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Preparation of Several Polypyrazolylborato Compounds. Structures of Two Phenyltrispyrazolylborato Complexes

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Several polypyrazolylborato compounds have been prepared. The structures of (phenyltrispyrazolylborato)(allyl)dicarbonylmolybdenum and (phenyltrispyrazolylborato)(cycloheptatriene)dicarbonylmolybdenum are described here. In both cases, the molybdenum atom is surrounded by an allyl, two carbonyl and three pyrazolyl groups. This provides the metal atom with a stable 18 electron configuration in contrast to a case where a similar arrangement of groups about the boron atom was used, namely, two phenyl and two pyrazolyl groups where the metal atom was left with a 16-electron configuration in its valence orbitals. The crystallographic data for $(\eta^3\text{-}C_3H_5)Mo(CO)_2pz_3BPh$ *(pz = 1-pyrazolyl, Ph = phenyl) are space group* $P\bar{1}$, $a = 10.021(2)$ Å, $b = 13.020(3)$ Å, $c = 8.546(3)$ Å, $\alpha = 95.70(2)^\circ$, $\beta = 107.42(2)^\circ$, $\gamma = 104.05(2)^\circ$, $V = 1014.0(5)$ Å^3, *and* $Z = 2$ *and those for* $(\eta^3\text{-}C_7H_7)Mo(CO)_2pz_3BPh$ *are space group* $P\bar{1}$, $a = 9.331(4)$ Å, $b = 10.459(3)$ Å, $c = 12.923(4)$ Å, $\alpha = 104.97(2)^\circ$, $\beta = 101.11(3)^\circ$, $\gamma = 98.15(3)^\circ$, $V = 1170.9(7)$ Å^3, *and* $Z = 2$.

Introduction

In the past few years, we have been interested in polypyrazolylborato molecules of the type (enyl)-$Mo(CO)_2pz_2BR_2$. When R is H [1] or C_2H_5 [2] and the enyl group is a η^3-allyl, it has been found that the electronically unsaturated molybdenum atom normally attains an 18-electron core by the formation of a 3 center-2 electron (3c–2e) bond formed by Mo···H–B or Mo···H–C interactions, respectively. However, in a crystallographic study of a molecule in which R is phenyl [3] we found no such interaction. That result was readily understood when a model of the molecule was built. It was found that a Mo···H–C interaction would demand sterically unacceptable orientations of the phenyl and pyrazolyl rings.

With this in mind, we attempted to prepare a molecule containing the diphenylbispyrazolylborato ligand while substituting the η^3-allyl by a cycloheptatrienyl ligand which could increase its hapticity from 3 to 5 and consequently, provide the molybdenum atom with a stable 18-electron configuration. Unfortunately, the desired compound never crystallized, despite persistent efforts, and an X-ray diffraction study was not possible.

We then decided to modify the set of four rings about the boron atom. An arrangement of groups similar to that present in the (pz_2BPh_2) ligand can be obtained by substitution of one phenyl group by a pyrazolyl group. Using the ligand Bpz_3Ph, two compounds, $(\eta^3\text{-}C_3H_5)Mo(CO)_2(pz_3BPh)$ and another in which the enyl group is C_7H_7 were prepared and their molecular structures determined. In these cases, stable 18-electron configurations for the molybdenum atoms were found.

Experimental

All operations were performed in an atmosphere of dry nitrogen or argon. Solvents were dried over potassium benzophenone and were distilled under nitrogen immediately before use or dried over molecular sieves and degassed by alternate freezing and thawing *in vacuo*. Infrared spectra were recorded on a Perkin–Elmer 237 spectrometer.

Synthesis of $(C_7H_7)Mo(CO)_2pz_2BPh_2$ *(1)*

$C_7H_7Mo(CO)_2I$ [4] (0.25 g) was stirred at room temperature with $Napz_2BPh_2$ [5] (0.30 g) in 3 ml of DMFA. The color changes rapidly from green to dark red. After 40 minutes the solvent is eliminated by vacuum, the benzene is added to the remaining red oil. This solution is filtered and chromatographed on neutral alumina using benzene as eluent. The dark orange compound that is obtained normally precipiated from the benzene solutions. IR (benzene) 1948 and 1870 cm^{-1} or 1960 and 1855 cm^{-1} in hexane.

