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Tetrakis(1-pyrazolyl)borato(triphenylphosphine)(carbonyl)(nitrosyl)molybdenum

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Abstract. $[(C_3H_3N_2)_4B]Mo[(C_6H_5)_3P](CO)(NO)$, triclinic, $P\bar{1}$, $a = 9{\cdot}058\,(5)$, $b = 13{\cdot}132\,(8)$, $c = 15{\cdot}020\,(8)$ Å; $\alpha = 86{\cdot}01\,(2)$, $\beta = 80{\cdot}26\,(2)$, $\gamma = 71{\cdot}65\,(2)°$, $U = 1671$ Å^3; $Z = 2$, $D_x = 1{\cdot}38$ g cm^{-3}. The structure was determined by Patterson methods and refined to an R of 0·057 for 3759 unique diffractometer data. The coordination around Mo is distorted octahedral, around B it is tetrahedral and around P, distorted tetrahedral. The four pyrazolyl rings are planar, as are the phenyl rings.

Table 1. *Atomic coordinates* (×10^4) *with standard deviations in parentheses*

	x	*y*	*z*		*x*	*y*	*z*
Mo	2887 (1)	1369 (1)	2379 (1)	N(14)	−299 (6)	−947 (3)	3502 (3)
P	1859 (1)	2896 (1)	1319 (1)	N(24)	−287 (8)	−1843 (5)	3103 (3)
B	669 (7)	−233 (4)	3085 (3)	C(34)	−1036 (8)	−2353 (5)	3712 (4)
N	3624 (6)	2147 (4)	3082 (3)	C(44)	−1531 (9)	−1817 (5)	4530 (4)
O(N)	4131 (7)	2589 (5)	3559 (4)	C(54)	−1060 (8)	−911 (4)	4384 (3)
C	4907 (7)	897 (4)	1709 (3)	C(15)	−301 (5)	3330 (3)	1430 (3)
O(C)	6213 (6)	538 (4)	1340 (4)	C(25)	−1060 (6)	2559 (4)	1407 (3)
N(11)	502 (5)	1705 (3)	3216 (3)	C(35)	−2662 (7)	2828 (5)	1541 (4)
N(21)	−260 (5)	949 (3)	3334 (3)	C(45)	−3582 (7)	3873 (6)	1699 (4)
C(31)	−1785 (8)	1409 (5)	3652 (4)	C(55)	−2848 (8)	4650 (5)	1726 (4)
C(41)	−2019 (7)	2476 (5)	3773 (4)	C(65)	−1223 (6)	4385 (4)	1583 (3)
C(51)	−574 (7)	2623 (4)	3497 (3)	C(16)	2459 (6)	2659 (3)	112 (3)
N(12)	2031 (5)	288 (3)	1643 (2)	C(26)	4047 (7)	2243 (4)	−236 (3)
N(22)	1079 (5)	−290 (3)	2057 (3)	C(36)	4519 (8)	2067 (4)	−1137 (4)
C(32)	727 (8)	−837 (5)	1420 (3)	C(46)	3438 (9)	2281 (5)	1711 (4)
C(42)	1502 (8)	−615 (5)	606 (3)	C(56)	1845 (9)	2696 (5)	−1386 (4)
C(52)	2277 (7)	84 (4)	771 (3)	C(66)	1371 (7)	2895 (4)	−482 (3)
N(13)	3309 (6)	−22 (3)	3293 (3)	C(17)	2255 (6)	4156 (3)	1443 (3)
N(23)	2226 (5)	−571 (3)	3480 (3)	C(27)	2021 (7)	4560 (4)	2302 (4)
C(33)	2789 (8)	−1423 (4)	4021 (4)	C(37)	2188 (8)	5544 (5)	2424 (4)
C(43)	4230 (9)	−1424 (5)	4192 (4)	C(47)	2642 (9)	6137 (5)	1696 (5)
C(53)	4511 (8)	−526 (5)	3721 (4)	C(57)	2919 (9)	5735 (5)	839 (5)
H(25)	−372 (11)	1747 (7)	1285 (6)	C(67)	2712 (7)	4758 (4)	710 (4)
H(35)	−3218 (13)	2225 (9)	1522 (7)	H(31)	−2520 (11)	1020 (12)	3830 (14)
H(45)	−4851 (13)	4082 (10)	1803 (8)	H(41)	−3040 (11)	2990 (13)	3920 (13)
H(55)	−3548 (13)	5461 (8)	1851 (8)	H(51)	−320 (13)	3310 (11)	3420 (12)
H(65)	−662 (11)	4991 (7)	1598 (6)	H(32)	150 (11)	−1340 (11)	1580 (12)
H(26)	4901 (12)	2064 (8)	207 (6)	H(42)	1220 (12)	−820 (13)	40 (8)
H(36)	5740 (14)	1751 (8)	−1394 (7)	H(52)	2760 (13)	430 (12)	320 (13)
H(46)	3819 (16)	2131 (8)	−2413 (7)	H(33)	2310 (13)	−1890 (12)	4310 (10)
H(56)	991 (15)	2868 (8)	−1835 (6)	H(43)	5020 (12)	−1880 (14)	4620 (9)
H(66)	148 (13)	3222 (8)	−230 (6)	H(53)	5450 (11)	−190 (13)	3770 (12)
H(27)	1684 (12)	4104 (7)	2870 (7)	H(34)	−1240 (13)	−3040 (11)	3640 (9)
H(37)	1980 (14)	5851 (8)	3087 (8)	H(44)	−2460 (11)	−1930 (12)	4990 (10)
H(47)	2787 (16)	6905 (8)	1795 (10)	H(54)	−1150 (12)	−350 (11)	4800 (11)
H(57)	3278 (16)	6190 (8)	273 (9)	H(67)	2910 (12)	4455 (7)	44 (7)

