCl_3$): δ 28.5 (indenyl-*C*H$_2$CH$_2$-N), 36.4 (N-*C*H$_3$), 37.9 (*C*H$_2$ in indenyl), 48.4 (N-*C*H$_2$-CH$_2$-indenyl), 118.5 (*C*H in imidazole), 122.3, 123.2 (*C*H in indenyl), 123.8 (*C*H in imidazole), 125.0, 126.1, 131.2 (C*H* in indenyl), 137.1 (N-*C*H-N), 138.4, 143.6, 143.9 (quaternary*C* in indenyl). HRMS (ESI) calcd for

$C_{15}H_{17}N_2$ $[M–Br]^+$: 225.1386, found: 225.1386.

Compound **1b** was prepared from 1-isopropylimidazole (304 mg, 2.76 mmol) and β-bromoethylindene (635 mg, 2.85 mmol) in the same manner as that for **1a**. Compound **1b** was isolated as orange oil (579 mg, 1.74 mmol, 63%). ^{1}H NMR (in $CDCl_3$): δ 1.53 (d, J = 6.9 Hz, 6H, CH_3 in *i*-Pr), 3.24 (t, J = 6.9 Hz, 2H, indenyl-CH_2CH$_2$-N), 3.33 (s, 2H, CH_2 in indenyl), 4.72 (t, J = 6.9 Hz, 2H, N-CH_2CH$_2$-indenyl), 4.78 (sept, J = 6.9Hz, 1H, CH in *i*-Pr), 6.41 (s, 1H, CH in indenyl), 7.19 (t, J = 7.6 Hz, 1H, CH in indenyl), 7.24 (t, J = 7.6 Hz, 1H, CH in indenyl), 7.34 (d, J = 7.6 Hz, 1H, CH in imidazole), 7.43 (d, J = 7.3 Hz, 1H, CH in imidazole), 7.46-7.48 (m, 2H, CH in indenyl), 10.43 (s, 1H, N-CH-N). ^{13}C{^{1}H} NMR (in $CDCl_3$): δ 22.9 (*C*H$_3$ in *i*-Pr), 28.5 (indenyl-*C*H$_2$CH$_2$-N), 37.9 (*C*H$_2$ in indenyl), 48.7 (N-*C*H$_2$-CH$_2$-indenyl), 53.1 (*C*H in *i*-Pr), 118.5 (*C*H in imidazole), 119.7, 122.5 (*C*H in indenyl), 123.9 (*C*H in imidazole), 125.0, 126.1, 131.4 (C*H* in indenyl), 135.8 (N-*C*H-N), 138.6, 143.8, 143.9 (quaternary*C* in indenyl). HRMS (ESI) calcd for $C_{17}H_{21}N_2$ $[M–Br]^+$: 253.1699, found: 253.1705.

