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Half-sandwich complexes with intramolecular amino group coordination: synthesis and structure of cationic molybdenum dienyl complexes

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Abstract

A half-sandwich cationic molybdenum dienyl complex with intramolecular amino group coordination was obtained from $\eta^5\text{-C}_5\text{H}_4\text{CH}_2\text{CH}_2\text{N}(\text{CH}_3)_2\text{Mo}(\text{CO})_2(\eta^3\text{-C}_6\text{H}_9)$ via hydride abstraction followed by CO extrusion. It crystallized as an orange monoclinic compound with $a = 10.4867(14)$, $b = 14.843(3)$, $c = 11.882(3)$ Å, $\beta = 94.057(15)^\circ$. The cyclohexadienyl ligand is shown as boat conformation. A second bidentate complex was isolated. Spectroscopic and elemental analysis data support the demethylation product of $[\eta^5\text{-C}_5\text{H}_4\text{CH}_2\text{CH}_2\text{N}(\text{CH}_3)\text{Mo}(\text{CO})(\eta^3\text{-C}_6\text{H}_8)]^+ \text{PF}_6^-$.

Introduction

Cationic molybdenum dienyl complexes are pertinent substrates for organic syntheses [1]. Electrophilic reactions of these complexes have been reported to show that the nucleophiles added to the diene from the non-metal side with high stereoselectivity [2]. Their application to the natural product syntheses has also been reported by Pearson et al. [3]. By using chiral allylic halide, derived from D-arabinose, Liebeskind [4] achieved the enantiomeric synthesis of cationic molybdenum dienyl complex.

We are interested in investigating amino group coordinated transition metal complexes, especially those containing cyclopentadienyl and amino bidentate complexes. They have been demonstrated in the syntheses of manganese dicarbonyl and molybdenum iodide complexes [5,6]. The cationic molybdenum dienyl complexes of this type are particularly interesting because the electrophilicity may be weakened as a result of amine coordination. The degree of weakening would be responsible for the latter nucleophilic addition. Also, the metal centre is now chiral. Reactions derived from these complexes may provide a model for studying

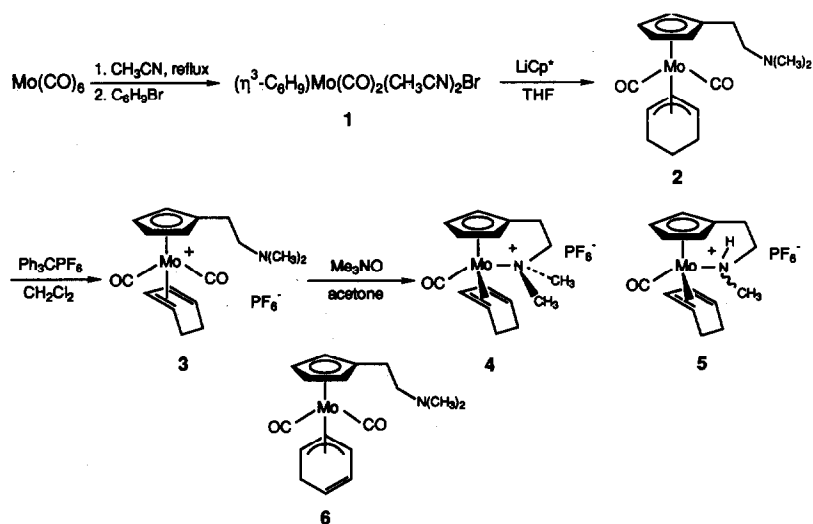
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diastereoselectivity. Here, we report the syntheses and structural studies of such complexes.

