**AN ENANTIOMERICALLY PURE ADAMANTYLIMIDO MOLYBDENUM
ALKYLIDENE COMPLEX. AN EFFECTIVE NEW CATALYST FOR
ENANTIOSELECTIVE OLEFIN METHATHESIS**

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Supporting Information

Infrared (IR) spectra were recorded on a Nicolet 210 spectrophotometer, $\nu_{\max}$ in cm^{-1} . Bands were characterized as broad (br), strong (s), medium (m), and weak (w). ^1H NMR spectra were recorded on a Varian INOVA 400 (400 MHz) spectrometer. Chemical shifts are reported in ppm from tetramethylsilane with the solvent resonance as the internal standard (CDCl_3 : δ 7.26). Data are reported as follows: chemical shift, multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, br = broad, m = multiplet, psd = pseudo), coupling constants (Hz), and integration. ^{13}C NMR spectra were recorded on a Varian INOVA 400 (100 MHz) spectrometer with complete proton decoupling. Chemical shifts are reported in ppm from tetramethylsilane with the solvent as the internal reference (C_6D_6 : δ 128.1, CDCl_3 : δ 77.2). Enantiomer ratios were determined by chiral HPLC (Chiral Technologies chiralpak AS (250 mm x 4.6 mm i.d.)) or chiral GLC (Alltech Associates CDGTA (30m x 0.25 mm i.d.)) in comparison with authentic racemic materials. Representative HPLC and GLC traces are included at the end of this section. Microanalyses were performed by Robertson Microlit Laboratories (Madison, NJ). High-resolution mass spectrometry was performed at the University of Illinois Mass Spectrometry Laboratories (Urbana-Champaign, IL).

Unless otherwise noted, all reactions were conducted in oven- (135 °C) or flame-dried glassware under an inert atmosphere of dry N_2 . All substrates for metathesis reactions were vigorously dried by repeated (three times) azeotropic distillation of water using anhydrous benzene under vacuum. Handling of all Mo catalysts was performed in a dry box. Benzene, toluene, and pentane were sparged with N_2 and then passed through an activated alumina column and an activated Cu column. Tetrahydrofuran, CH_2Cl_2 and Et_2O were sparged with N_2 and then passed through an activated alumina column. All reagents were purchased from Aldrich Chemical Company, Lancaster Synthesis, or Strem Chemicals, Inc. and were purified by appropriate methods prior to use. Mo complexes **1**¹, **2**, and **3**^{2,3} were prepared according to published procedures.

¹ Alexander, J. B.; La, D. S.; Cefalo, D. R.; Graf, D. D.; Hoveyda, A. H.; Schrock, R. R. *J. Am. Chem. Soc.* **1998**, *120*, 4041–4042.

² Aeilts, S. L.; Cefalo, D. R.; Bonitatebus, P. J., Jr.; Houser, J. H.; Hoveyda, A. H.; Schrock, R. R. *Angew. Chem., Int. Ed. Engl.* **2001**, *40*, 1452–1456.

Representative procedure for Mo-catalyzed AROM/CM. In a nitrogen filled glovebox, 40.0 mg **7a** (0.200 mmol) and 33.6 mg **8⁴** (0.200 mmol) was weighed into a 4 mL vial containing a stir bar and then diluted with 1.00 mL benzene. Catalyst **4** (7.20 mg, 0.0100 mmol, 0.05 equiv) was added and the vial was fitted with a teflon lined cap. The reaction was stirred at 22 °C for 6 h. The reaction was removed from the glovebox, and the volatiles were removed to give a viscous black oil. The crude product was purified by silica gel chromatography (50:1 pentane:Et₂O) affording **9a** as a colorless oil (53.0 mg, 78%).

2-[3-(2-Benzyloxy-3-vinyl-cyclopentyl)-allyl]-4,4,5,5-tetramethyl-[1,3,2]dioxaborolane (9a). IR (Neat): 3074 (w), 2974 (m), 2930 (m), 2867 (m), 1463 (m), 1319 (s), 1142 (s), 1097 (m); ¹H NMR (400 MHz, CDCl₃): δ 7.36-7.20 (m, 1H), 6.02-5.94 (m, 1H), 5.59-5.48 (m, 2H), 5.06-4.95 (m, 2H), 4.58-4.48 (ABq, *J* = 11.9, 2H), 3.71-3.69 (t, *J* = 4.0, 1H), 2.60-2.50 (br m, 2H), 1.80-1.67 (m, 4H), 1.66-1.64 (d, *J* = 6.2, 2H), 1.21 (s, 12H); ¹³C NMR (100 MHz, CDCl₃): δ 140.0, 131.7, 128.3, 127.9, 127.7, 127.4, 125.4, 114.5, 87.5, 83.3, 77.4, 73.6, 50.0, 49.2, 29.4, 29.0, 25.0; HRMS of **9a** calcd. for derived alcohol C₁₇H₂₂O₂ 258.16198. Found 258.16174

