

# Synthesis and characterization of dinuclear m- and o-xylene bridged complexes of cyclopentadienyltricarbonylmolybdenum and their derivatives. The crystal structure of bis(methylcyclopentadienyltricarbonylmolybdenum)- $\alpha, \alpha'$ - $\mu$ -o-xylene

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## Abstract

The reactions of R-CpMo(CO)<sub>3</sub>Li (R = H, Me, SiMe<sub>3</sub>) with  $\alpha, \alpha'$ -m- and o-dichloroxylenes gave bridged dinuclear complexes of R-CpMo(CO)<sub>3</sub> in high yield. All of them are stable to air and water, even stable to dilute acids and bases. Complex 5 crystallizes in the monoclinic system, space group  $P2_1/n$ , with  $a = 12.1811(17)$ ,  $b = 12.0634(24)$ ,  $c = 17.4496(22)$  Å,  $\beta = 105.674(11)^\circ$ ,  $V = 2468.8(7)$  Å<sup>3</sup>.  $R_F = 0.032$  and  $R_w = 0.030$ .

**Keywords:** Molybdenum; Cyclopentadienyl; Carbonyl; Xylenes

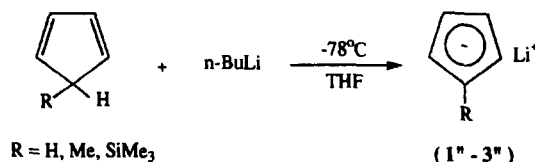
## 1. Introduction

Although a considerable number of  $\sigma$ -allyl derivatives [1,2] as well as  $\sigma$ -benzyl derivatives [3,4] of transition metals have been described, none of them is stable to air and water. Recently we prepared novel double benzyl bridged complexes of molybdenum. Both their physical properties and chemical properties are quite different from those of the early  $\sigma$ -benzyl type complex of molybdenum CpMo(CO)<sub>3</sub>CH<sub>2</sub>C<sub>6</sub>H<sub>5</sub> [3], prepared by King in 1965.

## 2. Results and discussion

Double benzyl type derivatives of molybdenum were prepared from the reactions of R-CpMo(CO)<sub>3</sub>Li (R =

H, Me, SiMe<sub>3</sub>) with  $\alpha, \alpha'$ -m- and o-dichloroxylenes in good yields. The salts (**1'**–**3'**) can be prepared easily from the reaction of R-Cp<sup>-</sup> (**1''**–**3''**) with Mo(CO)<sub>6</sub> in refluxing THF [5]. Instead of sodium, we employed more readily available n-butyl lithium as reagent to prepare R-Cp<sup>-</sup> (**1''**–**3''**):



R = H, Me, SiMe<sub>3</sub>

(**1''**–**3''**)

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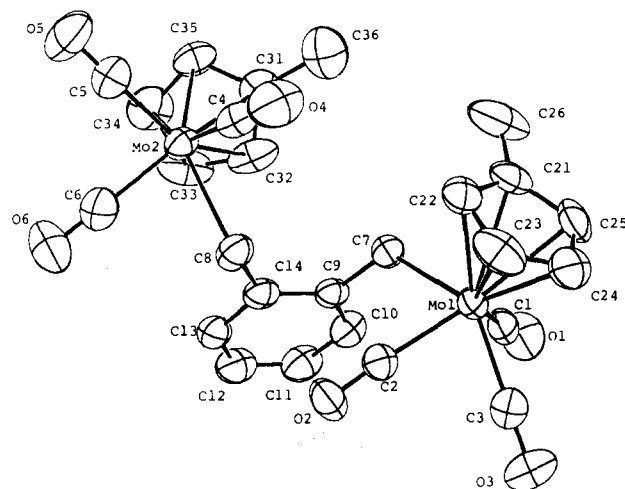
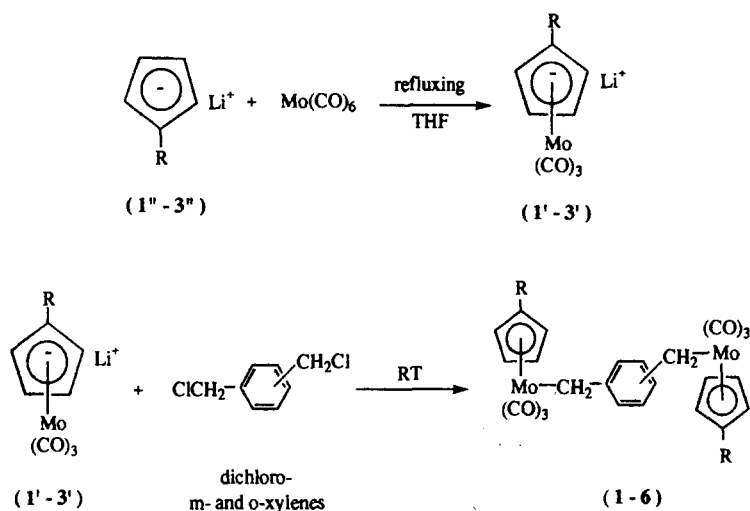
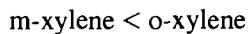
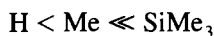