Introduction. Tetrakis(1-pyrazolyl)borate presents, in its coordination to a metal as a tridentate ligand, three chemically equivalent M—N bonds. Significant differences in these bonds have been reported, however, for some octahedral complexes (Holt, Holt & Watson, 1973; Kosby, Ganis & Avitabile, 1971) which could well be assigned to the *trans* effect. We now report the crystal structure of B(pz)$_4$(PPh$_3$)(CO)(NO)Mo, a compound which also presents the same characteristics.

Lattice parameters and integrated intensities were measured on a Norelco Pailred diffractometer with graphite-monochromated Mo $K\alpha$ radiation. A reddish-purple crystal, 0·45 × 0·25 × 0·20 mm, was mounted on a glass fibre and 4500 reflexions with $2\theta \le 45°$ were measured. Lp corrections were applied and the intensities averaged to give 3759 independent reflexions with $I > 3\sigma(I)$ used in the crystal structure analysis. The structure was elucidated by Patterson and Fourier methods. Atomic positional and thermal parameters were refined by blocked least squares with an overall isotropic temperature factor for the H atoms and the remaining atoms anisotropic. Complex neutral-atom scattering factors were employed and in the final stages the weighting scheme was $w = 1/\sigma^2(F)$. The refinement converged to $R' = \Sigma w^{1/2}\Delta/\Sigma w^{1/2}|F_o|$ = 0·054 after considering anisotropic thermal motion, anomalous dispersion correction for Mo, and H atom contributions. No absorption corrections were applied. The corresponding R was 0·057. The assignment of the CO and NO groups was made after studying the effect of interchanging C and N positions on the isotropic temperature factors. Positional parameters are given in Table 1* and the bond lengths and angles in Tables 2 and 3. A perspective view of the structure is shown in Fig. 1.

Table 2. *Bond distances* (Å)