Representative procedure for oxidation of allyl boronate (9a) to allylic alcohol (12). With no precautions taken to exclude air or moisture, a round bottom flask was charged with 53.0 mg **9a** (0.156 mmol), 500 μL 1:1 THF / EtOH, 250 μL 1.0M pH 7.00 (KH₂PO₄/ NaOH buffer), and 250 μL 30% aqueous H₂O₂ solution. The solution was stirred 5 mins at 22 °C. The reaction was quenched by dilution with 3.0 mL EtOAc, followed by addition of 1.0 mL saturated aqueous NaHCO₃ solution. The layers were separated, and the aqueous layer was washed with three 1.0 mL portions EtOAc. Combined EtOAc washes were dried with anhydrous Na₂SO₄ and concentrated. Purification by silica gel chromatography (10:1 hexanes:Et₂O) give **12** as a colorless oil (37.2 mg, 92%). IR (neat) 3370 (br m), 2955 (m), 2860 (m), 1262 (m), 1098 (s), 1035 (m), 916 (m), 727 (s); ¹H NMR (400 MHz, CDCl₃): δ 7.34-7.25 (m, 5H) 6.04-5.95 (ddd, *J* = 17.4, 10.3, 8.4 Hz, 1H), 5.81-5.70 (ddt, *J* = 15.4, 8.2, 1.3 Hz, 1H), 5.65-5.58 (dt, *J* = 15.4, 6.0 Hz, 1H), 5.09-5.02 (ddd, *J* = 17.2, 2.0, 0.9 Hz, 1H), 5.02-4.99 (dd, *J* = 10.3, 2.0 Hz, 1H), 4.59-4.55 (AB, *J* = 12.1 Hz, 1H), 4.49-4.43 (AB, *J* = 12.1 Hz, 1H), 4.05-4.02 (dd, *J* = 6.0, 6.0 Hz, 2H), 3.76-3.74 (t, *J* = 4.0 Hz, 1H), 2.64-2.53 (m, 2H), 1.83-1.74 (m, 4H); ¹³C NMR (100MHz, CDCl₃): δ 139.5, 133.9, 129.4, 128.4, 128.4, 128.0, 115.0, 87.2, 73.8, 64.1, 50.2, 48.5, 29.1, 28.9; Elem. Calcd. for **12** C₁₇H₂₂O₂ 258.16198. Found 258.16174

2-[3-(2-Methoxy-3-vinyl-cyclopentyl)-allyl]-4,4,5,5-tetramethyl-[1,3,2]dioxaborolane (9b). IR (Neat): 3074 (w), 2980 (m), 2930 (m), 2823 (m), 1646 (w), 1469 (m), 1331 (s), 1155 (m), 1111 (m), 972 (m); ¹H NMR (400 MHz, CDCl₃): δ 5.98-5.89 (ddd, *J* = 10.3, 8.1, 4.6 Hz, 1H), 5.55-5.49 (m, 2H), 5.06-4.96 (m, 2H), 3.43-3.41 (t, *J* = 5.0 Hz, 1H), 3.34 (s, 3H), 2.52 (br m, 2H), 1.75-1.63 (m, 2H), 1.75-1.63 (m, 6H), 1.22 (s, 12H); ¹³C NMR (100 MHz, CDCl₃): δ 139.7, 131.2, 125.4, 114.5, 89.7, 83.3, 60.1, 49.6, 48.8, 29.7, 29.2, 28.7, 24.9; Elem. Calcd. for **9b** C₁₇H₂₉BO₃: C, 69.87; H, 10.00. Found C, 69.60; H, 10.05.

³ Shrock, R. R.; Jamieson, J. Y.; Dolman, S. J.; Miller, S. A.; Bonitatebus, P. J., Jr.; Hoveyda, A. H., *Organometallics* **2002**, 21(2), 409-417.