S1-3. Preparation of 2a and 2b

Compound **1a** (3710 mg, 12.16 mmol) was put in a Schlenk tube, which was attached to a high-vacuum line. THF (ca. 50 mL) was added by a trap-to-trap-transfer technique at -78 °C. At this temperature, $LiBEt_3H$ (12.2 mL of its 1.0 M THF solution, 12.2 mmol) was added by syringe. Then the reaction mixture was allowed to warm to room temperature for 12 h. After removing the volatiles under reduced pressure, the residual oil was extracted with toluene (30 mL). The volatiles were removed under reduced pressure to give **2a** (3743 mg, 11.61 mmol, 95%). ^{1}H NMR (in $CDCl_3$): δ 0.50 (q, J = 7.6 Hz, 6H, BCH_2CH$_3$), 0.67 (t, J = 7.6 Hz, 9H, BCH$_2$CH_3), 3.03 (broad-t, J = 7.6 Hz, 2H, indenyl-CH_2CH$_2$-N), 3.35 (d, J = 1.5 Hz, 2H, CH_2 in indenyl), 3.85 (s, 3H, N-CH_3), 4.57 (t, J = 7.6 Hz, 2H, N-CH_2CH$_2$-indenyl), 6.26 (broad-s, 1H, CH in indenyl), 6.62 (d, J = 2.0 Hz, 1H, CH in imidazole), 6.65 (d, J = 2.0 Hz, 1H, CH in imidazole), 7.21-7.49 (m, 4H, CH in indenyl). ^{13}C{^{1}H} NMR (in $CDCl_3$): δ 11.5 (BCH$_2$*C*H$_3$), 14.4 (broad, B*C*H$_2$CH$_3$), 30.3 (indenyl-*C*H$_2$CH$_2$-N), 37.9 (N-*C*H$_3$), 38.0 (*C*H$_2$ in indenyl), 47.6 (N-*C*H$_2$-CH$_2$-indenyl), 118.6 (*C*H in indenyl), 119.8, 121.6 (*C*H in imidazole), 123.9, 124.8, 126.1, 130.2 (C*H* in indenyl), 140.1, 144.1, 144.4 (quaternary*C* in indenyl), 175.9 (broad, N-*C*-N). ^{11}B{^{1}H} NMR (in $CDCl_3$): δ -11.5. HRMS (ESI) calcd for $C_{15}H_{17}N_2$ $[M–BEt_3+H]^+$: 225.1386, found: 225.1395.

Compound **2b** was prepared from **1b** (705 mg, 2.12 mmol) and $LiBEt_3H$ (2.2 mL of its 1.0 M THF solution, 2.2 mmol) in the same manner as that for **2a**. Compound **2b** was isolated as a yellow solid (597 mg, 1.70 mmol, 80%). ^{1}H NMR (in $CDCl_3$): δ 0.51 (q, *J* = 7.3 Hz, 6H, BCH_2CH_3), 0.68 (t, *J* = 7.3 Hz, 9H, BCH_2CH_3), 1.38 (d, *J* = 6.6 Hz, 6H, CH_3 in *i*-Pr), 3.03 (dt, *J* = 1.3, 7.9 Hz, 2H, indenyl-CH_2CH_2-N), 3.36 (d, *J* = 1.3 Hz, 2H, CH_2 in indenyl), 4.56 (t, *J* = 7.9 Hz, 2H, N-CH_2CH_2-indenyl), 5.46 (sept, *J* = 6.6 Hz, 1H, C*H* in *i*-Pr), 6.26 (broad-s, 1H, C*H* in indenyl), 6.66 (d, *J* = 2.0 Hz, 1H, C*H* in imidazole), 6.85 (d, *J* = 2.0 Hz, 1H, C*H* in imidazole), 7.18-7.50 (m, 4H, C*H* in indenyl). ^{13}C{^{1}H} NMR (in $CDCl_3$): δ 11.6 (BCH_2CH_3), 14.4 (broad, BCH_2CH_3), 23.8 (CH_3 in *i*-Pr), 30.7 (indenyl-CH_2CH_2-N), 38.0 (CH_2 in indenyl), 47.7 (N-CH_2-CH_2-indenyl), 48.7 (*C*H in *i*-Pr), 115.4 (*C*H in imidazole), 118.6 (*C*H in indenyl), 120.9 (*C*H in imidazole), 123.8, 124.8, 126.1, 130.0 (C*H* in indenyl), 140.2, 144.1, 144.4 (quaternary*C* in indenyl), 174.3 (broad, N-*C*-N). ^{11}B{^{1}H} NMR (in $CDCl_3$): δ -11.3 . HRMS (ESI) calcd for $C_{17}H_{21}N_2$ $[M-BEt_3+H]^+$: 253.1699, found: 253.1710.

