each type. They attributed the variations to steric effects. In the present case we believe that electronic factors are likely to be at work, but it is not clear why a mixed species results. There would appear to be room for the two dmso ligands both to adopt the same bonding mode (S- or O-). The structure of this molecule exposes the inadequacy of our understanding of the behavior of ambidentate ligands.

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Bis(benzenethiolato)nitrosyl[tris(3,5-dimethyl-1-pyrazolyl)hydroborato]-molybdenum(II)

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Abstract. $[Mo(C_{15}H_{22}BN_6)(C_6H_5S)_2(NO)]$, $M_r = 641{\cdot}5$, monoclinic, $P2_1/n$, $a = 10{\cdot}898\,(1)$, $b = 18{\cdot}484\,(2)$, $c = 15{\cdot}989\,(2)$ Å, $\beta = 109{\cdot}81\,(1)°$, $V = 3030{\cdot}2$ Å^3, $Z = 4$, $D_x = 1{\cdot}41$, $D_m = 1{\cdot}41$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71073 Å, $\mu = 5{\cdot}9$ cm^{-1}, $F(000) = 1320$, $T = 293$ K, $wR = 0{\cdot}047$ for 3997 observed reflections. In each complex, the Mo atom is six-coordinate, ligated by a terminal NO group, a tridentate pyrazolylborate ligand and two benzenethiolate ligands. The structure is compared with that of similar complexes and the Mo—S bonding is discussed.

Introduction. The unusual stability of monomeric $[CpMo(NO)(SC_6H_5)_2]$ has been ascribed to $d\pi$–$p\pi$ bonding interactions between the thiolate lone pairs, primarily S 3p in character, and the empty Mo 4d_{xy} orbital (Ashby & Enemark, 1986). The extent of this interaction is revealed by the torsion angle ON—Mo—S—C; a torsion angle of 0 or 180° maximizes the d_{xy}–p overlap, whereas a torsion angle near 90° leads to an overlap of filled Mo d_{xz} and d_{yz} orbitals with S p orbitals and should not be favored. In $[CpMo(NO)(SC_6H_5)_2]$, these torsional angles are 12 and −174°. In the oxomolybdenum(V) complex $[\{HB(Me_2pz)_3\}MoO(SC_6H_5)_2]$ (Cleland, Barnhart, Yamanouchi, Collison, Mabbs, Ortega & Enemark, 1987) the d_{xz} and d_{yz} orbitals are empty and the complex adopts a structure with O—Mo—S—C torsional angles of −34 and −110°; similar torsion angles occur in $[\{HB(Me_2pz)_3\}MoO(OC_6H_5)_2]$, −49 and −90° (Kipke, Cleland, Roberts & Enemark, 1989). This conformation minimizes overlap of the S p orbital with the half-filled d_{xy} orbital and allows overlap with the empty d_{xz}, d_{yz} orbitals. It was also noted that the rotational conformations of the thiolate ligands place the S $p\pi$ orbitals in the same orientation as the $p\pi$ orbitals on the respective *trans* pyrazole ring, which raises the possibility that the electronic structure of the polypyrazolylborate ligand may influence the overall stereochemistry of the complex. We report here the structure of $[\{HB(Me_2pz)_3\}Mo(NO)(SC_6H_5)_2]$ and compare it to the previously described structures.

Experimental. The complex was prepared as described elsewhere (McCleverty, Drane, Bailey & Smith, 1983). Crystals were grown by slow evaporation of a dichloromethane–hexane solution of the complex.

