indicate the presence of (py·OH) oligomers in the product solutions.

The present results indicate that the iron(III) center must have a pyridine-like ligand for a hydroxide-induced electron transfer to occur (eq 1). This is confirmed by the failure of OH^- to induce the reduction of Fe^{III}TPP$^+$ in a 2,6-dimethylpyridine solution. Although a pyridine-like solvent, it is unable to bind to the iron(III) center because of steric hindrance.[13]

The reduction of Fe(III)–porphyrins by piperidine[5] or *n*-hexanethiol[7] is not feasible on thermodynamic grounds[14] and requires activation by deprotonation, either by base or by displacement of the protons by a good Lewis acid such as ferric ion. The hydroxide-induced reduction via pyridine, on the other hand, occurs by nucleophilic addition of OH^- to the aromatic ring. Although the net reaction (eq 1) is similar to that for other ligand-induced reductions of Fe(III)–porphyrins,[6,7] the activation of bound pyridine by OH^- to produce a strong reducing agent is unique.

Acknowledgment. This work was supported by the National Science Foundation under Grant No. CHE82-12299. We are grateful to Professors Joan Valentine (University of California, Los Angeles), Winslow Caughey (Colorado State University, Fort Collins), and Karl Kadish (University of Houston) for helpful discussions and to Dr. Richard Kondrat for the mass spectral analyses (departmental VG-ZAB mass spectrometer funded in part by a grant from the National Science Foundation).

Registry No. Fe^{III}TPP$^+$Cl$^-$, 14187-12-3; Fe^{III}TPP(py)$_2^+$, 60542-64-5; Fe^{II}TPP(py)$_2$, 16999-25-0; Fe^{III}TPP(Me-im)$_2^+$, 52155-25-6; Fe^{III}TPP(OH$^-$), 25482-26-2; Fe^{II}TPP, 16591-56-3; Fe^{III}TPP$^+$ClO$_4^-$, 59370-87-5; OH$^-$, 14280-30-9; (phenanthroline)iron(III) perchlorate, 14634-90-3; tris(bipyridyl)iron(III) perchlorate, 15388-50-8.

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(13) Walker, F. A. *J. Magn. Reson.* **1974**, *15*, 201.
(14) Unpublished electrochemical data.

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Synthesis of the Dinuclear Manganese(I) Species $[Mn_2(CO)_6(SPh)_3]^-$ and Its Oxidation to a Mixed-Valent Dimanganese(I,II) Complex

Sir:

Recently we described[1] the reactions of $Mo(CO)_6$ with alkane- and arenethiolates to yield the dinuclear Mo(0) species $[Mo_2(CO)_8(SR)_2]^{2-}$. These compounds were remarkable in that, although relatively simple in nature and in method of preparation, they had not been previously reported and also because they displayed a reversible two-electron oxidation in a single step. This latter behavior was attributed to metal–metal bond formation accompanied by structural rearrangement in the Mo(I) product $Mo_2(CO)_8(SR)_2$ to maintain an 18-electron configuration about each Mo. In an attempt to extend this type of chemistry to other metal systems, we have begun a study of the reactivity of dimanganese decacarbonyl with thiolate ions and herein describe some of our initial results.

Reaction of $Mn_2(CO)_{10}$ with 3 equiv of $[Et_4N][SPh]$ in MeCN for 18 h at 50 °C gave a yellow reaction mixture which, after concentration and addition of *i*-PrOH, deposited a yellow-orange crystalline solid.[2] Elemental analysis[3] and CO evolution data[4] were consistent with the formulation $[Et_4N][Mn_2(CO)_6(SPh)_3]$ (**1**), and plots of equivalent conductivity in MeCN vs. the square root of concentration were linear with slopes of ca. −200 units. This value is consistent with that for 1:1 electrolytes[1,5,6] and indicates that the above formulation (and not an oligomer of it) is correct for the product. The IR spectrum of **1** contains bands at 2000 (m), 1950 (sh), 1935 (s), and 1915 (s) cm^{-1}, consistent with the presence of terminal CO groups, but none below 1900 cm^{-1}, indicating the absence of bridging carbonyl. The combined spectral and analytical data suggest that the most reasonable formulation for the product consists of two $Mn(CO)_3$ fragments bridged by three μ-SPh groups, a structural arrangement that satisfies the 18-electron rule for both metals without invoking metal–metal bonding.

