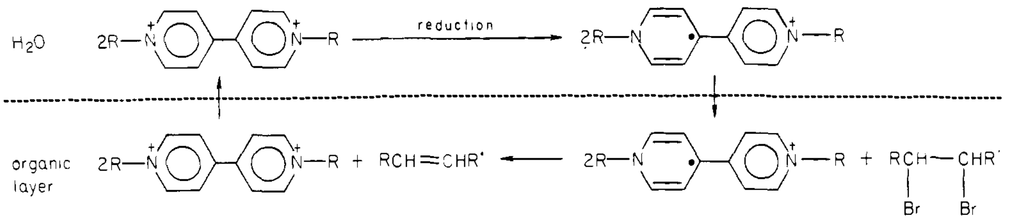
Table II. Two-Phase Dehalogenation of *vic*-Dihalide by Sodium Dithionite Using PrV^{2+} [a]

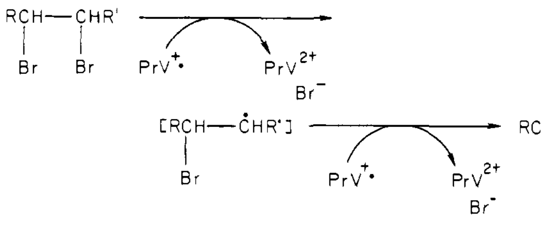
run	dihalide $[R_1CH(X)CH(X)R_2]$ config	time,[b] (min)	olefin	conv,[b] %	yield, %
1	X = Br, $R_1 = R_2$ = Ph erythro	40	*t*-PhC=CPh	100	97
2	X = Br, $R_1 = R_2$ = Ph threo	420	PhCH=CHPh[c]	100	
3	X = Br, R_1 = Ph, $R_2 = CO_2C_2H_5$ erythro	30	*t*-PhC=$CCO_2C_2H_5$	100	99
4	X = Br, R_1 = Ph, R_2 = H	150	PhC=C	100	84
5	X = Br, R_1 = Ph, $R_2 = CH_2OH$ erythro	30	*t*-PhC=CCH_2OH	100	94
6	X = Br, $R_1 = C_8H_{17}$, R_2 = H	720[d]	$C_8H_{17}C$=		24[e]
7	X = Br, $R_1 = CH_3$, $R_2 = CH_2OH$ erythro	330	*t*-CH_3C=CCH_2OH	100	97
8	X = Br, R_1, R_2 = $-(CH_2-)_6$	1440[d]	cyclooctene		47[e]
9	X = Br, R_1, R_2 = $-(CH_2-)_4$ threo	1620[d]	cyclohexene		42[e]
10	X = Cl, $R_1 = R_2$ = Ph erythro	1440[f]	*t*-PhC=CPh		34

[a] The reaction was performed with 1.4 mmol of $Na_2S_2O_4$, 1.5 mmol of K_2CO_3, 0.2 mmol of propylviologen, 1.0 mmol of dihalide in 20 mL of H_2O-CH_2Cl_2 (1:3) at room temperature. [b] Monitored by GLC every 10 or 30 min. [c] E/Z = 88/12. [d] Viologen was completely decomposed at the time indicated. [e] Product was not isolated, and the yield was determined by GLC [spontaneous dehalogenation of *vic*-dihalide did not occur at all in this condition (initial temp 80 °C; final temp 200 °C)] using 1-octanol as an internal standard. [f] Reaction was stopped at the time indicated.

Scheme I



Scheme II



methane (runs 3a-3c) dibromide was also recovered completely due to the insolubility of sodium dithionite. But *trans*-stilbene was obtained in the two-phase system using viologens (runs 4b-4f) and especially viologens having longer alkyl chains (runs 4d-4f) showed higher catalytic activity.[8] The debromination under the normal phase-transfer condition (run 5) did not proceed at the same condition as in the presence of viologens. It was demonstrated that longer alkyl chains containing viologens carried smoothly the electron to the dibromide through the heterophase, as indicated in Scheme I. Further, an electron transfer of viologen radical to the dibromide was confirmed by the examination of the effect of added dibromide on the reversibility of the cyclic voltammogram of PrV^{2+} (Figure 1).

