Preparative Procedures for New Compounds

Preparation of solutions of $[Mo_2Cp_2(\mu\text{-}Cl)(\mu\text{-}PA_2)(CO)_2]$ (1a-c). In a typical experiment, a solution of the complex $[Mo_2Cp_2(CO)_6]$ (0.050 g, 0.102 mmol) and a slight excess of $ClPA_2$ in 10 mL of toluene (X= Ph, Cy) or diglyme (X= OEt) were refluxed for 1h or 10 min respectively to give a dark green solution shown (by IR and ^{31}P NMR) to contain compounds **1a-c** as major species. All attempts to isolate these highly air-sensitive complexes resulted in their progressive decomposition, but removal of solvent from the reaction mixture gave a crude residue ready for further use.

Preparation of THF solutions of $[Mo_2Cp_2(\mu\text{-}PA_2)(\mu\text{-}CO)_2]^-$ (2a-c). In a typical experiment, the crude products obtained as described above (containing less than 0.1 mmol of complexes **1a-c**) were dissolved in THF (10 mL) and then stirred with $Li[BHEt_3]$ (2 mL of a 1M solution in THF, 0.2 mmol; reaction time: 10 min) or Na(Hg) (for **1b**: *ca.* 0.5 mL of a 0.5% amalgam, excess; reaction time: 30 min) to give dark red solutions shown (by IR and ^{31}P NMR) to contain the anions **2a-c** as major species, as the corresponding Li^+ or Na^+ salts. All attempts to isolate these highly air-sensitive complexes resulted in their progressive decomposition. These solutions, however, were ready for further use (in the case of **2b**, the solution can be filtered using a cannula).

Preparation of $[Mo_2Cp_2(\mu\text{-}H)(\mu\text{-}PCy_2)(CO)_2]$ (3). Solid $[NH_4]PF_6$ (0.100 g, 0.612 mmol) was added to a THF solution of **2a** (Li^+ salt), prepared from $[Mo_2Cp_2(CO)_6]$ (0.250 g, 0.510 mmol) as described above, and the mixture was stirred for 5 min to give a dark-brown solution. Solvent was then removed under vacuum and the residue then chromatographed on alumina (activity IV) at –20°C. Elution with dichloromethane:petroleum ether (1:4) gave a brown fraction which yielded, after removal of solvents under vacuum, compound **3** as a brown air-sensitive powder (0.125 g, 45% overall yield). Anal. Calcd for $C_{24}H_{33}Mo_2O_2P$: C, 49.65; H, 5.73. Found: C, 49.79; H, 6.37.

Preparation of $[Mo_2Cp_2(\mu\text{-}CH_3)(\mu\text{-}PCy_2)(CO)_2]$ (4). A THF solution of **2a** (Li^+ salt), prepared from $[Mo_2Cp_2(CO)_6]$ (0.050g, 0.102 mmol) as described above, was stirred with MeI (0.1 mL, 1.6 mmol) for 30 min to give a brown solution. Workup as described for **3** gave compound **4** as

a brown air-sensitive solid (0.020g, 35%). Anal. Calcd for $C_{25}H_{35}Mo_2O_2P$: C, 50.50; H, 5.94. Found: C, 50.08; H, 5.35.

Preparation of $[Mo_2Cp_2(\mu\text{-}COMe)(\mu\text{-}PCy_2)(\mu\text{-}CO)]$ (5). A THF solution of **2a** (Li^+ salt), prepared from $[Mo_2Cp_2(CO)_6]$ (0.050g, 0.102 mmol) as described above, was stirred with solid $[Me_3O]BF_4$ (0.030g, 0.202 mmol) for 10 min without noticeable changes in colour. Workup of the mixture as described for **3** (elution with dichloromethane) gave compound **5** as a red-rose air-sensitive solid (0.026g, 45%). Anal. Calcd for $C_{25}H_{35}Mo_2O_2P$: C, 50.50; H, 5.94. Found: C, 50.46; H, 5.84.

Preparation of $[Mo_2Cp_2(\mu\text{-}COEt)(\mu\text{-}PCy_2)(\mu\text{-}CO)]$ (5'). A THF solution of **2a** (Li^+ salt), prepared from $[Mo_2Cp_2(CO)_6]$ (0.050 g, 0.102 mmol) as described above, was stirred with Et_2SO_4 (50 μL, 0.374 mmol) for 20 min without noticeable changes in colour. Workup of the mixture as described for **3** (elution with dichloromethane) gave compound **5'** as a red-rose air-sensitive solid (0.027 g, 45%). Anal. Calcd for $C_{26}H_{37}Mo_2O_2P$: C, 51.68; H, 6.17. Found: C, 51.26; H, 5.88. The crystals used in the X-ray study were grown by slow diffusion of petroleum-ether into a toluene solution of the complex, at room temperature.

Preparation of $[Mo_2Cp_2(\mu\text{-}\eta^1\text{:}\eta^2\text{-}CMeCH_2)(\mu\text{-}PCy_2)(CO)_2]$ (6). A THF solution of **2a** (Li^+ salt), prepared from $[Mo_2Cp_2(CO)_6]$ (0.050g, 0.102 mmol) as described above, was stirred with an excess of allyl chloride (0.1 mL, 1.2 mmol) for 30 min to give an orange mixture. Workup as described for **3** (elution with dichloromethane:petroleum ether 1:3) gave compound **6** as a yellow-orange air-sensitive solid (0.027g, 45%). Anal. Calcd for $C_{27}H_{37}Mo_2O_2P$: C, 52.25; H, 6.01. Found: C, 52.16; H, 6.29.

