



Reactions of substituted tetramethylcyclopentadienes with molybdenum hexacarbonyl

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ABSTRACT

The reactions of the substituted tetramethylcyclopentadienes [C₅Me₄HR] [R = ethyl (**1**), *n*-propyl (**2**), *i*-propyl (**3**), cyclopentyl (**4**), cyclohexyl (**5**), 4-NMe₂Ph (**6**)] with Mo(CO)₆ in refluxing xylene gave the corresponding dinuclear metal carbonyl complexes [η⁵-C₅Me₄R]₂Mo₂(CO)₆ (**7–11**), while similar treatment of [C₅Me₄H(4-NMe₂Ph)] (**6**) with Mo(CO)₆ afforded an unexpected product [(η⁵-C₅Me₄(4-NMe₂-Ph))MoO₂]₂(μ-O) (**12**). All the new complexes were fully characterized. The molecular structures of **7**, **8**, **11** and **12** have been determined by X-ray single crystal diffraction.

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1. Introduction

The chemistry of metallocene metal complexes has been receiving much attention, one of considerable research is currently being focused on the synthesis and study of metal–metal bonded transition metal complexes, owing to their important role in many catalytic processes [1–4]. Cyclopentadienyls have been among the most important ligands in organo-transition metal chemistry, as they from a wide range of derivatives whose steric and electronic properties can be easily tailored by varying the ring substituents. These variations on the cyclopentadienyl unit not only open access to construct new compounds, but also have great influence on the metallocene catalytic activity [5–8]. To obtain a deeper insight into the steric and electric effects of substituents on the molecular structures and reactions of the corresponding biscyclopentadienyl binuclear metal carbonyl complexes, we have prepared a series of biscyclopentadienyl dimolybdenum complexes and determined their structures.

2. Experimental

2.1. General considerations

All manipulations were performed at an argon/vacuum manifold using standard Schlenk techniques. Solvents were dried by known procedures and used fresh distilled under nitrogen prior

to use. ¹H NMR spectra were recorded on a Bruker AV 500 instrument, while IR spectra were recorded as KBr disks on a FT IR 8900 spectrometer. X-ray measurements were made on a Bruker Smart APEX diffractometer with graphite monochromated Mo Kα (λ = 0.071073 nm) radiation. Elemental analyses were performed on a Vario EL III analyzer. The ligands [C₅Me₄HR] [R = ethyl (**1**), *n*-propyl (**2**), *i*-propyl (**3**), cyclopentyl (**4**), cyclohexyl (**5**), 4-NMe₂Ph (**6**)] were prepared as described in the literature [9,10].

2.2. Synthesis of complex [η⁵-C₅Me₄(CH₂CH₃)₂Mo₂(CO)₆ (**7**)

A solution of ligand C₅Me₄HCH₂CH₃ (**1**) (0.300 g, 2 mmol) and Mo(CO)₆ (0.528 g, 2 mmol) in 30 mL of xylene was refluxed for 12 h. After removal of solvent the residue was chromatographed on an alumina column. Eluent with petroleum ether/CH₂Cl₂ gave a dark-red band that afforded red crystals, yield: 0.373 g (56.6%), m.p. 169 °C (dec.). Anal. Calc. for C₂₈H₃₄Mo₂O₆: C, 51.08; H, 5.20. Found: C, 50.22; H, 5.13%. ¹H NMR (500 MHz, CDCl₃): δ 1.75(s, 6H, CH₃), 1.86(d, 12H, J = 4 Hz, C₅Me₄), 1.94(s, 4H, CH₂), 3.00(d, 12H, C₅Me₄). IR (ν_{CO}, cm^{−1}): 1919(s), 1890(m), 1865(s).

