$$[^-O_2\dot{N}Ph-X-CO_2Co^{III}(NH_3)_5]^+ \xrightarrow{k_{et}} [O_2NPh-X-CO_2Co^{II}(NH_3)_5]^+ \tag{2}$$

$$[O_2NPh-X-CO_2Co^{II}(NH_3)_5]^+ \xrightarrow{H^+} Co^{2+}(aq) + 5NH_4^+ + O_2NPh-X-CO_2^- \tag{3}$$

Values of $k_{et}$ for the ortho, meta, and para isomers of nitrobenzoato complexes are given in Table I. Reduction potentials of these nitro radicals, when uncoordinated, have been previously determined by electron-transfer reaction with duroquinone.[15] The potentials vary little and are in the range −0.396 V for the para isomer to −0.433 V for the meta analogue. The dissimilar isomeric trend and the $>10^3$-fold variation in the observed $k_{et}$ values for the complexed radical anions implicate a significant intrinsic (i.e., nonthermodynamic) component in the intramolecular electron-transfer process. That the redox potentials of the ortho, meta, and para isomers of dinitrobenzene and dinitrobenzoate free radicals are only 0.05–0.14 V less reducing than the nitrobenzoate analogues[15] provides a basis for generalization. Given the *same* donor, lead-in, and acceptor groups for the intramolecular electron-transfer process of all the complexes reported in Table I, it is unlikely that the $10^5$-fold variation in the observed $k_{et}$ values derives solely from an overall driving force difference dependent only on the bridging group. This group can, however, clearly change the approach to, and nature of, the transition state.

The $k_{et}$ trend observed for the nitrobenzoatao series ($o > p > m$) was previously explained[15] in terms of the significant electron spin density distribution at or adjacent to the lead-in group; the spin density is a maximum at the positions ortho and para to the nitro radical and negligible in the meta position. In the ortho case, direct nitro radical overlap with carboxylate and/or metal orbitals probably facilitates the transfer process. Undoubtedly, introduction of a $CH_2$ group greatly reduces the spin density at the lead-in carboxylate. However, close examination of Ealing CPK molecular model structures shows that the ortho overlap is also possible in the nitrophenylacetato case and that for the para isomer a configuration is possible in which the carboxylate group overlaps the ring carbon para to the nitro radical. Such overlap is eliminated in the more rigidly protruding *trans*-cinnamato series of complexes. The rate data do suggest, however, that the olefinic linkage may provide an effective means of delocalizing the spin density through the extended π system for the ortho and para isomers. In contrast, the meta derivative has no extended π conjugation, negligible spin density adjacent to the ethylenic linkage, and ostensibly no nitro radical–carboxylate nor –metal orbital overlap configurational possibilities. Consequently, the observed value of $k_{et} = 3.1\ s^{-1}$ for *m*-cinnamato may represent an unassisted transfer of an electron through the ligand bridging system.

Table I also includes values of $k_{et}$ for *p*-nitrophenylcarboxylate complexes having bridging groups varying in both length and flexibility. Examinations of the molecular models show that among the multitude of possible configurations, a *common* positional arrangement arises for X = $CH_2$, $CH_2CH_2CH_2$, and $CONHCH_2CONHCH_2$: proximity of a carboxylate oxygen to within ~1 Å of the para carbon of the aromatic ring (based on van der Waals radii). This configuration may be further enhanced by H-bonding interaction of the nitro oxygens with the ammonia hydrogens of the $Co(NH_3)_5$ moiety. Thus, despite the long-chain character of the bridging units, electron density can be directed to the lead-in group by effectively bypassing through-chain mediation. The degree of favorable π-orbital vertical overlap is most nearly attained for the dipeptide ligand structure; this complex exhibits a $k_{et}$ value larger than that of the two alkyl chain analogues. The more rigid monopeptide complex has *no* configuration that allows such orbital overlap. The further absence of direct configurational interaction at the metal or lead-in group and resonance delocalization through the bridging unit is consistent with the $k_{et}$ value for X = $CONHCH_2$, again reflecting an unassisted through-chain electron transfer.