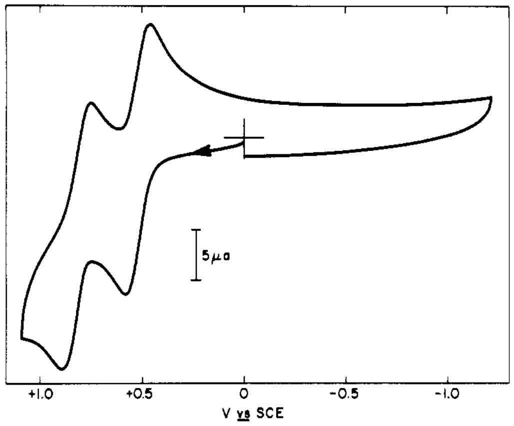
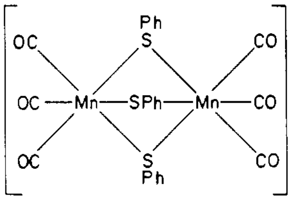


Figure 1. Cyclic voltammogram of a 1.41 mM solution of **1** in DMF at 200 mV/s scan rate and −45 °C.



The product is formally a Mn(I) dimer, and its synthesis was not anticipated from a Mn(0) starting material under anaerobic conditions. Although the mechanism of this reaction is unknown, it is pertinent to note that the halide analogues of **1**, $[Mn_2(CO)_6X_3]^-$, were prepared[7] by photolysis of $Mn_2(CO)_{10}$ in the presence of $[Et_4N]X$. Like the aforementioned molybdenum systems, it is remarkable that these simple dinuclear thiolate complexes have apparently not been reported previously. Other known[8,9] manganese–carbonyl–thiolate complexes include $[Mn_4(CO)_{12}(SR)_4]$ and $[Mn_2(CO)_8(SR)_2]$.

The electrochemistry of **1** is strikingly different from that of $[Mo_2(CO)_8(SPh)_2]^{2-}$. The cyclic voltammogram[10] of the man-

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(1) Zhuang, B.; McDonald, J. W.; Schultz, F. A.; Newton, W. E. *Organometallics* **1984**, *3*, 943.
(2) All reactions were carried out under an inert atmosphere by using degassed solvents and Schlenk tube techniques. On the basis of the proposed formulation, the yield was 45%. The synthetic method used here is virtually identical with that employed for $[Et_4N]_2[Mo_2(CO)_8(SR)_2]$ described in ref 1.
(3) Anal. Calcd. for $[Et_4N][Mn_2(CO)_6(SPh)_3]$ ($C_{32}H_{35}NO_6Mn_2S_3$): C, 52.24; H, 4.76; N, 1.90. Found: C, 52.24; H, 4.92; N, 1.97.
(4) The CO evolved on oxidation of the complex with excess Cl_2 was transferred by Toepler pumping to a calibrated manometric system and quantitated. The amount of CO collected was 97% of that predicted on the basis of this formulation.
(5) Davison, A.; Howe, D. V.; Shawl, E. T. *Inorg. Chem.* **1967**, *6*, 458.
(6) Callahan, K. P.; Cichon, E. J. *Inorg. Chem.* **1981**, *20*, 1941.
(7) Cihonski, J. L.; Walker, M. L.; Levenson, R. A. *J. Organomet. Chem.* **1975**, *102*, 335. These compounds were prepared in better yield from $Mn(CO)_5X$: Brisdon, B. J.; Edwards, D. A.; White, J. W. *J. Organomet. Chem.* **1978**, *161*, 233.
(8) (a) Jaintner, P. *J. Organomet. Chem.* **1981**, *210*, 353. (b) Jonson, B. F. G.; Pollick, P. J.; Williams, I. G.; Wojcicki, A. *Inorg. Chem.* **1968**, *7*, 831. (c) Abel, E. W.; Crosse, B. C. *J. Chem. Soc. A* **1966**, 1141.
(9) (a) Alper, H.; Paik, H.-N. *J. Organomet. Chem.* **1976**, *121*, 225. (b) Osborne, A. G.; Stone, F. G. A. *J. Chem. Soc. A* **1966**, 1143. (c) Treichel, P. M.; Morris, J. H.; Stone, F. G. A. *J. Chem. Soc. A* **1963**, 720.

