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The Crystal and Molecular Structure of Bis(cyclopentadienyldicarbonylchromium) (Cr≡Cr)

M. David Curtis and William M. Butler

Chemistry Department, University of Michigan, Ann Arbor, Michigan 38109 (U.S.A.)

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Abstract

The crystal structure of bis(cyclopentadienyldicarbonylchromium) has been determined by x-ray diffraction. The compound crystalizes in the triclinic system, space group $P\bar{1}$ (C_i^1, No. 2) with unit cell parameters: a, 7.829(3); b, 14.543(6); c, 6.588(2)Å; α, 94.67(3); β, 110.70(3); γ, 104.04°(3); V, 699.1(4)Å^3; Z=2. There are two independent molecules per unit cell located at the inversion centers at 0,0,0 and 0, 1/2, 0. The Cr-Cr bond distances are, respectively 2.200(3) and 2.230(3), thus supporting their formulation as triple bonds. The Cp-Cr-Cr angles in the two molecules are 165.0° and 158.7°, respectively. The structural features are compared with those of $Cp_2Mo_2(CO)_4$, which has a linear Cp-Mo-Mo-Cp axis; and the differences rationalized in terms of electronic interactions of the Cp-ligand with the orbitals of the M_2 unit. The differences observed in the structures of the two independent molecules are also related to the proposed bonding model and to packing considerations.

Many compounds with metal-metal multiple bonds have now been structurally characterized.[1-4] Most of the compounds so characterized are postulated to contain metal-metal quadruple bonds, although the structures of several compounds with bond order three have also been reported.[5-8] Most attention has been focused on the metal-metal bond length, but the multiple bonds also exert profound structural effects throughout the molecule. These effects are well recognized in the case of quadruple bonds (viz. the eclipsing of terminal ligands), but the structural effects of metal-metal triple bonds are not well recognized.

For example, the Mo≡Mo triple bond in $Cp_2Mo_2(CO)_4$ causes the Cp-Mo-Mo-Cp axis to be linear[8], but the $\acute{Cp}$-Cr-Cr-$\acute{Cp}$ ($\acute{Cp} = C_5Me_5$) axis in the analogous $Cp_2\acute{}Cr_2(CO)_4$ is highly bent (Cr-Cr-Cp = 158.7°). Even so, the latter angle is more open than that found in the compound $Cp_2Cr_2(CO)_6$ ($\angle$ Cr-Cr-Cp = 116.7). In both triply bonded molecules, $Cp_2Mo_2(CO)_4$ and $Cp_2\acute{}Cr(CO)_4$, the carbonyls are bent back over the metal-metal triple bond. The opened Cp´-Cr-Cr angle and the semi-bridging position of the carbonyls in $Cp_2\acute{}Cr_2(CO)_4$ were both ascribed to non-bonded repulsions between the methyl groups on the substituted cyclopentadienyl ring and the carbonyl groups.[5] However, the linear Cp-Mo-Mo axis and the semi-bridging position of the carbonyls in the sterically uncrowded molecule, $Cp_2Mo_2(CO)_4$, caused us to doubt that non-bonded repulsions were responsible for the unusual features noted in the structure of $Cp_2\acute{}Cr_2(CO)_4$. Indeed, it could be argued that non-bonded repulsions between the substituent methyl groups and the carbonyls were responsible for the observed bent, rather than linear, Cp´-Cr-Cr, structure.

In order to better resolve these interpretations, the crystal structure of the unsubstituted, parent compound, $Cp_2Cr_2(CO)_4$, was determined.

Table 1. Summary of crystal and intensity collection data.