⁴ Roush, W. R., Walts, A. E., Hoong, L. K., *J. Am. Chem. Soc.* **1985**, 107, 8186-8190

2-{3-[2-(*tert*-Butyl-dimethyl-silanyloxy)-3-vinyl-cyclopentyl]-allyl}-4,4,5,5-tetramethyl-[1,3,2]dioxaborolane (9c**). IR (Neat): 3068 (w), 2974 (m), 2955 (m), 2854 (m), 1646 (w), 1463 (m), 1369 (s), 1325 (s), 1255 (m), 1154 (s), 1104 (s), 979 (m); ¹H NMR (400 MHz, CDCl₃): δ 5.92-5.81 (ddd, *J* = 15.2, 10.0, 1.6 Hz, 1H), 5.44-5.41 (m, 2H), 5.02-4.97 (ddd, *J* = 15.2, 3.2, 1.6 Hz, 1H), 4.95-4.92 (dd, *J* = 10.0, 1.6 Hz, 1H), 3.91-3.89 (t, *J* = 3.6 Hz, 3H), 2.43 (br m, 2H), 1.73-1.69 (m, 4H), 1.66-1.64 (br d, 5.2 Hz, 2H), 1.23 (s, 12H), 0.89 (s, 9H), -0.02--0.03 (d, *J* = 2.0 Hz, 6H); ¹³C NMR (100 MHz, CDCl₃): δ 140.7, 134.2, 132.5, 125.0, 115.0, 114.2, 83.4, 83.2, 80.6, 51.3, 50.3, 28.9, 28.6, 26.2, 24.9, -3.8, -3.9; HRMS Calcd. for **9c** C₂₂H₄₀BO₃Si 391.28398. Found 391.28347**

Representative Chromatograms for Enantiomeric Excess Determination:

Chiralpak AS column, 1.0 mL min⁻¹, 190 nM, 97.5:2.5 Hexanes/Isopropanol

GLC CD-BPH column, 110 °C, 35 psi, 1.0 mL min⁻¹

GLC CD-BPH column, 140 °C, 15 psi, 1.0 mL min⁻¹

GLC CDGTA column, 105 °C, 15 psi, 1.0 mL min⁻¹

For Mo(NAd)(CHCMe₂Ph)[(S)-Biphen] (4)

- i. Fully labeled ORTEP drawing
- ii. Crystal data and structure refinement
 - iii. Atomic coordinates
 - iv. Bond lengths and angles
- v. Anisotropic thermal parameters
 - vi. Hydrogen coordinates

Table 1. Crystal data and structure refinement for 4.

Empirical formula	MoC ₄₄ H ₅₉ NO ₂	
Formula weight	729.86	
Temperature	183(2) K	
Wavelength	0.71073 Å	
Crystal system	Orthorhombic	
Space group	P2 ₁ 2 ₁ 2 ₁	
Unit cell dimensions	a = 10.9897(8) Å	α = 90°.
	b = 16.5354(13) Å	β = 90°.
	c = 21.4911(16) Å	γ = 90°.
Volume, Z	3905.3(5) Å ³ , 4	
Density (calculated)	1.241 Mg/m ³	
Absorption coefficient	0.372 mm ⁻¹	
F(000)	1552	
Crystal size	0.3 x 0.1 x 0.22 mm ³	
Theta range for data collection	2.26 to 20.00°.	
Index ranges	-9 ≤ h ≤ 10, -11 ≤ k ≤ 15, -20 ≤ l ≤ 20	
Reflections collected	11405	
Independent reflections	3645 [R _{int} = 0.0546]	
Completeness to theta = 20.00°	99.8 %	
Absorption correction	None	
Refinement method	Full-matrix least-squares on F ²	
Data / restraints / parameters	3645 / 0 / 446	
Goodness-of-fit on F ²	1.070	
Final R indices [I > 2σ(I)]	R ₁ = 0.0268, wR ₂ = 0.0626	
R indices (all data)	R ₁ = 0.0289, wR ₂ = 0.0633	
Absolute structure parameter	-0.07(3)	
Extinction coefficient	0.00000(15)	
Largest diff. peak and hole	0.230 and -0.164 e.Å ⁻³	

