The importance of the ion-pair formation is also supported by a different experiment in which the stereoselectivity is decreased by the use of a Tris–cacodylic acid buffer instead of phosphate buffer, as shown in Figure 3A. Because the Tris–cacodylate anion does not bind with cyt *c* but the phosphate anion does bind with cyt *c*,[11c,13] the greater stereoselectivity observed in the phosphate buffer would be attributed to the binding of the phosphate anion.

An interesting pH dependence of the stereoselectivity is observed; as shown in Figure 3, the stereoselectivity is very small at pH 7.0, disappears at pH = 6.0,[4] but increases considerably as the pH is lowered from 5.6 to 4.0.[4] A pH decrease causes protonation of the COO^- group of cyt *c*(II). This protonation weakens the hydrogen bond included in cyt *c*, which would induce the opening of the heme crevice and increase the stereoselectivity similarly to the increasing ethanol volume percent.[14] The protonation is also similar, to some extent, to the ion-pair formation. Thus, the protonated amino acid residues tend to approximate to $Co(acac)_3$ more easily than the deprotonated amino acid residues, which might be the other factor enhancing the stereoselectivity.

In conclusion, the first stereoselective electron-transfer reaction between cyt *c*(II) and $Co(acac)_3$ is observed in this work.[15] An interesting dependence of stereoselectivity on such reaction conditions as ethanol volume percent of the solvent, ionic strength, and pH is found. The stereoselectivity is expected to offer new information different from the redox potential, because the redox potential would be primarily influenced by a structural change of a heme center itself but the stereoselectivity would be sensitive to the situation around the active site.

Acknowledgment. We gratefully acknowledge the Ministry of Education, Science and Culture for a Grant-in-Aid (No. 61540451) and the Department of Physics, Faculty of Science, Kumamoto University, for use of the Jasco J-500C spectropolarimeter.

Registry No. cyt *c*(II), 9007-43-6; $Co(acac)_3$, 21679-46-9.

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(12) Even when the reactant is neutral, the electron-transfer reaction is considered to pass through the polarized transition state and the product is charged in many cases. Thus, the driving force of the electron-transfer reaction would be influenced by ionic strength, which would have some influence on the stereoselectivity. The relation between the stereoselectivity and the driving force of the reaction is important and an interesting issue to be examined. At the moment, we have no informative result about it, and a detailed discussion is omitted here.

(13) (a) Margalit, R.; Schejter, A. *Eur. J. Biochem.* **1973**, *32*, 492. (b) Barlow, G. H.; Hargoliash, E. *J. Biol. Chem.* **1966**, *241*, 1473.

(14) The reason that the stereoselectivity disappears at pH 6.0 is ambiguous and must be investigated in more detail.

(15) Stereoselectivity is also found in the electron-transfer reaction between cyt *c*(II) and $[Co(bpy)_3]^{3+}$. This stereoselectivity is larger than that found in this work (Sakaki, S. To be submitted for publication).

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Redox-Active Phenylene-Bridged Polymetallic σ,π Complexes Containing Iron and Chromium[1]

Research in our laboratories is centered on the synthesis and characterization of new phenylene-bridged polymetallic complexes. It is our hope that such complexes will serve as models for the production of linear-chain organometallic polymers which will serve as one-dimensional electronic conductors. Our model complexes have typically contained η^1-organometallic fragments such as Fp (Fp = $(\eta^5\text{-}C_5H_5)Fe(CO)_2$) σ-bound to an arene ring, and many have had an η^6-arene coordinated in a π fashion to an "$M(CO)_3$" group (M = Cr, Mo, W), e.g.

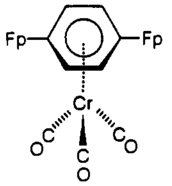

Table I. Electrochemical Data for the Oxidations of the Metalated $(arene)Cr(CO)_3$ Complexes[a]

arene[b]	$E°'$, V[c]	arene	$E°'$, V
1,4-C_6H_4(Cl)Fp (**1**)	+0.73	1,3-$C_6H_4Fp_2$ (**5**)	+0.45
C_6H_5Fp (**2**)	+0.64	1,4-$C_6H_4Fp_2$ (**6**)	+0.43
1,4-C_6H_4(Me)Fp (**3**)	+0.60	1,3,5-$C_6H_3Fp_3$ (**7**)	+0.28
1,4-C_6H_4(OMe)Fp (**4**)	+0.56		

[a] Complex concentrations of $(5-7) \times 10^{-4}$ M in dichloromethane/0.1 M $n\text{-}Bu_4NPF_6$; scan rate of 0.10 V s^{-1}; potentials vs SCE. [b] Fp = $(\eta^5\text{-}C_5H_5)Fe(CO)_2$. [c] Defined as the average of cathodic and anodic peak potentials.