Crystal dimensions (mm)	0.129 x 0.071 x 0.083
Space group	$P\bar{1}$
a,b,c (Å)	7.829(3), 14.543(6), 6.588(2)
α, β, γ (deg.)	94.67(3), 110.70(3), 104.04(3)
V(Å^3)	699.1(4)
Radiation	MoKα, monochromatized from graphite crystal
Takeoff angle	4°
μ(cm^{-1})	16.25
Transmission factors	.8772-.8953
Scan speed (deg/min)	2-12
Scan range, 2θ	$K\alpha_1$-0.8 to $K\alpha_2$ + 0.8
Ratio of background to peak scan time	0.8
Std. reflections	013, 061, $42\bar{1}$
2θ limit	55°
Total reflections collected	3100
Reflections with I > 3σ(I)	1216

Experimental and Results

The complex, $Cp_2Cr_2(CO)_4$, was synthesized by refluxing $Cp_2Cr_2(CO)_6$ in toluene according to the directions of Hackett et al.[9] Recrystallization from hot toluene afforded crystals suitable for diffraction. A crystal was mounted on a Syntex $P\bar{1}$ diffractometer. A summary of the crystal and intensity collection data is presented in Table 1. The crystal was triclinic, and a value of Z=2 gave a reasonable, calculated density of 1.72g cm^{-3}. The space group $P\bar{1}$ was then assumed

and later verified by the successful refinement.

A Patterson[10] map revealed that the two molecules each lay about two special positions at O,O,O and O, 1/2, O. Thus, one half of each molecule generates the asymmetric unit and two independent sets of bond distances and angles are obtained. Insertion of the Cr-atom positions from the Patterson map gave $R_1 = 0.30$ and a difference map revealed the non-hydrogen atom positions. Two full matrix refinements of the positional and isotropic thermal parameters gave $R_1 = 0.126$.[10] Refinement with all non-hydrogen atoms anisotropic and with anomalous

(Continued on p. 137)

Table 2. Fractional cell coordinates.[a]

atom	x	y	z
Cr1	0.0938(2)	0.5700(1)	0.1102(3)
Cr2	0.1324(2)	0.0498(1)	0.0047(3)
C1	0.2005(20)	-0.0171(9)	0.2608(23)
O1	0.2681(11)	-0.0436(6)	0.4107(13)
C2	0.0965(15)	-0.0746(8)	-0.1648(18)
O2	0.0971(10)	-0.1396(5)	-0.2613(12)
C3	-0.1419(15)	0.5553(7)	0.1335(17)
O3	-0.2780(10)	0.5563(5)	0.1609(13)
C4	-0.0237(17)	0.6020(8)	-0.1733(22)
O4	-0.0839(12)	0.6327(5)	-0.3250(13)
C5	0.2150(14)	0.6922(8)	0.3857(20)
C6	0.2747(14)	0.7220(7)	0.2274(21)
C7	0.3808(13)	0.6617(8)	0.1871(19)
C8	0.3910(14)	0.5976(8)	0.3350(19)
C9	0.2845(15)	0.6159(8)	0.4566(16)
C10	0.2353(16)	0.1426(9)	-0.1970(19)
C11	0.3802(17)	0.1057(8)	-0.0867(22)
C12	0.4350(14)	0.1347(8)	0.1365(22)
C13	0.3263(18)	0.1925(8)	0.1677(21)
C14	0.2030(15)	0.1993(8)	-0.0390(28)
H(5)	0.1322(0)	0.7207(0)	0.4489(0)
H(6)	0.2499(0)	0.7797(0)	0.1486(0)
H(7)	0.4415(0)	0.6647(0)	0.0716(0)
H(8)	0.4634(0)	0.5477(0)	0.3522(0)
H(9)	0.2614(0)	0.5788(0)	0.5739(0)
H(10)	0.1616(0)	0.1300(0)	-0.3653(0)
H(11)	0.4399(0)	0.0651(0)	-0.1574(0)
H(12)	0.5394(0)	0.1181(0)	0.2600(0)
H(13)	0.3374(0)	0.2260(0)	0.3176(0)
H(14)	0.1090(0)	0.2385(0)	-0.0646(0)

a) Hydrogen atoms numbered according to their bonded carbon. Hydrogen positions were not refined.

