Table VI. Structural Parameters of the $M_2(\mu\text{-Cl})_3$ Unit

	$R(M-M)$, Å	$\angle(M-Cl-M)$, deg	$R(M-(\mu\text{-Cl}))$, Å	ref
$Ti_2Cl_9{}^{3-}$	3.43	86.7	2.493	*a*
$V_2Cl_3(THF)_6{}^+$	2.97	73.8	2.477	this work
$Cr_2Cl_9{}^{3-}$	3.12	76.4	2.52	*b*

a Kirstenmacher, T. J.; Stucky, G. D. *Inorg. Chem.* **1971**, *10*, 122. *b* Wessel, G. J.; Ijdo, D. J. W. *Acta Crystallogr.* **1957**, *10*, 4661.

Whereas there is no direct V–V bonding in the d^2–d^2 dimer $[CpV(O_2CR)_2]_2$ (V···V $\cong$ 3.7 Å),[24,25] there are structural symptoms of incipient V–V bonding in $[V_2Cl_3(THF)_6]^+$. Thus, while the observed V–V distance of 2.97 Å is much longer than the 2.73 Å in $V_2(CO)_8(\mu\text{-PMe}_2)_2$,[26] the 2.46 Å in $Cp_2V_2(CO)_5$,[27] the 2.439 Å in $Cp_2V_2(C_8H_8)$,[5] and the 2.20 Å in $V_2[C_6H_3(OMe)_2]_4$,[28] it is shorter than the 3.255 Å in $[CpV(\mu\text{-Cl})PEt_3]_2$.[29] A useful standard for comparison is the set of isostructural face-shared octahedra listed in Table VI. The metal–metal separation in the d^0–d^0 dimer $[Ti_2Cl_9]^-$ is nearly 0.5 Å longer than in the d^3–d^3 species $[V_2Cl_3(THF)_6]^+$. Perhaps the most revealing comparison is with the d^3–d^3 dimer $Cr_2Cl_9{}^{3-}$, which has a longer M–M distance in spite of the fact that Cr(III) is inherently smaller than V(II). The contracted V–V distance suggests some bonding interaction between the metal atoms, which may be expected to manifest itself as magnetic coupling between the two d^3 centers. Antiferromagnetism is indeed observed, with an exchange integral ($-J$ = 75 cm^{-1}, calculated from the magnetic susceptibility between 30 and 270 K; Table I) considerably larger than in the isoelectronic $Cr_2Cl_6{}^{3-}$ systems[30] ($-J$ varies with the counterion between 4 and 11 cm^{-1}). A smaller exchange integral ($-J$ = 40 cm^{-1}) is also found for $[Et_3N]_3[V_2Cl_9]$, a V(III) complex anion with three halogen bridges.[31] In the V(II) complex the metal orbitals are larger and can give a better overlap, leading to a stronger magnetic interaction and the observed increase in the exchange integral, $-J$. For $[CpV(\mu\text{-Cl})PEt_3]_2$ (V–V = 3.255 Å[29]), the exchange integral ($-J$ = 110 cm^{-1}) is considerably larger, despite the fact that the distance between the metal centers is 0.3 Å longer. In the V(III) carboxylates, $[CpV(O_2CR)_2]_2$, the exchange between the d^2 centers is even stronger ($-J$ varies between 110 and 165 cm^{-1}, depending on R[32]), despite the fact that the V–V distance (3.63–3.70 Å)[24,25] is 0.6 Å larger than twice the covalent radius of vanadium. Strong superexchange through the four bridging carboxylate ligands was suggested as a mechanism for this unusually strong magnetic interaction.[25,32]