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

	x	y	z	U(eq)
Mo(1)	4532(1)	5166(1)	1961(1)	30(1)
N(1)	5449(3)	5639(2)	2490(1)	33(1)
O(1)	3860(2)	6024(2)	1413(1)	34(1)
O(2)	5282(2)	4147(2)	1642(1)	34(1)
C(1)	3243(3)	4743(3)	2424(2)	35(1)
C(2)	2652(3)	4590(2)	3051(2)	38(1)
C(3)	2882(5)	5312(3)	3473(2)	67(2)
C(4)	1262(4)	4489(3)	2957(2)	62(1)
C(5)	3198(4)	3846(3)	3357(2)	35(1)
C(6)	4390(5)	3629(3)	3279(2)	70(2)
C(7)	4905(5)	2968(4)	3559(3)	88(2)
C(8)	4248(6)	2504(3)	3951(2)	70(2)
C(9)	3053(6)	2681(4)	4039(2)	68(2)
C(10)	2533(5)	3347(3)	3751(2)	60(2)
C(11)	6283(4)	6104(2)	2875(2)	33(1)
C(12)	7315(4)	6411(3)	2458(2)	43(1)
C(13)	8214(4)	6912(3)	2852(2)	54(1)
C(14)	8741(4)	6383(3)	3361(2)	53(1)
C(15)	7722(4)	6079(3)	3776(2)	47(1)
C(16)	7065(5)	6794(3)	4064(2)	56(1)
C(17)	6530(4)	7321(3)	3547(2)	50(1)
C(18)	5633(4)	6822(2)	3163(2)	41(1)
C(19)	6819(4)	5579(3)	3396(2)	40(1)
C(20)	7546(4)	7628(3)	3134(2)	51(1)
C(21)	3242(4)	5547(3)	1009(2)	30(1)
C(22)	1984(4)	5667(3)	903(2)	34(1)
C(23)	1442(4)	5090(3)	539(2)	38(1)
C(24)	2022(4)	4426(3)	268(2)	33(1)
C(25)	3283(4)	4363(3)	339(2)	33(1)
C(26)	3891(3)	4927(3)	705(2)	28(1)
C(27)	5906(3)	4459(2)	1151(2)	27(1)
C(28)	5273(3)	4924(3)	717(2)	28(1)
C(29)	5895(3)	5341(2)	245(2)	30(1)
C(30)	7159(4)	5248(3)	202(2)	32(1)
C(31)	7737(4)	4725(3)	606(2)	33(1)
C(32)	7180(4)	4310(2)	1089(2)	32(1)
C(33)	1319(4)	6411(3)	1164(2)	45(1)
C(34)	1927(5)	7170(3)	920(3)	66(2)
C(35)	1323(5)	6416(3)	1868(2)	70(2)

C(36)	-14(4)	6439(3)	953(3)	80(2)
C(37)	7840(4)	3710(3)	1500(2)	37(1)
C(38)	9167(4)	3599(3)	1313(2)	63(2)
C(39)	7825(5)	3973(3)	2177(2)	60(2)
C(40)	7229(4)	2878(3)	1438(2)	59(1)
C(41)	1295(4)	3808(3)	-87(2)	45(1)
C(42)	3975(4)	3703(3)	11(2)	43(1)
C(43)	5208(4)	5851(3)	-220(2)	43(1)
C(44)	7877(4)	5715(3)	-274(2)	41(1)

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

Mo(1)-N(1)	1.709(3)
Mo(1)-C(1)	1.867(4)
Mo(1)-O(1)	1.986(3)
Mo(1)-O(2)	1.997(2)
N(1)-C(11)	1.453(5)
O(1)-C(21)	1.356(4)
O(2)-C(27)	1.360(4)
C(1)-C(2)	1.518(6)
C(2)-C(5)	1.519(6)
C(2)-C(3)	1.521(6)
C(2)-C(4)	1.549(6)
C(5)-C(6)	1.369(6)
C(5)-C(10)	1.389(6)
C(6)-C(7)	1.370(7)
C(7)-C(8)	1.350(7)
C(8)-C(9)	1.358(7)
C(9)-C(10)	1.387(7)
C(11)-C(18)	1.518(5)
C(11)-C(12)	1.532(5)
C(11)-C(19)	1.536(5)
C(12)-C(13)	1.543(6)
C(13)-C(14)	1.515(6)
C(13)-C(20)	1.520(6)
C(14)-C(15)	1.518(6)
C(15)-C(16)	1.518(6)
C(15)-C(19)	1.527(6)
C(16)-C(17)	1.529(6)
C(17)-C(20)	1.515(6)
C(17)-C(18)	1.528(6)
C(21)-C(26)	1.410(5)
C(21)-C(22)	1.415(6)
C(22)-C(23)	1.370(6)
C(22)-C(33)	1.537(6)
C(23)-C(24)	1.396(6)
C(24)-C(25)	1.398(6)
C(24)-C(41)	1.505(6)
C(25)-C(26)	1.390(6)
C(25)-C(42)	1.506(6)
C(26)-C(28)	1.518(5)
C(27)-C(28)	1.396(5)
C(27)-C(32)	1.427(5)
C(28)-C(29)	1.403(5)
C(29)-C(30)	1.400(5)
C(29)-C(43)	1.510(5)

