Reactivity of $Cp'_2Mo_2Co_2S_3(CO)_4$ with Diphosphines: Synthesis of $Cp'_2Mo_2Co_2S_3(\mu_3\text{-}CO)(\eta^2\text{-dmpe})_2$ and X-ray Structure of $[Cp'_2Mo_2Co_2S_3(\mu_3\text{-}CH)(\eta^2\text{-dmpe})_2]Cl$

Owen J. Curnow, M. David Curtis,* and Jeff W. Kampf

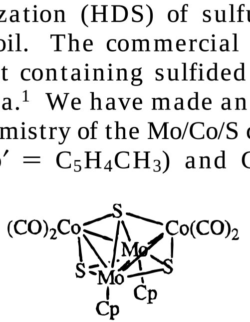
Department of Chemistry, Willard H. Dow Laboratories, The University of Michigan,
Ann Arbor, Michigan 48109-1055

Received December 12, 1996[⊗]

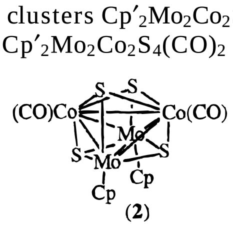
Treatment of the heterobimetallic sulfido cluster $Cp'_2Mo_2Co_2S_3(CO)_4$ (**1**) with the diphosphine compound bis(diphenylphosphino)methane (dppm) or bis(diphenylphosphino)ethane (dppe) gives the carbonyl-substituted adduct $Cp'_2Mo_2Co_2S_3(CO)_2(\eta^2\text{-dppm})$ (**11a**) or $Cp'_2Mo_2Co_2S_3(CO)_2(\eta^2\text{-dppe})$ (**11b**), respectively, whereas treatment of **1** with an excess of bis(dimethylphosphino)ethane (dmpe) gives the cluster $Cp'_2Mo_2Co_2S_3(\mu_3\text{-}CO)(\eta^2\text{-dmpe})_2$ (**12**) containing a triply-bridging carbonyl ligand ($\nu_{CO} = 1615\ cm^{-1}$). Cluster **12** readily reacts with dichloromethane to give the μ_3-methylidyne cluster $[Cp'_2Mo_2Co_2S_3(\mu_3\text{-}CH)(\eta^2\text{-dmpe})_2]^+$ (**14**). Cluster **14** was also characterized by an X-ray crystallographic analysis. Reaction of ^{13}CO-labeled **12** with CH_2Cl_2 and CD_2Cl_2 showed that the methylidyne atoms originate from the dichloromethane solvent. ^{13}CO-labeled **1** was prepared to 96% enrichment and revealed a resonance for the carbonyl ligands in the ^{13}C-NMR spectrum at δ 207 with $w_{1/2} = 940$ Hz. Cluster **11b** was found to desulfurize thiols and perform phosphinidene abstraction reactions via formation of the monomeric cluster $Cp'_2Mo_2Co_2S_4(\eta^2,\mu_2\text{-dppe})$ (**18**) or the oligomeric clusters $[Cp'_2Mo_2Co_2S_3(\mu_3\text{-}PPh)(\eta^2,\mu_2\text{-dppe})]_n$ (**19**), respectively.

Introduction

Research into the chemistry of heterobimetallic sulfido clusters, particularly of the Mo/Co combination, has been driven by its relevance to the catalytic hydrodesulfurization (HDS) of sulfur containing organics in crude oil. The commercial process commonly uses a catalyst containing sulfided Mo and Co supported on alumina.[1] We have made an extensive investigation on the chemistry of the Mo/Co/S clusters $Cp'_2Mo_2Co_2S_3(CO)_4$ (**1**) ($Cp' = C_5H_4CH_3$) and $Cp'_2Mo_2Co_2S_4(CO)_2$ (**2**), as



(**1**)



(**2**)

these clusters show activity similar to the commercial catalyst when supported on alumina[2] and desulfurization of organic sulfur compounds has been observed in homogeneous solution.[3−5]

The reactions between **1** and phosphines were investigated to gain more insight into the interaction of nucleophiles, including thiols, etc., with the cluster.[6−8] The clusters **3** were shown to be key intermediates in the nucleophilic carbonyl substitution reactions of cluster **1** to give clusters of the type $Cp'_2Mo_2Co_2S_3(CO)_3(L)$ (**4**) (Scheme 1).[6] Clusters of the formula $Cp'_2Mo_2Co_2S_3(CO)_4L$ (**3**) have been structurally characterized for L = PMe_3[6,7] and spectroscopically characterized for L = $PPhH_2$,[8] PPh_2H,[8] $P(OMe)_3$,[6] PPh_3,[6] CO,[7] RNC (R = Me, ^{t}Bu),[9] and SR^- (R = aryl).[4] The extent of carbonyl group substitution is dominated by electronic effects, with $PMe_3 < PR_3$ (R = OMe, Ph) < RNC (Scheme 1). Thus, treatment of **1** with PMe_3 reversibly gives a cluster of type **3**, whereas reactions with stronger π-acceptor phosphines proceeded to give the *cis* and *trans* isomers of the disubstituted cluster **8**. A maximum of three CO groups could be displaced with isocyanide ligands to give the trisubstituted cluster **10**. Replacement of all four carbonyl groups on cluster **1** with monodentate ligands has not been possible. This paper describes attempts to prepare a tetrasubstituted cluster by treatment of **1** with chelating diphosphine ligands, the μ_3-carbonyl containing cluster that was isolated in one of these reactions, and its further reaction with dichloromethane to give a cationic μ_3-methylidyne containing cluster.

Results and Discussion

Carbonyl Substitution Reactions. Treatment of cluster **1** with an excess of bis(diphenylphosphino)methane (dppm) or bis(diphenylphosphino)ethane (dppe) gave moderate yields (30−50%) of brown $Cp'_2Mo_2Co_2S_3(CO)_2(\eta^2\text{-dppm})$ (**11a**) or $Cp'_2Mo_2Co_2S_3(CO)_2(\eta^2\text{-dppe})$ (**11b**), respectively, after heating for several hours

---

[⊗] Abstract published in *Advance ACS Abstracts,* April 1, 1997.

[1] (a) Massoth, F. E. *Adv. Catal.* **1978**, *27*, 265. (b) *Proceedings of the 4th Int. Conference on the Chemistry and uses of Molybdenum*; Mitchell, P. C. H., Barry, H. F., Eds.; Reidel Publishing Co.: Dodrecht, Holland, 1983. (c) Ratnassamy, P.; Sivasanker, S. *Catal. Rev.—Sci. Eng.* **1980**, *22*, 401. (d) Prins, R.; De Beer, V. H. J.; Somorjai, G. A. *Catal. Rev.—Sci. Eng.* **1989**, *31*, 1. (e) Grange, P. *Catal. Rev.—Sci. Eng.* **1980**, *21*, 135.

[2] Curtis, M. D.; Penner-Hahn, J. E.; Schwank, J.; Baralt, O.; McCabe, D. J.; Thompson, L.; Waldo, G. *Polyhedron* **1988**, *7*, 2411.

[3] Riaz, U.; Curnow, O. J.; Curtis, M. D. *J. Am. Chem. Soc.* **1994**, *116*, 4357.

[4] (a) Druker, S. H.; Curtis, M. D. *J. Am. Chem. Soc.* **1995**, *117*, 6366. (b) Curtis, M. D.; Druker, S. H. *J. Am. Chem. Soc.* **1997**, *119*, 1027.