S1-4. Preparation of 4a

A solution of complex **3** (404 mg, 1.24 mmol) in THF (20 mL) was cooled to -78 ºC, and then a THF solution of lithiated **2a**, which was prepared by the reaction of **2a** (439 mg, 1.36 mmol) with *n*-BuLi (0.91 mL of its 1.5 M hexane solution, 1.37 mmol) at -78 ºC, was added. The reaction mixture was allowed to warm to room temperature. After several hours, the volatiles were removed under reduced pressure. The residual solid was extracted with toluene (25 mL) and then the filtrate was evaporated off under vacuum. The yellow solid was washed with pentane (10 mL x 2) and dried in vacuo to yield **4a** (569 mg, 1.08 mmol, 87%). Anal. Calcd for $C_{27}H_{37}BMoN_2O_2$: C, 61.38; H, 7.06; N, 5.30%. Found: C, 61.46; H, 6.85; N, 5.48%. IR (ν_{CO}, KBr) 1950, 1856. ^{1}H NMR (in $CDCl_3$): δ -0.77 (s, 1H, *anti*-CH_2 in metallyl), -0.73 (s, 1H, *anti*-CH_2 in metallyl), 0.50 (q, *J* = 6.6 Hz, 6H, BCH_2CH_3), 0.67 (t, *J* = 6.6 Hz, 9H, BCH_2CH_3), 1.42 (s, 3H, CH_3 in metallyl), 3.21-3.40 (m, 4H, *syn*- CH_2 in metallyl + indenyl-CH_2CH_2-N), 3.86 (s, 3H, N-CH_3), 4.43-4.67 (m, 2H, N-CH_2CH_2-indenyl), 5.52 (d, *J* = 2.6 Hz, 1H, C*H* in indenyl), 5.87 (d, *J* = 2.6 Hz, 1H, C*H* in indenyl), 6.62 (d, *J* = 2.0 Hz, 1H, C*H* in imidazole), 6.67 (d, *J* = 2.0 Hz, 1H, C*H* in imidazole), 6.93-7.15 (m, 4H, C*H* in indenyl). ^{13}C{^{1}H} NMR (in $CDCl_3$): δ 11.4 (BCH_2CH_3), 14.3 (broad, BCH_2CH_3), 23.2 (CH_3 in metallyl), 31.2 (indenyl-CH_2CH_2-N), 37.8 (N-CH_3), 50.2 (N-CH_2CH_2-indenyl), 57.3 (CH_2 in metallyl), 57.5 (CH_2 in metallyl), 75.8, 89.4 (*C*H in indenyl), 94.8 (CCH_3 in metallyl),

108.3 (quaternary*C* in indenyl), 112.3 (quaternary*C* in indenyl, overlapped), 120.1, 121.9, 122.6, 124.5, 124.7, 125.1 (C*H* in indenyl + *C*H in imidazole), 176.6 (broad, N-*C*-N), 240.3, 241.3 (*C*O). $^{11}B\{^1H\}$ NMR (in $CDCl_3$): δ -10.9.

