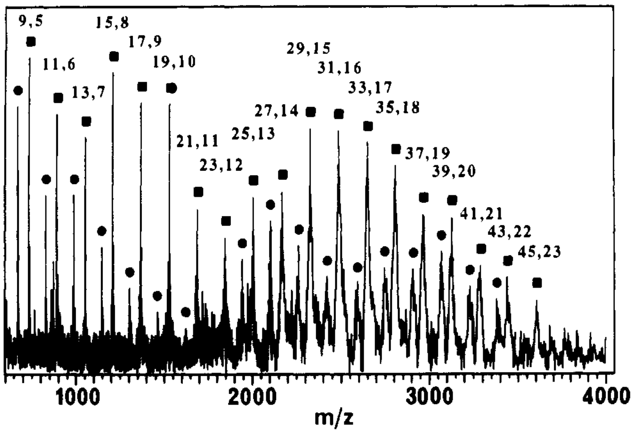

Figure 1. Part of the negative-ion FT-ICR broad-band mass spectrum of laser-ablated KCu_4S_3. Peak labels are *x,y* for ions $[Cu_xS_y]^-$. Peaks above *m/z* 1600 have not been unambiguously identified (see text) but are labeled as probable extensions of the low-mass series: ■, $x = 2y - 1$; ●, $x = 2y - 2$.

a lower intensity series of ions $[Cu_{2n-2}S_n]^-$, confirmed up to $n = 10$.

Samples of $Cu_{2-\delta}Se$ (synthetic Berzelianite) and of various mixtures of copper and selenium powders gave similar negative-ion spectra, but with different relative intensity patterns. Samples of both types gave significant amounts of the polyselenide ions Se_n^- ($n = 1, 2, 3, 4, 5, 8$). The negative-ion spectra of Cu_2Te (synthetic Weissite) and of mixtures of copper and tellurium powders were dominated by Te^-, which diminished the relative intensities of the $[Cu_xTe_y]^-$. Both sample types gave similar ions, but mixtures of copper and tellurium gave larger mass ions that cannot yet be unambiguously identified.[10]

There are two significant general conclusions about the cluster formation processes in these experiments. (i) The fact that the pattern of ions observed is largely independent of the composition of the sample ablated by the laser pulse suggests that reactions in the plasma during and immediately after ablation are the principal cluster formation processes. This is observed also for nickel chalcogenides[6a] and tantalum carbides.[11] It is established that high-power laser pulses cause immense heating of the sample, but the fact that the ions observed are present within milliseconds of the laser pulse causes us to believe that they are not formed by reactions of the molten elements. (ii) Combination of result i with the observation that the pattern of compositions of the negative ions $[Cu_xE_y]^-$ is largely independent of the chalcogen E (=S, Se, Te) indicates that this distribution of ions reflects the ion stabilities. Differences in relative intensities are presumably due to some differences in the plasma dynamics.

Features of the ion compositions are the excess of metal atoms ($x > y$) in all but the smallest ions (this occurs also for $[Ni_xS_y]^-$ ions on laser ablation of Ni_3S_2[6a]) and the series of ions $[Cu_{2n-1}E_n]^-$ that progress by addition of Cu_2E where nine or more Cu atoms are present. Experiments using samples with larger E:Cu ratios increase the proportions of $[E_y]^-$ ions, without changing the compositions of the $[Cu_xE_y]^-$ ions. $[Cu_6E_4]^-$ has large relative intensity, which bears comparison with the [*cuboctahedro*-Cu_{12}-*hexahedro*-S_8]$^{4-}$ ion characterized in the condensed phase.[12] The high Cu:E ratio may be contrasted to the composition of the [*tetrahedro*-Cu_4-*octahedro*-$(ER)_6$] clusters prevalent with chalcogenolate ligands.[13] The element ratios in the ion series $[Cu_{2n-1}E_n]^-$ (observed also for silver[6b]) resemble those of the core compositions of the copper selenide clusters $[Cu_{29}Se_{15}(PPr_3^i)_{12}]$, $[Cu_{30}Se_{15}(PPr_3^i)_{12}]$, and $[Cu_{36}Se_{18}(PBu_3^t)_{12}]$ recently reported by Fenske.[14] We note that the probable assignment of the most intense peak (*m/z* 2322) in the higher mass envelope in Figure 1 is $[Cu_{29}S_{15}]^-$, the same as the core composition of $[Cu_{29}Se_{15}(PPr_3^i)_{12}]$.[14] Freiser has recently reported that laser ablation of copper oxide, CuO, gives different results.[15] The positive ions include the series $[Cu_{2n}O_n]^+$, $n = 1$–6, as well as some ions $[Cu_{2n+1}O_n]^+$, $[Cu_{2n-1}O_n]^+$, and $[Cu_{2n-2}O_n]^+$, while the negative ions fall in several series $[Cu_{2n-m}O_n]^-$, $m = 2$–5.