The debromination of the various *vic*-dibromides was also tried under the same condition as run 4d in Table I. The results are summarized in Table II. Transisomers were obtained selectively from erythrodibromides, whereas trans and cis mixtures were given from *threo*-1,2-dibromo-1,2-diphenylethane (run 2). These results seems to show that the debromination may proceed by the two-step one-electron transfer not by E_2 mechanism. The dibromides that have phenyl (runs 1-5) or alkyl (run 7) groups were debrominated quantitatively. On the other hand, cyclic dibromides (runs 8 and 9) or 1,2-dibromoalkane (run 9) were not completely debrominated, and activity of viologen as the catalyst decreased gradually. Further, *vic*-dichloride (run 10) was also found to be dechlorinated. The debromination of bromodiphenylmethane was further carried out under the same condition to isolate tetraphenylethane in 50% yield. This reductive dimerization indicates that carbon radical as the intermediate is formed by the reduction of a carbon-bromine bond with $V^{+\bullet}$. The debromination described in this paper may proceed by the process in Scheme II.

Registry No. EV^{2+}, 46713-38-6; PrV^{2+}, 46903-41-7; HpV^{2+}, 47503-76-4; DdV^{2+}, 20462-61-7; *erythro*-PhCH(Br)CH(Br)Ph, 13440-24-9; *threo*-PhCH(Br)CH(Br)Ph, 70764-40-8; *erythro*-PhCH(Br)CH(Br)-$CO_2C_2H_5$, 30983-70-1; $PhCH(Br)CH_2Br$, 93-52-7; *erythro*-PhCH(Br)-$CH(Br)CH_2OH$, 83263-29-0; $C_8H_{17}CH(Br)CH_2Br$, 28467-71-2; *erythro*-$CH_3CH(Br)CH(Br)CH_2OH$, 54899-03-5; $CH_2(CH_2)_5CH(Br)CH$-(Br), 29974-69-4; *threo*-$CH_2(CH_2)_3CH(Br)CH(Br)$, 7429-37-0; *erythro*-PhCH(Cl)CH(Cl)Ph, 15951-99-2; *trans*-PhCH=CHPh, 103-30-0; *trans*-PhCH=CH($CO_2C_2H_5$, 4192-77-2; PhCH=CH_2, 100-42-5; *trans*-PhCH=$CHCH_2OH$, 872-05-9; *trans*-CH_3CH=$CHCH_2OH$, 504-61-0; cyclooctene, 931-88-4; cyclohexene, 110-83-8.

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(8) $C_3V^{+\bullet}$-$C_9V^{+\bullet}$ are soluble in water and extracted into CH_2Cl_2 from water.

Nucleophilic Additions to a Metal Carbonyl Cation: Synthesis of Metallocarboxylate and Formyl Complexes of Molybdenum

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Reactions of nucleophiles with metal carbonyl complexes have been the subject of intense scrutiny in recent years.[2] Special interest has centered on hydroxide[3] and hydride[4] additions since these produce intermediates thought to be generated in catalytic processes relating to carbon monoxide conversion.[5]

However, efforts to synthesize metallocarboxylic acids by OH^- addition to metal carbonyl complexes have resulted in few instances of isolated and fully characterized products.[6] Previous methods

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(1) University Predoctoral Fellow.

