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Synthesis and characterization of dinuclear *p*-, *m*- and *o*-xylyl bridged complexes of molybdenum and tungsten. The crystal structures of μ-*p*-xylyl-*bis*(η^5-pentamethyl-cyclopentadienyltricarbonylmolybdenum) and μ-m-xylyl-bis(η^5-pentamethylcyclopentadienyltricarbonyltungsten)

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Abstract—The reactions of η^5-Cp*M(CO)$_3$Na (M = Mo, W) with α,α'-*p*-, *m*- and *o*-dichloro-xylenes yielded *p*-, *m*- and *o*-xylyl bridged dinuclear complexes of η^5-Cp*M(CO)$_3$ in high yields. All of such new complexes are stable to air and water, even stable in dilute acids and bases. © 1997 Elsevier Science Ltd

Keywords: molybdenum; tungsten; carbonyls; xylyl bridged complexes

A considerable number of σ- and π-allyl derivatives [1–2] as well as σ- and π-benzyl derivatives [3–4] of transition metals have been described, none of them to stable to air and water. Recently we prepared a series of dinuclear xylyl bridged complexes of molybdenum [5–6], which possess physical and chemical properties that are quite different from those of the first benzyl type complexes of molybdenum [η^5-Cp(CO)$_3$)MoCH$_2$C$_6$H$_5$]^3 which was prepared by King in 1967. However, some of these xylyl bridged complexes of molybdenum are not soluble in common organic solvents. For example, α,α'-μ-*p*-xylyl-*bis* (cyclopentadienyltricarbonylmolybdenum) [6] does not dissolve in ether and THF, and almost not in acetone and dichloro-methane. On the other hand, the corresponding complex α,α'-μ-*p*-xylyl-*bis*(pentamethylcyclopentadienyltricarbonylmolybdenum) is not only stable to air and water, also quite soluble in organic solvents such as chloroform and acetone etc. We believe that complexes **1–6** are suitable starting materials for photochemical reactions to see if a double σ- to π-benzyl rearrangement is possible [3,7]. Now we wish to report xylyl bridged complexes of molybdenum and tungsten.

EXPERIMENTAL

Physical measurements

Infrared spectra were recorded on Jasco FT/IR 300 E spectrometer. ^{1}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 done at the Analysis Center of National Cheng Kung University.

Materials and preparations

Mo(CO)$_6$(Aldrich). W(CO)$_6$ (Aldrich), η^5-Cp*Na (Aldrich), α,α'-dichloro-*p*-xylene (Aldrich), α,α'-dichloro-m-xylene (Aldrich) and α,α'-dichloro-o-xylene

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(Aldrich) and organic solvents were routinely purified by standard procedures [8].

α,α′-μ-p-xylyl-bis(pentamethylcyclopentadienyltricarbonylmolybdenum) (**1**)

A three-necked 100 cm^3 round bottom flask was equipped with a dry ice bath, a reflux condenser topped with a nitrogen inlet, an addition funnel, a magnetic stirrer and a one-inch teflon-coated stirring bar. Into this flask was placed 0.88 g (0.0033 mol) of $Mo(CO)_6$, 6.7 mL of Cp*Na (0.5 M in THF) and 40 cm^3 of dry THF. The mixture was heated to reflux for 14 h. The resulting orange solution was cooled to room temperature. With stirring 0.31 g (0.00177 mol) of α,α′-dichloro-*p*-xylene was added. The mixture was stirred for 1 h. After removal of the solvent the residue was washed with distilled water (3 × 10 cm^3) and benzene (3 × 10 mL). The resulting product was dried *in vacuo* overnight to afford complex **1** as a yellow solid (0.98 g, 81%). m.p. 131–135°C (dec.). Found: c, 55.39; H, 5.30. Calc. for $C_{34}H_{38}Mo_2O_6$: C, 55.58; H, 5.18. (Mass (*m*/*z*): 736 (M+2), 734 (M), 678 (M—2CO), 622 (M—4CO), 594 (M—5CO), 566 (M—6CO).