Table 3. Anisotropic thermal parameters.[b]

atom[a]	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
Cr1	0.0144(4)	0.0035(1)	0.0255(7)	-0.0003(2)	0.0075(4)	-0.0013(2)
Cr2	0.0165(5)	0.0045(1)	0.0330(8)	-0.0017(2)	0.0107(5)	0.0013(2)
C1	0.0369(41)	0.0068(9)	0.0306(48)	0.0090(16)	0.0064(36)	-0.0003(16)
O1	0.0234(19)	0.0090(6)	0.0254(26)	0.0051(9)	0.0033(18)	0.0087(11)
C2	0.0274(31)	0.0049(7)	0.0268(38)	0.0083(12)	0.0072(27)	0.0030(13)
O2	0.0293(21)	0.0032(4)	0.0384(28)	0.0028(7)	0.0172(20)	-0.0021(9)
C3	0.0196(27)	0.0055(7)	0.0250(36)	0.0028(11)	0.0095(26)	0.0012(12)
O3	0.0168(18)	0.0072(5)	0.0439(32)	0.0032(8)	0.0144(20)	-0.0026(10)
C4	0.0256(31)	0.0057(8)	0.0401(50)	0.0067(13)	0.0183(34)	0.0057(16)
O4	0.0445(28)	0.0052(5)	0.0272(29)	0.0040(9)	0.0067(23)	0.0048(10)
C5	0.0179(27)	0.0046(7)	0.0365(44)	0.0016(11)	0.0041(28)	-0.0033(14)
C6	0.0162(25)	0.0036(6)	0.0430(48)	0.0012(10)	0.0036(29)	0.0049(14)
C7	0.0107(22)	0.0055(7)	0.0392(46)	0.0036(10)	0.0051(26)	0.0038(15)
C8	0.0132(23)	0.0057(7)	0.0332(41)	0.0058(10)	0.0011(25)	0.0005(14)
C9	0.0189(25)	0.0059(7)	0.0204(34)	-0.0009(11)	-0.0015(24)	0.0016(13)
C10	0.0206(29)	0.0069(8)	0.0288(41)	-0.0010(12)	0.0059(28)	0.0060(15)
C11	0.0220(29)	0.0056(7)	0.0401(49)	-0.0000(12)	0.0183(32)	0.0019(15)
C12	0.0144(26)	0.0054(7)	0.0445(51)	0.0014(11)	-0.0008(28)	0.0084(17)
C13	0.0248(33)	0.0039(7)	0.0391(49)	-0.0011(12)	0.0138(33)	-0.0021(14)
C14	0.0149(27)	0.0043(7)	0.0699(69)	0.0028(11)	0.0095(37)	0.0096(18)
H(5)	4.68(0)					
H(6)	4.35(0)					
H(7)	3.74(0)					
H(8)	4.03(0)					
H(9)	4.29(0)					
H(10)	4.85(0)					
H(11)	4.83(0)					
H(12)	4.63(0)					
H(13)	4.70(0)					
H(14)	5.00(0)					

a) Hydrogen atoms numbered according to the bonded carbon. Hydrogen parameters were not refined.

b) The form of the anisotropic thermal correction is: $\exp[-(h^2\beta_{11} + k^2\beta_{22} + \ell^2\beta_{33} + 2hk\beta_{12} + 2h\ell\beta_{13} + 2k\ell\beta_{23})]$.