Metal chalcogenides are notoriously nonmolecular in structure, and yet the phenomena and applications cited in the introduction relate to molecular clusters, nanocrystallites, and to the control of structural molecularity. A standard method for avoiding or regulating the condensation of molecular clusters is the use of obstructive terminal ligands in designed syntheses.[14,16] However, complexes such as $[Cu_{29}Se_{15}(PPr_3^i)_{12}]$, $[Cu_{30}Se_{15}(PPr_3^i)_{12}]$, $[Cu_{36}Se_{18}(PBu_3^t)_{12}]$, and $[Ni_{20}Te_{18}(PEt_3)_{12}]$[16] are thermally unstable relative to the nonmolecular binary metal chalcogenides.[14,16] Our experiments, which allow preservation of the molecular metal chalcogenide ions in the gas phase, provide an alternative entry to examination of their fundamental chemistry. We are currently investigating the reactivities of these ions by collisional activation and ion-molecule reactions.

Acknowledgment. This research was supported by the Australian Research Council. K.J.F. acknowledges the award of an Australian National Research Fellowship.

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(11) McElvany, S. W.; Cassady, C. J. *J. Phys. Chem.* **1990,** *94,* 2057.
(12) Betz, P.; Krebs, B.; Henkel, G. *Angew. Chem., Int. Ed. Engl.* **1984,** *23,* 311
(13) Dance, I. G. *Polyhedron* **1986,** *5,* 1037.
(14) Fenske, D.; Krautscheid, H.; Balter, S. *Angew. Chem., Int. Ed. Engl.* **1990,** *29,* 769.
(15) Gord, J. R.; Bemish, R. J.; Freiser, B. S. *Int. J. Mass Spectrom. Ion Processes* **1990,** *102,* 115.
(16) Brennan, J. G.; Siegrist, T.; Stuczynski, S. M.; Steigerwald, M. L. *J. Am. Chem. Soc.* **1989,** *111,* 9240.

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Received December 28, 1990

An Isostructural Series of Molecular Metamagnets: The Organometallic Charge-Transfer Salts Decamethylmanganocenium Bis[bis(trifluoromethyl)ethylenedithiolato]nickelate, -palladate, and -platinate

Despite substantial efforts,[1] the search for ferromagnetically ordered molecular charge-transfer salts has yielded only two such compounds: $[Fe(Cp^*{}_2)][TCNE]$[2,3] and $[Mn(Cp^*)_2][TCNQ]$.[4] In addition, $[Fe(Cp^*)_2][TCNQ]$ is a metamagnet,[5] a compound