Preparation of $[Mo_2Cp_2(\mu\text{-}PCy_2)\{\mu\text{-}P(OEt)_2\}(CO)_2]$ (7). A THF solution of **2a** (Li^+ salt), prepared from $[Mo_2Cp_2(CO)_6]$ (0.050g, 0.102 mmol) as described above, was stirred with $ClP(OEt)_2$ (20 μL, 0.14 mmol) for 20 min, to give a brown-greenish mixture. Workup as described for **3** (chromatography on Florisil, elution with dichloromethane:petroleum ether 1:3) gave compound **7** as a green air-sensitive powder. Anal. Calcd for $C_{29}H_{44}Cl_2Mo_2O_2P$ ($7.CH_2Cl_2$): C, 44.58; H, 5.68. Found: C, 44.14; H, 5.39.

Preparation of $[Mo_2AuCp_2(\mu\text{-}PCy_2)(CO)_2\{P(p\text{-}tol)_3\}]$ (8). A THF solution of **2a** (Li^+ salt), prepared from $[Mo_2Cp_2(CO)_6]$ (0.050g, 0.102 mmol) as described above, was stirred with $[AuCl\{P(p\text{-}tol)_3\}]$ (0.060g, 0.112 mmol) for 10 min to give a black mixture. Solvent was then removed under vacuum, the residue was extracted with toluene and the solution was filtered using a cannula. Removal of solvent from the filtrate and washing of the residue with petroleum ether gave compound **8** as a brown-greenish solid of medium purity. All attempts to further purify this crude product resulted in its progressive decomposition.

X-Ray Structure Determination for Compound 5'

Formula: $Mo_2PO_2C_{26}H_{37}$, Triclinic, *P*-1, a = 17.489 (5) Å, b = 15.118 (5) Å, c = 10.254(5) Å, α = 91.75(5)°, β = 92.51(5)°, γ = 72.57(5)°, V = 2583.7(17) Å^3, Fw = 604.41, Z = 4, D_{calcd}= 1.554 Mg m^{-3}, $F(000)$ = 1232, λ = 0.71073 Å, μMoKα = 10.52 cm^{-1}. Intensity data were collected at r.t. on a Bruker AXS Smart 1000 single-crystal diffractometer , equipped with area detector, using a graphite monochromated MoKα radiation; 31274 reflections were measured (with θ in the range 1.41-27.62°) and included in the structure refinement. The structure was solved by Patterson and Fourier methods and refined by full-matrix least-squares procedures (based on F_o^2), with anisotropic thermal parameters in the last cycles of refinement for all non-hydrogen atoms. Two crystallographically independent molecules are present. The hydrogen atoms were introduced into the geometrically calculated positions and refined riding on the corresponding parent atoms. The refinement converged at $wR2$ = 0.1481 for all data and 564 variables; $R1$ = 0.0551 for 17413 reflections with $I > 2\sigma(I)$; GOF = 0.851. The SHELX-97 system of computer programs was used. (SHELX-97. Programs for Crystal Structure Analysis (Release 97-2). Sheldrick, G.M., Institüt für Anorganische Chemie der Universität, Tammanstrasse 4, D-3400 Göttingen, Germany, **1998**)

Crystal Data for $[Mo_2Cp_2(\mu\text{-}COEt)(\mu\text{-}PCy_2)(\mu\text{-}CO)]$ (5')

Table 1. Crystal data and structure refinement for $[Mo_2Cp_2(\mu\text{-}COEt)(\mu\text{-}PCy_2)(\mu\text{-}CO)]$ (5')

Empirical formula	$C_{26}\ H_{37}\ Mo_2\ O_2\ P$
Formula weight	604.41
Temperature	293(2) K
Wavelength	0.71073 Å
Crystal system, space group	triclinic, P-1
Unit cell dimensions	a = 17.489(5) Å α= 91.75(5)°.
	b = 15.118(5) Å β= 92.51(5)°.
	c = 10.254(5) Å γ= 72.57(5)°.
Volume	2583.7(17) $Å^3$
Z, Calculated density	4, 1.554 Mg/m^3
Absorption coefficient	1.052 mm^{-1}
F(000)	1232
Crystal size	0.12 x 0.21 x 0.09 mm
Theta range for data collection	1.22 to 27.62 °.
Limiting indices	$-22 \le h \le 22$, $-19 \le k \le 19$, $-13 \le l \le 13$
Reflections collected / unique	31414 / 31417 [R(int) = 0.0000]
Completeness to θ = 27.62	70.8 %
Absorption correction	None
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	31417 / 0 / 564
Goodness-of-fit on F^2	0.584
Final R indices [I>2σ (I)]	R1 = 0.0561, wR2 = 0.1501
R indices (all data)	R1 = 0.0952, wR2 = 0.1880
Largest diff. peak and hole	1.585 and -0.536 e. $Å^{-3}$

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$ x 10^3) for **5'**. U(eq) is defined as one third of the trace of the orthogonalized Uij tensor.