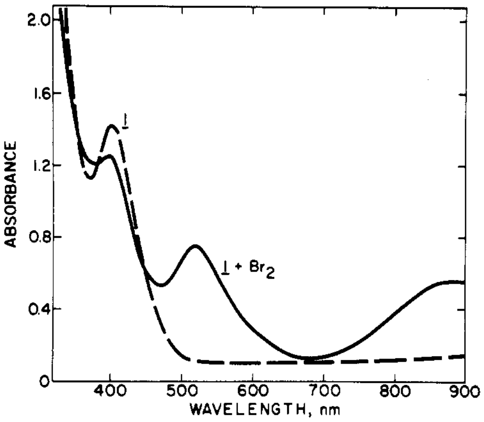

Figure 2. Electronic spectral change on Br_2 oxidation of **1** to **at** −32 °C in CH_2Cl_2. The concentration of **1** was 0.054 mM, and the spectra were obtained in a low-temperature cell with a 5.7-cm path length.

ganese species (Figure 1) exhibits reversible oxidation waves at +0.52 and +0.82 V vs. SCE with current parameters[11] consistent with sequential one-electron oxidations to form $[Mn_2(CO)_6(SPh)_3]$ (**2**) and $[Mn_2(CO)_6(SPh)_3]^+$ (**3**) as shown in eq 1, whereas the molybdenum complex is simultaneously oxidized by two electrons under similar conditions (eq 2).[13] This latter multielectron behavior[1] (and that for related phosphido-bridged iron carbonyl species[14]) is thought to result from metal–metal bond formation and might have been anticipated for **1**, since generation of a Mn–Mn bond gives an 18-electron configuration about each metal in **3**. The fact that this behavior is *not* observed is likely due to steric hindrance in the μ-SPh bridges, making metal–metal bond formation less favorable. Comparison of the dinuclear Mo and Mn species demonstrates the striking effect of the nature of the bridge on the redox properties of these metal–carbonyl–thiolate systems.

$$[Mn_2(CO)_6(SPh)_3]^- \underset{+e^-}{\overset{-e^-}{\rightleftharpoons}} [Mn_2(CO)_6(SPh)_3] \underset{+e^-}{\overset{-e^-}{\rightleftharpoons}} [Mn_2(CO)_6(SPh)_3]^+ \quad (1)$$

$$[Mo_2(CO)_8(SPh)_2]^{2-} \rightleftharpoons [Mo_2(CO)_8(SPh)_2] + 2e^- \quad (2)$$

When the CV experiment was carried out at room temperature, the i_{pa}/i_{pc} ratio of both couples shown in Figure 1 became greater than unity at slow scan rates, indicating some thermal instability of **2** and **3**. At −45 °C, however, this increase in peak current ratio was not observed, and thus attempts were made to generate the one-electron-oxidized species **2** in situ at low temperature.[15] Addition of 1 equiv of Br_2 to solutions of **1** in CH_2Cl_2 or MeCN at −40 °C results in the immediate generation of a deep red reaction mixture that is relatively stable at low temperature and that yields a cyclic voltammogram identical with that of **1**.[16] The electronic spectral change characterizing the oxidation of **1** to **2** is shown in Figure 2, with the latter species characterized by new bands at 519 and 880 nm. Likewise, after Br_2 oxidation of **1** at −40 °C, an aliquot of the reaction mixture was rapidly transferred to a 1-mm-i.d. capillary tube and the fluid-solution EPR spectrum of the oxidized species obtained. These spectra (Figure 3) are characterized by a symmetric 11-line pattern indicative of a single spin interacting with two equivalent ^{55}Mn nuclei ($I = {}^5/_2$) and confirm that the in-situ-generated complex **2** is the postulated Mn(I)–Mn(II) species $[Mn_2(CO)_6(SPh)_3]$. It is evident from Figure 3 that, although the overall shape of the hyperfine pattern varies slightly with solvent, neither the center position ($g = 2.017$) nor the ^{55}Mn hyperfine parameter ($A = 33$ G) is a function of the medium. The net $S = {}^1/_2$ ground state is likely due to spin coupling between the Mn(I) and Mn(II) entities, although it is not known at present whether these metals are high or low spin.

