solution and refinement. The dimensions, unit cell parameters, and other crystal data are given in Table V. Throughout data collection four standard reflections were measured every 50 reflections to monitor stability. The structures were solved by direct methods using MULTAN 78 for **1a** and MULTAN 76 for **1b**. *E* maps revealed the positions of the silicon and carbon atoms. Subsequent electron density difference maps revealed overlapping positions for the disordered toluene solvate. Further electron density difference maps revealed the nonsolvent hydrogen atoms. In the final cycles of refinement all non-hydrogen atoms were assumed to vibrate anisotropically and all hydrogen atoms were assumed to vibrate isotropically. The final *R* values ($R_1 = \sum||F_o| - |F_c||/\sum|F_o|$; $R_2 = [\sum w(|F_o| - |F_c|)^2/\sum w(F_o)^2]^{1/2}$) are also included in Table V. The final atomic coordinates for **1a** and **1b** are listed in Tables VI and VII, respectively.

Acknowledgment. Research was supported by the Air Force Office of Scientific Research, Air Force Systems Command, USAF, under Grant No. AFOSR-82-0067 and the National Science Foundation (Grant CHE80-00256). The United States government is authorized to reproduce and distribute reprints for governmental purposes notwithstanding any copyright notation thereon.

Registry No. **1a**, 80785-72-4; **1b**, 88526-23-2; **2a**, 79184-72-8; **2b**, 88526-27-6; **3a**, 79184-71-7; **3b**, 89486-28-2; **4a**, 88957-30-6; **4b**, 89486-29-3; **5a**, 84537-22-4; **5b**, 89486-30-6; MesLi, 5806-59-7; (Mes)$_2$,siCl$_2$, 5599-27-9; (Mes)SiCl$_3$, 17902-75-9; (Mes)(*t*-Bu)SiCl$_2$, 89486-31-7; bromomesitylene, 576-83-0.

Supplementary Material Available: Final anisotropic thermal parameters and listings of $10|F_o|$ and $10|F_c|$ for **1a** and **1b** (31 pages). Ordering information is given on any current masthead page.

Communications

Synthesis and Structural Characterization of $[Fe_4(\mu_3\text{-}S)_3(\mu_3\text{-}S_2)Cp_4][MoOCl_4(thf)]$. A New Fe–S Cluster with Different Modes of Coordination at the Iron Sites

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Received November 22, 1983

Summary: Reaction of $Fe_4S_6(Cp)_4$ with $MoOCl_3(thf)_2$ generates $[Fe_4(\mu_3\text{-}S)_3(\mu_3\text{-}S_2)Cp_4][MoOCl_4(thf)]$. Structural data show that the cation contains a distorted Fe_4S_5 core with a triply bridging S_2^{2-} ligand and with one Fe having expanded its ligation to four sulfurs. EPR and Mössbauer studies lead to the core formal electronic description [3 Fe^{III}, 1 Fe^{II}].

Recent findings show that natural[1-3] Fe–S clusters are not confined to the well-studied species having planar 2Fe–2S and cubane-type [4Fe–4S] core units.[4]

This has triggered a series of synthetic efforts to obtain heretofore unknown polynuclear Fe/S geometries, which have resulted in the isolation and structural characterization of $[Et_4N][Fe_3S(O\text{-}xyl)_3]$,[5] $[Et_4N]_3[Fe_3S_4(SPh)_4]$,[6] $Fe_4S_6(Cp)_4$,[7] $[Fe_6S_8(PEt_3)_6][BPh_4]_2$,[8] and $[Et_4N][Fe_6S_9(SEt)_2]$.[6,9]

Moreover, [4Fe–4S] clusters of a novel type have been identified at the "P" sites of nitrogenase,[10] with unique and so far unexplained spectral and magnetic properties. It has been suggested[10,11] that the protein could achieve this by use of non-thiolate ligands at the Fe sites, or geometric distortion of the 4Fe–4S core from tetrahedral geometry, or expansion to pentacoordination for the Fe atoms.

While investigating the reactivity of Mo compounds vs. $Fe_4S_6Cp_4$, we isolated a solid of composition $[Fe_4S_5Cp_4][MoOCl_4(thf)]$ (I). We report here the synthesis and structural characterization of this interesting cluster, where a S_2^{2-} ligand bonded to three Fe sites is present and one of the Fe's has expanded its ligation to four S and one Cp.

