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Synthesis and characterization of the linear M—Hg—M trimetallic complexes [η^5-$Bz_5C_5(CO)_3M]_2Hg$ and [η^5-$HO_2CC_5H_4(CO)_3M]_2Hg$ (M = Cr, Mo, W). The X-ray crystal structure of [η^5-$Bz_5C_5(CO)_3W]_2Hg$

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Abstract—The η^5-Bz_5C_5 ligand-containing trimetallic complexes [η^5-$Bz_5C_5(CO)_3M]_2Hg$ (M = Cr, Mo, W) were synthesized from the *in situ* reaction of phenylmercuric chloride with corresponding transition metal lithium salts [η^5-$Bz_5C_5(CO)_3M$] Li in THF, whereas the η^5-$HO_2CC_5H_4$ ligand-containing trimetallic complexes [η^5-$HO_2CC_5H_4(CO)_3M]_2Hg$ were synthesized through hydrolysis of their corresponding esters in the presence of KOH/EtOH/H_2O, followed by acidification. These compounds were characterized by elemental analysis, IR, ^1H NMR and MS spectroscopy, while the structure of [η^5-$Bz_5C_5(CO)_3W]_2Hg$ was determined by an X-ray diffraction analysis. Copyright © 1996 Elsevier Science Ltd

Keywords: η^5-pentabenzylcyclopentadienyl; η^5-carboxycyclopentadienyl; group 6 metals; mercury; crystal structure.

In view of the theoretical and practical importance of metal–metal bonded compounds [1], the organometallic complexes containing M—Hg—M (M = Cr, Mo, W) structural moieties have so far received considerable attention [2–4]. However, although the trimetallic M—Hg—M complexes containing mono-substituted cyclopentadienyl ligand have been known for several years [2–4], the study concerning their chemical reactions and synthesis regarding the M—Hg—M complexes containing poly-substituted cyclopentadienyl ligand has not been reported as yet. In order to further understanding of the properties of this kind of complex containing mono-substituted cyclopentadienyl ligand and to prepare the novel M—Hg—M complexes carrying η^5-pentabenzylcyclopentadienyl ligand η^5-Bz_5C_5, we initiated this study. Herein we report the synthesis and characterization of the new complexes [η^5-$HO_2CC_5H_4(CO)_3M]_2Hg$ and [η^5-$Bz_5C_5(CO)_3M]_2Hg$ (M = Cr, Mo, W) including the single-crystal structure of [η^5-$Bz_5C_5(CO)_3W]_2Hg$ determined by X-ray diffraction analysis.

RESULTS AND DISCUSSION

Synthesis and characterization of [η^5-$HO_2CC_5H_4(CO)_3M]_2Hg$ (2a–2c)

On the basis of our work for synthesizing [η^5-$RC_5H_4(CO)_3M]_2Hg$ (M = Cr, Mo, W; R = CO_2Me, CO_2Et) (**1**) [3], an effort has been made to study further the preparation of their hydrolysis derivatives **2**. Fortunately, it was found that the M—Hg—M structural unit in the starting complexes **1** is quite stable towards both acidic and alkaline conditions; the starting complexes **1**, after the action of NaOH/EtOH/H_2O and followed by acidification of the intermediate potassium salt with HCl/H_2O, gave rise to the cor-

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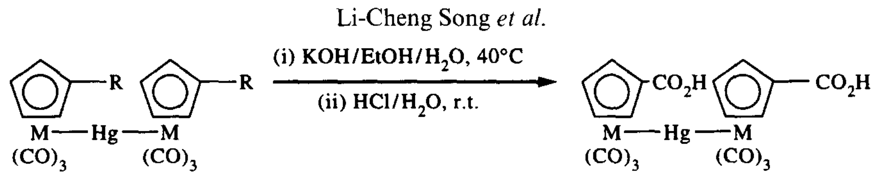


1 **2**

$R = CO_2Me, CO_2Et$

2a : M = Cr; **2b** : M = Mo; **2c** ; M = W

Scheme 1.

responding derivatives **2** in moderate yields, as shown in Scheme 1.

