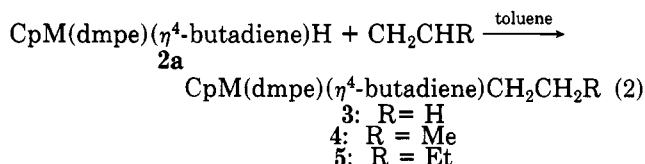


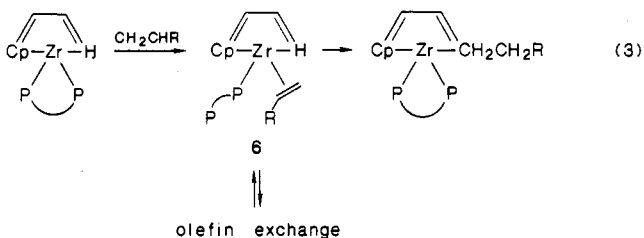
equatorial position, was located by difference Fourier methods and satisfactorily refined [Zr-H = 1.80 (5) Å; Cp(centroid)-Zr-H = 97.4 (3)°].

In the  $^1\text{H}$  NMR spectrum<sup>10</sup> the characteristic terminal hydride resonance appears as a low-field triplet [ $\delta = 7.20$  ppm ( $J_{\text{P-H}} = 5.2$  Hz) (**2a**);  $\delta = 7.27$  ppm ( $J_{\text{P-H}} = 5.2$  Hz) (**2b**)]. A broad and intense band in the IR spectrum is tentatively assigned to the M-H stretching mode [1420  $\text{cm}^{-1}$  (**2a**); 1460  $\text{cm}^{-1}$  (**2b**)].

Compound **2a** rapidly reacts with  $\alpha$ -olefins (1 atm, room temperature) to give terminally bonded alkyl derivatives CpM(dmpe)( $\eta^4$ -butadiene)R in crystalline form and good yield<sup>10</sup> (eq 2). Internal olefins such as *cis*-2-butene and terminally disubstituted olefins like isobutene fail to react with these hydride complexes.



A puzzling question arises from the possible insertion pathway into the Zr-H bond because no empty orbitals are available on **2a** for the necessary olefin pre-coordination.<sup>11</sup> However, dissociation of one of the two phosphorus atoms of the chelating phosphine might possibly provide the empty coordination site (eq 3).<sup>12</sup> In a



subsequent step, hydride transfer to the coordinated olefin transforms intermediate **6** into the final insertion product. Preliminary evidence for the formation of **6** is provided by the ability of the alkyl derivatives **3**, **4**, and **5** to reversibly transform into each other by reaction with an excess of the corresponding  $\alpha$ -olefin (1 atm, room temperature) (eq 4). The equilibrium shown in eq 4 can be approached from both sides of eq 4 and is easily shifted by adding or removing an olefin.

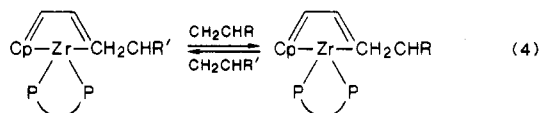
The present results suggest that the hydrozirconation reaction might in fact be an equilibrium. The situation should be similar to the insertion of olefins into the Nb-H

(9) (a) Erker, G.; Wicher, J.; Engel, K.; Rosenfeldt, F.; Dietrich, W.; Kruger, C. *J. Am. Chem. Soc.* **1980**, *102*, 6344. (b) Erker, G.; Berg, K.; Kruger, C.; Muller, G.; Angermund, K.; Benn, R.; Schroth, G. *Angew. Chem., Int. Ed. Engl.* **1984**, *23*, 455. (c) Erker, G.; Muhlerbernd, T.; Benn, R.; Rufinska, A.; Tsay, Y.; Kruger, C. *Angew. Chem., Int. Ed. Engl.* **1985**, *24*, 32. (d) Kruger, C.; Muller, G. *Organometallics* **1985**, *4*, 215. (e) Blenkins, J.; Hessen, B.; van Bolhuis, F.; Wagner, A. J.; Teuben, J. H. *Organometallics* **1987**, *6*, 459. (f) Yasuda, H.; Nakamura, A. *Angew. Chem., Int. Ed. Engl.* **1987**, *26*, 723.

(10) The structure of all the complexes has been assigned on the basis of their spectroscopic and analytical details (available as supplementary material) and direct synthesis from **1** and the corresponding alkyllithium or Grignard reagents. The presence of terminally bonded alkyl chains has been demonstrated by NMR techniques [attached proton test (APT-NMR)].