2.3. Synthesis of complex [η⁵-C₅Me₄(CH₂CH₂CH₃)₂Mo₂(CO)₆ (**8**)

Using a procedure similar to that described above, reaction of ligand C₅Me₄HCH₂CH₂CH₃ (**2**) with Mo(CO)₆ in refluxing xylene afforded complex **8** as red crystals in 57.8% yield. m.p. 175 °C (dec.). Anal. Calc. for C₃₀H₃₈Mo₂O₆: C, 52.49; H, 5.58. Found: C, 52.28; H, 5.66%. ¹H NMR (CDCl₃): δ 2.26 (t, J = 8.0 Hz, 4H, C₅Me₄-CH₂), 1.92–1.98 (m, 24H, C₅Me₄), 1.34–1.50 (m, 4H, CH₂), 0.91(t,

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$J = 7.5$ Hz, 6H, CH₃). IR (ν_{CO} , cm⁻¹): 1906(s), 1927(s), 1868(s), 1896(s).

2.4. Synthesis of complex $[\eta^5\text{-C}_5\text{Me}_4\text{CH}(\text{CH}_3)_2]_2\text{Mo}_2(\text{CO})_6$ (**9**)

Using a procedure similar to that described above, reaction of ligand $\text{C}_5\text{Me}_4\text{HCH}(\text{CH}_3)_2$ (**3**) with $\text{Mo}(\text{CO})_6$ in refluxing xylene afforded complex **9** as red crystals in 30.4% yield. m.p. 121–122 °C. *Anal.* Calc. for $\text{C}_{30}\text{H}_{38}\text{Mo}_2\text{O}_6$: C, 52.49; H, 5.58. Found: C, 52.60; H, 5.49%. ^1H NMR (CDCl_3): δ 2.03–2.18 (m, 2H, CH), 1.93–1.97 (m, 24H, C_5Me_4), 1.30 (d, $J = 7.0$ Hz, 12H, CH₃). IR (ν_{CO} , cm⁻¹): 1927(s), 1863(s), 1830(s).

2.5. Synthesis of complex $[\eta^5\text{-C}_5\text{Me}_4\text{CH}(\text{CH}_2)_4]_2\text{Mo}_2(\text{CO})_6$ (**10**)

Using a procedure similar to that described above, reaction of ligand $\text{C}_5\text{Me}_4\text{HCH}(\text{CH}_2)_4$ (**4**) with $\text{Mo}(\text{CO})_6$ in refluxing xylene afforded complex **10** as red crystals in 43.1% yield. m.p. 182–183 °C. *Anal.* Calc. for $\text{C}_{34}\text{H}_{42}\text{Mo}_2\text{O}_6$: C, 55.29; H, 5.73. Found: C, 55.41; H, 5.68%. ^1H NMR (CDCl_3): δ 2.11–2.23 (m, 2H, $\text{C}_5\text{Me}_4\text{CH}$), 1.92–1.97 (m, 24H, C_5Me_4), 1.67–1.72 (m, 16H, $(\text{CH}_2)_4$). IR (ν_{CO} , cm⁻¹): 1919(s), 1905(s), 1890(s).

2.6. Synthesis of complex $[\eta^5\text{-C}_5\text{Me}_4\text{CH}(\text{CH}_2)_5]_2\text{Mo}_2(\text{CO})_6$ (**11**)

Using a procedure similar to that described above, reaction of ligand $\text{C}_5\text{Me}_4\text{HCH}(\text{CH}_2)_5$ (**5**) with $\text{Mo}(\text{CO})_6$ in refluxing xylene afforded complex **11** as red crystals in 84% yield. m.p. 158 °C (dec.). *Anal.* Calc. for $\text{C}_{36}\text{H}_{46}\text{Mo}_2\text{O}_6$: C, 56.40; H, 6.05. Found: C, 56.29; H, 5.89. ^1H NMR (CDCl_3): δ 2.39 (t, $J = 11.5$ Hz, 2H, CH), 1.84–2.14 (m, 24H, C_5Me_4), 1.20–1.54 (m, 20H, $(\text{CH}_2)_5$). IR (ν_{CO} , cm⁻¹): 1935(m), 1921(m), 1898(s), 1888(m), 1869(s), 1830(m).