Products **2a**–**2c** are air-stable yellow solids and soluble in THF, DMSO and acetone but not in CH_2Cl_2 and other non-polar solvents. All of them were fully characterized by elemental analysis, IR, ^1H NMR and mass spectroscopies. In the IR spectra of **2a**–**2c**, there are three strong to very strong absorption bands in the range of 1884–1967 cm^{-1}, characteristic of their terminal carbonyls, one strong absorption band at about 1680 cm^{-1} and one broad band at about 3430 cm^{-1} characteristic of their carbonyl and hydroxyl group in the carboxylic substituent, respectively. Their ^1H NMR spectra have a pair of apparent triplets, in which the downfield triplet has been assigned to the H(2, 5) protons of the substituted cyclopentadienyl ring, while the upfield triplet is assigned to the H(3, 4) protons. Additionally, the triplet assigned to H(2, 5) or H(3, 4) is located in the lowest field in the case of the tungsten compound **2c**, the intermediate field for the molybdenum compound **2b** and in the highest field for the chromium compound **2a** and there are larger differences in chemical shifts between the corresponding triplets of **2a** and **2b** than between those of **2b** and **2c**. It follows that the ^1H NMR behaviour of the functionally substituted cyclopentadienyl ring of **2a**–**2c** are similar to that of starting complexes and other electron-withdrawing functional analogs [2, 3, 5]. It is worth pointing out that the ^1H NMR spectrum of the carboxylic functionality was not observed in the field as low as $\delta = 15$ ppm, perhaps due to the D/H exchange between **2** and the solvent DMSO.

Synthesis and characterization of $[\eta^5\text{-}Bz_5C_5(CO)_3M]_2Hg$ (**4a**–**4c**)

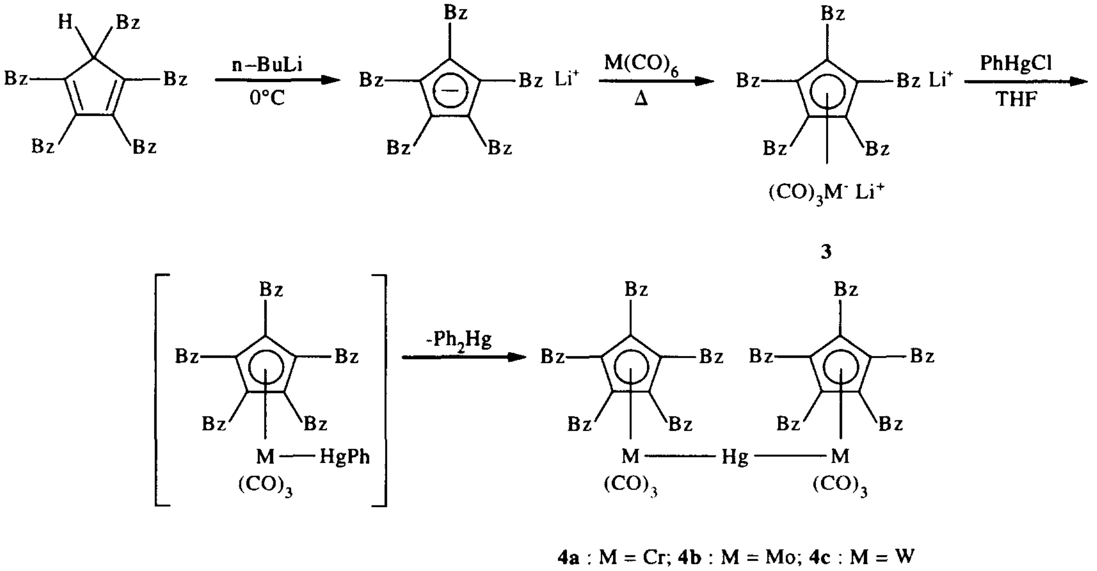
Similar to the lithium or sodium salts of monosubstituted cyclopentadienyltricarbonyl group 6 metal anions [2–4], the pentabenzylcyclopentadienyl analogs $\eta^5\text{-}Bz_5C_5(CO)_3MLi$ (**3**) derived from sequential reactions of pentabenzylcyclopentadiene, *n*-BuLi and group 6 metal carbonyls $M(CO)_6$, reacted with phenylmercuric chloride in THF to give the expected trimetallic compounds **4a**–**4c**. According to the related mechanistic study [3], the compounds **4a**–**4c** were most likely produced by a symmetrization process of the active bimetallic intermediate $\eta^5\text{-}Bz_5C_5M(CO)_3\text{-}HgPh$ formed by a condensation reaction of **3** with PhHgCl, as shown in Scheme 2.