A pattern of mechanisms of intramolecular electron transfer emerges from the rate data and configurational considerations. The passage of electron density from the donor to the acceptor site can occur either through the bridging unit or via a path that effectively bypasses it. π bonding within the bridge may facilitate through-chain transfer because of favorable extended π conjugation between the donor and lead-in sites. In the absence of such resonance assistance, the through-chain rate of transfer is markedly decreased. Consistent with the similarity of the two lowest observed $k_{et}$ values must be the dominance of fundamental rate-determining parameters: Franck–Condon barriers, acceptor/lead-in orbital symmetry matching, and overall driving force. Indeed, the considerable lower intramolecular rates of similarly accepting binuclear complexes may reflect lower overall thermodynamic drives in those cases.[6]

A favorable disposition of the donor to the acceptor or lead-in site may well create a more facile path of electron transfer than is offered by the through-chain alternative. We propose the most effective configurations to be those that allow orbital overlap between the nitro group and the metal center, the nitro group and the coordinated carboxylate, and the para ring carbon and the carboxylate. In addition, there is the possibility of electron transfer without direct orbital overlap; an outer-sphere transfer mechanism of related phenomena has been previously proposed.[19] Apart from the obvious relevance of these results to the "chemical" mechanism of electron transfer,[20] the coordinated ligand radical species serve as models for even more complex intramolecular electron transfer. In the case of electron transfer in large biomolecules, the contributions of "through-chain" and "bypass" transfer must be assessed.

**Registry No.** $[^-O_2\dot{N}Ph\text{-}o\text{-}CH_2CO_2Co^{III}(NH_3)_5]^+$, 74298-07-0; $[^-O_2\dot{N}Ph\text{-}m\text{-}CH_2CO_2Co^{III}(NH_3)_5]^+$, 74298-08-1; $[^-O_2\dot{N}Ph\text{-}p\text{-}CH_2CO_2Co^{III}(NH_3)_5]^+$, 74298-09-2; $[^-O_2\dot{N}Ph\text{-}o\text{-}CH{=}CHCO_2Co^{III}(NH_3)_5]^+$, 74298-10-5; $[^-O_2\dot{N}Ph\text{-}m\text{-}CH{=}CHCO_2Co^{III}(NH_3)_5]^+$, 74298-11-6; $[^-O_2\dot{N}Ph\text{-}p\text{-}CH{=}CHCO_2Co^{III}(NH_3)_5]^+$, 74298-12-7; $[^-O_2\dot{N}Ph\text{-}p\text{-}CH_2CH_2CH_2CO_2Co^{III}(NH_3)_5]^+$, 74298-13-8; $[^-O_2\dot{N}Ph\text{-}p\text{-}CONHCH_2CO_2Co^{III}(NH_3)_5]^+$, 74298-14-9; $[^-O_2\dot{N}Ph\text{-}p\text{-}CONHCH_2CONHCH_2CO_2Co^{III}(NH_3)_5]^+$, 74298-15-0.

---

(19) K. Wieghardt, H. Cohen, and D. Meyerstein, *Ber. Bunsenges. Phys. Chem.*, **82**, 388 (1978).

(20) A. Haim, *Acc. Chem. Res.*, **8**, 264 (1975).

---

Contribution from the Central Leather Research Institute,
Adyar, Madras 600 020, India

## Substitution Lability of Chromium(III) Complexes with Ground-State Distortion

D. Rajendra Prasad, T. Ramasami,* D. Ramaswamy,
and M. Santappa

*Received March 4, 1980*

The substitution inertness of the $d^3$ chromium(III) ion in aquo-ligand environments has already been established.[1] However, there are some exceptions in aquo(ethylenedi-

---

(1) Swaddle, T. W. *Coord. Chem. Rev.* **1974**, *14*, 217.