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(1) (a) Miller, J. S.; Epstein, A. J.; Reiff, W. M. *Chem. Rev.* **1988,** *88,* 201-220. (b) Miller, J. S.; Epstein, A. J.; Reiff, W. M. *Science* **1988,** *240,* 40-47; *Acc. Chem. Res.* **1988,** *21,* 114-120. (c) Breslow, R. *Pure Appl. Chem.* **1982,** *54,* 927-938; *Mol. Cryst. Liq. Cryst.* **1985,** *125,* 261-267. (d) For other approaches to new types of organic/molecular magnetically ordered solids, see: Kahn, O. *J. Chim. Phys.-Chim. Biol.* **1988,** *85,* 1113-1118. Caneschi, A.; Gatteschi, D.; Sessoli, R. *Acc. Chem. Res.* **1989,** *22,* 392-398. Korshak, Y. V.; Ovchinnikov, A. A.; Shapino, A. M.; Medvedeva, T. V.; Spector, V. N. *JETP Lett.* (*Engl. Transl.*) **1986,** *43,* 399. Fujita, I.; Teki, Y.; Takui, T.; Kinoshita, T.; Itoh, K.; Miko, F.; Sawaki, Y.; Iwamura, H.; Izuoka, A.; Sugawara, T. *J. Am. Chem. Soc.* **1990,** *112,* 4074-4075. Allemand, P.-M.; Srdanov, G.; Wudl, F. *J. Am. Chem. Soc.* **1990,** *112,* 9391-9392. Torrance, J. B.; Bagus, P. S. Johannsen, I.; Nazzal, A. I.; Parkin, S. S.. P.; Batail, P. *J. Appl. Phys.* **1988,** *63,* 2962-2965. Dougherty, D. A. *Acc. Chem. Res.* **1991,** *24,* 88-94.
(2) Abbreviations: $Cp^* = [C_5(CH_3)_5]^-$; TCNQ = 7,7,8,8-tetracyanoquinodimethanide; TCNE = tetracyanoethyleneide; tfd = bis(trifluoromethyl)ethylenedithiolate.
(3) Miller, J. S.; Calabrese, J. C.; Rommelmann, H.; Chittipeddi, S. R.; Zhang, J. H.; Reiff, W. M.; Epstein, A. J. *J. Am. Chem. Soc.* **1987,** *109,* 769-781.
(4) Broderick, W. E.; Thompson, J. A.; Day, E. P.; Hoffman, B. M. *Science* **1990,** *249,* 401-403.

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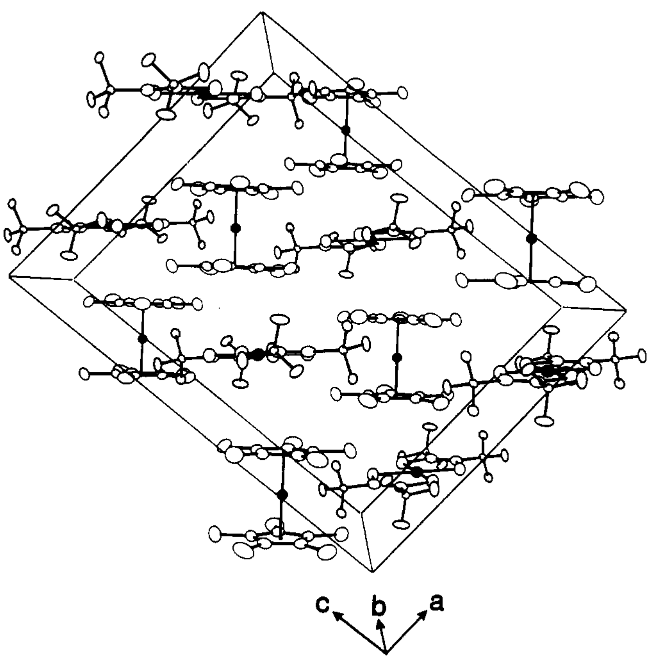

Figure 1. Packing diagram of **1**, showing a perspective view of the ...$D^+A^-D^+A^-$... stacking arrangement in the *ac* plane.

