

Figure 5. $^{95,97}\text{Mo}$ hyperfine splitting constant vs. the square of the d_{xy} coefficient.

qualitatively with the EPR observations. However, we may proceed further and compare the coefficients for the d_{xy} orbital from complex to complex with the hyperfine splittings in the EPR spectral lines stemming from the ^{95}Mo and ^{97}Mo nuclei. The magnitudes of these splittings have been determined.^{2,3} We would expect the splittings to increase in magnitude with increase in metal d character of the highest occupied orbital. Since the character of the highest occupied orbital is determined in each calculation, we can plot the *square* of the d_{xy} coefficient (which determines the percentage d contribution) against the observed splitting constants. Figure 5 shows such a plot, and it can be seen that there is effectively a straight-line relationship between the splitting constant and the square of the d_{xy} coefficient. Only the case of the *i-mnt* complex appears to be anomalous, and at present we can advance no reason for this deviation.

Electronic Spectra. The electronic absorption spectra of the complexes depend on the energy level sequences around the highest occupied and lowest unoccupied orbital regions; thus, it is germane to inquire whether any correlation should exist between these properties and $E_{1/2}$ values. Orbital levels alone are not sufficient to determine absorption energies, but where there is no orbital degeneracy and localization is strong (and hence configuration interaction is reduced), a qualitative result is reliable. The latter conditions apply to these complexes. One sees that the energy gaps are little changed from complex to complex, and hence, the electronic absorption spectra will not be as sensitive to ligand structure as are $E_{1/2}$ values and core-electron binding energies. This expectation is demonstrated by the charge-transfer absorptions of the ethylenedithiolate complexes, which correspond to the almost constant spacings *A* (for ligand \rightarrow metal charge transfer) or *B* (for metal \rightarrow ligand charge transfer) in Figure 3. Experimentally, the charge-transfer energies observed for Mo(V)- and Mo(VI)-ethylenedithiolate complexes^{2,3} cover a range of ≤ 0.12 eV, in agreement with the prediction that these properties are not highly sensitive to ligand structure. There are other recent observations,^{13,14} which have been rationalized qualitatively on a similar basis, of families of structurally related transition-metal complexes exhibiting a large range in $E_{1/2}$ values but only small changes in electronic absorption energies.

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Some Reactions of Tetrakis(μ -acetato)dichromium(II) with Potentially Chelating Ligands: Two Unexpected Products

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Reactions of $\text{Cr}_2(\text{O}_2\text{CCH}_3)_4$, under mild conditions, with $\text{NaOCH}_2\text{CH}_2\text{N}(\text{CH}_3)_2$ and with $\text{Na}[\text{B}(\text{C}_2\text{H}_5)_2(2\text{-pyrazolyl})_2]$ have been examined, and crystalline products, **1** and **2**, respectively, have been isolated in good yields. In neither case did we find a simple substitution product. Compound **1** consists of $\text{Cr}_4(\text{O}_2\text{CCH}_3)_4(\text{OCH}_2\text{CH}_2\text{NMe}_2)_4$ molecules, which have crystallographic $\bar{4}$ (S_4) symmetry. Two $\text{Cr}_2(\mu\text{-O}_2\text{CCH}_3)_2$ units, in which the O_2CC planes are approximately orthogonal and $\text{Cr}-\text{Cr} = 2.531$ (2) Å, form a bisphenoid of Cr atoms, and there is a square set of oxygen atoms (from $\text{OCH}_2\text{CH}_2\text{NMe}_2$ groups) between the upper and lower Cr_2 units. The N atoms are coordinated to the Cr atoms along the extensions of the $\text{Cr}-\text{Cr}$ bonds. This compound crystallizes in space group $P4_2/n$ with two tetranuclear formula units in a unit cell of dimensions $a = 13.269$ (2) Å, $c = 10.611$ (2) Å, and $V = 1868$ (1) Å³. Compound **2** contains mononuclear $\text{Cr}(\text{pz})_2\text{BEt}_2$ molecules residing on crystallographic inversion centers. This bright orange, square-planar Cr(II) complex is air stable and crystallizes in space group $P2_1/c$ with the following unit cell parameters: $a = 10.154$ (4) Å, $b = 8.507$ (1) Å, $c = 13.624$ (4) Å, $\beta = 93.30$ (3)°, and $V = 1175$ (1) Å³. The Cr-N distances are identical within experimental error: 2.058 (4) and 2.061 (4) Å. The stability of the compound may be in part due to the fact that one ethyl group from each ligand is positioned above and below the CrN_4 coordination plane.