We recently reported the synthesis and spectroscopic characterization of a representative set of these novel tri- and tetrametallic complexes and confirmed their structures by subjecting one of these compounds to a single-crystal X-ray crystallographic study.[1,2] We now wish to communicate the results of our preliminary electrochemical investigations on an extended series of the Fe/Cr complexes.

Cyclic voltammetry at a stationary platinum-bead electrode reveals that the complexes **1**–**7**[2,3] (Table I) exhibit simple one-electron reversible oxidations in dichloromethane solution (containing 0.1 M $n\text{-}Bu_4NPF_6$, vs SCE).[6,7] These oxidation processes are diffusion-controlled and exhibit cathodic–anodic peak separations of 60–70 mV (at a scan rate of 0.1 V s^{-1}). A comparison of peak separations and wave heights with those of ferrocene[8] established the one-electron nature of these oxidations, i.e.

$$(\text{arene})\text{Cr(CO)}_3 \underset{+e^-}{\overset{-e^-}{\rightleftharpoons}} [(\text{arene})\text{Cr(CO)}_3]^{\bullet +} \qquad (1)$$

(as did controlled-potential electrolysis of **3**). These oxidations are clean and produce remarkably stable radical cations. Thus, when a representative example, $(\eta^6\text{-}1,4\text{-}C_6H_4MeFp)Cr(CO)_3$, is treated with 1 equiv of $AgPF_6$ in dichloromethane, a thermally stable but air-sensitive blue paramagnetic complex, $[(\eta^6\text{-}1,4\text{-}C_6H_4MeFp)Cr(CO)_3]^{\bullet +}PF_6^-$, is produced; i.e.[9]

$$(\eta^6\text{-}1,4\text{-}C_6H_4(\text{Me})\text{Fp})\text{Cr(CO)}_3 + \text{AgPF}_6 \xrightarrow{CH_2Cl_2} [(\eta^6\text{-}1,4\text{-}C_6H_4(\text{Me})\text{Fp})\text{Cr(CO)}_3]\text{PF}_6 + \text{Ag} \qquad (2)$$

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(1) Organometallic Complexes with Electronic Bridges. 4. Part 3: Hunter, A. D.; McLernon, J. L. *Organometallics*, in press.

(2) Hunter, A. D. *Organometallics* **1989**, *8*, 1118.

(3) The $(\eta^6\text{-}1,4\text{-}C_6H_4(X)Fp)Cr(CO)_3$ complexes (X = H, Me, Cl, OMe) were synthesized from the corresponding fluoro- or chloroarene complexes by the nucleophilic displacement of fluoride or chloride by Fp^-.[4,5]

(4) Richter-Addo, G. B.; Hunter, A. D.; Wichrowska, N. Manuscript in preparation.

(5) Heppert, J. A.; Morgenstern, M. A.; Scherubel, D. M.; Takusagawa, F.; Shaker, M. R. *Organometallics* **1988**, *7*, 1715.

(6) The electrochemical cell used for the cyclic voltammetry experiments was similar to that previously described (Legzdins, P.; Wassink, B. *Organometallics* **1984**, *3*, 1811). Under the experimental conditions employed, ferrocene is reversibly oxidized in dichloromethane at +0.47 V vs SCE: cathodic–anodic peak potential separation, 60 mV; $i_{p,c}/i_{p,a}$ = 1.0; scan rate, 0.1 V s^{-1}.

(7) The complexes exhibit other oxidation waves at higher potentials, but these lead to extensive coating of the electrode surface, making analyses of these waves difficult. In addition, no reduction waves are observed in the cyclic voltammograms of these complexes up to −1.8 V vs SCE.

(8) Gagne, R. R.; Koval, C. A.; Lisensky, G. C. *Inorg. Chem.* **1980**, *19*, 2854.