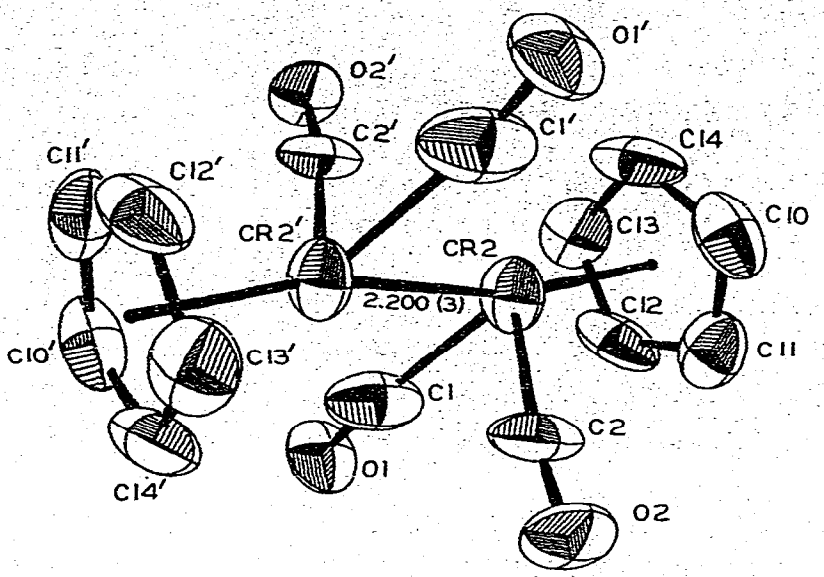


Fig. 1 ORTEP drawing of the $Cp_2Cr_2(CO)_4$ molecule located at 0,0,0. Thermal ellipsoids are contoured at the 50% probability level.

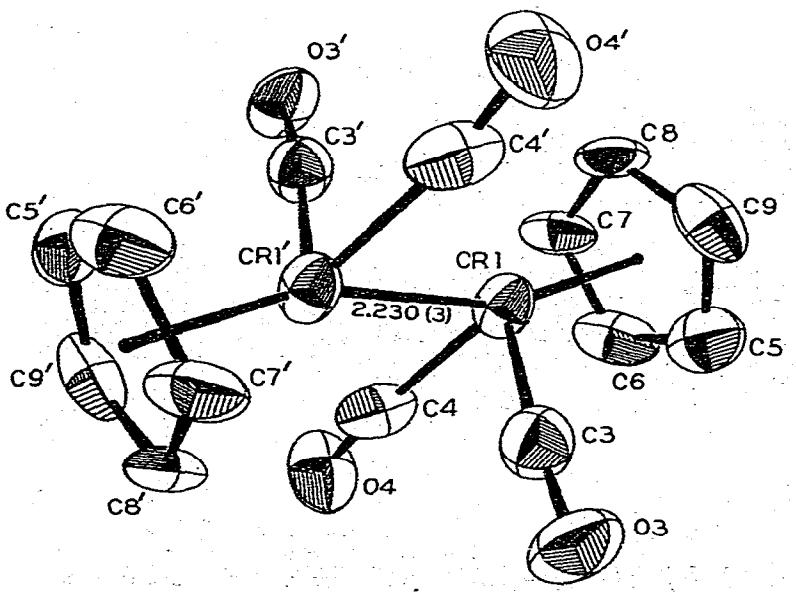


Fig. 2 ORTEP drawing of the $Cp_2Cr_2(CO)_4$ molecule located at 0,1/2,0. Thermal ellipsoids are contoured at the 50% probability level.

dispersion included for Cr converged in two more cycles at $R_1 = .067$ and $R_2 = .074$. A difference map at this stage did not show clear hydrogen peaks although electron density was obvious in the regions expected for the hydrogen atoms. Therefore, H-positions were calculated[10] with the C-H distance set at 1.0Å and with B-values set one unit higher than that of the contiguous carbon. These H-atoms were included in the Fourier synthesis but not refined. The structure analysis then converged at $R_1 = 0.060$ and $R_2 = 0.064$. The highest peaks in the final difference map were 0.89 and 0.87 $eÅ^{-3}$ and were shadows around

Table 4. Interatomic Distances and Bond Angles[a]