[5] Riaz, U.; Curnow, O. J.; Curtis, M. D. *J. Am. Chem. Soc.* **1991**, *113*, 1416.

[6] Curnow, O. J.; Kampf, J. W.; Curtis, M. D.; Shen, J.-K.; Basolo, F. *J. Am. Chem. Soc.* **1994**, *116*, 224.

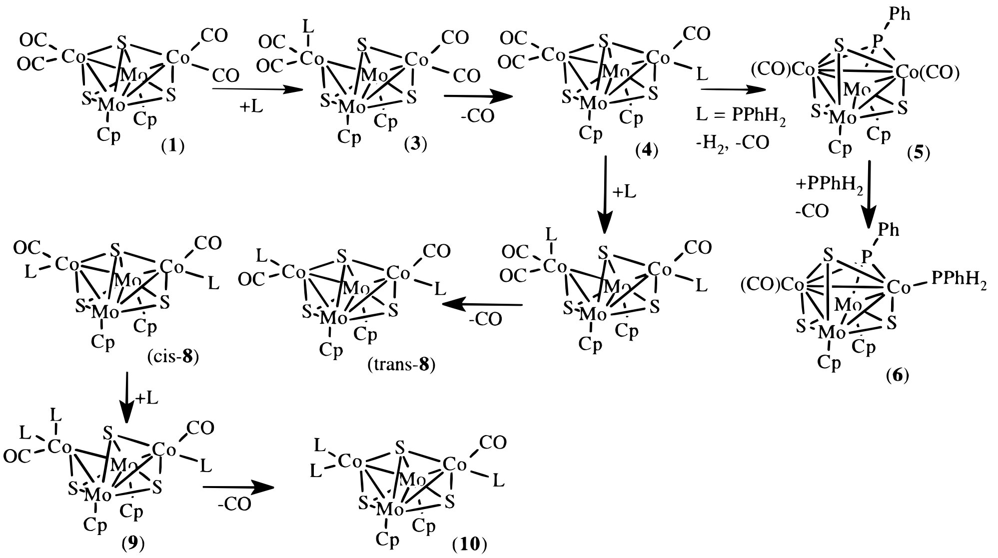
[7] Curnow, O. J.; Kampf, J. W.; Curtis, M. D.; Mueller, B. L. *Organometallics* **1992**, *11*, 1984.

[8] Curnow, O. J.; Kampf, J. W.; Curtis, M. D. *Organometallics* **1991**, *10*, 2546.

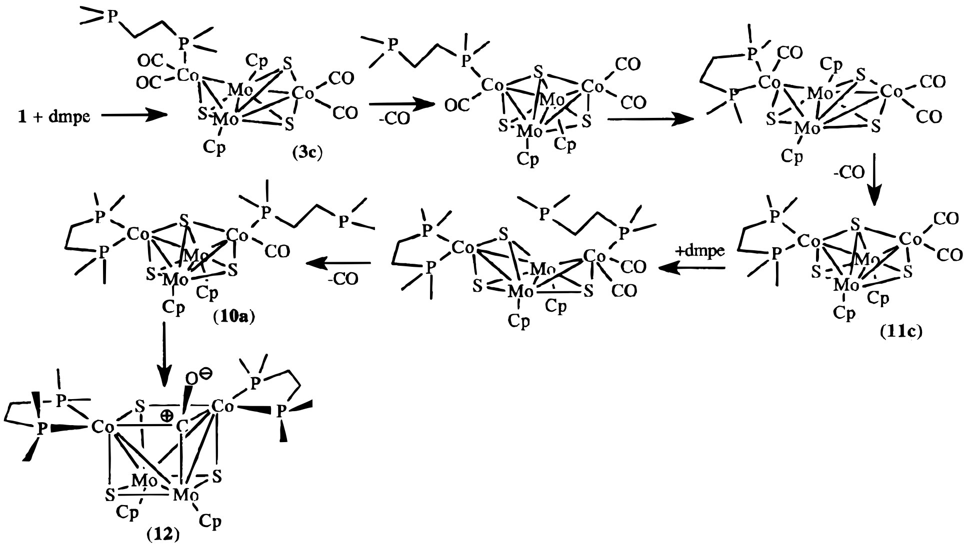
[9] Curtis, M. D.; Curnow, O. J. *Organometallics* **1994**, *13*, 2489.

S0276-7333(96)01045-X CCC: $14.00

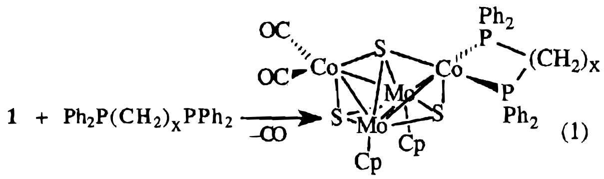
Scheme 1



Scheme 2



followed by chromatography down an alumina column (eq 1). The C_s symmetry of clusters **11a** and **11b**, in

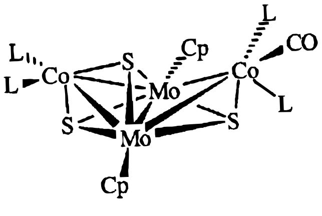


(x = 1, **11a**; x = 2, **11b**)

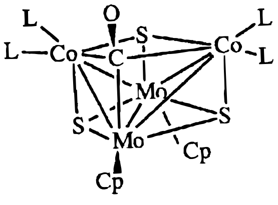
which a mirror plane passes through the Co atoms, was indicated by the ^{1}H-NMR pattern for the cyclopentadienyl ligands (one set of ABCD resonances and one CH$_3$ resonance) and the single phosphorus environment in the ^{31}P-NMR spectrum (δ 85.9 for **11b**). The infrared spectrum showed two peaks (ν_{CO} = 1975 and 1927 cm^{-1}) corresponding to the symmetric and asymmetric stretches. These are the first disubstituted clusters based on **1** in which both CO ligands on the same Co atom are replaced by other ligands.

Addition of an excess of bis(dimethylphosphino)ethane (dmpe) to **1** at ambient temperature in benzene rapidly produced a dark red/brown solution typical of clusters of type **3**, with a characteristic infrared spectrum (ν_{CO} = 1991 (s), 1952 (ms), 1942 (ms), 1922 (m, sh) cm^{-1}) (cluster **3c**, Scheme 2). After 2 h of heating at 45 °C, the solution infrared spectrum showed bands at 1973 (s), 1923 (ms), and 1905 (ms) cm^{-1}. The first two bands are attributed to the cluster Cp′$_2$Mo$_2$Co$_2$S$_3$(CO)$_2$(η^2-dmpe) (**11c**), by analogy with the infrared spectra of **11a** and **11b**, while the third peak is assigned to Cp′$_2$Mo$_2$Co$_2$S$_3$(CO)(η^1-dmpe)(η^2-dmpe) (**10a**) based on a comparison with the triisocyanide clusters of type **10**, in which ν_{CO} is observed at ca. 1918 cm^{-1}. Heating the solution to reflux overnight gave a dark green solution, from which the cluster Cp′$_2$Mo$_2$Co$_2$S$_3$(μ_3-CO)(η^2-dmpe)$_2$ (**12**), containing a triply-bridging carbonyl ligand, was isolated in 85% yield. The C_s symmetry of cluster **12**, in which a mirror plane passes through the two Mo atoms, was confirmed by the two A$_2$B$_2$ patterns for the cyclopentadienyl ring protons and two methyl resonances in the ^{1}H-NMR spectrum. The ^{13}C-NMR spectrum was also consistent with this structural assignment. The ^{31}P-NMR spectrum showed two broad resonances at δ 58.0 ($w_{1/2}$ = 240 Hz) and 50.1 ($w_{1/2}$ = 160 Hz). The peaks are broadened by coupling to the quadrupolar Co nuclei. A ν_{CO} band attributed to a triply-bridging carbonyl appears at 1615 cm^{-1} in the infrared spectrum. Confirmation of this assignment was provided by a shift of this band to 1577 cm^{-1} for the ^{13}CO analog and the observation of a broad resonance in the ^{13}C-NMR spectrum at δ 223 ($w_{1/2} = 28$ Hz). The composition of **12** was also confirmed by the FAB-MS, for which the isotope pattern for the parent ion was in excellent agreement with the calculated pattern.