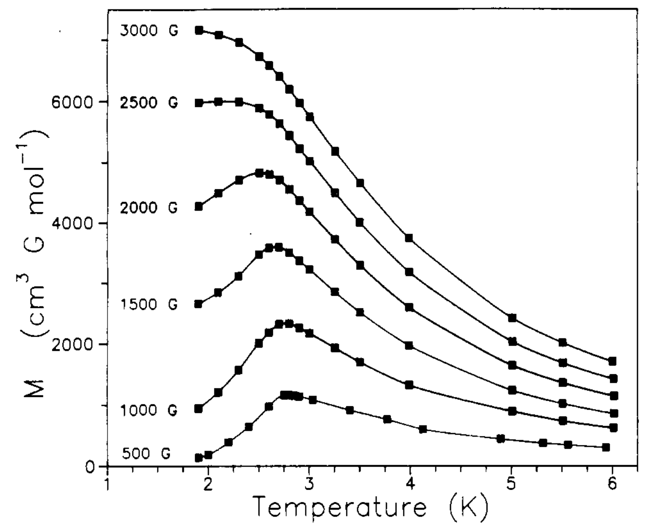

Figure 2. Field-cooled magnetization data for **2** in applied fields (H_A) as indicated.

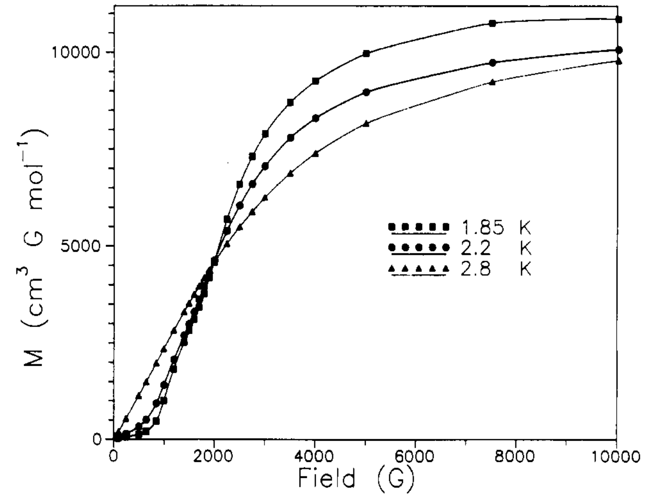

Figure 3. Magnetization isotherms for **2** at temperatures as indicated.

with predominantly ferromagnetic couplings that shows a field-induced transition from an antiferromagnetic to a ferromagnetic ground state.[6] We now report that the use of the $S = 1$ cation $[Mn(Cp^*)_2]^+$ with a transition-metal bis(dichalcogolene) complex as the anion[7] has given a series of three charge-transfer molecular crystals, each of which displays cooperative magnetic behavior. Each of the compounds $[Mn(Cp^*)_2]^+[M(tfd)_2]^-$ (M = Ni (**1**), Pd (**2**), Pt (**3**); tfd = bis(trifluoromethyl)ethylenedithiolato) is a metamagnet. The failure of the corresponding CT salt (**4**) of the $S > {}^1/_2$ $[Fe(Cp^*)_2]^+$ cation to display magnetic ordering[8] supports our proposal[4] that the use of higher spin components will increase the ordering temperatures, T_c. To illustrate, for a given exchange coupling, $T_c \propto S(S + 1)$ in mean-field theory.[6c]

The structure of the 1:1 CT salt **1** was determined by single-crystal X-ray crystallography.[9] The crystal lattice of **1** (Figure 1) consists of an array of parallel stacks of alternating $[Mn(Cp^*)_2]^+$ cations ($[D^+]$) and $[Ni(tfd)_2]^-$ anions ($[A^-]$) parallel to [101]. This ...$D^+A^-D^+A^-$... stack arrangement is similar to that found in other decamethylmetallocenium CT salts,[1,3-5] and in fact **1** is isostructural to the decamethylferrocenium analogue[8] (**4**). There are no contacts less than the sum of the van der Waals radii between D^+ and A^- in **1**. The Mn–Ni distance within the ...$D^+A^-D^+A^-$... stack is 6.043 Å. The salts **2** and **3** were found to be isostructural to **1** by comparison of powder X-ray data for the three compounds.