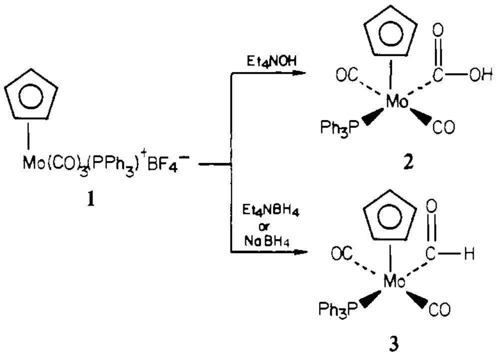
(2) (a) See, for example: Davies, S. G.; Green, M. L. H.; Mingos, D. M. P. *Tetrahedron* **1978** *34*, 3047. (b) Clack, D. W.; Monshi, M.; Kane-Maguire, L. A. P. *J. Organomet. Chem.* **1976**, *107*, C40. (c) Brown, D. A.; Chester, J. P.; Fitzpatrick, N. H. *Ibid.* **1978**, *158*, C21. (d) Birch, A. J.; Stephenson, G. R. *Ibid.* **1981**, *218*, 91. (e) Brown, D. A.; Chester, J. P.; Fitzpatrick, N. J. *Inorg. Chem.* **1982**, *21*, 2723. (f) Brown, D. A.; Chawla, S. K.; Glass, W. K.; Hussein, F. M., *Ibid.* **1982**, *21*, 2726. (g) Block, T. F.; Fenske, R. F.; Casey, C. P. *J. Am. Chem. Soc.* **1980**, *98*, 441.

(3) See: Ford, P. C. *Acc. Chem. Res.* **1981**, *14*, 31 and references cited therein.

(4) Gladysz, J. A. *Adv. Organomet. Chem.* **1982**, *20*, 1.

(5) See, for example: (a) Masters, C. *Adv. Organomet. Chem.* **1979**, *17*, 61. (b) Rofer-DePoorter, C. K. *Chem. Rev.* **1981**, *81*, 447. (c) Blackborow, J. R.; Daroda, R. J.; Wilkinson, G. *Coord. Chem. Rev.* **1982**, *43*, 17.

to prepare the neutral acids or formyl complexes have generally involved direct addition of the nucleophile to solutions of a metal carbonyl cation. We have used a somewhat different procedure to do the conversions indicated below:



In each case indicated above, cation **1** was slurried with methanol (in which it is sparingly soluble), and a methanol solution of the nucleophile was added to this slurry. Within a few minutes reaction was complete and the product, insoluble in CH_3OH, could be collected by vacuum filtration. The metallocarboxylic acid **2**, formed in 96% yield, is a yellow powder:[8] mp 145–153 °C dec; IR (Nujol) ν(CO) 1955 (m), 1862 (s), and 1616 (m) cm^{-1}; ^{1}H NMR (CD_2Cl_2) δ 5.08 (d, J_{PH} = 1.3 Hz) and 7.42 (m), relative areas 5:16, respectively; ^{13}C NMR (CD_2Cl_2, −7 °C) δ 95.47 (s), 128.89 (s), 129.03 (s), 130.91 (s), 133.23 (br, s), 209.52 (d, J_{PC} = 11.2 Hz), 238.49 (d, J_{PC} = 25.8 Hz), relative intensities of these two doublets are 1:2, respectively. The equivalence of the terminal CO ligands in the carbon spectrum data establishes a trans relationship between PPh_3 and COOH unambiguously,[9] and the IR and ^{1}H NMR spectra are also in accord with spectral characteristics reported for other trans complexes of the general formula $CpMo(CO)_2(PPh_3)X$ (Cp = η^5-C_5H_5).[10] The carbonyl resonances of **2** are similar to those of other metallocarboxylic acids,[6d,11] but the aromatic carbons do not show a typical pattern for triphenylphosphine ligands in metal complexes.[12] Several attempts were made to generate acid **2** in homogeneous media (e.g., acetone/water) by addition of aqueous NaOH or Et_4NOH. All of these efforts resulted in formation of an intractable purple solid of unknown structure.

As with some other metallocarboxylic acids,[6] treatment of **2** with CF_3COOH converts it back to the cation, $CpMo(CO)_3(PPh_3)^+$. Metallocarboxylic acids are not uniform in their behavior toward bases, however. Some have been reported to decarboxylate readily in the presence of bases such as Et_3N[6b,g,i] while others are stable in basic media;[6c,d] differences in behavior toward aqueous base as compared with Et_3N in acetone have also been noted.[6g] Furthermore, one compound apparently does not decarboxylate readily upon either heating or treatment with Et_3N.[6f] Compound **2** does not decarboxylate upon treatment with Et_3N in acetone at room temperature and shows no evidence of rapid decarboxylation in aqueous acetone with added NaOH. Also, there is no spectral evidence for ionization to the cation in polar solvents such as DMF as was reported for the related iron complex $CpFe(CO)(PPh_3)COOH$.[6c] However, as noted above, the hydroxyl group in **2** is labile in the presence of a strong acid. Furthermore, treatment of **2** with Et_4NBH_4 reduces it to formyl complex **3** in 70% yield; borohydrides do not reduce organic carboxylic acids.