Compounds **4a**–**4c** are air-stable yellow crystalline solids, whose melting points increase in the order of **4a** < **4b** < **4c**. The yields of **4a**–**4c** are much lower than those of the analogs containing $\eta^5\text{-}C_P$ or $\eta^5\text{-}RC_5H_4$ ligand [2–4], the reason for which could be attributed to the special electronic and steric effects of $\eta^5\text{-}Bz_5C_5$ ligand [6–10] relative to those of $\eta^5\text{-}C_P$ and η^5-mono-substituted cyclopentadienyl ligand. For **4a**–**4c** all the characterizing data of the elemental analysis, IR and ^1H NMR spectroscopy are in good agreement with the structures shown in Scheme 2. However, it is worth noting that compounds **4a**–**4c** contain not only terminal carbonyls, but also bridging carbonyls. This is because in each of their IR spectra there is one strong absorption band at about 1860 cm^{-1} besides two absorption bands at the region of 1884–1959 cm^{-1}.

X-ray crystallography

In order to determine the structure of compounds **4a**–**4c** and establish the type of carbonyls in these compounds, an X-ray diffraction analysis for **4c** was undertaken. To the best of our knowledge, this is the first single-crystal molecular structure determination of an M—Hg—M trinuclear organometallic containing $\eta^5\text{-}Bz_5C_5$ ligand, although those of $\eta^5\text{-}Bz_5C_5$-containing mononuclear [8–10] and dinuclear [11] organometallics and $\eta^5\text{-}C_P$ [2, 12] or $\eta^5\text{-}RC_5H_4$ [3,4] containing M—Hg—M organometallics are known. Table 1 lists the crystallographic data. Table 2 and Table 3 list the selected bond lengths and bond angles, respectively. Figure 1 shows its molecular structure.

As seen from Fig. 1 complex **4c** consists of one mercury atom and two identical organometallic structural units $\eta^5\text{-}Bz_5C_5(CO)_3W$. The central Hg atom is located on a crystallographic inversion center so that the W—Hg—W skeleton is exactly linear ($\angle W\text{—}Hg\text{—}W = 180°$) and two substituted cyclopentadienyl rings are parallel and lie in a *trans* position with respect to the linear W—Hg—W axis. This is also the case for the ethoxycarbonylcyclopentadienyl molybdenum analog [3] but different from its parent compound, which is non-centrosymmetric and has a *cis* configuration of cyclopentadienyl ligands with respect to the W—Hg—W axis [2]. The bond angles



4a : M = Cr; **4b** : M = Mo; **4c** : M = W

Scheme 2.

Table 1. Crystal data and intensity collection details for **4c**

Empirical formula	$C_{86}H_{70}HgO_6W_2$
Formula weight	1767.79
Crystal system	monoclinic
Space group	$P2_1/n$(#14)
Lattice parameters	
a (Å)	12.536(1)
b (Å)	15.180(4)
c (Å)	18.322(4)
β (°)	94.04(1)
V (Å^3)	3478(1)
Z	2
D_{calc} (g cm^{-3})	1.69
F(000)	1724
μ (Mo-K_α) (cm^{-1})	56.25
Radiation	Mo-K_α, $\lambda = 0.71069$ Å graphite-monochromated
Temperature (°C)	23
$2\theta_{max}$(°)	49.9
No. of observation ($I \geq 3\sigma(I)$)	4202
No. of variables	430
Residuals R and Rw	0.042, 0.047
Goodness of fit indicator	1.07
Maximum shift in final cycle	0.00
Largest peak in final diff. map (eÅ^{-3})	0.80

Table 2. Selected bond lengths (Å) for **4c**

W—Hg	2.7532(7)	C(2)—C(3)	1.44(1)
W—C(03)	1.93(1)	C(2)—C(20)	1.52(1)
W—C(01)	1.94(1)	C(3)—C(4)	1.45(1)
W—C(02)	1.98(1)	C(3)—C(30)	1.48(1)
W—C(5)	2.310(9)	C(4)—C(5)	1.43(1)
W—C(1)	2.325(9)	C(4)—C(40)	1.51(1)
W—C(4)	2.336(8)	C(5)—C(50)	1.52(1)
W—C(2)	2.362(9)	C(10)—C(11)	1.51(1)
W—C(3)	2.376(8)	C(11)—C(16)	1.35(1)
O(1)—C(01)	1.16(1)	C(11)—C(12)	1.40(2)
O(2)—C(02)	1.16(1)	C(12)—C(13)	1.35(2)
O(3)—C(03)	1.17(1)	C(13)—C(14)	1.36(2)
C(1)—C(2)	1.42(1)	C(14)—C(15)	1.33(2)
C(1)—C(5)	1.44(1)	C(15)—C(16)	1.36(2)
C(1)—C(10)	1.52(1)	Hg—C(01)	2.843(9)