2.7. Synthesis of complex $[\eta^5\text{-C}_5\text{Me}_4(4\text{-NMe}_2\text{Ph})\text{MoO}_2]_2(\mu\text{-O})$ (**12**)

Using a procedure similar to that described above, reaction of ligand $[\text{C}_5\text{Me}_4\text{H} 4\text{-NMe}_2\text{Ph}]$ (**6**) with $\text{Mo}(\text{CO})_6$ in refluxing xylene afforded complex **12** as orange crystals in 60.2% yield. m.p. 165(dec.) °C. *Anal.* Calc. for $\text{C}_{34}\text{H}_{44}\text{Mo}_2\text{N}_2\text{O}_5$: C, 54.26; H, 5.89. Found: C, 53.96; H, 5.78%. ^1H NMR (500 MHz, CDCl_3): δ 2.09(s, 24H, C_5Me_4), 2.97(s, 12H, N CH₃), 6.72(d, 4H, $J = 8$ Hz, C_6H_4), 7.19(d, 4H, $J = 8$ Hz, C_6H_4). IR ($\text{Mo}=\text{O}$, $\text{Mo}=\text{O}$, $\text{Mo}-\text{O}-\text{Mo}$ cm⁻¹): 910(s), 878(s), 758(s).

2.8. Crystallographic studies

Single crystals of complexes **7**, **8**, **11** and **12** suitable for X-ray diffraction were obtained from the slow evaporation of hexane-dichloromethane solution. All X-ray crystallographic data were collected on a Bruker Smart APEX diffractometer with graphite monochromated Mo $K\alpha$ ($\lambda = 0.71073$ Å) radiation using the ϕ/ω scan technique. The structures were solved by direct methods and refined by full-matrix least-squares procedures based on F^2 using the SHELXL-97 program system. The crystal data and summary of X-ray data collection are presented in Table 1.

3. Results and discussion

3.1. Synthesis of **7–12**

Treatment of the compounds $[\text{C}_5\text{Me}_4\text{HR}]$ [$\text{R} = \text{ethyl}$ (**1**), n -propyl (**2**), i -propyl (**3**), cyclopentyl (**4**), cyclohexyl (**5**)] with $\text{Mo}(\text{CO})_6$ under refluxing xylene for 12 h afforded the normal Mo–Mo single bonded dinuclear complexes **7–11** (Scheme 1).

The IR spectra of complexes **7–11** showed similar patterns and characteristic absorption peaks for strong CO in the terminal $\nu(\text{CO})$ region. The ^1H NMR spectra of complex **7** showed two doublet peaks for the four methyls protons while the ^1H NMR spectra of complexes **8–11** showed one multiple peak for the four methyls protons, respectively.

However, when ligand $[\text{C}_5\text{Me}_4\text{HR}]$ [$\text{R} = 4\text{-NMe}_2\text{Ph}$ (**6**)] reacted with $\text{Mo}(\text{CO})_6$ under refluxing xylene for 12 h, the unexpected dinuclear oxo-bridging complex **12** was obtained.

The IR of **12** showed three characteristic peaks at 910, 878 and 758 cm⁻¹, corresponding to the two terminal Mo=O stretching modes and a bridging antisymmetric Mo–O–Mo stretching mode, respectively. The ^1H NMR spectrum of complex **12** showed one singlet peak for the four methyls protons, two group doublet peaks for the phenyl protons and one singlet peak for the N-methyls protons.

Complex **12** is an unexpected product and the structure is unusual. Because our team has never observed similar reaction of substituted tetramethylcyclopentadienes with metal carbonyl complex, we are puzzled by this reaction. After consulting a large number of literatures [11–13], since the mechanistic is unconfirmed, we suggest the following mechanistic pathway shown in Scheme 2. when ligand **6** reacted with $\text{Mo}(\text{CO})_6$ under refluxing xylene for 12 h, the product $[\eta^5\text{-C}_5\text{Me}_4(4\text{-NMe}_2\text{Ph})]_2\text{Mo}_2(\text{CO})_4$ ($\text{Mo}\equiv\text{Mo}$) was firstly obtained, subsequent treatment of this dimer with petroleum ether/ CH_2Cl_2 as eluent exposed to the air, the reaction was occur by oxidation of the carbonyl complex $[\eta^5\text{-C}_5\text{Me}_4(4\text{-NMe}_2\text{Ph})]_2\text{Mo}_2(\text{CO})_4$ ($\text{Mo}\equiv\text{Mo}$) with O_2 in CH_2Cl_2 , then carbonyl loss to give a single oxygen bridged dimer $[\eta^5\text{-C}_5\text{Me}_4(4\text{-NMe}_2\text{Ph})\text{MoO}_2]_2(\mu\text{-O})$. This reaction illustrated that the $\text{Mo}\equiv\text{Mo}$ triple bonded complex is not stable, very easy to react with O_2 in the air.