α,α′-μ-m-xylyl-bis(pentamethylcyclopentadienyltricarbonylmolybdenum) (**2**)

The procedure described above was followed by using 0.88 g (0.0033 mol) of $Mo(CO)_6$, 6.7 cm^3 (0.5 M in THF) of Cp*Na, 0.31 g (0.00167 mol) of α,α′-dichloro-*m*-xylene and 30 cm^3 of dry THF. The complex **2** was isolated as a yellow solid (0.87 g, 72%). m.p. 119–123°C (dec.). Found: 55.43; H, 5.26. Calc. for $C_{34}H_{38}Mo_2O_6$: 55.58; H, 5.18 Mass (*m*/*z*): 736 (M+2), 734 (M), 706 (M—CO), 678 (M—2CO), 622 (M—4CO), 566 (M—6CO).

α,α′-μ-o-xylyl-bis(pentamethylcyclopentadienyltricarbonylmolybdenum) (**3**)

The procedure described above was followed by using 0.88 g (0.0033 mol) of $Mo(CO)_6$, 6.7 mL (0.5 M in THF) of Cp*Na, 0.31 g (0.00167 mol) of α,α′-dichloro-*o*-xylene and 30 cm^3 of dry THF. The complex **3** was isolated as a yellow solid (0.55 g, 46%). m.p. 102–105°C (dec.) found: C, 55.39; H, 5.28. Calc. for $C_{34}H_{38}Mo_2O_6$: C, 55.58; H, 5.18. Mass (*m*/*z*): 736 (M+2), 734 (M), 678 (M—2CO), 622 (M—4CO), 566 (M—6CO).

α,α′-μ-p-xylyl-bis(pentamethylcyclopentadienyltricarbonyltungsten) (**4**)

The procedure described above was followed by suing 1.17 g (0.0033 mol) of $W(CO)_6$, 6.7 mL (0.5 M in THF) of Cp*Na, 0.31 g (0.00167 mol) of α,α′-dichloro-*p*-xylene and 30 cm^3 of dry THF. The complex **4** was isolated as a yellow solid (1.17 g, 78%). m.p. 164–169°C (dec.). Found: C, 44.76; H, 4.27. Calc. for $C_{34}H_{38}W_2O_6$: 44.83; H, 4.16 Mass (*m*/*z*): 912 (M+2), 910 (M), 882 (M—CO), 826 (M—3CO), (M—4CO).

α,α′-μ-m-xylyl-bis(pentamethylcyclopentadienyltricarbonyltungsten) (**5**)

The procedure described above was followed by using 1.17 g (0.0033 mol) of $W(CO)_6$, 6.7 mL (0.5 M in THF) of Cp*Na, 0.31 g (0.00167 mol) of α,α′-dichloro-*m*-xylene and 30 cm^3 of dry THF. The complex **5** was isolated as a yellow solid (1.06 g, 71%). m.p. 138–142°C (dec.). Found: C, 44.81; H, 4.29. Calc. for $C_{34}H_{38}W_2O_6$: C, 44.83; H, 4.16. Mass (*m*/*z*): 912 (M+2), 910 (M), 882 (M—CO), 854 (M—2CO), 826 (M—3CO), 798 (M—4CO), 742 (M—6CO).

α,α′-μ-o-xylyl-bis(pentamethylcyclopentadienyltricarbonyltungsten) (**6**)