Introduction

It is true generally that the reactions of compounds containing $\text{M}\equiv\text{M}$ (triple) and $\text{M}\equiv\text{M}$ (quadruple) bonds may proceed either in conservative ways, i.e., with retention of the initial multiple bond, or in various nonconservative ways, i.e.,

with modification or outright loss of the initial M-M multiple bond.^{1,2} The tendency for nonconservative reactions to occur

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is particularly great with the dichromium tetracarboxylates, $\text{Cr}_2(\text{O}_2\text{CR})_4$, where the $\text{Cr}-\text{Cr}$ bond is relatively weak and, hence, unusually reactive.

In this paper we report two new examples of nonconservative reactions of $\text{Cr}_2(\text{O}_2\text{CCH}_3)_4$. In the first case, in an attempt to displace several of the CH_3CO_2^- ligands by the chelating ligand $\text{OCH}_2\text{CH}_2\text{NMe}_2$, a tetranuclear product, $\text{Cr}_4(\text{O}_2\text{CCH}_3)_4(\text{OCH}_2\text{CH}_2\text{NMe}_2)_4$ (**1**) was obtained. In fact, the $\text{Cr}-\text{Cr}$ bond, still formally a quadruple bond, is retained, and the local stereochemistry about the Cr_2^{4+} units is still of the original type, but a more complex overall structure has been formed. In the reaction with $\text{Na}[\text{B}(\text{pz})_2\text{Et}_2]$, where, again, the planned goal was simple replacement of some (or all) of the CH_3CO_2^- ligands with chelating ligands, such a replacement was achieved, but with concomitant loss of the $\text{Cr}-\text{Cr}$ bond, so that a mononuclear product, $\text{Cr}((\text{pz})_2\text{BEt}_2)_2$ (**2**), resulted.

Experimental Section

All reactions, manipulations, and crystallizations were performed under an atmosphere of dry argon. All solvents were dried and deoxygenated prior to use. $\text{NaOCH}_2\text{CH}_2\text{N}(\text{CH}_3)_2$ was prepared by mixing stoichiometric amounts of sodium methoxide and (dimethylamino)ethanol in methanol and $\text{Na}(\text{pz})_2\text{BEt}_2$ by the method of Trofimenko.³

Preparation and Crystallization of $\text{Cr}_4(\text{O}_2\text{CCH}_3)_4(\text{dimethylaminoethanol})_4$ (1**).** $\text{NaOCH}_2\text{CH}_2\text{N}(\text{CH}_3)_2$ (0.44 g, 4 mmol) was dissolved in 40 mL of THF. To this rapidly stirred solution was added 0.68 g (2 mmol) of anhydrous chromium(II) acetate, and the mixture was stirred overnight. After filtration through Celite the clear red solution was reduced in volume to ca. 10 mL. Dark red, air-sensitive needles, yield 0.5 g (63%), were obtained after several days of standing at -10°C . The structural unit in the crystals was shown by X-ray crystallography (vide infra) not to be that of the desired dichromium(II) chelate complex, $\text{Cr}_2(\text{O}_2\text{CCH}_3)_2(\text{OCH}_2\text{CH}_2\text{N}(\text{CH}_3)_2)_2$, but instead that of a tetranuclear complex with the same empirical formula, $\text{Cr}_4(\text{O}_2\text{CCH}_3)_4(\text{OCH}_2\text{CH}_2\text{N}(\text{CH}_3)_2)_4$.

Preparation and Crystallization of $\text{Cr}_2((\text{pz})_2\text{BEt}_2)_2$ (2**).** In a manner similar to that described for compound **1** $\text{Na}(\text{pz})_2\text{BEt}_2$ (0.91 g, 4 mmol) and $\text{Cr}_2(\text{OAc})_4$ (0.34 g, 1 mmol) were stirred overnight in 25 mL of THF. After filtration of the orange solution into a tubular flask, 20 mL of hexane was carefully layered onto the THF solution. Slow diffusion (7 days) of the two layers afforded 0.55 g, 60%, of bright orange, air-stable crystals. Again, the structural unit in the isolated product was not that of the expected dinuclear complex, $\text{Cr}_2((\text{pz})_2\text{BEt}_2)_2$, but, instead, as shown by X-ray crystallography, it is monomeric $\text{Cr}((\text{pz})_2\text{BEt}_2)_2$ analogous to $\text{Cr}(\text{H}_2\text{B}(\text{pz})_2)_2$ previously obtained⁴ from the reaction of CrCl_2 and $\text{KH}_2\text{B}(\text{pz})_2$.

