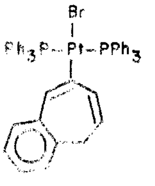
complexes, the probable mechanism for formation of **7–9** is that given in Scheme II. However, the known tendency for Pt(0) to insert into carbon–bromine bonds suggested an insertion–elimination mechanism (Scheme III) as a possible alternative. To evaluate this possibility, the $(Ph_3P)_2Pt$ insertion products **18–20** were synthesized, characterized, and subjected to the reaction conditions. In no case was any reaction observed.



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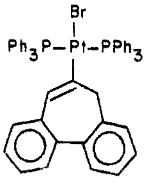

20

In summary, $(Ph_3P)_3Pt$ effectively traps cycloheptatetraenes to give allene complexes, one of which requires at least 17 kcal/mol to convert to its carbene isomer. This is in marked contrast to Fp^+, Rp^+ (Rp = $RuCp(CO)_2$), or $(CO)_5W$ complexes of the parent and in one case (M = Fp^+) the monobenzoannelated ring corresponding to **8**, all of which exist solely as carbene complexes. This marked difference between the d^6 and d^{10} metals rests primarily on the availability of a significantly lower lying LUMO in the former.[10]

Acknowledgment. Support for this work by the National Science Foundation is gratefully acknowledged. W.R.W. also acknowledges a fellowship from the Proctor and Gamble Co. We thank the Center for Instructional Research Computing Activities, University of Florida, for a grant of computer time. High-field NMR spectra were obtained on a Nicolet NT-300 spectrometer, operating at a field of 7 T; therefore acknowledgment is made to the Instrument Program, Chemistry Division, National Science Foundation, for financial assistance in the purchase of this instrument.

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(19) **18**: tetrakis(triphenylphosphine)platinum (1.07 g, 0.86 mmol) was weighed into a round-bottom flask equipped with a magnetic stirrer and a nitrogen inlet. A mixture of isomeric bromocycloheptatrienes (1.0 g, 5.85 mmol) in 50 mL of benzene was added and the mixture stirred overnight. The solvent was removed in vacuo and the product triturated with 50 mL of ether. The precipitate was filtered and dried in vacuo to give **18**, devoid of the 2- and 3-isomers (0.680 g, 89%): mp 216.0–216.5 °C dec IR (KBr) 3050 w, 3010 w, 1571 w, 1505 m, 1480 m, 1430 s, 1385 m, 1180 m, 1100 s, 1030 m, 1000 m, 750 m, 700 s, 500 s cm^{-1}; ^1H NMR (100 MHz, $CDCl_3$) δ 2.23 (td, $^3J_{PtH} = 39$ Hz, $J_{HH} = 7.1$ Hz, H7), 4.30 (dt, $J = 6.84$ Hz, $J = 6.0$ Hz, 1 H), 5.6 (t, $J_{PtH} = 32$ Hz, 1 H), 5.64 (s, 2 H), 6.0 (s, 3 H), 7.2 (Ph_3P, 18 H), 7.7 (Ph_3P, 12 H); ^{13}C NMR (75 MHz, $CDCl_3$) 80.5 (C7), 115.4, 122.9, 124.58, 127.70 (t, $^3J_{PC} = 5.27$ Hz, Ph_3P, meta), 130.1 (s, Ph_3P, para), 131.0 (t, $^2J_{PtC} = 46$ Hz, Ph_3P, ipso), 134.3, 134.45, 135.2 (m, Ph_3P, ortho), 139.4 (Cl); ^{31}P NMR (121.5 MHz, $CDCl_3$) δ 21.34 (t, $^1J_{PtP} = 3215$ Hz). Anal. Calcd for $C_{43}H_{37}BrP_2Pt$: C, 57.94; H, 4.15. Found: C, 57.76; H, 4.23.