Results and discussion

Substituted cyclopentadienyl complex **2** was prepared in 78% yield by adding lithium(dimethylaminoethyl)cyclopentadienylide [6] to a THF solution of $(\eta^3\text{-C}_6\text{H}_9)\text{Mo(CO)}_2(\text{CH}_3\text{CN})_2\text{Br}$ [2,7] (see Scheme 1). Complex **2** revealed similar spectroscopic properties to that of the unsubstituted $(\eta^5\text{-C}_5\text{H}_5)\text{Mo(CO)}_2(\eta^3\text{-C}_6\text{H}_9)$ [2], except for the dimethylaminoethyl side chain. Attempts to effect intramolecular ligand substitution by irradiation, heating or trimethylamine *N*-oxide treatment, were unsuccessful. In realizing that the low carbonyl stretching frequency of **2** at 1929 and 1844 cm^{-1} led to the difficulty with the dissociative substitution reaction [8], we turned to transforming **2** to a more susceptible cyclohexadienyl cationic complex. Treatment of **2** with triphenylcarbenium hexafluorophosphate provided cyclohexadienyl complex **3** as a yellow solid. Now, the carbonyl stretching frequency appeared at 2016 and 1957 cm^{-1} . This made CO removal easier. Treatment of **3** with trimethylamine *N*-oxide effected carbon monoxide removal with concomitant intramolecular amino group coordination to give complex **4** in 58% yield. A small amount of deprotonation product **6** was found in the reaction mixture. Trimethylamine, which was generated during the reaction, may be responsible for the deprotonation. This could be supported by the analogous reaction of treating **3** with triethylamine. In this operation, only complex **6** was isolated.

Upon intramolecular amino group coordination, molybdenum became a chiral centre. The ^1H NMR spectrum showed diastereotopic *N,N*-dimethyl resonances at δ 2.74 (s) and δ 2.38 (s) compared to the solitary methyl singlet resonance of complex **2** at δ 2.16 (s). Four cyclopentadienyl protons were also revealed as non-equivalent resonances at δ 6.59–6.55 (m), 5.87 (br, s), 5.55–5.48 (m), and δ



Scheme 1.

Table 1

Crystal data and refinement details for complex 4

Formula	$C_{16}H_{22}NOMoPF_6$	Transmission factors (max, min)	1.000, 0.975
F.W.	485.26	2θ (max)	44.8°
Crystal system	Monoclinic	Octants	h, k, l -11-11, 0-15, 0-12
Space group	$P2_1/n$	No. of data collected	2556
a (Å)	10.4867(14)	No. of unique data	2404; 2070 with $l > 2.0\sigma(l)$
b (Å)	14.843(3)		
c (Å)	11.882(3)	K	0.000100
V (Å ³)	1844.8(6)	R, R_w	0.036, 0.037
Z	4		
D_c (g cm ⁻³)	1.747	GOF	2.07
$F(000)$	975.73	Maximum shift/ σ ratio	0.600
Crystal dimension (mm ³)	0.40 × 0.34 × 0.30	ρ_{max} (e Å ⁻³)	0.400

4.58–4.54 (m). This phenomenon is similar to that of the molybdenum iodide complex reported previously [6].

A single crystal of **4** was grown in a 1:1 solution of dichloromethane and diethyl ether at -20°C . A monoclinic orange crystal was subjected to an X-ray diffraction study (Tables 1, 2). Exo-conformation of cyclohexadienyl group was realized (see Fig. 1). The bond length of C(12)–C(13) (1.386 Å) is slightly shorter (0.02 Å) than that of C(11)–C(12) and C(13)–C(14). The bond length of Mo–N (2.375 Å) is similar to that of the molybdenum iodide complex (2.38 Å). A linear geometry of molybdenum–carbonyl is consistent with the bond angle of Mo–C(1)–O(1) (172.8°). The carbonyl group resides on the top of the cyclohexadienyl group and bisects the cyclohexadienyl ring, as can be seen from the equal bond angles of C(1)–Mo–C(11), C(1)–Mo–C(14) and C(1)–Mo–C(12), C(1)–Mo–C(13) (see Fig. 1 and Table 3). The N–Mo bond is perpendicular to the Mo–C(1) (92°) and Mo–C(13) (91.98°) bonds. The torsion angle of 172.6° of C(7)–C(2)–C(3)–C(4), suggests that the C(7)–C(2) bond is bent 7.4° away from the cyclopentadienyl plane. The cyclohexadienyl ring is a boat conformation with two planes of C(11)–C(12)–C(13)–C(14) and C(14)–C(15)–C(16)–C(11) in an angle of about 42° (see Table 4).