(11) See, for example: (a) Lauher, J. W.; Hoffmann, R. *J. Am. Chem. Soc.* **1976**, *98*, 1729. (b) Doherty, N. M.; Bercaw, J. E. *J. Am. Chem. Soc.* **1985**, *107*, 2670.

(12) Whereas the possibility of slippage of the Cp ring cannot be ruled out, a  $\eta^4$ -butadiene =  $\eta^2$ -butadiene rearrangement process seems quite improbable on the basis of the simple consideration that  $\eta^2$ -butadiene should react not dissimilarly from a normal  $\eta^2$ -bonded  $\alpha$ -olefin. The insertion or exchange products expected in this case have never been observed.



bond of permethylniobocene(III) olefin hydride complexes,<sup>11b</sup> where the presence of  $\beta$ -hydrogens in the alkyl chain is a prerequisite for exchange. In agreement with that, CpZr( $\eta^4$ -butadiene)(dmpe)CH<sub>3</sub> does not react with olefins (ethylene, propylene), even after long reaction times and using severe reaction conditions (5 atm, 80 °C).

Further study of the chemistry of these hydrides is in progress.

**Supplementary Material Available:** Tables of crystal data, anisotropic thermal parameters, atomic coordinates, and bond lengths and angles and spectroscopic and analytical data (14 pages); a listing of calculated and observed structure factors (20 pages). Ordering information is given on any current masthead page.

### Regio- and Stereospecific Conversion of Chromium Dienes into Olefins via Anionic Allyl Complexes

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**Summary:** Pseudooctahedral chromium diene complexes react with trialkylborohydride reagents to give allylic anions resulting from addition of H<sup>-</sup> at the least crowded position and then rearrangement to the syn isomer. The product allylic anions can be isolated as tetraethylammonium salts and they react in CD<sub>3</sub>CN solution with electrophiles to give free olefins.

There are relatively few examples of isolable diene complexes that can be conveniently made in large quantities and manipulated to give functionalized products.<sup>3</sup> We have begun research on the easily prepared chromium diene complexes of the stoichiometry Cr(CO)<sub>3</sub>(P(OMe)<sub>3</sub>)( $\eta^4$ -diene) and find that it is possible to convert the dienes into *trans*-olefins stereospecifically.

The required starting materials are made photolytically with a reaction developed by Kreiter.<sup>4</sup> The diene complexes react with trialkylborohydrides MBHR<sub>3</sub> (M = Li, K; R = ethyl, *sec*-butyl)<sup>5</sup> in THF at reduced temperature. The use of potassium as a counterion permits the isolation of good yields of pure, crystalline derivatives after counterion exchange with tetraethylammonium bromide in THF at -20 °C (eq 1).<sup>6,7</sup>

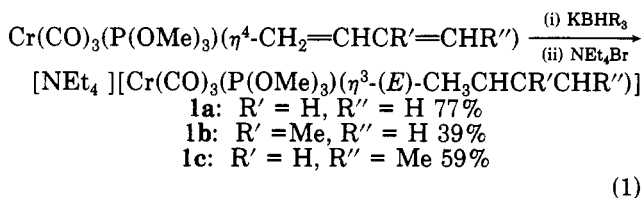
(1) New York University.

(2) Merck, Sharp, and Dohme.

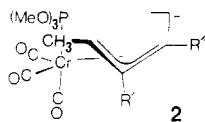
(3) (a) Davies, S. G. *Organotransitionmetal Complexes in Organic Synthesis*; Pergamon: Elmsford, 1982. (b) Pearson, A. J. *Metallo-Organic Chemistry*; Wiley: New York, 1985. (c) Collman, J. P.; Hegedus, L. S.; Norton, J. R.; Finke, R. G. *Principles and Applications of Organotransitionmetal Chemistry*, 2nd ed.; University Science: Mill Valley, CA, 1987.

(4) (a) Kreiter, C. F. *Adv. Organomet. Chem.* **1986**, *26*, 297. (b) Kotzian, M.; Kreiter, C. G.; Ozkar, S. *J. Organomet. Chem.* **1982**, *229*, 29. (c) Kotzian, M.; Kreiter, C. G.; Michael, G.; Ozkar, S. *Chem. Ber.* **1983**, *116*, 3637. (d) Kreiter, C. G.; Kotzian, M.; Schubert, U.; Bau, R.; Bruck, M. A. *Z. Naturforsch.* **1984**, *39B*, 1553. (e) Kreiter, C. G.; Kotzian, M. *J. Organomet. Chem.* **1985**, *289*, 295.