W—C(01)—O(1) and W—C(02)—O(2) [174(1) and 177(1)°, respectively] and Hg—W—C(01) and Hg—W—C(02) [72.2(3) and 69.9(3)°, respectively] indicate that the C(01)—O(1) and C(02)—O(2) carbonyl ligands are linear semi-bridging. Since the W—C(01) and Hg···C(01) distances D_1 and D_2 are 1.94(1) and 2.84(3) Å, for C(01)—O(1) the bridge asymmetry parameter $\alpha = 0.47$; since W—C(02) and Hg···C(02) distances D_1 and D_2 are 1.98(1) and 2.78(6) Å, for C(02)—O(2) $\alpha = 0.41$ ($\alpha = D_2 - D_1/D_1$) [13–14]. Apparently, the second W atom also has attached two corresponding carbonyls since **4c** is centrosymmetric. So, the fact that the semi-bridging cabonyls exist in **4c** is in good agreement with its IR spectroscopic data.

It is worth pointing out that for each of the two η^5-Bz_5C_5 ligands in complex **4c** four phenyl groups bonded to C(10), C(30), C(40) and C(50) of the benzyl substituents are situated on the opposite side of the tungsten atom with respect to substituted cyclopentadienyl ring, while the phenyl group attached to C(20) of the benzyl substituent approaches the tungsten atom. Similar to the mononuclear analogs of cobalt [6], manganese [7] and molybdenum [10], this

Table 3. Selected bond angles (°) for **4c**

W*—Hg—W	180.00	C(3)—C(4)—W	73.6(5)
C(01)—W—C(02)	102.7(4)	C(40)—C(4)—W	122.2(6)
C(01)—W—C(5)	101.1(4)	C(4)—C(5)—C(1)	107.2(8)
C(01)—W—C(1)	135.9(4)	C(4)—C(5)—C(50)	124(1)
C(01)—W—C(4)	92.5(4)	C(4)—C(5)—W	73.1(5)
C(01)—W—C(2)	151.2(4)	C(1)—C(5)—C(50)	128(1)
C(01)—W—C(3)	117.8(4)	C(1)—C(5)—W	72.5(5)
C(1)—W—Hg	137.6(2)	C(50)—C(5)—W	122.2(6)
C(4)—W—Hg	95.6(2)	C(03)—W—Hg	130.3(3)
C(2)—W—Hg	103.4(2)	C(01)—W—Hg	72.2(43)
C(3)—W—Hg	80.5(2)	C(02)—W—Hg	69.9(3)
W*—Hg—C(01)	139.4(3)	W*—Hg—C(02)	138.1(3)

