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**CRYSTAL STRUCTURES OF DIMOLYBDENUM CARBONYL COMPLEXES  
CONTAINING THE CYCLOPENTADIENYL—THIENYL LIGAND****J.-W. Wang<sup>1</sup>, L.-J. Tian<sup>2</sup>, Z.-H. Ma<sup>3</sup>, K.-M. Guo<sup>2</sup>, Z.-G. Han<sup>2</sup>, X.-Z. Zheng<sup>2</sup>, J. Lin<sup>2</sup>**<sup>1</sup>*Department of Chemistry, Shanxi Normal University, Linfen, People's Republic of China,  
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Reactions of thienyl side-chain-functionalized cyclopentadienyl ligands  $C_5H_5C(R_1R_2)C_4H_3S$  [ $R_1 = R_2 = CH_3$  (**1**);  $R_1 = CH_3$ ,  $R_2 = C_2H_5$  (**2**);  $R_1 = R_2 = C_2H_5$  (**3**);  $R_1, R_2 = (CH_2)_5$  (**4**)] with  $Mo(CO)_6$  in refluxing xylene gave the corresponding cyclopentadienyl dimolybdenum carbonyl complexes:  $[(\eta^5-C_5H_4)C(R_1R_2)(C_4H_3S)Mo(CO)_3]_2$  [ $R_1 = R_2 = CH_3$  (**5**);  $R_1 = CH_3$ ,  $R_2 = C_2H_5$  (**6**);  $R_1 = R_2 = C_2H_5$  (**7**);  $R_1, R_2 = (CH_2)_5$  (**8**)]. They are characterized by elemental analysis, IR and  $^1H$  NMR spectra. The molecular structures of (**5**) and (**8**) are determined by single crystal X-ray diffraction.

**Keywords:** cyclopentadienyl—thienyl, molybdenum carbonyl, crystal structure.

**INTRODUCTION**

Substituted cyclopentadienyl anions occupy a prominent place in organometallic chemistry, serving as versatile ligands for main group elements, transition metals, and actinides [1]. Seemingly subtle changes in cyclopentadienyl ligand substitution can have profound consequences on chemical reactivity. In recent years, the chemistry of cyclopentadienyl metal complexes containing a donor functionalized side chain has been receiving much attention [2–4]. However, the coordination chemistry of S-donor ligands have hardly been explored. If we introduce the thiophene moiety into a cyclopentadienyl ring, it is envisaged that the thiophene-cyclopentadienyl ligand may serve as a hemilabile ligand. The S atom of thiophene can act as a good two electron donor site and can coordinate to a variety of metals [5–8]. The thienyl side-chain-functionalized cyclopentadienyl ligands can coordinate to the metal center by  $\eta^5$  mode for cyclopentadienyl and by  $\eta^1$  mode for the S atom [9–11]. The interest in novel reactions led us to investigate the reactivity of  $Mo(CO)_6$  with  $C_5H_5C(R_1R_2)C_4H_3S$  [ $R_1 = R_2 = CH_3$  (**1**);  $R_1 = CH_3$ ,  $R_2 = C_2H_5$  (**2**);  $R_1 = R_2 = C_2H_5$  (**3**);  $R_1, R_2 = (CH_2)_5$  (**4**)]. Meanwhile, the structures of the complexes were characterized.

**EXPERIMENTAL**

Schlenk and vacuum line techniques were employed for all manipulations. All solvents were distilled from appropriate drying agents under argon prior to use. Methylene chloride was distilled over  $P_2O_5$  under nitrogen. Furthermore, the chromatographic spectra were measured using dichloromethane and petroleum ether as eluent.  $^1H$  NMR spectra were recorded on a Bruker AV 500 instrument, while IR spectra were recorded on a FT-IR 8900 spectrometer as KBr pellets and elemental analyses were recorded on a VARIO EL III analyzer.