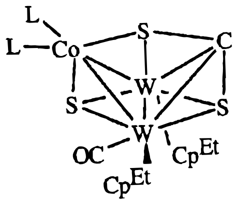
Cluster **12** is the first example of a third structural type (**B**) for the class of compounds of the general formula $Cp'_2M_2Co_2S_3(CO)L_4$, where M = Mo or W and L is a neutral two-electron-donor ligand. For M = Mo and L = π-acceptor ligand e.g., CO, structural type **A** is observed, in which the metals adopt an open butterfly arrangement and no ligands or atoms bridge the two hinge Co atoms. A second structural type (**C**) was recently reported for M = W and L = CO, $Cp^{Et}{}_2W_2Co_2S_3(CO)_5$ ($Cp^{Et} = C_5Me_4Et$, **13**), in which the butterfly is partially closed (one sulfur atom bridges the two hinge Co atoms) and one carbonyl ligand is terminally bound to a W atom.[10] This structure is presumably adopted because the greater π basicity of $(C_5Me_4Et)W$ *vs* $(C_5H_4Me)Mo$ makes coordination of the π-acidic CO ligand to the W atom more favorable than to a Co atom, despite the steric bulk of the Cp^{Et} ligand. In **12**, however, the significantly stronger σ-donor properties of the dmpe ligands compared to those of CO increases the electron density on the Co atoms to the point that Co−(μ_3-CO) bonding becomes important in draining excess charge from the Co atoms. Cluster **12** has a 62-electron closed butterfly or "cubane" type structure with a sulfur atom and a CO ligand bridging the two hinge Co atoms. On the basis of a cluster electron count of 62, no Co−Co bond is expected.



(A)

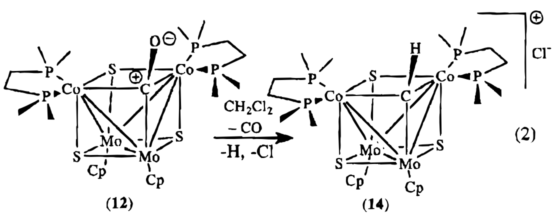


(B)



(C)

Reaction of $Cp'_2Mo_2Co_2S_3(\mu_3\text{-}CO)(\eta^2\text{-dmpe})_2$ (12) with CH_2Cl_2. When cluster **12** was refluxed in dichloromethane overnight, a brown/black solution was obtained, from which the cationic cluster $[Cp'_2Mo_2Co_2S_3(\mu_3\text{-}CH)(\eta^2\text{-dmpe})_2]^+$ (**14**) was obtained in moderate yield (~50%) with chloride as the counterion (eq 2). As with cluster **12**, cluster **14** exhibits C_s symmetry with the mirror plane through the Mo atoms (two A_2B_2 patterns for the cyclopentadienyl ring protons and two methyl resonances in the ^1H-NMR spectrum). The triply-bridging methylidyne proton appeared as a deshielded triplet at δ 8.60 with coupling to two of the phosphorus atoms ($^3J_{PH} = 19$ Hz). This coupling was assigned to the *cis* phosphorus atoms, for which the P−Co−C−H dihedral angles average 3.5°, rather than to the *trans* phosphorus atoms, for which the P−Co−C−H dihedral angles average 51.3°. $^3J_{PH}$ values typically approach zero as the dihedral angle nears 90° and maximizes near 0° and 180°.[11] The composition of **14** was also confirmed by FAB-MS.



(12) (14) (2)

In order to trace the origin of the methylidyne C and H atoms, cluster **12** was prepared with a ^{13}C-labeled carbonyl ligand and treated with CH_2Cl_2. This gave unlabeled cluster **14** as the product (the resonance for the methylidyne proton in the ^1H-NMR spectrum was unchanged). Treatment of labeled **12** with CD_2Cl_2 gave a ^1H-NMR spectrum with no methylidyne proton resonance. Thus, it can be concluded that the C and H atoms of the methylidyne group originated from the dichloromethane solvent. Although tricobalt−methylidyne clusters have been isolated from the reaction of $Co_2(CO)_8$ with $CHBr_3$,[12] this appears to be the first example of formation of a methylidyne cluster from CH_2Cl_2. The mechanism for the formation of **14** is not known.

X-ray Structure of $[Cp'_2Mo_2Co_2S_3(\mu_3\text{-}CH)(\eta^2\text{-dmpe})_2]Cl\cdot 2C_6H_6$ ($14\cdot 2C_6H_6$). The structure of **14** was confirmed by an X-ray crystallographic analysis. Selected bond distances and angles are given in Tables 1 and 2. The solid state structure contains one unique cluster cation, one chloride anion, and two molecules of benzene in the unit cell. Figure 1 shows a thermal ellipsoid plot of the cluster cation with the atomic labeling scheme as well as the cluster core. The benzene molecules do not enter into any significant interactions with either the cluster cation or chloride anion. The chloride anion's closest contacts are nonbonding to the cluster via the dmpe and Cp' ligands (Cl−C = 3.65(2) and 3.66(2) Å for the dmpe methyl groups C21 and C17, respectively, and 3.69(2) Å for the Cp' ring carbon atom C9). The Co−Co distance of 3.147(3) Å is too long for a significant bonding interaction.[13] A sulfur atom caps each of the $CoMo_2$ triangular faces under the wings, while a third sulfur atom bridges the two Co atoms and one of the Mo atoms and the methylidyne carbon atom bridges both Co atoms and the other Mo atom. Additionally, each Co atom has a chelating dmpe ligand and each Mo atom has an η^5-methylcyclopentadienyl ligand.

With the exception of the Co−Co distance, the cluster core of **14** is remarkably similar to that of the cubane

---

[10] Mansour, M. A.; Curtis, M. D.; Kampf, J. W. *Organometallics* **1997**, *16*, 275.

[11] *Phosphorus-31 NMR Spectroscopy in Stereochemical Analysis*; Verkade, J. G., Quin, L. D., Eds.; VCH Publishers, Inc.: Deerfield Beach, FL, 1987.

[12] Seyferth, D.; Hallgren, J. E.; Hung, P. L. K. *J. Organomet. Chem.* **1973**, *50*, 265.

[13] Curtis, M. D.; Riaz, U.; Curnow, O. J.; Kampf, J. W.; Rheingold, A. L.; Haggerty, B. S. *Organometallics* **1995**, *14*, 5337.