19: mp 218–219 °C dec; 61% yield; IR (KBr) 3065, 3020 w, 1610 w, 1485 s, 1440 s, 1390 s, 1100 s, 750 m, 700 s, 540 s cm^{-1}; ^1H NMR (100 MHz, $CDCl_3$) δ 1.6 (d, $^3J_{HH} = 3$ Hz, 47), 4.68 (m, 1 H), 5.8 (td, $^3J_{PtH} = 38$ Hz, $J_{HH} = 10$ Hz), 6.4–7.1 (m, 5 H), 7.2 (18 H, Ph_3P), 7.7 (12 H, Ph_3P); ^{13}C NMR (25 MHz, CD_2Cl_2) δ 33.3 (C7), 121 (t, $^2J_{PtC} = 72$ Hz), 124.7, 126.1, 126.2, 126.5, 128.1 (d, $^3J_{PC} = 4.9$ Hz, Ph_3P, meta), 130.4, 130.5 (s, Ph_3P, para), 131.3 (td, $^2J_{PtC} = 28$ Hz, $^1J_{PC} = 15.9$ Hz, Ph_3P, ipso), 134.4, 135.5 (d, $^2J_{PC} = 6.1$ Hz, Ph_3P, ortho), 139.1, 142.8 (d, $^2J_{PC} = 17.1$ Hz, C4); ^{31}P NMR (121.5 MHz, $CDCl_3$) δ 22.0 (t, $^1J_{PtP} = 3189.4$ Hz).

20: mp 281–283 °C dec; 86% yield; IR (KBr) 3060 w, 1590 m, 1485 s, 1440 s, 1390 s, 1195 w, 1105 s, 1000 m, 840 m, 755 s, 695 s, 520 s cm^{-1}; ^1H NMR (100 MHz, $CDCl_3$) δ 5.50 (t, $^3J_{PtH} = 68.4$ Hz, 2 H, H7), 6.6 (m), 7.2 (b s, Ph_3P); ^{13}C NMR (75 MHz, $CDCl_3$) δ 45.6 (C7), 123.4 (CH), 124.9 (CH), 125.6 (CH), 126.5 (CH), 127.0 (CH), 127.3 (CH), 127.7 (Ph_3P, meta), 128.4, 129.1, 130.0 (Ph_3P, para), 132.1 (t, $^3J_{PC} = 7.2$ Hz, C2), 135.1 (vt, $^2J_{PC} = 6$ Hz, Ph_3P, ortho), 136.9, 139.2, 139.6, 139.8, 147.1 (t, $^2J_{PC} = 17.9$ Hz, C1); ^{31}P NMR (121.5 MHz, $CDCl_3$) δ 22.7 (t, $^1J_{PtP} = 3190$ Hz).

(Fulvalene)dimolybdenum Pentacarbonyl Diphosphine: A Dinuclear Organometallic Zwitterion

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Summary: Treatment of (η^5:η^5-fulvalene)dimolybdenum hexacarbonyl ($FvMo_2(CO)_6$, **1**) with $P(CH_3)_3$ or $(CH_3)_2PCH_2P(CH_3)_2$ gives the dinuclear zwitterions $FvMo_2(CO)_5L_2$ (**2a,b**) in a reaction that does not proceed via the metal–metal bonded $FvMo_2(CO)_5L$ as an intermediate. Complex **2b** was characterized by X-ray crystallography. The zwitterionic species show reactivity patterns consistent with the charge-localized structure: the negatively charged metal center undergoes attack by electrophiles (**2a,b**), whereas the positively charged center undergoes reduction with one-electron reductants and $LiAlH_4$ (**2a**). When treated with an excess of $P(CH_3)_3$, **2a** and **2b** undergo decomplexation to form $FvMo(CO)_2L_2$ (**12a,b**) in which only one Cp moiety of the Fv ligand is complexed to Mo.