Magnetization data for **1–3** were measured with a SQUID magnetometer.[12] An analysis of field-cooled magnetization data recorded from 300 to 1.85 K at 500 G reveals that for temperatures down to 50 K **1–3** obey the Curie–Weiss law, $\chi = C/(T - \Theta)$, with ferromagnetic Weiss constants Θ = 2.6, 3.7, and 1.9 K, respectively. Room-temperature values $\chi_M T$ = 2.03, 1.82, and 1.91 cm^3 K mol^{-1} for **1–3** correspond to $\mu_{eff} = (3k/N)^{1/2}(\chi_M T)^{1/2}$ = 4.03, 3.81, and 3.91 μ_B, respectively. These values of μ_{eff} are slightly greater than the spin-only value (3.32 μ_B) calculated for an uncorrelated two-spin system with $S_{D^+} = 1$, $S_{A^-} = {}^1/_2$, and $g = 2$ for both spins. Because the average g values for the $[M(tfd)_2]^-$ anions are only slightly greater than the spin-only value,[13] the enhanced values for μ_{eff} are attributed to an orbital contribution

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(5) (a) Candela, G. A.; Swartzendruber, L. J.; Miller, J. S.; Rice, M. J. *J. Am. Chem. Soc.* **1979**, *101*, 2755–2756. (b) Miller, J. S.; Reis, A. H., Jr.; Gebert, E.; Ritsko, J. J.; Salaneck, W. R.; Kovnat, L.; Cape, T. W.; Van Duyne, R. P. *J. Am. Chem. Soc.* **1979**, *101*, 7111–7113. (c) Miller, J. S.; Zhang, J. H.; Reiff, W. M.; Dixon, D. A.; Preston, L. D.; Reis, A. H., Jr.; Gebert, E.; Extine, M.; Troup, J.; Epstein, A. J.; Ward, M. D. *J. Phys. Chem.* **1987**, *91*, 4344–4360.

(6) (a) Carlin, R. L. *Magnetochemistry*; Springer Verlag: New York, 1986. Carlin, R. L.; vanDuyneveldt, A. J. *Acc. Chem. Res.* **1980**, *13*, 231–236. (b) DeFotis, G. C.; McGhee, E. M.; Bernal, I.; Losee, D. B. *J. Appl. Phys.* **1987**, *61*, 3298–3300. (c) Smart, J. S. *Effective Field Theories of Magnetism*; W. B. Saunders: Philadelphia, 1966.

(7) Broderick, W. E.; Thompson, J. A.; Godfrey, M. R.; Sabat, M.; Hoffman, B. M.; Day, E. P. *J. Am. Chem. Soc.* **1989**, *111*, 7656–7657.

(8) Miller, J. S.; Calabrese, J. C.; Epstein, A. J. *Inorg. Chem.* **1989**, *28*, 4230–4238.

(9) The CT salts of **1–3** were prepared by addition of a concentrated acetonitrile solution of $[Mn(Cp^*)_2][PF_6]$[10] to a methanol solution of the appropriate tetraethylammonium metal bis-tfd salt.[11] In each case, brown crystals deposited on standing. Crystals of **1** suitable for single-crystal X-ray analysis were obtained by recrystallization from acetone. Crystallographic data for **1**: $C_{28}H_{30}F_{12}S_4MnNi$, $M_r = 848.4$; monoclinic, space group $C2/c$ (No. 15); $a = 14.302$ (2), $b = 12.697$ (2), and $c = 18.415$ (1) Å; $\beta = 94.63$ (1)°; $V = 3333$ (1) Å^3; $Z = 4$; $\rho_{calcd} = 1.67$ g·cm^3; $\mu = 12.57$ cm^{-1}. The structure was solved by a combination of direct methods and Fourier syntheses in TEXSAN 4.0. Full-matrix least-squares refinement gave the final agreement factors, $R(F) = 0.044$ and $R_w(F) = 0.062$ for 2486 reflections (with $F^2 \geq 3\sigma(F^2)$) measured between $4 \leq 2\theta \leq 50$ at −120 °C (Mo Kα radiation, λ = 0.71069 Å).