3.2. Crystal structures

The selected bond parameters for complexes **7**, **8**, **11** and **12** are presented in Table 2 and their molecular structures are depicted in Figs. 1–4.

The structures of complexes **7**, **8**, and **11** are shown in Figs. 1–3, all the complexes consist of two $(\text{C}_5\text{Me}_4\text{R})\text{Mo}(\text{CO})_3$ units, each of the molybdenum atoms is coordinated with an η^5 -cyclopentadienyl and three terminal CO ligands. All the complexes disposed in the *trans* conformation and linked by a metal–metal bond, these complexes lying on a crystallographic inversion. Two independent but chemically equivalent molecules appear in the unit cell. The fifth coordination position is occupied by a cyclopentadienyl ring that is essentially planar. For complex **7**, disorder on the location of Mo has been shown in Fig. 1, the Mo1–Mo1i distance is 0.3272 nm, the Mo1'–Mo1'i distance is 0.2504 nm. The Mo1–CEN distance is 0.2024 nm. The disorder share of Mo1 is 0.929 and Mo1' is 0.071. For complex **11**, the conformation of the cyclohexyl ring has shown a very steady chair form. The structural parameters of complexes **7**, **8** and **11** are identical to those in $[\eta^5\text{-C}_5\text{H}_4\text{-R}\text{Mo}(\text{CO})_3]_2$, Mo–Mo bond distances for the complexes **7**, **8**, and **11** are 0.3273, 0.3305 and 0.3291 nm, respectively, which are comparable to other metal–metal bond distances found in this kind of $[\eta^5\text{-C}_5\text{H}_4\text{R}\text{Mo}(\text{CO})_3]_2$ [$\text{R} = \text{Ph-Me}$, (0.3283 nm); $\text{R} = \text{Ph-Ome}$ (0.3307 nm) [14]; $\text{R} = \text{benzyl}$ (0.3266 nm [15]; $\text{R} = n\text{-butyl}$ (0.3286 nm) [16]]. However, Mo–Mo bond distances for the complexes **7**, **8**, and **11** are longer than that reported for *trans*- $[\eta^5\text{-C}_5\text{H}_5\text{-Mo}(\text{CO})_3]_2$ [0.3235(1) nm] [17] and $[(\eta^5\text{-C}_5\text{H}_4\text{Pr})\text{Mo}(\text{CO})_3]_2$ [0.3222(5) nm] [18], which indicate that the substituents of the cyclopentadienyl ring have steric hindrance.

The molecular structure for complex **12** is shown in Fig. 4. Complex **12** is a pentaoxo dimer, and crystallizes in the monoclinic space group $P2(1)/c$. Every molecule contains two molybdenum atoms connected with a single oxygen bridge. The two cyclopentadienyl planes are parallel. The complex is centrosymmetric with a

Table 1
Crystal data and structure refinement parameters for complexes **7**, **8**, **11** and **12**.