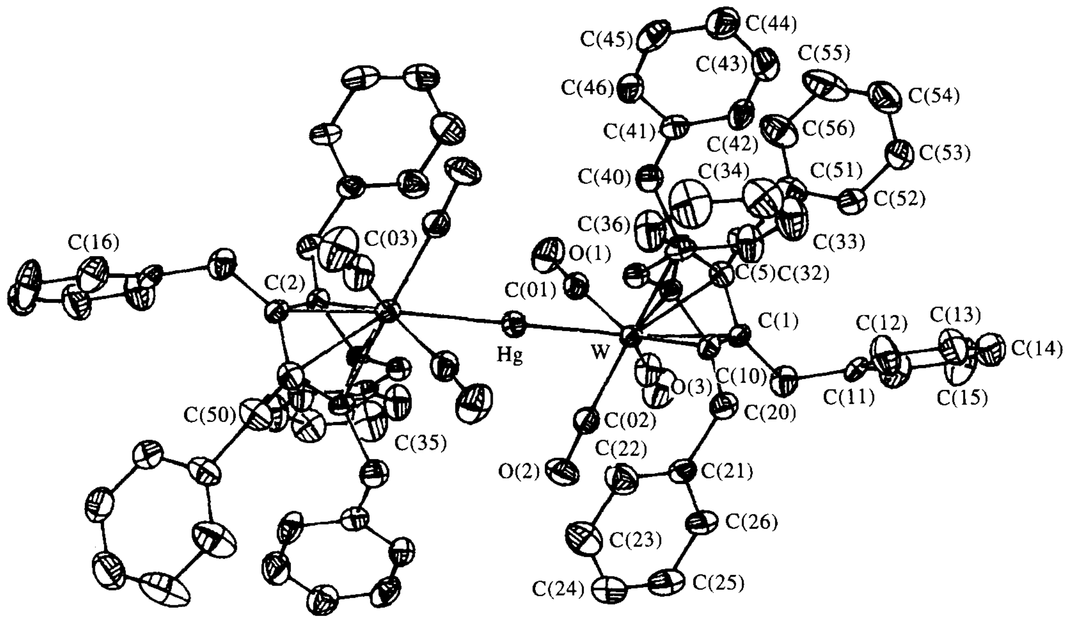


Fig. 1. Molecular structure of **4c**.

could be possibly caused by the smallest gap between the two phenyl groups bound to C(10) and C(30). However, this is not the case because the non-bonding distance of C(11)···C(31) (6.235 Å) lies midway between those of C(11)···C(41) (6.508 Å) and C(31)···C(51) (6.203 Å). So, besides the intramolecular non-bonding interactions, the intermolecular interactions could also be one of the reasons for this geometric arrangement of the five phenyl groups in complex **4c**. However, the real reasons remain unclear and further study is needed.

EXPERIMENTAL

The reactions were carried out under highly prepurified tank nitrogen. Diglyme and THF were rigorously dried and deoxygenated by distillation from Na/benzophenone ketyl. Bz_5C_5H [10], *n*-BuLi [15] and $[\eta^5\text{-}RO_2CC_5H_4(CO)_3M]_2Hg$ (M = Cr, Mo, W; R = CO_2Me, CO_2Et) [3] were prepared according to the literature. $M(CO)_6$(M = Cr, Mo, W) was purchased from Strem Chemicals Inc. and PhHgCl from Alfa Ventron. Silica gel for column chromatography was of about 300–400 mesh. Silica gel for TLC was GF-254 (10–40 μm). IR spectra were recorded on a Nicolet FT-IR 5DX spectrophotometer, ^1H NMR spectra on a Jeol FX 90Q spectrometer. Elemental analysis and MS determinations were performed by a 240 C analyzer and HP 5988A spectrometer, respectively. Melting points were determined on a Yanaco MP-500 apparatus.

Synthesis of $[\eta^5\text{-}HO_2CC_5H_4(CO)_3Cr]_2Hg$ (**2a**)

A 100 cm^3 two-necked flask equipped with a magnetic stir-bar, a serum cap and a reflux condenser, was charged with 0.697 g (0.96 mmol) of $[\eta^5\text{-}EtO_2$

$CC_5H_4(CO)_3Cr]_2Hg$, 20 cm^3 of C_2H_5OH and a solution prepared from 0.291 g (5.19 mmol) KOH and 10 cm^3 of distilled water. The reaction mixture was stirred for 8–10 h at about 40°C until TLC showed no starting complex left. After cooling to room temperature, the reaction mixture was filtered and to the filtrate was added about 50 cm^3 of distilled water followed by treatment with concentrated HCl until the yellow solid precipitated completely. The solid was filtered out and washed first with distilled water until the filtrate reached to the neutral and then washed with CH_2Cl_2 several times. Drying of the washed solid *in vacuo* gave 0.311 g (48%) of **2a** as a yellow solid, m.p. 158°C (dec.). Found: C, 31.6; H, 1.6. Calc. for $C_{18}H_{10}Cr_2HgO_{10}$: C, 31.3; H, 1.5%. IR (KBr, disc): νOH, 3435 (br.), $\nu_{C\equiv O}$, 1959(vs), 1915(s), 1893(vs), $\nu_{C=O}$, 1688(s) cm^{-1}. ^1H NMR (DMSO, δ, ppm): 5.29 [t, 4H, 2(H^3, H^4)], 5.67 [t, 4H, 2(H^2, H^5)]. MS(EI, Cr^{52}), *m/z* (relative intensity): 245 [$HO_2CC_5H_4Cr(CO)_3^+$, 7.3], 217[$HO_2CC_5H_4Cr(CO)_2^+$, 15.7], 202(Hg^+, 62.5), 189[$HO_2CC_5H_4Cr(CO)^+$, 17.2], 161($HO_2CC_5H_4Cr^+$, 56.8), 109($HO_2CC_5H_4^+$, 0.4), 65($C_5H_5^+$, 12.6), 52(Cr^+, 60.7), 45(CO_2H^+, 2.9).