0020-1669/80/1319-3181$01.00/0

aminetetraacetato)chromium(III), $Cr^{III}(EDTA)(H_2O)^-$,[2] and in aquohydroxo[tetrakis(*p*-sulfonatophenyl)porphine]chromium(III), $Cr^{III}(TPPS)(H_2O)OH$,[3] where the substitution of water ligand occurs at stopped-flow rates ($t_{1/2}$ = 30 ms–70 s). In the complex $Cr^{III}(EDTA)(H_2O)^-$, EDTA is a pentadentate ligand and Cr(III) is in a six-coordinate (undistorted) octahedral geometry[4] with a carboxylate ion being free. The carboxylate ion is believed to form a seven-coordinate intermediate leading to the labilization of the aquo ligand by altering the energy of the transition state.[2] On the other hand, the effects of distortion in the ground-state geometry on the substitution behavior of Cr(III) have not yet been systematically investigated due to the scarcity of suitable Cr(III) complexes.[5] One of the well-established examples of a distorted octahedral Cr(III) complex is diaquo[*N*,*N'*-ethylenebis(salicylideniminato)]chromium(III) perchlorate, $[Cr(salen)(H_2O)_2]ClO_4$.[6]

The X-ray structure of the complex reveals an axial distortion with Cr(III) being slightly above the plane of the "salen" ring donor atoms.[7] We report here the kinetics of the equilibration of thiocyanate and azide ions with the complex $Cr(salen)(H_2O)_2^+$ and its conjugate base.

## Experimental Section

The complex $[Cr(salen)(H_2O)_2]ClO_4$ was prepared by standard procedures.[6,7] The complexes [Cr(salen)($H_2O$)NCS] and [Cr(salen)($H_2O$)$N_3$] were prepared by reacting $[Cr(salen)(H_2O)_2]ClO_4$ with lithium thiocyanate and sodium azide, respectively, and identified by their UV–visible spectra and elemental analyses. The concentrations of lithium thiocyanate and sodium azide were estimated according to standard methods.[8]

The apparent equilibrium constant, *K*, for the overall reaction of $Cr(salen)(H_2O)_2^+$ and its conjugate base with $NCS^-$ and $N_3^-$ was measured with the use of a Beckman Model 26 spectrophotometer at 320 nm as a function of hydrogen ion concentration: $[H^+] = (0.40–10) \times 10^{-6}$ M for $NCS^-$ and $[H^+] = (0.63–20) \times 10^{-7}$ M for $N_3^-$. At any particular $[H^+]$, *K* may be obtained from average absorptivities, $\epsilon_R$ of reactants ($[Cr(salen)(H_2O)_2]^+$ and its conjugate base) and $\epsilon_P$ of products ([Cr(salen)($H_2O$)X] and its conjugate base), as given by eq 1 where $\epsilon_{obsd}$ is the molar absorptivity of a mixture

$$\epsilon_{obsd} = (\epsilon_R + \epsilon_P K[X^-])(1 + K[X^-])^{-1} \quad (1)$$

of reactants and products at any given $[H^+]$ and $[X^-] = [NCS^-]$ or $[N_3^-]$.

The kinetics of the overall equilibration process were measured by using an Aminco stopped-flow spectrophotometer in the concentration ranges $[NCS^-]$ = 0.012–0.25 M, $[N_3^-]$ = 0.012–0.25 M, [Cr(III)] = $2 \times 10^{-4}$ M, $[H^+] = (0.40–10) \times 10^{-6}$ M for $NCS^-$ and $(0.63–20) \times 10^{-7}$ M for $N_3^-$ (*I* = 1.0 M ($LiClO_4$) and *T* = 30 °C). The pseudo-first-order plots were linear to about 4 half-lives, and only one kinetic step was observed on the flow time scale.

## Results

The overall equilibrium constant, *K*, as well as the rate of equilibration, $k_{eq}$, exhibited a marked dependence on $[H^+]$.