Reaction of $Fe_4S_6Cp_4$[7] with $MoOCl_3(thf)_2$[12] in dichloromethane (CH_2Cl_2) at room temperature under argon gives instantenously complex I,[13] presumably by reaction of Mo with one of the disulfide groups, followed by S abstraction.

$$Fe_4S_6(Cp)_4 + 2MoOCl_3(thf)_2 \rightarrow [Fe_4S_5Cp_4][Mo^{V}OCl_4(thf)] + (1/n)[Mo^{VI}OSCl_2]_n$$

Its infrared spectrum displays $\nu(Mo{=}O) = 957$ (m) cm^{-1}, $\nu(Mo{-}Cl) = 330$ (sh), 305 (s) cm^{-1}, $\nu(S{-}S) = 525$ (w) cm^{-1}, and $\nu(Fe{-}S) = 466$ (m) cm^{-1}.[14] The UV–visible spectrum

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(13) In an argon-filled glovebox, 0.348 g (0.5 mmol) of $Fe_4S_6Cp_4$ in 100 mL of CH_2Cl_2 is added to a solution (20 mL) of 0.357 mg (1 mmol) of $MoOCl_3(thf)_2$ also in CH_2Cl_2, and the resulting mixture is stirred for 15 min. The brown solid is filtered, washed with CH_2Cl_2, and dried under vacuum. Complex I is extracted with acetonitrile, whereby a brown residue is left, which is insoluble in most organic solvents and tentatively formulated as $MoOSCl_2$ of polymeric structure (diamagnetic; $\nu(Mo{-}O{-}Mo) = 700$ (m) cm^{-1}, $\nu(Mo{-}S{-}Mo) = 455$ (m) cm^{-1}, $\nu(Mo{-}Cl) = 330$ (s) cm^{-1}. Complex I is soluble, but decomposes in chlorinated solvents and is recrystallized from CH_3CN–THF–hexane (35% yield). Elemental analyses were performed by Le Service Central d'Analyse, CNRS, Vernaison. Satisfactory analytical data were obtained for C, H, S, Cl, Fe, Mo.

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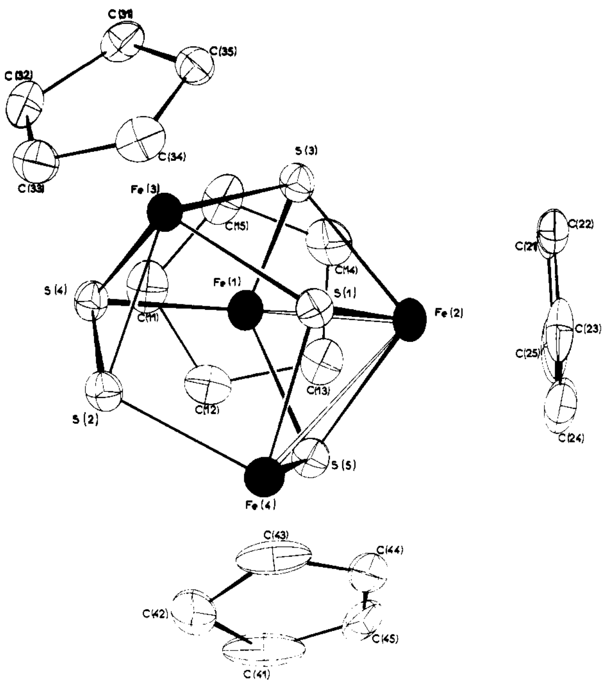

Figure 1. ORTEP drawing of the $[Fe_4S_5Cp_4]^+$ cation. Thermal ellipsoids are plotted at the 20% level. Hydrogen atoms are omitted for clarity.

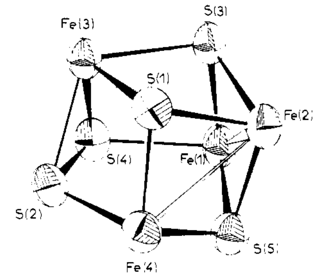

Figure 2. ORTEP drawing of the Fe_4S_5 core of the $[Fe_4S_5Cp_4]^+$ cation. Thermal ellipsoids are plotted at the 50% level.

is characterized by absorptions at 430, 360, 285, and 210 nm.