Decarboxylation of acid **2** occurs smoothly upon prolonged (4 h) stirring in a methanol slurry at room temperature, providing the known hydride **4**, $CpMo(CO)_2(PPH_3)H$[14] (92% yield), which is also sparingly soluble in methanol. The relative intensities of the IR ν(CO) bands (in Nujol) indicate that this product may be primarily trans also. However, the compound is fluxional[15] in solution thus making the assignment of trans stereochemistry tenuous.

There are two previous reports of hydride reductions of cation **1** (as the PF_6 salt). The first report,[16] a number of years ago, described the conversion of **1** to the methyl complex $CpMo(CO)_2(PPh_3)CH_3$ after prolonged treatment with excess $NaBH_4$. The second report described the low-temperature conversion of **1** to formyl complex **3** by reduction with $LiEt_3BH$.[17] Under these conditions the formyl complex was reported to be unstable. We have prepared **3** by addition of Et_4NBH_4 or $NaBH_4$ to a methanol slurry of cation **1**. The product precipitated from solution within a few minutes, as it was formed, as a yellow powder, which could be collected by vacuum filtration under a blanket of N_2; it is not highly air sensitive. Compound **3**,[18] obtained in this way in 89% yield, has the following properties: mp 155–160 °C dec; IR (Nujol) ν(CO) 1945 (m), 1854 (s), and 1596 (m) cm^{-1}. NMR and solution IR spectra of **3** could be obtained at ambient temperature only by adding either PPh_3 or Et_4NBH_4 to the solvent prior to the addition of **3**; the formyl complex is not reduced further by short exposure to Et_4NBH_4, nor is the stereochemistry changed

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(6) (a) Deeming, A. J.; Shaw, B. L. *J. Chem. Soc. A* **1969**, 443. (b) Casey, C. P.; Andrews, M. H.; Rinz, J. E. *J. Am. Chem. Soc.* **1979**, *103*, 741. (c) Grice, N.; Kao, S. C.; Pettit, R. *Ibid.* **1979**, *103*, 1627. (d) Catellini, M.; Halpern, J. *Inorg. Chem.* **1980**, *19*, 566. (3) Gibson, D. H.; Hsu, W.-L.; Ahmed, F. U. *J. Organomet. Chem.* **1981**, *215*, 379. (f) Tam, W.; Lin, G.-Y.; Wong, W. K.; Kiel, W. A.; Wong, V. K.; Gladysz, J. A. *J. Am. Chem. Soc.* **1982**, *104*, 141. (g) Sweet, J. R.; Graham, W. A. G. *Organometallics* **1982**, *1*, 982. (h) Alton, J. G.; Kane-Maguire, L. A. P. *J. Organomet. Chem.* **1983**, *246*, C23. (i) Barrientos-Penna, C. F.; Gilchrist, A. B.; Sutton, D. *Organometallics* **1983**, *2*, 1265.

(7) Cation **1** was prepared by treating $CpMo(CO)_2(PPh_3)Cl$ with $AgBF_4$ while bubbling CO through a solution of the chloride in CH_2Cl_2. Recrystallization of the crude product in CH_2Cl_2/hexane afforded yellow needles: IR (Nujol) 2055 (m), 1990 (m), 1960 (s) cm^{-1}. The spectrum compares favorably with that of the PF_6 salt [(Nujol) 2025 (s), 1995 (m), 1955 (s) cm^{-1}] reported previously: Treichel, P. M.; Barnett, K. W.; Shubkin, R. L. *J. Organomet. Chem.* **1967**, *7*, 449.