(10) Robbins, J. L.; Edelstein, N. M.; Cooper, S. R.; Smart, J. C. *J. Am. Chem. Soc.* **1979**, *101*, 3853–3857.

(11) (a) Davison, A.; Edelstein, N.; Holm, R. H.; Maki, A. H. *Inorg. Chem.* **1964**, *3*, 814–823. (b) Davison, A.; Howe, D. V.; Shawl, E. T. *Inorg. Chem.* **1967**, *6*, 458–463.

(12) Magnetization data were corrected for contributions due to the sample holder and core diamagnetism, which were estimated from Pascal's constants.

(13) $[Ni(tfd)_2]^-$, $\langle g \rangle = 2.062$; $[Pd(tfd)_2]^-$, $\langle g \rangle = 2.024$; $[Pt(tfd)_2]^-$, $\langle g \rangle = 2.039$.[11a]

to the g factor of $[\mathrm{Mn(Cp^*)_2}]^+$.[4]

As **1**–**3** are cooled below 20 K in a field of 500 G, χ_M *and* $\chi_M T$ increase rapidly, which together with the positive Θ values observed for **1**–**3** indicate that ferromagnetic interactions dominate. However, as shown in Figure 2, χ_M for **2** reaches a maximum in the range 2–3 K and then decreases sharply, signaling an antiferromagnetic phase transition. Similar behavior is observed for **1** and **3** and the Néel temperatures are T_N = 2.4, 2.8, and 2.3 K for **1**–**3**, respectively. The presence of significant ferromagnetic interactions in these materials, as evidenced in the susceptibilities above T_N, along with the previous observations that $[\mathrm{Mn(Cp^*)_2}]^+$ CT salts typically exhibit large magnetic anisotropies,[3,14] suggested that **1**–**3** might exhibit metamagnetic behavior,[6] where the application of a relatively weak magnetic field causes a transition from an antiferromagnetic to a ferromagnetic ground state. We therefore investigated the field dependence of the magnetization in the range 1.85–10 K.

Field-cooled magnetization curves for **2** (Figure 2) demonstrate that **2** undergoes such a field-induced transition. As H_A is increased from 500 G, the maximum in χ_M broadens, shifts to lower temperatures, and finally disappears for $H_A > 2500$–3000 G, demonstrating the transition from antiferromagnetic to ferromagnetic behavior; similar data are obtained for **1** and **3**. Figure 3 presents magnetization isotherms for **2** recorded at 1.85, 2.20, and 2.80 K. As the temperature is reduced, the isotherms become increasingly sigmoidal. At the lowest temperature, T = 1.85 K, M first increases slowly with H_A, as for an antiferromagnet and then increases rapidly in a field-induced phase transition to the ferromagnetic state, as expected for a metamagnet with $T < T_N$.[6] The critical field at this temperature, defined as the extrapolation of the transition region to $M = 0$, is H_c = 800 G.

In the T = 1.85 K isotherm the magnetization attains the value M = 12 500 emu G/mol at the highest field employed (40 000 G; data not shown). As is typical of metamagnets where T is not far below T_N,[5a,6b] even at this field M has not fully reached the saturation magnetization value, $M_{\mathrm{sat}} = N\beta[g_D S(\mathrm{D}^+) + g_A S(\mathrm{A}^-)] \approx 16\,800$ emu G/mol, expected for the ferromagnetic state when $S(\mathrm{D}^+) = 1$ and $S(\mathrm{A}^-) = {}^1/_2$ (g = 2 for both spins). However, it is far larger than $M_{\mathrm{sat}} = N\beta[g_D S(\mathrm{D}^+) - g_A S(\mathrm{A}^-)] \approx 5600$ emu G/mol expected if the field-induced transition instead were to a high-field state that is ferrimagnetic. In addition, M_{sat} is expected to be lower than the theoretical value owing to the polycrystalline nature of the sample and the expected anisotropy for a metamagnet material.