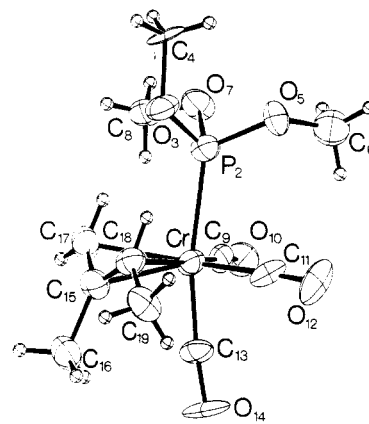
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The NMR spectra of the tetraethylammonium salts are consistent with  $\eta^3$ -allyl complexes **1** formed by hydride attack on a terminal, unsubstituted position on the diene and rearrangement of the carbon backbone to place any substituents in syn (*E*) positions. A single stereoisomer is observed in the case of the (*E*)-2-buten-1-yl and (*E*)-3-penten-2-yl derivatives **1a** and **1c**, derived from 1,3-butadiene and (*E*)-1,3-pentadiene, respectively. The  $^1\text{H}$  NMR spectrum of the latter includes a triplet at  $\delta$  4.22 for the hydrogen at the 2-position coupled to two equivalent anti hydrogens, while a multiplet is observed for the 2-hydrogen in the case of the (*E*)-2-buten-1-yl derivative because of additional coupling to a single syn proton. The resonance of the anti hydrogen of the  $\text{CH}_2$  group appears at  $\delta$  0.60 with a very large coupling to phosphorus ( $^3J_{\text{P-H}} = 18$  Hz). This is consistent with the open face of the allyl being pointed at the phosphite (in the  $\alpha$ -position of the pseudooctahedron, as in **2**) in analogy to the parent diene complexes<sup>4c</sup> and in contrast to certain isoelectronic neutral manganese allyl complexes.<sup>8</sup>



There are two isomers present in unequal but reproducible amounts in samples of the (*E*)-2-methyl-2-buten-1-yl derivative **1b**, derived from isoprene. They are, we suggest, related by rotation about the chromium-allyl bond and, from examination of partially enriched samples, do not interconvert at an appreciable rate.<sup>9</sup> An X-ray diffraction study of a single crystal (selected from a mixture of the two isomers) confirms the assigned regiochemistry (Figure 1).<sup>10</sup> The geometry about the chromium atom is dis-



**Figure 1.** ORTEP drawing of the anion in  $[\text{NEt}_4][\text{Cr}(\text{CO})_3(\text{P}(\text{OMe})_3)(\eta^3\text{-}(E)\text{-2-methyl-2-buten-1-yl})]$  showing thermal ellipsoids at 30% probabilities and the atom numbering scheme. Significant bond distances ( $\text{\AA}$ ) and angles ( $^\circ$ ): Cr1-P2, 2.312 (2); Cr1-C9, 1.809 (6); Cr1-C11, 1.807 (6); Cr1-C13, 1.799 (6); Cr1-C15, 2.206 (7); Cr1-C17, 2.247 (7); Cr1-C18, 2.296 (6); C9-O10, 1.183 (8); C11-O12, 1.165 (8); C13-O14, 1.173 (8); C15-C16, 1.49 (1); C15-C17, 1.39 (1); C15-C18, 1.397 (9); C18-C19, 1.48 (1); P2-Cr1-C9, 87.3 (2); P2-Cr1-C11: 89.5 (2); P2-Cr1-C13, 167.8 (2); C9-Cr1-C11, 101.4 (3); C9-Cr1-C13, 83.3 (3); C11-Cr1-C13, 84.8 (3); C16-C15-C18, 121.9 (6).

torted octahedron with a close contact between the 2-methyl group and the carbonyl trans to the phosphite. The steric crowding in this complex is apparently the reason why this derivative is the least stable of the three allylic anions we have made.

