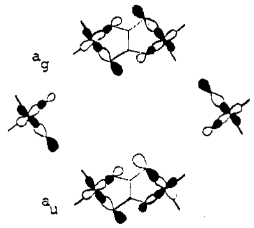
in which the S–N overlaps are negative (see below).



In the B isomer, the delocalization of the unpaired electrons toward sulfur and nitrogen atoms is much the same as in the A isomer. But, owing to both the deviation from planarity of the binuclear unit (see Figure 3) and the slight lengthening of S–N distances (2.702 and 2.682 Å instead of 2.674 Å), the σ S–N overlaps are not so well favored and the Δ energy separation becomes smaller than in the A isomer.

In conclusion, we wish to emphasize that the design of new bridging ligands more efficient than the dithiooxamidato to propagate the interactions in polynuclear systems, in which the transition ions are far away from each other, may be considered as a challenge for inorganic chemists. We would accept with pleasure to compete in this goal.

Registry No. $[\{Cu_2[S_2C_2(NCH_2CH_2OH)_2](H_2O)(SO_4)\}(H_2O)]_2$, 71276-85-2.

Supplementary Material Available: A listing of structure factor amplitudes (16 pages). Ordering information is given on any current masthead page.

References and Notes

(1) Our colleague C. Chauvel died in an accident on April 9, 1978, just after he obtained the single crystals of the title compound used for the structure study. We would like to dedicate to him this paper, his last paper.
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Structural Determination of $[(C_5H_5)_2Mo(H)CO][(C_5H_5)Mo(CO)_3]$

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On the basis of an X-ray diffraction study at −125 °C, $[Cp_2Mo(H)CO][CpMo(CO)_3]$ has been established to crystallize in space group $P2_1/c$ with $a = 6.370$ (2) Å, $b = 10.226$ (3) Å, $c = 27.233$ (11) Å, and $\beta = 84.58$ (1)°. Refinement of 3931 nonzero reflections leads to $R(F) = 0.040$ and $R_w(F) = 0.064$; all hydrogen atoms were refined. The anion $CpMo(CO)_3^-$ suffers only minor distortion from a symmetric "piano stool" configuration with average distances from Mo to cyclopentadienyl and carbonyl carbons of 2.381 and 1.930 Å; the MoCO groups are within 3σ of being linear. In the cation $Cp_2Mo(H)CO^+$, the corresponding distances are 2.291 and 1.991 Å. Comparison of these parameters, as well as the C–O distances, supports the idea that back-bonding is enhanced in low oxidation states. Comparison of the structural data for $Cp_2Mo(H)CO^+$ with the corresponding values in Cp_2MoH_2 indicates minimal π donation from CO to Mo(IV) in the cation.

Introduction

Compounds containing one metallic element in disparate oxidation states are unusual, if for no other reason than their potential to react by comproportionation to equalize their oxidation states. Our recent study[1] of the mechanism of carbonylation of Cp_2MoH_2 revealed the participation of a variety of bimetallic hydrogen-transfer reactions between molybdenum complexes in formal oxidation states IV, II, and I. For example, both reactions 1 and 2 yield compound I, of

$$Cp_2MoCO + CpMoH(CO)_3 \rightarrow I \qquad (1)$$

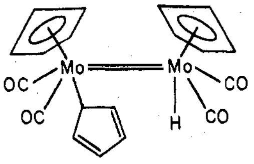
$$Cp_2MoH_2 + {}^1/_2[CpMo(CO)_3]_2 + CO \rightarrow I \qquad (2)$$

empirical formula $Cp_3Mo_2H(CO)_4$. Comproportionation or electronegativity equalization reactions necessitate consideration of a structure such as A for compound I. The structural study reported here was undertaken to resolve the actual nature of compound I.

Table I. Crystal Data for $[(C_5H_5)_2Mo(H)CO][(C_5H_5)Mo(CO)_3]$

formula	$C_{19}H_{16}O_4Mo_2$
color	orange
crystal dimensions, mm	0.19 × 0.12 × 0.25
space group	$P2_1/c$
a,[a] Å	6.370 (2)
b,[a] Å	10.226 (3)
c,[a] Å	27.233 (11)
β,[a] deg	84.58 (1)
Z	4
V, Å^3	1765.8
ρ(calcd), g cm^{-3}	1.878
mol wt	499.21
λ, Å	0.71069
μ(Mo Kα), cm^{-1}	14.07

[a] Cell dimensions at −125 °C using 12 reflections.