The ligands  $C_5H_5C(R_1R_2)C_4H_3S$  [ $R_1 = R_2 = CH_3$  (**1**);  $R_1 = CH_3, R_2 = C_2H_5$  (**2**);  $R_1 = R_2 = C_2H_5$  (**3**);  $R_1, R_2 = (CH_2)_5$  (**4**)] were synthesized according to the literature procedures [12].

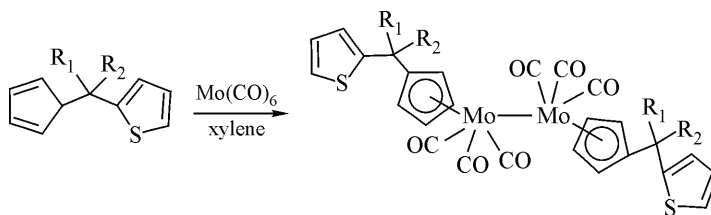
**Synthesis of  $[(\eta^5-C_5H_4)C(CH_3)_2C_4H_3SMo(CO)_3]_2$  complex (**5**).** A xylene solution (30 ml) of ligand (**1**) (2.5 mmol, 0.48 g) and  $Mo(CO)_6$  (2.0 mmol, 0.53 g) was stirred and refluxed for 16 h. Volatiles were removed under reduced pressure. The residue was dissolved in methylene chloride and subjected to an alumina column. Elution with  $CH_2Cl_2$ /petroleum ether ( $v:v = 1:1$ ) afforded a red band. Concentration of the fraction gave bright-red crystals (**5**). Yield: 0.53 g (72 %). m.p.: 193 °C.  $C_{30}H_{26}Mo_2O_6S_2$ : Found: C, 48.9; S, 8.6; H, 3.5. Calcd: C, 48.8; S, 8.7; H, 3.6. IR (KBr,  $cm^{-1}$ ): 1884(s), 1903(w), 1913(s), 1928(s), 1942(w), 1950(s).  $^1H$  NMR (500 MHz,  $CDCl_3$ ,  $\delta$ , ppm): 7.13 (d,  $J = 4.9$  Hz, 2H,  $C_4H_3S$ ), 6.90 (m, 2H,  $C_4H_3S$ ), 6.86 (d,  $J = 4.5$  Hz, 2H,  $C_4H_3S$ ), 5.11 (m, 4H,  $C_5H_4$ ), 5.35 (m, 4H,  $C_5H_4$ ), 1.52 (s, 12H,  $CH_3$ ).