Table 1. Selected Bond Lengths (Å) for $[Cp'_2Mo_2Co_2S_3(\mu_3\text{-}CH)(\eta^2\text{-dmpe})_2]Cl\cdot 2C_6H_6$ (14·$2C_6H_6$)

Mo1−Cp1[a]	2.061(3)	Mo2−Cp2[a]	2.041(3)
Mo1−Mo2	2.784(2)	Mo2−Co1	2.722(3)
Mo1−Co1	2.713(3)	Mo2−Co2	2.703(2)
Mo1−Co2	2.730(2)	Mo2−C25	1.97(1)
Mo1−S1	2.331(3)	Mo2−S2	2.309(4)
Mo1−S2	2.344(4)	Mo2−S3	2.317(4)
Mo1−S3	2.352(5)	Co2−S1	2.224(5)
Co1−S1	2.222(4)	Co2−S3	2.190(4)
Co1−S2	2.176(2)	Co2−C25	2.02(1)
Co1−C25	2.12(1)	Co2−P3	2.155(5)
Co1−P1	2.213(5)	Co2−P4	2.217(4)
Co1−P2	2.192(5)		

[a] Cp1 = centroid of C1−C5, Cp2 = centroid of C7−C11.

Table 2. Selected Bond Angles (deg) for $[Cp'_2Mo_2Co_2S_3(\mu_3\text{-}CH)(\eta^2\text{-dmpe})_2]Cl\cdot 2C_6H_6$ (14·$2C_6H_6$)

Mo1−Mo2−Co1	59.04(6)	Co1−Mo1−Co2	70.66(7)
Mo1−Mo2−Co2	59.64(6)	Co1−Mo1−S1	51.6(1)
Mo1−Mo2−S2	53.8(1)	Co1−Mo1−S2	50.34(9)
Mo1−Mo2−S3	54.0(1)	Co1−Mo1−S3	105.2(1)
Mo1−Mo2−C25	88.3(4)	Co1−Mo2−Co2	70.92(6)
Mo1−Co1−Mo2	61.62(6)	Co1−Mo2−S2	50.46(9)
Mo1−Co1−S2	55.3(1)	Co1−Mo2−S3	106.0(1)
Mo1−Co1−S2	56.0(1)	Co1−Mo2−C25	50.7(4)
Mo1−Co1−P1	101.2(1)	Co1−S1−Co2	90.1(1)
Mo1−Co1−P2	173.1(1)	Co1−C25−Co2	98.8(6)
Mo1−Co1−C25	87.3(4)	Co2−Mo1−S1	51.4(1)
Mo1−Co2−Mo	61.64(5)	Co2−Mo1−S2	104.4(1)
Mo1−Co2−S1	54.99(9)	Co2−Mo1−S3	50.4(1)
Mo1−Co2−S3	55.8(1)	Co2−Mo2−S2	106.2(1)
Mo1−Co2−P3	170.8(2)	Co2−Mo2−S3	51.0(1)
Mo1−Co2−P4	102.6(1)	Co2−Mo2−C25	48.2(4)
Mo1−Co2−C25	88.8(3)	S1−Mo1−S2	101.8(1)
Mo1−S1−Co1	73.11(9)	S1−Mo1−S3	101.6(1)
Mo1−S1−Co2	73.6(1)	S1−Co1−S2	111.1(2)
Mo1−S2−Mo2	73.5(1)	S1−Co1−P1	91.9(2)
Mo1−S2−Co1	73.7(1)	S1−Co1−P2	128.3(1)
Mo1−S3−Mo2	73.2(1)	S1−Co1−C25	80.1(3)
Mo1−S3−Co2	73.8(1)	S1−Co2−S3	110.6(2)
Mo2−Mo1−Co1	59.34(6)	S1−Co2−P3	121.7(2)
Mo2−Mo1−Co2	58.72(5)	S1−Co2−P4	94.6(2)
Mo2−Mo1−S1	92.1(1)	S1−Co2−C25	82.2(4)
Mo2−Mo1−S2	52.7(1)	S2−Mo1−S3	101.2(2)
Mo2−Mo1−S3	52.8(1)	S2−Mo2−S3	103.4(2)
Mo2−Co1−S1	95.2(1)	S2−Mo2−C25	101.1(4)
Mo2−Co1−S2	54.9(1)	S2−Co1−P1	96.7(2)
Mo2−Co1−P1	151.4(1)	S2−Co1−P2	120.5(1)
Mo2−Co1−P2	111.5(1)	S2−Co1−C25	100.8(3)
Mo2−Co1−C25	46.0(3)	S3−Mo2−C25	99.1(4)
Mo2−Co2−S1	95.68(9)	C3−Co2−P3	127.5(2)
Mo2−Co2−S3	55.3(1)	S3−Co2−P4	94.4(2)
Mo2−Co2−P3	112.1(1)	S3−Co2−C25	101.8(3)
Mo2−Co2−P4	149.7(1)	P1−Co1−P2	84.9(2)
Mo2−Co2−C25	46.6(3)	P1−Co1−C25	162.3(3)
Mo2−S2−Co1	74.7(1)	P2−Co1−C25	87.8(4)
Mo2−S3−Co2	73.7(1)	P3−Co2−P4	85.9(2)
Mo2−C25−Co1	83.3(4)	P4−Co2−C25	163.6(3)
Mo2−C25−Co2	85.2(6)		

clusters $[Cp'_2Mo_2Co_2S_4(CO)_2]^-$ (**2**$^-$)[14] and $Cp'_2Mo_2Co_2S_4(CO)_2$ (**2**)[13] which have one and two fewer cluster core electrons, respectively: For all three clusters, the Mo−S distances average 2.33(2) Å, while the Co−Mo distances average 2.72(1), 2.74(1), and 2.70(5) Å, respectively, and there are two sets of Co−S distances which average 2.223(1), 2.242(7), and 2.214(6), and 2.18(1), 2.172(4), and 2.175(3) Å, respectively (the shorter distances are to the $CoMo_2$ bridging S atoms). The similar distances in **14**, **2**, and **2**$^-$ seems quite surprising given the differing cluster core oxidation states (+12 versus +11 and +10, respectively), Co coordination geometry and ligand type (a bidentate dmpe ligand with no Co−Co bond versus one CO ligand and a Co−Co bond), and the replacement of a bridging sulfido atom by a bridging methylidyne group.

The Co−C25 bond distances are significantly asymmetric: Co1−C25 = 2.12(1) Å and Co2−C25 = 2.02(1) Å. Each of these distances is longer than the Mo−C25 bond length, 1.97(1) Å, and when corrected for the difference in the covalent radii (ca. 0.15 Å in these clusters[13,15]), the Co−C distances are considerably longer than expected for a Co−C single bond. These bonds are probably elongated as a result of the large Co−Co distance. In the cluster $Co_3(CO)_9(\mu_3\text{-}CH)$,[16] the Co−Co distances are typical of Co−Co bonds (2.48 *vs* 3.15 Å in **14**), and the Co−C distances are significantly shorter at an average of 1.895 Å. The Mo−C25 distance is 0.1 Å shorter than that found in $Cp_3Mo_3(CO)_6(\mu_3\text{-}CH)$ (2.07 Å)[17] and approaches the distances found in some compounds with Mo=C double bonds, e.g., 1.98 Å in $Cp_2Mo_2(CO)_3(CAr_2)(N_2CAr_2)$.[18] These distances suggest that the μ_3-methylidyne group is behaving more like a terminal Mo−methylidyne that is weakly interacting with the Co atoms. The Co−S bonds, on the other hand, do not appear to be affected at all, apparently the larger S atoms are able to accommodate the increase in the Co−Co distance. There is likewise little difference in the bond distances to Mo1 as compared to Mo2.