In order to obtain a complete information on the Fe–S core structure, a single-crystal X-ray diffraction study of complex I was performed[15] (see Figures 1 and 2 and Table I). The molecule is constructed of discrete $[Fe_4(\mu_3\text{-}S)_3(\mu_3\text{-}S_2)Cp_4]$ cations and $[MoOCl_4(thf)]$ anions. The geometry of $[MoOCl_4(thf)]^-$ is essentially the same as already reported.[16] Its interatomic distances and bond angles are available as supplementary material.

Table I. Selected Geometric Parameters for $[Fe_4(\mu_3\text{-}S)_3(\mu_3\text{-}S_2)Cp_4]^+$

atom type	bond length, Å	atom type	bond angle, deg
Fe(1)-S(3)	2.189 (5)	S(3)-Fe(1)-S(5)	99.6 (2)
Fe(1)-S(4)	2.218 (3)	S(4)-Fe(1)-S(5)	95.2 (2)
Fe(1)-S(5)	2.186 (4)	S(1)-Fe(2)-S(5)	95.8 (2)
Fe(2)-S(1)	2.240 (4)	S(3)-Fe(2)-S(5)	98.9 (2)
Fe(2)-S(3)	2.188 (4)	S(1)-Fe(4)-S(5)	96.8 (2)
Fe(2)-S(5)	2.208 (5)	S(2)-Fe(4)-S(5)	95.7 (2)
Fe(3)-S(1)	2.284 (4)	S(3)-Fe(1)-S(4)	81.4 (2)
Fe(3)-S(2)	2.276 (5)	S(1)-Fe(2)-S(3)	79.0 (2)
Fe(3)-S(3)	2.277 (3)	S(1)-Fe(3)-S(2)	77.3 (2)
Fe(3)-S(4)	2.271 (4)	S(1)-Fe(3)-S(3)	76.3 (2)
Fe(4)-S(1)	2.210 (5)	S(3)-Fe(3)-S(4)	78.4 (2)
Fe(4)-S(2)	2.194 (4)	S(1)-Fe(4)-S(2)	80.6 (2)
Fe(4)-S(5)	2.200 (4)	S(1)-Fe(3)-S(4)	110.2 (2)
S(2)-S(4)	2.067 (4)	S(2)-Fe(3)-S(4)	111.2 (2)
Fe(1)-Fe(2)	2.723 (3)	S(2)-Fe(3)-S(3)	54.1 (2)
Fe(2)-Fe(4)	2.866 (3)	Fe(2)-S(1)-Fe(3)	98.2 (2)
Fe(1)···Fe(3)	3.369 (3)	Fe(3)-S(1)-Fe(4)	98.4 (2)
Fe(1)···Fe(4)	3.721 (3)	Fe(3)-S(2)-Fe(4)	99.1 (2)
Fe(2)···Fe(3)	3.420 (3)	Fe(1)-S(3)-Fe(3)	97.9 (2)
Fe(3)···Fe(4)	3.402 (3)	Fe(2)-S(3)-Fe(3)	100.0 (2)
		Fe(2)-S(4)-Fe(3)	97.3 (2)
		Fe(2)-S(1)-Fe(4)	80.2 (2)
		Fe(1)-S(3)-Fe(2)	76.9 (2)
		Fe(1)-S(5)-Fe(2)	76.6 (2)
		Fe(2)-S(5)-Fe(4)	81.1 (2)
		Fe(1)-S(5)-Fe(4)	116.1 (2)
		Fe(4)-S(2)-S(4)	112.3 (2)
		Fe(1)-S(4)-S(2)	111.7 (2)
		Fe(3)-S(2)-S(4)	62.8 (2)
		Fe(3)-S(4)-S(2)	63.1 (2)