Preparation of $(C_7H_7)Mo(CO)_2(pz)pz_2BPh_2$ *(2)*

This compound is easily obtained by addition of pyrazole to the previous compound. The reaction is instantaneous upon stirring in benzene. IR (benzene) 1935 and 1858 cm^{-1} or 1945 and 1820 cm^{-1} in hexane.

Synthesis of (η^3-C_3H_5)Mo(CO)$_2$pz$_3$BPh (3)

Li(pz$_3$BPh) was prepared by heating and stirring Li(H$_3$BPh) [5] (4.0 g) with pyrazole (9g) until gas evolution had ceased. Since pyrazole sublimes, a condenser attached to a steam line was used. After the reaction was completed, hot hexane was added to the reaction flask. After filtration, the white solid was heated under vacuum at 100 °C for about 6 hr.

For the preparation of (pz$_3$BPh)(η^3-C_3H_5)(CO)$_2$-Mo, Li(Pz$_3$BPh) (1.0 g) was added to a suspension of 1.00 g of Mo(CO)$_6$ in 15 ml of dry and oxygen free acetone or DMF. This was stirred and refluxed for about 20 minutes and allowed to return to room temperature. Allyl bromide (0.50 g) was then added and the solution refluxed until gas evolution had ceased. After this, the solvent was eliminated by vacuum and the residue was extracted with a few milliliters of benzene and chromatographed on neutral alumina using benzene as eluent. Crystals suitable for X-ray studies were grown from a dichloromethane solution.

Synthesis of (η^3-C_7H_7)Mo(CO)$_2$pz$_3$BPh (4)

Li(pz$_3$BPh) (1.0 g) was stirred at room temperature with C_7H_7Mo(CO)$_2$I (1.2 g) in 15 ml of DMF. After about 20 minutes the solvent was eliminated by vacuum and the residue was extracted with benzene and then chromatographed as in the previous compound.

Crystal Data and Structure Determination for (η^3-C_3H_5)Mo(CO)$_2$pz$_3$BPh

A yellow crystal of (phenyltrispyrazolylborato)-(allyl)dicarbonylmolybdenum measuring about 0.15 X 0.15 X 0.25 mm was mounted on a glass fiber and examined on a Syntex P$\bar{1}$ four-circle automated diffractometer. Axial photographs and ω scans of several intense reflections showed that the crystal was good. At small scattering angles the peak widths at half-height were $< 0.2°$.

For calculation of the lattice parameters, fifteen of the strongest reflections in the range $20° < 2\theta$ (Mo-Kα) $< 31°$ were selected to give a variety of crystal orientations. Based on angular settings for the reflections, the refined lattice parameters obtained from the Syntex software package are (Mo Kα, λ = 0.71073 Å) a = 10.021(2) Å, b = 13.020(3) Å, c = 8.546(3) Å, α = 95.70(2)°, β = 107.42(2)°, γ = 104.05(2)°, and V = 1014.0(5) Å^3. For the triclinic space group P$\bar{1}$ with Z = 2 and a molecular weight of 482.17 g mol^{-1}, the calculated density is 1.579 g cm^{-3}.

Intensity data were collected at 23 ± 1° using graphite-monochromatized Mo Kα radiation and a θ–2θ scan rate varying from 4 to 24°min, depending on the intensity of the reflection. Background measurements were made at both limits of each scan. Of the 3947 integrated intensities collected in the range $0° < 2\theta$ (Mo Kα) $< 52°$, 3108 unique observations with $I > 3\sigma(I)$ were retained as observed data and corrected for Lorentz and polarization effects. Three standard reflections measured every 100 data points were stable and showed no significant time dependence. The linear absorption coefficient of the compound is 6.7 cm^{-1} and no absorption correction was applied.