^{13}CO-Enriched $Cp'_2Mo_2Co_2S_3(CO)_4$ (1). The ^{13}CO-labeled cluster **1** was prepared by exchanging the carbonyl ligands with labeled CO, as described in the Experimental Section. The extent of ^{13}CO enrichment was determined to be 96% by fitting the calculated and observed intensities of the nine most intense peaks of the parent ion in the EI mass spectrum (Figure 2). Spectral subtraction of the infrared spectra of the partially- and nonenriched clusters allowed the determination of the infrared spectrum of the monolabeled cluster $Cp'_2Mo_2Co_2S_3(^{12}CO)_3(^{13}CO)$. The four bands at ν_{CO} = 2003 (m), 1979 (s), 1953 (m), and 1921 (m) cm^{-1} in the monolabeled compound correspond to the A_1, B_1, B_2, and A_2 modes, respectively, for the unlabeled C_{2v} symmetric species. The A_2 mode is not observed in the unlabeled compound, but the reduction in symmetry in the monosubstituted cluster makes this band infrared active. The spectrum of the 96%-labeled material showed four infrared bands at ν_{CO} = 1986 (w), 1963 (m), 1942 (s), and 1910 (ms) cm^{-1}. The first weak band is assigned to the symmetric A_1 stretch of the trisubstituted cluster $Cp'_2Mo_2Co_2S_3(^{12}CO)(^{13}CO)_3$, with the remaining three bands (B_1, B_2, and A_2) being buried under the strong bands of the fully labeled cluster. Hence, the next three bands are assigned to the A_1, B_1, and B_2 stretches of the fully labeled cluster $Cp'_2Mo_2Co_2S_3(^{13}CO)_4$. A comparison of the differences between the bands of the unlabeled cluster and the fully labeled cluster confirms these assignments: for $Cp'_2Mo_2Co_2S_3(^{12}CO)_4$, 2009 ($A_1$) − 21 = 1988 cm^{-1} ($B_1$) and 1988 ($B_1$)

---

(14) Curtis, M. D.; Druker, S. H.; Goossen, L.; Kampf, J. W. *Organometallics* **1997**, *16*, 231−235.

(15) Curtis, M. D.; Williams, P. D.; Butler, W. M. *Inorg. Chem.* **1988**, *27*, 2853.

(16) Leung, P.; Coppens, P.; McMullan, R. K.; Koetzle, T. F. *Acta Crystallogr., Sect. B* **1981**, *37*, 1347.

(17) Akita, M.; Noda, K.; Moro-oka, Y. *Organometallics* **1994**, *13*, 4145.

(18) Messerle, L.; Curtis, M. D. *J. Am. Chem. Soc.* **1982**, *104*, 889.

(19) Brunner, H.; Wachter, J. *J. Organomet. Chem.* **1982**, *240*, C41.

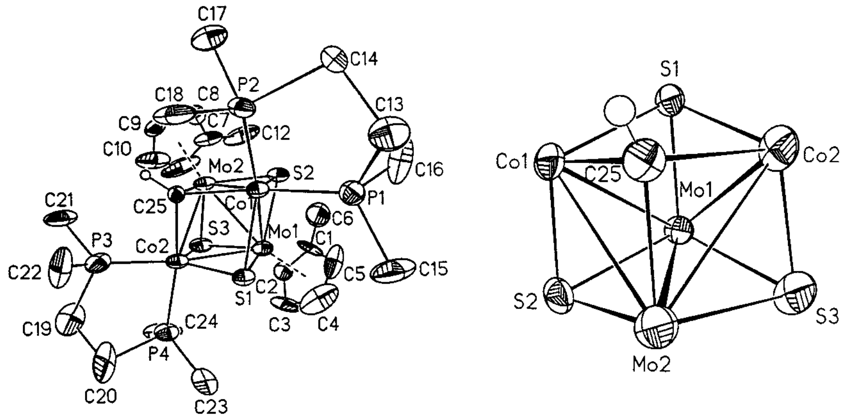


Figure 1. (left) Thermal ellipsoid plot of $[Cp'_2Mo_2Co_2S_3(\mu_3\text{-}CH)(\eta^2\text{-dmpe})_2]^+$ (**14**) showing the atom labeling scheme. (right) Thermal ellipsoid plot of the cluster core.

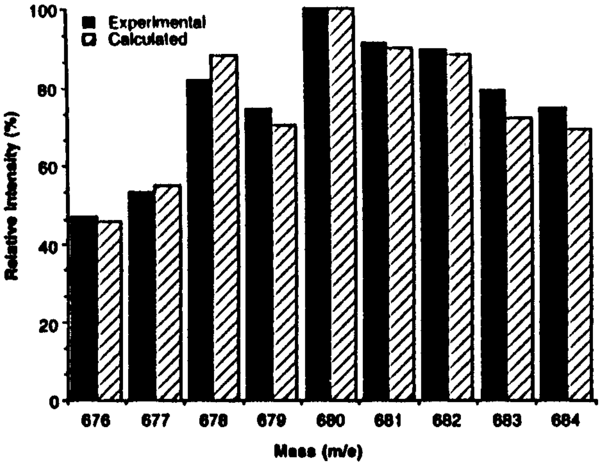


Figure 2. Calculated (96% enrichment) and experimental isotope patterns for the nine most intense peaks of the parent ion of the ^{13}CO-enriched cluster **1**.

$- 34 = 1954$ cm^{-1} (B_2); whereas for $Cp'_2Mo_2Co_2S_3(^{13}CO)_4$, 1963 ($A_1$) $- 21 = 1942$ cm^{-1} (B_1) and 1942 (B_1) $- 32 = 1910$ cm^{-1} (B_2).

The preparation of ^{13}CO-enriched **1** allowed, for the first time, one to observe the ^{13}C resonance of the carbonyl ligands of **1** in the ^{13}C-NMR spectrum. This was found at δ 207 with a half-width of 930 Hz. The breadth of this resonance, attributed to the highly anisotropic environment of the carbonyl ligands in the presence of the quadrupolar Co nuclei, accounts for our earlier difficulties in observing it. The ^{31}P resonances of the dmpe ligands in **12** were similarly broad (240 and 160 Hz). In contrast, the μ_3-CO ligand, which is in a much more symmetric environment, has a relatively narrow half-width of 28 Hz for the ^{13}C resonance.

Desulfurization and Phosphinidene-Abstraction Reactions. The diphosphine-substituted clusters were originally synthesized in the hope that a chelating ligand in the Co atoms would stabilize one or more intermediates in the cluster-mediated, thiol desulfurization reactions described in the Introduction. Thus, we have investigated the reactions of cluster **11a** with $(CH_3)_2CHCH_2CH_2SH$ in refluxing toluene (Scheme 3). Initially, a product with one ν_{CO} at 1955 cm^{-1} was formed. This intermediate is believed to be $Cp'_2Mo_2Co_2S_4(CO)(\eta^1\text{-dppe})$ (**15**) since $Cp'_2Mo_2Co_2S_4(CO)(^tBuNC)$ has a single carbonyl stretch at 1953 cm^{-1}. The final product shows no carbonyl stretches in the infrared spectrum, while the NMR spectrum shows one A_2B_2 pattern for the Cp′ ring protons (δ 5.58 and 5.25), one singlet for the Cp′ methyl protons (δ 2.01), and one broad multiplet for the CH_2CH_2 protons at δ 4.22 (twisting of the six-membered ring and coupling to the P atoms may account for the broadening). This suggests the C_{2v} symmetric cluster $Cp'_2Mo_2Co_2S_4(\eta^2,\mu_2\text{-dppe})$ (**18**) in which the dppe ligand bridges both Co atoms. The reaction of the cubane cluster **2** with dppe at low temperature, followed by slow warming to room temperature, gave the same intermediate species **15** ($\nu_{CO} = 1955$ cm^{-1}). However, the final product showed several singlets in the $CpCH_3$ region suggesting that intermolecular substitution by the free phosphine may be occurring to form oligomeric species (**17**).