The electronic spectrum of the $[V_2Cl_3(THF)_6]^+$ cation in CH_2Cl_2/THF (340–1200 nm) is typical of spin-allowed, Laporte-forbidden transitions. The spectrum is remarkably similar to that reported for authentic (from electrochemical reduction) $VCl_2\cdot2THF$ in THF.[18] This, taken with the observed antiferromagnetic for the latter,[18] leads us to suggest for authentic $VCl_2\cdot2THF$ in solution a structure similar to the one reported here, e.g. $[V_2Cl_3(THF)_6]Cl$. When the observed spectrum is idealized to V(II) in an octahedral ligand field, the maxima are assigned as $^4A_{2g} \rightarrow {}^4T_{2g}$ (ν = 10.0 × 10^3 cm^{-1}), $^4A_{2g} \rightarrow {}^4T_{1g}(F)$ (ν = 15.6 × 10 cm^{-1}), and $^4A_{2g} \rightarrow {}^4T_{1g}(P)$ (ν = 24.5 × 10^3 cm^{-1}). From these data, values for $10Dq$ (10.0 × 10^3 cm^{-1}) and the electron repulsion parameter, B (814 cm^{-1}), can be calculated.[33] These predict $^4A_{2g} \rightarrow {}^4T_{1g}(P)$ at 26.2 × 10^3 cm^{-1}. The actual local symmetry (C_{3v}) is evident as shoulders in the high-energy band due to splitting of the $T_{1g}(P)$ level, while the lower energy bands are too broad ($\Delta\nu_{1/2}$ = (2–3) × 10^3 cm^{-1}) for normal high-symmetry d–d transitions.

Conclusions. The precipitated solid from the zinc reduction of $VCl_3\cdot3THF$ has precisely the average solution composition resulting from this one-electron reduction: $VCl_2\cdot{}^1/_2ZnCl_2\cdot3THF$. The isolation of this unexpectedly complicated product is probably due more to its low solubility than to its exceptional stability. Spin coupling within the two d^3 centers in the cation is significant compared to analogous systems, but nevertheless only comparable to thermal energies in the range 100–300 K and thus weaker than, for example, a hydrogen bond. At the same time, this weak interaction is in part responsible for the dimeric aggregation that is proposed to facilitate multielectron reduction of N_2 by V(II), thereby avoiding high-energy or radical nitrogenous intermediates.[7]

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Thionitrosyl and Bridging Sulfide Complexes of Dicarbonyl[hydridotris(3,5-dimethylpyrazolyl)borato]metal (Metal = Molybdenum and Tungsten)

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The few transition-metal–thionitrosyl complexes that are known have displayed some remarkable chemical and physical properties in relation to those of their more common metal-nitrosyl analogues.[1-8] There is considerable interest in ex-

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tending the limited range of known metal–thionitrosyl species, particularly through triads of the transition-metal series. The reagent $N_3S_3Cl_3$ has been shown to react with $CpCr(CO)_3^-$ ($Cp = \eta^5$-$C_5H_5^-$) in THF to give $CpCr(CO)_2NS^{1,2}$ and $(CpCr(CO)_2)_2(\mu$-$S)^3$ but only serves to oxidize the analogous Mo and W anions to the $CpM(CO)_3Cl$ and $(CpM(CO)_3)_2$ complexes.[9] We have investigated alternatives to the cyclopentadienyl systems that might help to inhibit the apparent oxidizing power of $N_3S_3Cl_3$ and therefore provide a complete series of group 6 NS complexes for characterizing metal–NS interactions. An attractive approach was to use the polypyrazolylborate type ligand, introduced some years ago by Trofimenko[10–12] and further investigated by McCleverty.[13,14] The hydridotris(pyrazolyl)borate ligand is similar to cyclopentadienyl in its formal charge and effective occupation of three coordination sites at the metal. Complexes with this ligand have been shown in some cases to surpass the stability and chemical diversity of their Cp analogues.[15] More recently, the work of Enemark and co-workers has shown the ability of the $HB(Me_2Pz)_3^-$ ligand to stabilize a variety of mononuclear oxomolybdenum(V) complexes[16] ($HB(Me_2Pz)_3^- =$ hydridotris(3,5-dimethylpyrazolyl)borate). It was our objective to determine whether the $HB(Me_2Pz)_3^-$ ligand could retard oxidation of the tricarbonyl anions and allow the production of the NS complexes.

The potassium or tetraethylammonium salts of $HB(Me_2Pz)_3M(CO)_3^-$ (M = Mo, W) react in THF with $N_3S_3Cl_3$ in a way similar to the Legzdins synthesis of $CpCr(CO)_2NS$. As in the synthesis of $CpCr(CO)_2NS$, a bridging sulfide complex is also produced in Scheme I.