A three dimensional Patterson map showed the position of the molybdenum atom. A difference Fourier synthesis based on the refined molybdenum position revealed all the rest of the non-hydrogen atoms. All atoms were assigned isotropic thermal parameters and least-squares refinement then gave discrepancy indices of $R_1 = \Sigma||F_0| - |F_c||/\Sigma|F_0|$ = 0.061 and $R_2 = [\Sigma w(|F_0| - |F_c|)^2/\Sigma w|F_0|^2]^{1/2}$ = 0.080. Refinement was continued with anisotropic thermal parameters for the molybdenum atom and isotropic thermal parameters for the rest of the non-hydrogen atoms to convergence at R_1 = 0.053, R_2 = 0.071, and the error in an observation of unit weight = 1.6. No attempt was made to locate the hydrogen atoms. The function minimized was $\Sigma w(|F_0| - |F_c|)^2$ where p = 0.06 was used in the expression for the weights [7, 8]. Scattering factors were from ref. 9. Corrections for anomalous scattering by molybdenum were taken from Cromer and Liberman [10]. For the programs used, see reference 11. A final difference map was judged to be free of significant features. No unusual trends were observed in an analysis of $\Sigma w(|F_0| - |F_c|)^2$ as a function of $|F_0|$, reflection number, $\lambda^{-1} \sin\theta$, or various classes of indices. A list of observed and calculated structure factors is available from the Editor upon request.

Data and Structure Refinement for (η^3-C_7H_7)Mo-(CO)$_2$pz$_3$BPh.

A red–orange crystal measuring about 0.10 X 0.15 X 0.30 mm was mounted on a glass capillary.

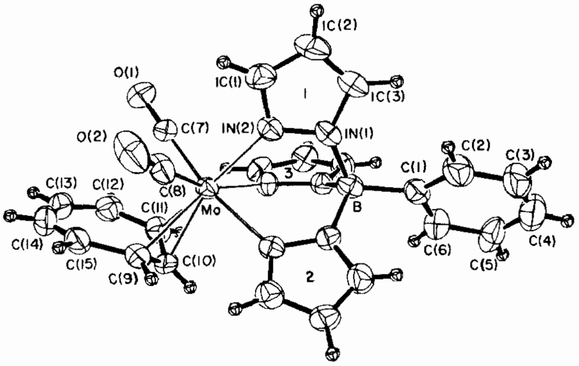

Fig. 1. An ORTEP view of the (η^3-C_7H_7)Mo(CO)$_2$(pz$_3$BPh) molecule. The atom numbering scheme used in the tables is defined. The atoms are represented by 50% probability thermal ellipsoids.

TABLE I. Positional and Thermal Parameters[a,b] for $[PhB(pz)_3][\eta^3\text{-}C_3H_5](CO)_2Mo$.

Atom	x	y	z	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
Mo	0.17582(5)	−0.15902(4)	0.17136(6)	59.8(6)	35.5(3)	103.8(9)	10.9(3)	28.1(5)	13.9(4)