X-ray Data Collection and Structure Determination. For both crystals the intensity data were collected at room temperature on an Enraf-Nonius CAD-4 automated diffractometer to $2\theta = 50^\circ$ by using the $\omega-2\theta$ method and a scan range determined by $\Delta\omega = (0.66 + 0.35 \tan \theta)$, **1**, and $(0.60 + 0.35 \tan \theta)$, **2**, with a 25% extension at either end for background determination. Standard data-collection procedures have been previously summarized,⁵ and the pertinent crystallographic parameters for both crystals are listed in Table I. Data were processed, averaged for equivalent and overlapping reflections, and corrected for Lorentz and polarization effects; all calculations required to solve and refine the structures were carried out on a PDP 11/60 computer at B. A. Frenz and Associates, College Station, TX. Tables of thermal parameters and observed and calculated structure factors for both compounds are available as supplementary material, as are hydrogen atom positions and thermal parameters for compound **2**.

Structure of $\text{Cr}_4(\text{O}_2\text{CCH}_3)_4(\text{OCH}_2\text{CH}_2\text{N}(\text{CH}_3)_2)_4$ (1**).** A dark red needle measuring $0.5 \times 0.2 \times 0.1$ mm was sealed under nitrogen in

Table I. Crystallographic Data and Data Collection Procedures

	$\text{Cr}_4\text{O}_{12}\text{N}_4\text{C}_{24}\text{H}_{52}$ (1)	$\text{CrN}_8\text{C}_{20}\text{B}_2\text{H}_{32}$ (2)
M_r	796.70	458.15
space group	$P4_2/n$ (2nd setting)	$P2_1/c$
a , Å	13.269 (2)	10.154 (4)
b , Å	13.269 (2)	8.507 (1)
c , Å	10.611 (2)	13.624 (4)
V , Å ³	1868.3 (9)	1175 (1)
β , deg	90.00 (0)	93.30 (3)
Z	2	2
ρ_c , g/cm ³	1.395	1.295
$\mu(\text{Mo K}\alpha)$, cm ⁻¹	12.364	4.968
cryst size, mm	$0.5 \times 0.2 \times 0.1$	$0.3 \times 0.2 \times 0.1$
diffractometer	Enraf-Nonius CAD-4	
radiation	graphite-monochromated Mo K α ($\lambda = 0.71073$ Å)	
collection range	$+h, +k, +l$ ($0 \leq 2\theta \leq 50^\circ$)	$+h, +k, \pm l$ ($0 \leq 2\theta \leq 50^\circ$)
scan type	$\omega-2\theta$	$\omega-2\theta$
scan width	$0.66 + 0.35 \tan \theta$	$0.60 + 0.35 \tan \theta$
max counting time, s	30	120
X-ray exposure time, h	54	54
% cryst dec	0	0
no. of unique data	1794	2056
no. of data with $I \geq 3\sigma(I)$	835	974
ρ	0.05	0.05
no. of variables	100	142
R_1	0.052	0.057
R_2	0.065	0.066
esd	1.834	1.662
largest shift ^a	0.01	0.00
largest peak, ^b e/Å ³	0.39	0.41

^a Largest parameter shift in final refinement cycle. ^b Largest peak in a final difference Fourier.

a glass capillary with use of degassed epoxy cement. Data were collected for a tetragonal crystal system and revealed systematic absences (hkl , $h+k=2n+1$, and $00l$, $l=2n+1$) that uniquely determined the space group as $P4_2/n$. The observed cell volume was consistent with the presence of four dinuclear (or, of course, two tetranuclear) entities in the unit cell. The position of the unique chromium atom, situated close to a $\bar{4}$ symmetry element at $1/4, 1/4, 1/4$, was determined from the Patterson map on a general position with the second origin setting for $P4_2/n$. Four cycles of least-squares refinement of this atom position along with the associated isotropic temperature factor produced residuals of

$$R_1 = \sum ||F_o| - |F_c|| / \sum |F_o| = 0.412$$

$$R_2 = [\sum w(|F_o| - |F_c|)^2 / \sum w|F_o|^2]^{1/2} = 0.445$$

Refinement of the Cr atom position followed by a difference Fourier map revealed the positions of the remaining 10 atoms. Further least-squares refinement with anisotropic thermal parameters for all 11 atoms led to final discrepancy factors listed in Table I. No effort was made to locate the hydrogen atoms. The final difference Fourier map showed no significant residual electron density.