Fig. 1. Molecular structure of complex 5.

The reactions of  $\text{R-CpMo(CO)}_3\text{Li}$  with  $\alpha, \alpha'$ -m- and o-dichloroxylylene in 2:1 molar proportions lead to the formation of anti-type dinuclear  $\alpha, \alpha'$ -m- and o-xylene bridged complexes  $[\text{R-CpMo(CO)}_3]_2(\mu\text{-CH}_2\text{C}_6\text{H}_4\text{-CH}_2\text{-})$  (**1-6**) in good yield. These complexes are stable to air and water, complexes **1, 2, 4, 5** even being stable in dilute acids and bases ( $\sim 10^{-3}$  M).

The solubility of these complexes in common solvents such as diethyl ether and dichloromethane depends on two factors: (1) the substituents R on the Cp ring and (2) the types of xylene. In general, the solubility of complexes **1-6** varies obviously with the following order:

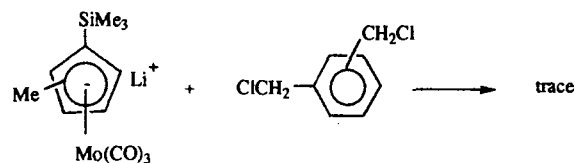


A characteristic feature of all these yellow complexes is the presence of two intense  $\nu(\text{CO})$  bands in the region  $2005\text{--}1920\text{ cm}^{-1}$ . Electron donating substituents on the cyclopentadienyl ring cause a little decrease in the CO stretching frequency, not to the extent observed in the IR spectra for  $\text{Mo(CO)}_3(\eta^6\text{-arene})$  complexes [6]. In addition,  $^1\text{H-NMR}$  spectroscopy also reveals characteristic upfield shifts for the methylene protons upon complexation to the moiety  $\text{R-CpMo(CO)}_3$ . The coordination chemical shift  $\Delta\delta$  is about 1.7 ppm (i.e. from  $\sim 4.5$  to  $\sim 2.8$  ppm). The exact mass was observed in the mass spectra for complexes **2-6**, but not for complex **1**.

The crystal structure of complex **5** consists of discrete molecules of  $[\text{CH}_2\text{Mo(CO)}_3\text{Cp}']_2(\mu\text{-o-CH}_2\text{C}_6\text{H}_4\text{CH}_2\text{-})$ . Fig. 1 shows the ORTEP molecular plot, the plane made up of o- $\text{C}_6\text{H}_4(\text{CH}_2)_2$  being within a deviation of  $0.019(4)$  Å with two metal substituents at the *anti*-positions, presumably steric in origin. The displacements of Mo centers from the plane are  $1.942(5)$  Å for Mo1 and  $-2.243(4)$  Å for Mo2. Mo1 is  $2.026(2)$  Å from the centroid of the attached MeCp ring. For Mo2,