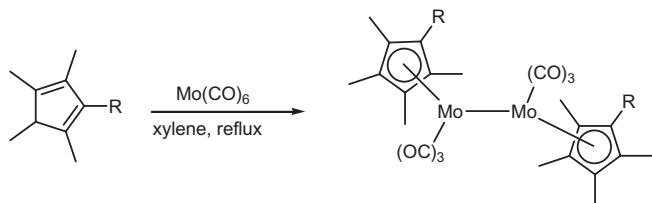
Complex	7	8	11	12
Empirical formula	C ₂₈ H ₃₄ Mo ₂ O ₆	C ₃₀ H ₃₈ Mo ₂ O ₆	C ₃₆ H ₄₆ Mo ₂ O ₆	C ₃₄ H ₄₄ Mo ₂ N ₂ O ₅
Formula weight	658.43	686.48	766.61	650.37
T (K)	298(2)	298(2)	298(2)	298(2)
Crystal system	monoclinic	monoclinic	monoclinic	monoclinic
Space group	P2(1)/n	P2(1)/n	P2(1)/n	P2(1)/c
a (nm)	0.92417(14)	0.89270(17)	0.8954(5)	1.6454(3)
b (nm)	0.90929(14)	0.93258(18)	2.0743(11)	0.78203(16)
c (nm)	1.6505(3)	1.8089(4)	0.9166(5)	1.3233(3)
α (°)	90	90	90	90
β (°)	97.646(2)	90.593(2)	93.175(7)	109.344(3)
γ (°)	90	90	90	90
V (nm ³)	1.3746(4)	1.5058(5)	1.6999(15)	1.6066(6)
Z	2	2	2	2
F(000)	668	700	788	772
D _{calc} (g/cm ³)	1.591	1.514	1.498	1.556
Crystal dimensions (mm)	0.48 × 0.36 × 0.20	0.48 × 0.42 × 0.03	0.26 × 0.21 × 0.20	0.49 × 0.37 × 0.03
θ Range (°)	2.40–25.50	2.25–25.50	2.43–25.50	2.62–25.02
Reflection collected (R _{int})	6822 (0.0208)	7432	8376 (0.0482)	6515 (0.0329)
Independent reflections	2545	2802	3151	2835
R _{int}	0.0208	0.0317	0.0482	0.0329
Parameters	178	177	203	202
Goodness of fit (GOF) on F ²	1.120	1.076	1.090	1.062
R ₁ , wR ₂ [I > 2σ (I)]	R ₁ = 0.0266, wR ₂ = 0.0651	R ₁ = 0.0306, wR ₂ = 0.0811	R ₁ = 0.0558, wR ₂ = 0.1170	R ₁ = 0.0383, wR ₂ = 0.0933
R ₁ , wR ₂ (all data)	R ₁ = 0.0314, wR ₂ = 0.0671	R ₁ = 0.0338, wR ₂ = 0.0852	R ₁ = 0.0736, wR ₂ = 0.1244	R ₁ = 0.0474, wR ₂ = 0.0983
CCDC deposition No.	832372	883602	833999	834001

Complex **7**: $w = 1/[\sigma^2(F_o^2) + (0.0280 p)^2 + 0.7464 p]$, where $p = (F_o^2 + 2F_c^2)/3$.

Complex **8**: $w = 1/[\sigma^2(F_o^2) + (0.0479 p)^2 + 0.3556 p]$, where $p = (F_o^2 + 2F_c^2)/3$.

Complex **11**: $w = 1/[\sigma^2(F_o^2) + (0.0484 p)^2 + 3.4886 p]$, where $p = (F_o^2 + 2F_c^2)/3$.

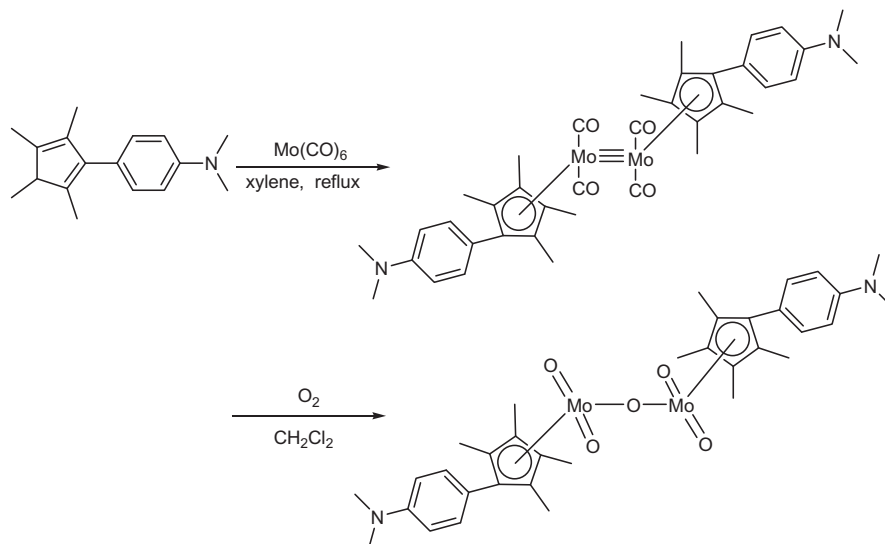
Complex **12**: $w = 1/[\sigma^2(F_o^2) + (0.0550 p)^2 + 0.4486 p]$, where $p = (F_o^2 + 2F_c^2)/3$.