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Green block, dimensions 0·40 × 0·40 × 0·30 mm mounted on a quartz fiber with epoxy. D_m by flotation ($ZnBr_2$ and H_2O). Syntex $P2_1$ diffractometer, cell constants from 24 reflections with $22 < 2\theta < 25°$. The unit cell of highest metric symmetry is orthorhombic, but axial photographs along the *a* axis do not have mirror symmetry. Consequently, the structure was solved in the monoclinic cell. Systematic absences identified space group $P2_1/n$. Data collection using $\theta/2\theta$ scans, $2\theta_{max} = 50°$, reflections with indices $hk \pm l$ collected. Three standard reflections collected every 97 reflections. No decay observed. No absorption corrections ($\mu = 5·9\ cm^{-1}$). 5531 unique reflections, 3997 with $F_o^2 > 3\sigma(F_o^2)$ used in the refinement. $R_{int} = 0·017$. Position of Mo atom determined from Patterson map, other non-H atoms from succeeding difference maps. H atoms included at calculated positions and not refined. Final refinement on *F* (3997 reflections, 352 parameters) resulted in $R = 0·031$, $wR = 0·047$, $S = 1·33$. Weights for each reflection were assigned as $w = 4F_o^2/[\sigma^2(I) + (pF_o^2)^2]$ with *p* set to 0·04. $(\Delta/\sigma)_{max} = 0·05$. $\Delta\rho_{max} = 0·31$, $\Delta\rho_{min} = -0·31\ e\ Å^{-3}$. Atomic scattering factors from Cromer & Waber (1974) and anomalous-dispersion factors from Cromer (1974). Programs *SDP* (Frenz, 1978) run on a MicroVAXII.

Discussion. The six-coordinate Mo atom is ligated in a pseudo-octahedral coordination geometry. One face of the octahedron is occupied by the three N atoms of the pyrazolylborate ligand, the opposite face by the S atoms of the benzenethiolate ligands and by the N of the linear nitrosyl group [Mo—N—O = 179·1 (2)°]. The nitrosyl group exerts only a weak *trans* influence, with Mo—N(11) [2·244 (2) Å] *trans* to Mo—NO lengthened 0·03 Å relative to Mo—N(21) [2·205 (2) Å] and Mo—N(31) [2·216 (2) Å]. Fig. 1 illustrates the structure and the labeling of the ligand atoms; Table 1 lists the final positional and equivalent isotropic thermal parameters;* Table 2 gives selected bond distances and angles for the complex. The bond distances and angles in the pyrazolylborate ligand are unremarkable.

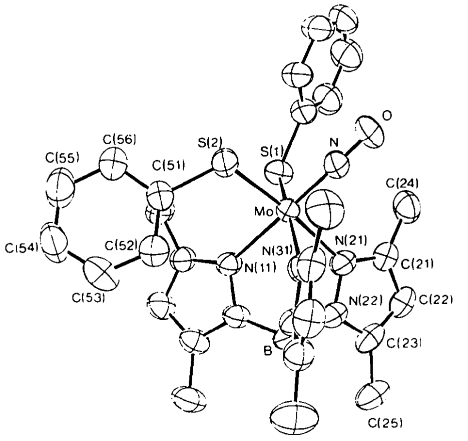

Fig. 1. View of [{$HB(Me_2pz)_3$}Mo(NO)(SC_6H_4)$_2$] showing the atomic labeling scheme. H atoms are omitted for clarity. The numbering of the atoms in the unlabeled pyrazole and benzene rings parallels that shown for the labeled rings.

Table 1. *Positional and equivalent isotropic thermal parameters*

Anisotropically refined atoms are given in the form of the equivalent isotropic displacement parameter defined as $B_{eq} = 8\pi^2(U_{11} + U_{22} + U_{33})/3$.