Structure of $\text{Cr}((\text{pz})_2\text{BEt}_2)_2$ (2**).** Diffraction data for **2** were collected for a monoclinic crystal system. Subsequent examination of the data revealed systematic absences consistent with the space group $P2_1/c$: $h0l$, $l=2n+1$; $0k0$, $k=2n+1$. Since the cell volume was appropriate for only two formula units, the metal atom was required to be located on an inversion center. Reformulation of the anticipated tightly bound dimeric moiety as a monomer was demanded. From a three-dimensional Patterson function the position of the chromium atom was located at the 0, 0, 0 inversion center. Three cycles of least-squares refinement of the chromium atom position followed by a difference Fourier map led to location of all non-hydrogen atoms in the complex. A subsequent Fourier synthesis revealed all hydrogen atoms except those of the methyl carbon. Hydrogen atom contributions were included in F_c values, but they were not refined. Inclusion of these atoms in the refinement gave convergence at $R_1 = 0.057$ and $R_2 = 0.066$.

Final positional parameters of non-hydrogen atoms for **1** and **2** are given in Tables II and III, respectively. The thermal parameters for these structures are available as supplementary tables S1 and S2, and

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Table II. Positional Parameters and Their Estimated Standard Deviations for $\text{Cr}_4(\text{CH}_3\text{COO})_4((\text{CH}_3)_2\text{NCH}_2\text{CH}_2\text{O})_4$ (1)^a

atom	x	y	z
Cr(1)	0.15736 (8)	0.22729 (8)	0.1163 (1)
O(1)	0.1432 (4)	0.3383 (4)	-0.0145 (5)
O(2)	0.3069 (4)	0.3666 (4)	-0.0283 (5)
O(3)	0.3215 (3)	0.3815 (3)	0.2439 (5)
N(1)	0.2079 (5)	0.5257 (5)	0.3896 (6)
C(1)	0.2175 (5)	0.3793 (5)	-0.0646 (7)
C(2)	0.1996 (7)	0.4473 (6)	-0.1794 (8)
C(3)	0.2944 (8)	0.5546 (7)	0.3163 (10)
C(4)	0.3116 (8)	0.4870 (6)	0.2074 (10)
C(5)	0.1123 (8)	0.5588 (8)	0.3323 (12)
C(6)	0.2182 (10)	0.5697 (7)	0.5200 (10)

^a Estimated standard deviations in the least significant digits are shown in parentheses.

Table III. Positional Parameters and Their Estimated Standard Deviations for Non-Hydrogen Atoms in $\text{Cr}((\text{pz})_2\text{BET}_2)_2$ (2)^a

atom	x	y	z
Cr	0.0000 (0)	0.0000 (0)	0.0000 (0)
N(11)	-0.1685 (5)	0.0984 (6)	-0.0649 (4)
N(12)	-0.2690 (4)	0.0085 (7)	-0.1044 (3)
N(21)	-0.0237 (5)	-0.1875 (6)	-0.0952 (4)
N(22)	-0.1458 (5)	-0.2386 (6)	-0.1282 (3)
C(1)	-0.2637 (6)	-0.1990 (8)	0.0350 (5)
C(2)	-0.2568 (9)	-0.3710 (10)	0.0670 (6)
C(3)	-0.4016 (6)	-0.2536 (8)	-0.1390 (5)
C(4)	-0.5318 (6)	-0.2173 (10)	-0.0937 (6)
C(11)	-0.3521 (6)	0.1008 (8)	-0.1588 (5)
C(12)	-0.3063 (6)	0.2528 (8)	-0.1537 (5)
C(13)	-0.1907 (6)	0.2468 (8)	-0.0947 (5)
C(21)	-0.1283 (6)	-0.3537 (8)	-0.1955 (5)
C(22)	0.0037 (6)	-0.3762 (8)	-0.2073 (5)
C(23)	0.0647 (6)	-0.2711 (8)	-0.1418 (5)
B	-0.2744 (7)	-0.1724 (9)	-0.0817 (5)

^a Estimated standard deviations in the least significant digits are shown in parentheses.