S1-5. Preparation of 5a

Complex **4a** (61 mg, 0.12 mmol) and pyridine (5 mL) were put in a Schlenk tube. After being refluxed for 4 h, the volatiles were removed under reduced pressure. The residual solid was dissolved in CH_2Cl_2 (ca 3 mL). This solution was loaded on an Al_2O_3 column (ϕ 10 x 40 mm) and eluted with CH_2Cl_2/hexane (1/1). The red band was collected and dried in vacuo to give **5a** as a red solid (39 mg, 0.097 mmol, 81%). Anal. Calcd for $C_{20}H_{22}MoN_2O$: C, 59.70; H, 5.51; N, 6.96%. Found: C, 59.39; H, 5.53; N, 6.79%. IR (ν_{CO}, KBr) 1794, 1773. ^1H NMR (in $CDCl_3$): δ -1.24 (s, 1H, *anti*-CH_2 in metallyl), -0.17 (s, 1H, *anti*-CH_2 in metallyl), 1.63 (s, 3H, C*H*$_3$ in metallyl), 1.90 (d, *J* = 3.6 Hz, 1H, *syn*-CH_2 in metallyl), 2.22 (ddd, *J* = 14.2, 12.2, 3.6 Hz, 1H, indenyl-C*H*$_2$CH$_2$-N), 2.85 (d, *J* = 3.6 Hz, 1H, *syn*-CH_2 in metallyl), 3.32 (ddd, *J* = 14.2, 3.3, 2.3 Hz, 1H, indenyl-C*H*$_2$CH$_2$-N), 3.50 (s, 3H, N-C*H*$_3$), 4.15-4.34 (m, 2H, N-C*H*$_2$CH$_2$-indenyl), 5.21 (d, *J* = 2.6 Hz, 1H, C*H* in indenyl), 5.44 (d, *J* = 2.6 Hz, 1H, C*H* in indenyl), 6.82 (d, *J* = 2.0 Hz, 1H, C*H* in imidazole), 7.05 (d, *J* = 2.0 Hz, 1H, C*H* in imidazole), 6.84-7.12 (m, 4H, C*H* in indenyl). $^{13}C\{^1H\}$ NMR (in $CDCl_3$): δ 24.7 (*C*H$_3$ in metallyl), 27.4 (indenyl-*C*H$_2$CH$_2$-N), 39.6 (N-*C*H$_3$), 52.4 (N-*C*H$_2$CH$_2$-indenyl), 53.8 (*C*H$_2$ in metallyl), 59.8 (*C*H$_2$ in metallyl), 73.8, 89.6 (*C*H in indenyl), 93.6 (quaternary*C* in metallyl), 103.9, 108.3, 110.8 (quaternary*C* in indenyl), 121.2, 121.8, 122.2, 122.5, 123.2, 127.1 (*C*H in indenyl + *C*H in imidazole), 201.0 (N-*C*-N), 260.6 (CO).

S1-6. Preparation of 7a

Complexes **4a** (91 mg, 0.17 mmol) and **6** (80 mg, 0.17 mmol) ware put in a Schlenk tube, which was attached to a high-vacuum line. Heptane (ca. 10 mL) was added by a trap-to-trap-transfer technique at -78 °C. After being refluxed for 2.5 h, a yellow solid was precipitated. The resulting yellow precipitates were isolated by filtration, washed with hexane, and dried in vacuo to give **7a** (102 mg). The yellow filtrate was cooled to form further yellow precipitates, which was collected and dried in vacuo to give **7a** (12 mg). Complex **6a** was totally obtained in 82% yield (114 mg, 0.14 mmol). Anal. Calcd for $C_{39}H_{38}Mo_2N_4O_4$: C, 57.22; H, 4.68; N, 6.84%. Found. C, 57.33; H, 4.68; N, 6.85%. IR (ν_{CO}, KBr) 1949, 1914,