Atom	x	y	z	B, Å^2	Atom	x	y	z	B, Å^2
1N(1)	−0.0948(5)	−0.3719(4)	0.0794(6)	2.76(8)	C(7)	0.3350(8)	−0.2193(6)	0.1944(9)	4.1(1)
2N(1)	−0.1682(5)	−0.2060(4)	0.0949(6)	2.55(8)	O(1)	0.4328(7)	−0.2581(5)	0.2257(8)	6.7(1)
3N(1)	−0.0977(5)	−0.2680(4)	−0.1541(6)	2.82(8)	C(8)	0.2656(7)	−0.1302(5)	0.4108(8)	3.2(1)
1N(2)	0.0498(5)	−0.3235(4)	0.1702(6)	2.72(8)	O(2)	0.3239(6)	−0.1139(4)	0.5555(7)	5.0(1)
2N(2)	−0.0362(5)	−0.1307(4)	0.1770(6)	2.68(8)	B	−0.1804(7)	−0.3053(6)	−0.0318(9)	2.8(1)
3N(2)	0.0475(5)	−0.2149(4)	−0.1026(6)	2.94(9)	C(1)	−0.3478(7)	−0.3661(5)	−0.1411(7)	3.0(1)
1C(3)	−0.1367(7)	−0.4687(5)	0.1218(8)	3.5(1)	C(2)	−0.3986(7)	−0.4762(6)	−0.2021(9)	3.7(1)
2C(3)	−0.2676(7)	−0.1902(5)	0.1630(8)	3.2(1)	C(3)	−0.5442(8)	−0.5255(6)	−0.3059(9)	4.3(1)
3C(3)	−0.1514(7)	−0.2894(5)	−0.3239(8)	3.6(1)	C(4)	−0.6394(8)	−0.4637(6)	−0.3533(9)	4.2(1)
1C(2)	−0.0196(8)	−0.4845(6)	0.2408(9)	4.1(1)	C(5)	−0.5895(8)	−0.3536(6)	−0.3003(9)	4.3(1)
2C(2)	−0.2031(8)	−0.1003(6)	0.2882(9)	3.8(1)	C(6)	−0.4452(8)	−0.3049(6)	−0.1957(9)	3.8(1)
3C(2)	−0.0403(8)	−0.2504(6)	−0.3843(9)	4.0(1)	C(9)	0.2649(8)	0.0304(6)	0.2656(9)	4.5(1)
1C(1)	0.0938(7)	−0.3927(5)	0.2670(8)	3.5(1)	C(10)	0.2593(8)	−0.0028(6)	0.0996(10)	4.4(1)
2C(1)	−0.0587(7)	−0.0666(5)	0.2935(8)	3.4(1)	C(11)	0.3600(9)	−0.0603(6)	0.0830(10)	4.7(1)
3C(1)	0.0807(7)	−0.2063(5)	−0.2450(8)	3.7(1)					

[a] All values include estimated standard deviations in parentheses. [b] The anisotropic temperature factors are of the form $\exp[-(\beta_{11}h^2 + \beta_{22}k^2 + \beta_{33}l^2 + 2\beta_{12}hk + 2\beta_{13}hl + 2\beta_{23}kl)]$. Values for the anisotropic factors have been multiplied by 10^4.

Cell constants were obtained by carefully centering 15 reflections (22 ± 1 °C) in the range $20° < 2\theta < 29°$. A least-squares refinement (Mo Kα, λ = 0.71073) gave a = 9.331(4) Å, b = 10.459(3) Å, c = 12.923(4) Å, α = 104.97(2)°, β = 101.11(3)°, γ = 98.15(3)°, and V = 1170.9(7) Å^3.

The data collection procedure and treatment of the data were similar to that given above. The linear absorption coefficient is 5.9 cm^{-1} and no correction for absorption was made. Of the 3074 reflections collected in the range $0° < 2\theta$ (Mo Kα) $< 45°$, 2384 unique observations with $I > 3\sigma(I)$ were retained as observed data. For the triclinic space group $P\bar{1}$, with Z = 2 and a molecular weight of 532.23 g mol^{-1}, the calculated density is 1.511 g cm^{-3}.

The position of the molybdenum atom was obtained from a three dimensional Patterson map. A difference Fourier map based on the refined position of the molybdenum atom gave the position of all the rest of the non-hydrogen atoms. In the last cycles of refinement, the positions of the hydrogen atoms were calculated at a distance of 0.95 Å from the carbon atoms and included in the calculation of structure factors. Refinement was continued, converging at R_1 = 0.030 and R_2 = 0.038, and the error in an observation of unit weight = 0.87. A final difference map was judged to be free of significant features. A table of observed and calculated structure factor amplitudes is available.

Results

The molecular structure of compound 4 is shown in Figure 1, which also defines the numbering scheme for both compounds 3 and 4. Positional and temperature parameters are listed in Tables I and II. The interatomic distances are given in Table III and the angles in Table IV.