The ligand-induced disproportionation of $[CpMo(CO)_3]_2$ to $[CpMoL_2(CO)_2]^+[CpMo(CO)_3]^-$ (particularly for L = phosphine) has been known for some time.[1] Recently, Tyler and co-workers have studied the mechanism of the photochemically induced disproportionation in detail.[2] We have been interested in the chemistry of carbonyl (η^5:η^5-fulvalene)dimetal (FvM_2) complexes,[3] in which the metals are forced to remain in close proximity, even after the metal–metal bond is broken. We report here the thermal reaction of $FvMo_2(CO)_6$ (**1**) with the strongly donating phosphines $P(CH_3)_3$ and $(CH_3)_2PCH_2P(CH_3)_2$ (dmpm), leading to the first dinuclear organometallic zwitterions $FvMo_2(CO)_5L_2$ (**2a,b**). The reactivity of these complexes, one of which has been structurally characterized, clearly reflects the zwitterionic nature of these systems.

Treatment of **1** with $P(CH_3)_3$ in THF (0 °C, 48 h) gave $FvMo_2(CO)_5[P(CH_3)_3]_2$ (**2a**) in 63% yield.[4] A similar product was obtained with dmpm (**2b**, 45 °C, 4 h, 77%).[4] To our knowledge, these are the first examples of dinuclear, metal-centered organometallic zwitterions. Because of its novelty an X-ray structural characterization was undertaken of **2b**[4] (Figure 1). The Mo–Mo bond has been broken, and the two metal centers are coordinated to the fulvalene ligand in a trans manner. The average Mo1–CO bond length, 1.974 Å, is considerably larger than the av-

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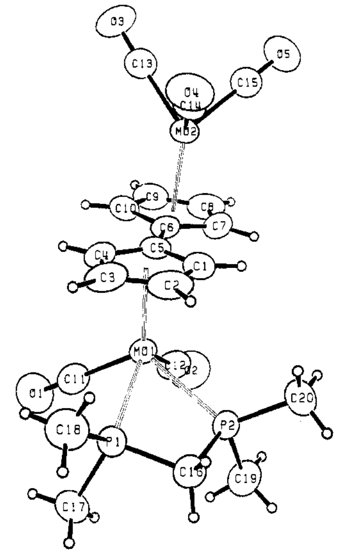
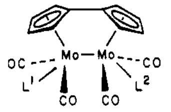
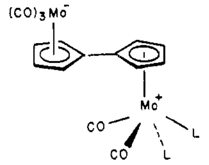


Figure 1. ORTEP drawing of **2b**. Ellipsoids are scaled to represent the 50% probability surface.

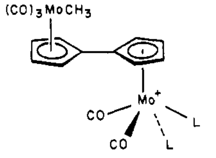
erage Mo2–CO bond length of 1.927 Å. This is consistent with stronger back-donation of electrons from Mo2 to CO than from Mo1. Similarly, the average Mo1–C(ring) distance, 2.328 Å, is shorter than the Mo2–C(ring) distance, 2.391 Å, as would be expected if Mo1 is electron poor compared to Mo2. We find it surprising that, even though conjugated, the molecule prefers the charge-localized structure rather than an alternative $L_2(CO)_2$Mo1(Cp diene)–Mo2$(CO)_3$(fulvene) arrangement, which also gives each metal 18 valence electrons. Both $M(CO)_2L_2$(diene)[5] and $M(CO)_3(\eta^6$-fulvene)[6] complexes (L = PR_3; M = Cr, Mo, W) are well-known. Compounds **2a** and **2b** exhibit reactivity consistent with their zwitterionic structure. They can both be methylated to **3a**[4] (CH_3I, THF, room temperature, 1 h) and **3b**[4] (CH_3CN, room temperature, 5 min), respectively. The methylene protons in **2b** undergo H–D exchange with CD_3CN in the presence of $NaBH_4$ or NaO-H.[7] Reduction of **2b** with $LiAlH_4$ in THF yields an intractable mixture of products, **4b** being only a minor component. On the other hand, **2a** undergoes smooth reduction of one carbonyl group to methyl ($LiAlH_4$, THF, −20 °C, 1 h), giving **4a**.[4] Methylation of **2a** with CH_3I followed by $LiAlH_4$ reduction gave the dimethyl complex **5**.[4] To our knowledge, these conversions are unprecedented in dinuclear complex chemistry,[8] boding well for other novel chemistry.