1874, 1822. ^{1}H NMR (in $CDCl_3$): δ -0.84 (s, 1H, *anti*-CH_2 in metallyl), -0.73 (s, 1H, *anti*-CH_2 in metallyl), 1.39 (s, 3H, C*H*$_3$ in metallyl), 1.66 (d, *J* = 9.6 Hz, 1H, *anti*-CH_2 in allyl), 1.77 (d, *J* = 9.6 Hz, 1H, *anti*-CH_2 in allyl), 3.11-3.22 (m, 1H, indenyl-C*H*$_2$CH$_2$-N), 3.32 (d, *J* = 3.3 Hz, 1H, *syn*-CH_2 in metallyl), 3.37 (d, *J* = 3.3 Hz, 1H, *syn*-CH_2 in metallyl), 3.31-3.44 (m, 1H, indenyl-C*H*$_2$CH$_2$-N), 3.44 (s, 3H, N-C*H*$_3$), 3.65-3.97 (m, 2H, N-C*H*$_2$CH$_2$-indenyl), 4.00-4.25 (m, 3H, *syn*-C*H*$_2$ in allyl + C*H* in allyl), 5.37 (broad-s, C*H* in indenyl), 5.82 (d, *J* = 3.0 Hz, 1H, C*H* in indenyl), 6.71–7.10 (m, 16H, Ph + C*H* imidazole + C*H* in indenyl), 8.37 (s, 1H, C*H* in amidinato). ^{13}C{^{1}H} NMR (in $CDCl_3$): δ 23.2 (*C*H$_3$ in metallyl), 30.0 (indenyl-*C*H$_2$CH$_2$-N), 38.9 (N-*C*H$_3$), 51.8 (N-*C*H$_2$CH$_2$-indenyl), 57.0 (*C*H$_2$ in metallyl), 57.4 (*C*H$_2$ in metallyl), 59.1, 61.2 (*C*H$_2$ in allyl), 75.4 (*C*H in indenyl), 85.2 (*C*H in allyl), 89.0 (*C*H in indenyl-5ring), 94.9 (quaternary*C* in metallyl), 108.1, 112.2, 112.2 (quaternary*C* in indenyl), 118.4, 118.8, 120.4, 121.2, 121.5, 122.9, 122.9, 124.3, 124.4, 124.8, 129.0, 129.1, 147.6, 147.9 (Ph + *C*H in indenyl + *C*H in imidazole), 154.7 (amidinato-*C*H), 193.0 (N-*C*-N), 229.4, 229.7, 240.4, 241.4 (*C*O).

S2. Single-crystal X-ray crystallography

S2-1. Experimental procedure for X-ray analyses

Suitable single crystals were obtained by recrystallization from hexane (**4a**) or from toluene/hexane (**5a** and **7a**) and are individually mounted on glass fibers. Indexing was performed from 3 oscillations, which were exposed for 30 seconds (**4a**), 45 seconds (**5a**), and 90 seconds (**7a**), respectively, using a Rigaku RAXIS-RAPID Imaging Plate diffractometer with graphite-monochromated Cu-*K*α radiation (λ = 1.54187 Å). The crystal-to-detector distance was 127.40 mm. The data were collected at a temperature of 23 ± 1 °C to a maximum 2θ value of 136.5 º. The collected oscillation images were 24 for **4a**, 24 for **5a**, and 36 for **7a**. Readout was performed in the 0.100 mm pixel mode.

Crystallographic data and the results of measurement are summarized in Table S1. The structures were solved by direct methods (SIR92)[5] for all complexes, and expanded using Fourier techniques (DIRDIF99).[6] Least-square refinements were carried out using SHELXL97.[7] All of the non-hydrogen atoms were refined anisotropically. Hydrogen atoms were introduced at the ideal positions and refined using the riding model. All calculations were performed using the CrystalStructure crystallographic software package.[8]

Table S1. Summery of Crystal Data for Complexes **4a**, **5a**, and **7a**

		4a	**5a**	**7a**
Empirical Formula		$C_{27}H_{37}BMoN_2O_2$	$C_{20}H_{22}MoN_2O$	$C_{39}H_{38}Mo_2N_4O_4$
Formula weight		528.35	402.35	818.63
Crystal color, habit		yellow, needle	red, plate	yellow, needle
Crystal size/mm		0.50 x 0.25 x 0.18	0.25 x 0.18 x 0.08	0.20 x 0.10 x 0.08
Crystal system		monoclinic	monoclinic	monoclinic
Space group		$P2_1/c$ (#14)	$P2_1/c$ (#14)	$P2_1/a$ (#14)
Lattice parameters	a/Å	6.8287(15)	8.7708(11)	7.411(2)
	b/Å	26.711(5)	16.5056(17)	38.544(8)
	c/Å	14.680(3)	12.1411(13)	13.169(3)
	β/º	97.025(5)	91.388(10)	76.26(2)
	V/Å^3	2657.6(9)	1757.1(3)	3654.0(15)
Z		4	4	4
D_c/g cm^{-3}		1.320	1.521	1.488
F_{000}		1104.00	824.00	1664.00
μ(Cu-$K\alpha$)/cm^{-1}		42.118	61.458	59.673
Reflection measured		22903	13487	29621
Independent reflections (R_{int})		4646 (0.055)	3066 (0.073)	6121 (0.111)
No. variables		299	218	443
Reflection/parameter ratio		15.54	14.06	13.82
Residuals: R; Rw		0.0538; 0.1130	0.0419; 0.0832	0.1616; 0.1044
Residuals: $R1$ ($I > 2.0\sigma(I)$)		0.0393	0.0334	0.0594
Goodness of fit indicator		1.076	1.050	0.870
$\delta\rho_{max, min}$/e Å^{-3}		0.59, -0.46	0.44, -0.65	0.74, -0.45