C(30)-C(31)	1.380(5)
C(30)-C(44)	1.506(6)
C(31)-C(32)	1.388(6)
C(32)-C(37)	1.514(6)
C(33)-C(35)	1.512(6)
C(33)-C(34)	1.515(6)
C(33)-C(36)	1.535(6)
C(37)-C(39)	1.519(6)
C(37)-C(38)	1.523(6)
C(37)-C(40)	1.537(6)
N(1)-Mo(1)-C(1)	105.37(17)
N(1)-Mo(1)-O(1)	106.66(13)
C(1)-Mo(1)-O(1)	107.54(14)
N(1)-Mo(1)-O(2)	111.80(13)
C(1)-Mo(1)-O(2)	100.34(15)
O(1)-Mo(1)-O(2)	123.54(11)
C(11)-N(1)-Mo(1)	172.8(3)
C(21)-O(1)-Mo(1)	98.7(2)
C(27)-O(2)-Mo(1)	98.9(2)
C(2)-C(1)-Mo(1)	149.3(3)
C(1)-C(2)-C(5)	110.5(3)
C(1)-C(2)-C(3)	109.2(3)
C(5)-C(2)-C(3)	108.1(4)
C(1)-C(2)-C(4)	108.9(4)
C(5)-C(2)-C(4)	111.0(3)
C(3)-C(2)-C(4)	109.1(4)
C(6)-C(5)-C(10)	115.0(4)
C(6)-C(5)-C(2)	122.5(4)
C(10)-C(5)-C(2)	122.5(4)
C(5)-C(6)-C(7)	123.4(5)
C(8)-C(7)-C(6)	120.4(5)
C(7)-C(8)-C(9)	118.8(5)
C(8)-C(9)-C(10)	120.5(5)
C(9)-C(10)-C(5)	121.8(5)
N(1)-C(11)-C(18)	110.4(3)
N(1)-C(11)-C(12)	108.1(3)
C(18)-C(11)-C(12)	109.2(3)
N(1)-C(11)-C(19)	111.0(3)
C(18)-C(11)-C(19)	108.9(3)
C(12)-C(11)-C(19)	109.3(3)
C(11)-C(12)-C(13)	109.4(4)
C(14)-C(13)-C(20)	110.4(4)
C(14)-C(13)-C(12)	109.3(4)
C(20)-C(13)-C(12)	109.1(4)
C(13)-C(14)-C(15)	109.5(4)

C(14)-C(15)-C(16)	109.4(4)
C(14)-C(15)-C(19)	110.1(4)
C(16)-C(15)-C(19)	109.3(4)
C(15)-C(16)-C(17)	109.3(4)
C(20)-C(17)-C(18)	109.9(4)
C(20)-C(17)-C(16)	109.5(4)
C(18)-C(17)-C(16)	109.5(4)
C(11)-C(18)-C(17)	109.8(3)
C(15)-C(19)-C(11)	109.5(4)
C(17)-C(20)-C(13)	109.1(4)
O(1)-C(21)-C(26)	117.8(3)
O(1)-C(21)-C(22)	120.7(4)
C(26)-C(21)-C(22)	121.5(4)
C(23)-C(22)-C(21)	114.7(4)
C(23)-C(22)-C(33)	124.0(4)
C(21)-C(22)-C(33)	121.2(4)
C(22)-C(23)-C(24)	126.0(4)
C(23)-C(24)-C(25)	117.7(4)
C(23)-C(24)-C(41)	120.2(4)
C(25)-C(24)-C(41)	122.1(4)
C(26)-C(25)-C(24)	119.2(4)
C(26)-C(25)-C(42)	120.5(4)
C(24)-C(25)-C(42)	120.2(4)
C(25)-C(26)-C(21)	120.4(3)
C(25)-C(26)-C(28)	119.2(4)
C(21)-C(26)-C(28)	120.0(4)
O(2)-C(27)-C(28)	118.5(3)
O(2)-C(27)-C(32)	120.1(4)
C(28)-C(27)-C(32)	121.4(4)
C(27)-C(28)-C(29)	120.7(3)
C(27)-C(28)-C(26)	120.7(4)
C(29)-C(28)-C(26)	118.3(4)
C(30)-C(29)-C(28)	118.5(4)
C(30)-C(29)-C(43)	120.9(4)
C(28)-C(29)-C(43)	120.5(3)
C(31)-C(30)-C(29)	118.9(4)
C(31)-C(30)-C(44)	120.5(3)
C(29)-C(30)-C(44)	120.6(4)
C(30)-C(31)-C(32)	125.2(4)
C(31)-C(32)-C(27)	114.7(4)
C(31)-C(32)-C(37)	123.3(4)
C(27)-C(32)-C(37)	121.9(4)
C(35)-C(33)-C(34)	109.9(4)
C(35)-C(33)-C(36)	107.3(5)
C(34)-C(33)-C(36)	107.1(4)
C(35)-C(33)-C(22)	111.6(4)