In a typical preparation 1.3 g of $K^+HB(Me_2Pz)_3Mo(CO)_3^-$ (2.5 mmol)[11,13] was dissolved in dry, N_2-saturated THF (40 mL), and the solution was cooled to 0 °C. To this rapidly stirred slurry was added dropwise a solution of 0.2 g of N_3-S_3Cl_3[17] (0.83 mmol) in 10 mL of THF. The reaction evolved gas and turned a dark reddish brown color. The reaction mixture was allowed to warm to room temperature over a 1-h period. The solvent was then removed in vacuo, and the residue was slurried in a minimum amount of benzene and filtered through a short (2 × 4 cm) alumina column (activity III). The benzene eluent was concentrated in vacuo to a saturated solution, and 5 mL portions were syringed to the top of a medium-pressure chromatography column (1.5 × 25 cm) packed with silica gel.[18] The column was eluted with a 1:1

mixture of benzene/hexane. The first red band through the column was the thionitrosyl complex followed by the green bridging sulfide complex.[19] Several other colored bands followed but were found to contain no carbonyl IR bands and were not characterized. The individual collections were combined for each complex and were pumped to dryness. The final products were obtained by recrystallization from CH_2Cl_2/pentane. Yield: $HB(Me_2Pz)_3Mo(CO)_2NS$, 0.30 g (24%); $(HB(Me_2Pz)_3Mo(CO)_2)_2(\mu$-$S)$, 0.17 g (15%). Anal.[20] Calcd for $C_{17}H_{22}N_7O_2SBMo$: C, 41.23; H, 4.48; N, 19.80. Found: C, 41.20, H, 4.37; N, 20.11. Calcd for $C_{34}H_{44}N_{12}O_4SB_2Mo_2$: C, 43.89; H, 4.77; N, 18.07. Found: C, 43.91; H, 4.63; N, 17.77.

The tungsten complexes were prepared in a similar way. Yield: $HB(Me_2Pz)_3W(CO)_2NS$, 10%; $(HB(Me_2Pz)_3W(CO)_2)_2(\mu$-$S)$, ca. 1%. Anal. Calcd for $C_{17}H_{22}N_7O_2SBW$: C, 35.01; H, 3.80; N, 16.81. Found: C, 35.22; H, 3.60; N, 16.53. The relatively small amounts of the W–μ-sulfide complex isolated prevented a satisfactory analysis, but the complex was identified on the basis of its characteristics (IR spectrum, color, NMR) similar to those of the Mo analogue (vide infra).

Attempts at producing the $HB(Me_2Pz)_3Cr(CO)_2NS$ complex from $N_3S_3Cl_3$ and the corresponding Cr carbonyl anion have so far been unsuccessful. It is apparent from the work of Trofimenko and work in this laboratory that the $HB(Me_2Pz)_3Cr(CO)_2NO$ complex cannot be obtained from the treatment of the chromium tricarbonyl anion with ClNO but can only be produced by using an alkyl nitrite reagent.[11] McCleverty has commented on the susceptibility of the tris(pyrazolyl)borate ligand to oxidation in certain complexes.[13] The smaller metal in the chromium complex may be overprotected by the bulky hydridotris(3,5-dimethylpyrazolyl)borate ligand, causing competing reactions to occur preferentially.

The dark violet thionitrosyl compounds of Mo and W dissolve in solvents such as $CHCl_3$, CH_2Cl_2, C_6H_6, and acetone to give blood red solutions that are stable for weeks in air. The Mo–NS complex decomposes at temperatures above 240 °C, while the W–NS complex decomposes at temperatures above 280 °C. In comparison, the corresponding nitrosyl complexes decompose at temperatures above 270 and 335 °C, respectively. Like their nitrosyl analogues, the thionitrosyl complexes sublime in vacuo at temperatures above 200 °C. The IR and ^1H NMR data for these complexes, along with the data for the corresponding nitrosyl complexes (this work), are given in Table I. Their IR spectra show the expected dicarbonyl pattern of two strong bands, and the [hydridotris(3,5-dimethylpyrazolyl)borato]metal IR resonances match very closely with those of the corresponding nitrosyl complex. The NS stretch appears in the 1125–1140-cm^{-1} region (Table I). The green bridging sulfide complexes show an IR pattern for the carbonyl ligands similar to that of the $(CpCr(CO)_2)_2(\mu$-$S)$ complex in both solid (KBr) and solution spectra.