In addition to **4**, a substantial amount of another bidentate complex was isolated ($\sim 10\%$). The ^1H NMR spectrum revealed only one methyl doublet at δ 2.47 (3H, d, $J = 6.2$ Hz). The four cyclopentadienyl protons also showed as non-equivalent resonances at δ 6.63–6.59 (1H, m); 5.91–5.88 (1H, m), 5.25–5.21 (1H, m); 5.04–4.99 (1H, m). Elemental analysis showed that this complex contained one methylene less than that of **4**. Therefore, the new bidentate complex was tentatively assigned as **5**. The relative stereochemistry of the methyl group is undefined. The mechanism of demethylation is not yet clear. However, it may occur before intramolecular complexation, because complex **4** remained unchanged during treatment with various Lewis acids or bases.

Reactions of **4** with various electrophiles (LiBEt_3H , MeMgBr , CH_2CHMgBr) gave complex mixtures. When **4** was treated with benzylmagnesium bromide, a

Table 2

Atomic coordinates and B_{iso}

Atom	x	y	z	B_{iso}
Mo	0.16599(3)	0.18344(2)	0.19908(3)	2.43(2)
P	0.78417(13)	0.50542(9)	0.28162(10)	3.72(6)
N	0.12228(4)	0.2367(3)	0.3810(3)	3.6(2)
O(1)	0.3851(4)	0.3198(3)	0.1718(4)	6.7(2)
F(1)	0.8258(4)	0.6054(2)	0.3083(3)	8.9(2)
F(2)	0.6836(3)	0.5404(3)	0.1876(2)	7.3(2)
F(3)	0.8872(3)	0.5000(2)	0.1904(3)	6.5(2)
F(4)	0.8826(4)	0.4700(3)	0.3763(3)	8.8(2)
F(5)	0.7445(4)	0.4055(2)	0.2551(3)	8.5(2)
F(6)	0.6805(3)	0.5125(3)	0.3725(3)	7.6(2)
C(1)	0.2998(5)	0.2740(3)	0.1847(4)	4.0(2)
C(2)	0.2062(5)	0.0639(3)	0.3262(4)	3.9(2)
C(3)	0.1269(6)	0.0295(3)	0.2370(5)	4.3(3)
C(4)	0.1944(6)	0.0366(3)	0.1396(5)	4.5(3)
C(5)	0.3134(6)	0.0748(4)	0.1662(5)	4.9(3)
C(6)	0.3212(5)	0.0919(4)	0.2832(5)	4.6(3)
C(7)	0.1698(9)	0.0799(5)	0.4437(5)	6.1(4)
C(8)	0.1914(7)	0.1788(5)	0.4701(5)	5.9(3)
C(9)	0.1696(8)	0.3311(5)	0.4063(6)	5.7(3)
C(10)	-0.0146(6)	0.2351(5)	0.3997(5)	5.2(3)
C(11)	0.1400(6)	0.2086(4)	0.0018(4)	4.6(3)
C(12)	0.0286(6)	0.1745(4)	0.0457(5)	4.5(3)
C(13)	-0.0272(5)	0.2268(4)	0.1254(4)	3.8(2)
C(14)	0.0378(5)	0.3092(3)	0.1495(4)	3.8(2)
C(15)	0.0882(6)	0.3646(4)	0.0553(5)	4.3(3)
C(16)	0.1463(7)	0.3057(4)	-0.0310(5)	5.1(3)