it is  $2.018(3)$  Å. The Mo-C(CO) lengths average  $1.978(8)$  Å, very close to the average of those in  $\text{FvMo}_2(\text{CO})_5(\text{dmpm})$ ,  $1.974(8)$  Å [7]. The mean Mo-C distance for the MeCp ligand of  $2.400(4)$  Å is longer than the corresponding distance in  $\text{MoCp}(\text{C}_4\text{Ph}_4)\text{Cl}_2$ ,  $2.340(14)$  Å [8]. Unlike the  $\alpha, \alpha'$ -p-xylene complex  $[\text{1-Me-3-TMS-CpMo(CO)}_3]_2(\mu\text{-p-CH}_2\text{C}_6\text{H}_4\text{CH}_2\text{-})$  [9], the two Mo-C(CH<sub>2</sub>) distances in **5** are not the same. The Mo1-C(H<sub>2</sub>) distance of  $2.383(4)$  Å is identical with the corresponding distance in  $[\text{1-Me-3-TMS-CpMo(CO)}_3]_2(\mu\text{-p-CH}_2\text{C}_6\text{H}_4\text{CH}_2\text{-})$ ,  $2.383(19)$  Å, and close to that in the complex  $[\text{Mo}_2\{(\text{CH}_2)_2\text{PMe}_2\}_4]$ ,  $2.31(2)$  Å [10]. The Mo2-C(CH<sub>2</sub>) distance is longer,  $2.421(4)$  Å. Both complex **5** and  $[\text{1-Me-3-TMS-CpMo(CO)}_3]_2(\mu\text{-p-CH}_2\text{C}_6\text{H}_4\text{CH}_2\text{-})$  crystallize in monoclinic space groups and are with two  $\text{R-CpMo(CO)}_3$  groups at the *anti*-positions.

Attempts to prepare m- and o- $[\text{1-Me-3-TMS-CpMo(CO)}_3]_2(\mu\text{-CH}_2\text{C}_6\text{H}_4\text{CH}_2\text{-})$  were unsuccessful. The products were obtained in yields less than 5%. However,  $[\text{1-Me-3-TMS-CpMo(CO)}_3]_2(\mu\text{-p-CH}_2\text{C}_6\text{H}_4\text{CH}_2\text{-})$  was obtained in high yield [9].



### 3. Conclusion

In this work we prepared and characterized the  $\alpha, \alpha'$ -m- and o-bridged binuclear molybdenum complexes **1-6**. We also identified the molecular structure of complex **5** to be an *anti*-conformer in the solid state.

## 4. Experimental

### 4.1. General information

All solvents were routinely purified by standard procedures and stored under nitrogen [11]. All reactions were carried out in dry solvents under oxygen-free nitrogen. Commercial reagents including  $\text{Mo}(\text{CO})_6$ ,  $^n\text{BuLi}$ ,  $\text{Me}_3\text{SiCl}$ , cyclopentadiene dimer, methylcyclopentadiene dimer,  $\alpha, \alpha'$ -dichloro-*m*-xylene and  $\alpha, \alpha'$ -dichloro-*o*-xylene purchased from Aldrich Chemicals Co.  $\text{Me}_3\text{SiC}_5\text{H}_5$  [12] and  $\text{Me}_3\text{SiC}_5\text{H}_4\text{Me}$  [13] were prepared according to literature procedures.

Infrared spectra were recorded on a Jasco FT/IR 300 E spectrometer,  $^1\text{H-NMR}$  spectra were registered on an AC 400 Bruker NMR spectrometer. Mass spectra were measured on a Finnigan 1015 D GC-MS spectrometer. Elemental analyses were carried out at the Analysis Center of the National Cheng Kung University.

### 4.2. X-ray crystallographic analyses for complex 5

Yellow crystals of complex 5 were prepared by diffusion of hexane into a concentrated  $\text{CH}_2\text{Cl}_2$  solution of complex 5 under nitrogen and at  $4^\circ\text{C}$ . The single crystal X-ray diffraction measurements were performed on a Nonius CAD-4 automated diffractometer using graphite monochromated  $\text{Mo K}\alpha$  radiation. 25 high-angle reflections ( $15.08^\circ < 2\theta < 34.63^\circ$ ) were used in a least-squares fit to obtain accurate cell constants. The monoclinic space group  $P2_1/n$  was assigned on the basis of the systematic absences. Diffraction intensities were collected up to  $2\theta < 45^\circ$  using  $\theta/2\theta$  scan technique. The reflections with  $I_o > 2.5 \sigma(I_o)$  were judged as observations and used for solution and structure refinement. Data were corrected for Lorentz-polarization factors. An empirical absorption correction based on a series of  $\Psi$  scans was applied to the data. The structure was solved by direct methods [14] and refined by a full-matrix least-squares routine [15] with anisotropic thermal parameters for all non-hydrogen atoms. All of the hydrogen atoms were placed isotropically at their calculated positions and fixed in the calculations. For a summary of crystal data and refinement details, see Table 1. Selected bond distances and angles are listed in Tables 2 and 3. Selected atomic coordinates are listed in Table 4.