(8) Anal. Calcd for $C_{26}H_{21}O_4PMo$: C, 59.55; H, 4.04; P, 5.91. Found: C, 59.76; H, 4.29; P, 6.09.

(9) The carbon spectra of the cis halides in the series $CpMo(CO)_2(PPh_3)X$ show two carbonyl resonances, the lower field signal having J_{PC} of approximately 28 Hz and the other having J_{PC} of ~5 Hz. The trans complex $CpMo(CO)(PPh_3)_2Cl$ shows a doublet for the carbonyl with J = 27.2 Hz while *trans*-$CpMo(CO)_2(PPh_3)I$ shows a single resonance for the two equivalent carbonyls with J_{PC} = 27.4 Hz. From these comparisons, carbonyl groups trans to triphenylphosphine in related complexes are expected to have significantly smaller J_{PC} values than the carbonyl groups that are cis to the phosphine ligand (see: Todd, L. J.; Wilkinson, J. R.; Hickey, D. L.; Barnett, K. W. *J. Organomet. Chem.* **1978**, *154*, 151). This study also included *trans*-$CpMo(CO)_2(PPh_3)COCH_3$, which showed a lower carbon chemical shift for the acetyl carbonyl than the terminal carbonyls but about the same J_{PC} value (11.7 Hz) for the acetyl carbonyl as we observe for the carboxyl carbon in **2**. The acetyl carbon appeared at much lower field than the carboxyl carbon in **2**; the same trend is observed for ketones relative to carboxylic acids (see: Levy, G. C.; Lichter, R. L.; Nelson, G. L. "Carbon-13 Nuclear Magnetic Resonance Spectroscopy", 2nd ed.; Wiley-Interscience: New York, 1980; Chapter 5).

(10) Compounds having this general structure, with X = hydrogen or halogen, show the cyclopentadienyl peak as a doublet (J = 1–2 Hz) in the trans isomer but show a singlet for the cis isomer; see: (a) Faller, J. W.; Anderson, A. S. *J. Am. Chem. Soc.* **1970**, *92*, 5852. (b) Beach, D. L.; Barnett, K. W. *J. Organomet. Chem.* **1975**, *97*, C27. (c) Alway, D. G.; Barnett, K. W. *Inorg. Chem.* **1980**, *19*, 1533. The IR spectra of halides in this series show two ν(CO) bands with the higher frequency band more intense in the cis isomer (see ref 10b,c).

(11) The J_{PC} values in metallocarboxylic acids with phosphine ligands appear to be quite sensitive to the nature of the metal atom. For example, $CpFe(CO)(PPh_3)COOH$ (first reported by Pettit[6c]) shows carbonyl resonances of equal intensity at δ 220.0 (J_{PC} = 30.4 Hz) and 214.3 (J_{PC} = 34.6 Hz) while the related chloride shows its single resonance at 220.32 (J_{PC} = 32.7 Hz). On the other hand, $CpRu(CO)(PPh_3)COOH$ shows carbonyl resonances of equal intensity at 204.36 (J_{PC} = 18.3 Hz) and 199.0 (J_{PC} = 19.2 Hz). Furthermore, the carbon resonances for the triphenylphosphine ligands in the iron and ruthenium complexes show the typical pattern[12] of one relatively large J_{PC} value of 40–60 Hz, two smaller ones of about 10 Hz each, and one near 0 Hz (presumably $^1J_{PC}$, $^{2,3}J_{PC}$, and $^4J_{PC}$, respectively). Other characteristics of the iron and ruthenium complexes will be reported separately (Gibson, D. H.; Ong, T.-S., unpublished results).

(12) See, for a discussion of related systems: Jolly, P. W.; Mynott, R. *Adv. Organomet. Chem.* **1981**, *19*, 257.

(13) Darensbourg, D. J.; Froelich, J. A. *J. Am. Chem. Soc.* **1978**, *100*, 3300.

(14) Manning, A. R. *J. Chem. Soc. A* **1968**, 651.

(15) See ref 10a.

(16) Treichel, P. M.; Shubkin, R. L. *Inorg. Chem.* **1967**, *6*, 1328.