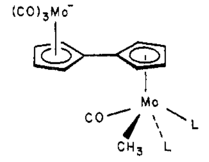
1 $L^1 = L^2 = CO$
8 $L^1 = L^2 = P(CH_3)_3$
11 $L^1 = CO; L^2 = P(CH_3)_3$



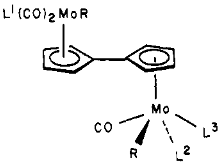
2a $L = P(CH_3)_3$
2b $L = 1/2\,(CH_3)_2PCH_2P(CH_3)_2$



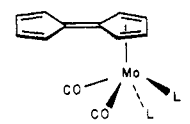
3a $L = P(CH_3)_3$
3b $L = 1/2\,(CH_3)_2PCH_2P(CH_3)_2$



4a $L = P(CH_3)_3$
4b $L = 1/2\,(CH_3)PCH_2P(CH_3)_2$



5 $R = CH_3; L^1 = CO; L^2 = L^3 = P(CH_3)_3$
6 $R = H; L^1 = L^2 = L^3 = CO$
7 $R = H; L^1 = L^2 = P(CH_3)_3; L^3 = CO$
10 $R = CH_3; L^1 = L^2 = CO; L^3 = P(CH_3)_3$



12a $L = P(CH_3)_3$
12b $L = 1/2\,(CH_3)_2PCH_2P(CH_3)_2$

Sodium amalgam reduction of **1** followed by protonation with CF_3COOH gave the dihydride $FvMo_2(CO)_6H_2$ (**6**).[9] Treatment of **6** with $P(CH_3)_3$ yielded the phosphine-substituted dihydride **7**,[4] which undergoes rapid cis–trans isomerization at each metal center, as seen by NMR.[10]