Scheme 1. Synthesis of the complexes **7–11**.

Mo(1)–O(1)–Mo(1i) angle of 180°, the Mo(1)–O(1)–Mo(1i) is a colinear bond at an oxygen atom, the linearity of the Mo(1)–

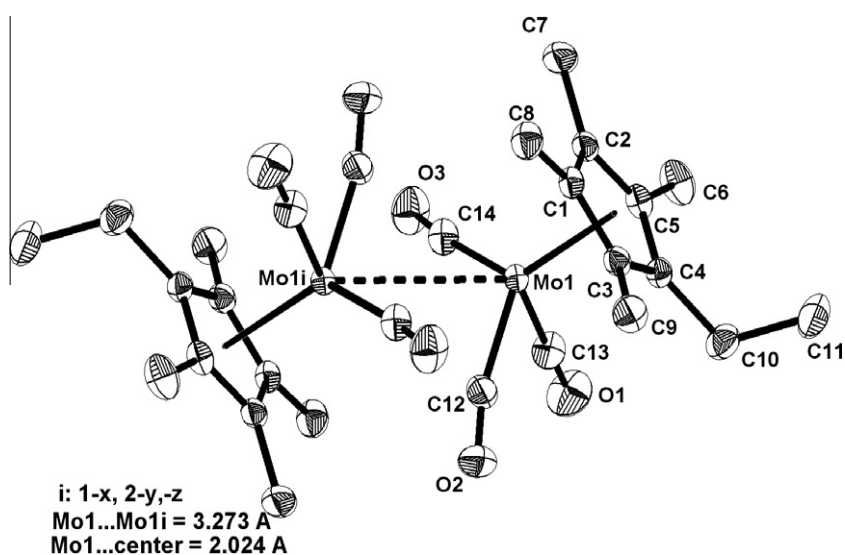
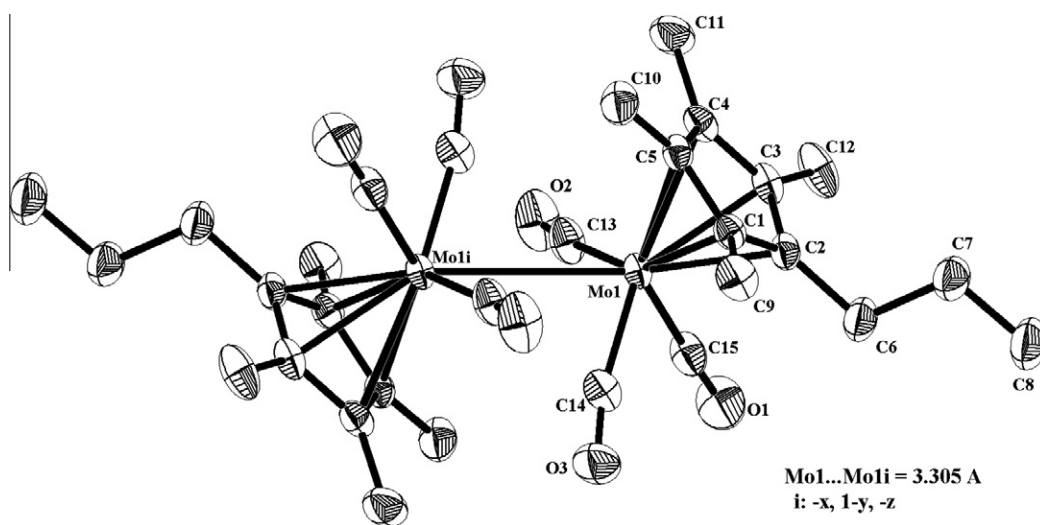
O(1)–Mo(1i) is first observed in our experiments, the linear Mo(1)–O(1)–Mo(1i) bridge bond was attributed to π -bonding after the possible consequences of steric interactions were considered. In fact, a linear, centrosymmetric arrangement in a [(MoO₂)₂O] unit was once previously observed in [(η^5 -C₅Me₅)MoO₂]₂(μ -O) [11]. In complex **12**, the bond distances of Mo(1)–O(1) and Mo(1i)–O(1) are completely equal (0.18836 nm), the terminal Mo=O distances (average 0.1706 nm) are much longer than [(η^5 -C₅Me₅)MoO₂]₂(μ -O) (average 0.167 nm)], the long bond distances of terminal Mo=O bonds have been suggested to decrease O–O repulsion and result in O–Mo–O angles larger than 90°. Further investigation of the reactivity and stability of this complex is in progress.