(9) (a) IR (Nujol mull): ν_{CO} 2056 (s), 2034 (m), 1970 (s, br), 1930 (m, br) cm^{-1}. Compare with those of **3**: 2014 (s), 1966 (s), 1932 (s), 1862 (sh), 1850 (s), 1840 (sh), 1818 (sh) cm^{-1}. Anal. Calcd for $C_{17}H_{12}O_5PF_6CrFe$: C, 37.16; H, 2.19. Found: C, 36.84; H, 2.13. This complex exhibits an unresolved (broad) ESR signal at 298 K in dichloromethane. (b) A similar but less thermally stable complex, $[\mathbf{4}]^{\bullet +}BF_4^-$, can be prepared by the analogous reaction of **4** with $AgBF_4$ in dichloromethane.

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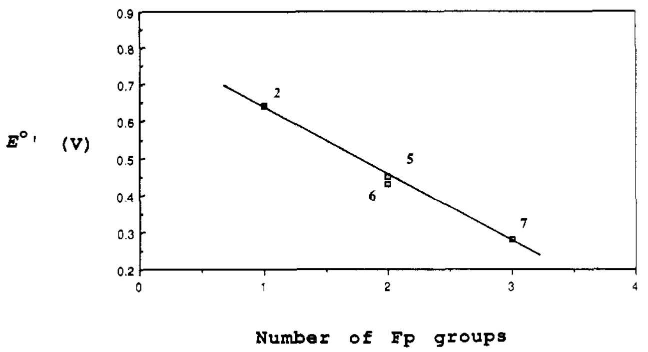

Figure 1. Plot of $E^{\circ\prime}$ vs the number of Fp groups in the $(\eta^6\text{-}C_6H_{6-n}Fp_n)Cr(CO)_3$ complexes **2**, **5**, **6**, and **7** (where *n* is 1, 2, 2, and 3, respectively).

High chemical reversibility is observed in the processes (1), as judged by the cathodic-to-anodic peak current ratios ($i_{p,c}/i_{p,a}$) of ≥0.96 in this solvent. The formal oxidation potentials ($E^{\circ\prime}$) for these complexes are in the +0.2–0.8-V range and are assigned to oxidations involving orbitals of the chromium center.[10] The magnitude of $E^{\circ\prime}$ for the bimetallic complexes **1–4** decreases as a function of the overall electron-donating ability of the modified arene ring, i.e. $C_6H_4(Cl)Fp > C_6H_5Fp > C_6H_4(Me)Fp > C_6H_4(OMe)Fp$. This trend is in accord with the observed carbonyl-stretching frequencies for the $Cr(CO)_3$ fragment in these complexes[4,5] and provides additional evidence for the electron being removed from a HOMO having significant chromium character. In addition, the observation that these complexes are more readily oxidized (by −0.22 to −0.24 V) than are their analogues which do not contain a Fp group (i.e. where arene = C_6H_5X and X = Cl, H, Me, OMe)[11] clearly indicates that substantial transfer of electron density from Fe to Cr is occurring in these complexes.

An approximate linear additivity of electron donation from the Fp groups in the complexes $(\eta^6\text{-}C_6H_{6-n}Fp_n)Cr(CO)_3$ (n = 1–3) is observed by cyclic voltammetry for the complexes **2**, **5**, and **7** (see Figure 1) involving only meta substitution, as is also seen by IR spectroscopy.[1,2] This feature reflects the cumulative donor effects of the added Fp groups, with each additional Fp group shifting the redox potential by about −0.2 V. Inspection of the cyclic voltammetry data (Table I) reveals that the 1,4-substituted arene in **6** is more electron donating than is the 1,3-substituted arene in compound **5** (by 0.02 V, which is comparable to a methylation of an organic arene bound to the $Cr(CO)_3$ group).[10d,11] Thus, the 1,4-substitution geometry for the π donors in these complexes seems to lead to enhanced electron transfer to the Cr center.[12,13]