The addition of the hydride to a terminal position is contrary to the expectation that a homoallyl, not an  $\eta^3$ -allyl, would be the thermodynamic product of nucleophilic addition,<sup>11</sup> since it is well-known that the  $d^6$   $\text{M}(\text{CO})_4$  fragment prefers coordination to a nonconjugated diene.<sup>12</sup> However, the binding preference is *inverted* in the presence of trimethyl phosphite: we have demonstrated that *mer*- $\text{Cr}(\text{CO})_3(\text{P}(\text{OMe})_3)(\eta^4\text{-1,5-cyclooctadiene})$  reacts with conjugated dienes to give the *fac*- $\text{Cr}(\text{CO})_3(\text{P}(\text{OMe})_3)(\eta^4\text{-1,3-diene})$  products.<sup>13</sup> It is also noteworthy that the substituents on the terminal positions of the allyl groups are in the syn position only, probably formed through a  $\pi\text{-}\sigma\text{-}\pi$  rearrangement driven by the steric crowding of this metal ligand environment. Steric crowding is also the reason, we feel, why only 2-methyl-2-buten-1-yl is formed from isoprene in this system, in contrast with the 1-methyl-2-buten-1-yl ligand obtained in the case of  $[\text{Ru}(\text{C}_5\text{H}_5)(\text{CO})(\eta^4\text{-isoprene})]^+$ <sup>14</sup> and  $[\text{Mo}(\text{C}_5\text{H}_5)(\text{CO})_2(\eta^4\text{-isoprene})]^+$ <sup>15,16</sup>

Treatment of acetonitrile solutions of the tetraethylammonium salts of the allyl anions with an excess an electrophile gives the corresponding *trans*-olefin stereospecifically, as determined by  $^1\text{H}$  NMR.<sup>17</sup> In the case of

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(15) Faller, J. W.; Rosan, A. M. *J. Am. Chem. Soc.* **1977**, *99*, 4858.

(16) This specificity may be under thermodynamic or kinetic control, a distinction we are examining through labeling studies: Wink, D. J.; Wang, N.-F., research in progress.

(6) In a typical reaction, 0.65 g (2 mmol) of  $\text{Cr}(\text{CO})_3(\text{P}(\text{OMe})_3)((E)\text{-1,3-pentadiene})$  in 10 mL of THF was cooled in a dry ice/2-propanol bath. One equivalent of potassium tri-*sec*-butylborohydride (Aldrich, 1.0 M in THF, 2.0 mL) was added while the solution was magnetically stirred. A milky precipitate forms quickly. After 5 min, a suspension of 1.5 g (7 mmol) of  $\text{NEt}_4\text{Br}$  in 10 mL of THF is added and the mixture is stirred 4 h in the dry ice bath and then stored in a freezer at  $-20^\circ\text{C}$  overnight. The yellow supernatant is filtered from the white solids at  $-20^\circ\text{C}$ , and an equal volume of ether is added on top of the solution. Fine yellow needles (0.54 g, 59%) of  $[\text{NEt}_4][\text{Cr}(\text{CO})_3(\text{P}(\text{OMe})_3)(\eta^3\text{-}(E,E)\text{-3-penten-2-yl})]$  form as the ether mixes into the THF over 48 h at  $-20^\circ\text{C}$ . Samples for elemental analysis were recrystallized from THF at  $-20^\circ\text{C}$ . A similar procedure with 0.60 g (1.9 mmol) of  $\text{Cr}(\text{CO})_3(\text{P}(\text{OMe})_3)(1,3\text{-butadiene})$  yielded 0.64 g of  $[\text{NEt}_4][\text{Cr}(\text{CO})_3(\text{P}(\text{OMe})_3)(\eta^3\text{-}(E)\text{-2-buten-1-yl})]$  (77%) and with 1.80 g (5.5 mmol) of  $\text{Cr}(\text{CO})_3(\text{P}(\text{OMe})_3)(2\text{-methyl-1,3-butadiene})$  yielded 0.98 g of  $[\text{NEt}_4][\text{Cr}(\text{CO})_3(\text{P}(\text{OMe})_3)(\eta^3\text{-}(E)\text{-2-methyl-2-buten-1-yl})]$  (39%).

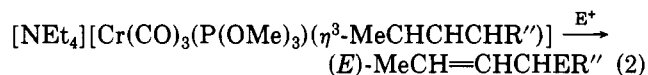
(7) While  $\text{NEt}_4\text{Br}$  is only sparingly soluble in THF, it does undergo the exchange reaction in the presence of organometallic anions: Wink, D. J. Ph.D. Thesis, Harvard University, Aug 1985.

(8) Paz-Sandoval, M.; Powell, P.; Drew, M. G. B.; Perutz, R. N. *Organometallics* **1984**, *3*, 1026.