Why are **1**–**3** metamagnets, whereas the Fe analogue, **4**, is not? We take the high-temperature behavior of χ_M, along with the appearance of a metamagnetic transition, to indicate that the $\mathrm{D^+}$–$\mathrm{A^-}$ intrachain coupling in **1**–**3** is strong and ferromagnetic, as in $[\mathrm{Mn(Cp^*)_2}][\mathrm{TCNQ}]$,[4] but that weak interchain antiferromagnetic coupling leads to the metamagnetic behavior and field-dependent ground state demonstrated in Figure 2. In this case one might expect[15] $T_N \propto |E_{\mathrm{intra}}E_{\mathrm{inter}}|^{1/2}$, where E_{intra} is the intrachain near-neighbor $\mathrm{D^+}$–$\mathrm{A^-}$ interaction, $E_{\mathrm{intra}} \propto S_D S_A J_{DA}$, and E_{inter} is the much weaker, effective interchain interaction energy. We propose that an increase in E_{intra} in **1**–**3** caused by the increase in spin for $\mathrm{D^+}$ from $S = {}^1/_2$ in **4** to $S = 1$, along with possible increases in J_{DA}, contributes to the observation of a magnetically ordered state for **1**–**3**. It is hoped that detailed magnetic–structural correlations will clarify the mechanism of spin coupling within these salts and rationalize the variation in T_N.

Acknowledgment. We thank Prof. E. P. Day and Mr. Jack Vaughey for technical assistance. This work has been supported by the Solid State Chemistry Program of the National Science Foundation (Grant DMR-8818599) and by the Northwestern University Materials Research Center under the NSF-MRL program (Grant DMR-8821571).

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(14) Broderick, W. E.; Day, E. P.; Hoffman, B. M. Unpublished results.

(15) (a) Villain, J.; Loveluck, J. M. *J. Phys. Lett.* **1977**, *38*, L77–L80. (b) Caneschi, A.; Gatteschi, D.; Renard, J. P.; Rey, P.; Sessoli, R. *Inorg. Chem.* **1989**, *28*, 3314–3319.

Supplementary Material Available: A table of positional parameters and B_{eq} values (Å^2) for the atoms of **1** (1 page). Ordering information is given on any current masthead page.

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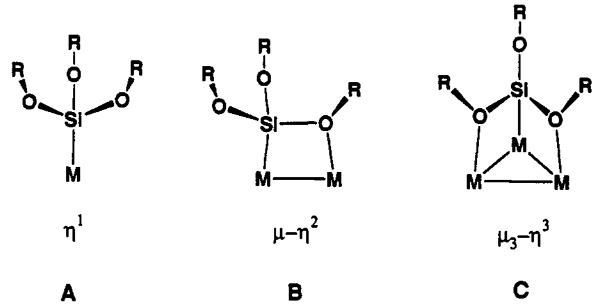
Received February 13, 1991

Unusual Coordination Properties of Trialkoxysilyl Groups in Metal Cluster Complexes. Synthesis, Structure, and Reactivity of $\mathrm{Os_3(CO)_9[\mu_3\text{-}\eta^3\text{-}Si(OEt)_3](\mu\text{-}H)}$

Alkoxysilanes are widely used in metal-catalyzed hydrosilation reactions because of their high reactivity.[1] In most cases, trialkoxysilyl groups coordinate to metal atoms in the conventional η^1 mode (A) that is commonly found for trialkyl- and triarylsilyl groups. Recent reports by Braunstein et al. of the first examples of μ-η^2-siloxyl groups (B) in binuclear metal complexes have provided the first evidence that the alkoxy groups are not always innocent spectators in metal complexes.[2] We now wish to report the first example of a μ_3-η^3-siloxyl ligand (C) in a trinuclear metal complex as further evidence of the unusual coordination properties of these ligands.