Trends similar to those observed in dichloromethane for the oxidation potentials, $E^{\circ\prime}$, of the complexes **1–7** are observed in acetonitrile, a typical coordinating solvent. Since the oxidations of most (arene)$Cr(CO)_3$ complexes are irreversible in coordinating solvents, we were interested to find that the radical cations of **1–7** display remarkable persistence in acetonitrile. Indeed, the degree of this stabilization is markedly enhanced by increased metalation of the arene ring.[15] For example, the trisubstituted complex **7** has a chemically reversible oxidation ($i_{p,c}/i_{p,a}$ = 1.00) even at 0.10 V s^{-1}, while the disubstituted complexes **5** and **6** have somewhat lower $i_{p,c}/i_{p,a}$ values of 0.90 at this scan rate.[18] The monosubstituted complexes **3** and **4** show some degree of chemical reversibility, but the decomposition of their radical cations is greater in these cases. A cyclic voltammogram illustrating such a decomposition (that of complex **4**) is shown in Figure 2. When the anodic scan is reversed at +0.77 V, a coupled redox wave[19] is observed at −0.06 V, which is attributable to the formation of $[(CH_3CN)_3Cr(CO)_3]^{\bullet+}$. This assignment was verified by obtaining a cyclic voltammogram of authentic $(CH_3CN)_3Cr(CO)_3$[20] in acetonitrile. Under identical experimental conditions, the $(CH_3CN)_3Cr(CO)_3^{0/+}$ couple occurs at $E^{\circ\prime}$ = −0.06 V vs SCE (ΔE = 81 mV, $i_{p,c}/i_{p,a}$ = 0.80 at 0.1 V s^{-1}). This result is in perfect agreement with our proposal that the $(CH_3CN)_3Cr(CO)_3^{\bullet+}$ complex is indeed formed during the oxidative decomposition of our (arene)$Cr(CO)_3$ complexes.[21,22] Detailed studies of the redox couples over a variety of scan rates (0.05–2.0 V s^{-1}) established an E_rC_i electrode process for the oxidation of **4**, i.e.

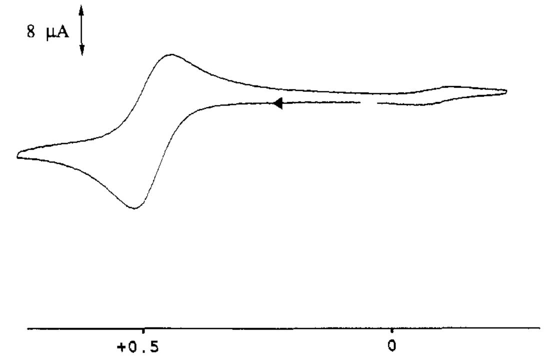

Figure 2. Cyclic voltammogram of $(\eta^6\text{-}C_6H_4(OMe)Fp)Cr(CO)_3$ in acetonitrile at a platinum-bead electrode (scan rate 1.0 V s^{-1}).

$$(\eta^6\text{-}1,4\text{-}C_6H_4(OMe)Fp)Cr(CO)_3 \underset{+e^-}{\overset{-e^-}{\rightleftharpoons}} [(\eta^6\text{-}1,4\text{-}C_6H_4(OMe)Fp)Cr(CO)_3]^{\bullet+} \quad (3)$$

$$[(\eta^6\text{-}1,4\text{-}C_6H_4(OMe)Fp)Cr(CO)_3]^{\bullet+} \xrightarrow{k_f} 1,4\text{-}C_6H_4(OMe)Fp + [(CH_3CN)_3Cr(CO)_3]^{+} \quad (4)$$

A rate constant for the pseudo-first-order follow-up chemical reaction (eq 3) of k_f' = 1.01 s^{-1} ($k_f' = k_f[CH_3CN]$) was obtained from an analysis of $i_{p,c}/i_{p,a}$ values for the electrochemical reaction described by eq 2 over the 0.1–1.00 V s^{-1} scan rate range. Complex **3** is also decomposed (k_f' = 1.02 s^{-1}) upon oxidation by this mechanism. In contrast, however, the oxidations of complexes **1** and **2** in this solvent do not show any chemical reversibility even at 1.0 V s^{-1}. Thus, the rates of the nucleophilic decomposition

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(10) Many other (arene)$Cr(CO)_2L$ complexes (L = CO, unsaturated hydrocarbon, phosphine) also undergo reversible oxidations in this potential range. For leading references, see: (a) Zoski, C. G.; Sweigart, D. A.; Stone, N. J.; Rieger, P. H.; Mocellin, E.; Mann, T. F.; Mann, D. R.; Gosser, D. K.; Doeff, M. M.; Bond, A. M. *J. Am. Chem. Soc.* **1988**, *110*, 2109. (b) Van Order, N., Jr.; Geiger, W. E.; Bitterwolf, T. E.; Rheingold, A. L. *J. Am. Chem. Soc.* **1987**, *109*, 5680. (c) Connelly, N. G.; Geiger, W. E. *Adv. Organomet. Chem.* **1984**, *23*, 64. (d) Lloyd, M. K.; McCleverty, J. A.; Connor, J. A.; Jones, E. M. *J. Chem. Soc., Dalton Trans.* **1973**, 1768 and references cited therein.