	x	*y*	*z*	B_{eq}(Å^2)
Mo	−0·00362 (2)	0·15329 (2)	0·23791 (2)	3·239 (5)
S(1)	−0·07034 (9)	0·22985 (5)	0·11364 (6)	4·18 (2)
S(2)	−0·09387 (8)	0·20935 (5)	0·33717 (6)	4·20 (2)
O	0·2360 (2)	0·2363 (2)	0·3310 (2)	6·05 (7)
N	0·1400 (2)	0·2023 (2)	0·2934 (2)	3·95 (6)
N(11)	−0·1866 (2)	0·0935 (1)	0·1616 (2)	3·45 (6)
N(12)	−0·1806 (3)	0·0183 (1)	0·1599 (2)	3·58 (6)
N(21)	0·0865 (3)	0·0810 (2)	0·1655 (2)	3·69 (6)
N(22)	0·0462 (3)	0·0092 (1)	0·1516 (2)	3·69 (6)
N(31)	0·0532 (2)	0·0587 (2)	0·3279 (2)	3·65 (6)
N(32)	0·0113 (3)	−0·0097 (2)	0·2958 (2)	3·70 (6)
C(11)	−0·3136 (3)	0·1104 (2)	0·1251 (2)	3·76 (7)
C(12)	−0·3866 (3)	0·0483 (2)	0·1010 (2)	4·27 (8)
C(13)	−0·3018 (3)	−0·0085 (2)	0·1241 (2)	4·10 (8)
C(14)	−0·3656 (4)	0·1867 (2)	0·1136 (3)	5·08 (9)
C(15)	−0·3295 (4)	−0·0885 (2)	0·1155 (3)	5·6 (1)
C(21)	0·1756 (3)	0·0902 (2)	0·1253 (2)	4·00 (7)
C(22)	0·1923 (3)	0·0255 (2)	0·0869 (2)	4·51 (8)
C(23)	0·1113 (3)	−0·0241 (2)	0·1047 (2)	4·38 (8)
C(24)	0·2391 (4)	0·1610 (2)	0·1232 (3)	5·40 (9)
C(25)	0·0954 (4)	−0·1028 (2)	0·0798 (3)	5·7 (1)
C(31)	0·1114 (3)	0·0517 (2)	0·4168 (2)	4·40 (8)
C(32)	0·1047 (4)	−0·0195 (2)	0·4402 (2)	4·92 (9)
C(33)	0·0435 (3)	−0·0573 (2)	0·3629 (2)	4·56 (8)
C(34)	0·1732 (4)	0·1133 (3)	0·4763 (3)	6·0 (1)
C(35)	0·0170 (6)	−0·1363 (3)	0·3509 (3)	7·3 (1)
C(41)	0·0135 (3)	0·3135 (2)	0·1342 (2)	4·34 (8)
C(42)	0·0371 (4)	0·3509 (2)	0·2125 (3)	5·14 (9)
C(43)	0·1017 (4)	0·4170 (2)	0·2246 (3)	6·5 (1)
C(44)	0·1422 (4)	0·4448 (2)	0·1578 (4)	7·7 (1)
C(45)	0·1129 (5)	0·4084 (3)	0·0793 (4)	8·0 (1)
C(46)	0·0492 (4)	0·3437 (3)	0·0672 (3)	6·1 (1)
C(51)	−0·2437 (3)	0·1716 (2)	0·3359 (2)	3·92 (7)
C(52)	−0·2667 (3)	0·0979 (2)	0·3332 (2)	4·47 (8)
C(53)	−0·3863 (4)	0·0725 (2)	0·3332 (3)	5·8 (1)
C(54)	−0·4820 (4)	0·1195 (3)	0·3355 (3)	6·6 (1)
C(55)	−0·4598 (4)	0·1925 (3)	0·3385 (3)	6·5 (1)
C(56)	−0·3400 (3)	0·2195 (2)	0·3404 (3)	5·16 (9)
B(1)	−0·0496 (4)	−0·0205 (2)	0·1948 (3)	3·83 (9)

The two benzenethiolate ligands occupy different environments as indicated by the ON—Mo—S—C torsional angles which are −16 and −168°. These angles are essentially identical with those of [CpMo(NO)(SC_6H_5)$_2$], and quite different from those of the oxomolybdenum(V) complexes of $HB(Me_2pz)_3^-$ (Table 3). The similarity of the ON—Mo—S—C torsion angles for [CpMo(NO)(SC_6H_5)$_2$] and [{$HB(Me_2pz)_3$}Mo(NO)(SC_6H_5)$_2$] indicate that the electronic structure of the polypyrazolylborate ligand has little influence on the overall stereochemistry of

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* Lists of structure factors, anisotropic thermal parameters, bond distances and angles and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51901 (17 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 2. *Selected bond distances* (Å) *and angles* (°)

Numbers in parentheses are e.s.d.'s of the least significant digits.