Discussion

As indicated before, we were unable to obtain suitable crystals of $C_7H_7Mo(CO)_2pz_2BPh_2$. However, infrared spectra suggest that the compound can be prepared and is even stable to oxygen for short periods of time. Its behavior in the presence of excess pyrazole in solution is typical [12] for that type of compound. There is a slight change in color. This is due to coordination of the pyrazole molecule to the molybdenum atom. The pyrazole adduct was characterized by X-ray crystallography [13] and found to have a structure very similar to that of (diethylbispyrazolylborato) (pyrazolato) (η^3-allyl) (dicarbonyl)-molybdenum [14].

With respect to $(\eta^3\text{-}C_3H_5)Mo(CO)_2pz_3BPh$ and $(\eta^3\text{-}C_7H_7)Mo(CO)_2pz_3BPh$, it was found that their structures are essentially the same, insofar as possible.

TABLE II. Positional and Thermal Parameters[a,b] for $[PhB(pz)_3][\eta^3\text{-}C_7H_7](CO)_2Mo$.

Atom	x	y	z	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
Mo	−0.13570(4)	−0.11731(4)	0.16429(3)	90.5(6)	58.1(4)	41.7(3)	20.4(3)	21.1(3)	16.1(2)
1N(1)	−0.0482(4)	0.1338(3)	0.3839(3)	118(5)	82(4)	37(3)	38(4)	15(3)	9(3)
2N(1)	−0.1540(4)	0.1102(3)	0.2820(3)	87(5)	74(4)	49(3)	20(4)	12(3)	13(3)
3N(1)	−0.0732(4)	0.2046(3)	0.2158(3)	94(5)	59(4)	49(3)	22(4)	20(3)	17(3)
1N(2)	−0.1243(4)	0.0029(3)	0.3342(3)	118(6)	67(4)	47(3)	31(4)	26(3)	16(3)
2N(2)	0.1094(4)	−0.0225(3)	0.2219(3)	89(5)	71(4)	51(3)	26(3)	19(3)	17(3)
3N(2)	−0.1566(4)	0.0872(3)	0.1422(3)	90(5)	59(4)	45(3)	13(4)	10(3)	17(3)
1C(3)	−0.0875(5)	0.1812(5)	0.4788(4)	155(8)	110(6)	44(3)	45(6)	35(4)	13(4)
2C(3)	0.3040(6)	0.1403(5)	0.3103(4)	105(7)	97(6)	63(4)	15(5)	3(4)	3(4)
3C(3)	−0.1360(5)	0.3078(5)	0.2000(4)	125(7)	66(5)	69(4)	26(5)	24(5)	18(4)
1C(2)	−0.1831(7)	0.0819(6)	0.4946(4)	189(9)	142(7)	57(4)	55(7)	59(5)	30(5)
2C(2)	0.3586(5)	0.0303(5)	0.2689(4)	78(7)	121(7)	75(4)	33(5)	12(4)	9(4)
3C(2)	−0.2588(5)	0.2598(5)	0.1144(4)	97(7)	86(6)	79(4)	36(5)	10(4)	38(4)
1C(1)	−0.2046(6)	−0.0268(5)	0.4028(4)	128(7)	100(6)	63(4)	27(5)	34(4)	34(4)
2C(1)	0.2328(5)	−0.0686(5)	0.2155(4)	111(7)	88(5)	64(4)	36(5)	18(4)	22(4)
3C(1)	−0.2681(5)	0.1230(5)	0.0815(4)	82(6)	97(6)	54(3)	12(5)	4(4)	33(4)
C(7)	−0.3499(5)	−0.1727(4)	0.1379(4)	114(8)	76(5)	56(4)	7(5)	38(4)	10(3)
O(1)	0.5238(4)	−0.2052(4)	0.1282(3)	113(6)	134(5)	101(3)	−11(4)	44(3)	2(3)
C(8)	−0.1401(6)	−0.2692(5)	0.2253(4)	180(9)	70(5)	57(4)	33(5)	30(5)	11(4)
O(2)	−0.1539(6)	−0.3589(3)	0.2627(3)	412(11)	85(4)	80(3)	73(5)	84(5)	46(3)
B	0.0457(6)	0.2035(5)	0.3178(4)	102(7)	90(6)	42(4)	31(5)	17(4)	18(4)
C(1)	0.1432(5)	0.3525(5)	0.3832(4)	102(7)	90(6)	53(4)	31(5)	13(4)	5(4)
C(2)	0.2050(6)	0.3967(6)	0.4969(4)	149(8)	140(7)	61(4)	26(6)	12(5)	11(4)
C(3)	0.3002(6)	0.5228(7)	0.5486(5)	138(9)	168(8)	81(5)	9(7)	−3(6)	−26(6)
C(4)	0.3375(7)	0.6038(6)	0.4871(6)	144(9)	104(7)	134(7)	−5(6)	9(7)	−14(6)
C(5)	0.2843(6)	0.5618(5)	0.3743(6)	133(8)	78(6)	130(6)	−4(6)	6(6)	22(6)
C(6)	0.1896(6)	0.4373(5)	0.3235(5)	132(8)	99(6)	70(4)	5(6)	8(4)	13(4)
C(9)	−0.0638(6)	−0.3054(4)	0.0546(4)	112(7)	83(6)	60(4)	26(5)	26(4)	5(4)
C(10)	−0.0793(6)	−0.2022(5)	0.0041(4)	106(7)	86(5)	50(3)	−4(5)	36(4)	4(4)
C(11)	−0.2187(6)	−0.1766(5)	−0.0343(4)	154(8)	83(5)	41(3)	22(5)	21(4)	19(3)
C(12)	−0.3547(6)	−0.2746(5)	−0.0923(4)	120(7)	121(7)	50(3)	19(6)	17(4)	17(4)
C(13)	−0.3868(6)	−0.4073(5)	−0.1034(4)	140(8)	110(7)	59(4)	−13(6)	25(4)	−1(4)
C(14)	−0.2947(6)	−0.4826(5)	−0.0517(4)	181(9)	65(5)	80(5)	−7(6)	42(6)	10(4)
C(15)	−0.1617(6)	−0.4397(5)	0.0168(4)	154(8)	70(5)	67(4)	28(5)	41(5)	13(4)