Atom	x	y	z	U(eq)
Mo(1A)	2150(1)	9377(1)	781(1)	54(1)
Mo(2A)	1411(1)	9482(1)	-1356(1)	58(1)
P(1A)	2843(1)	8904(1)	-1205(1)	61(1)
O(1A)	1050(2)	8077(3)	487(4)	81(1)
O(2A)	1298(2)	11412(2)	155(3)	72(1)
C(1A)	1360(3)	8634(4)	167(5)	64(1)
C(2A)	1506(3)	10507(3)	-123(5)	59(1)
C(3A)	804(3)	12041(3)	-793(6)	77(2)
C(4A)	647(4)	13000(4)	-336(7)[a]	107(2)
C(5A)	2024(4)	9151(6)	2982(5)	88(2)
C(6A)	2751(4)	8583(4)	2704(5)	76(2)
C(7A)	3229(4)	9109(5)	2363(6)	90(2)
C(8A)	2781(5)	10032(5)	2440(6)	92(2)
C(9A)	2040(4)	10037(5)	2849(6)	98(2)
C(10A)	1213(4)	9209(5)	-3638(6)	93(2)
C(11A)	815(4)	8702(5)	-2925(6)	85(2)
C(12A)	208(3)	9353(5)	-2266(6)	88(2)
C(13A)	249(4)	10241(5)	-2558(6)	92(2)
C(14A)	871(4)	10149(5)	-3387(6)	94(2)
C(15A)	3423(3)	7673(4)	-1498(6)	71(2)
C(16A)	2907(4)	7116(4)	-2014(7)	100(2)
C(17A)	3383(4)	6104(5)	-2220(8)	115(3)
C(18A)	3808(5)	5690(4)	-973(7)	108(2)
C(19A)	4343(4)	6229(5)	-495(8)	125(3)
C(20A)	3875(4)	7252(4)	-276(6)	100(2)
C(21A)	3436(3)	9606(4)	-1931(5)	81(2)
C(22A)	3369(4)	9605(5)	-3370(6)	108(2)
C(23A)	3810(4)	10268(6)	-3909(7)	129(3)
C(24A)	4659(5)	10010(6)	-3387(9)	145(3)
C(25A)	4720(4)	10015(6)	-1993(8)	122(3)
C(26A)	4295(4)	9365(5)	-1398(6)	94(2)
Mo(1B)	8418(1)	6109(1)	-4441(1)	55(1)
Mo(2B)	8039(1)	4733(1)	-3802(1)	56(1)
P(1B)	7102(1)	6255(1)	-3688(1)	60(1)
O(1B)	9393(2)	5118(2)	-1986(3)	74(1)
O(2B)	8389(2)	4605(2)	-6704(3)	76(1)
C(1B)	8907(3)	5234(3)	-2869(5)	59(1)
C(2B)	8312(3)	5032(3)	-5538(5)	60(1)
C(3B)	8657(3)	5024(4)	-7778(5)	84(2)
C(4B)	8639(4)	4463(5)	-8954(6)	114(2)
C(5B)	8503(4)	7238(4)	-5919(6)	76(2)
C(6B)	8253(4)	7714(4)	-4742(6)	76(2)
C(7B)	8867(4)	7395(4)	-3801(6)	75(2)
C(8B)	9497(4)	6719(4)	-4412(8)	88(2)
C(9B)	9276(4)	6630(4)	-5683(7)	88(2)
C(10B)	7973(6)	3617(4)	-2271(6)	92(2)
C(11B)	8580(5)	3213(4)	-3107(9)	98(2)
C(12B)	8229(6)	3208(4)	-4361(8)	107(3)
C(13B)	7431(6)	3569(5)	-4260(8)	106(2)
C(14B)	7282(5)	3833(4)	-2997(9)	99(2)
C(15B)	6169(4)	6640(5)	-4724(6)	99(2)
C(16B)	5584(4)	6148(7)	-4415(8)	156(4)
C(17B)	4804(5)	6554(10)	-5271(11)	246(8)
C(18B)	4862(5)	6623(9)	-6492(9)	173(5)
C(19B)	5419(4)	7145(7)	-6766(8)	174(4)
C(20B)	6184(5)	6821(9)	-5875(10)	270(8)

C(21B)	6743(3)	6821(4)	-2099(6)	71(2)
C(22B)	6937(6)	6194(5)	-1042(7)	173(4)
C(23B)	6647(7)	6644(5)	288(8)	169(4)
C(24B)	6915(4)	7438(5)	613(7)	102(2)
C(25B)	6680(6)	8098(5)	-480(8)	159(4)
C(26B)	6983(6)	7626(5)	-1785(6)	138(3)

Table 3. Bond lengths [Å] and angles [°] for **5'**.