Mo—S(1)	2·3452 (9)	Mo—N(31)	2·216 (2)
Mo—S(2)	2·3702 (9)	S(1)—C(41)	1·769 (3)
Mo—N	1·766 (2)	S(2)—C(51)	1·770 (3)
Mo—N(11)	2·244 (2)	O—N	1·193 (3)
Mo—N(21)	2·205 (2)		

S(1)—Mo—S(2)	102·92 (3)	N—Mo—N(11)	177·4 (1)
S(1)—Mo—N	94·80 (9)	N—Mo—N(21)	95·7 (1)
S(1)—Mo—N(11)	82·72 (6)	N—Mo—N(31)	94·2 (1)
S(1)—Mo—N(21)	89·23 (7)	N(11)—Mo—N(21)	83·53 (9)
S(1)—Mo—N(31)	164·75 (7)	N(11)—Mo—N(31)	88·03 (9)
S(2)—Mo—N	86·38 (8)	N(21)—Mo—N(31)	77·66 (9)
S(2)—Mo—N(11)	94·93 (6)	Mo—S(1)—C(41)	112·2 (1)
S(2)—Mo—N(21)	167·49 (7)	Mo—S(2)—C(51)	114·2 (1)
S(2)—Mo—N(31)	89·89 (7)	Mo—N—O	179·1 (2)

the complex. Whenever filled d_{xz} and d_{yz} orbitals exist, as in the $\{Mo(NO)\}^4$ complexes, the torsional angles adopt values near 0 and 180° to maximize d_{xy}–p overlap (Ashby & Enemark, 1986).

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Table 3. *Torsion angles X*—Mo—S—C (°) *in selected complexes*

			Reference
[{HB(Me$_2$pz]$_3$}Mo(NO)(SC$_6$H$_5$)$_2$]	−16	−168	This work
[(η^5-C$_5$H$_5$)Mo(NO)(SC$_6$H$_5$)$_2$]	12	−174	(1)
[{HB(Me$_2$pz)$_3$}MoO(SC$_6$H$_5$)$_2$]	−34	−110	(2)
[{HB(Me$_2$pz)$_3$}MoO(OC$_6$H$_5$)$_2$]	−49	−90	(3)

References: (1) Ashby & Enemark (1986). (2) Cleland *et al.* (1987). (3) Kipke *et al.* (1989).

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Structure of Triphenyltin(IV) 2-Aminophenyl Sulfide

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Abstract. (2-Aminobenzenethiolato-*S*)triphenyltin(IV), [Sn(C$_6$H$_5$)$_3$(C$_6$H$_6$NS)], $M_r = 474{\cdot}17$, monoclinic, $P2_1/a$, $a = 9{\cdot}074$ (1), $b = 19{\cdot}062$ (1), $c = 13{\cdot}119$ (1) Å, $\beta = 106{\cdot}78$ (1)°, $V = 2172{\cdot}5$ (4) Å^3, $Z = 4$, $D_x = 1{\cdot}44$ Mg m^{-3}, λ(Cu $K\alpha_1$) = 1·54056 Å, $\mu = 10{\cdot}55$ mm^{-1}, $F(000) = 952$, $R = 0{\cdot}044$, $wR = 0{\cdot}037$ for 2360 [$I_{\rm net} > 2{\cdot}5\sigma(I_{\rm net})$] reflections. The geometry at the Sn atom is tetrahedral and the 2-aminobenzenethiol ligand is monodentate. The compound is an uncommon example of a molecule containing an $^-$S—CH═CH—NH$_2$ ligand grouping that is neither chelating nor bridging.

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