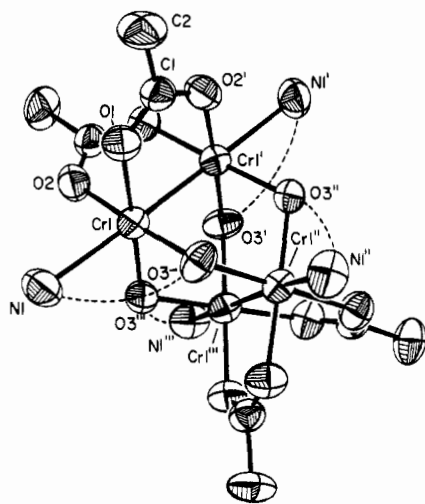


Figure 1. Semischematic view of the $\text{Cr}_4(\text{O}_2\text{CCH}_3)_4(\text{OCH}_2\text{CH}_2\text{NMe}_2)_4$ molecule in which the $-\text{CH}_2\text{CH}_2-$ and CH_3 groups of the $\text{OCH}_2\text{CH}_2\text{NMe}_2$ ligands have been omitted for clarity. Associated O and N atoms are connected by dashed lines. Atoms are represented by 30% thermal ellipsoids.

for 2 supplementary tables S3 and S4 give positional and thermal parameters for hydrogen atoms as well as bond lengths and angles in which hydrogen atoms are involved. Tables of structure factors are also available as supplementary material.

Results and Discussion

Compound 1. This substance consists of tetranuclear molecules, $\text{Cr}_4(\text{O}_2\text{CCH}_3)_4(\text{OCH}_2\text{CH}_2\text{NMe}_2)_4$, residing on positions of $\bar{4}$ (S_4) crystallographic symmetry. Figure 1 shows the molecule in a semischematic way, with only the O and N

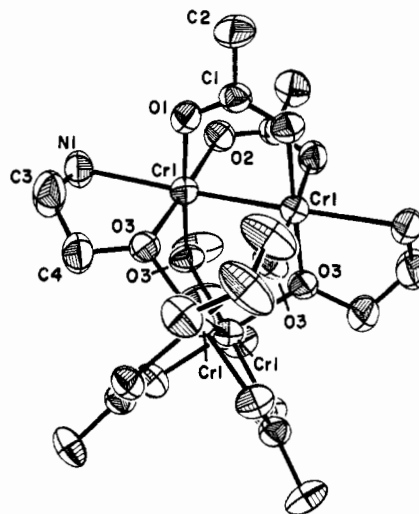


Figure 2. Different view of the $\text{Cr}_4(\text{O}_2\text{CCH}_3)_4(\text{OCH}_2\text{CH}_2\text{NMe}_2)_4$ molecule, with the *N*-methyl groups omitted for clarity.

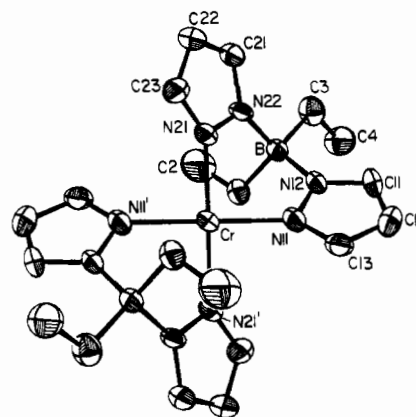


Figure 3. ORTEP computer drawing of the $\text{Cr}((\text{C}_2\text{H}_5\text{N}_2)_2\text{B}(\text{C}_2\text{H}_5)_2)_2$ molecule showing the atom-labeling scheme. The Cr atom resides on a center of symmetry.