Bond	r(Å)	Bond	r(Å)
Cr1-Cr1′	2.230(3)	Cr2-CR2′	2.200(3)
Cr1-C3	1.868(11)	Cr2-C1	1.990(15)
Cr1-C4	1.920(14)	Cr2-C2	1.950(11)
Cr1-C5	2.190(10)	Cr2-C10	2.181(11)
Cr1-C6	2.218(10)	Cr2-C11	2.219(10)
Cr1-C7	2.172(10)	Cr2-C12	2.200(10)
Cr1-C8	2.187(10)	Cr2-C13	2.173(10)
Cr1-C9	2.178(10)	Cr2-C14	2.179(10)
Cr1···C3′	2.515(11)	Cr2···C1′	2.474(14)
Cr1···C4′	2.530(12)	Cr2···C2′	2.459(11)
Cr1-Cp[b]	1.842(11)	Cr2-Cp[b]	1.847(11)
C3-03	1.145(11)	C1-01	1.093(12)
C4-04	1.123(12)	C2-02	1.096(11)
C5-C6	1.347(16)	C10-C11	1.370(16)
C6-C7	1.415(14)	C11-C12	1.374(16)
C7-C8	1.398(14)	C12-C13	1.385(16)
C8-C9	1.397(14)	C13-C14	1.391(16)
C9-C5	1.394(15)	C14-C10	1.406(17)

Ave. C-C: 1.390 ± .026
Ave. Cr1-CO: 1.894 ± .037
Ave. Cr1-CO′: 2.523 ± .011
Ave. Cr1-Cp: 2.189 ± .018

Ave. C-C: 1.385 ± .014
Ave. Cr2-CO: 1.970 ± .028
Ave. Cr2-CO′: 2.466 ± .011
Ave. Cr2-Cp: 2.190 ± .019

Table 4. (continued)

Bonds	Angle°	Bonds	Angle°
Cr1-C3-O3	172.6(9)	Cr2-C1-O1	167.9(12)
Cr1-C4-O4	171.1(10)	Cr2-C2-O2	171.4(10)
Cr1′-Cr1-Cp[b]	158.7(4)	Cr2′-Cr2-Cp[b]	165.0(4)
Cr1′-Cr1-C3	75.2(3)	Cr2′-Cr2-C1	72.2(4)
Cr1′-Cr1-C4	74.7(3)	Cr2′-Cr2-C2	72.4(3)
Cr1′-Cr1-C5	160.7(4)	Cr2′-Cr2-C10	140.2(3)
Cr1′-Cr1-C6	161.8(4)	Cr2′-Cr2-C11	158.4(3)
Cr1′-Cr1-C7	136.4(3)	Cr2′-Cr2-C12	158.5(3)
Cr1′-Cr1-C8	125.6(3)	Cr2′-Cr2-C13	140.4(4)
Cr1′-Cr1-C9	135.8(3)	Cr2′-Cr2-C14	132.0(3)
C3-Cr1-C4	85.8(5)	C1-Cr2-C2	83.6(5)
C5-C6-C7	107.8(1.0)	C10-C11-C12	108.9(1.2)
C6-C7-C8	107.7(1.0)	C11-C12-C13	108.1(1.1)
C7-C8-C9	107.2(0.9)	C12-C13-C14	108.0(1.1)
C8-C9-C5	107.5(1.0)	C13-C14-C10	107.1(1.0)
C9-C5-C6	109.7(1.1)	C14-C10-C11	107.8(1.0)
Ave. C-C-C: 108.0 ± 1.0		Ave. C-C-C: 108.0 ± .6	
Ave. Cr1′-Cr1-CO: 75.0 ± 0.4		Ave. Cr2′-Cr2-CO: 72.3(5)	
Ave. Cr1-C-O: 171.8 ± 1.3		Ave. Cr2-C-O: 169.6 ± 2.5	

a) Standard deviations from variance-covariance matrix are in parentheses. Standard Deviations reported as "±" were computed from the formula, $\sigma = (\Sigma(x-\bar{x})^2/(n-1))^{1/2}$.

b) Cp in this table stands for the centroid of the cyclopentadienyl ring.