[a] All values include estimated standard deviation in parentheses. [b] All values for the temperature factors have been multiplied by 10^4. The anisotropic temperature factors are of the form $\exp[-(\beta_{11}h^2 + \beta_{22}k^2 + \beta_{33}l^2 + 2\beta_{12}hk + 2\beta_{13}hl + 2\beta_{23}kl)]$.

In these molecules, the molybdenum atom is surrounded by an allyl, two carbonyl and three pyrazolyl groups. The pyrazolyl groups are bonded in mutually *cis* positions, as might have been expected considering the ability of the trispyrazolylborato ligands to form that arrangement [15–18]. This provides the metal atom a stable 18-electron configuration.

As pointed out in the Introduction, the orientation of the phenyl group in this molecule is of interest since the arrangement of atoms about the boron atom is somewhat similar to that found in electronically unsaturated molecules containing the pz_2BPh_2 ligand, such as $(pz_2BPh_2)[CH_2C(CH_3)CH_2](CO)_2Mo$ [3], where electron donation from a C–H bond to the molybdenum atom is prevented by steric problems, the angle between the planes formed by the phenyl group and pyrazolyl 1 is 33.7° for compound 3 and 35.2° for compound 4. This is not precisely what was expected. From model analysis of molecules containing the pz_2BPh_2 ligand, an angle near 90° was predicted. Apparently, the observed conformation avoids the crowding that would have resulted had the phenyl group been at a position near 90°.