Mo—P	2·512 (1)	C(65)—C(15)	1·386 (6)
Mo—N	1·849 (5)	C(16)—C(26)	1·390 (8)
Mo—C	1·870 (6)	C(26)—C(36)	1·361 (7)
Mo—N(11)	2·235 (5)	C(36)—C(46)	1·364 (10)
Mo—N(12)	2·246 (4)	C(46)—C(56)	1·386 (11)
Mo—N(13)	2·188 (4)	C(56)—C(66)	1·370 (8)
P—C(15)	1·839 (5)	C(66)—C(16)	1·389 (8)
P—C(16)	1·821 (5)	C(17)—C(27)	1·387 (7)
P—C(17)	1·828 (5)	C(27)—C(37)	1·375 (8)
B—N(21)	1·552 (6)	C(37)—C(47)	1·379 (10)
B—N(22)	1·527 (6)	C(47)—C(57)	1·379 (10)
B—N(23)	1·546 (8)	C(57)—C(67)	1·385 (9)
B—N(14)	1·513 (8)	C(67)—C(17)	1·393 (7)
N—O	1·182 (8)	C(31)—H(31)	0·954 (15)
C—O	1·180 (8)	C(41)—H(41)	0·958 (16)
N(11)—N(21)	1·363 (6)	C(51)—H(51)	0·994 (15)
N(11)—C(51)	1·332 (7)	C(32)—H(32)	0·959 (15)
N(21)—C(31)	1·339 (9)	C(42)—H(42)	1·005 (14)
C(31)—C(41)	1·370 (9)	C(52)—H(52)	0·916 (18)
C(41)—C(51)	1·374 (9)	C(33)—H(33)	0·905 (16)
N(12)—N(22)	1·368 (6)	C(43)—H(43)	1·057 (16)
N(12)—C(52)	1·323 (5)	C(53)—H(53)	1·089 (15)
N(22)—C(32)	1·370 (7)	C(34)—H(34)	0·992 (7)
C(32)—C(42)	1·364 (7)	C(44)—H(44)	1·037 (8)
C(42)—C(52)	1·376 (9)	C(54)—H(54)	0·974 (16)
N(13)—N(23)	1·373 (7)	C(25)—H(25)	1·063 (10)
N(13)—C(53)	1·327 (9)	C(35)—H(35)	1·068 (14)
N(23)—C(33)	1·353 (7)	C(45)—H(45)	1·081 (14)
C(33)—C(43)	1·372 (11)	C(55)—H(55)	1·074 (12)
C(43)—C(53)	1·403 (9)	C(65)—H(65)	1·074 (11)
N(14)—N(24)	1·353 (7)	C(26)—H(26)	1·065 (12)
N(14)—C(54)	1·383 (7)	C(36)—H(36)	1·064 (14)
N(24)—C(34)	1·318 (9)	C(46)—H(46)	1·064 (12)
C(34)—C(44)	1·393 (9)	C(56)—H(56)	1·072 (14)
C(44)—C(54)	1·377 (9)	C(66)—H(66)	1·066 (13)
C(15)—C(25)	1·396 (7)	C(27)—H(27)	1·066 (12)
C(25)—C(35)	1·364 (9)	C(37)—H(37)	1·065 (13)
C(35)—C(45)	1·376 (10)	C(47)—H(47)	1·080 (13)
C(45)—C(55)	1·388 (10)	C(57)—H(57)	1·070 (15)
C(55)—C(65)	1·384 (9)	C(67)—H(67)	1·068 (12)

Table 3. *Bond angles* (°)

P—Mo—N	95·5 (2)	N(22)—C(32)—C(42)	106·9 (5)
P—Mo—C	95·0 (2)	C(32)—C(42)—C(52)	106·6 (5)
P—Mo—N(11)	90·9 (1)	N(12)—C(52)—C(42)	110·7 (5)
P—Mo—N(12)	90·5 (1)	Mo—N(13)—N(23)	120·0 (3)
C—Mo—N	88·7 (2)	Mo—N(13)—C(53)	132·4 (4)
N—Mo—N(11)	95·4 (2)	N(23)—N(13)—C(53)	107·7 (5)
N—Mo—N(13)	93·4 (2)	B—N(23)—N(13)	122·8 (4)
C—Mo—N(12)	92·1 (2)	B—N(23)—C(33)	128·6 (5)
C—Mo—N(13)	93·5 (2)	N(13)—N(23)—C(33)	108·6 (4)
N(11)—Mo—N(12)	83·2 (1)	N(23)—C(33)—C(43)	108·8 (5)
N(11)—Mo—N(13)	80·0 (2)	C(33)—C(43)—C(53)	105·4 (6)
N(12)—Mo—N(13)	80·5 (2)	N(13)—C(53)—C(43)	109·6 (6)
P—Mo—N(13)	167·8 (1)	B—N(14)—N(24)	123·1 (5)
N—Mo—N(12)	173·9 (2)	B—N(14)—C(54)	125·9 (5)
C—Mo—N(11)	172·5 (2)	N(24)—N(14)—C(54)	110·0 (5)
Mo—P—C(15)	112·4 (1)	N(14)—N(24)—C(34)	106·5 (6)
Mo—P—C(16)	117·5 (2)	N(24)—C(34)—C(44)	111·7 (6)
Mo—P—C(17)	117·9 (2)	C(34)—C(44)—C(54)	105·1 (5)
C(15)—P—C(16)	102·3 (2)	N(14)—C(54)—C(44)	106·8 (5)
C(15)—P—C(17)	102·2 (2)	P—C(15)—C(25)	118·9 (3)
C(16)—P—C(17)	102·4 (2)	P—C(15)—C(65)	122·9 (4)
N(21)—B—N(22)	107·9 (4)	C(65)—C(15)—C(25)	118·1 (4)
N(21)—B—N(23)	109·2 (4)	C(15)—C(25)—C(35)	121·3 (5)
N(21)—B—N(14)	109·0 (4)	C(25)—C(35)—C(45)	120·8 (6)
N(22)—B—N(23)	108·0 (4)	C(35)—C(45)—C(55)	118·7 (6)
N(22)—B—N(14)	114·2 (4)	C(45)—C(55)—C(65)	120·8 (6)
N(23)—B—N(14)	108·4 (4)	C(55)—C(65)—C(15)	120·3 (5)
Mo—N—O	176·0 (5)	P—C(16)—C(26)	119·8 (4)
Mo—C—O	174·6 (5)	P—C(16)—C(66)	121·9 (4)
Mo—N(11)—N(21)	120·8 (3)	C(66)—C(16)—C(26)	118·4 (5)
Mo—N(11)—C(51)	131·4 (4)	C(16)—C(26)—C(36)	120·6 (5)
N(21)—N(11)—C(51)	106·0 (4)	C(26)—C(36)—C(46)	120·5 (6)
B—N(21)—N(11)	119·6 (4)	C(36)—C(46)—C(56)	120·4 (6)
B—N(21)—C(31)	130·7 (5)	C(46)—C(56)—C(66)	119·2 (6)
N(11)—N(21)—C(31)	109·5 (5)	C(56)—C(66)—C(16)	120·9 (5)
N(21)—C(31)—C(41)	108·1 (6)	P—C(17)—C(27)	118·7 (4)
C(31)—C(41)—C(51)	105·4 (6)	P—C(17)—C(67)	123·0 (4)
N(11)—C(51)—C(41)	110·7 (5)	C(67)—C(17)—C(27)	118·2 (5)
Mo—N(12)—N(22)	123·7 (3)	C(17)—C(27)—C(37)	120·9 (5)
Mo—N(12)—C(52)	129·9 (3)	C(27)—C(37)—C(47)	120·7 (6)
N(22)—N(12)—C(52)	106·4 (4)	C(37)—C(47)—C(57)	119·2 (7)
B—N(22)—N(12)	117·0 (4)	C(47)—C(57)—C(67)	120·3 (7)
B—N(22)—C(32)	133·6 (5)	C(57)—C(67)—(17)	120·7 (6)
N(12)—N(22)—C(32)	109·4 (4)		