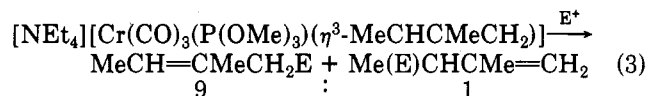
(9) The absence of resonances in the region of the  $^1\text{H}$  NMR spectrum downfield from  $\delta$  4, characteristic of a proton on the 2-position of an allyl, indicates the absence of a possible 1-methyl-2-buten-1-yl isomer.

(10) Crystals of tetraethylammonium tricarboxylate  $(\eta^3\text{-}(E,E)\text{-2-methyl-2-buten-1-yl})$ (trimethyl phosphite)chromium(1-),  $\text{C}_{19}\text{H}_{36}\text{CrN}_4\text{O}_7$ , formed with space group symmetry  $P2_12_12_1$  and cell constants  $a = 9.332$  (1)  $\text{\AA}$ ,  $b = 12.356$  (3)  $\text{\AA}$ , and  $c = 20.836$  (6)  $\text{\AA}$  with  $Z = 4$ . Of 1865 reflections measured, 1395 had  $I > 3\sigma(I)$ . Least-squares refinement of the positions and anisotropic thermal parameters of all the non-hydrogen atoms to minimize the function  $\Delta = w(|F_o| - |F_c|)^2$  with  $w = 1/(\sigma(F_o))^2$  gave  $R = 0.040$  and  $R_w = 0.047$ . Further information is provided in the supplementary material.

CF<sub>3</sub>CO<sub>2</sub>H and CO<sub>2</sub>, production of the simple olefin or β,γ-unsaturated acid is essentially instantaneous, while the reaction with SiMe<sub>3</sub>Cl takes a few hours. The addition occurs exclusively at the less hindered carbon for **1a** and **1b** (eq 2) and predominantly (>9:1) for **1c** (eq 3).



E = CO<sub>2</sub><sup>-</sup>, SiMe<sub>3</sub>, H



(17) In a typical reaction, treatment of a solution of the η<sup>3</sup>-2-buten-1-yl complex with an excess of trifluoroacetic acid produces an NMR spectrum with resonances at δ 5.41 (1 H, q) and 1.57 (3 H), indicative of *trans*-2-butene. There are no resonances in the regions δ 5.0–4.0 or 2.1–2.0, consistent with the absence of *cis*-2-butene or 1-butene. Similarly, treatment of a solution of the η<sup>3</sup>-2-buten-1-yl complex with carbon dioxide produces an NMR spectrum with resonances at δ 5.3–5.2 (2 H, m, 3,4-H), 2.60 (2 H, d of d, 2-CH<sub>2</sub>), and 1.62 (3 H, d, 5-CH<sub>3</sub>) indicative of 3-pentenoate with the formation of no terminal olefins such as 2-methyl-3-butenolate. Finally, when ca. 2 equiv of SiMe<sub>3</sub>Cl (Aldrich) is added to a solution of the anion in CD<sub>3</sub>CN, a slow color change from yellow to orange occurs. Examination by <sup>1</sup>H NMR indicates that the starting material is consumed in 1 h and (*Z*)-2-butenyltrimethylsilane is formed: δ 5.39 (1 H, m, 2- or 3-H), 5.24 (1 H, m, 2- or 3-H), 1.58 (3 H, d of qu, 4-CH<sub>3</sub>), 1.35 (2 H, d of t, CH<sub>2</sub>), -0.07 (9 H, s, Si(CH<sub>3</sub>)<sub>3</sub>). The other allyl anions studied react in a similar manner to give the appropriate olefins; spectroscopic details are presented in the supplementary material.

These simple regio- and stereospecific transformations of a coordinated diene into an olefin by the stepwise addition of hydride and an electrophile represent the first use of this metal ligand environment for the controlled transformation of hydrocarbons. Variation of the electrophile in the second step (a strategy also suggested by Brookhart's work with manganese dienes<sup>18</sup>) presents an alternative to the strategy of nucleophilic functionalization developed for cobalt<sup>19</sup> and molybdenum<sup>20</sup> dienes.

**Acknowledgment.** We are grateful to the donors of the Petroleum Research Fund, administered by the American Chemical Society, and the ARCO Foundation for financial support of this research.

**Supplementary Material Available:** Spectroscopic data for all new compounds, tables of the atomic positional and thermal parameters, complete bond distances and angles, and drawings of the complete asymmetric unit and atomic packing (9 pages); a listing of observed and structure factor amplitudes (7 pages). Ordering information is given on any current masthead page.