The reaction of **11a** with $PPhH_2$ followed a similar route as the reaction with $(CH_3)_2CHCH_2CH_2SH$; a product with one ν_{CO} at 1940 cm^{-1} was formed initially. This product was found to be stable enough to obtain a ^1H-NMR spectrum that is very similar to that of $Cp'_2Mo_2Co_2S_3(\mu_3\text{-PPh})(CO)(PPhH_2)$;[8] it consists of two ABCD patterns for the cyclopentadienyl ring protons and two resonances for the methyl protons. Thus, the product probably has the structure $Cp'_2Mo_2Co_2S_3(\mu_3\text{-PPh})(CO)(\eta^1\text{-dppe})$ (**16**), with only one of the phosphorus atoms coordinated to the cluster. Upon workup, **16** reacted further and the final product showed a complex ^1H-NMR spectrum probably due to the formation of oligomers (**19**). The steric bulk of the phosphinidene phenyl group presumably disfavors a dppe ligand bridging across the cluster.

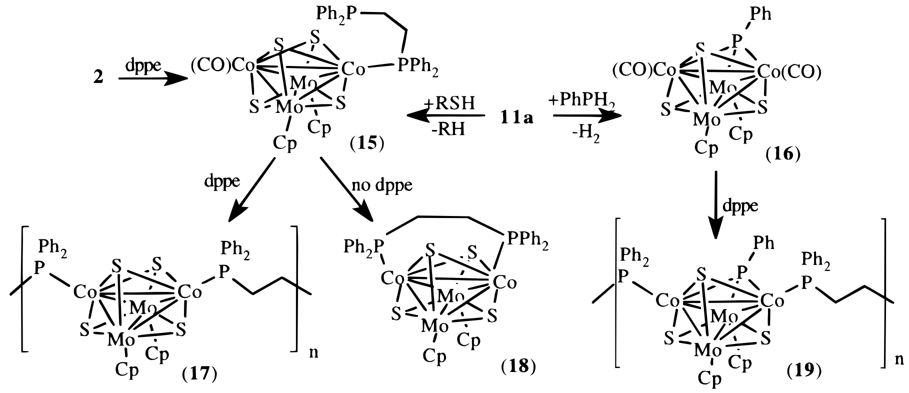
Conclusions

The strong σ-donor and chelating properties of dmpe have been shown to give rise to a new structural type of cluster for the class of compounds which have the formula $Cp'_2Mo_2Co_2S_3(CO)(L)_4$. This new structural type contains a carbonyl ligand triply-bridging an open-sided $MoCo_2$ metal triangle. This μ_3-carbonyl cluster underwent an unexpected and facile reaction with dichloromethane, via loss of the carbonyl ligand, to form a cationic cluster containing a μ_3-methylidyne ligand that also spans an open-sided $MoCo_2$ metal triangle. Determination of the mechanism of this reaction awaits a more detailed investigation.

Experimental Section

General Considerations. All manipulations and reactions were carried out under a nitrogen atmosphere by use of

Scheme 3



standard Schlenk-line techniques. Reagent grade solvents were dried and distilled prior to use: Toluene and benzene from Na/benzophenone; dichloromethane and hexane from CaH$_2$. Cp$'_2$Mo$_2$Co$_2$S$_3$(CO)$_4$[14] and Cp$'_2$Mo$_2$Co$_2$S$_4$(CO)$_2$[19] were prepared by published procedures. All other reagents were purchased from Aldrich Chemical Co. ^{1}H- and ^{13}C-NMR data were collected on a Bruker AM-300 or Bruker WM-360 spectrometer. ^{31}P-NMR data were collected on a General Electric GN-500 NB spectrometer. Infrared spectra were obtained on a Nicolet 5-DXB spectrometer. Mass spectra were collected on a Finnegan 4021 quadrupole mass spectrometer. Elemental analyses were done by Galbraith Laboratories Inc.

Preparation of Cp$'_2$Mo$_2$Co$_2$S$_3$(CO)$_2$(η^2-dppe) (11a). Cluster **1** (0.733 g, 1.08 mmol) and dppe (0.746 g, 1.87 mmol) were dissolved in 50 mL of CH$_2$Cl$_2$ and refluxed for 14 h. Elution down a 25 cm column of alumina with 1:1 CH$_2$Cl$_2$/hexane yielded 0.52 g of brown product **11a** (47% yield). ^{1}H NMR (C$_6$D$_6$): δ 7.79 (m, 4H, Ph*H*), 7.66 (m, 4H, Ph*H*), 7.0 (m, 12H, Ph*H*), 5.47 (m, 2H), 4.87 (m, 2H), 4.72 (m, 2H), 4.57 (m, 2H, ABCD pattern for Cp*H*), 2.13 (d, J_{PH} = 18 Hz, 4H, C*H*$_2$C*H*$_2$), 1.84 (s, 6H, CpC*H*$_3$). ^{31}P NMR (C$_6$D$_6$): δ 85.9. IR (benzene): ν_{CO} 1975 (s), 1927 (s) cm^{-1}. Anal. Calcd for C$_{40}$H$_{38}$Co$_2$Mo$_2$O$_2$P$_2$S$_3$: C, 47.17; H, 3.76. Found: C, 47.15; H, 3.30.

Preparation of Cp$'_2$Mo$_2$Co$_2$S$_3$(CO)$_2$(η^2-dppm) (11b). Cluster **1** (0.098 g, 0.145 mmol) and dppm (0.111 g, 0.290 mmol) were dissolved in 40 mL of benzene and refluxed for 5 h. Elution down a 30 cm column of alumina with 1:1 CH$_2$Cl$_2$/hexane yielded 0.05 g of brown product **11b** (34% yield). ^{1}H NMR (C$_6$D$_6$): δ 7.6 (m, 8H, Ph*H*), 6.9 (m, 12H, Ph*H*), 5.33 (m, 2H), 5.00 (m, 2H), 4.96 (m, 2H), 4.67 (m, 2H, ABCD pattern for Cp*H*), 4.44 (t, J_{PH} = 9.5 Hz, 2H, C*H*$_2$), 1.80 (s, 6H, CpC*H*$_3$). IR (benzene): ν_{CO} 1975 (s), 1927 (s) cm^{-1}. Anal. Calcd for C$_{39}$H$_{36}$Co$_2$Mo$_2$O$_2$P$_2$S$_3$: C, 46.63; H, 3.61. Found: C, 46.69; H, 3.90.