**Synthesis of complexes (**6**–**8**).** Using similar procedures as described above, the reactions of ligand (**2**–**4**) with  $Mo(CO)_6$  gave (**6**–**8**) as red complexes in 75 %, 83 %, and 88 % yields respectively. (**6**): m.p. 199 °C.  $C_{32}H_{30}Mo_2O_6S_2$ : Found: C, 50.2; S, 8.3; H, 4.0. Calcd: C, 50.1; S, 8.4; H 3.9. IR (KBr,  $cm^{-1}$ ): 1890(w), 1906(w), 1923 (s), 1933(w), 1945 (s), 1958 (s).  $^1H$  NMR (500 MHz,  $CDCl_3$ ,  $\delta$ , ppm): 7.17 (d,  $J = 5.0$  Hz, 2H,  $C_4H_3S$ ), 6.93 (m, 2H,  $C_4H_3S$ ), 6.89 (d,  $J = 4.7$  Hz, 2H,  $C_4H_3S$ ), 5.06 (m, 4H,  $C_5H_4$ ), 5.36 (m, 4H,  $C_5H_4$ ), 1.67 (s, 6H,  $CH_3$ ), 1.30 (m, 4H,  $CH_2$ ), 0.83 (t,  $J = 7.5$  Hz, 6H,  $CH_2CH_3$ ). (**7**): m.p. 209 °C.  $C_{34}H_{34}Mo_2O_6S_2$ : Found: C, 51.3; S, 8.0; H, 4.2. Calcd: C, 51.4; S, 8.1; H, 4.3. IR (KBr,  $cm^{-1}$ ): 1894 (s), 1910(w), 1923(s), 1935(s), 1943(w), 1950(s).  $^1H$  NMR (500 MHz,  $CDCl_3$ ,  $\delta$ , ppm): 7.20 (d,  $J = 4.9$  Hz, 2H,  $C_4H_3S$ ), 7.01 (m, 2H,  $C_4H_3S$ ), 6.99 (d,  $J = 3.8$  Hz, 2H,  $C_4H_3S$ ), 5.02 (m, 4H,  $C_5H_4$ ), 5.30 (m, 4H,  $C_5H_4$ ), 2.08 (m,  $J = 7.5$  Hz, 8H,  $CH_2$ ), 0.86 (t,  $J = 7.5$  Hz, 12H,  $CH_3$ ). (**8**): m.p. 210–211 °C.  $C_{36}H_{34}Mo_2O_6S_2$ : Found: C, 52.9; S, 7.9; H, 4.1. Calcd: C, 52.8; S, 8.0; H, 4.2. IR (KBr,  $cm^{-1}$ ): 1890(s), 1902(s), 1932(w), 1948(s), 1954(w), 1960(s).  $^1H$  NMR (500 MHz,  $CDCl_3$ ,  $\delta$ , ppm): 7.13 (d,  $J = 4.9$  Hz, 2H,  $C_4H_3S$ ), 7.05 (m, 2H,  $C_4H_3S$ ), 6.96 (d,  $J = 4.5$  Hz, 2H,  $C_4H_3S$ ), 5.18 (m, 4H,  $C_5H_4$ ), 4.99 (m, 4H,  $C_5H_4$ ), 1.26–1.80 (m, 20H,  $(CH_2)_5$ ).

**Crystal structure determination.** Single crystals of (**5**) and (**8**) suitable for the X-ray structure analysis were obtained from hexane-dichloromethane solutions at room temperature. Diffraction intensities were collected at 298 K with Bruker SMART APEX(II)-CCD or Bruker Smart 1000 CCD, using graphite-monochromated  $MoK_\alpha$  radiation ( $\varphi/\omega$  scans,  $\lambda = 0.71073 \text{ \AA}$ ). Semiempirical absorption corrections were applied for all complexes using the SADABS program. The structures were solved by direct methods and refined by full-matrix least squares. All calculations were performed using the SHELXL-97 program. Additional details of the data collection and structure refinement are given in Table 1.

## RESULTS AND DISCUSSION

**Synthesis of complexes (**5**–**8**).** Upon thermal treatment of  $C_5H_5CR_1R_2C_4H_3S$  ligands [ $R_1 = R_2 = CH_3$  (**1**);  $R_1 = CH_3, R_2 = C_2H_5$  (**2**);  $R_1 = R_2 = C_2H_5$  (**3**);  $R_1, R_2 = (CH_2)_5$  (**4**)] with  $Mo(CO)_6$  under refluxing xylene for 16 h, the corresponding Mo–Mo bonded dinuclear complexes  $(C_5H_4CR_1R_2C_4H_3S)_2Mo_2(CO)_6$  were synthesized. The equations of the reaction were as follows (Scheme 1). The four complexes are sensitive towards air and moisture in the solution, but in the solid state, they are relatively stable.