A

Experimental Section

A crystal obtained in low yield from reaction 2 was used for data collection. Unit cell dimensions and other crystal data are collected in Table I. Intensity data[2] were collected at −125 °C. A total of 8101 data were collected in the range $4° \leq 2\theta \leq 50°$. After reduction[3] and averaging of equivalent reflections, a final set of 4093 unique data was obtained, of which 3931 were nonzero, 3675 had $I > \sigma(I)$, and 3486 had $I > 2.33\sigma(I)$.

The structure was solved by using a combination of direct methods and Patterson and Fourier techniques. Some initial difficulty in locating the two Mo atoms was, in retrospect, attributable to the fact that the heavy atoms had almost identical y coordinates and were separated by about 0.25 Å along the z direction. All atoms were located and refined, including the hydride hydrogen atom.

Final residues for 3931 nonzero reflections are $R(F) = 0.040$ and $R_w(F) = 0.064$. The "goodness of fit" in the final cycle of refinement was 1.749, and the largest Δ/σ for a nonhydrogen parameter (O(2)) was 0.26; the largest value was 1.1 for a hydrogen atom. The ratio of observations to variables was 3931/285 = 13.8. An isotropic extinction correction parameter was included in the refinement (1.5 (8) × 10^{-7}).

Refined fractional coordinates and equivalent isotropic thermal parameters appear in Table II. Anisotropic B's and hydrogen positions are available as supplementary material. Bond lengths and angles are displayed in Tables III and IV. Least-squares planes and associated angles appear in Table V.

The carbon atoms within each five-carbon ring deviate by less than one standard deviation from the respective least-squares plane. Within experimental error, the refined hydrogen positions are coplanar with the five-carbon planes. The HCC angles range from 101 (6) to 151 (7)° and average 126°. Carbon–hydrogen distances range from 0.74 (6) to 1.16 (13) Å and average 0.99 Å. The atoms Mo(2), H(16), C(4), and O(4) are coplanar within experimental error.

Interionic C···C, C···O, and O···O contacts exceed 3.07 Å and interionic cyclopentadienyl C···H "contacts" (recognizing the systematic error in locating hydrogen atoms with X-rays) exceed 3.00 Å. Atom C(2) of the anion experiences three hydrogen contacts in the range 2.73–2.88 Å; similarly O(2) has one cyclopentadienyl hydrogen contact (2.29 Å to H(7) related by x, $0.5 - y$, $0.5 + z$) notably shorter than those for other oxygen atoms of the anion. This is apparently responsible for the observed angular displacement of ligand C(2)O(2) (Tables IV and V). In the cation, the shortest inter-ring contact not involving hydrogen is C(23)–C(31) at 3.08 Å. Other contacts include H(16)–C(4) (2.18 Å), H(16)–C(25) (2.42 Å), and C(4)–C(21) (2.65 Å).

Results and Discussion

Crystals of $(C_5H_5)_3Mo_2H(CO)_4$ contain discrete $Cp_2Mo(H)CO^+$ (Figure 1) and $CpMo(CO)_3^-$ ions (Figure 2). The shortest metal–metal separations are 5.902 Å (cation to anion), 6.338 Å (cation to cation), and 6.370 Å (anion to anion); there is, therefore, little evidence for ion pairing between oppositely charged ions in the solid. The anion has a "piano stool" structure, while the cation is structurally similar to the isoelectronic species $Cp_2Nb(H)CO$.[4] Detailed comparison to the niobium compound is not justifiable due to the large errors associated with that determination. The rings in the cation are essentially eclipsed (Figure 3) as they are in $Cp_2Nb(H)CO$. If planes are defined by using points M(1) and M(2) (see footnote a, Table IV) and individual carbon atoms, all dihedral angles vary by less than 1° from their average (7.9°).