### 4.3. Bis(cyclopentadienyltricarbonylmolybdenum)- $\alpha, \alpha'$ - $\mu$ -*m*-xylene 1

A three-necked 100 ml round bottomed flask was equipped with a dry ice bath, a reflux condenser topped with a nitrogen inlet, an addition funnel, a magnetic stirrer and a 1 in teflon-coated stirring bar. Into this flask was placed 0.66 g (0.01 mol) of fresh cyclopenta-

Table 1  
Crystallographic data and refinement details for compound 5

Empirical formula	$\text{C}_{26}\text{H}_{22}\text{Mo}_2\text{O}_6$
Molar mass (g)	622.33
Color	Yellow
Crystal size ( $\text{mm}^3$ )	$0.44 \times 0.31 \times 0.13$
Crystal system	Monoclinic
Space group	$P2_1/n$
<i>a</i> (Å)	12.1811(17)
<i>b</i> (Å)	12.0634(24)
<i>c</i> (Å)	17.4496(22)
$\beta$ (deg)	105.67(11)
<i>V</i> (Å <sup>3</sup> )	2468.8(7)
<i>Z</i>	4
<i>F</i> (000)	1239.55
$\lambda$ (Å)	0.71069
$D_{\text{calc}}$ ( $\text{g cm}^{-3}$ )	1.674
$\mu$ ( $\text{mm}^{-1}$ )	1.03
$2\theta$ (max)	44.9
Scan mode	$\theta/2\theta$
<i>h, k, l</i> range	0–13; 0–15; 0–16
No. of reflections	3392
No. of unique reflections	3214
No. of reflections with $I > 2.5\sigma(I)$	2667
$R_F^a$	0.032
$R_w^a$	0.030
GOF	1.49

$$R_F = \frac{\sum(F_o - F_c)}{\sum(F_o)}$$

$$R_w = \left[ \frac{\sum\theta(F_o - F_c)^2}{\sum(\omega F_o^2)} \right]^{1/2}$$

Table 2  
Selected bond distances (Å) for compound 5

Mo(1)–C(1)	1.971(5)
Mo(1)–C(2)	1.993(4)
Mo(1)–C(3)	1.975(4)
Mo(1)–C(7)	2.383(4)
Mo(1)–C(21)	2.385(4)
Mo(1)–C(22)	2.385(4)
Mo(1)–C(23)	2.336(4)
Mo(1)–C(24)	2.322(4)
Mo(1)–C(25)	2.363(4)
Mo(2)–C(4)	1.978(5)
Mo(2)–C(5)	1.970(5)
Mo(2)–C(6)	1.982(5)
Mo(2)–C(8)	2.421(4)
Mo(2)–C(31)	2.397(4)
Mo(2)–C(32)	2.370(4)
Mo(2)–C(33)	2.319(5)
Mo(2)–C(34)	2.304(4)
Mo(2)–C(35)	2.333(4)
C(7)–C(9)	1.482(5)
C(8)–C(14)	1.486(6)
C(9)–C(10)	1.403(5)
C(9)–C(14)	1.407(6)
C(10)–C(11)	1.367(6)
C(11)–C(12)	1.381(7)
C(12)–C(13)	1.377(7)
C(13)–C(14)	1.399(6)
C(21)–C(26)	1.491(6)
C(31)–C(36)	1.482(7)

diene and 30 ml of dry THF. The solution was stirred and cooled to  $-78^{\circ}\text{C}$  and 6.25 ml of  ${}^n\text{BuLi}$  (1.6 M) was added dropwise. After the reaction was completed (about 30 min), 2.64 g (0.01 mol) of  $\text{Mo}(\text{CO})_6$  was added, and the mixture was heated to reflux for 18 h. The resulting deep orange solution was cooled to room temperature. With stirring 0.86 g (0.005 mol) of  $\alpha, \alpha'$ -dichloro-*m*-xylene was added. The mixture was stirred for 1 h. After removal of the solvent the residue was washed three times with 10 ml of distilled water, then three times with 10 ml benzene. The product was isolated as yellow solids, in 68% yield (1.998 g). IR ( $\text{CH}_2\text{Cl}_2$ ): 1925 (vs), 2012 (s) ( $\nu$  CO).  ${}^1\text{H-NMR}$  ( $\text{CDCl}_3$ ):  $\delta$  7.18–7.08 (m, 4H), 5.13 (s, 10H), 2.88 (s, 4H). Anal. Found: C, 48.58; H, 3.14.  $\text{C}_{24}\text{H}_{18}\text{Mo}_2\text{O}_6$  Calc.: C, 48.48; H, 3.03%.