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(19) (a) Sautet, P.; Eisenstein, O.; Nicholas, K. M. *Organometallics* 1987, 6, 1845. (b) Barinelli, L. S.; Tao, K.; Nicholas, K. M. *Organometallics* 1986, 5, 588.

(20) (a) Pearson, A. J.; Blystone, S. L.; Roden, B. A. *Tetrahedron Lett.* 1987, 22, 2459. (b) Pearson, A. J.; Khan, Md. N. I.; Clardy, J. C.; Cunniff, H. J. *Am. Chem. Soc.* 1985, 107, 2748.

## Book Reviews

**Reactions of Coordinated Ligands. Vol. 1.** Edited by P. S. Braterman. Plenum, New York. 1986. xi + 1052 pages. \$135.00

Organotransition-metal chemistry has, since the discovery of ferrocene in 1951, attained considerable importance in organic synthesis, in both stoichiometric and catalytic processes. It is transformations of organic groups or molecules while they are coordinated to one or more metal centers that generally are involved in such chemistry. Such coordination often results in modifications of the reactivity of the free ligand—enhancing its reactivity toward a specified reagent or changing (hopefully for the better) the regioselectivity or stereoselectivity of its reaction with this reagent. Or, on the other hand, perhaps through coordination, free ligand reactivity can be “turned off” at some functional group in the molecule. In other instances, coordination to a metal center may serve to stabilize an otherwise fugitive species and permit its chemistry to be developed. In all of these processes reactions at coordinated ligands are involved. The present book is the first volume of a two-volume set in which this subject is developed in some detail by means of contributed chapters by experts in the areas about which they write. Volume 1 covers in 16 chapters the chemistry of carbon-bonded organic ligands. Volume 2 will cover the chemistry of heteroatom-bound ligands. The book is not as monumental as its 1052 pages might lead one to believe since it is a photoreproduction of double-spaced typescripts.

The 16 chapters in this book cover the following topics, all, as already noted, involving carbon-bonded organic ligand reactivity: 1-, 2-, and 3-carbon ligands, by P. S. Braterman (alkyl, alkene, and η<sup>3</sup>-allyl complexes); reactions of 1-carbon ligands in complexes of macrocycles, by M. D. Johnson; alkylidene complexes of the earlier transition metals, by R. R. Schrock; carbene complexes of groups VIA, VIIA, and VIII, by K.-H. Dötz; mechanistic aspects of the olefin metathesis reaction, by M. Leconte, J. M. Basset, F. Quignard, and C. Larroche; general aspects of carbonylation and related chemistry, by P. S. Braterman; promotion effects in transition-metal-catalyzed carbonylation, by G. P. Chiusoli, G.

Salerno, and M. Foa; hydride transfer to coordinated CO and related ligands, by C. E. L. Headford and W. R. Roper; reactions of coordinated isocyanides, by G. Crociani; formation and reactions of metallacycles, by G. Ingrosso; nucleophilic attack on coordinated alkenes, by J.-E. Bäckvall; asymmetric additions to double bonds, by P. A. Chaloner and D. Parker; reactions of coordinated acetylenes, by J. L. Davidson; reactions of coordinated dienes, by J. A. S. Howell; reactions of 5-carbon and larger ligands, by P. Powell.

Inspection of these chapter titles shows that the editor has achieved a good, but not complete, coverage of the subject of reactions of coordinated organic ligands. Another chapter that might have been included is one that deals with reactions of μ<sub>1</sub>-, μ<sub>2</sub>-, and μ<sub>3</sub>-carbyne complexes (why stop at carbenes?). Also, the chemistry of coordinated carbenium ions has received a good bit of study, and this interesting topic is not included. It is important to note that exhaustive coverage of the 16 topics mentioned above was not possible. Indeed, many of these topics would require a separate book of their own if anything even approaching exhaustive coverage were intended. Thus these chapters for the most part should be read as introductions to their respective subject.

This book was published in 1986. One might hope to find that its chapters cover the literature of their topics through the end of 1984. This, however, is not the case. The literature cutoff date seems to have been, in general, 1981–1982. Later work (from 1983, 1984 mostly) is brought in many of the chapters in a concluding section entitled “More Recent Developments”. Some of these sections even include references from the 1980 and 1981 literature. As with many multiauthor efforts, in this case there also must have been problems in completing the job. Nevertheless, if one takes into consideration that this book is not as up-to-date as it might have been, the reader will find it a useful one whose chapters will serve as very good introductions to the 16 topics covered.

The abbreviations used in this book are defined in a list following the last chapter and there is a subject index.

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