Synthesis of [η^5-$HO_2CC_5H_4(CO)_3Mo]_2Hg$ (**2b**)

The same procedure for **2a** was followed, but the reagents were changed as follows: 0.597 g (0.74 mmol) of [η^5-$MeO_2CC_5H_4(CO)_3Mo]_2Hg$ and 0.201 g (3.59 mmol) of KOH; or 0.75 g (0.90 mmol) of [η^5-$EtO_2CC_5H_4(CO)_3Mo]_2Hg$ and 0.219 g (3.91 mmol) of KOH. The former reaction gave 0.449 g (78%) of **2b,** while the latter reaction gave 0.589 g (84%) of **2b,** as a yellow solid, m.p. 195°C (dec.). Found: C, 27.7; H. 1.3. Calc. for $C_{18}H_{10}HgMo_2O_{10}$: C, 27.8; H, 1.3%. IR (KBr, disc): ν_{OH}, 3443 (br.), $\nu_{C\equiv O}$, 1967(vs), 1922(s), 1893(vs), $\nu_{C=O}$, 1680(s) cm^{-1}. ^1H NMR (DMSO, δ, ppm): 5.76[t, 4H, 2(H^3, H^4)], 6.14 [t, 4H, 2(H^2, H^5)]. MS(EI, MO^{98}), *m/z* (relative intensity): 291[$HO_2CC_5H_4Mo(CO)_3^+$, 4.0], 263[$HO_2CC_5H_4Mo(CO)_2^+$, 2.9], 247[$C_5H_5Mo(CO)_3^+$, 1.2], 235[$HO_2CC_5H_4Mo(CO)^+$, 2.8], 207($HO_2CC_5H_4Mo^+$, 3.2), 202(Hg^+, 26.2), 66($C_5H_6^+$, 31.4), 45(CO_2H^+, 7.1).

Synthesis of [(η^5-$HO_2CC_5H_4(CO)_3W]_2Hg$ (**2c**)

The same procedure for **2a** was followed, but the reagents were changed as follows: 0.272 g (0.277 mmol) of [η^5-$MeO_2CC_5H_4(CO)_3W]_2Hg$ and 0.069 g (1.23 mmol) of KOH. 0.205 g (78%) of **2c** was given as a yellow solid, m.p. 224°C (dec.). Found: C, 22.7; H, 1.0; Calc. for $C_{18}H_{10}HgO_{10}W$: C, 22.7; H, 1.1%. IR (KBr, disc): ν_{OH}, 3418 (br.), $\nu_{C\equiv O}$, 1967(vs), 1909(s), 1885(vs), $\nu_{C=O}$, 1688(s) cm^{-1}. ^1H NMR (DMSO, δ, ppm): 5.84 [t, 4H, 2(H^3, H^4)], 6.20 [t, 4H, 2(H^2, H^5)]. MS(EI, W^{185}), *m/z* (relative intensity): 202(Hg^+, 40.3), 185(W^+, 18.5), 109($HO_2CC_5H_4^+$, 28.9), 64($C_5H_4^+$, 14.0), 45(CO_2H^+, 38.5).

Synthesis of [η^5-$Bz_5C_5(CO)_3Cr]_2Hg$ (**4a**)