#### 4.4. Bis(methylcyclopentadienyltricarbonylmolybdenum)- $\alpha, \alpha'$ - $\mu$ -*m*-xylene 2

In a three-necked 100 ml spherical flask equipped as described above was added 0.04 g (0.005 mol) of fresh methylcyclopentadiene and 30 ml of dry THF. The solution was stirred and cooled to  $-78^{\circ}\text{C}$  and 6.25 ml of  ${}^n\text{BuLi}$  (1.6 M) was added dropwise. After the reaction was completed (about 1 h), 1.32 g (0.005 mol) of  $\text{Mo}(\text{CO})_6$  was added, and the mixture was heated to reflux for 21 h. The resulting deep orange solution was cooled to room temperature. With stirring 0.45 g (in excess) of  $\alpha, \alpha'$ -dichloro-*m*-xylene was added. The mixture was stirred for 45 min. The treatment was the same as for the reaction above. The product consisted of yellow powder, yield 63% (0.97 g). IR ( $\text{CH}_2\text{Cl}_2$ ): 2008 (s), 1923 (vs)  $\text{cm}^{-1}$  ( $\nu$  CO).  ${}^1\text{H-NMR}$  ( $\text{CDCl}_3$ ):  $\delta$  7.12–7.06 (m, 4H), 5.11 (d, 4H, 2.3 Hz), 5.06 (d, 4H), 2.87 (s, 4H), 1.97 (s, 6H). Mass ( $m/z$ ): 623 (M + 1), 622 (M), 594 (M–CO), 566 (M–2CO). Anal. Found: 49.82; H, 3.64.  $\text{C}_{26}\text{H}_{22}\text{Mo}_2\text{O}_6$  Calc.: C, 50.18; H, 3.56%.

#### 4.5. Bis(trimethylsilylcyclopentadienyltricarbonylmolybdenum)- $\alpha, \alpha'$ - $\mu$ -*m*-xylene 3

In a three-necked 100 ml spherical flask equipped as described above was added 0.46 g (0.0033 mol) of trimethylsilylcyclopentadiene and 30 ml of dry THF. The solution was stirred and cooled to  $-78^{\circ}\text{C}$  and 2.06 ml of  ${}^n\text{BuLi}$  (1.6 M) was added dropwise. After the reaction was completed (about 1 h), 0.872 g (0.0033 mol) of  $\text{Mo}(\text{CO})_6$  was added, and the mixture was heated to reflux for 24 h. The resulting orange solution was cooled to room temperature. With stirring 0.289 g (0.00165 mol) of  $\alpha, \alpha'$ -dichloro-*m*-xylene was added. The mixture was stirred for 40 min. The treatment was the same as for the reaction above. The product consisted of yellow powder, yield 52% (0.63 g). IR ( $\text{CH}_2\text{Cl}_2$ ): 2005 (s), 1920 (vs)  $\text{cm}^{-1}$  ( $\nu$  CO), 1249 (s)

$\text{cm}^{-1}$  ( $\nu$  SiMe<sub>3</sub>).  ${}^1\text{H-NMR}$  ( $\text{CDCl}_3$ ):  $\delta$  7.08–7.02 (m, 4H), 5.18 (d, 4H, 2.6 Hz), 4.98 (d, 4H), 0.08 (s, 18H). Mass ( $m/z$ ): 739 (M + 1), 738 (M), 710 (M–CO), 682