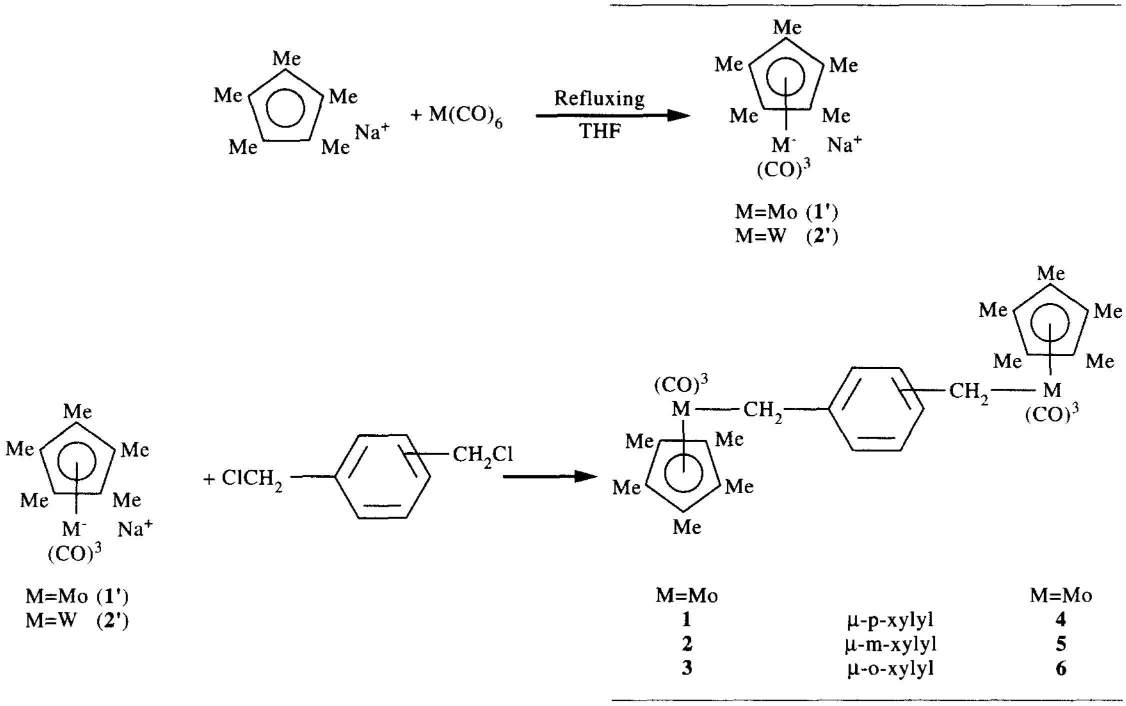
The procedure described above was followed by using 1.17 g (0.0033 mol) of $W(CO)_6$, 6.7 cm^3 (0.5 M in THF) of Cp*Na, 0.31 g (0.00167 mol) of α,α′-dichloro-o-xylene and 30 mL of dry THF. The complex **6** was isolated as a yellow solid (0.87 g, 58%). m.p. 121–124°C (dec.). Found: C, 44.84; H, 4.34. Calc. for $C_{34}H_{38}W_2O_6$: 44.83; H, 4.16. Mass (*m*/*z*): 911 (M+1), 910 (M), 854 (M—4CO), 742 (M—6CO).

X-ray single-crystal structural determination

Yellow crystals of complex **1** and **5** were prepared by diffusion of hexane into a concentrated CH_2Cl_2 solutions of **1** and **5** under nitrogen at 4°C. The single crystal X-ray diffraction measurements were performed on a Nonious CAD-4 automated diffractometer using graphite monochromated Mo-Kα radiation. 25 High-angle reflections ($14.53 < 2\theta < 30.91°$) (for complex **1**) and ($16.92 < 2\theta < 36.02°$) (for complex **5**) were used, respectively, in a least-squares fit to obtain accurate cell constants. The monoclinic space group $P2_1/n$ and orthorombic space group *P bca* were assigned on the basis of the systematic absences. Diffraction intensities were collected up to $2\theta < 49.8°$ (for complex **1**) and $2\theta < 44.8°$ (for complex **5**) using $\theta/2\theta$ scan technique. The reflections with $I_0 > 2.0\sigma\ (I_0)$ 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 ψ scan was applied to the data. The structure was solved by direct methods [9], and refined by a full-matrix least-squares routine [10] with anisotropic thermal parameters for all non-hydrogen atoms. All of the 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 2–4.

RESULTS AND DISCUSSION

The reactions of η^5-Cp*M(CO)$_3$Na (M = Mo, W) with α,α'-p-,α,α'-m- and α,α'-*o* dichloroxylenes in 2 : 1 molar proportions lead to formation of *anti*-type xylyl bridged dinuclear complexes of molybdenum (**1**–**3**) and tungsten (**4**–**5**) in high yields. All of these complexes are soluble in CH_2Cl_2 and $CHCl_3$, and stable to air and water, even stable in dilute bases ($\sim 10^{-2}$ M in ethanol). The precursors **1′** and **2′** were prepared from the reactions η^5-Cp*Na with Mo(CO)$_6$ and W(CO)$_6$, respectively:



Complexes (**1**–**6**) are not soluble in non-polar organic solvents such as hexane and benzene. However, they are quite soluble in CH_2Cl_2, $CHCl_3$, ethyl acetate and actone. In general, the solubility of complexes (**1**–**6**) varies in following order:

$$o > m > p$$

$$W > \mathrm{Mo}$$

A characteristic feature of all these complexes is the presence of two intense ν(CO) bands in the region 2010–1908 cm^{-1}[5–7] (see Table 6). In addition, ^{1}H-NMR spectra also reveal characteristic upfield shifts for the methylene protons upon complexation to the moiety Cp*M(CO)$_3$ coordination chemical shift is about 2.3 ppm (i.e. from ~4.5 to ~2.2 ppm) [5–7]. We have also tried hard to take variable temperature ^{1}H NMR for complexes **1**–**6**, unfortunately, we failed to observe fluxional behavior for them. The ^{1}H NMR data for 1–6, see Table 5.

The crystal structure of complex **1** and complex **5** consist of discrete molecules of *p*-$C_6H_4[CH_2Mo(CO)_3Cp^*]_2$ and m-$C_6H_4[CH_2W(CO)_3Cp^*]_2$. Figure 1 is the ORTEP plot of *p*-$C_6H_4[CH_2Mo(CO)_3Cp^*]_2$. The main molecular plane can be considered as that of p-$C_6H_4(CH_2)$ with two Cp*Mo(CO)$_3$ at the anti-position. The Mo—C(O) lengths average 1.976(4) Å is very close to the average corresponding lengths in the complex $[\{Mo(CO)_3\}_2(\eta^6,\eta'^6C_{14}H_{14}]$[11] of 1.962(5) Å. The mean Mo—C distance for the Cp* ligand of 2.349(3) Å is quite close to the corresponding length in the $Cp^*_2Mo_2(\mu\text{-}S_2)(\mu\text{-}S)_2$ of 2.344(2) Å [12]. The mean Mo—C(H$_2$) distance in **1** of 2.366(3) Å is very close to that in the complex $[Cp'Mo(CO)_3]_2(\mu\text{-}o\text{-}CH_2C_6H_4CH_2)$ of 2.383(4) Å [5]. Figure 2 is the ORTEP plot of m-$C_6H_4[CH_2W(CO)_3Cp^*]_2$, the plane made up of *m*-$C_6H_4(CH_2)_2$ also with two Cp*W(CO)$_3$ at the anti-positions. The W—C(O) lengths average 1.955(13) Å is a little longer than the average corresponding length in $[FvW_2(CO)_6]^{-2}$ (Fv = Fulvalene) of 1.929(5) Å [10]. The mean W—C distance for the Cp* ligand of 2.342(11) Å is very close to the average corresponding distances in *p*-$C_6H_4[CH_2W(CO)_3Cp^*]_2$ of 2.348(8) Å. The mean W—C(H$_2$) distance for **5**, 2.3605(11) Å, is almost identical with the average corresponding distance in *p*-$C_6H_4[CH_2W$