(17) (a) Tam, W.; Wong, W.-K.; Gladysz, J. A. *J. Am. Chem. Soc.* **1979**, *101*, 1589. (b) Tam, W.; Lin, G.-Y.; Gladysz, J. A. *Organometallics* **1982**, *1*, 525.

(18) Anal. Calcd for $C_{26}H_{21}O_3Mo$: C, 61.43; H, 4.16; P, 6.09. Found: C, 61.37; H, 4.18; P, 5.89.

as evidenced by the IR spectrum. The ^{1}H NMR spectrum (CD_2Cl_2 with added Et_4NBH_4) of **3** shows a doublet at δ 5.10 (J_{PH} = 1.0 Hz), a multiplet followed by a singlet centered at 7.31, and a doublet at 14.89 (J_{PH} = 4.0 Hz) with relative areas 5:15:1. The spectral data thus indicate that **3** is the trans isomer also. As reported previously,[16] reaction of **1** with excess $NaBH_4$ in THF yields $CpMo(CO)_2(PPh_3)CH_3$ (**5**). However, treatment of **3** with $NaBH_4$ does not lead directly to **5**; this reaction is under further study.

Although anionic formyl complexes are known to be potent hydride donors, the neutral complexes are apparently less active (as might be expected).[4] However, hydride transfer does occur from CpRe(NO)(CO)CHO to benzaldehyde,[19] although the reaction appears to require acid catalysis. Formyl complex **3** behaves as a hydride donor to $Ph_3C^+BF_4^-$ and is converted to **1** in 70% yield.

Formyl complex **3** is labile in solution at room temperature, converting cleanly to $CpMo(CO)_3H$ within a few minutes (as also suggested by the observations of Gladysz[17]). Added triphenylphosphine slows, but does not stop, this conversion; the inhibiting effect of Et_4NBH_4 is not yet understood. Since we have determined that **3** has trans stereochemistry, it appears that a formyl group must be regarded as a trans labilizing ligand in this series also.[20]

Work is in progress to define the chemistry of **2** and **3**, as well as related systems, further.

Acknowledgment. Partial support of this work by the National Science Foundation (CHE-79-26044), the University of Louisville Graduate Research Council, and the Arts and Sciences Research Committee is gratefully acknowledged.

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(20) The tris(3,5-dimethylphenyl) phosphite complex $Et_4N^+[(ArO)_3P](CO)_3FeCHO^-$ decomposed with exclusive loss of the phosphite ligand (Casey, C. P.; Newmann, S. M.; Andrews, M. A.; McAlister, D. R. *Pure Appl. Chem.* **1980**, *52*, 625). This complex was shown to have trigonal-bipyramidal geometry with the formyl group trans to the phosphite. The corresponding triphenyl phosphite complex decomposes less selectively, but with preferential loss of the phosphite ligand.

A Sulfur-Bridged Iron(III) Porphyrin–Copper(II) System: Relevance to Cytochrome *c* Oxidase

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The properties of the active site of the resting form of cytochrome *c* oxidase (CcO) must be accounted for in terms of a unit containing a copper(II) atom and an iron(III) heme.[1] Strong ligand-mediated antiferromagnetic coupling between these metal atoms has been proposed[2-6] to explain the magnetic properties of the CcO active site. Potential model compounds[7-12] involving imidazolate and oxide bridging ligands have not successfully reproduced these magnetic properties.

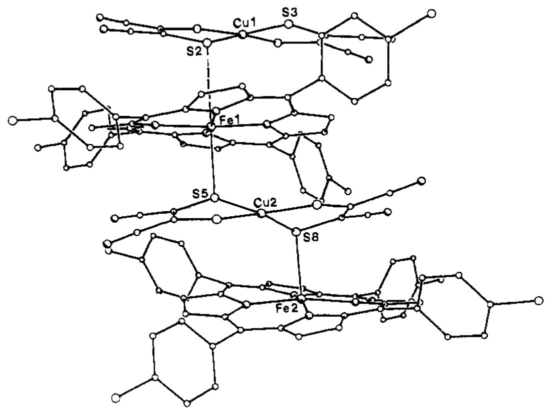

Figure 1. View of the metal-containing anionic unit of **2**. Atoms have been drawn as spheres of arbitrary radius.