Mo(1A)-C(2A)	1.974(5)	Mo(2B)-P(1B)	2.395(2)
Mo(1A)-C(1A)	2.091(5)	P(1B)-C(21B)	1.861(6)
Mo(1A)-C(9A)	2.307(6)	P(1B)-C(15B)	1.857(6)
Mo(1A)-C(5A)	2.323(6)	O(1B)-C(1B)	1.195(5)
Mo(1A)-C(8A)	2.341(6)	O(2B)-C(2B)	1.333(5)
Mo(1A)-C(6A)	2.376(5)	O(2B)-C(3B)	1.448(6)
Mo(1A)-P(1A)	2.395(2)	C(3B)-C(4B)	1.458(7)
Mo(1A)-C(7A)	2.381(6)	C(5B)-C(9B)	1.404(8)
Mo(1A)-Mo(2A)	2.4793(15)	C(5B)-C(6B)	1.404(7)
Mo(2A)-C(2A)	2.011(5)	C(6B)-C(7B)	1.394(7)
Mo(2A)-C(1A)	2.073(6)	C(7B)-C(8B)	1.414(8)
Mo(2A)-C(13A)	2.332(6)	C(8B)-C(9B)	1.357(8)
Mo(2A)-C(14A)	2.373(6)	C(10B)-C(14B)	1.348(9)
Mo(2A)-C(12A)	2.323(5)	C(10B)-C(11B)	1.377(9)
Mo(2A)-C(11A)	2.355(5)	C(11B)-C(12B)	1.400(9)
Mo(2A)-P(1A)	2.3922(17)	C(12B)-C(13B)	1.344(10)
Mo(2A)-C(10A)	2.392(6)	C(13B)-C(14B)	1.357(9)
P(1A)-C(21A)	1.879(5)	C(15B)-C(20B)	1.223(9)
P(1A)-C(15A)	1.856(5)	C(15B)-C(16B)	1.484(8)
O(1A)-C(1A)	1.190(6)	C(16B)-C(17B)	1.560(10)
O(2A)-C(2A)	1.332(5)	C(17B)-C(18B)	1.270(10)
O(2A)-C(3A)	1.443(6)	C(18B)-C(19B)	1.465(11)
C(3A)-C(4A)	1.459(7)	C(19B)-C(20B)	1.547(9)
C(5A)-C(9A)	1.359(8)	C(21B)-C(26B)	1.425(8)
C(5A)-C(6A)	1.341(8)	C(21B)-C(22B)	1.424(9)
C(6A)-C(7A)	1.377(8)	C(22B)-C(23B)	1.541(9)
C(7A)-C(8A)	1.382(8)	C(23B)-C(24B)	1.438(10)
C(8A)-C(9A)	1.378(9)	C(24B)-C(25B)	1.485(9)
C(10A)-C(14A)	1.388(8)	C(25B)-C(26B)	1.535(9)
C(10A)-C(11A)	1.417(9)		
C(11A)-C(12A)	1.396(7)	C(2A)-Mo(1A)-C(1A)	92.7(2)
C(12A)-C(13A)	1.406(8)	C(2A)-Mo(1A)-C(9A)	95.8(2)
C(13A)-C(14A)	1.382(8)	C(1A)-Mo(1A)-C(9A)	118.8(3)
C(15A)-C(20A)	1.503(7)	C(2A)-Mo(1A)-C(5A)	122.3(2)
C(15A)-C(16A)	1.479(7)	C(1A)-Mo(1A)-C(5A)	93.8(2)
C(16A)-C(17A)	1.521(8)	C(9A)-Mo(1A)-C(5A)	34.1(2)
C(17A)-C(18A)	1.506(9)	C(2A)-Mo(1A)-C(8A)	100.3(2)
C(18A)-C(19A)	1.474(8)	C(1A)-Mo(1A)-C(8A)	150.9(2)
C(19A)-C(20A)	1.533(8)	C(9A)-Mo(1A)-C(8A)	34.5(2)
C(21A)-C(22A)	1.475(8)	C(5A)-Mo(1A)-C(8A)	57.3(2)
C(21A)-C(26A)	1.517(7)	C(2A)-Mo(1A)-C(6A)	151.4(2)
C(22A)-C(23A)	1.562(8)	C(1A)-Mo(1A)-C(6A)	101.7(2)
C(23A)-C(24A)	1.496(9)	C(9A)-Mo(1A)-C(6A)	55.5(2)
C(24A)-C(25A)	1.428(9)	C(5A)-Mo(1A)-C(6A)	33.15(19)
C(25A)-C(26A)	1.551(8)	C(8A)-Mo(1A)-C(6A)	56.50(19)
Mo(1B)-C(2B)	2.001(5)	C(2A)-Mo(1A)-P(1A)	87.61(15)
Mo(1B)-C(1B)	2.099(5)	C(1A)-Mo(1A)-P(1A)	88.58(15)
Mo(1B)-C(8B)	2.335(5)	C(9A)-Mo(1A)-P(1A)	152.1(2)
Mo(1B)-C(9B)	2.332(6)	C(5A)-Mo(1A)-P(1A)	149.8(2)
Mo(1B)-C(7B)	2.370(5)	C(8A)-Mo(1A)-P(1A)	117.6(2)
Mo(1B)-C(5B)	2.358(5)	C(6A)-Mo(1A)-P(1A)	117.04(17)
Mo(1B)-C(6B)	2.384(5)	C(2A)-Mo(1A)-C(7A)	132.1(2)
Mo(1B)-P(1B)	2.4040(16)	C(1A)-Mo(1A)-C(7A)	133.7(2)
Mo(1B)-Mo(2B)	2.4775(11)	C(9A)-Mo(1A)-C(7A)	56.0(2)
Mo(2B)-C(2B)	1.963(5)	C(5A)-Mo(1A)-C(7A)	56.0(2)
Mo(2B)-C(1B)	2.076(5)	C(8A)-Mo(1A)-C(7A)	34.0(2)
Mo(2B)-C(12B)	2.287(5)	C(6A)-Mo(1A)-C(7A)	33.66(19)
Mo(2B)-C(13B)	2.339(6)	P(1A)-Mo(1A)-C(7A)	101.98(17)
Mo(2B)-C(11B)	2.329(6)	C(2A)-Mo(1A)-Mo(2A)	52.20(15)
Mo(2B)-C(10B)	2.371(6)	C(1A)-Mo(1A)-Mo(2A)	53.11(15)
Mo(2B)-C(14B)	2.351(6)	C(9A)-Mo(1A)-Mo(2A)	141.51(16)