Cr1 and Cr2, respectively.* No other significant features were noted.

Transmission factors were calculated[10] for a variety of

*The table of structure factors has been deposited as NAPS Document No. 03267 (seven pages). Order from ASIS/NAPS, c/o Microfiche Publications, P.O. Box 3513, Grand Central Station, New York, N.Y. 10017. A copy may be secured by citing the document number, remitting $6.00 for photocopies or $4.00 for microfiche. Advance payment is required. Make checks payable to Microfiche Publications.

crystal settings and the small range in values indicated an absorption correction was not required. Standard reflections, measured after every 50 peaks, showed no decrease in intensity.

Atomic positional and temperature parameters are listed in Tables 2 and 3, respectively. Figures 1 and 2 are ORTEP plots of the independent molecules at 0,0,0 and 0, 1/2, 0, respectively. Figure 3 is a stereoview of the unit cell contents. Bond distances and angles are collected in Table 4, and Table 5 gives the equations of the least-squares planes containing the metal atoms and two carbonyls in each molecule.

Table 5. Least squares planes containing the metal atoms and carbonyls.

Plane[a]	Atom	Distance[b], Å (x 10^4)
1	Cr1'	-4
	Cr1	-26
	C3	77
	03	-47
2	Cr1'	8
	Cr1	53
	C4	-163
	04	102
3	Cr2'	6
	Cr2	24
	C1	-82
	01	52
4	Cr2'	2
	Cr2	16
	C2	-49
	02	31

a) Equations defining the planes: (1) .0240 x ÷.6426y - .7658z -4.6714=0; (2) .9207x -.2980y -.2519z + 2.1640=0; (3) .3671x -.7452y -.5567z -.0015=0; (4) -.2125x +.4988y -.8403z -.0009=0

b) Normal distance of atoms to the planes.

Discussion

The structure of the unsubstituted chromium complex $Cp_2Cr_2(CO)_4$, is remarkably similar to the structure of $(C_5Me_5)_2Cr(CO)_4$ determined earlier.[5] In fact, the anomolous features noted for the latter compound, and ascribed to steric crowding by the methyl groups, are present to the same or greater extent in the unsubstituted parent. Thus, the opening of the Cr-Cr-Cp angle is the same or greater, cf. 158.7° in the permethyl derivative vs. 158.7° and 165.0° in the two structures determined here. The Cr'-Cr-CO angles, while tending to be smaller in the unsubstituted complex (ave. 73.6±1.5 vs. 76.0±4.3 in $(C_5Me_5)_2Cr_2(CO)_4$), are not statistically different, due primarily to the large difference in the two Cr-Cr-CO angles found in the permethyl structure.

Hence, it is clear that these unusual features are the result of electronic effects associated with the Cr≡Cr triple