(11) In our hands, the $E^{\circ\prime}$ values for the related $(C_6H_5X)Cr(CO)_3$ complexes in dichloromethane are +0.97, +0.87, +0.84, and +0.78 V for X = Cl, H, Me, and OMe, respectively.

(12) The Fp group is a strong π-donor as well as a good σ-donor to an arene ring. See ref 1 and work cited therein.

(13) It is well-known that, in monosubstituted benzenes, the π-donor effect is of greater importance at the para position that at the meta position. In fact, this may result in quinoid character to the arene linkage in such species.[1,4,14]

(14) (a) Hehre, W. J.; Taft, R. W.; Topsom, R. D. *Prog. Phys. Org. Chem.* **1976**, *12*, 159. (b) Bromilow, J.; Brownlee, R. T. C.; Craik, D. J.; Sadek, M.; Taft, R. W. *J. Org. Chem.* **1980**, *45*, 2429 and references therein. (c) Hunter, A. D.; Szigety, A. *Organometallics*, in press.

(15) Metalation of the arene in some (arene)$Cr(CO)_3$ complexes by main-group substituents (e.g. SnR_3; R = alkyl, aryl)[16,17] results in moderate stabilization of their radical cations.

(16) Rieke, R. D.; Tucker, I.; Milligan, S. N.; Wright, D. R.; Willeford, B. R.; Radonovich, L. J.; Eyring, M. W. *Organometallics* **1982**, *1*, 938.

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(18) Our 1,4- and 1,3-substituted complexes show similar stabilities, in contrast to what might be expected; see for example: Ikeshoji, T.; Parker, V. D. *Bull. Chem. Soc. Jpn.* **1984**, *57*, 1112.

(19) The low intensity of the second redox couple is due to the fact that, in the return scan, no electrolysis of **4** is taking place, and the $[(CH_3CN)_3Cr(CO)_3]^+$ product complex diffuses away from the electrode surface.

(20) Tate, D. P.; Knipple, W. R.; Augl, J. M. *Inorg. Chem.* **1962**, *1*, 433.

(21) The $(CH_3CN)_3Cr(CO)_3^{\bullet+}$ complex has only recently been detected electrochemically in related decompositions of other (arene)- and (thiophene)chromium tricarbonyl complexes.[22]

(22) Rourke, F.; Crayston, J. A. *J. Chem. Soc., Chem. Commun.* **1988**, 1264.

reactions appear to be inversely proportional to the electron richness of the Cr centers. If all these observations are taken together, this trend in the rate of decomposition of the generated radical cations is consistent with that in the $E^{\circ\prime}$ and $\nu_{CO}(Cr(CO)_3)$ values of the parent complexes and is a reflection of the electron-transfer abilities of the arene and Fp groups.

It is evident from this work that polymetalation of the arene in the title complexes leads to remarkable stability of their respective radical cations, even in a coordinating solvent such as acetonitrile. Experiments to obtain the stable radical cations of all of the title complexes and to elucidate the origin of this stabilization are currently in progress.

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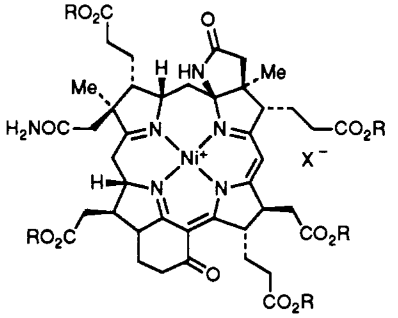
The First Structural Characterization of a Nickel(I) Macrocyclic System: Structure of Nickel(I) Diphenyldi-*p*-tolyl-21-thiaporphyrin

Factor F_{430} (**1**), the prosthetic group of methyl-coenzyme M reductase, contains nickel ion in a macrocycle with N_4 coordination.[1] Observation of an ESR signal in whole cells of *Methanobacterium thermoautotrophicum* has been attributed to Factor F_{430} in the intact, active enzyme, and the *g* values and hyperfine coupling constants of this signal are consistent with the presence of Ni^I (or Ni^{III}).[2] Reduction of factor F_{430} pentamethyl ester has been shown to yield a reduced species ($g = 2.065, 2.074, 2.250$) in which reduction is localized on the metal and the tetrahydrocorphin ring system is unaltered.[3]