Scheme 2. Synthesis of the complex **12**.

Table 2Selected bond distances (nm) and angles (°) for complexes **7**, **8**, **11** and **12**.

7		8		11		12	
Mo(1)–Mo(1i)	0.3273	Mo–Mo(1i)	0.3305	Mo–Mo(1i)	0.3291	Mo(1)–C(1)	0.2349(3)
Mo(1)–C(1)	0.2383(3)	Mo(1)–C(1)	0.2378(2)	Mo(1)–C(1)	0.2313(5)	Mo(1)–C(2)	0.2426(4)
Mo(1')–C(1)	0.2227(5)	Mo(1)–C(2)	0.2313(2)	Mo(1)–C(2)	0.2385(5)	Mo(1i)–O(1))	0.18836(5)
Mo(1')–C(2)	0.1998(8)	Mo(1)–C(3)	0.2321(3)	Mo(1)–C(3)	0.2409(5)	Mo(1)–O(1)	0.18836(5)
Mo(1)–C(2)	0.2426(3)	Mo(1)–C(4)	0.2380(3)	Mo(1)–C(4)	0.2381(5)	Mo(1)–O(2)	0.1708(3)
Mo(1')–Mo(1'i)	0.2504(12)	Mo(1)–C(5)	0.2425(3)	Mo(1)–C(5)	0.2313(5)	Mo(1)–O(3)	0.1704(3)
Mo(1')–C(3)–Mo(1)	18.5(4)	C(2)–Mo(1)–C(1)	35.26(9)	C(2)–Mo(1)–C(1)	35.20(17)	C(2)–Mo(1)–C(1)	35.20(17)
Mo(1')–C(1)–Mo(1)	18.4(4)	C(2)–Mo(1)–C(3)	35.88(10)	C(2)–Mo(1)–C(3)	34.53(18)	C(2)–Mo(1)–C(3)	33.17(12)
C(5)–Mo(1)–C(1)	35.25(10)	C(1)–Mo(1)–C(3)	58.67(9)	C(1)–Mo(1)–C(3)	58.08(17)	C(1)–Mo(1)–C(3)	56.92(12)
C(3)–Mo(1)–C(1)	57.86(9)	C(2)–Mo(1)–C(4)	58.77(9)	C(2)–Mo(1)–C(4)	58.16(18)	C(2)–Mo(1)–C(4)	55.80(12)
O(1)–C(13)–Mo(1)	178.9(3)	C(3)–Mo(1)–C(5)	58.05(9)	C(3)–Mo(1)–C(5)	58.16(17)	O(1)–Mo(1)–C(5)	117.39(8)
O(1)–C(13)–Mo(1')	177.7(3)	C(1)–Mo(1)–Mo(1i)	111.31(6)	C(1)–Mo(1)–C(16)	139.8(2)	Mo(1)–O(1)–Mo(1i)	180.0

**Fig. 1.** The molecular structure of complex **7**. Ellipsoids correspond to 30% probability. Hydrogen atoms are omitted for clarity.**Fig. 2.** The molecular structure of complex **8**. Ellipsoids correspond to 30% probability. Hydrogen atoms are omitted for clarity.