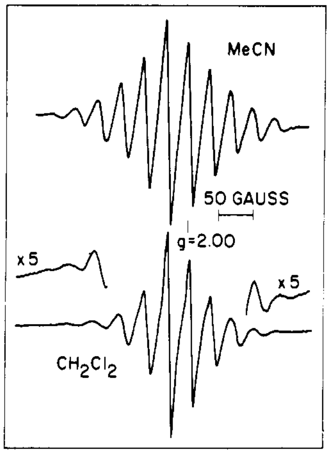

Figure 3. Fluid-solution EPR spectra of **2** in CH_2Cl_2 and MeCN at −40 °C (microwave power 5 mW with 9-G modulation amplitude (100-kHz modulation frequency); microwave frequency 9.15 GHz).

To our knowledge, **2** is the first well-characterized Mn(I)–Mn(II) mixed-valent species reported, and, as such, a comparison of its spectral properties with those of the known[17] Mn(III)–Mn(IV) dinuclear species $[Mn_2O_2(bpy)_4]^{3+}$ and $[Mn_2O_2(phen)_4]^{3+}$ is of interest. The work of Cooper and co-workers[18] has conclusively demonstrated that these latter complexes are examples of the class II mixed-valent designation of Robin and Day,[19] whose classifications were later refined by Wong and Schatz.[20] This class II assignment was based on the observation of both complex EPR spectra with at least 16 lines (indicating that the two metals possess discrete oxidation states) and electronic spectra containing a low-energy band at 830 nm assigned to a Mn(III)–Mn(IV) transition. Complex **2**, by contrast, seemingly fits best in class IIIA of the Robin and Day scheme,[19] where complete delocalization of the unpaired spin is realized but a low-energy transition in the visible/near-IR spectral region is still predicted. The 11-line EPR signal of **2** is consistent with the first criterion, while the second is met by the 880-nm band shown in Figure 2. The hyperfine splitting parameter of 33 G for **2** is much lower than either of the values for the Mn(III)–Mn(IV) species ($A_1 = 167$ G; $A_2 = 79$ G)[16] or the typical value of ca. 80 G, which we measure for Mn(II) in DMF. The basis for this dramatic difference is not

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(10) Cyclic voltammetry experiments were carried out with a CV-1A potentiostat from Bioanalytical Systems (BAS) and a three-electrode cell using 0.1 M $[Bu_4N]BF_4$ as the supporting electrolyte. The working electrode was a glassy-carbon disk frm BAS, and the reference electrode was an aqueous SCE separated from the sample solution by a salt bridge.

(11) The value of $i_{pa}/v^{1/2}AC$ for the one-electron oxidation of $[Mn_2(CO)_6(SPh)_3]^-$ at 52 °C was 591 A cm $S^{1/_2}$ $V^{-1/_2}$ mol^{-1} compared to values of 545 for the one-electron oxidation of the known[12] $Mo[S_2C_2(CN)_2]_4{}^{3-}$ and 1315 for the two-electron oxidation[1] of $[Mo_2(CO)_8(SPh)_2]^{2-}$.

(12) Smith, D. A.; McDonald, J. W.; Finklea, H. O.; Ott, V. R.; Schultz, F. A. *Inorg. Chem.* **1982**, *21*, 3825.

(13) It is also noted that no solvolysis of the oxidized product was indicated for the manganese system, as evidenced by the lack of additional reduction waves at more negative potential, a characteristic feature[1] of the $[Mo_2(CO)_8(SPh)_2]^{2-/0}$ couple.