The 250-MHz proton NMR data for the NS complexes show characteristics similar to other systems containing the $HB(Me_2Pz)_3$ ligand.[13] The thionitrosyl complexes have a plane of symmetry as shown in Figure 1, and the proton resonances appear as sharp signals with relative intensities of 2:1, representing the two pyrazolyl groups cis to the thionitrosyl substituent and the one pyrazolyl group trans to the thionitrosyl. It is clear that the hydridotris(3,5-dimethyl-

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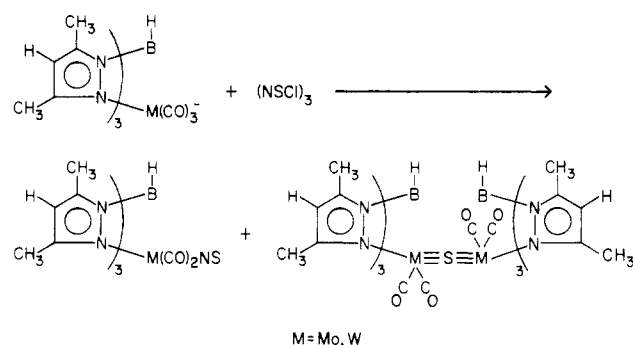(20) Microanalysis by MicAnal, Tucson, AZ 85717.

Scheme I

M = Mo, W

Table I. Spectroscopic Data

	IR,^{a} cm^{-1}	^{1}H NMR,^{b,d} δ	assgnt^e
HB(Me$_2$Pz)$_3$Mo(CO)$_2$NO	CO:^{c} 1997, 1905	5.84 (2)	b
	NO:^{c} 1650	5.73 (1)	b'
		2.46 (6)	c
		2.36 (6)	a
		2.33 (3)	c'
		2.32 (3)	a'
HB(Me$_2$Pz)$_3$Mo(CO)$_2$NS	CO:^{c} 2003, 1924	5.88 (2)	b
	NS:^{f} 1125, 1136	5.66 (1)	b'
		2.62 (6)	c
		2.37 (6)	a
		2.28 (3)	c'
		2.23 (3)	a'
HB(Me$_2$Pz)$_3$W(CO)$_2$NO	CO:^{c} 1993, 1884	5.88 (2)	b
	NO:^{c} 1640	5.76 (1)	b'
		2.49 (6)	c
		2.39 (9)	a, c'
		2.32 (3)	a'
HB(Me$_2$Pz)$_3$W(CO)$_2$NS	CO:^{c} 1988, 1902	5.92 (2)	b
	NS:^{f} 1127, 1140	5.70 (1)	b'
		2.66 (6)	c
		2.38 (6)	a
		2.29 (3)	c'
		2.26 (3)	a'
(HB(Me$_2$Pz)$_3$Mo(CO)$_2$)$_2$- (μ-S)	CO:^{g} 1984, 1929, 1891, 1868	5.76 (2)^h	b
		5.58 (1)	b'
		2.40 (6)	c
		2.23 (12)	a,^{i} c', a'
(HB(Me$_2$Pz)$_3$W(CO)$_2$)$_2$- (μ-S)	CO:^{g} 1962, 1902, 1863, 1838	5.77 (2)^j	b
		5.63 (1)	b'
		2.41 (6)	c
		2.29 (3)	c'
		2.27 (3)	a'
K$^+$(HB(Me$_2$Pz)$_3$)$^-$		5.83 (3)	b
		2.25 (18)	a, c

^{a} Perkin-Elmer 983 IR spectrophotometer. ^{b} Bruker WM-250 NMR, in CDCl$_3$, Me$_4$Si reference. ^{c} KBr pellet. ^{d} Integrations in parentheses. ^{e} See Figure 1 and text. ^{f} Two peaks observed in KBr pellet also observed in CH$_2$Cl$_2$ solutions. ^{g} CH$_2$Cl$_2$ solution. ^{h} Slightly broadened. ^{i} Broad. ^{j} Broad; resonance of protons a not observed.

pyrazolyl)borate ligand is not undergoing rotation on this time scale. The average proton resonances show small (0.02–0.03 ppm) deshielding upon proceeding from the nitrosyl to the thionitrosyl complexes and similar shifts when proceeding from Mo to W.