**Table I.** Spectrophotometrically Determined Equilibrium Constants for the Equilibration of $X^-$ ($NCS^-$ and $N_3^-$) with $Cr(salen)(H_2O)_2^+$

| $X^- = NCS^-$ | | $X^- = N_3^-$ | |
|---|---|---|---|
| $10^6[H^+]$/M | $K/M^{-1}$ | $10^7[H^+]$/M | $K/M^{-1}$ |
| 10.00 | 2.0 | 20.00 | 30.0 |
| 4.00 | 2.8 | 10.00 | 16.1 |
| 2.00 | 3.3 | 2.50 | 9.4 |
| 0.90 | 6.5 | 1.25 | 6.7 |
| 0.40 | 7.3 | 0.63 | 4.9 |

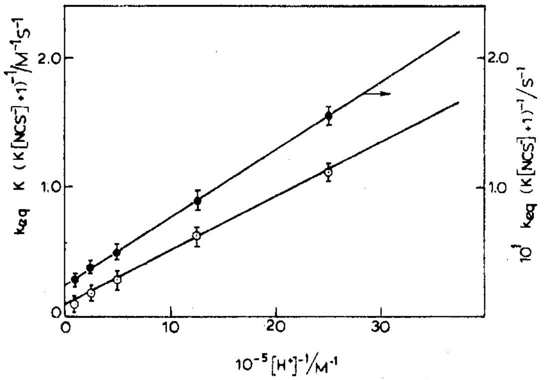


**Figure 1.** Dependence of $k_{eq}K(1 + K[NCS^-])^{-1}$ and $k_{eq}(1 + K[NCS^-])^{-1}$ on $[H^+]^{-1}$ (*T* = 30 °C, *I* = 1.0 M).

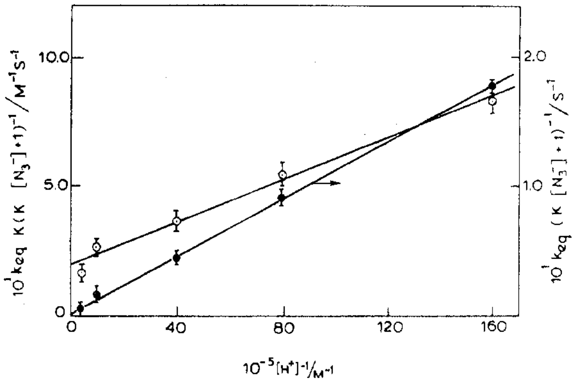
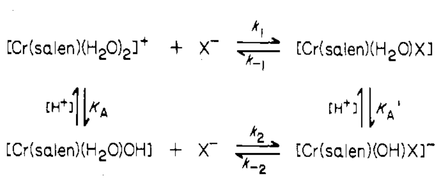


**Figure 2.** Dependence of $k_{eq}K(1 + K[N_3^-])^{-1}$ and $k_{eq}(1 + K[N_3^-])^{-1}$ on $[H^+]^{-1}$ (*T* = 30 °C, *I* = 1.0 M).

The equilibration rate, $k_{eq}$, varied linearly with $[NCS^-]$ or $[N_3^-]$ at any particular $[H^+]$. The overall reaction scheme can be written as in eq 2.



(2)

On the basis that $[H^+] \gg K_A$ ($5 \times 10^{-9}$ M), the rate law for eq 2 can be derived as in eq 3, where $[X^-] = [NCS^-]$ or

$$k_{eq} = k_{-1} + k_{-2}K_A'[H^+]^{-1} + (k_1 + k_2K_A[H^+]^{-1})[X^-] \quad (3)$$

$[N_3^-]$ and the overall equilibrium constant, *K*, can be expressed as in eq 4, where $K_1 = k_1/k_{-1}$ and $K_2 = k_2/k_{-2}$.

$$K = (K_1[H^+] + K_2K_A)([H^+] + K_A)^{-1} \quad (4)$$

The kinetically determined overall equilibrium constants $(k_1 + k_2K_A/[H^+])(k_{-1} + k_{-2}K_A'/[H^+])^{-1}$ at various $[H^+]$ were in satisfactory agreement (within a factor of 2) with the spectrophotometrically estimated value given in Table I. The

---

[2] Ogino, H.; Shimura, M.; Tanaka, N. *Inorg. Chem.* **1979**, *18*, 2497 and references therein. Sulfab, Y.; Taylor, R. S.; Sykes, A. G. *Ibid.* **1976**, *15*, 2388.