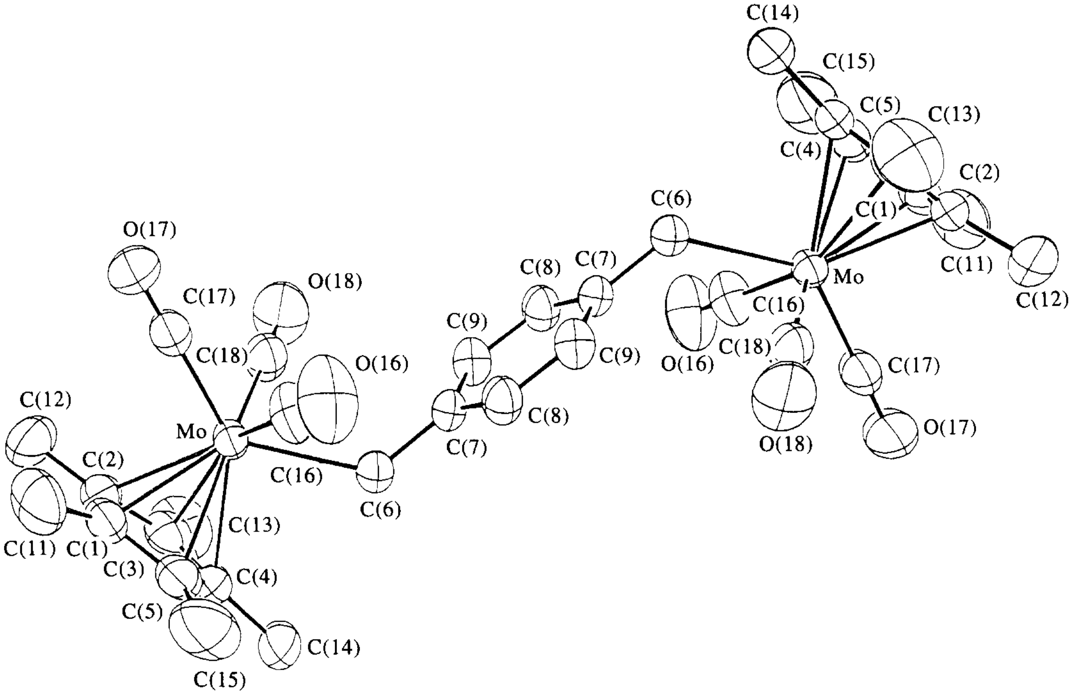


Fig. 1. Molecular Structure of Complex **1**.

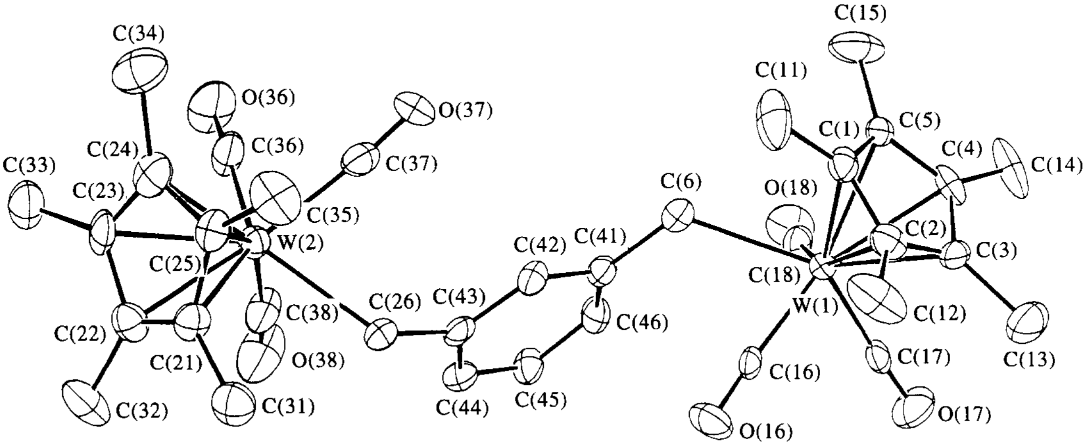


Fig. 2. Molecular Structure of complex **5**.

Table 1. Crystallographic data and refinement details for complexes **1** and **5**

	1	**5**
Empirical formula	$C_{34}H_{38}Mo_2O_6$	$C_{34}H_{38}W_2O_6$
Molar mass (g)	734	910
Crystal system	monoclinic	orthorhombic
Space group	$P2_1/n$	$P\,bca$
Crystal size (mm^3)	$0.56 \times 0.25 \times 0.25$	$0.50 \times 0.50 \times 0.31$
a (Å)	8.8179(22)	15.869(3)
b (Å)	12.8968(16)	14.2852(8)
c (Å)	15.3531(13)	29.0731(20)
β (°)	103.346(14)	
V (Å^3)	1698.8(5)	6590.5(12)
Z	4	8
T (K)	298	298
D_{calc} (g cm^{-3})	1.436	1.835
λ (Å)	0.70930	0.70930
μ (mm^{-1})	7.587	71.666
F (000)	748	3503
Scan type	$\theta/2\theta$	$\theta/2\theta$
2θ max (°)	50.0	45.0
h, k, l range	(−10; 10) (0; 15) (0; 18)	(0; 17) (0; 15) (0;31)
No. of unique reflections	2992	4281
No. of data with I > 2σ(I)	2463	3251
$R_F{}^a$	0.031	0.036
$R_W{}^a$	0.037	0.043
GOF	1.54	1.86