Table IV. Bond Distances (Å) and Bond Angles (deg) for $\text{Cr}_4(\text{CH}_3\text{COO})_4((\text{CH}_3)_2\text{NCH}_2\text{CH}_2\text{O})_4$ (1)^a

Distances			
Cr(1)-Cr(1')	2.531 (2)	O(2)-C(1)	1.258 (6)
-O(1)	2.033 (4)	O(3)-C(4)	1.459 (8)
-O(2)	2.033 (4)	N(1)-C(3)	1.438 (9)
-O(3)	1.999 (4)	-C(5)	1.473 (9)
-O(3''')	2.007 (4)	-C(6)	1.509 (9)
-N(1)	2.444 (5)	C(1)-C(2)	1.534 (8)
O(1)-C(1)	1.245 (7)	C(3)-C(4)	1.48 (1)
Angles			
Cr(1')-Cr(1)-O(1)	85.3 (1)	Cr(1)-O(2)-C(1)	122.2 (4)
-O(2)	85.4 (1)	Cr(1)-O(3)-Cr(1'')	113.7 (2)
-O(3)	92.0 (1)	-C(4)	121.7 (4)
-O(3''')	95.8 (1)	Cr(1'')-O(3)-C(4)	112.8 (4)
-N(1)	172.2 (2)	Cr(1)-N(1)-C(3)	99.6 (4)
O(1)-Cr(1)-O(2)	87.1 (2)	-C(5)	112.1 (5)
-O(3)	177.2 (2)	-C(6)	113.5 (4)
-O(3''')	91.7 (2)	C(3)-N(1)-C(5)	112.6 (7)
-N(1)	88.1 (2)	-C(6)	108.7 (7)
O(2)-Cr(1)-O(3)	92.1 (2)	C(5)-N(1)-C(6)	109.9 (7)
-O(3''')	178.3 (2)	O(1)-C(1)-O(2')	123.9 (6)
-N(1)	98.5 (2)	-C(2)	118.3 (6)
O(3)-Cr(1)-O(3''')	89.2 (1)	O(2')-C(1)-C(2)	117.9 (6)
O(3)-Cr(1)-N(1)	94.6 (2)	N(1)-C(3)-C(4)	112.6 (7)
O(3''')-Cr(1)-N(1)	80.2 (2)	O(3)-C(4)-C(3)	112.8 (6)
Cr(1)-O(1)-C(1)	122.4 (4)		

^a Numbers in parentheses are the estimated standard deviations

atoms of the $\text{OCH}_2\text{CH}_2\text{NMe}_2$ groups present, while Figure 2 presents another view of the molecule in which the con-

Table V. Bond Distances (Å) and Angles (deg) Involving Non-Hydrogen Atoms in $\text{Cr}((\text{pz})_2\text{BEt}_2)_2$ (2)^a

Distances			
Cr-N(11)	2.058 (4)	C(1)-C(2)	1.527 (9)
-N(21)	2.061 (4)	-B	1.603 (9)
N(11)-N(12)	1.361 (6)	C(3)-C(4)	1.523 (8)
-C(13)	1.342 (8)	-B	1.625 (9)
N(12)-C(11)	1.343 (7)	C(11)-C(12)	1.375 (7)
-B	1.571 (8)	C(12)-C(13)	1.385 (7)
N(21)-N(22)	1.365 (6)	C(21)-C(22)	1.372 (7)
-C(23)	1.335 (7)	C(22)-C(23)	1.384 (8)
N(22)-C(21)	1.360 (8)		
-B	1.587 (9)		
Angles			
N(11)-Cr-N(11')	180.0 (2)	C(21)-N(22)-B	131.9 (5)
-N(21)	88.9 (2)	C(2)-C(1)-B	114.7 (5)
-N(21')	91.1 (2)	C(4)-C(3)-B	113.8 (5)
N(21)-Cr-N(21')	180.0 (2)	N(12)-C(11)-C(12)	108.8 (4)
Cr-N(11)-N(12')	121.8 (3)	C(11)-C(12)-C(13)	105.5 (4)
-C(13)	129.3 (4)	N(11)-C(13)-C(12)	109.4 (5)
N(12)-N(11)-C(13)	107.5 (4)	N(22)-C(21)-C(22)	110.3 (5)
N(11)-N(12)-C(11)	108.8 (4)	C(21)-C(22)-C(23)	103.8 (5)
-B	120.4 (4)	N(21)-C(23)-C(22)	111.2 (5)
C(11)-N(12)-B	130.8 (5)	N(12)-B-N(22)	103.3 (5)
Cr-N(21)-N(22)	121.6 (3)	-C(1)	109.5 (5)
-C(23)	130.8 (3)	-C(3)	110.9 (5)
N(22)-N(21)-C(23)	107.3 (5)	N(22)-B-C(1)	109.6 (5)
N(21)-N(22)-C(21)	107.4 (5)	-C(3)	107.8 (5)
-B	120.5 (5)	C(1)-B-C(3)	115.0 (5)

^a Numbers in parentheses are the estimated standard deviations in the last significant digits.

necting carbon atoms are also shown. Bond distances and angles are listed in Table IV. Because of the high symmetry, relatively few of these are required to constitute a complete set of independent values.