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[4] All new compounds gave satisfactory spectral and/or analytical data. **2a**: 63%, orange-brown crystals, mp 242 °C dec; ^1H NMR (300 MHz for all spectra, CD_3CN) δ 1.64 ("d", J = 10.3 Hz, 18 H), 5.06 (t, J = 2.4 Hz, 2 H), 5.22 (m, 2 H), 5.50 (t, J = 2.4 Hz, 2 H), 5.53 (m, 2 H); $^{31}P\{^1H\}$ NMR (121 MHz for all spectra, CD_3CN) δ 19.2 (s); CIMS, m/e (relative intensity) 584 (M^+ − CO, highest mass peak in isotope envelope, 28.3), 77 (100); IR (THF) 1955, 1906, 1871, 1795 cm^{-1}. On the basis of the relative intensity of the 1955 and 1871 cm^{-1} absorptions, we believe that the two $P(CH_3)_3$ groups are coordinated to Mo in a trans manner: Manning, A. R. *J. Chem. Soc. A* **1967**, 1984. **2b**: 77%, brown crystals, mp 243 °C dec; ^1H NMR (CD_3CN) δ 1.68 ("t", J = 5.7 Hz, 6 H), 1.69 ("t", J = 5.7 Hz, 6 H), 3.65 (m, 2 H), 5.04 (t, J = 2.4 Hz, 2 H), 5.41 (m, 2 H), 5.49 (t, J = 2.4 Hz, 2 H), 5.69 (t, J = 2.1 Hz, 2 H); $^{31}P\{^1H\}$ NMR (CD_3CN) δ −23.4 (s); CIMS, m/e (relative intensity) 568 (M^+ − CO, 3.4), 61 (100); IR (CH_3CN) 1983, 1904, 1788 cm^{-1}; X-ray, crystal size 0.20 × 0.23 × 0.40 mm, orthorombic Laue symmetry, space group $P2_12_12_1$, a = 9.1049 (8) Å, b = 12.2598 (14) Å, c = 20.1606 (18) Å, V = 2250.4 (7) $Å^3$, μ_{calcd} = 12.55 cm^{-1}, d_{calcd} = 1.76 g cm^{-3}, radiation Mo Kα ($\bar{\lambda}$ = 0.71073 Å), scan range 3° $\leq 2\theta \leq$ 45°, reflections collected 1725, unique 1641 with $F^2 > 3\sigma(F^2)$, R = 0.0164, R_w = 0.0242. **12a**: 92%, orange-red crystals, mp 180 °C dec; ^1H NMR (CD_3CN) δ 1.40 ("d", J = 10.0 Hz, 18 H), 4.99 (m, 2 H), 5.20 (t, J = 2.2 Hz, 2 H), 5.63 (t, J = 2.2 Hz, 2 H); 5.90 (t, J = 2.8 Hz, 2 H); $^{31}P\{^1H\}$ NMR (CD_3CN) δ 19.4 (s); ^{13}C NMR (75 MHz, CD_2Cl_2) δ 18.9 (dq, J = 31, 130 Hz), 78.8 (d, J = 175 Hz), 86.0 (d, J = 175 Hz), 104.5 (s), 109.1 (d, J = 158 Hz), 110.5 (d, J = 161 Hz), 133.7 (s), 236.6 (t, J = 28 Hz); CIMS, m/e (relative intensity) 432 (M^+, 1.6), 77 (100); IR (THF) 1936, 1852 cm^{-1} (see note to **2a**). **12b**: 93%, red crystals, mp 195 °C dec; ^1H NMR (CD_3CN) δ 1.10 ("t", J = 5.4 Hz, 6 H), 1.60 ("t", J = 5.4 Hz, 6 H), 3.33 (m, 2 H), 5.01 (t, J = 2.1 Hz, 2 H), 5.39 (m, 2 H), 5.64 (m, 2 H), 5.92 (t, J = 2.5 Hz, 2 H); $^{31}P\{^1H\}$ NMR (CD_3CN) δ −16.2 (s); CIMS, m/e (relative intensity) 416 (M^+, 1.8), 55 (100); IR (THF) 1959, 1884 cm^{-1}.

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Photolysis of **7** (THF, 300 nm, 2 h) led to loss of H_2 and formation of the disubstituted metal–metal bonded complex **8**[4,11a,c] (structure ascertained by X-ray analysis). This transformation is interesting in view of the current efforts to observe dinuclear dihydrogen evolution as a model for such processes on surfaces.[11b] Sodium amalgam reduction of **2a** (THF, room temperature, 3 h) caused loss of $P(CH_3)_3$ to give the dianion **9**,[4] which could be methylated (CH_3I, THF, 0 °C, 5 min) to provide the dimethyl species **10**[4] in a dynamic equilibrium of cis and trans isomers around the phosphine-substituted Mo center. Protonation of **9** (CF_3COOH, THF, 0 °C, 2 min) followed by photolysis (300 nm, 2 h) gave the monosubstituted metal–metal bonded complex **11**.[4,11] Irradiation of **11** for a longer period of time (8–10 h) resulted in a 1:2:1 mixture of **1**, **11**, and **8**. Irradiation of **5** furnished **8** (ca. 80%, NMR).