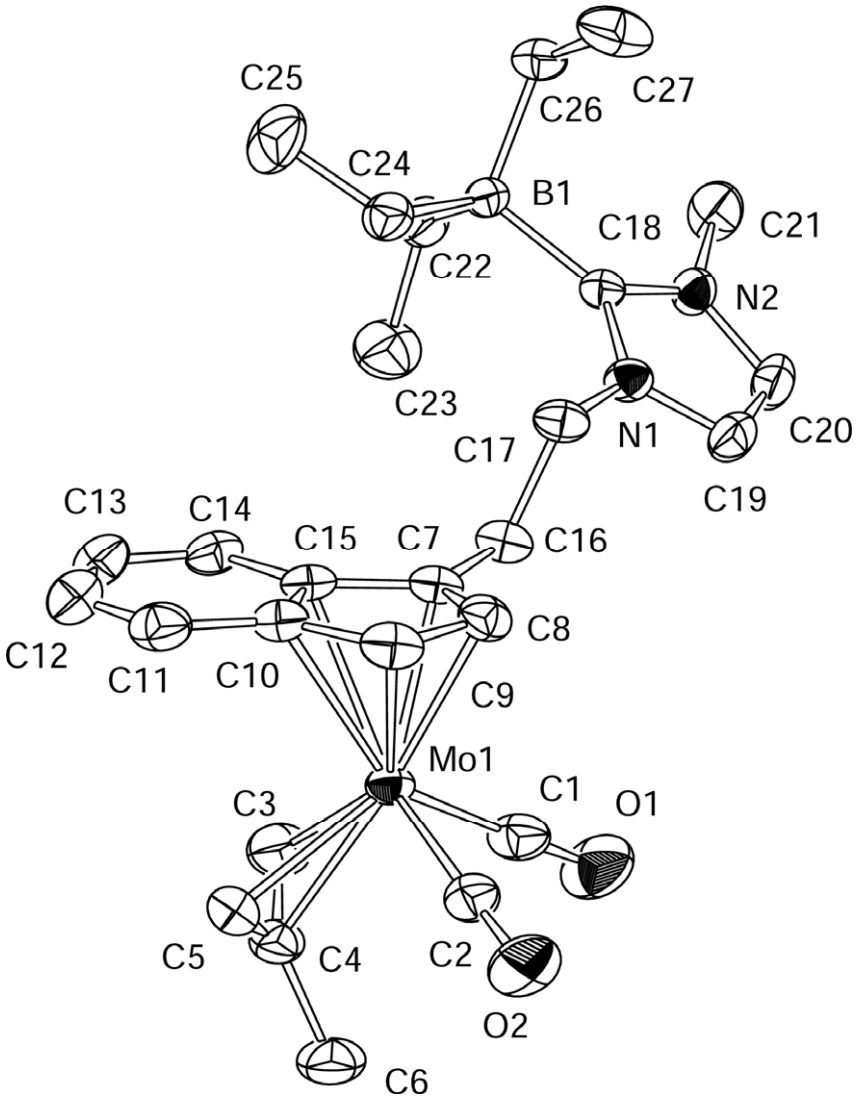

Figure S1. ORTEP drawing of **4a** (30% probability of thermal ellipsoids) showing the numbering system. All hydrogen atoms are omitted for clarity.