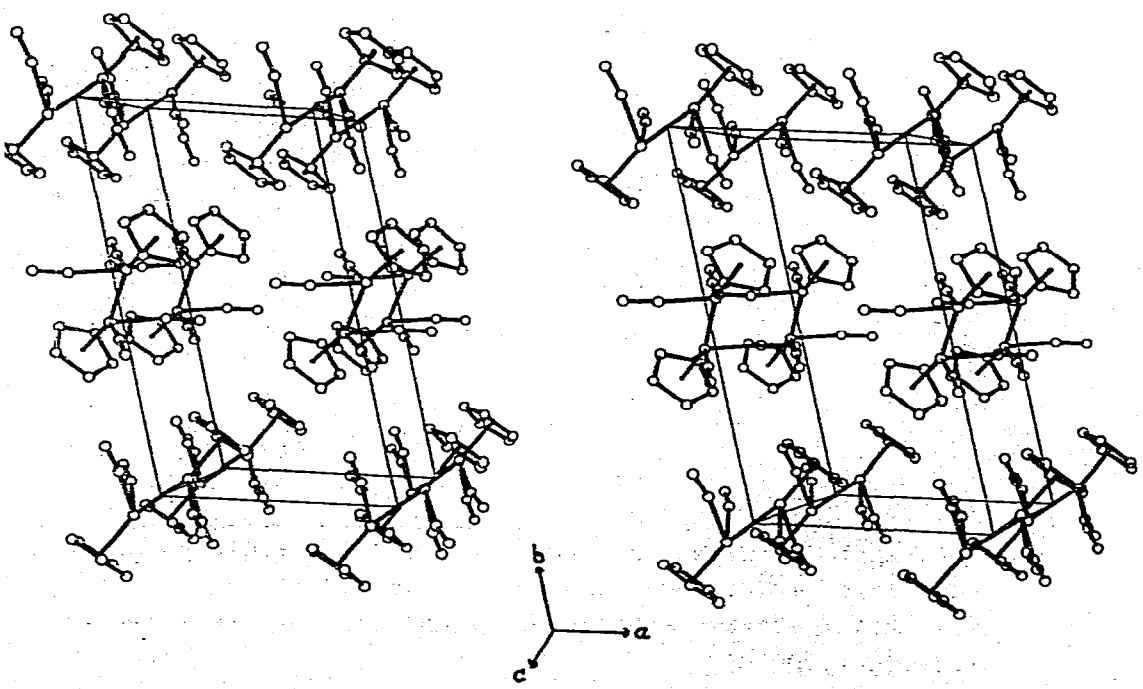
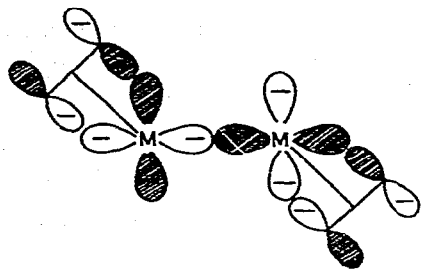


Fig. 3 ORTEP stereoview of unit cell contents.

(a) LINEAR



(b) BENT (135°)

Fig. 4 (a) Interaction of the Cp-ring e_1-orbitals with the metal-metal π-system; and (b) with the metal-metal σ-system.

bond, and are not due to steric crowding. This view is reinforced by the structure of the molybdenum compound, $Cp_2Mo_2(CO)_4$, in which these anomalies are carried to the extreme.[8]

The problem of why the complexes, $Cp_2Cr_2(CO)_4$ and $(C_5Me_5)_2Cr_2(CO)_4$ are bent whereas the complex, $Cp_2Mo_2(CO)_4$, is "linear" cannot be answered definitively in the absence of reliable MO calculations.

In compounds of the type being considered, the strongest interaction between the Cp-rings and the metal centers arises from the electron donation from filled e_1-orbitals on the ring to vacant d-orbitals on the metal. In the linear structure, the electron donation is to the metal-metal π^* orbitals. At a Cp-M-M angle, ω, of 135°, the ring e_1-orbitals donate e-density into the metal-metal σ^*-orbital (see Fig. 4).

The interaction of the M_2 fragment with the cyclopentadienyls will raise the energy of the metal d-orbitals regardless of the Cp-M-M angle. However, in the linear configuration, the π-system energy is raised higher relative to the σ. The opposite effect prevails at $\omega = 135°$; the σ-system is raised relative

to the π. In other words, as the Cp-M-M angle is decreased from 180°, the metal-metal π-energies will fall and the metal-metal σ-energy will rise. The angle, ω, at which a minimum occurs will depend on the relative rates of changes in the σ- and π- bond energies, and these in turn depend on the metal-ring overlap and the energy separation between the metal and the ring orbitals.

The Cr-Cr bond lengths, 2.230(3) and 2.200(3), in the two structures determined here are significantly shorter than the Cr-Cr distance, 2.280(2), found in the permethyl derivative. The pentamethylcyclopentadienyl ligand should be more electron donating than the unsubstituted ring, leading to higher populations in those orbitals with a metal-metal anti-bonding component, and increasing the metal-metal distance as observed.