[3] Fleischer, E. B.; Krishnamurthy, M. *J. Am. Chem. Soc.* **1971**, *93*, 3784.

[4] (a) Hoard, J. L.; Kennard, C. H. L.; Smith, G. S. *Inorg. Chem.* **1963**, *2*, 1316. (b) Lin, G. H. Y.; Leggett, J. D.; Wing, R. M. *Acta Crystallogr., Sect. B* **1973**, *B29*, 1023.

[5] Deutsch, E.; Asher, L. E. *Inorg. Nucl. Chem. Lett.* **1977**, *13*, 91. Stein, C.; Bouma, S.; Carlson, J.; Cornelius, C.; Maeda, J.; Weschler, C.; Deutsch, E.; Hodgson, K. O. *Inorg. Chem.* **1976**, *15*, 1183.

[6] Yamada, S.; Iwasaki, K. *Inorg. Chim. Acta* **1971**, *5*, 3 and references therein.

[7] Coggon, P.; McPhail, A. T.; Mabbs, F. E.; Richards, A.; Thornley, A. S. *J. Chem. Soc. A* **1970**, 3296.

[8] Vogel, A. I. "A Text Book of Quantitative Inorganic Analysis", 3rd ed.; Longmans, Green and Co.: London, 1962; pp 275, 645.

rearrangement of eq 3 as a function of $K$ gave (5) and (6).

$$k_1 + k_2K_A[H^+]^{-1} = k_{eq}K(K[X^-] + 1)^{-1} \qquad (5)$$

$$k_{-1} + k_{-2}K_A'[H^+]^{-1} = k_{eq}(K[X^-] + 1)^{-1} \qquad (6)$$

The plots of $k_{eq}K(K[X^-] + 1)^{-1}$ and $k_{eq}(K[X^-] + 1)^{-1}$ against $[H^+]^{-1}$ were linear as shown in Figures 1 and 2 and gave the values $k_1 = 0.10$ ($NCS^-$) and 0.20 $M^{-1}\ s^{-1}$ ($N_3^-$), $k_2K_A = 42 \times 10^{-8}$ ($NCS^-$) and $3.8 \times 10^{-8}\ s^{-1}$ ($N_3^-$), $k_{-1} = 0.024$ ($NCS^-$) and 0.001 $s^{-1}$ ($N_3^-$), and $k_{-2}K_A' = 52 \times 10^{-8}$ ($NCS^-$) and $1.1 \times 10^{-8}$ M $s^{-1}$ ($N_3^-$).