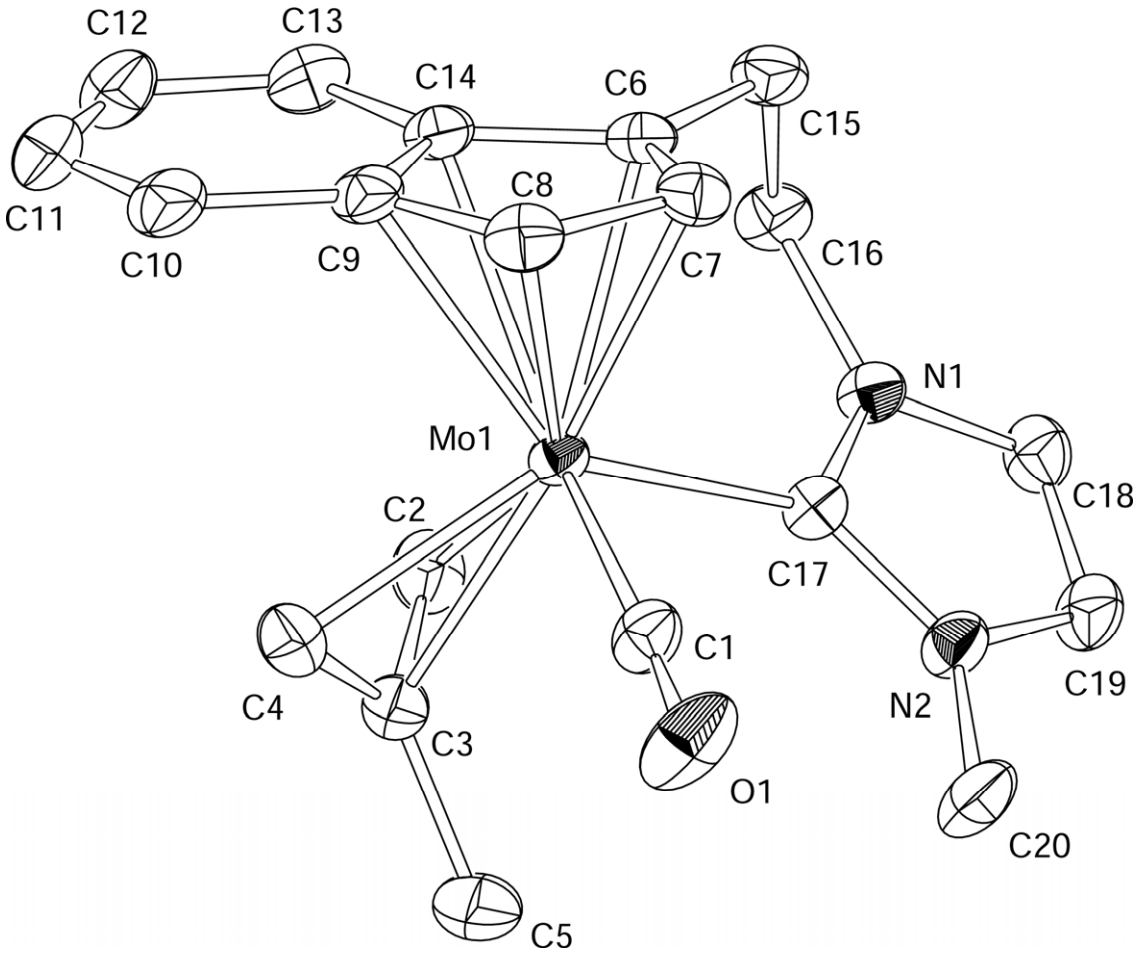

Figure S2. ORTEP drawing of **5a** (30% probability of thermal ellipsoids) showing the numbering system. All hydrogen atoms are omitted for clarity.