C(5A)-Mo(1A)-Mo(2A)	141.78(16)	C(6A)-Mo(1A)-Mo(2A)	152.73(13)
C(8A)-Mo(1A)-Mo(2A)	150.77(16)		
P(1A)-Mo(1A)-Mo(2A)	58.75(5)	C(7A)-C(8A)-Mo(1A)	74.6(3)
C(7A)-Mo(1A)-Mo(2A)	160.69(17)	C(9A)-C(8A)-Mo(1A)	71.4(3)
C(2A)-Mo(2A)-C(1A)	92.2(2)	C(5A)-C(9A)-C(8A)	109.6(6)
C(2A)-Mo(2A)-C(13A)	100.7(2)	C(5A)-C(9A)-Mo(1A)	73.6(3)
C(1A)-Mo(2A)-C(13A)	120.2(2)	C(8A)-C(9A)-Mo(1A)	74.1(4)
C(2A)-Mo(2A)-C(14A)	108.2(2)	C(14A)-C(10A)-C(11A)	109.0(6)
C(1A)-Mo(2A)-C(14A)	148.6(2)	C(14A)-C(10A)-Mo(2A)	72.3(3)
C(13A)-Mo(2A)-C(14A)	34.2(2)	C(11A)-C(10A)-Mo(2A)	71.2(3)
C(2A)-Mo(2A)-C(12A)	124.4(2)	C(12A)-C(11A)-C(10A)	106.6(6)
C(1A)-Mo(2A)-C(12A)	91.1(2)	C(12A)-C(11A)-Mo(2A)	71.4(3)
C(13A)-Mo(2A)-C(12A)	35.2(2)	C(10A)-C(11A)-Mo(2A)	74.1(3)
C(14A)-Mo(2A)-C(12A)	57.8(2)	C(11A)-C(12A)-C(13A)	107.9(6)
C(2A)-Mo(2A)-C(11A)	157.9(2)	C(11A)-C(12A)-Mo(2A)	73.9(3)
C(1A)-Mo(2A)-C(11A)	94.9(2)	C(13A)-C(12A)-Mo(2A)	72.7(3)
C(13A)-Mo(2A)-C(11A)	57.8(2)	C(14A)-C(13A)-C(12A)	108.9(6)
C(14A)-Mo(2A)-C(11A)	57.8(2)	C(14A)-C(13A)-Mo(2A)	74.6(3)
C(12A)-Mo(2A)-C(11A)	34.71(18)	C(12A)-C(13A)-Mo(2A)	72.1(3)
C(2A)-Mo(2A)-P(1A)	86.87(15)	C(10A)-C(14A)-C(13A)	107.5(7)
C(1A)-Mo(2A)-P(1A)	89.10(15)	C(10A)-C(14A)-Mo(2A)	73.8(4)
C(13A)-Mo(2A)-P(1A)	149.02(19)	C(13A)-C(14A)-Mo(2A)	71.3(4)
C(14A)-Mo(2A)-P(1A)	114.93(19)	C(20A)-C(15A)-C(16A)	110.5(5)
C(12A)-Mo(2A)-P(1A)	148.64(17)	C(20A)-C(15A)-P(1A)	110.9(4)
C(11A)-Mo(2A)-P(1A)	114.07(17)	C(16A)-C(15A)-P(1A)	112.1(4)
C(2A)-Mo(2A)-C(10A)	139.9(2)	C(15A)-C(16A)-C(17A)	111.7(5)
C(1A)-Mo(2A)-C(10A)	127.4(2)	C(18A)-C(17A)-C(16A)	111.1(6)
C(13A)-Mo(2A)-C(10A)	56.4(2)	C(19A)-C(18A)-C(17A)	109.1(6)
C(14A)-Mo(2A)-C(10A)	33.9(2)	C(18A)-C(19A)-C(20A)	111.2(6)
C(12A)-Mo(2A)-C(10A)	57.1(2)	C(15A)-C(20A)-C(19A)	111.5(5)
C(11A)-Mo(2A)-C(10A)	34.7(2)	C(22A)-C(21A)-C(26A)	113.3(5)
P(1A)-Mo(2A)-C(10A)	98.94(18)	C(22A)-C(21A)-P(1A)	112.2(4)
C(2A)-Mo(2A)-Mo(1A)	50.87(14)	C(26A)-C(21A)-P(1A)	113.3(4)
C(1A)-Mo(2A)-Mo(1A)	53.79(14)	C(21A)-C(22A)-C(23A)	109.8(5)
C(13A)-Mo(2A)-Mo(1A)	145.88(17)	C(24A)-C(23A)-C(22A)	110.2(6)
C(14A)-Mo(2A)-Mo(1A)	156.12(18)	C(25A)-C(24A)-C(23A)	112.9(7)
C(12A)-Mo(2A)-Mo(1A)	140.45(17)	C(24A)-C(25A)-C(26A)	112.3(6)
C(11A)-Mo(2A)-Mo(1A)	145.82(18)	C(21A)-C(26A)-C(25A)	109.5(5)
P(1A)-Mo(2A)-Mo(1A)	58.88(6)	C(2B)-Mo(1B)-C(1B)	91.0(2)
C(10A)-Mo(2A)-Mo(1A)	157.30(17)	C(2B)-Mo(1B)-C(8B)	125.8(2)
C(21A)-P(1A)-C(15A)	105.7(3)	C(1B)-Mo(1B)-C(8B)	92.9(2)
C(21A)-P(1A)-Mo(2A)	118.24(19)	C(2B)-Mo(1B)-C(9B)	100.8(2)
C(15A)-P(1A)-Mo(2A)	124.01(17)	C(1B)-Mo(1B)-C(9B)	118.7(2)
C(21A)-P(1A)-Mo(1A)	121.6(2)	C(8B)-Mo(1B)-C(9B)	33.8(2)
C(15A)-P(1A)-Mo(1A)	120.6(2)	C(2B)-Mo(1B)-C(7B)	158.0(2)
Mo(2A)-P(1A)-Mo(1A)	62.38(6)	C(1B)-Mo(1B)-C(7B)	99.3(2)
C(2A)-O(2A)-C(3A)	118.2(4)	C(8B)-Mo(1B)-C(7B)	34.96(19)
O(1A)-C(1A)-Mo(2A)	142.7(4)	C(9B)-Mo(1B)-C(7B)	57.2(2)
O(1A)-C(1A)-Mo(1A)	144.2(4)	C(2B)-Mo(1B)-C(5B)	105.9(2)
Mo(2A)-C(1A)-Mo(1A)	73.09(18)	C(1B)-Mo(1B)-C(5B)	150.1(2)
O(2A)-C(2A)-Mo(1A)	135.0(4)	C(8B)-Mo(1B)-C(5B)	57.2(2)
O(2A)-C(2A)-Mo(2A)	148.0(4)	C(9B)-Mo(1B)-C(5B)	34.8(2)
Mo(1A)-C(2A)-Mo(2A)	76.93(18)	C(7B)-Mo(1B)-C(5B)	57.1(2)
O(2A)-C(3A)-C(4A)	110.6(5)	C(2B)-Mo(1B)-C(6B)	137.1(2)
C(9A)-C(5A)-C(6A)	107.9(6)	C(1B)-Mo(1B)-C(6B)	131.5(2)
C(9A)-C(5A)-Mo(1A)	72.3(4)	C(8B)-Mo(1B)-C(6B)	57.3(2)
C(6A)-C(5A)-Mo(1A)	75.6(3)	C(9B)-Mo(1B)-C(6B)	57.3(2)
C(5A)-C(6A)-C(7A)	108.7(5)	C(7B)-Mo(1B)-C(6B)	34.09(17)
C(5A)-C(6A)-Mo(1A)	71.3(3)	C(5B)-Mo(1B)-C(6B)	34.44(18)
C(7A)-C(6A)-Mo(1A)	73.4(3)	C(2B)-Mo(1B)-P(1B)	87.09(15)
C(8A)-C(7A)-C(6A)	108.1(6)	C(1B)-Mo(1B)-P(1B)	88.79(14)
C(8A)-C(7A)-Mo(1A)	71.4(3)	C(8B)-Mo(1B)-P(1B)	147.03(19)
C(6A)-C(7A)-Mo(1A)	73.0(3)	C(9B)-Mo(1B)-P(1B)	150.88(18)
C(7A)-C(8A)-C(9A)	105.7(6)	C(7B)-Mo(1B)-P(1B)	112.36(16)