## Discussion

Although there are two replaceable aquo ligands in the complex $[Cr(salen)(H_2O)_2]^+$, only monosubstitution occurred on the stopped-flow time scale. This is consistent with the widely differing $Cr^{III}–OH_2$ bond lengths in the complex, viz., 1.923 and 2.085 Å.[7] With the conjugate base of $[Cr(salen)(H_2O)_2]^+$, the observed single-stage kinetics is to be expected because the direct substitution of $OH^-$ by other nucleophiles is considered unlikely in this class of compounds. The observed direct dependence of the forward rates on $[X^-]$ at any $[H^+]$ does not provide compelling evidence for dissociative mechanisms for the reactions of either $Cr(salen)(H_2O)_2^+$ or its conjugate base, though X-ray structure data would suggest dissociative processes. However the similarity of the $k_1$ terms for the $Cr(salen)(H_2O)_2^+$ reactions with $NCS^-$ (0.10 $M^{-1}\ s^{-1}$) and $N_3^-$ (0.20 $M^{-1}\ s^{-1}$), after making allowance for the statistical factor for $N_3^-$,[9] is in keeping with a dissociative $I_d$-type process with small ion-pairing constants. Assumption of low ion-pairing constants for the reactions of uninegative ions like $NCS^-$ and $N_3^-$ with $Cr(salen)(H_2O)_2^+$ and its conjugate base will be consistent with the low charges on the complex reactants. The reverse rates for the complexes $[Cr(salen)(H_2O)X]$ with $X^- = NCS^-$ (0.024 $s^{-1}$) and $N_3^-$ (0.001 $s^{-1}$) and their equilibrium constants [$NCS^-$ (2.2 $M^{-1}$) and $N_3^-$ (50 $M^{-1}$)] are also in keeping with a dissociative mechanism involving rate-limiting $M–OH_2$ bond dissociation. On the other hand, magnetic measurements on the conjugate base of $Cr(salen)(H_2O)_2^+$ as a solid indicate a five-coordinate [Cr(salen)OH] structure in the ground state.[7] If the conjugate base were to be six-coordinate in solution and the mechanism involved rate-limiting loss of the sixth ligand, viz., water, the two similarly charged anions $NCS^-$ and $N_3^-$ should have similar $k_2K_A$ terms. The values obtained for $k_2K_A$ ($4.2 \times 10^{-7}$ M $s^{-1}$ for $NCS^-$ and $3.8 \times 10^{-8}$ M $s^{-1}$ for $N_3^-$) were markedly different. On the other hand, if the conjugate base were to be a five-coordinate [Cr(salen)OH] complex in solution also, the nucleophiles such as $N_3^-$ and $NCS^-$ may undergo bimolecular addition reactions to give six-coordinate products with large variations in the $k_2$ term. In light of the recent reports on the interesting features of complexes with a five-coördinate structure,[10] further work is in progress to distinguish between the two possible mechanisms, viz., nucleophilic substitution and bimolecular addition for the reactions of the conjugate base of $[Cr(salen)(H_2O)_2]^+$.

**Acknowledgment.** The authors thank the Director of CLRI for his permission to publish the work and for a grant to D.R.P.

**Registry No.** $Cr(salen)(H_2O)_2^+$, 47248-17-9; $NCS^-$, 302-04-5; $N_3^-$, 14343-69-2.

---

(9) Nishizawa, M.; Saito, K. *Inorg. Chem.* **1978**, *17*, 3676. Patai, S., Ed. "The Chemistry of The Azido Group"; Wiley-Interscience: New York, 1971.

(10) Ramasami, T.; Espenson, J. H. *Inorg. Chem.* **1980**, *19*, 1523.

---

Contribution from the Department of Chemistry,
Northwestern University, Evanston, Illinois 60201

# Comparisons of Ir–Cl Stretching Frequencies and $^{31}P$ Chemical Shifts in *trans*-$IrCl[P(C_6H_5)_3]_2L$ Complexes

K. Dahl Schramm, T. H. Tulip, and James A. Ibers*

*Received March 6, 1980*

Many techniques have been used to probe the trans and, recently, cis (bond-weakening) influences of ligands in transition-metal complexes.(1) For square-planar Pt(II) complexes in particular extensive direct (structural)[2,3] and indirect (spectroscopic) information has been amassed. These indirect indicators include Pt–halide and –hydride stretching frequencies,[4,5] $^{195}Pt–^{31}P$ and $–^1H$ coupling constants,[6–8] and $^{195}Pt$ and $^1H$ chemical shifts.[8,9] Although analogous square-planar Ir(I) complexes have attracted much attention,[10] trans-influence data for these compounds are scarce. Many of the spectroscopic indicators available in Pt(II) systems do not apply to Ir(I) complexes. In the absence of structural results, of which there are only a few reports, we have been forced to use the Ir–Cl stretching frequency as our sole indicator of trans influence in complexes of the type *trans*-$IrCl(PPh_3)_2L$ [Ph = $C_6H_5$; L = $N_2Ar^+$, $N_2C_5X_4$ (Ar = aryl; X = Cl, Br)].[11,12] In an effort to find additional trans-influence probes for these complexes, we have examined their $^{31}P$ NMR chemical shifts. We reasoned that within a closely related series of complexes differences in this parameter will arise predominantly from variations in the inductive-shielding contribution of the metal. These shieldings should reflect relative metal-centered electron densities which, although not themselves direct measures of the trans influences of the ligands, will be closely related as both are functions of the effective L → M charge transfer. The obvious ease with which the $^{31}P$ NMR method can be applied to complexes difficult or impossible to isolate or stable only at low temperature and even to mixtures of complexes makes it a very attractive alternative to infrared spectroscopy which generally requires isolable, relatively pure solids. Here we describe the results of our comparisons of Ir–Cl stretching frequencies and $^{31}P$ chemical shifts for a series of complexes, *trans*-$IrCl(PPh_3)_2L$.