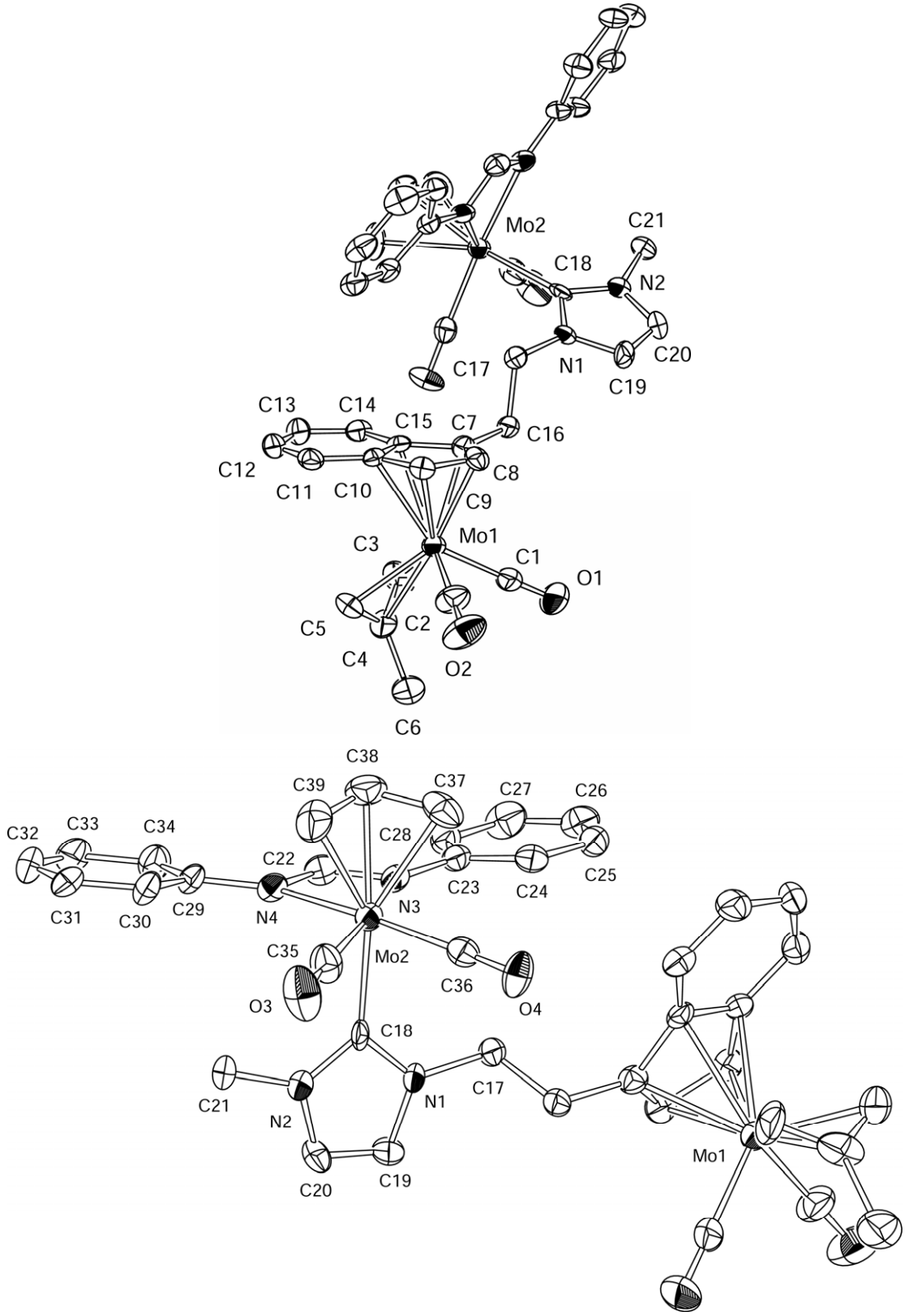

Figure S3. ORTEP drawings of **7a** (30% probability of thermal ellipsoids) showing the numbering system. All hydrogen atoms are omitted for clarity.

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