The reaction of $\mathrm{Os_3(CO)_{10}(NCMe)_2}$ with $\mathrm{HSi(OEt)_3}$ at 25 °C for 1 h has yielded the complex $\mathrm{Os_3(CO)_{10}(NCMe)[Si(OEt)_3](\mu\text{-}H)}$ (**1**; 84%). Compound **1** was characterized by IR, ^{1}H NMR, and single-crystal X-ray diffraction analyses.[3] The crystal contains two independent but structurally similar molecules in the asymmetric crystal unit.[4,5] An ORTEP drawing of the molecular structure of one of these is shown in Figure 1. The molecule consists of a triangular triosmium cluster with an η^1-$\mathrm{Si(OEt)_3}$ ligand, type A, coordinated to only one metal atom,

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(1) (a) Lewis, L. N. *J. Am. Chem. Soc.* **1990**, *112*, 5998. (b) Harrod, J. F.; Chalk, A. J. In *Organic Synthesis via Metal Carbonyls*; Wender, I., Pino, P., Eds.; John Wiley: New York, 1977. (c) Ojima, I.; Clos, N.; Donovan, R. J.; Ingallina, P. *Organometallics* **1990**, *9*, 3127.

(2) (a) Braunstein, P.; Knorr, M.; Tiripicchio, A.; Tiripicchio Camellini, M. *Angew. Chem., Int. Ed. Engl.* **1989**, *28*, 1361. (b) Braunstein, P.; Knorr, M. *New J. Chem.* **1990**, *14*, 583. (c) Braunstein, P.; Knorr, M.; Piana, H.; Schubert, U. *Organometallics* **1991**, *10*, 828.

(3) A solution of $\mathrm{Os_3(CO)_{10}(NCMe)_2}$, 103.9 mg (0.111 mmol), in 60 mL of $\mathrm{CH_2Cl_2}$ was allowed to react with 61.7 μL (0.334 mmol) of triethoxysilane at 25 °C for 1 h. The product was separated by TLC using a 3/2 $\mathrm{CH_2Cl_2}$/hexane solvent mixture to yield 98.8 mg (84% yield) of **1**. IR, ν(CO) (cm^{-1}), in hexane: 2103 (m), 2065 (vs), 2041 (vs), 2021 (vs), 2008 (s), 2000 (vs), 1988 (vs), 1976 (m). ^{1}H NMR, δ, in $\mathrm{CDCl_3}$: 3.89 (q, $\mathrm{C}H_2$, 6 H), 2.57 (s, $\mathrm{NCC}H_3$, 3 H), 1.27 (t, $\mathrm{C}H_3$, 9 H), −16.40 (s, OsH, 1 H). Satisfactory elemental analyses (C, H, N) have been obtained. Yellow crystals of **1** were grown from a $\mathrm{CH_2Cl_2}$/petroleum ether solution by slow evaporation of solvent at 25 °C.

(4) Crystal data for **1**: space group = $P\bar{1}$, a = 16.630 (5) Å, b = 20.062 (7) Å, c = 9.501 (2) Å, α = 101.04 (3)°, β = 103.22 (2)°, γ = 106.26°, Z = 4, 3603 reflections, R = 0.051.

(5) Diffraction measurements were made on a Rigaku AFC6S fully automated four-circle diffractometer using graphite-monochromatized Mo Kα radiation. All data were processed on a Digital Equipment Corp. VAXstation 3520 computer by using the TEXSAN structure solving program library (version 5.0) obtained from the Molecular Structure Corp., The Woodlands, TX. An absorption correction was applied.

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