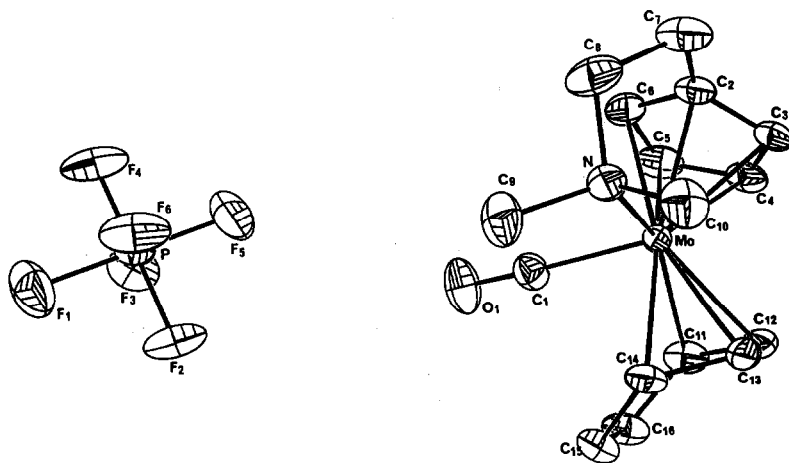


Fig. 1. The ORTEP drawing of complex 4.

Table 3

Selected bond lengths (Å) and bond angles (°) in complex 4

Mo–N	2.375(4)	N–C(8)	1.505(7)
Mo–C(1)	1.960(5)	N–C(9)	1.508(7)
Mo–C(2)	2.349(4)	N–C(10)	1.474(7)
Mo–C(3)	2.371(5)	O(1)–C(1)	1.142(6)
Mo–C(4)	2.317(5)	C(11)–C(12)	1.407(9)
Mo–C(5)	2.287(5)	C(11)–C(16)	1.495(8)
Mo–C(6)	2.294(5)	C(12)–C(13)	1.386(8)
Mo–C(11)	2.371(5)	C(13)–C(14)	1.419(7)
Mo–C(12)	2.245(5)	C(14)–C(15)	1.514(7)
Mo–C(13)	2.244(5)	C(15)–C(16)	1.509(8)
Mo–C(14)	2.351(5)		
N–Mo–C(1)	92.00(17)	C(2)–C(7)–C(8)	107.6(5)
N–Mo–C(2)	72.73(15)	N–C(8)–C(7)	110.5(5)
N–Mo–C(3)	95.77(17)	Mo–N–C(8)	109.7(3)
N–Mo–C(6)	88.60(18)	Mo–C1–O(1)	172.8(4)
N–Mo–C(13)	91.98(17)	Mo–C(2)–C(7)	115.8(4)
C(1)–Mo–C(11)	80.72(21)	C(3)–C(2)–C(7)	126.1(6)
C(1)–Mo–C(12)	113.10(21)	C(6)–C(2)–C(7)	125.2(6)
C(1)–Mo–C(13)	113.67(20)	C(11)–C(12)–C(13)	117.0(5)
C(1)–Mo–C(14)	80.43(19)	C(12)–C(13)–C(14)	113.6(5)

reasonable amount of bibenzyl was obtained. The further scope and limitations of the reactions of 4 are now under investigation.

Experimental

All reactions were performed under argon by Schlenk techniques. Tetrahydrofuran (THF) and *n*-hexane were distilled from sodium benzophenone ketyls; methylene chloride (CH₂Cl₂) was distilled from CaH₂. Infrared solution spectra were recorded on a Perkin–Elmer 882 infrared spectrophotometer using 0.1 mm cells with CaF₂ windows. Melting points were determined by using a Yanaco model MP micro melting point apparatus and are uncorrected. ¹H NMR (200 MHz) and ¹³C NMR (50 MHz) were obtained with a Bruker AC-200 FT spectrophotometer. All chemical shifts are reported in parts per million (ppm) relative