C(5B)-Mo(1B)-P(1B)	116.05(17)
C(6B)-Mo(1B)-P(1B)	98.08(15)
C(2B)-Mo(1B)-Mo(2B)	50.63(15)
C(1B)-Mo(1B)-Mo(2B)	53.16(14)
C(8B)-Mo(1B)-Mo(2B)	142.31(15)
C(9B)-Mo(1B)-Mo(2B)	145.08(15)
C(7B)-Mo(1B)-Mo(2B)	148.53(15)
C(5B)-Mo(1B)-Mo(2B)	153.97(15)
C(6B)-Mo(1B)-Mo(2B)	156.76(14)
P(1B)-Mo(1B)-Mo(2B)	58.74(5)
C(2B)-Mo(2B)-C(1B)	92.8(2)
C(2B)-Mo(2B)-C(12B)	92.9(2)
C(1B)-Mo(2B)-C(12B)	124.1(3)
C(2B)-Mo(2B)-C(13B)	102.9(2)
C(1B)-Mo(2B)-C(13B)	152.3(2)
C(12B)-Mo(2B)-C(13B)	33.8(2)
C(2B)-Mo(2B)-C(11B)	117.8(3)
C(1B)-Mo(2B)-C(11B)	95.8(2)
C(12B)-Mo(2B)-C(11B)	35.3(2)
C(13B)-Mo(2B)-C(11B)	56.6(3)
C(2B)-Mo(2B)-C(10B)	149.8(2)
C(1B)-Mo(2B)-C(10B)	99.7(2)
C(12B)-Mo(2B)-C(10B)	57.4(2)
C(13B)-Mo(2B)-C(10B)	56.2(2)
C(11B)-Mo(2B)-C(10B)	34.1(2)
C(2B)-Mo(2B)-C(14B)	135.6(3)
C(1B)-Mo(2B)-C(14B)	130.1(3)
C(12B)-Mo(2B)-C(14B)	56.2(2)
C(13B)-Mo(2B)-C(14B)	33.6(2)
C(11B)-Mo(2B)-C(14B)	55.7(3)
C(10B)-Mo(2B)-C(14B)	33.2(2)
C(2B)-Mo(2B)-P(1B)	88.20(15)
C(1B)-Mo(2B)-P(1B)	89.60(14)
C(12B)-Mo(2B)-P(1B)	146.2(3)
C(13B)-Mo(2B)-P(1B)	113.3(2)
C(11B)-Mo(2B)-P(1B)	153.0(2)
C(10B)-Mo(2B)-P(1B)	119.0(2)
C(14B)-Mo(2B)-P(1B)	101.22(19)
C(2B)-Mo(2B)-Mo(1B)	52.00(13)
C(1B)-Mo(2B)-Mo(1B)	54.05(15)
C(12B)-Mo(2B)-Mo(1B)	141.48(17)
C(13B)-Mo(2B)-Mo(1B)	151.40(19)
C(11B)-Mo(2B)-Mo(1B)	142.40(19)
C(10B)-Mo(2B)-Mo(1B)	152.36(15)
C(14B)-Mo(2B)-Mo(1B)	160.25(18)
P(1B)-Mo(2B)-Mo(1B)	59.10(4)
C(21B)-P(1B)-C(15B)	100.8(3)
C(21B)-P(1B)-Mo(2B)	121.68(19)
C(15B)-P(1B)-Mo(2B)	123.9(3)
C(21B)-P(1B)-Mo(1B)	122.71(19)
C(15B)-P(1B)-Mo(1B)	124.0(2)
Mo(2B)-P(1B)-Mo(1B)	62.16(5)
C(2B)-O(2B)-C(3B)	119.1(4)
O(1B)-C(1B)-Mo(2B)	144.8(4)
O(1B)-C(1B)-Mo(1B)	142.4(4)
Mo(2B)-C(1B)-Mo(1B)	72.80(18)
O(2B)-C(2B)-Mo(2B)	134.8(3)
O(2B)-C(2B)-Mo(1B)	147.7(4)
Mo(2B)-C(2B)-Mo(1B)	77.37(18)
C(4B)-C(3B)-O(2B)	108.5(5)
C(9B)-C(5B)-C(6B)	107.3(6)
C(9B)-C(5B)-Mo(1B)	71.6(3)
C(6B)-C(5B)-Mo(1B)	73.8(3)
C(7B)-C(6B)-C(5B)	107.9(5)
C(7B)-C(6B)-Mo(1B)	72.4(3)
C(5B)-C(6B)-Mo(1B)	71.8(3)
C(6B)-C(7B)-C(8B)	107.4(5)
C(6B)-C(7B)-Mo(1B)	73.5(3)
C(8B)-C(7B)-Mo(1B)	71.2(3)
C(7B)-C(8B)-C(9B)	108.6(6)
C(7B)-C(8B)-Mo(1B)	73.9(3)
C(9B)-C(8B)-Mo(1B)	73.0(3)
C(5B)-C(9B)-C(8B)	108.9(6)
C(5B)-C(9B)-Mo(1B)	73.6(3)
C(8B)-C(9B)-Mo(1B)	73.2(4)
C(14B)-C(10B)-C(11B)	106.8(6)
C(14B)-C(10B)-Mo(2B)	72.6(4)
C(11B)-C(10B)-Mo(2B)	71.3(4)
C(10B)-C(11B)-C(12B)	107.4(7)
C(10B)-C(11B)-Mo(2B)	74.6(4)
C(12B)-C(11B)-Mo(2B)	70.7(3)
C(13B)-C(12B)-C(11B)	107.6(6)
C(13B)-C(12B)-Mo(2B)	75.3(4)
C(11B)-C(12B)-Mo(2B)	74.0(3)
C(14B)-C(13B)-C(12B)	108.0(7)
C(14B)-C(13B)-Mo(2B)	73.7(3)
C(12B)-C(13B)-Mo(2B)	71.0(4)
C(13B)-C(14B)-C(10B)	110.2(7)
C(13B)-C(14B)-Mo(2B)	72.7(4)
C(10B)-C(14B)-Mo(2B)	74.2(4)
C(20B)-C(15B)-C(16B)	115.3(7)
C(20B)-C(15B)-P(1B)	121.6(6)
C(16B)-C(15B)-P(1B)	112.0(5)
C(15B)-C(16B)-C(17B)	108.4(7)
C(18B)-C(17B)-C(16B)	118.8(10)
C(17B)-C(18B)-C(19B)	111.2(9)
C(18B)-C(19B)-C(20B)	110.2(8)
C(15B)-C(20B)-C(19B)	123.0(8)
C(26B)-C(21B)-C(22B)	110.5(6)
C(26B)-C(21B)-P(1B)	114.3(5)
C(22B)-C(21B)-P(1B)	112.9(4)
C(21B)-C(22B)-C(23B)	114.1(6)
C(24B)-C(23B)-C(22B)	113.1(7)
C(23B)-C(24B)-C(25B)	108.7(7)
C(24B)-C(25B)-C(26B)	111.4(6)
C(21B)-C(26B)-C(25B)	114.3(6)