EXAFS studies of CcO have suggested that the iron/copper active site pair may be bridged by thiolate sulfur.[13] We have described[14] a trinuclear complex (**1**) involving two $Fe(TPP)^+$ (TPP = *meso*-tetraphenylporphyrin) moieties coupled to $Cu(MNT)_2^{2-}$ (MNT^{2-} = *cis*-1,2-dicyano-1,2-ethylenedithiolate). Although single crystals were not obtained for **1**, EXAFS studies[15] favored sulfur atom bridges between iron(III) and copper(II). Appreciable antiferromagnetic coupling was not observed for **1**. Instead, the lack of a significant EPR signal at 77 K was interpreted as due to an extreme broadening of the copper(II) signal by rapidly relaxing $S = {}^3/_2$ iron.

A slight change in synthetic conditions[16] has permitted isolation of $(TBA)\{[Fe(p\text{-}Cl_4TPP)]_2[Cu(MNT)_2]_2\}\cdot 3C_6H_6$ (**2**) as a crystalline compound. Single-crystal X-ray diffraction[17] has established (Figure 1) that **2** contains a pair of $Fe(p\text{-}Cl_4TPP)^+$ units that sandwich a $Cu(MNT)_2^{2-}$ anion. This anion is bound strongly to both Fe1 and Fe2 through cis sulfur atoms S5 and S8 (Fe2–S8 = 2.444 (2), Fe1–S5 = 2.549 (2) Å). Six-coordination about Fe1 is completed by a weak bond to S2 (Fe1–S2 = 2.956 (2) Å) of an additional $Cu(MNT)_2^-$ anion, but Fe2 is five-coordinate

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(1) Malmström, B. G. In "Metal Ion Activation of Dioxygen"; Spiro, T. G., Ed.; Wiley: New York, 1980; Chapter 5.

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(15) Powers, L., personal communication, 1982.

(16) Under N_2 atmosphere, 80.6 mg (0.10 mmol) of $Fe(p\text{-}Cl_4TPP)$ ($p\text{-}Cl_4TPP$ = *meso*-tetra(*p*-chlorophenyl)porphyrin) and 67.4 mg (0.12 mmol) of $(TBA)[Cu(MNT)_2]$ (TBA^+ = tetra-*n*-butylammonium ion) were mixed in 50 mL of benzene and stirred for 6 h. Filtration removed the excess $(TBA)_2[Cu(MNT)_2]$ product. Vapor diffusion of *n*-heptane into the filtrate for 1 month (under N_2 atmosphere) yielded a mixture of crystals: (a) the major product, ostensibly the Fe–Cu–Fe trimer analogous to that previously reported; (b) some $(TBA)[Cu(MNT)_2]$ and/or $(TBA)_2[Cu(MNT)_2]$; (c) approximately 10% yield of the tetranuclear derivative **2** described in this Communication, as efflorescent dark brown crystals. Magnetic and Mössbauer data for **2** are not presently available for comparison with the properties of **1**, although such studies are planned. Solution spectra of **1** and **2** are qualitatively similar.

(17) For **2** ($C_{138}H_{102}N_{17}S_8Cu_2Fe_2Cl_8$), a = 13.378 (3) Å, b = 17.356 (2) Å, c = 28.918 (7) Å, α = 104.05 (2)°, β = 99.62 (2)°, γ = 98.38 (2)° at −130 °C. Space group $P\bar{1}$, Z = 2, μ(calcd) = 9.2 cm^{-1}. A small (0.42 mm × 0.46 mm × 0.30 mm) crystal yielded 16078 unique observed ($I > 2\sigma(I)$) reflections on the R3m/E diffractometer. The refinement (individual anisotropic thermal parameters for most non-hydrogen atoms, idealized phenyl and benzene rings with isotropic thermal parameters, no H atoms) of 1042 parameters (using the R3m/E SHELXTL software) converged at R = 0.063, R_w = 0.069, GOF = 1.72.

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