Scheme 1. Synthesis of complexes **5**–**8**

$R_1 = CH_3, R_2 = CH_3$  (**5**);  $R_1 = CH_3, R_2 = CH_2CH_3$  (**6**);  $R_1 = R_2 = C_2H_5$  (**7**);  $R_1, R_2 = (CH_2)_5$  (**8**)

Table 1

## Crystal data and summary of X-ray collection for (5) and (8)

Complex	5	8
Empirical formula	C <sub>30</sub> H <sub>26</sub> Mo <sub>2</sub> O <sub>6</sub> S <sub>2</sub>	C <sub>36</sub> H <sub>34</sub> Mo <sub>2</sub> O <sub>6</sub> S <sub>2</sub>
Formula weight	738.50	818.63
Temperature, K	298(2)	298(2)
Wavelength, Å	0.71073	0.71073
Crystal system	Monoclinic	Triclinic
Space group	<i>P</i> 2 <sub>1</sub> / <i>c</i>	<i>P</i> $\bar{1}$
<i>a</i> , <i>b</i> , <i>c</i> , Å	8.3642(6), 11.0864(8), 16.2907(12)	8.7350(16), 13.507(3), 15.420(3)
$\alpha$ , $\beta$ , $\gamma$ , deg.	90, 103.4740(10), 90	71.861(2), 73.836(2), 87.655(2)
<i>V</i> , Å <sup>3</sup>	1.46904(18)	1.6584(5)
<i>Z</i>	4	2
<i>D</i> <sub>c</sub> , g/cm <sup>-3</sup>	1.670	1.639
$\mu$ , mm <sup>-1</sup>	1.037	0.928
<i>F</i> (000)	740	828
Crystal size, mm	0.15×0.10×0.05	0.49×0.24×0.11
$\theta$ range, deg.	2.24—27.50	1.45—25.50
Reflections collected/unique	8437 / 3285 [ <i>R</i> <sub>int</sub> = 0.0254]	8699 / 6054 [ <i>R</i> <sub>int</sub> = 0.0297]
Completeness to $\theta = 27.50$ %	97.4	98.0
Max. and min. transmission	0.9500 and 0.8599	0.9048 and 0.6592
Data / restraints / parameters	3285 / 0 / 225	6054 / 7 / 415
Goodness-of-fit on <i>F</i> <sup>2</sup>	1.093	1.045
Final <i>R</i> indices [ <i>I</i> > 2 $\sigma$ ( <i>I</i> )]	<i>R</i> <sub>1</sub> = 0.0458, <i>wR</i> <sub>2</sub> = 0.1336	<i>R</i> <sub>1</sub> = 0.0468, <i>wR</i> <sub>2</sub> = 0.1348
<i>R</i> indices (all data)	<i>R</i> <sub>1</sub> = 0.056, <i>wR</i> <sub>2</sub> = 0.1421	<i>R</i> <sub>1</sub> = 0.0585, <i>wR</i> <sub>2</sub> = 0.1503
$\rho$ min / max, e·nm <sup>-3</sup>	-1259 / 1743	-1145 / 1311

$$^5w = 1/[\sigma^2(F_0^2) + (0.0735p)^2 + 3.4725p], \text{ where } p = (F_0^2 + 2F_c^2)/3.$$

$$^8w = 1/[\sigma^2(F_0^2) + (0.0923p)^2 + 0.4560p], \text{ where } p = (F_0^2 + 2F_c^2)/3.$$

The IR spectra of all four complexes showed similar patterns and displayed characteristic absorption peaks of six strong CO in the terminal  $\nu(\text{CO})$  region. In the <sup>1</sup>H NMR spectra of complexes **5**—**8**, three resonances were separately observed at  $\delta = 6.86$ — $7.20$  ppm for thiophene protons. When compared to the corresponding values in the references, all four complexes showed three similar patterns and displayed characteristic absorption peaks of thiophene, which was substituted at the 2-position ( $\delta$ , ppm, 6.84—7.20). Two multiple peaks for the cyclopentadienyl proton signals appear at  $\delta = 4.99$ — $5.30$  ppm, which is consistent with the chemical shifts of known cyclopentadienyl.