Table II. Fractional Coordinates and Equivalent Isotropic Thermal Parameters for $[Cp_2Mo(H)CO][CpMo(CO)_3]$

atom	10^4x	10^4y	10^4z	$10B_{iso}$, Å^2
Mo(1)	5833 (1)	3154 (0)	3245 (0)	18
Mo(2)	1879 (1)	2905 (0)	696 (0)	16
O(1)	2806 (6)	5251 (4)	3726 (2)	40
O(2)	7641 (7)	2700 (5)	4238 (2)	33
O(3)	2312 (7)	1241 (5)	3640 (2)	45
O(4)	2652 (7)	5392 (4)	33 (2)	36
C(1)	3956 (7)	4465 (5)	3545 (2)	24
C(2)	6943 (8)	2902 (5)	3871 (2)	23
C(3)	3618 (8)	1949 (5)	3487 (2)	28
C(4)	2327 (8)	4497 (5)	278 (2)	27
C(11)	8434 (18)	2220 (9)	2666 (3)	61
C(12)	9090 (10)	3521 (11)	2771 (2)	51
C(13)	7634 (10)	4352 (6)	2578 (2)	34
C(14)	6120 (10)	3636 (7)	2380 (2)	35
C(15)	6561 (14)	2335 (8)	2432 (3)	47
C(21)	1393 (10)	2176 (6)	−74 (2)	32
C(22)	3519 (9)	1846 (6)	24 (2)	27
C(23)	3360 (9)	976 (5)	423 (2)	28
C(24)	1158 (8)	716 (5)	563 (2)	27
C(25)	−11 (9)	1448 (6)	254 (2)	33
C(31)	4042 (14)	2743 (8)	1312 (3)	46
C(32)	2003 (19)	2413 (8)	1513 (3)	57
C(33)	703 (12)	3507 (8)	1485 (2)	42
C(34)	1944 (9)	4515 (5)	1272 (2)	28
C(35)	4021 (10)	4055 (6)	1168 (2)	35

Table III. Bond Distances (Å) in $[Cp_2Mo(H)CO][CpMo(CO)_3]$

$Cp_2Mo(H)CO^+$		$CpMo(CO)_3^-$	
Mo(2)–C(4)	1.991 (5)	Mo(1)–C(1)	1.926 (5)
		Mo(1)–C(2)	1.923 (5)
		Mo(1)–C(3)	1.941 (5)
		av Mo–C(CO)	1.930 (10)[a]
Mo(2)–C(21)	2.276 (5)	Mo(1)–C(11)	2.378 (7)
Mo(2)–C(22)	2.291 (5)	Mo(1)–C(12)	2.367 (6)
Mo(2)–C(23)	2.281 (5)	Mo(1)–C(13)	2.392 (6)
Mo(2)–C(24)	2.321 (5)	Mo(1)–C(14)	2.396 (6)
Mo(2)–C(25)	2.323 (5)	Mo(1)–C(15)	2.373 (7)
Mo(2)–C(31)	2.274 (6)	av Mo–C(C_5H_5)	2.381 (11)[a]
Mo(2)–C(32)	2.290 (7)		
Mo(2)–C(33)	2.291 (6)		
Mo(2)–C(34)	2.277 (5)		
Mo(2)–C(35)	2.286 (5)		
av Mo–C(C_5H_5)	2.291 (18)[a]		
C(4)–O(4)	1.140 (7)	C(1)–O(1)	1.164 (6)
		C(2)–O(2)	1.151 (7)
		C(3)–O(3)	1.152 (7)
		av C–O	1.156 (7)[a]
C(21)–C(22)	1.445 (9)	C(11)–C(12)	1.432 (15)
C(21)–C(25)	1.416 (9)	C(11)–C(15)	1.410 (15)
C(22)–C(23)	1.398 (8)	C(12)–C(13)	1.397 (11)
C(23)–C(24)	1.443 (8)	C(13)–C(14)	1.362 (9)
C(24)–C(25)	1.394 (8)	C(14)–C(15)	1.370 (12)
C(31)–C(32)	1.403 (14)	av C–C	1.394 (26)[a]
C(31)–C(35)	1.398 (10)		
C(32)–C(33)	1.398 (13)		
C(33)–C(34)	1.393 (10)		
C(34)–C(35)	1.407 (8)		
av C–C	1.410 (19)[a]		
Mo(2)–H(16)	1.76 (8)		

[a] Esd's on average values are calculated by using the scatter formula $\sigma(\mathrm{av}) = [\Sigma(d_i - \bar{d})^2/(N-1)]^{1/2}$, where d_i is one of N individual values and $\bar{d}$ is the average of all values.