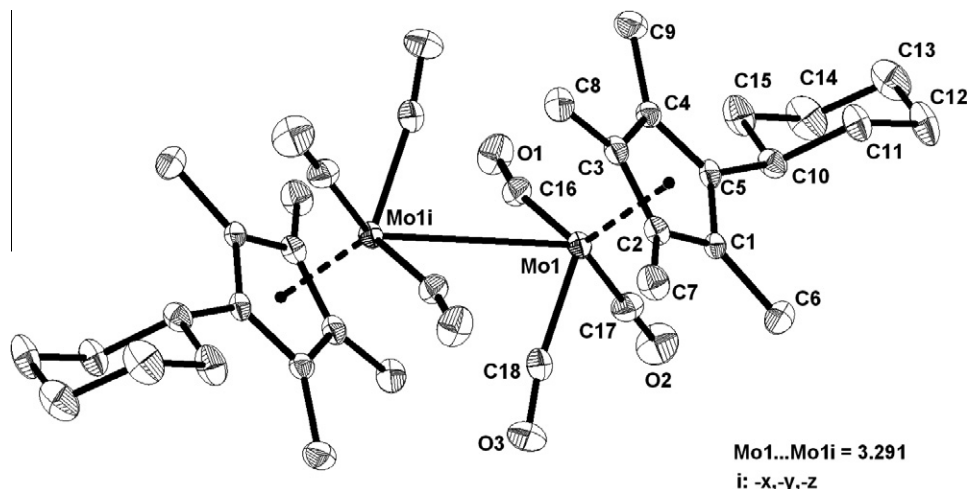


Fig. 3. The molecular structure of complex 11. Ellipsoids correspond to 30% probability. Hydrogen atoms are omitted for clarity.

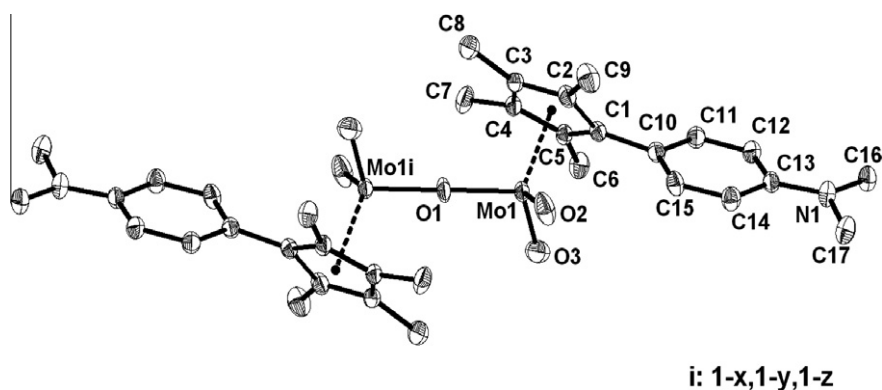


Fig. 4. The molecular structure of complex 12. Ellipsoids correspond to 30% probability. Hydrogen atoms are omitted for clarity.

4. Conclusions

In summary, six new metal carbonyl complexes have been synthesized by the reactions of substituted tetramethylcyclopentadienes with Mo(CO)_6 in refluxing xylene, five of them are normal product but one of them is unexpected product. The results revealed the ligands coordinate to the molybdenum center by η^5 model. Comparing the range of structures that have been determined for complexes of the type $\text{Cp}^*_2\text{Mo}_2(\text{CO})_6$, we find that the same overall structure with six terminal CO groups is presented in all compounds. We also see that the Cp^* ligands are *trans* in the dimeric structures in the solid state. The change of substituent of cyclopentadienyl has some influence on the Mo–Mo bond length. The $\text{Mo}\equiv\text{Mo}$ triple bonded complex was not stable, very easy to react with O_2 in the air.

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Appendix A. Supplementary material

CCDC 832372, 883602, 833999 and 834001 contain the supplementary crystallographic data for **7**, **8**, **11** and **12**. These data can be

obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif. Supplementary data associated with this article can be found, in the online version, at <http://dx.doi.org/10.1016/j.ica.2013.01.010>.

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