**Crystal structure of complexes (5) and (8).** X-ray diffraction analysis shows that in (**5**) and (**8**), each molybdenum atom is coordinated to  $\eta^5$ -cyclopentadienyl, and an intramolecular sulfur atom of the thienyl ring is not involved in coordination to the atoms. The five-membered heterocycle of thiophene is electron-rich; it is relatively difficult for thiophene to become an anion by losing hydrogen. The lone pair in the S *p*-orbital overlaps with each *p*-orbital of the other four carbon atoms to form a closed conjugated system, resulting in reducing its ability to coordinate to metal. The thiophene moiety is linked to the Cp ring through a carbon bridge at the 2-position. Selected bond lengths and angles are listed in Tables 2 and 3.

The crystal structures of complexes (**5**) and (**8**) (Figs. 1, 2) indicate that each of them has a *trans* conformation with six terminal carbonyl ligands. Two cyclopentadienyl rings and two thienyl rings are

Table 2

Selected bond distances (Å) and angles (deg.) for complex (5)

Bond distances					
Mo1—Mo1i	3.242	C(3)—C(4)	1.422(7)	Mo(1)—C(3)	2.356(5)
C(1)—C(5)	1.431(9)	C(4)—C(5)	1.420(7)	Mo(1)—C(4)	2.348(5)
C(1)—C(2)	1.410(9)	Mo(1)—C(1)	2.352(6)	Mo(1)—C(5)	2.321(5)
C(2)—C(3)	1.400(8)	Mo(1)—C(2)	2.371(6)	C(11)—S(1)	1.640(14)
Angles					
O(1)—C(13)—Mo(1)	172.1(5)	C(13)—Mo(1)—C(5)	107.6(2)	C(4)—C(6)—C(8)	111.6(5)
C(11)—C(12)—C(10)	116.4(8)	C(9)—C(6)—C(7)	109.6(5)	C(4)—C(6)—C(7)	110.9(4)
C(15)—Mo(1)—C(13)	76.5(2)	O(2)—C(14)—Mo(1)	173.6(5)	C(2)—C(3)—Mo(1)	73.4(3)
C(13)—Mo(1)—C(14)	107.2(2)	C(12)—C(11)—S(1)	114.9(7)	C(5)—C(4)—C(3)	106.5(5)

Table 3

Selected bond distances (Å) and angles (deg.) for complex (8)

Bond distances					
Mo1—Mo2	3.2241(6)	C(3)—C(4)	1.417(6)	C(3)—Mo(1)	2.374(4)
C(1)—C(5)	1.431(5)	C(4)—C(5)	1.408(5)	C(4)—Mo(1)	2.350(4)
C(1)—C(2)	1.426(6)	C(1)—Mo(1)	2.318(4)	C(5)—Mo(1)	2.347(4)
C(2)—C(3)	1.417(6)	C(2)—Mo(1)	2.350(4)	C(30)—S(1)	1.710(4)
Angles					
C(2)—C(1)—C(5)	108.7(4)	C(26)—C(27)—C(28)	109.7(5)	C(12)—C(13)—C(14)	114.0(5)
C(4)—C(3)—C(2)	107.7(4)	C(18)—Mo(1)—C(1)	106.64(15)	C(14)—C(15)—S(2)	111.9(4)
C(6)—C(12)—S(2)	122.3(3)	C(2)—C(1)—Mo(1)	73.5(2)	C(17)—Mo(1)—C(18)	76.36(18)
O(6)—C(16)—Mo(1)	174.4(4)	C(4)—C(3)—Mo(1)	71.6(2)	C(36)—Mo(2)—C(34)	79.62(19)