A 100 cm^3 two-necked flask equipped with a magnetic stir-bar, a serum cap and a reflux condenser, was charged with 1.24 g (2.4 mmol) of η^5-Bz_5C_5H and 20 cm^3 of diglyme. To this mixture was added an equimolar aliquot of *n*-BuLi/hexane at 0°C and the solution turned to deep purple. After continuous stirring for 15 min, 0.538 g (2.4 mmol) of $Cr(CO)_6$ was added and the reaction mixture heated at about 130°C overnight. Solvent was removed at reduced pressure and 20 cm^3 of THF was added to give a THF solution of the salt η^5-$Bz_5C_5(CO)_3CrLi$. To this salt, 0.70 g (2.4 mmol) of PhHgCl was added at about 0°C and the solution turned to bright-yellow rapidly. After stirring for an additional 1 h, solvent was removed at reduced pressure to give a residue, which was extracted with CH_2Cl_2. The extracts were subjected to column chromatography and TLC separation with 50% CH_2Cl_2/petroleum ether as eluent, followed by recystallization from 50% CH_2Cl_2/petroleum ether to give 0.045 g (3%) of **4a** as a yellow solid, m.p. 140–142°C. Found: C, 68.2; H, 4.7. Calc. for $C_{86}H_{70}Cr_2HgO_6$: C. 68.7; H, 4.7%. IR (KBr, disc): $\nu_{C\equiv O}$, 1942(vs), 1885(s), 1860(vs) cm^{-1}. ^1H NMR ($CDCl_3$, δ, ppm): 3.90(s, 20H, $10CH_2$), 6.40–7.32(m, 50H, $10C_6H_5$).

Synthesis of [η^5-$Bz_5C_5(CO)_3Mo]_2Hg$ (**4b**)

A 100 cm^3 two-necked flask equipped with a magnetic stir-bar, a serum cap and a reflux condenser, was charged with 1.24 g (2.4 mmol) of η^5-Bz_5C_5H and 20 cm^3 of THF. The mixture was stirred and cooled to 0°C. To this mixture was added an equimolar aliquot of *n*-BuLi/hexane at 0°C. After stirring for 15 min, 0.62 g (2.4 mmol) of $Mo(CO)_6$ was added and the reaction mixture refluxed for about 24 h to give the salt η^5-$Bz_5C_5(CO)_3MoLi$. To this salt was added 0.70 g (2.4 mmol) of PhHgCl at about 0°C and the reaction mixture stirred for an additional 1 h. After the same work-up as that in the preparation of **4a,** 0.085 g (5%) of **4b** was given as a yellow solid, m.p. 188–189°C. Found: C. 64.8; H, 4.4. Calc. for $C_{86}H_{70}HgMo_2O_6$: C, 64.9; H, 4.4%. IR (KBr, disc): $\nu_{C\equiv O}$, 1959(vs), 1909(s), 1868(vs) cm^{-1}. ^1H NMR ($CDCl_3$, δ, ppm): 3.97(s, 20H, $10CH_2$). 6.70–7.27(m, 50H, $10C_6H_5$).

Synthesis of [η^5-$Bz_5C_5(CO)_3W]_2Hg$ (**4c**)

The same procedure for **4a** was followed, but use of 0.84 g (2.4 mmol) of $W(CO)_6$ instead of $Cr(CO)_6$ to give 0.121 g (6%) of **4c** as a yellow solid, m.p. 200–202°C. Found: C, 58.4; H, 3.8. Calc. for $C_{86}H_{70}HgO_6W_2$: C, 58.4; H, 4.0%. IR (KBr, disc): $\nu_{C\equiv O}$, 1950(vs), 1893(s), 1860(vs) cm^{-1}. ^1H NMR ($CDCl_3$, δ, ppm): 4.00(s, 20H, $10CH_2$), 6.63–7.25(m, 50H, $10C_6H_5$).

Single-crystal structure determination of $[\eta^5\text{-Bz}_5\text{C}_5(\text{CO})_3\text{W}]_2\text{Hg}$ (**4c**)

A yellow crystal measuring $0.16 \times 0.20 \times 0.50$ mm^3 obtained by recrystallization from CH_2Cl_2/hexane in a refrigerator, was mounted on a glass fiber and placed on an Enraf-Nonius CAD4 diffractometer equipped with a graphite monochromator. A total of 6386 independent reflections were collected at room temperature with Mo-K_α ($\lambda = 0.71069$ Å) radiation by ω-2θ scan mode, of which 4202 independent reflections with $I \geqslant 3\sigma(I)$ were considered to be observed and used in the subsequent refinement. Data were corrected for Lp factors.

The positions of tungsten and mercury atoms were found in an E-map by direct methods and the remaining non-hydrogen atoms found in the succeeding difference Fourier syntheses. The final refinement by full-matrix least-squares method used anisotropic thermal parameters for non-hydrogen atoms. All calculations were performed on a MICRO-VAX I computer using TEXSAN program system.

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