## Experimental Section

All procedures were carried out under inert atmosphere. The complexes *trans*-$IrCl(PPh_3)_2L$, where L = CO,[13] CS,[14] $N_2$,[15] $C_2H_4$,[16] $PPh_3$,[17] $NO^+$,[18] $N_2Ar^+$,[11] and $N_2C_5Cl_4$,[12] were prepared by literature

---

(1) Appleton, T. G.; Clark, H. C.; Manzer, L. E. *Coord. Chem. Rev.* **1973**, *10*, 335.

(2) (a) Mason, R.; Meek, D. W. *Angew. Chem., Int. Ed. Engl.* **1978**, *17*, 183. (b) McWeeny, R.; Mason, R.; Towl, A. D. C. *Discuss. Faraday Soc.* **1969**, *47*, 20.

(3) Manojlović-Muir, Lj.; Muir, K. W.; Solomun, T. *J. Organomet. Chem.* **1977**, *142*, 265.

(4) Adams, D. M.; Chatt, J.; Gerratt, J.; Westland, A. D. *J. Chem. Soc.* **1964**, 734.

(5) Chatt, J. *Proc. Chem. Soc., London* **1962**, 318.

(6) Tau, K. D.; Meek, D. W. *Inorg. Chem.* **1979**, *18*, 3574.

(7) (a) Mather, G. G.; Pidcock, A.; Rapsey, G. J. N. *J. Chem. Soc., Dalton Trans.* **1973**, 2095. (b) Hitchcock, P. B.; Jacobson, B.; Pidcock, A. *Ibid.* **1977**, 2038.

(8) Powell J.; Shaw, B. L. *J. Chem. Soc.* **1965**, 3879.

(9) Dean, R. R.; Green, J. C. *J. Chem. Soc. A* **1968**, 3047.

(10) Vaska, L. *Acc. Chem. Res.* **1968**, *1*, 335.

(11) Haymore, B. L.; Ibers, J. A. *J. Am. Chem. Soc.* **1973**, *95*, 3052.

(12) Schramm, K. D.; Ibers, J. A. *J. Am. Chem. Soc.* **1978**, *100*, 2932; *Inorg. Chem.* **1980**, *19*, 1231.

(13) Collman, J. P.; Sears, C. T.; Kubota, M. *Inorg. Synth.* **1968**, *11*, 101.

(14) Kubota, M. *Inorg. Synth.* **1979**, *19*, 206.

(15) Collman, J. P.; Kubota, M.; Vastine, F. D.; Sun, J. Y.; Kang, J. W. *J. Am. Chem. Soc.* **1968**, *90*, 5430.

(16) van der Ent, A.; Onderdelinden, A. L. *Inorg. Chim. Acta* **1973**, *7*, 203.

(17) Bennett, M. A.; Milner, D. L. *J. Am. Chem. Soc.* **1969**, *91*, 6983.

(18) Fitzgerald, R. J.; Lin, H.-M. W. *Inorg. Synth.* **1976**, *16*, 41.

0020-1669/80/1319-3183$01.00/0