The $CpMo(CO)_3^-$ anion suffers some small distortion. While distances from Mo(1) to carbonyl carbons differ by less than 2.5σ, intra-ring carbon–carbon and Mo(1)–ring carbon distances differ by up to 4σ. Since these distortions do not reflect the maximum potential intraionic symmetry of this species, they are attributed to packing forces between ions (see Experimental Section). Comparable distortions were observed in $[N(n\text{-}Bu)_4][CpMo(CO)_3]$.[5] Carbon–oxygen distances are equal with experimental error.

The geometry of the cation is exceptionally close to that of Cp_2MoH_2 (Table VI),[6] in terms of both the dihedral angle

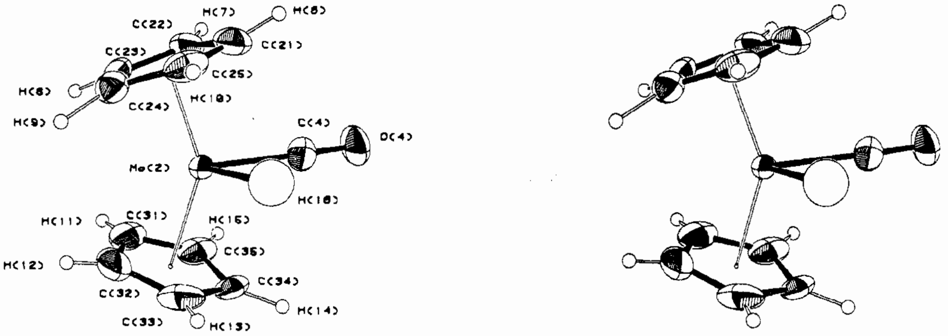


Figure 1. Stereoscopic view of $Cp_2Mo(H)CO^+$. Ring-hydrogen temperature factors have been arbitrarily reduced.

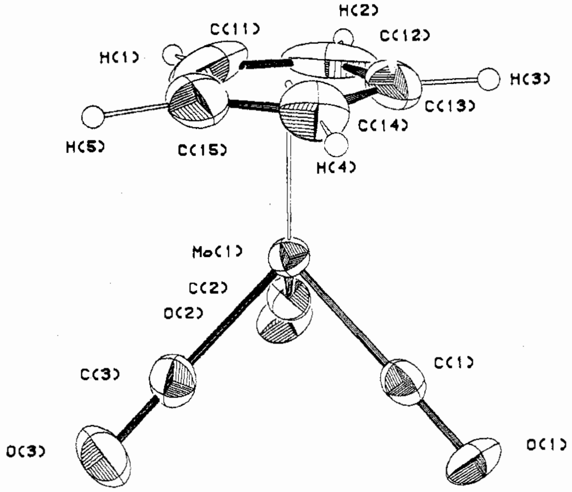


Figure 2. Perspective view of $CpMo(CO)_3^-$.

between the cyclopentadienyl planes and the angle between the noncyclopentadienyl ligands. This result is even more significant in view of the prediction[7] that significant π-acceptor character in the metal–ligand (L) bond in a $Cp_2M(H)L$ complex should increase the inter-C_5H_5 dihedral angle. However, the carbonyl stretching frequency in $Cp_2Mo(H)CO^+$ is high (2020 cm^{-1}), so it appears that there is actually minimal back-bonding to Mo(IV) in this complex. The absence of any systematic asymmetry in metal–cyclopentadienyl bond lengths implies that the steric requirements of CO in this weakly bonded environment are modest.