Preparation of Cp$'_2$Mo$_2$Co$_2$S$_3$(μ_3-CO)(η^2-dmpe)$_2$ (12). Cluster **1** (0.875 g, 1.29 mmol) was dissolved in 30 mL of benzene, and dmpe (0.6 mL, 3.6 mmol) was added with stirring to give a dark red/brown solution of Cp$'_2$Mo$_2$Co$_2$S$_3$(CO)$_4$(η^1-dmpe) (ν_{CO} = 1991 (s), 1952 (ms), 1942 (ms), 1922 (m, sh) cm^{-1}). After 2 h at 45 °C, the solution infrared spectrum shows bands at 1973 (s) and 1923 (ms) cm^{-1} attributed to Cp$'_2$Mo$_2$Co$_2$S$_3$(CO)$_2$(η^2-dmpe) and at 1905 (ms) cm^{-1} which may be due to Cp$'_2$Mo$_2$Co$_2$S$_3$(η^1-dmpe)(CO)(η^2-dmpe). The solution was allowed to reflux overnight to give a dark green solution of **12** containing no stretches in the terminal ν_{CO} region. A dark green powder of **12** was obtained by recrystallization from benzene/hexane (0.98 g, 85% yield). ^{1}H NMR (C$_6$D$_6$): δ 5.31 (2H), 5.18 (2H), 4.87 (2H), 4.81 (2H, 2A$_2$B$_2$ pattern for Cp*H*), 2.62 (s, 3H, CpC*H*$_3$), 2.30 (s, 3H, CpC*H*$_3$), 2.05 (d, $^2J_{PH}$ = 7.7 Hz, 6H, PC*H*$_3$), 1.13 (d, $^2J_{PH}$ = 6.4 Hz, 12H, PC*H*$_3$), 0.65 (d, $^2J_{PH}$ = 7.7 Hz, 6H, PC*H*$_3$). ^{31}P{^{1}H} NMR (C$_6$D$_6$): δ 58.0 ($w_{1/2}$ = 240 Hz), 50.1 ($w_{1/2}$ = 160 Hz). ^{13}C{^{1}H} NMR (C$_6$D$_6$): δ 92.13 (s), 91.39 (s), 91.36 (s), 90.93 (s, tertiary *C*$_5$H$_4$CH$_3$), 32.39 (dd, J_{PC} = 20 and 27 Hz, P$-$*C*H$_2$$-$), 31.67 (dd, J_{PC} = 18 and 28 Hz, P$-$*C*H$_2$$-$), 21.21 (d, $^1J_{PC}$ = 11.5 Hz), 20.00 (d, $^1J_{PC}$ = 16.0 Hz), 17.50 (d, $^1J_{PC}$ = 17.0 Hz), 12.67 (d, $^1J_{PC}$ = 16.5 Hz), 16.92 (s, Cp*C*H$_3$), 17.71 (s, Cp*C*H$_3$). IR (benzene): ν_{CO} 1615 cm^{-1}. FAB-MS: Parent ion peak at 892 *m/e*. Anal. Calcd for C$_{25}$H$_{46}$Co$_2$Mo$_2$OP$_4$S$_3$: C, 33.65; H, 5.20. Found: C, 32.98; H, 5.45.

Preparation of Cp$'_2$Mo$_2$Co$_2$S$_3$(μ_3-^{13}CO)(dmpe)$_2$. This was prepared in a similar way to unlabeled Cp$'_2$Mo$_2$Co$_2$S$_3$(μ_3-CO)(η^2-dmpe)$_2$ (**9**), but starting from Cp$'_2$Mo$_2$Co$_2$S$_3$(^{13}CO)$_4$. ^{13}C-{^{1}H} NMR (C$_6$D$_6$): δ 223 ($w_{1/2}$ = 28 Hz, μ_3-^{13}*C*O). IR (benzene): ν_{CO} 1577 cm^{-1}.

Preparation of [Cp$'_2$Mo$_2$Co$_2$S$_3$(μ_3-CH)(η^2-dmpe)$_2$]Cl (14). Cluster **12** (0.080 g, 0.089 mmol) was dissolved in CH$_2$Cl$_2$ (35 mL) and heated to reflux for 24 h to give a brown/black solution. The solvent was removed, and the residue was washed with toluene. Recrystallization by slow evaporation of a CH$_2$Cl$_2$/toluene solution gave a black solid of **14** (0.05 g, 61% yield). ^{1}H NMR (CD$_3$OD): δ 8.60 (t, J_{PH} = 19 Hz, 1H, C*H*), 5.39 (2H), 5.21 (2H), 5.05 (2H), 4.96 (2H, 2A$_2$B$_2$ pattern for Cp*H*), 2.29 (s, 3H, CpC*H*$_3$), 2.25 (s, 3H, CpC*H*$_3$), 2.04 (d, $^2J_{PH}$ = 8.8 Hz, 6H, PC*H*$_3$), 1.44 (d, $^2J_{PH}$ = 8.7 Hz, 6H, PC*H*$_3$), 1.02 (d, $^2J_{PH}$ = 9.5 Hz, 6H, PC*H*$_3$), 0.79 (d, $^2J_{PH}$ = 9.6 Hz, 6H, PC*H*$_3$), 2.13 (m, 2H, CH$_2$), 1.72 (m, 2H, CH$_2$), 1.58 (m, 4H, CH$_2$). FAB-MS: Parent ion peak at 877 *m/e*. Anal. Calcd for C$_{25}$H$_{47}$ClCo$_2$Mo$_2$P$_4$S$_3$: C, 32.89; H, 5.19. Found: C, 32.75; H, 5.42.

Reaction of Cp$'_2$Mo$_2$Co$_2$S$_3$(μ_3-^{13}CO)(η^2-dmpe)$_2$ with CH$_2$Cl$_2$. Cp$'_2$Mo$_2$Co$_2$S$_3$(μ_3-^{13}CO)(η^2-dmpe)$_2$ was dissolved in CH$_2$Cl$_2$ (35 mL) and heated to reflux for 24 h to give a brown/black solution. The solvent was then removed, and an NMR spectrum was taken in CD$_3$OD, which showed the product to be **14** with an unlabeled methylidyne carbon. There was also a broad resonance at $-$4.4 ppm ($w_{1/2}$ ~ 330 Hz).

Reaction of Cp$'_2$Mo$_2$Co$_2$S$_3$(μ_3-^{13}CO)(η^2-dmpe)$_2$ with CD$_2$Cl$_2$. Cp$'_2$Mo$_2$Co$_2$S$_3$(μ_3-^{13}CO)(η^2-dmpe)$_2$ (~10 mg) was dissolved in CD$_2$Cl$_2$ in an NMR tube. The initial spectrum shows [Cp$'_2$Mo$_2$Co$_2$S$_3$(μ_3-CD)(η^2-dmpe)$_2$]$^+$. ^{1}H-NMR (CD$_2$Cl$_2$): δ 5.33 (2H), 5.17 (2H), 4.99 (2H), 4.87 (2H, 2A$_2$B$_2$ pattern for Cp*H*), 2.27 (s, 3H, CpC*H*$_3$), 2.24 (s, 3H, CpC*H*$_3$), 2.04 (d, $^2J_{PH}$ = 8.7 Hz, 6H, PC*H*$_3$), 1.42 (d, $^2J_{PH}$ = 8.6 Hz, 6H, PC*H*$_3$), 0.99 (d, $^2J_{PH}$ = 9.4 Hz, 6H, PC*H*$_3$), 0.77 (d, $^2J_{PH}$ = 9.6 Hz, 6H, PC*H*$_3$).