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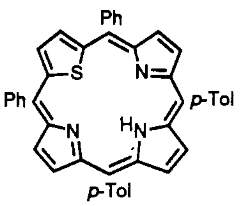

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It has generally been assumed that reduction of Ni^{II} within a macrocycle would lead to expansion of the Ni–ligand distances.[4-7] In considering the redox potentials for a wide range of Ni^{II} tetraaza macrocycles, Busch and co-workers[4] noted a correlation with core size and reasoned that larger cores better accommodate the (presumably) larger Ni^I and facilitated reduction. In examining the causes for varying degrees of unsaturation in biological macrocycles (e.g. porphyrin, chlorin, hydrocorphin), Stolzenberg and co-workers[5] have focused on hole size differences as determining the relative stability of different metal ion oxidation states and have estimated that a Ni^I–N distance would be optimal in excess of 2.1 Å. Here we report on the structure of a Ni^I complex of a macrocyclic ligand, diphenyldi-*p*-tolyl-21-thiaporphyrin (**2**, (SDPDTP)H), that allows determination of Ni–N distances within this environment along with direct comparison to a corresponding Ni^{II} complex, Ni^{II}(STTP)Cl (STTP is the monoanion of tetra-*p*-tolyl-21-thiaporphyrin).[8]

Brown, paramagnetic ($g = 2.030, 2.040, 2.109$ in frozen toluene) crystals of Ni^I(SDPDTP) were obtained as described previously[9]

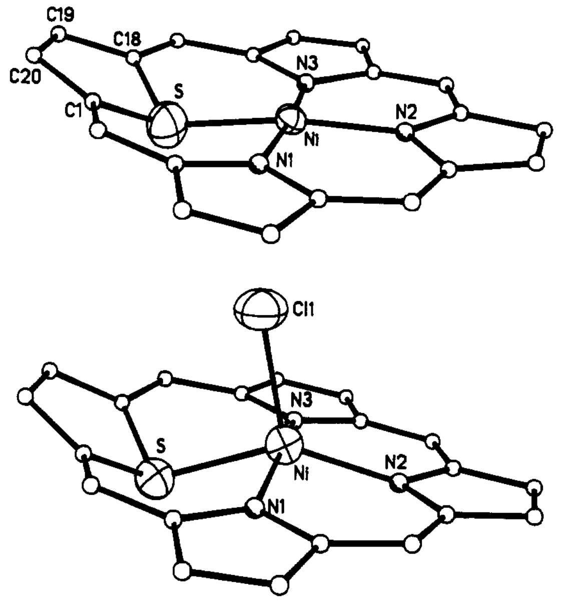

Figure 1. Comparison of the thiaporphyrin inner cores (phenyl and *p*-tolyl groups omitted) for (top) Ni^I(SDPDTP) and (bottom) Ni^{II}(STTP)Cl.

Table I. Characteristic Distances (Å) and Angles (deg)

	Ni^I-(SDPDTP)	Ni^{II}-(STPP)Cl[d]	Cu^{II}-(STPP)Cl[d]
	Distances		
M–N(1)	2.015 (12)	2.094 (3)	2.055 (6)
M–N(3)	2.014 (12)	2.084 (3)	2.057 (7)
M–N(2)	1.910 (14)	1.963 (4)	1.962 (8)
M–S	2.143 (6)	2.296 (1)	2.335 (2)
$M–N_3$[a]	0.042	0.295	0.274
	Angles		
S–M–N(2)	170.6 (4)	146.0 (1)	144.4 (2)
N(1)–M–N(3)	173.2 (6)	162.8 (1)	163.7 (2)
C(1)–S–C(18)	92.9 (9)	92.4 (5)	91.3 (5)
C(1)–S–M	119.4 (6)	107.6 (1)	106.3 (3)
C(18)–S–M	118.0 (7)	108.6 (1)	107.3 (3)
C_1SC_{18} vs M–S[b]	45.9	63.3	65.6
$C_1C_{18}C_{19}C_{20}$ vs C_1SC_{18}[c]	14.6	13.4	14.7

[a] Distance of the metal from the N_3 plane. [b] Angle between the M–S line and the C_1SC_{18} plane. [c] Angle of folding of the thiophene ring. [d] Data from ref 6.

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