Table 4

Selected torsion angles (°) in complex 4

C(9)–N–C(8)–C(7)	–170.8(7)	C(6)–Mo–C(12)–C(13)	149.8(4)
C(7)–C(2)–C(3)–C(4)	172.6(7)	C(6)–C(2)–C(3)–C(4)	0.1(3)
C(3)–C(2)–C(6)–C(5)	0.0(3)	C(2)–C(3)–C(4)–C(5)	–0.1(3)
C(7)–C(2)–C(6)–C(5)	–172.6(7)	C(3)–C(4)–C(5)–C(6)	0.1(3)
C(2)–C(7)–C(8)–N	53.4(4)	C(4)–C(5)–C(6)–C(2)	–0.1(3)
C(16)–C(11)–C(12)–C(13)	–43.7(4)	C(12)–C(11)–C(16)–C(15)	41.8(4)
C(13)–C(14)–C(15)–C(16)	–40.3(4)	C(11)–C(12)–C(13)–C(14)	0.8(3)
C(12)–Mo–C(5)–C(6)	–159.0(5)	C(12)–C(13)–C(14)–C(15)	41.6(4)
C(13)–Mo–C(5)–C(6)	–147.9(5)	C(14)–C(15)–C(16)–C(11)	–1.0(3)
C(5)–Mo–C(12)–C(13)	167.2(4)		

to Me₄Si. Elemental analyses were obtained on a Perkin–Elmer 2400 CHN elemental analyzer. High resolution and normal mass spectra were recorded on a VG 70-250S mass spectrophotometer.

Preparation of $\eta^5\text{-C}_5\text{H}_4\text{CH}_2\text{CH}_2\text{N}(\text{CH}_3)_2\text{Mo}(\text{CO})_2(\eta^3\text{-C}_6\text{H}_9)$ (2)

Molybdenum carbonyl (17.16 g, 65 mmol) was suspended in CH₃CN (150 ml), and the suspension was heated under reflux for 4 h. After the formation of the Mo(CO)₃(CH₃CN)₃, the heating source was removed and 3-bromocyclohexene [9] (10.5 g, 7.5 ml, 65 mmol) was added dropwise to the hot solution over 10 min. The mixture was stirred for 1 h. ($\eta^3\text{-C}_6\text{H}_9$)Mo(CO)₂(CH₃CN)₂Br was formed as a yellow precipitate. The solvent was removed under a vacuum and then THF (100 ml) was added.

A THF solution of lithium(dimethylamino)cyclopentadienylide (136 ml, 0.5 M) was added to the above suspension at room temperature over 30 min. After stirring for 2 h, the reaction mixture was filtered. The filtrate was concentrated and then flash chromatographed [10] on silica gel 60 (230–400 mesh, 5% CH₃OH in CH₂Cl₂). The yellow band was collected. After evaporation to dryness, a mixture of liquid and solids was obtained. The solid material was found to be the corresponding ammonium salt, generated due to the acidity of the silica gel. The mixture was dissolved with CH₂Cl₂ (400 ml) and washed once with aqueous sodium carbonate. After evaporation, 2 was obtained as an orange liquid, 18.86 g (78% yield). TLC (silica gel): $R_f = 0.24$ (5% CH₃OH in CH₂Cl₂). IR (CH₂Cl₂): 1929s, 1844s cm⁻¹. ¹H NMR (C₃D₆O, 200 MHz): δ 5.35 (2H, t, $J = 2.2$ Hz, Cp–H's); 5.27 (2H, t, $J = 2.2$ Hz, Cp–H's); 4.12 (1H, t, $J = 7$ Hz, H₂), 3.56–3.52 (2H, m, H₁ + H₃); 2.38–2.34 (4H, m); 2.16 (6H, s); 1.98–1.80 (2H, m, H_{4 β} + H_{6 β}); 1.66–1.52 (2H, m, H_{4 α} + H_{6 α}); 0.98–0.83 (1H, m, H_{5 β}); 0.42–0.15 (1H, m, H_{5 α}). ¹H NMR (CDCl₃, 200 MHz): δ 5.18 (4H, s, Cp–H's); 3.90 (1H, t, $J = 7$ Hz, H₂); 3.55–3.50 (2H, m, H₁ + H₃); 2.44–2.30 (4H, m); 2.25 (6H, s); 2.00–1.82 (2H, m, H_{4 β} + H_{6 β}); 1.72–1.60 (2H, m, H_{4 α} + H_{6 α}); 1.05–0.90 (1H, m, H_{5 β}); 0.46–0.20 (1H, m, H_{5 α}). ¹³C NMR (CDCl₃, 50 MHz): δ 235.9 (CO \times 2); 112.0 (C, Cp); 92.6 (CH \times 2, Cp), 89.7 (CH \times 2, Cp); 60.6 (CH₂); 59.1 (CH, C₂); 56.2 (CH \times 2, C₁ and C₃); 45.3 (CH₃ \times 2); 26.5 (CH₂); 21.4 (CH₂ \times 2, C₄ and C₆); 18.8 (CH₂, C₅). Mass spectra at 18 eV, m/e (rel. intensity (%)): 371 (30, M⁺), 343 (6, M⁺ – CO), 315 (85, M⁺ – 2CO), 313 (100, M⁺ – CH₂N(CH₃)₂), 290 (17, M⁺ – C₆H₉). High resolution mass. Found: 371.0792. C₁₇H₂₃NO₂Mo calc.: 371.0783.