The structural arrangement of $[Fe_4(\mu_3\text{-}S)_3(\mu_3\text{-}S_2)Cp_4]^+$ will be discussed in relation to the already structurally investigated $[Fe_4S_xCp_4]^{n+}$ clusters[17-20] ($x = 4, 6$; $n = 0, 1, 2$). Three different types of iron atoms are present: (a) Fe(1) and Fe(4), each bonded to three sulfur atoms and interacting with one iron atom; (b) Fe(2), bonded to three sulfur atoms and interacting with two iron atoms; (c) Fe(3), bonded to four sulfur atoms and not involved in any Fe–Fe bond. Like $Fe_4S_4Cp_4$, $[Fe_4S_4Cp_4]^+$, $Fe_4S_6Cp_4$, and $[Fe_4S_6Cp_4]^+$, the present cluster shows two Fe–Fe bonds and four nonbonding Fe–Fe distances. In our case however, Fe(2) is involved in both Fe–Fe bonds, and Fe(3) is not involved in any, whereas in the above-mentioned examples all four iron atoms are involved in one Fe–Fe bond each.

The sulfur atoms are present as three triply bridging S and one triply bridging S_2 group. The way in which the disulfide is coordinated to the three iron atoms is different from the way found in $Fe_4S_6Cp_4$.[20] As in $Mn_4S_4(CO)_{15}$,[21] the S_2 ligand is "side-on" bonded to one metal atom, while each of its sulfurs is bonded to one other metal atom. In $Fe_4S_6Cp_4$, one of the S_2 sulfur atoms is bonded to two iron atoms, while the other sulfur is bonded to a third iron atom.

The Fe–S distances divide into two sets: those "short" bonds involving Fe(1), Fe(2), and Fe(4) range from 2.186 (4) to 2.240 (4) Å, while the four "long" Fe(3)–S bonds range from 2.271 (4) to 2.284 (4) Å. This increase of coordination number around Fe(3), together with the replacement of a sulfide by a disulfide, accounts for the

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[15] A black platelike crystal was sealed under argon in a Lindemann capillary. Crystal data: space group $P2_12_12_1$ $M_r = 969.9$, $a = 28.253$ (4) Å, $b = 14.972$ (2) Å, $c = 7.487$ (7) Å, $V = 3167.0$ Å^3, $d_{calcd} = 2.03$ g cm^{-3}, $Z = 4$, λ(Mo K$_\alpha$) = 0.7107 Å, μ(Mo K$_\alpha$) = 28.1 cm^{-1}. Data were collected on an Enraf-Nonius CAD-4 diffractometer, by the ω-scan technique. Of the 4645 reflections collected, 2925 independent reflections with $I > 2\sigma(I)$ were used and corrected for decay (17%) and for Lorenz-polarization effects. An absorption correction could not be applied due to the irregular shape of the crystal. A ψ-scan absorption correction was applied, but did not improve the R values; no secondary extinction correction was applied. The structure was solved by direct methods using the program MULTAN and refined by full-matrix least-squares procedures: $R = 0.057$ and $R_w = 0.052$. Hydrogen atom positions were calculated with a C–H distance of 0.95 Å ($B = 4.0$ Å^2) and not refined. All non-hydrogen atoms were refined with anisotropic thermal parameters.

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distortion from a "conventional" Fe_4S_4 core.

A cation of the same stoichiometry and with PF_6^- as counterion has been reported by Vergamini and Kubas.[7] This, however, has been obtained by using a different pathway from ours, namely by *slow* S abstraction, by air oxidation, from $Fe_4S_6Cp_4$ to form $[Fe_4S_5Cp_4]^{2+}$, which is then added to the neutral species $Fe_4S_5Cp_4$ to give the monocation. Moreover, these authors propose, on the basis of NMR studies, a different structure of the Fe_4S_5 core, with the S_2 group bound to only two iron atoms. At the present state of knowledge, one cannot assume whether these two cations do have the same structure or not.

When examined in frozen CH_3CN solutions at 4.2 K and at fairly low concentration (<1 mmol/L), complex I exhibits an EPR rhombic spectrum of $S = {}^1/_2$ type, having principal g values at 2.188, 1.999, and 1.968. The temperature dependence of the spectrum resembles that of Fe_4S_4 sites in both proteins and model compounds,[22] and the signals are totally unobservable above 140 K, indicating a rapid electronic spin relaxation rate. A concentration dependence is also observed: when the cluster concentration is increased up to 10 mmol/L, the signals are broadened, indicating an extensive intercluster magnetic interaction. The Mo(V) anion is evidenced by the presence at low T (4.2–150 K, 5–10 mmol/L) of three signals having $g_x = 1.913$, $g_y = 1.939$, and $g_z = 1.978$ (the latter is best visible at 150 K, when the cluster signals have disappeared). At higher T (190 K) a strong isotropic signal ($S = {}^1/_2$) is visible at $g = 1.942$ ($=(g_x + g_y + g_z)/3$) with $A(^{95,97}Mo) = 44$ G, which is consistent with the presence of a monomeric Mo(V) species.