The above model has an interesting application to the differences observed in the two independent structures determined for $Cp_2Cr_2(CO)_4$. As the angle, ω, decreases from 180° toward 135°, electron density is shifted from molecular orbitals with a metal-metal π* component to molecular orbitals with a metal-metal σ* component. One could argue that population of the latter molecular orbital would exert a more pronounced weakening (and lengthening) of the metal-metal bond. This is precisely what is observed in the two structures presented here. The molecule with the more open angle, viz. Cr2′-Cr2-Cp = 165.0(4) vs. Cr1′-Cr1 - Cp = 158.7(4), has the shorter Cr-Cr bond, viz. 2.200(3) vs. 2.230(3). The difference in Cr-Cr bond lengths, .03Å, is some seven times the standard deviation of the difference, $\sigma_d = (\sigma_1^2 + \sigma_2^2)^{1/2}$.

It appears that packing forces are responsible for the distortion of the molecule at 0,1/2,0 relative to the one at 0,0,0. From Fig. 3, it can be seen that the carbonyl groups

on the molecule at 0,0,0 point toward the Cp-rings on the molecule at 0,1/2,0 in a manner which would tend to compress the Cr1´-Cr1-Cp angle. The short intermolecular contacts, O1-H6 (2.92), O1-H5(3.39), O2-H5(2.80), and O2-H6(3.00) are all in a direction such that both the angles, Cr1´-Cr1-Cp and Cr2´-Cr2-CO, would be compressed. The contacts, O3-H14´(3.17), O4-H14´(3.14), and O4-H13´(3.19), all tend to open the Cr2´-Cr2-Cp angle and to compress the CR1´-Cr1-CO angle. Thus, the differences observed in the two independent structures determined here can be rationalized on the basis of the bonding model presented and the intermolecular forces present in the crystal.

The carbonyl ligands in $Cp_2Cr_2(CO)_4$ form an acute Cr´-Cr-CO angle (ave. = 73.6 ± 1.5) and form an unsymmetric bridge. If we define the "asymmetry parameter", α , to be $(d_2-d_1)/d_1$, where d_2 = long M···C distance, d_1 = short M-C distance, then a plot of α vs. θ, the M-C-O angle, gives two smooth curves.[11] One curve has θ essentially invariant with respect to α ; and the other curve shows a decrease in θ with decreasing α . The latter behavior is typical of normal bridging carbonyls, i.e. carbonyls which act as two electron donor ligands. The former behavior is characteristic of a class of bridging carbonyls which act as incipient 4-electron donors, two electrons being donated as usual by the CO-lone pair and the other two from the CO π-bond. The average value of the asymmetry parameter for $Cp_2Cr_2(CO)_4$ is 0.291. This value, when plotted against the average value of θ gives a point which is clearly on the "4-electron donor curve".[11] Hence, we postulate some electron donation from the carbonyl π-orbitals into the Cr-Cr π^* orbitals. A similar interaction of the carbonyls with the Mo≡Mo triple bond has been postulated for $Cp_2Mo_2(CO)_4$.[11]

Two of the Cr'-Cr-CO angles in $Cp_2Cr_2(CO)_6$ are also quite acute (ave. = 71.2°).[13] However, the asymmetry parameter,[13] $\alpha = 0.728$, is considerably larger and a plot of α vs. θ lies in the terminal CO-region as expected.[11]

It is interesting to note that the chemical reactivities of $Cp_2Cr_2(CO)_4$ and $Cp_2Mo_2(CO)_4$ also differ markedly. While the latter reacts with many soft nucleophiles and acetylenes[8,14] to yield well-defined products, the former either yields intractable mixtures[9] or, in the case of acetylenes, fails to react at RT.[12] These differences in chemical reactivity of the metal-metal triple bonds may also be related to the same factors which are responsible for the different structures of the chromium and molybdenum derivatives, and further work is in progress to elucidate this point.

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