approximately parallel. At the same time, the structures are symmetrical ( $C_i$ ) and similar to *trans*- $[(\eta^5\text{-C}_5\text{H}_5)\text{Mo}(\text{CO})_3]_2$  [13]. For *trans*- $[(\eta^5\text{-C}_5\text{H}_5)\text{Mo}(\text{CO})_3]_2$ , five carbon atoms in cyclopentadienyl are equivalent. In the  $^1\text{H}$  NMR spectra there are two single peaks of cyclopentadienyl. As for  $[(\eta^5\text{-C}_5\text{H}_4\text{CR}_1\text{R}_2\text{C}_4\text{H}_3\text{S})\text{Mo}(\text{CO})_3]_2$  [ $\text{R}_1 = \text{R}_2 = \text{CH}_3$  (**5**);  $\text{R}_1, \text{R}_2 = (\text{CH}_2)_5$  (**8**)], the substituent destroys the symmetry of the cyclopentadienyl ring, so that in the  $^1\text{H}$  NMR spectra there are two multiple peaks of cyclopentadienyl. Meanwhile, five carbon atoms become unequal, therefore, their extent of bonding to the Mo atom also becomes unequal. For *trans*- $[(\eta^5\text{-C}_5\text{H}_5)\text{Mo}(\text{CO})_3]_2$ , the longest and the shortest C—C

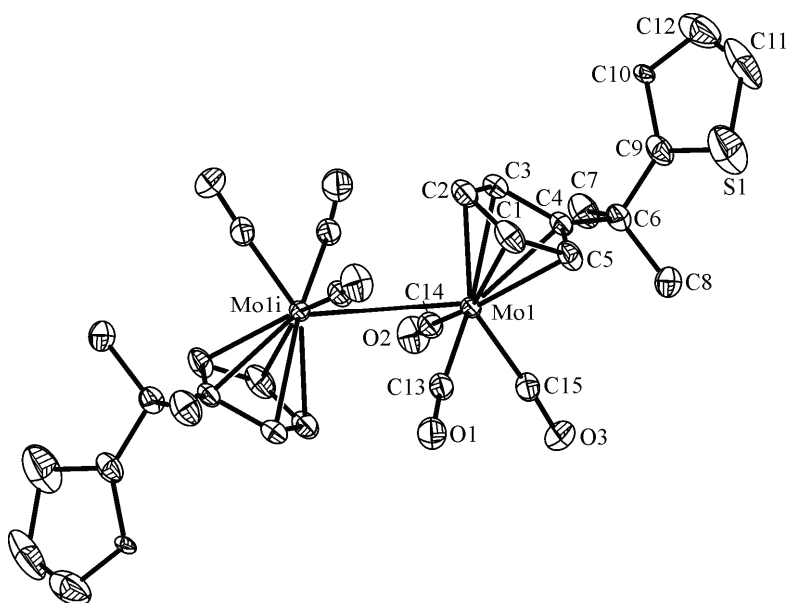


Fig. 1. Molecular structure of complex 5.

Ellipsoids correspond to 30 % probability.  
Hydrogen atoms are omitted for clarity