Table 3  
Selected bond angles (deg) for compound 5

C(1)–Mo(1)–C(2)	107.23(15)
C(1)–Mo(1)–C(3)	79.02(18)
C(1)–Mo(1)–C(7)	74.03(15)
C(1)–Mo(1)–C(21)	105.32(16)
C(1)–Mo(1)–C(22)	139.14(16)
C(1)–Mo(1)–C(23)	151.10(17)
C(1)–Mo(1)–C(24)	117.26(18)
C(1)–Mo(1)–C(25)	94.82(16)
C(2)–Mo(1)–C(3)	76.48(16)
C(2)–Mo(1)–C(7)	75.92(14)
C(2)–Mo(1)–C(21)	134.55(16)
C(2)–Mo(1)–C(22)	103.71(15)
C(2)–Mo(1)–C(23)	100.43(16)
C(2)–Mo(1)–C(24)	127.42(18)
C(2)–Mo(1)–C(25)	157.93(15)
C(3)–Mo(1)–C(7)	133.14(15)
C(3)–Mo(1)–C(21)	141.05(16)
C(3)–Mo(1)–C(22)	134.66(18)
C(3)–Mo(1)–C(23)	99.86(18)
C(3)–Mo(1)–C(24)	85.55(16)
C(3)–Mo(1)–C(25)	107.67(17)
C(7)–Mo(1)–C(21)	83.60(13)
C(7)–Mo(1)–C(22)	88.57(15)
C(7)–Mo(1)–C(23)	121.89(17)
C(7)–Mo(1)–C(24)	140.99(14)
C(7)–Mo(1)–C(25)	112.12(15)
C(4)–Mo(2)–C(5)	78.17(18)
C(4)–Mo(2)–C(6)	108.77(19)
C(4)–Mo(2)–C(8)	70.81(15)
C(4)–Mo(2)–C(31)	94.90(16)
C(4)–Mo(2)–C(32)	112.14(19)
C(4)–Mo(2)–C(33)	147.15(20)
C(4)–Mo(2)–C(34)	145.86(18)
C(4)–Mo(2)–C(35)	110.88(17)
C(5)–Mo(2)–C(6)	77.92(19)
C(5)–Mo(2)–C(8)	126.50(16)
C(5)–Mo(2)–C(31)	114.82(17)
C(5)–Mo(2)–C(32)	145.10(17)
C(5)–Mo(2)–C(33)	127.86(20)
C(5)–Mo(2)–C(34)	94.32(20)
C(5)–Mo(2)–C(35)	87.64(17)
C(6)–Mo(2)–C(8)	72.56(16)
C(6)–Mo(2)–C(31)	155.33(18)
C(6)–Mo(2)–C(32)	124.91(19)
C(6)–Mo(2)–C(33)	97.40(20)
C(6)–Mo(2)–C(34)	101.92(19)
C(6)–Mo(2)–C(35)	133.68(18)
C(8)–Mo(2)–C(31)	110.37(14)
C(8)–Mo(2)–C(32)	87.72(14)
C(8)–Mo(2)–C(33)	99.75(16)
C(8)–Mo(2)–C(34)	134.56(17)
C(8)–Mo(2)–C(35)	143.80(15)
Mo(1)–C(7)–C(9)	122.87(24)
Mo(2)–C(8)–C(14)	111.32(24)
Mo(1)–C(1)–O(1)	175.3(3)
Mo(1)–C(2)–O(2)	176.1(3)
Mo(1)–C(3)–O(3)	179.1(4)
Mo(2)–C(4)–O(4)	177.2(4)
Mo(2)–C(5)–O(5)	179.3(4)
Mo(2)–C(6)–O(6)	178.2(4)

(M–2CO), 626 (M–4CO). Anal. Found: C, 48.84; H, 5.02. C<sub>30</sub>H<sub>34</sub>Mo<sub>2</sub>Si<sub>2</sub>O<sub>6</sub>. Calc.: C, 48.78; H, 4.61%.

#### 4.6. Bis(cyclopentadienyltricarbonylmolybdenum)- $\alpha,\alpha'$ - $\mu$ -o-xylene 4

In a three-necked 100 ml spherical flask equipped as described above was added 0.33 g (0.005 mol) of fresh cyclopentadiene and 30 ml of dry THF. The solution was stirred and cooled to  $-78^{\circ}\text{C}$  and 3.13 ml of <sup>n</sup>BuLi (1.6 M) was added dropwise. After the reaction was completed (about 30 min), 1.32 g (0.005 mol) of Mo(CO)<sub>6</sub> was added, and the mixture was heated to reflux for 18 h. The resulting deep orange solution was cooled to room temperature. With stirring 0.43 g (0.0025 mol) of  $\alpha,\alpha'$ -dichloro-o-xylene was added. The mixture was stirred for 1 h. The treatment was the same as for the reaction above. The product consisted of yellow powder, yield 49% (0.73 g). IR (CH<sub>2</sub>Cl<sub>2</sub>): 2013 (s), 1926 (vs) cm<sup>-1</sup> ( $\nu$  CO). <sup>1</sup>H-NMR (CDCl<sub>3</sub>):  $\delta$  7.15–7.08 (m, 4H), 5.13 (s, 10H), 2.85 (s, 4H). Mass ( $m/z$ ): 595 (M + 1), 594 (M), 566 (M–CO), 538 (M–2CO). Anal. Found: 48.35; H, 3.17. C<sub>24</sub>H<sub>18</sub>Mo<sub>2</sub>O<sub>6</sub>. Calc.: C, 48.48; H, 3.03%.