(14) (a) Collman, J. P.; Rothrock, R. K.; Finke, R. G.; Moore, E. J.; Rose-Munch, F. *Inorg. Chem.* **1982**, *21*, 146. (b) Ginsberg, R. E.; Rothrock, R. K.; Finke, R. G.; Collman, J. P.; Dahl, L. F. *J. Am. Chem. Soc.* **1979**, *101*, 6550.

(15) Because **3** is expected to be EPR silent, no attempts were made to generate this species in situ.

(16) The stability of **2** is greatest in CH_2Cl_2 where red solutions are stable indefinitely at −40 °C. Even at low temperature, reaction mixtures in MeCN change to yellow within 15–20 min, and in DMF the red color due to **2** fades very rapidly. The greater stability of **2** in the noncoordinating CH_2Cl_2 suggests that the decomposition of this complex involves solvolysis.

(17) (a) Nyholm, R. S.; Turco, A. *Chem. Ind.* (*London*) **1960**, 74. (b) Plaskin, P. M.; Stouter, R. C.; Mathew, M.; Palenick, G. J. *J. Am. Chem. Soc.* **1972**, *94*, 2121. (c) Morrison, M. M.; Sawyer, D. T. *J. Am. Chem. Soc.* **1977**, *99*, 257.

(18) (a) Cooper, S. R.; Calvin, M. *J. Am. Chem. Soc.* **1977**, *99*, 6623. (b) Cooper, S. R.; Dismukes, G. C.; Klein, M. P.; Calvin, M. *J. Am. Chem. Soc.* **1978**, *100*, 7248.

(19) Robin, M. B.; Day, P. *Adv. Inorg. Chem. Radiochem.* **1967**, *10*, 247.

(20) Wong, K. Y.; Schatz, P. N. *Prog. Inorg. Chem.* **1981**, *28*, 369.

presently understood, but efforts are in progress to produce additional EPR-active low-valent Mn complexes to test its generality.

The Mn(III)–Mn(IV) dimers served as useful prototypes for studies[21] of the EPR spectrum exhibited by the Mn in photosystem II of photosynthesis (PS II), where it is now postulated that either a Mn(III)–Mn(III) or Mn(II)–Mn(III) dinuclear species is present.[21] Although **2** is not a realistic model for the Mn site in PS II, a study of its spectral properties increases the limited body of knowledge available on mixed-valent Mn complexes and in this sense may aid in the general understanding of the biological system.

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Registry No. 1, 96212-29-2; **2**, 96212-30-5; **3**, 96212-31-6; $Mn_2(CO)_{10}$, 10170-69-1.

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(21) (a) Abramowicz, D. A.; Dismukes, G. C. *Biochim. Biophys. Acta* **1984**, *765*, 318. (b) Dismukes, G. C.; Siderer, Y. *Proc. Natl. Acad. Sci. U.S.A.* **1981**, *78*, 274.

(22) Contribution No. 861.

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A Test of Superconductivity vs. Molecular Disorder in (BEDT-TTF)$_2$X Synthetic Metals: Synthesis, Structure (298, 120 K), and Microwave/ESR Conductivity of (BEDT-TTF)$_2$I$_2$Br

Sir:

For the first time two *ambient*-pressure synthetic metal superconductors have been reported in a single organic radical-cation donor system, with two different *symmetric* trihalide anions, and based on BEDT-TTF,[1] abbreviated "ET". They are β-(ET)$_2$I$_3$, with a reported superconducting transition temperature[2-6] (T_c) of 1.2–1.6 K, and β-(ET)$_2$IBr$_2$, with a T_c (2.3–2.8 K) twice[7,8] that of the I_3^- derivative. These two systems are isostructural with a characteristic structural motif of an extended "corrugated sheet" network of short *inter*stack $S\cdots S$ ($d_{S\cdots S} < 3.60$ Å) interaction distances.[9-11] The $S\cdots S$ orbital overlap within the two-dimensional *inter*stack network comprises the main electron conduction pathway in these synthetic metals, thereby constituting the conduction band.[12] Only β-(ET)$_2$I$_3$ possesses a "modulated"[13-15] structure, and a disordered $-CH_2$ group, with the former property being observed for the first time in any organic superconductor. However, these structural novelties do *not* appreciably affect the overall pattern of $S\cdots S$ orbital overlaps or the occurrence of superconductivity. In β-(ET)$_2$IBr$_2$, which is completely ordered at 125 K and contains no structural modulations, the IBr_2^- anion is ~8% shorter than the I_3^- anion and as a result the network $S\cdots S$ distances are *systematically* shortened due to cation–anion $[-CH_2\cdots(X_3)^-]$ interactions.[7] These findings suggest that one might prepare a new anion derivative, with a length *intermediate* between that of I_3^- and IBr_2^-, such as the *asymmetric* (I–I–Br)$^-$ anion,[16] which might also have $S\cdots S$ distances and T_c (expectedly at ambient pressure) intermediate between those of the two parent superconducting salts. More importantly, the (I$_2$Br)$^-$ anion provides a unique test of the effect of the dipolar anion and molecular order/disorder on the superconducting ground state in these unusual systems. While the (I–I–I)$^-$ and (Br–I–Br)$^-$ derivatives of ET are centrosymmetric and crystallographically ordered, the isostructural (ET)$_2$(I–I–Br) derivative *requires* a disordered anion configuration in the crystal due to its site symmetry ($\bar{1}$). Thus an "average" (I$_2$Br)$^-$ anion configuration results from a superposition of two allowed site-packing configurations, viz. (I–I–Br)$^-$ and (Br–I–I)$^-$. Structural disorder of this type generates a *random* electrostatic potential throughout the lattice, which is "sensed" by the electrons in the conducting radical-cation $S\cdots S$ network, or the corresponding $Se\cdots Se$ network in (TMTSF)$_2$X

Table I. Selected Intermolecular Interstack $S\cdots S$ Distances (all $D \le 3.6$ Å) for β-(BEDT-TTF)$_2$X (X = I_3^-, I_2Br^-, IBr_2^-) at 298 and 120 K

interstack contact		dist		
		β-(ET)$_2$I$_3$[a]	β-(ET)$_2$I$_2$Br	β-(ET)$_2$IBr$_2$
$S(5)\cdots S(2)$	298 K	3.574 (2)[b]	3.558 (3)	3.559 (2)
	120 K	3.555 ± 0.033	3.522 (1)	3.514 (2)
	diff[c]	0.019	0.036	0.045
$S(7)\cdots S(8)$	298 K	3.598 (2)	3.582 (3)	3.560 (2)
	120 K	3.534 ± 0.022	3.548 (1)	3.510 (2)
	diff	0.064	0.035	0.050
$S(5)\cdots S(6)$	298 K	3.600 (2)	3.582 (3)	3.593 (2)
	120 K	3.551 ± 0.048	3.578 (1)	3.551 (2)
	diff	0.049	0.034	0.042
$S(5)\cdots S(7)$	298 K	3.628 (2)	3.611 (3)	3.572 (2)
	120 K	3.548 ± 0.033	3.530 (1)	3.498 (2)
	diff	0.080	0.052	0.074
$S(6)\cdots S(8)$	298 K	3.593 (2)	3.578 (3)	3.564 (2)
	120 K	3.552 ± 0.094	3.515 (2)	3.504 (2)
	diff	0.041	0.062	0.060

[a] Each distance in the modulated structure[13] of β-(ET)$_2$I$_3$ at 120 K is an *average* value accompanied by the range allowed by the modulation and cannot be *directly* compared[27] to analogous distances in β-(ET)$_2$I$_2$Br or β-(ET)$_2$IBr$_2$, which are not modulated. [b] Estimated standard deviations are enclosed in parentheses and are ±0.003–0.005 for the average distances at 120 K for β-(ET)$_2$I$_3$. [c] The differences cited are those between 298 and 120 K values.

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(1) BEDT-TTF or "ET" is bis(ethylenedithio)tetrathiafulvalene, $C_{10}S_8H_8$.