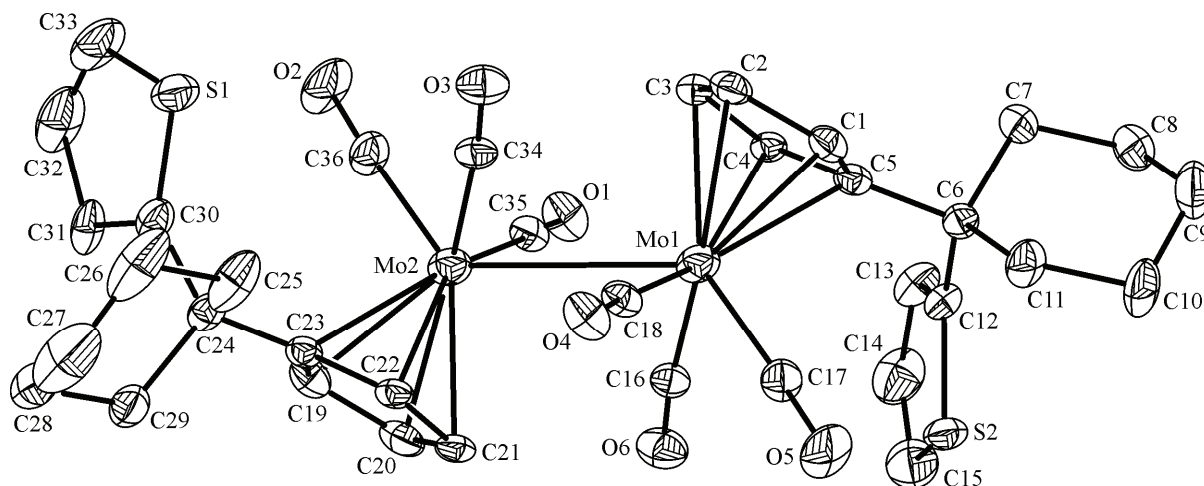


Fig. 2. Molecular structure of complex **8**.

Ellipsoids correspond to 30 % probability. Hydrogen atoms are omitted for clarity

bond lengths in the same cyclopentadienyl ring are 1.442 Å and 1.370 Å respectively. As for  $[(\eta^5\text{-C}_5\text{H}_4\text{CR}_1\text{R}_2\text{C}_4\text{H}_3\text{S})\text{Mo}(\text{CO})_3]_2$  [ $\text{R}_1 = \text{R}_2 = \text{CH}_3$  (**5**);  $\text{R}_1, \text{R}_2 = (\text{CH}_2)_5$  (**8**)], the longest and the shortest C—C bond lengths are 1.431(9) Å (**5**), 1.431(5) Å (**8**) and 1.400(8) Å (**5**), 1.408(5) Å (**8**) respectively. These data reveal that the substituents introduced make the aromaticity of the cyclopentadienyl ring enhanced [ 14 ].

The bond distance data for Mo—Mo in (**5**) and (**8**) are 3.242 Å and 3.2241(6) Å respectively; they are little longer than the bonds in *trans*- $[(\eta^5\text{-C}_5\text{H}_4(\text{i-Pr})\text{Mo}(\text{CO})_3]_2$  [3.222(5) Å] [ 14 ] and *trans*- $[(\eta^5\text{-C}_5\text{H}_4\text{Ph})\text{Mo}(\text{CO})_3(\mu\text{-CN})]_2$  [3.212(1) Å] [ 15 ], but shorter than those in *trans*- $[(\eta^5\text{-C}_5\text{Me}_5)\text{Mo}(\text{CO})_3]_2$  [3.281(1) Å] [ 16 ], *trans*- $[(\eta^5\text{-C}_5\text{Me}_4\text{PhMe})\text{Mo}(\text{CO})_3]_2$  [3.283 Å], and *trans*- $[(\eta^5\text{-C}_5\text{Me}_4\text{PhOMe})\times \times \text{Mo}(\text{CO})_3]_2$  [3.307 Å] [ 17 ]. The bond distance data show that cyclopentadienyl substituents have a certain effect on the Mo—Mo bond length.

## CONCLUSIONS

Four new dinuclear metal carbonyl complexes have been synthesized by reactions of thienyl side-chain-functionalized cyclopentadienyl with  $\text{Mo}(\text{CO})_6$  in refluxing xylene. The results clearly reveal that the ligands coordinate only to the Mo(I) center by  $\eta^5$  mode for cyclopentadienyl rather than by  $\eta^1$  mode to the S atom because of the weak S-donor thiophene ligand. The substituent introduced can make the aromaticity of the cyclopentadienyl ring change. The Cp\* (where Cp\* = substituted cyclopentadienyl ligand) ligands are *trans* in the dimeric structures in the solid state. A change in the substituents has a certain effect on the Mo—Mo bond length.

**Supplementary materials.** Crystallographic data for the structural analysis have been deposited with the Cambridge Crystallographic Data Centre, CCDC Nos. 760943 and 779684 for complexes (**5**) and (**8**), respectively. Copies of this information may be obtained free of charge from The Director, CCDC, 12 Union Road, Cambridge CB2 1EZ, UK, fax: +44 1223 336 033, e-mail: deposit@ccdc.cam.ac.uk or www: <http://www.ccdc.cam.ac.uk>.

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