#### 4.7. Bis(methylcyclopentadienyltricarbonylmolybdenum)- $\alpha,\alpha'$ - $\mu$ -o-xylene 5

In a three-necked 100 ml spherical flask equipped as described above was added 0.04 g (0.005 mol) of fresh methylcyclopentadiene and 30 ml of dry THF. The

solution was stirred and cooled to  $-78^{\circ}\text{C}$  and 3.25 ml of <sup>n</sup>BuLi (1.6 M) was added dropwise. After the reaction was completed (about 1 h), 1.32 g (0.005 mol) of Mo(CO)<sub>6</sub> was added, and the mixture was heated to reflux for 21 h. The resulting deep orange solution was cooled to room temperature. With stirring 0.45 g (in excess) of  $\alpha,\alpha'$ -dichloro-o-xylene was added. The mixture was stirred for 50 min. The treatment was the same as for the reaction above. The product consisted of yellow powder, yield 42% (0.65 g). IR (CH<sub>2</sub>Cl<sub>2</sub>): 2009 (s), 1923 (vs) cm<sup>-1</sup> ( $\nu$  CO). <sup>1</sup>H-NMR (CDCl<sub>3</sub>):  $\delta$  7.11–7.04 (m, 4H), 5.09 (d, 4H, 2.4 Hz), 5.06 (d, 4H), 2.83 (s, 4H). Mass ( $m/z$ ): 623 (M + 1), 622 (M), 594 (M–CO), 566 (M–2CO), 175 (Cp'Mo). Anal. Found: C, 49.85; H, 3.50. C<sub>26</sub>H<sub>22</sub>Mo<sub>2</sub>O<sub>6</sub>. Calc.: C, 50.18; H, 3.56%.

#### 4.8. Bis(trimethylsilylcyclopentadienyltricarbonylmolybdenum)- $\alpha,\alpha'$ - $\mu$ -o-xylene 6

In a three-necked 100 ml spherical flask equipped as described above was added 0.46 g (0.0033 mol) of trimethylsilylcyclopentadiene and 30 ml of dry THF. The solution was stirred and cooled to  $-78^{\circ}\text{C}$  and 2.06 ml of <sup>n</sup>BuLi (1.6 M) was added dropwise. After the reaction was completed (about 1 h), 0.872 g (0.0033 mol) of Mo(CO)<sub>6</sub> was added, and the mixture was heated to reflux for 24 h. The resulting orange solution was cooled to room temperature. With stirring 0.289 g (0.00165 mol) of  $\alpha,\alpha'$ -dichloro-o-xylene was added. The mixture was stirred for 40 min. The treatment was

Table 4  
Non-H atomic coordinates for compound 5

Atom	x	y	z	B <sub>iso</sub>
Mo1	0.22979(3)	0.15543(3)	0.02344(2)	2.88(1)
Mo2	-0.01363(3)	0.09149(3)	0.02741(2)	3.52(2)
C1	0.2607(3)	0.3159(4)	0.0246(2)	3.9(2)
C2	0.0611(4)	0.1363(3)	-0.0099(2)	3.3(2)
C3	0.1884(4)	0.1882(4)	-0.0917(3)	4.7(2)
C4	0.0814(4)	-0.0203(4)	0.2625(2)	4.3(2)
C5	-0.0453(4)	-0.0430(4)	0.3518(3)	5.2(2)
C6	-0.1760(4)	0.0628(4)	0.2422(3)	5.2(2)
C7	0.1687(3)	0.2292(3)	0.1318(2)	3.6(2)
C8	-0.0360(3)	0.1125(3)	0.1560(2)	3.9(2)
C9	0.0572(3)	0.2851(3)	0.1212(2)	3.2(2)
C10	0.0473(4)	0.3981(3)	0.1013(2)	4.1(2)
C11	-0.0513(4)	0.4563(3)	0.0938(2)	4.8(2)
C12	-0.1462(4)	0.4026(4)	0.1047(3)	4.9(2)
C13	-0.1403(4)	0.2916(4)	0.1237(2)	4.1(2)
C14	-0.0394(3)	0.2314(3)	0.1330(2)	3.4(2)
C21	0.3810(3)	0.0792(4)	0.1266(2)	4.2(2)
C22	0.2950(4)	-0.0019(4)	0.1008(3)	4.5(2)
C23	0.2885(4)	-0.0286(4)	0.0216(3)	5.2(2)
C24	0.3661(4)	0.0377(4)	-0.0026(3)	5.2(2)
C25	0.4240(3)	0.1034(4)	0.0620(3)	4.7(2)
C31	0.1446(4)	0.1994(4)	0.3719(2)	4.3(2)
C32	0.0732(5)	0.2689(4)	0.3149(2)	5.3(3)
C33	-0.0340(5)	0.2747(4)	0.3313(3)	6.4(3)
C34	-0.0281(5)	0.2094(5)	0.3981(3)	6.4(3)
C35	0.0803(4)	0.1618(4)	0.4225(2)	4.9(2)