Preparation of Cp$'_2$Mo$_2$Co$_2$S$_3$(^{13}CO)$_4$. Unlabeled **1** (0.70 g, 1.03 mmol) was dissolved in toluene (50 mL) in a 100 mL Schlenk flask and cooled to $-$94 °C. The flask was then evacuated and refilled with ^{13}CO (from a 1 L flask at ~1 atm) and stirred for 15 min at $-$94 and 45 °C each. Spectral subtraction of the infrared spectrum gave peaks at 2003 (m), 1979 (s), 1953 (m), and 1921 (m) cm^{-1}, which are assigned to the monolabeled species Cp$'_2$Mo$_2$Co$_2$S$_3$(^{12}CO)$_3$(^{13}CO). This procedure was then repeated several times. Recrystallization from CH$_2$Cl$_2$/hexane gave 0.61 g (87% yield) of Cp$'_2$Mo$_2$Co$_2$S$_3$(^{13}CO)$_4$. ^{13}C{^{1}H} NMR (C$_6$D$_6$): δ 207 ($w_{1/2}$ = 930 Hz, ^{13}*C*O), 114 (quaternary *C*$_5$H$_4$CH$_3$), 100 (tertiary *C*$_5$H$_4$CH$_3$), 98 (tertiary *C*$_5$H$_4$CH$_3$), 15 (C$_5$H$_4$*C*H$_3$). Best fit of mass spectrum (EI) gives Cp$'_2$Mo$_2$Co$_2$S$_3$(^{12}CO)$_{0.16}$(^{13}CO)$_{3.84}$, which corresponds to

Table 3. Crystallographic Data for $[Cp'_2Mo_2Co_2S_3(\mu_3\text{-}CH)(\eta^2\text{-dmpe})_2]Cl\cdot 2C_6H_6$ ($14\cdot 2C_6H_6$)

formula	$C_{25}H_{47}P_4ClS_3Co_2Mo_2\cdot 2C_6H_6$
mw	1069.15
color and habit	black rectangular plate
crystal dimens (mm)	0.14 × 0.68 × 0.32
a, b, c (Å)	19.60(1), 10.981(5), 21.289(8)
α, β, γ (deg)	90, 107.60(3), 90
vol (Å^3), Z	4368(3), 4
density (calcd)	1.62(6)
system, space group	monoclinic, $P2_1/n$
radiation	Mo Kα ($\lambda = 0.710\ 73$ Å), Lp corrected, graphite monochromator
temperature	175 K
μ (cm^{-1})	16.6
max/min transmission	0.666/0.388
abs corr	empirical
diffractometer	Siemens R3m/v, equipped with LT-2
2θ range; octants used	5−30°; $+h$, $+k$, $\pm l$ 30−50°; $+h$, $+k$, $\pm l$
max indices	h, 0/24; k, 0/14; l, −26/26
no. of data collected	10 832
no. of unique reflns	7720, $R_{\text{int}} = 0.0145$
no. of refined reflns, $F_o \geq 8\sigma(F)$	6001
$R = \Sigma\lvert F_o\rvert - \lvert F_c\rvert)/\Sigma\lvert F_o\rvert$	0.1193
$R_w = [\Sigma(w\lvert F_o - F_c\rvert)^2/\Sigma w(F_o)^2]^{1/2}$, $w = \sigma^2(F_o)$	0.0788
gof	11.3
residual electron density (e/Å^3)	+1.58/−1.67

96% enrichment. IR (benzene): ν_{CO} 1986 (w), 1963 (m), 1942 (s), 1910 (ms) cm^{-1}.

Reaction of $Cp'_2Mo_2Co_2S_3(CO)_2(\eta^2\text{-dppe})$ with $(CH_3)_2CHCH_2CH_2SH$. Cluster **11a** (0.047 g, 0.046 mmol) and $(CH_3)_2CHCH_2CH_2SH$ (0.70 mL, 5.6 mmol) were dissolved in 20 mL of toluene and refluxed. After 2 h, only one ν_{CO} at 1955 cm^{-1} was observed, due to $Cp'_2Mo_2Co_2S_4(CO)(\eta^1\text{-dppe})$ (**15**); this peak disappeared after 6 h. The solvent was removed to give a dark green product which is believed to have the formula $Cp'_2Mo_2Co_2S_4(\eta^2,\mu_2\text{-dppe})$ (**18**). ^{1}H NMR (C_6D_6): δ 5.58 (t, J = 2.3 Hz, 4H), 5.25 (t, J = 2.3 Hz, 4H, A_2B_2 pattern for Cp*H*), 4.22 (m, 4H, CH_2CH_2), 2.01 (s, 6H, $CpCH_3$). Attempts to purify the product by chromatography or recrystallization were unsuccessful due to decomposition.

Reaction of $Cp'_2Mo_2Co_2S_4(CO)_2$ with dppe. Cluster **2** (0.124 g, 0.190 mmol) was dissolved in 500 mL of toluene and cooled to −50 °C. A toluene solution of dppe (0.076 g, 0.190 mmol) was added, and the solution was allowed to warm to room temperature. The solution was stirred for a total of 30 h. A ν_{CO} absorption at 1955 cm^{-1}, assigned to $Cp'_2Mo_2Co_2S_4(CO)(\eta^1\text{-dppe})$ (**15**), was observed after 5 h. The solvent was removed to give a green/brown product. No ν_{CO} absorptions were observed in the final product, and the NMR spectrum showed at least 7 singlets in the $CpCH_3$ region.

Reaction of $Cp'_2Mo_2Co_2S_3(CO)_2(\eta^2\text{-dppe})$ with $PhPH_2$. Cluster **11a** (0.093 g, 0.091 mmol) and $PhPH_2$ (2.5 mL of 0.35 M, 0.875 mmol) were dissolved in 25 mL of toluene and refluxed for 2 h. The infrared showed only one ν_{CO} at 1940 cm^{-1}, due to $Cp'_2Mo_2Co_2S_3(\mu^3\text{-PPh})(CO)(\eta^1\text{-dppe})$ (**16**). Attempts to purify this product for further analysis were unsuccessful. ^{1}H NMR (C_6D_6): δ 5.42 (m, 2H), 5.17 (m, 1H), 5.13 (m, 1H), 5.08 (m, 2H), 4.97 (m, 1H), 4.93 (m, 1H, 2ABCD pattern for Cp*H*), 1.91 (s, 3H, $CpCH_3$), 1.85 (s, 3H, $CpCH_3$).

Molecular Structure of $[Cp'_2Mo_2Co_2S_3(\mu_3\text{-}CH)(\eta^2\text{-dmpe})_2]Cl\cdot 2C_6H_6$ ($14\cdot 2C_6H_6$). A black rectangular plate suitable for an X-ray structure determination was obtained from CH_2Cl_2/C_6H_6 and mounted on a glass fiber. The structure was solved from the Patterson map obtained from the SHELXTL PLUS system and then submitted to a full-matrix least-squares refinement minimizing the function $\Sigma w(\lvert F_o - F_c\rvert)^2$. Atoms C2, C6, C25, and C31 were refined isotropically, all other non-hydrogen atoms were allowed to refine anisotropically. Hydrogen atoms were treated with the riding model $d_{C-H} = 0.96$ Å with isotropic U(H) refined to 0.089(9). Data were also collected in the range 50−55°, however, the increased resolution did not improve the model but rather lead to residuals ~1.5% higher than those reported above. In the final refinement, reflections in the 50−55° range were suppressed. Table 3 gives a summary of the crystal data and the data collection and refinement parameters.

Acknowledgment. We thank the National Science Foundation for support of this research under Grant No. CHE-9205018. O.J.C. also thanks the Horace H. Rackham School of Graduate Studies and the Department of Chemistry for Yates and Riggs Fellowships.

Supporting Information Available: Tables of structural determination parameters, atomic coordinates, anisotropic thermal parameters, bond lengths, and bond angles (10 pages). Ordering information is given on any current masthead page.

OM9610455