The arrangement of groups about the boron atom observed in this structure appears inadequate for the molecule containing the pz_2BPh_2 ligand mentioned above due to steric interactions caused by the substitution of a 5 atom pyrazolyl ring by a 6 atom phenyl ring. This is coupled with the fact that the Mo–1N(2) distance is shorter than the expected Mo–C distance if a C–H interaction were to occur, thus allowing the formation of a less strained boat conformation formed by the Mo–N–N–B–N–N atoms.

Another thing that should be noticed in this molecule is the appearance of significantly different molybdenum–nitrogen distances ranging from

TABLE III. Interatomic Distances (Å).[a–d]

Atoms	Distance	Atoms	Distance
Mo–1N(2)	2.204(4)	C(7)–O(1)	1.152(5)
	2.202(5)		1.181(9)
Mo–2N(2)	2.256(4)	C(8)–O(2)	1.164(5)
	2.251(5)		1.171(8)
Mo–3N(2)	2.260(3)	C(1)–C(2)	1.396(7)
	2.251(5)		1.386(9)
Mo–C(7)	1.938(5)	C(2)–C(3)	1.400(8)
	1.908(7)		1.408(10)
Mo–C(8)	1.946(5)	C(3)–C(4)	1.356(9)
	1.928(6)		1.387(10)
Mo–C(9)	2.373(4)	C(4)–C(5)	1.376(9)
	2.371(8)		1.377(10)
Mo–C(10)	2.226(4)	C(5)–C(6)	1.384(7)
	2.222(8)		1.399(10)
Mo–C(11)	2.418(4)	C(6)–C(1)	1.394(7)
	2.343(8)		1.407(9)
Mo–B	3.423(5)	C(9)–C(10)	1 408(7)
	3.420(7)		1.421(10)
B–1N(1)	1.567(6)	C(10)–C(11)	1.389(7)
	1.558(8)		1.423(10)
B–2N(1)	1.554(6)	C(11)–C(12)	1.446(7)
	1.555(8)	C(12)–C(13)	1.341(7)
B–3N(1)	1.554(6)	C(13)–C(14)	1.431(8)
	1.560(8)	C(14)–C(15)	1.319(7)
B–C(1)	1 610(7)	C(15)–C(9)	1.468(7)
	1.606(9)		

Pyrazolyl Rings

	Ring 1	Ring 2	Ring 3
N(1)–N(2)	1.372(5)	1.362(5)	1.364(5)
	1.372(7)	1.371(6)	1.361(7)
N(2)–C(1)	1.333(6)	1.319(6)	1.336(5)
	1.346(8)	1.340(8)	1.361(8)
C(1)–C(2)	1.374(7)	1.378(7)	1.368(6)
	1.379(9)	1.389(9)	1.370(9)
C(2)–C(3)	1.355(7)	1.351(7)	1.360(7)
	1.377(9)	1.378(9)	1.371(9)
C(3)–N(1)	1.340(6)	1.345(6)	1.341(6)
	1.348(8)	1.337(8)	1.361(8)

[a] In cases where two distances are given, the first value corresponds to [PhB(pz)$_3$][η^3-C$_7$H$_7$](CO)$_2$Mo and the second value corresponds to [PhB(pz)$_3$][η^3-C$_3$H$_5$](CO)$_2$Mo. [b] Numbers in parentheses are the estimated standard deviations in the least significant digits. [c] Atoms are labeled as in Figure 1. [d] Rings 1, 2 and 3 are identified in Figure 1 by the numbers on the pyrazolyl rings.

TABLE IV. Bond Angles (deg.)