C(34)-C(33)-C(22)	109.1(4)
C(36)-C(33)-C(22)	111.7(4)
C(32)-C(37)-C(39)	111.4(4)
C(32)-C(37)-C(38)	112.6(4)
C(39)-C(37)-C(38)	107.3(4)
C(32)-C(37)-C(40)	109.1(3)
C(39)-C(37)-C(40)	109.5(4)
C(38)-C(37)-C(40)	106.7(4)

Table 4. Anisotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for **4**. The anisotropic displacement factor exponent takes the form: $-2\pi^2 [h^2 a^{*2} U^{11} + \dots + 2 h k a^* b^* U^{12}]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
Mo(1)	28(1)	30(1)	33(1)	1(1)	0(1)	1(1)
N(1)	34(2)	33(2)	31(2)	4(2)	-6(2)	3(2)
O(1)	32(2)	33(2)	37(2)	2(2)	-5(1)	-2(2)
O(2)	30(2)	31(2)	40(2)	4(1)	4(2)	2(2)
C(1)	27(2)	31(2)	48(3)	3(2)	-3(2)	7(2)
C(2)	38(3)	34(3)	43(3)	-2(3)	12(2)	0(2)
C(3)	93(4)	50(4)	57(3)	-8(3)	26(3)	4(3)
C(4)	39(3)	72(3)	74(4)	24(3)	16(3)	20(3)
C(5)	33(3)	36(3)	37(3)	0(2)	2(2)	0(3)
C(6)	55(4)	74(4)	79(4)	41(3)	8(3)	3(3)
C(7)	62(4)	106(5)	96(5)	60(4)	10(3)	35(4)
C(8)	91(5)	67(4)	52(4)	14(3)	-6(4)	26(4)
C(9)	82(5)	64(4)	57(4)	20(3)	6(3)	-3(4)
C(10)	59(3)	65(4)	57(3)	10(3)	19(3)	10(3)
C(11)	31(2)	32(3)	36(3)	0(2)	0(2)	0(2)
C(12)	39(3)	48(3)	43(3)	5(2)	6(2)	-3(2)
C(13)	35(3)	68(4)	59(3)	5(3)	5(2)	-14(3)
C(14)	40(3)	48(3)	69(3)	-2(3)	-16(3)	3(3)
C(15)	53(3)	43(3)	44(3)	9(3)	-19(3)	-4(3)
C(16)	57(3)	58(4)	54(3)	-14(3)	0(3)	-18(3)
C(17)	46(3)	37(3)	66(4)	-20(3)	5(3)	6(3)
C(18)	34(3)	37(3)	54(3)	-4(2)	3(3)	-2(2)
C(19)	50(3)	32(3)	39(3)	5(2)	-3(2)	2(3)
C(20)	53(3)	32(3)	68(4)	11(3)	-11(3)	-10(3)
C(21)	30(3)	30(3)	30(3)	3(2)	-2(2)	-7(2)
C(22)	29(3)	31(3)	41(3)	5(2)	0(2)	-4(2)
C(23)	22(2)	48(4)	45(3)	15(3)	-6(2)	-6(3)
C(24)	27(3)	37(3)	36(3)	3(2)	1(2)	-2(3)
C(25)	30(3)	37(3)	33(3)	2(2)	5(2)	-2(3)
C(26)	30(3)	28(3)	27(2)	4(2)	-1(2)	-1(2)
C(27)	25(3)	24(2)	33(3)	-2(2)	6(2)	-7(2)
C(28)	22(3)	34(3)	30(2)	-5(2)	-4(2)	2(2)
C(29)	33(3)	28(3)	29(3)	-2(2)	-3(2)	-3(2)
C(30)	29(3)	34(3)	34(3)	-9(3)	5(2)	-4(3)
C(31)	19(2)	36(3)	43(3)	-7(3)	1(2)	-3(2)
C(32)	31(3)	25(3)	40(3)	-6(2)	-1(2)	-4(2)
C(33)	39(3)	28(3)	67(4)	4(3)	-5(3)	6(2)
C(34)	65(3)	45(3)	89(4)	5(3)	-10(3)	11(3)
C(35)	84(4)	69(4)	57(4)	0(3)	21(3)	31(3)
C(36)	36(3)	63(4)	140(5)	-37(4)	-23(3)	16(3)