[a] $R_F = \Sigma(F_o - F_c)/\Sigma(F_o)$; $R_W = [\Sigma\theta(F_o - F_c)^2/\Sigma(\omega F_o^2)]^{1/2}$

Table 2. Bond distances (Å) for complexes **1** and **5**

1		**5**	
Mo—C(1)	2.322(3)	W(1)—C(1)	2.389(11)
Mo—C(2)	2.322(3)	W(1)—C(2)	2.346(11)
Mo—C(3)	2.354(3)	W(1)—C(3)	2.307(11)
Mo—C(4)	2.391(3)	W(1)—C(4)	2.308(10)
Mo—C(5)	2.359(4)	W(1)—C(5)	2.323(10)
Mo—C(6)	2.366(3)	W(1)—C(6)	2.355(11)
Mo—C(16)	1.980(4)	W(1)—C(16)	1.972(12)
Mo—C(17)	1.969(4)	W(1)—C(7)	1.959(14)
Mo—C(18)	1.979(4)	W(1)—C(18)	1.935(14)
C(1)—C(2)	1.410(6)	W(2)—C(21)	2.392(12)
C(1)—C(5)	1.416(6)	W(2)—C(22)	2.340(12)
C(2)—C(3)	1.424(5)	W(2)—C(23)	2.321(11)
C(3)—C(4)	1.407(6)	W(2)—C(24)	2.299(12)
C(5)—C(6)	1.403(6)	W(2)—C(25)	2.338(12)
C(6)—C(7)	1.480(5)	W(2)—C(26)	2.366(11)
		C(1)—C(2)	1.376(17)
		C(1)—C(5)	1.414(17)
		C(2)—C(3)	1.385(20)
		C(3)—C(4)	1.439(18)
		C(4)—C(5)	1.404(17)
		C(21)—C(22)	1.363(18)
		C(21)—C(25)	1.428(19)
		C(22)—C(23)	1.447(19)
		C(23)—C(24)	1.410(21)
		C(24)—C(25)	1.472(19)

Table 3. Selected bond angles (°) for complex **1** and complex **5**

1		**5**	
C(1)—Mo—C(16)	105.72(15)	C(1)—W(1)—C(16)	106.4(4)
C(1)—Mo—C(17)	89.57(14)	C(1)—W(1)—C(17)	143.3(4)
C(1)—Mo—C(18)	139.18(15)	C(1)—W(1)—C(18)	128.5(5)
C(2)—Mo—C(16)	139.99(15)	C(2)—W(1)—C(16)	91.4(4)
C(2)—Mo—C(17)	89.40(14)	C(2)—W(1)—C(17)	112.8(5)
C(2)—Mo—C(18)	105.14(15)	C(2)—W(1)—C(18)	155.9(4)
C(3)—Mo—C(16)	151.30(17)	C(3)—W(1)—C(16)	110.1(4)
C(3)—Mo—C(17)	121.17(16)	C(3)—W(1)—C(17)	86.1(4)
C(3)—Mo—C(18)	95.73(15)	C(3)—W(1)—C(18)	130.5(5)
C(4)—Mo—C(16)	117.42(17)	C(4)—W(1)—C(16)	146.3(4)
C(4)—Mo—C(17)	145.63(13)	C(4)—W(1)—C(17)	94.7(4)
C(1)—Mo—C(18)	119.18(16)	C(4)—W(1)—C(18)	99.1(4)
C(5)—Mo—C(16)	94.94(16)	C(5)—W(1)—C(16)	141.1(4)
C(5)—Mo—C(17)	120.94(15)	C(5)—W(1)—C(17)	129.2(4)
C(5)—Mo—C(18)	152.61(16)	C(5)—W(1)—C(18)	98.9(4)
C(6)—Mo—C(16)	75.65(15)	C(21)—W(2)—C(36)	144.0(5)
C(6)—Mo—C(17)	133.35(14)	C(21)—W(2)—C(37)	121.2(5)
C(6)—Mo—C(18)	75.03(19)	C(21)—W(2)—C(38)	114.5(6)
C(16)—Mo—C(17)	79.11(19)	C(22)—W(2)—C(36)	117.4(5)
C(16)—Mo—C(18)	109.33(17)	C(22)—W(2)—C(37)	151.9(5)
C(17)—Mo—C(18)	77.33(17)	C(22)—W(2)—C(38)	95.2(5)
Mo—C(6)—C(7)	124.75(23)	C(23)—W(2)—C(36)	86.2(5)
Mo—C(16)—O(16)	176.6(4)	C(23)—W(2)—C(37)	132.8(5)
Mo—C(17)—O(17)	178.3(4)	C(23)—W(2)—C(38)	108.7(5)
Mo—C(18)—O(18)	176.2(4)	C(24)—W(2)—C(36)	89.7(5)
		C(24)—W(2)—C(37)	99.7(5)
		C(24)—W(2)—C(38)	143.8(6)
		C(25)—W(2)—C(36)	124.0(5)
		C(25)—W(2)—C(37)	93.2(5)
		C(25)—W(2)—C(38)	149.6(6)
		W(1)—C(6)—C(41)	123.0(8)
		W(1)—C(16)—O(16)	176.7(9)
		W(1)—C(17)—O(17)	177.3(10)
		W(1)—C(18)—O(18)	174.4(10)
		W(2)—C(26)—O(43)	122.0(7)
		W(2)—C(36)—O(36)	176.4(13)
		W(2)—C(37)—O(37)	173.6(11)
		W(2)—C(38)—O(38)	173.6(13)