Table 4. Anisotropic displacement parameters ($A^2 \times 10^3$) for **5'**. The anisotropic displacement factor exponent takes the form:
$-2\pi^2 [h^2 a^{*2} U_{11} + ... + 2 h k a^* b^* U_{12}]$

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
Mo(1A)	56(1)	53(1)	50(1)	5(1)	1(1)	-14(1)
Mo(2A)	50(1)	72(1)	50(1)	3(1)	4(1)	-14(1)
P(1A)	52(1)	71(1)	59(1)	6(1)	6(1)	-16(1)
O(1A)	76(2)	83(3)	95(3)	14(2)	1(2)	-40(2)
O(2A)	79(2)	59(2)	73(2)	10(2)	-9(2)	-12(2)
C(1A)	60(3)	69(3)	61(3)	-2(3)	4(3)	-18(3)
C(2A)	55(3)	67(3)	56(3)	10(2)	5(2)	-20(3)
C(3A)	78(4)	63(4)	87(4)	23(3)	-4(3)	-16(3)
C(4A)	113(5)	71(4)	117(6)	11(4)	-17(5)	0(4)
C(5A)	89(5)	131(6)	51(4)	5(4)	1(3)	-43(5)
C(6A)	102(5)	58(3)	69(4)	14(3)	-13(3)	-26(4)
C(7A)	70(4)	118(6)	80(5)	9(4)	-24(3)	-30(4)
C(8A)	145(7)	76(5)	78(4)	13(3)	-33(4)	-69(5)
C(9A)	100(5)	90(5)	76(5)	-26(4)	-21(4)	11(4)
C(10A)	91(5)	118(6)	61(4)	-18(4)	2(4)	-20(5)
C(11A)	82(4)	113(5)	57(4)	-10(4)	-16(3)	-27(4)
C(12A)	59(4)	133(6)	74(4)	-11(4)	0(3)	-35(4)
C(13A)	60(4)	120(6)	78(5)	5(4)	-25(3)	-4(4)
C(14A)	95(5)	130(6)	59(4)	28(4)	-16(4)	-35(5)
C(15A)	56(3)	86(4)	73(4)	1(3)	8(3)	-23(3)
C(16A)	84(4)	76(4)	124(6)	-24(4)	-19(4)	-6(3)
C(17A)	118(6)	91(5)	130(7)	-18(5)	-16(5)	-26(4)
C(18A)	128(6)	80(5)	112(6)	0(4)	19(5)	-21(5)
C(19A)	118(6)	96(5)	129(7)	-3(5)	-38(5)	12(5)
C(20A)	106(5)	68(4)	99(5)	-2(3)	-28(4)	9(4)
C(21A)	59(3)	115(5)	71(4)	18(3)	7(3)	-27(3)
C(22A)	107(5)	164(7)	74(5)	15(4)	8(4)	-70(5)
C(23A)	89(5)	190(8)	117(6)	74(6)	6(5)	-48(5)
C(24A)	122(7)	207(10)	131(8)	11(7)	39(6)	-82(7)
C(25A)	118(6)	160(8)	119(7)	-12(6)	31(5)	-88(6)
C(26A)	87(4)	124(6)	83(5)	15(4)	0(4)	-50(4)
Mo(1B)	55(1)	54(1)	59(1)	7(1)	1(1)	-21(1)
Mo(2B)	71(1)	54(1)	50(1)	3(1)	1(1)	-28(1)
P(1B)	56(1)	65(1)	63(1)	10(1)	-2(1)	-24(1)
O(1B)	77(2)	78(2)	68(3)	10(2)	-13(2)	-25(2)
O(2B)	96(3)	87(3)	57(2)	-3(2)	10(2)	-42(2)
C(1B)	56(3)	62(3)	54(3)	4(3)	5(3)	-11(3)
C(2B)	62(3)	54(3)	65(3)	3(2)	-15(3)	-19(2)
C(3B)	92(4)	116(5)	53(4)	14(3)	3(3)	-45(4)
C(4B)	148(6)	132(6)	68(5)	-14(4)	22(4)	-49(5)
C(5B)	96(5)	74(4)	72(4)	26(3)	-3(4)	-44(4)
C(6B)	84(4)	56(3)	94(5)	9(3)	7(4)	-28(3)
C(7B)	88(4)	68(4)	79(4)	7(3)	-9(4)	-40(3)
C(8B)	68(4)	72(4)	132(6)	31(4)	-19(4)	-34(3)
C(9B)	85(5)	73(4)	118(6)	-8(4)	33(4)	-42(4)
C(10B)	160(7)	74(4)	55(4)	10(3)	-14(5)	-58(5)
C(11B)	113(6)	53(4)	132(7)	28(4)	-14(5)	-29(4)
C(12B)	189(8)	54(4)	94(6)	-21(4)	73(6)	-54(5)
C(13B)	168(8)	104(6)	81(6)	5(4)	-11(5)	-96(6)
C(14B)	135(7)	72(4)	114(6)	3(4)	43(6)	-62(5)
C(15B)	83(4)	148(6)	81(5)	53(4)	-25(4)	-55(4)
C(16B)	118(6)	258(11)	132(7)	105(7)	-59(5)	-117(7)
C(17B)	115(7)	510(20)	177(12)	121(14)	-52(8)	-179(12)
C(18B)	121(7)	325(15)	107(7)	-9(9)	-22(6)	-124(9)
C(19B)	106(6)	303(13)	136(8)	121(8)	-41(5)	-92(7)