the same as for the reaction above. The product consisted of yellow powder, yield 36% (0.44 g). IR ( $\text{CH}_2\text{Cl}_2$ ): 2006 (s), 1923 (vs)  $\text{cm}^{-1}$  ( $\nu$  CO), 1249 (s)  $\text{cm}^{-1}$  ( $\text{SiMe}_3$ ).  $^1\text{H-NMR}$  ( $\text{CDCl}_3$ ):  $\delta$  7.06–7.01 (m, 4H), 5.16 (d, 4H, 3.1 Hz), 4.95 (d, 4H), 0.08 (s, 18H). Mass ( $m/z$ ): 739 (M + 1), 738 (M), 710 (M–CO), 682 (M–2CO), 626 (M–4CO), 598 (M–5CO). Anal. Found: C, 49.12; H, 4.71.  $\text{C}_{30}\text{H}_{34}\text{Mo}_2\text{Si}_2\text{O}_6$  Calc.: C, 48.78; H, 4.61%.

### 5. Supplementary material available

Complete bond distances, bond angles, atomic coordinates and anisotropic temperature factors are available from See Lin and Lin-Kang Liu.

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### References

- [1] M.L.H. Green and P.L.I. Hagy, *Adv. Organomet. Chem.*, **2** (1964) 325.
- [2] E.W. Abel and S. Moorhouse, *J. Chem. Soc., Dalton Trans.*, (1973) 1706.
- [3] R.B. King and A. Frronzaglia, *J. Am. Chem. Soc.*, **88** (1966) 709.
- [4] J.S. Roberts and K.J. Klaunde, *J. Am. Chem. Soc.*, **99** (1976) 2509.
- [5] S. Lin, C.Y. Lin and H.Y. Wang, *J. Chin. Chem. Soc.*, **33** (1986) 261.
- [6] C. Barbeau and J. Turcotte, *Can J. Chem.*, **54** (1976) 1612.
- [7] M. Tilset, K.P.C. Vollhardt and R. Boese, *Organometallics*, **13** (1994) 3146.
- [8] O.J. Curnow, W. Hirpo, W.M. Butler and M.D. Curtis, *Organometallics*, **12** (1993) 4479.
- [9] S. Lin, S.B.T. Nyuyen, R.H. Grubbs and J.W. Ziller, unpublished work.
- [10] F.A. Cotton, B.E. Hanson, W.H. Ilsley and G.W. Rice, *Inorg. Chem.*, **18** (1979) 2713.
- [11] D.D. Perrin, W.L.F. Armarego and D.R. Perrin, *Purification of Laboratory Chemicals*, Pergamon, New York, 1980.
- [12] H.P. Fritz and C.G. Kreiter, *J. Organomet. Chem.*, **4** (1965) 313.
- [13] A. Darison and P.E. Rakta, *J. Am. Chem. Soc.*, **90** (1968) 4479.
- [14] P. Main, in G.M. Sheldrick, C. Krueger and R. Goddard (eds.), *Crystallographic Computing 3: Data Collection, Structure Determination, Proteins and Databases*, Clarendon, Oxford, 1985, p. 206.
- [15] E.J. Gabe, F.L. Lee and Y. Lepage, in G.M. Krueger and R. Goddard (eds.), *Crystallographic Computing 3: Data Collection, Structure Determination, Proteins and Databases*, Clarendon, Oxford, 1985, p. 167.