Preliminary Mössbauer experiments on I in the solid state at 4.2 K, without an external magnetic field, display one doublet with isomer shift (IS) of 0.31–0.35 mm/s (vs. Fe metal at 300 K), quadrupole splitting (QS) of 1.075 mm/s and a line width of 0.40 mm/s. When the temperature is increased, from 1.4 to 77 K, the hyperfine parameters do not change.

These values are intermediate between the parameters corresponding to Fe^{2+} and Fe^{3+} ions with tetrahedral coordination and indicate a charge delocalization in the Fe_4S_5 core. The IS magnitude has been shown[23] to follow a nearly linear correlation with the average oxidation state of the iron atoms. This implies that in our case we have $Fe^{2.75+}$ (3 Fe^{III}, 1 Fe^{II}), although the spectra show no evidence of nonequivalent Fe atoms. The line-width invariance when changing the temperature indicates that, even at $T = 1.4$ K, the Mössbauer spectrum is not magnetically split. This observation confirms that this magnetic cluster, with a magnetic moment of $\mu = 2.62$ at 5 K and in the solid state, has a very short electronic spin relaxation time as compared with the Lazmoz nuclear precession, because of spin–spin interaction between the clusters.

Electron counting arguments are in support of assigning different formal oxidation states to the Fe centers. Assuming each Cp^- contributes six electrons to the metal, each S^{2-} six electrons, and the S_2^{2-} eight electrons, then, to derive an 18-electron configuration at each metal center (71 electrons for $[Fe_4S_5Cp_4]^+$), we assign oxidation states of 3 Fe^{3+} and 1 Fe^{2+}. This observation, joined to the EPR results giving a $g_{av} > 2$, suggests the presence of a $[Fe_4S_5]^{3+}$ type core in compound I, which can therefore be considered as a minimal model for $Hipip_{ox}$.[22]

Detailed magnetic susceptibility and Mössbauer studies are currently under way to gain information about spin–relaxation processes and to try to visualize any magnetic inequivalence of the structurally different Fe(3) site.

Registry No. I, 89016-76-2; $Fe_4S_6Cp_4$, 72256-41-8; $MoOCl_3(thf)_2$, 20529-42-4; Fe, 7439-89-6.

Supplementary Material Available: Tables of all positional and thermal parameters, additional interatomic distances and bond angles, and observed and calculated structure factors for compound I (21 pages). Ordering information is given on any current masthead page.

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Unusual Stabilization of Formylmetal Complexes

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Received November 14, 1983

Summary: The exceptional stabilization of a variety of transient formylmetal complexes can be achieved by the judicious addition of tri-*n*-butyltin hydride and other hydrogen donors. The saturation effect of these additives (illustrated in Figure 1) dictates that alternative pathways involving nonchain decompositions must be identified in addition to the efficient radical-chain mechanism demonstrated in the previous study.

Metal catalysis of carbon monoxide reduction has recently centered around formylmetal complexes as one of the prime intermediates along the reaction pathways.[1] Although a variety of formylmetal species has been generated by the action of various hydridic reducing agents on metal carbonyls of different structural types,[2] their role as intermediates in the catalytic hydrogenations of carbon monoxide has not been established owing to their apparent instabilities. As part of our interest in the reduction of metal carbonyls,[3] we recently noted a highly efficient radical-chain mechanism for the conversion of the formylrhenium complex *cis*-$(OC)_5ReRe(CO)_4CHO^-(Bu_4N)^+$ to the corresponding hydridorhenium complex, i.e.,[4]

$$(OC)_5ReRe(CO)_4C({=}O)H^- \rightarrow (OC)_5ReRe(CO)_4H^- + CO \quad (1)$$

The catalytic turnover number in this system is very high (>400), and the radical chain could be triggered thermally with azo compounds and by photochemical or electro-

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