Preparation of $[\eta^5\text{-C}_5\text{H}_4\text{CH}_2\text{CH}_2\overline{\text{N}(\text{CH}_3)_2\text{Mo}(\text{CO})(\eta^3\text{-C}_6\text{H}_9)}]^+\text{PF}_6^-$ (4) and $[(\eta^5\text{-C}_5\text{H}_4\text{CH}_2\text{CH}_2\overline{\text{NH}(\text{CH}_3)_2\text{Mo}(\text{CO})(\eta^3\text{-C}_6\text{H}_9)})]^+\text{PF}_6^-$ (5)

A powder of triphenylcarbenium hexafluorophosphate (2.91 g, 7.5 mmol) was added portionwise to a stirred orange solution of 2 (1.845 g, 5 mmol) in CH₂Cl₂ (30 ml) at 0°C over 10 min. The mixture was then stirred at room temperature for 14 h. The desired product was formed as a straw precipitate. Et₂O (30 ml) was added. Solids were collected and washed twice with Et₂O (30 ml \times 2). After evaporation to dryness, 3 was obtained as a straw powder. IR (acetone): 2016s, 1957s cm⁻¹. ¹H NMR (C₃D₆O, 200 MHz): δ 6.15–6.10 (2H, m); 6.05–6.00 (4H, m, Cp–H's); 5.12–5.05 (2H, m); 3.70–3.55 (4H, m); 3.17 (6H, s); 2.38–2.28 (2H, m); 2.13–2.03 (2H, m).

Straw powder of **3** was dissolved in acetone (50 ml), and then the solution was cooled in an ice-water bath. Crystals of anhydrous Me_3NO [11] (412 mg, 5.5 mmol) were added in one portion to the above stirred yellow solution. After stirring for 10 min, the infrared spectrum indicated complete disappearance of **3**. Acetone was removed under reduced pressure. The residue was subjected to flash chromatography (silica gel 60, 230–400 mesh, 40% acetone in EtOAc at the beginning followed by 5% MeOH in CH_2Cl_2). Compounds **4** and **5** were obtained.