Table 4. Selected Bond distances (Å) for μ-*p*-xylyl-[η^5-Cp*W(CO)$_3$]$_2$

W(1)—C(1)(Cp*)	2.372(8)
W(1)—C(2)(Cp*)	2.330(8)
W(1)—C(3)(Cp*)	2.337(8)
W(1)—C(4)(Cp*)	2.358(8)
W(1)—C(5)(Cp*)	2.385(8)
W(2)—C(21)(Cp*)	2.332(8)
W(2)—C(22)(Cp*)	2.389(8)
W(2)—C(23)(Cp*)	2.368(9)
W(2)—C(24)(Cp*)	2.317(8)
W(2)—C(25)(Cp*)	2.329(8)
W(1)—C(6)(CH$_2$)	2.351(8)
W(2)—C(26)(CH$_2$)	2.372(8)
W(1)—C(16)(O)	1.969(10)
W(1)—C(17)(O)	1.976(9)
W(1)—C(18)(O)	1.982(9)
W(2)—C(36)(O)	1.989(9)
W(2)—C(37)(O)	1.962(10)
W(2)—C(38)(O)	1.996(10)

Table 5. ^{1}H-NMR spectra data compounds **1**–**6**

Compounds^a	Phenylene	Methylene	Methyl (ppm)
1	7.01 (s, 4H)	2.22 (s, 4H)	1.91 (s, 30H)
2	6.92–6.77 (m, 4H)	2.21 (s, 4H)	1.92 (s, 30H)
3	6.90–6.74 (m, 4H)	2.20 (s, 4H)	1.91 (s, 30H)
4	6.94 (s, 4H)	2.35 (s, 4H)	2.03 (s, 30H)
5	7.09–6.74 (m, 4H)	2.18 (s, 4H)	2.10 (s, 30H)
6	7.07–6.73 (m 4H)	2.25 (s, 4H)	2.12 (s, 30H)

^{a}Measured in $CDCl_3$ solutions.

Table 6. Infrared spectra data for compounds **1**–**6**

Compounds^a	ν(C=O) (cm^{-1})
1	2002 (s), 1919 (vs)
2	2003 (s), 1921 (vs)
3	2005 (s), 1923 (vs)
4	1992 (s), 1911 (vs)
5	1996 (s), 1912 (vs)
6	1998 (s), 1914 (vs)

^{a}Measured in CH_2Cl_2 solutions.

$(CO)_3Cp^*]$ of 2.3615(8) Å (see Table 4). Furthermore, all structural parameters are very reasonable if compared to the literature values [11–13].

In summary, we have prepared and characterized the α,α'-*p*-, *m*- and *o*-xylyl bridged dinuclear new complexes of molybdenum and tungsten **1**–**6**. We also identified the molecular structures of complexes **1** and **5** to be anti conformers in solid states.

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