The $\bar{4}$ (or S_4) symmetry axis is a perpendicular bisector of both Cr-Cr bonds. The set of bridging oxygen atoms is planar within experimental error, though planarity is not required by the S_4 symmetry. The local symmetry of each Cr_2 unit and its ligands is essentially the same as that characteristic of quadruply bonded $\text{M}_2\text{X}_8\text{L}_2$ units in general. Each Cr atom is surrounded by a set of six nearest neighbors that form a distorted octahedron. There are four oxygen atoms that form a virtually square array with the Cr atom at the center. Perpendicular to this plane are the Cr-Cr bond and the Cr-N bond. Thus, it is reasonable to compare the local Cr_2 unit and its associated ligands in this molecule with molecules such as

$\text{Cr}_2(\text{O}_2\text{CH})_4(\text{py})_2$ and $\text{Cr}_2(\text{O}_2\text{CCF}_3)_4(\text{Et}_2\text{O})_2$, whose structures have previously been reported.⁶

The Cr-Cr distance, 2.531 (2) Å, is still within the previously established range for Cr⁴⁺-Cr bonds in $\text{Cr}_2(\text{O}_2\text{CR})_4\text{L}_2$ compounds, but just barely. The upper end of that range is provided by $\text{Cr}_2(\text{O}_2\text{CCF}_3)_4(\text{Et}_2\text{O})_2$, in which the Cr-Cr distance is 2.541 (1) Å. The great length of the Cr-Cr distance in the present case cannot be attributed to any unusual strength of the axial Cr-N bonds, at least insofar as such strength might be indicated by bond length. The Cr-N bond length here, 2.444 (5) Å, is much greater than that in $\text{Cr}_2(\text{O}_2\text{CH})_4(\text{py})_2$, in which a Cr-N bond length of 2.308 (3) Å is associated with a Cr-Cr bond length of 2.408 (1) Å. Evidently the replacement of two bridging CH_3CO_2 groups by the $\text{O}_2\text{Cr}-\text{CrO}_2$ unit allows, or causes, an increase in the Cr-Cr bond length, regardless of the other factors at work.

Compound 2. The reaction of $\text{Cr}_2(\text{O}_2\text{CCH}_3)_4$ with ligand or ligand precursor molecules to give mononuclear chromium(II) (or occasionally Cr^{III}) products is, of course, well-known.¹ In the present case, the result is perhaps not surprising.

There is no known example of a $(\text{Cr}^4\text{Cr})^{4+}$ compound that does not contain bridging ligands except for the $[\text{Cr}_2(\text{CH}_3)_8]^{4+}$ ion. Thus, given that the four CH_3CO_2^- ligands are to be replaced by the $[\text{B}(\text{pz})_2\text{Et}_2]^-$ ligands, a severing of the Cr⁴⁺-Cr bond to give two square $\text{Cr}((\text{pz})_2\text{BEt}_2)_2$ molecules is a predictable result. The fact that $\text{Cr}((\text{pz})_2\text{BEt}_2)_2$, which is a truly four-coordinate complex, is also stable in air is of interest and may be due, in part, to a protective effect resulting from the positioning of one ethyl group over each side of the CrN_4 plane, as shown in Figure 3. The bond lengths and angles, listed in Table V, are all of expected magnitudes.

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Supplementary Material Available: Listings of thermal parameters for non-hydrogen atoms and positional and thermal parameters for hydrogen atoms for 1 and 2, bond lengths and angles involving hydrogen atoms for 2, and observed and calculated structure factors for 1 and 2 (14 pages). Ordering information is given on any current masthead page.

(6) Cotton, F. A.; Extine, M. W.; Rice, G. W. *Inorg. Chem.* 1978, 17, 176.