Compound 4: 1.41 g (58% yield). TLC (silica gel): $R_f = 0.29$ (5% CH_3OH in CH_2Cl_2). Orange crystals, m.p. 150°C (dec). IR (CH_2Cl_2): 1955 cm^{-1} . $^1\text{H NMR}$ ($\text{C}_3\text{D}_6\text{O}$, 200 MHz): δ 6.59–6.55 (1H, m, Cp–H); 5.99 (1H, br.s, H_{12} or H_{13}); 5.87 (1H, br.s, Cp–H); 5.55–5.48 (2H, m, Cp–H and H_{11} or H_{14}); 4.58–4.54 (1H, m, Cp–H); 3.92–3.77 (1H, m, H_{8a}); 3.72–3.65 (1H, m, H_{13} or H_{12}); 2.91–2.81 (1H, m, H_{8b}); 2.74 (3H, s); 2.68–2.27 (5H, m); 2.38 (3H, s); 2.24–2.12 (2H, m). $^{13}\text{C NMR}$ ($\text{C}_3\text{D}_6\text{O}$, 50 MHz): δ 225.8 (CO); 137.0 (C, Cp); 98.3 (CH, Cp); 91.2 (CH, Cp); 88.6 (CH, C_{12} or C_{13}); 85.7 (CH, Cp); 81.7 (CH \times 2, Cp and C_{11} or C_{14}); 80.2 (CH, C_{13} or C_{12}); 77.2 (CH_2 , C_8); 66.0 (CH, C_{14} or C_{11}); 59.2 (CH_3); 53.6 (CH_3); 26.6 (CH_2); 26.2 (CH_2); 24.8 (CH_2). Anal. Found: C, 39.47; H, 4.52; N, 2.72. $\text{C}_{16}\text{H}_{22}\text{NOMoPF}_6$ calc.: C, 39.60; H, 4.57; N, 2.89%.

Compound 5: 0.135 g (6% yield). TLC (silica gel): $R_f = 0.25$ (5% CH_3OH in CH_2Cl_2). Yellow crystals, m.p. 195°C (dec). IR (acetone): 1954 cm^{-1} . $^1\text{H NMR}$ ($\text{C}_3\text{D}_6\text{O}$, 200 MHz): δ 6.63–6.59 (1H, m, Cp–H); 5.91–5.88 (1H, m, Cp–H); 5.64–5.60 (1H, m); 5.52–5.45 (1H, m); 5.25–5.21 (1H, m, Cp–H); 5.04–4.99 (1H, m, Cp–H); 5.55–4.90 (1H, br); 3.94–3.78 (1H, m); 3.52–3.46 (1H, m); 3.24–3.15 (1H, m); 2.87–2.33 (5H, m); 2.47 (3H, d, $J = 6.2$ Hz); 2.24–2.15 (2H, m). $^{13}\text{C NMR}$ ($\text{C}_3\text{D}_6\text{O}$, 50 MHz): δ 224.8 (CO); 137.4 (C); 98.1 (CH); 90.1 (CH); 89.8 (CH); 89.1 (CH); 84.0 (CH); 80.5 (CH); 77.0 (CH); 69.5 (CH_2); 64.1 (CH); 44.4 (CH_3); 26.5 (CH_2); 25.5 (CH_2); 24.8 (CH_2). Anal. Found: C, 38.38; H, 4.20; N, 3.15. $\text{C}_{15}\text{H}_{20}\text{NOMoPF}_6$ calc.: C, 38.23; H, 4.28; N, 2.97%.

*Crystal structure of $[\eta^5\text{-C}_5\text{H}_4\text{CH}_2\text{CH}_2\overline{\text{N}(\text{CH}_3)_2}\text{Mo}(\text{CO})(\eta^3\text{-C}_6\text{H}_8)]^+\text{PF}_6^-$ (**4**)*

A single crystal of **4a** was grown in a 1:1 solution of dichloromethane and diethyl ether at -20°C . Diffraction measurements were made on an Enraf–Nonius CAD-4 diffractometer by use of graphite-monochromated Mo-K_α radiation ($\lambda = 0.7093\text{ \AA}$) in the θ – 2θ scan mode. Unit cell dimensions were obtained by least-squares refinement with use of 25 centred reflections for which $14.43^\circ < 2\theta < 33.03^\circ$. Other crystal data and refinement details are listed in Table 1.

Supplementary material available. Lists of crystal data and refinement details (1 page), atomic coordinates and B_{iso} (1 page), bond lengths and angles (2 pages) and torsion angles of **4** are available from T.-F.W.

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