C(20B)	134(7)	530(20)	218(12)	238(14)	-113(8)	-202(11)
C(21B)	65(4)	63(4)	82(4)	11(3)	-1(3)	-15(3)
C(22B)	347(14)	81(5)	94(6)	-9(4)	96(7)	-59(7)
C(23B)	313(13)	107(6)	89(6)	13(5)	78(7)	-54(8)
C(24B)	110(5)	117(6)	75(5)	-2(4)	-9(4)	-28(5)
C(25B)	310(13)	87(5)	99(6)	-16(5)	51(7)	-85(7)
C(26B)	274(11)	113(6)	66(4)	-9(4)	39(6)	-112(7)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$ x 10^3) for **5'**.

Atom	x	y	z	U(eq)
H(3A1)	300	11902	-940	116(11)
H(3A2)	1073	11957	-1615	116(11)
H(4A1)	321	13407	-980	151(11)
H(4A2)	1146	13138	-198	151(11)
H(4A3)	371	13085	469	151(11)
H(5A)	1587	8972	3224	100(5)
H(6A)	2907	7939	2737	100(5)
H(7A)	3760	8884	2122	100(5)
H(8A)	2946	10544	2254	100(5)
H(9A)	1615	10566	3009	100(5)
H(10A)	1637	8952	-4186	100(5)
H(11A)	933	8060	-2901	100(5)
H(12A)	-158	9223	-1728	100(5)
H(13A)	-88	10799	-2244	100(5)
H(14A)	1031	10629	-3717	100(5)
H(15A)	3817	7657	-2155	111(12)
H(16A)	2654	7369	-2838	161(5)
H(16B)	2486	7158	-1408	161(5)
H(17A)	3023	5757	-2530	161(5)
H(17B)	3774	6056	-2882	161(5)
H(18A)	3419	5702	-322	161(5)
H(18B)	4119	5049	-1130	161(5)
H(19A)	4752	6181	-1125	161(5)
H(19B)	4609	5973	319	161(5)
H(20A)	3500	7304	413	161(5)
H(20B)	4245	7593	3	161(5)
H(21A)	3181	10248	-1650	111(12)
H(22A)	2808	9808	-3653	161(5)
H(22B)	3606	8981	-3709	161(5)
H(23A)	3803	10228	-4855	161(5)
H(23B)	3533	10903	-3653	161(5)
H(24A)	4944	9396	-3714	161(5)
H(24B)	4917	10442	-3708	161(5)
H(25A)	4482	10642	-1666	161(5)
H(25B)	5281	9821	-1714	161(5)
H(26A)	4577	8724	-1617	161(5)
H(26B)	4302	9435	-454	161(5)
H(3B1)	8308	5651	-7904	116(11)
H(3B2)	9198	5052	-7588	116(11)
H(4B1)	8819	4729	-9674	151(11)
H(4B2)	8101	4445	-9141	151(11)
H(4B3)	8986	3844	-8821	151(11)
H(5B)	8211	7311	-6709	100(5)
H(6B)	7765	8163	-4613	100(5)
H(7B)	8862	7590	-2932	100(5)
H(8B)	9982	6390	-4011	100(5)
H(9B)	9587	6232	-6298	100(5)
H(10B)	8028	3721	-1378	100(5)
H(11B)	9123	2985	-2881	100(5)
H(12B)	8499	2995	-5124	100(5)
H(13B)	7049	3628	-4937	100(5)
H(14B)	6777	4122	-2677	100(5)
H(15B)	5916	7258	-4348	111(12)
H(16C)	5463	6231	-3497	161(5)
H(16D)	5806	5489	-4599	161(5)
H(17C)	4455	6174	-5148	161(5)
H(17D)	4535	7168	-4927	161(5)
H(18C)	4338	6932	-6879	161(5)

H(18D)	5050	6007	-6885	161(5)
H(19C)	5160	7801	-6619	161(5)
H(19D)	5563	7051	-7674	161(5)
H(20C)	6532	6271	-6289	161(5)
H(20D)	6451	7297	-5905	161(5)
H(21B)	6156	7033	-2187	111(12)
H(22C)	7514	5920	-979	161(5)
H(22D)	6701	5698	-1230	161(5)
H(23C)	6066	6832	273	161(5)
H(23D)	6841	6187	963	161(5)
H(24C)	7493	7246	753	161(5)
H(24D)	6673	7734	1409	161(5)
H(25C)	6899	8610	-307	161(5)
H(25D)	6100	8348	-539	161(5)
H(26C)	6788	8070	-2477	161(5)
H(26D)	7564	7454	-1757	161(5)