Atoms	Angle	Atoms	Angle
Mo–C(7)–O(1)	176.2(6)	C(1)–B–3N(1)	111.6(4)
	172.7(7)		107.8(5)
Mo–C(8)–O(2)	174.6(6)	B–C(1)–C(2)	124.0(5)
	177.7(5)		123.1(5)
Mo–C(10)–C(9)	77.9(3)	B–C(1)–C(6)	119.2(4)
	77.8(4)		119.3(6)
Mo–C(10)–C(11)	80.4(3)	C(6)–C(1)–C(2)	116.1(5)
	76.5(5)		117.2(6)
C(7)–Mo–C(8)	81.5(2)	C(1)–C(2)–C(3)	121.8(6)
	79.1(3)		121.4(6)
C(7)–Mo–C(9)	103.9(2)	C(2)–C(3)–C(4)	119.7(6)
	110.1(3)		120.2(7)
C(7)–Mo–C(10)	106.0(2)	C(3)–C(4)–C(5)	120.4(6)
	101.7(3)		119.4(7)
C(7)–Mo–C(11)	76.9(2)	C(4)–C(5)–C(6)	119.8(6)
	66.3(3)		120.2(7)
C(8)–Mo–C(9)	66.7(2)	C(5)–C(6)–C(1)	122.1(5)
	71.4(3)		121.5(6)
C(8)–Mo–C(10)	101.9(2)	C(9)–C(10)–C(11)	121.1(4)
	103.5(3)		115.6(7)
C(8)–Mo–C(11)	115.3(2)	C(10)–C(11)–C(12)	127.4(5)
	102.9(3)	C(11)–C(12)–C(13)	129.4(5)
1N(1)–B–2N(1)	106.8(4)	C(12)–C(13)–C(14)	126.7(5)
	103.6(5)	C(13)–C(14)–C(15)	128.9(5)
1N(1)–B–3N(1)	103.4(3)	C(14)–C(15)–C(9)	129.8(5)
	107.7(5)	C(15)–C(9)–C(10)	124.9(5)
2N(1)–B–3N(1)	110.6(4)		
	109.8(5)		
C(1)–B–1N(1)	116.2(4)		
	116.5(5)		
C(1)–B–2N(1)	108.0(4)		
	111.3(5)		

	Pz ring 1	Pz Ring 2	Pz Ring 3
N(2)–Mo–C(7)	90.4(2)	169.3(2)	91.8(2)
	84.9(2)	162.8(2)	101.9(3)
N(2)–Mo–C(8)	84.6(2)	100.5(2)	161.6(2)
	92.2(2)	94.7(2)	171.3(2)
N(2)–Mo–C(9)	145.0(2)	86.3(1)	131.7(2)
	154.4(2)	82.5(2)	115.8(2)
N(2)–Mo–C(10)	163.0(2)	83.9(2)	96.4(2)
	163.8(2)	95.4(2)	84.9(2)
N(2)–Mo–C(11)	153.9(2)	111.2(1)	79.4(1)
	143.6(2)	130.9(2)	85.3(2)
N(2)–N(1)–C(3)	108.7(4)	108.4(4)	109.0(3)
	108.8(5)	110.0(5)	109.3(5)
N(1)–C(3)–C(2)	109.3(5)	110.0(4)	109.3(4)
	109.1(6)	108.5(6)	109.0(6)
C(3)–C(2)–C(1)	105.3(5)	103.8(4)	104.7(4)
	105.0(6)	104.9(6)	104.8(6)
C(2)–C(1)–N(2)	110.7(5)	112.0(4)	111.4(4)
	110.7(6)	110.6(6)	111.5(4)
C(1)–N(2)–N(1)	105.9(4)	105.9(4)	105.6(3)
	106.3(5)	105.9(5)	105.4(5)
Mo–N(2)–N(1)	125.6(3)	119.9(2)	122.2(2)
	125.9(3)	121.8(3)	120.4(4)
Mo–N(2)–C(1)	127.8(3)	134.2(3)	129.7(3)
	127.5(4)	128.4(4)	134.2(4)
B–N(1)–N(2)	118.2(3)	124.1(3)	121.1(3)
	118.0(5)	120.9(4)	123.2(5)
B–N(1)–C(3)	132.5(4)	127.4(4)	127.9(4)
	132.8(5)	128.0(5)	127.3(5)

2.202(5) to 2.256(4). This effect has been observed in previous structures. The pattern observed is the same, namely that the longer Mo–N distances occur when there is a carbonyl group *trans* to the nitrogen atom.

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Supplementary Material Available (32 pages)

Listings of structure factor amplitudes available from the Editor upon request.

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