We find that no reaction occurs when **11** is treated with an excess of $P(CH_3)_3$ (THF, room temperature), proving that **11** is not an intermediate in the formation of **2a** from **1**. The reaction is facile even in the dark, indicating that initial homolysis of the metal–metal bond is not required.[12]

If **1** is exposed to an excess of $P(CH_3)_3$ (≥6 equiv) in CH_3CN, the formation of **2a** is instant and quantitative (NMR). Surprisingly, a subsequent slower transformation takes place (room temperature, 2 days) to give the known *fac*-$Mo(CO)_3[P(CH_3)_3]_3$[13] as well as the novel $FvMo(CO)_2[P(CH_3)_3]_2$ (**12a**).[4] In this unprecedented process, the product contains a fulvalene ligand in which only one of the Cp moieties is complexed to Mo. On the basis of ^{13}C NMR data for **12a**,[4] we can exclude a trimethylenemethane structure. The spectral data[4] indicate a considerable contribution of the dipolar resonance structure $L_2(CO)_2Mo^+(Cp)(Cp^-)$ to **12a**. This finding is corroborated by the facile H–D exchange (acetone-d_6) of the uncomplexed half of the Fv ligand and its facile reduction with $LiAlH_4$ to $FvMo(CO)(CH_3)[P(CH_3)_3]_2$ (**13**; NMR, 75%).[4] The complex **12b** is formed and reacts similarly. These decomplexation reactions, which can be reversed by addition of metal carbonyls, constitute the first examples of ring slippage[14] in a fulvalene system and open up the way to a designed synthesis of heterodinuclear complexes.

Acknowledgment. This work was supported by the Director, Office of Energy Research, Office of Basic Energy Sciences, Chemical Sciences Division of the U.S. Department of Energy, under Contract DE-AC03-76SF00098. The crystal structure analysis was carried out by Dr. F. J. Hollander, U.C. Berkeley X-ray Crystallographic Facility. M.T. is the recipient of a Fellowship from the Royal Norwegian Council for Scientific and Industrial Research.

Supplementary Material Available: Complete yields, melting points, and spectral data on **3**–**5** and **7**–**11**, a listing of positional and thermal parameters, and tables of bond lengths and angles, torsional angles, and structure factors on **2b** (20 pages). Ordering information is given on any current masthead page.

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[11] (a) See also: Alt, H. G.; Mahmoud, K. A.; Rest, A. J. *Angew. Chem., Int. Ed. Engl.* **1983**, *22*, 544; *Angew. Chem. Suppl.* **1983**, 803. (b) Waleh, A.; Loew, G. H.; Mueller-Westerhoff, U. T. *Inorg. Chem.* **1984**, *23*, 2859. Moore, M. F.; Wilson, S. R.; Hendrickson, D. N.; Mueller-Westerhoff, U. T. *Ibid.* **1984**, *23*, 2918. Saillard, J.-Y.; Hoffmann, R. *J. Am. Chem. Soc.* **1984**, *106*, 2006. Mueller-Westerhoff, U. T.; Nazzal, A. *Ibid.* **1984**, *106*, 5381. Susuki, K. *J. Less-Common Met.* **1983**, *89*, 183. Paal, Z.; Menon, P. G. *Catal. Rev.—Sci. Eng.* **1983**, *25*, 229. (c) Details of the X-ray analyses are to be submitted: Cass, M. E.; Kretchmar, S. A.; Turowski, P. N., unpublished.

[12] We have found that treatment of $[CpMo(CO)_3]_2$ with $P(CH_3)_3$ gives $[CpMo(CO)_3^-][CpMo(CO)_2[P(CH_3)_3]_2^+]$ even in the dark. Provided that the ligand is nucleophilic enough, no irradiation[2] is needed to initiate the disproportionation.

[13] Mathieu, R.; Lenzi, M.; Poilblanc, R. *Inorg. Chem.* **1970**, *9*, 2030.

[14] For Cp ring slippage, see: Casey, C. P.; O'Connor, J. M.; Haller, K. J. *J. Am. Chem. Soc.* **1985**, *107*, 1241. Faller, J. W.; Crabtree, R. H.; Habib, A. *Organometallics* **1985**, *4*, 929 and the references therein.