C(37)	23(3)	40(3)	47(3)	2(2)	1(2)	5(2)
C(38)	38(3)	62(4)	90(4)	21(3)	5(3)	19(3)
C(39)	61(3)	68(4)	52(3)	2(3)	-13(3)	22(3)
C(40)	53(3)	35(3)	88(4)	8(3)	-6(3)	0(3)
C(41)	32(3)	55(3)	49(3)	-2(3)	-6(2)	-10(3)
C(42)	43(3)	45(3)	39(3)	-5(2)	-2(2)	-6(2)
C(43)	38(3)	50(3)	41(3)	7(2)	1(2)	-5(2)
C(44)	35(3)	45(3)	41(3)	3(2)	6(2)	-6(2)

Table 5. Hydrogen coordinates ($\times 10^4$) and isotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for **4**.

	x	y	z	U(eq)
H(1)	2714	4521	2132	42
H(3A)	2526	5213	3874	100
H(3B)	2524	5787	3294	100
H(3C)	3743	5392	3519	100
H(4A)	864	4485	3354	93
H(4B)	1104	3989	2745	93
H(4C)	958	4931	2712	93
H(6)	4876	3947	3023	84
H(7)	5713	2837	3477	106
H(8)	4606	2071	4158	84
H(9)	2580	2354	4295	81
H(10)	1716	3463	3822	72
H(12A)	6985	6743	2127	52
H(12B)	7736	5956	2271	52
H(13)	8874	7109	2586	65
H(14A)	9319	6691	3606	63
H(14B)	9166	5928	3177	63
H(15)	8066	5742	4107	56
H(16A)	7629	7109	4312	68
H(16B)	6419	6603	4334	68
H(17)	6103	7781	3733	60
H(18A)	4977	6633	3428	50
H(18B)	5284	7156	2838	50
H(19A)	6171	5385	3664	48
H(19B)	7229	5113	3219	48
H(20A)	8106	7954	3376	61
H(20B)	7211	7964	2805	61
H(23)	613	5145	466	46
H(31)	8567	4645	549	39
H(34A)	1500	7637	1071	99
H(34B)	1911	7167	474	99
H(34C)	2756	7187	1061	99
H(35A)	2148	6427	2015	105
H(35B)	926	5938	2019	105
H(35C)	899	6886	2015	105
H(36A)	-384	6929	1101	120
H(36B)	-442	5982	1120	120
H(36C)	-50	6425	507	120
H(38A)	9587	4106	1350	95
H(38B)	9209	3413	890	95

H(38C)	9542	3207	1582	95
H(39A)	8228	3573	2426	90
H(39B)	6998	4030	2314	90
H(39C)	8239	4481	2217	90
H(40A)	7225	2716	1009	88
H(40B)	6408	2909	1588	88
H(40C)	7674	2487	1678	88
H(41A)	476	3998	-138	68
H(41B)	1290	3307	138	68
H(41C)	1657	3726	-489	68
H(42A)	4063	3839	-421	64
H(42B)	3538	3203	48	64
H(42C)	4765	3646	197	64
H(43A)	4359	5854	-114	64
H(43B)	5311	5629	-629	64
H(43C)	5516	6394	-211	64
H(44A)	7887	6277	-163	61
H(44B)	7506	5653	-676	61
H(44C)	8696	5513	-288	61