(2) Yagubskii, E. B.; Shchegolev, I. F.; Laukhin, V. N.; Kononovich, P. A.; Karatsovnik, M. W.; Zvarykina, A. V.; Buravov, L. I., *Pis'ma, Zh. Eksp. Teor. Fiz.* **1984**, *39*, 12; *JETP Lett.* (*Engl. Transl.*) **1984**, *39*, 12.

(3) Williams, J. M.; Emge, T. J.; Wang, H. H.; Beno, M. A.; Copps, P. T.; Hall, L. N.; Carlson, K. D.; Crabtree. G. W. *Inorg. Chem.* **1984**, *23*, 2558.

(4) Crabtree, G. W.; Carlson, K. D.; Hall, L. N.; Copps, P. T.; Wang, H. H.; Emge, T. J.; Beno, M. A.; Williams, J. M. *Phys. Rev. B: Condens. Matter* **1984**, *30*, 2958.

(5) Carlson, K. D.; Crabtree, G. W.; Hall, L. N.; Copps, P. T.; Wang, H. H.; Emge, T. J.; Beno, M. A.; Williams, J. M. *Mol. Cryst. Liq. Cryst.* **1985**, *119*, 357.

(6) Azevedo, L. J.; Venturini, E. L.; Schirber, J. E.; Williams, J. M.; Wang, H. H.; Emge, T. J. *Mol. Cryst. Liq. Cryst.* **1985**, *119*, 389.

(7) Williams, J. M.; Wang, H. H.; Beno, M. A.; Emge, T. J.; Sowa, L. M.; Copps, P. T.; Behroozi, F.; Hall, L. N.; Carlson, K. D.; Crabtree, G. W. *Inorg. Chem.* **1984**, *23*, 3839.

(8) Carlson, K. D.; Crabtree, G. W.; Hall, L. N.; Behroozi, F.; Copps, P. T.; Sowa, L. M.; Nunez, L.; Firestone, M. A.; Wang, H. H.; Beno, M. A.; Emge, T. J.; Williams, J. M. *Mol. Cryst. Liq. Cryst.* **1985**, *125*, 159.

(9) In the (ET)$_2$X, X = (I$_3$)$^-$,[3,10,11] (I$_2$Br)$^-$, and IBr_2^-,[7,8] systems, the intermolecular *intra*stack $S\cdots S$ interaction distances are longer than the van der Waals radius sum of 3.6 Å while several *inter*stack distances are shorter. The degree to which these distances can be correlated with the degree of orbital overlap interaction is presently under investigation.[12]

(10) Kaminskii, V. F.; Prokhorova, T. G.; Shibaeva, R. P.; Yagubskii, E. B. *Pis'ma Zh. Eksp. Teor. Fiz.* **1984**, *39*, 15; *JETP Lett.* (*Engl. Transl.*) **1984**, *39*, 17.

(11) Mori, T.; Kobayashi, Y.; Sasaki, Y.; Kobayashi, H.; Saito, G.; Inokuchi, H. *Chem. Lett.* **1984**, 957.

(12) Whangbo, M.-H.; Williams, J. M.; Leung, P. C. W.; Beno, M. A.; Emge, T. J.; Wang, H. H.; Carlson, K. D.; Crabtree, G. W., work in progress.

(13) Leung, P. C. W.; Emge, T. J.; Beno, M. A.; Wang, H. H.; Williams, J. M.; Petricek, V.; Coppens, P. *J. Am. Chem. Soc.* **1984**, *106*, 7644.

(14) Williams, J. M.; Leung, P. C. W.; Emge, T. J.; Wang, H. H.; Beno, M. A.; Petricek, V.; Coppens, P. *Mol. Cryst. Liq. Cryst.* **1985**, *119*, 347.

(15) Emge, T. J.; Leung, P. C. W.; Beno, M. A.; Schultz, A. J.; Wang, H. H.; Sowa, L. M.; Williams, J. M. *Phys. Rev. B: Condens. Matter* **1984**, *30*, 6780.

(16) Wells, A. F. "Structural Inorganic Chemistry", 5th ed.; Oxford University Press: New York, 1984; p 396.

0020-1669/85/1324-1736$01.50/0