The Mo–C distances in Cp_2MoH_2[6] vary significantly (0.088 Å, 44σ). In the present study of the less symmetric species $Cp_2Mo(H)CO^+$ these distances are equal within experimental error for ring 3 and vary by 0.047 Å (7σ) for ring 2. Another indication of asymmetry of ring-to-metal bonding is given by the angle formed between the intersection of the line from Mo to the ring midpoint and the ring least-squares plane. For ring 3, this angle is 89.6°, while for ring 2 it is 88.6°. In Cp_2MoH_2, this angle is 80°. The distortions found for $Cp_2Mo(H)CO^+$ are thus small (of the size observed in Cp_2TaH_3) and of uncertain origin. Carbon–carbon distances, while normal, show considerably less spread in ring 3 (1σ) than in ring 2 (4σ).

Table IV. Intramolecular Angles (deg) in $[Cp_2Mo(H)CO][CpMo(CO)_3]$

$Cp_2Mo(H)CO^+$		$CpMo(CO)_3^-$	
		C(1)–Mo(1)–C(2)	89.0 (2)
		C(1)–Mo(1)–C(3)	84.0 (2)
		C(2)–Mo(1)–C(3)	86.0 (2)
		av	86.3 (25)[b]
M(2)[a]–Mo(2)–H(16)	99.9	M(1)–Mo(1)–C(1)	128.2
M(3)–Mo(2)–H(16)	106.6	M(1)–Mo(1)–C(2)	126.0
M(2)–Mo(2)–C(4)	105.9	M(1)–Mo(1)–C(3)	129.2
M(3)–Mo(2)–C(4)	105.0	av	127.8
M(2)–Mo(2)–M(3)	144.5		
C(4)–Mo(2)–H(16)	70.7 (25)		
Mo(2)–C(4)–O(4)	177.6 (5)	Mo(1)–C(1)–O(1)	179.4 (4)
		Mo(1)–C(2)–O(2)	177.1 (5)
		Mo(1)–C(3)–O(3)	178.5 (6)
		av	178.3 (12)[b]
C(22)–C(21)–C(25)	107.8 (5)	C(12)–C(11)–C(15)	106.8 (7)
C(21)–C(22)–C(23)	107.1 (5)	C(11)–C(12)–C(13)	105.8 (7)
C(22)–C(23)–C(24)	108.7 (5)	C(12)–C(13)–C(14)	110.0 (7)
C(23)–C(24)–C(25)	107.6 (5)	C(13)–C(14)–C(15)	108.8 (6)
C(21)–C(25)–C(24)	108.8 (5)	C(11)–C(15)–C(14)	108.5 (7)
C(32)–C(31)–C(35)	107.9 (6)	av	108.0 (17)[b]
C(31)–C(32)–C(33)	108.5 (7)		
C(32)–C(33)–C(34)	107.5 (7)		
C(33)–C(34)–C(35)	108.8 (6)		
C(31)–C(35)–C(34)	107.4 (6)		
av	108.0 (6)[b]		

[a] M(*i*) signifies the midpoint of the ring formed by carbons C(*iN*), *N* = 1–5. [b] See footnote *a*, Table III.

The hydride hydrogen in $Cp_2Mo(H)CO^+$ was located and refined in this study. While the Mo–H(16) distance is within experimental error of the more accurate determination in Cp_2MoH_2, there is some evidence for angular misplacement of H(16) in our work. Thus, while the line Mo(2)–C(4) makes equal angles (105°) to a line from Mo(2) to each ring midpoint, H(16) does not (99° and 106°). This also leads to an inequality in the angles between the Mo(2),C(4),O(4),-H(16) plane and the ring planes (Table V). The carbonyl ligand in the cation is clearly displaced off the twofold axis it would presumably occupy in Cp_2MoCO; the line

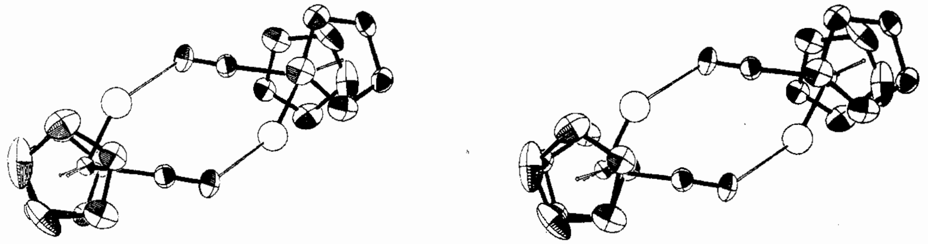


Figure 3. Stereoview of two $Cp_2Mo(H)CO^+$ cations along the line between ring midpoints (M(2)–M(3)) of the ion at left.