When the substituent indicated by X in Figure 1 is –S– (HB(Me$_2$Pz)$_3$(CO)$_2$), rotation about the M–S–M bond destroys the plane of symmetry. The resonance of protons b of the cis pyrazolyls (see Figure 1) of the bridging sulfide complexes are slightly broadened at room temperature for the Mo complex and are very broad for the W complex. Likewise, one of the methyl proton resonances (assigned as protons a) of the cis pyrazolyls is very broad for the Mo complex and could not be observed at room temperature for the W complex.
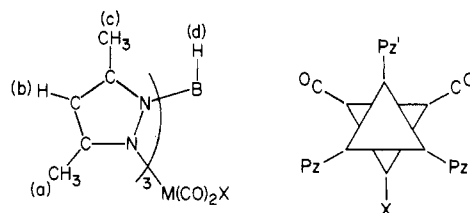

Figure 1. Proton labels in HB(3,5-Me$_2$Pz)$_3$M(CO)$_2$X complexes (X = NO, NS, or S(CO)$_2$M[HB(3,5-Me$_2$Pz)$_3$]).

The resonances of the protons on the trans pyrazolyls are not affected by rotation about the M–S–M bond and remain sharp in all spectra. It is interesting that the proton NMR of the (HBPz$_3$Mo(CO)$_2$)$_2$ dimer, with the much shorter distance between metal atoms, did not give evidence of rotation about the metal–metal bond.[15] Further multinuclear NMR and other characterization of these NS complexes in comparison to their NO analogues are part of a separate study.[21]

The main point is that the HB(Me$_2$Pz)$_3$M(CO)$_3^-$ anions with M = Mo and W allow simple production of NS substitution products while the cyclopentadienyl analogues do not. It is apparent that the nitrogen electron pair donors of the hydridotris(pyrazolyl)borate ligand are more effective than cyclopentadienyl at imposing pseudooctahedral electronic symmetry at the metal center,[14,15] and the nitrogens are probably better donors in comparison to cyclopentadienyl.[22–24] The steric requirements of HBPz$_3^-$ are somewhat similar to the pentamethylcyclopentadienyl ligand, and only one metal dimer complex with HBPz$_3^-$ ligands on the metals is presently known.[15] In the chemistry we report here, the pyrazolyl groups have methyl substituents in the 3 and 5 positions, and this greatly increases the steric requirements of the ligand. Curtis has commented on the importance of steric factors in facilitating decarbonylation,[25] and the steric crowding around the metal atom will make formation of (HB(Me$_2$Pz)$_3$M(CO)$_3$)$_2$ and HB(Me$_2$Pz)$_3$M(CO)$_3$Cl less favorable. The steric bulk of the HB(Me$_2$Pz)$_3^-$ ligand apparently protects the metal from competing oxidation reactions while facilitating smooth displacement of the carbonyl ligand with the thionitrosyl species. These features of [hydridotris(3,5-dimethylpyrazolyl)borato]metal chemistry promise many additional interesting applications of this ligand in inorganic and organometallic chemistry.

Registry No. HB(Me$_2$Pz)$_3$Mo(CO)$_2$NO, 24899-04-5; HB-(Me$_2$Pz)$_3$Mo(CO)$_2$NS, 90605-26-8; HB(Me$_2$Pz)$_3$W(CO)$_2$NO, 24899-06-7; HB(Me$_2$Pz)$_3$W(CO)$_2$NS, 90605-27-9; (HB-(Me$_2$Pz)$_3$Mo(CO)$_2$)$_2$(μ-S), 90641-19-3; (HB(Me$_2$Pz)$_3$W(CO)$_2$)$_2$(μ-S), 90623-45-3; K$^+$HB(Me$_2$Pz)$_3$Mo(CO)$_3^-$, 90623-43-1; (Et$_4$N)[HB-(Me$_2$Pz)$_3$Mo(CO)$_3$], 22357-70-6; K$^+$HB(Me$_2$Pz)$_3$W(CO)$_3^-$, 90623-44-2; (Et$_4$N)[HB(Me$_2$Pz)$_3$W(CO)$_3$], 22357-72-8; K$^+$(HB(Me$_2$Pz)$_3$)$^-$, 17567-17-8; N$_3$S$_3$Cl$_3$, 18428-81-4.

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