Table V. Distances (Å) from Least-Squares Planes and Dihedral Angles (deg) in $[Cp_2Mo(H)CO][CpMo(CO)_3]$

plane no.	atoms defining plane	atom	dist of atom from plane
1	C(11), C(12), C(13), C(14), C(15)	Mo(1)	2.065
		C(1)	3.284
		C(2)	3.166
		C(3)	3.291
		O(1)	4.025
		O(2)	3.800
		O(3)	4.040
2	C(21), C(22), C(23), C(24), C(25)	Mo(2)	1.955
3	C(31), C(32), C(33), C(34), C(35)	Mo(2)	1.948
4	Mo(2), C(4), O(4), H(16)	M(2)	1.879
		M(3)	1.832

plane nos.	dihedral angle	plane nos.	dihedral angle
2, 3	35.1	3, 4	19.8
2, 4	15.8		

Table VI. Comparison of Average Structural Parameters in $Cp_2Mo(H)L$ Species

	$Cp_2Mo(H)CO^+$	Cp_2MoH_2
Mo–C, Å	2.291 (5)	2.289 (2)
C–C, Å	1.410 (10)	1.421 (2)
Mo–H, Å	1.76 (8)	1.685 (3)
max carbon dev from C_5 plane, Å	0.014	0.004
Mo–C_5 plane, Å	1.952	1.942
dihedral angle between C_5 planes, deg	35.1	34.2
L–Mo–H, deg	71 (3) (L = CO)	75.5 (3) (L = H)

Mo(2)–C(4) makes an angle of 29.2° with the plane defined by M(2), M(3), and Mo(2). The hydride hydrogen suffers a greater displacement (41.3°) from this plane, consistent with its smaller size.

The formal metal oxidation number in the cation is +4, while it is zero in the anion. The intrinsic metal size is, therefore, expected to be larger in the anion. Consistent with this, the average Mo–C(cyclopentadienyl) distance is larger (0.090 Å, or 4.5 times the esd of the difference, using σ(av)) in the anion than in the cation, in spite of the greater crowding in the latter. The molybdenum-to-carbonyl carbon distances *contradict* this prediction, being 6σ shorter in the anion. This is consistent with greater back-donation to CO in the anion (the cation exhibits ν(CO) = 2020 cm^{-1} while the anion absorptions are all below 1890 cm^{-1}). In fact, the observed average Mo–CO distance of 1.930 Å appears to be shorter than all other compounds except $[N(n\text{-Bu})_4][CpMo(CO)_3]$,[5] in which the average is 1.909 Å.

Although there is no evidence for pairing between oppositely charged ions in the solid, atom O(4) in the cation has a 2.5 Å contact to the hydridic hydrogen of the cation related by ($-x$, $1-y$, $-z$); see Figure 3. The angle Mo(2)–H(16)–O(4) is 143°. Although the crystal symmetry necessitates that such contacts occur pairwise, it is surprising to find a metal-bound hydrogen (even on acidic complex[1]) as a participant in a hydrogen bond. In one form[8] of $[HIrNO(PPh_3)_3][ClO_4]$, in which the hydrogen was not located, there is a similar contact ($Ir\cdots OClO_3$) at 4.31 Å. The length of the O(4)···H(16) contacts in $[Cp_2Mo(H)CO][CpMo(CO)_3]$, and the lack of any spectroscopic effect on ν(CO) in the solid state, requires that any hydrogen bond be very weak if it exists at all.

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Registry No. $[Cp_2Mo(H)CO][CpMo(CO)_3]$, 69239-90-3.

Supplementary Material Available: Listings of calculated and observed structure factors, anisotropic thermal parameters, and refined hydrogen atom positions (39 pages). Ordering information is given on any current masthead page.

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