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* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 32604 (27 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

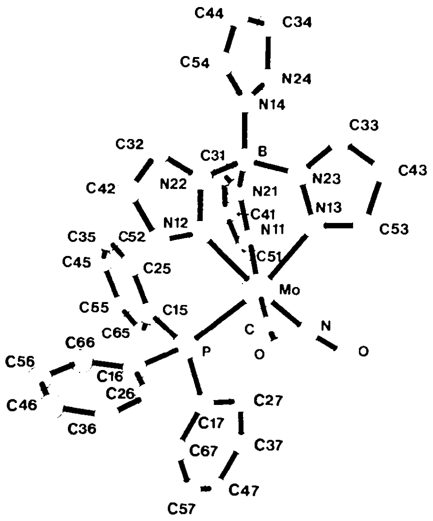

Fig. 1. The molecule of tetrakis(1-pyrazolyl)borato(triphenylphosphine)(carbonyl)(nitrosyl)molybdenum.

Discussion. The coordination around Mo is distorted octahedral. The Mo—N(pz) bonds *trans* to the CO and NO groups, 2·235 (5) and 2·246 (4) Å, are longer than the equivalent bond *trans* to the triphenylphosphine ligand, 2·188 (4) Å. The former bonds form the largest (pz)N—Mo—N(pz) angle, 83·2°. The asymmetry in the Mo—N(pz) bonds is similar to that in $HB(pz)_3[\eta^3\text{-}CH_2C(CH_3)CH_2](CO)_2Mo$ (Holt, Holt & Watson, 1973) 2·283 (4), 2·312 (3) and 2·207 (3) Å, with the longest bonds *trans* to the carbonyl groups. On the other hand, in $HB(pz)_3(CO)_2(NNC_6H_5)Mo$ (Avitabile, Ganis & Nemiroff, 1971) [2·231 (5) and 2·212 (4) Å *trans* to the CO groups *vs* 2·212 (4) Å *trans* to the NNC_6H_5] and in $Mo[HB(3,5\text{-}Me_2\text{-}4\text{-}ClC_3N_2)_3(NO)Cl(OPr^i)]$ (McCleverty, Seddon, Bailey & Walker, 1976) [2·239 (8), 2·211 (8), and 2·172 (8) Å *trans* to NO, OPr^i and Cl, respectively] where all the ligands are good acceptors, the differences are less significant, and in $HB(pz)_3Mo(CO)_2NO$ (Holt, Holt, Cavalito & Watson, 1976) where the CO and NO are symmetrically disordered, there are three equal M—N bonds, 2·213 (4) Å.

The coordination around the B atom is tetrahedral with mean N—B—N angles of 109·5°. The four pyrazolyl rings are planar, the deviation of any atom from the calculated mean plane being less than 0·014 Å. The bond distances and angles of the bidentate pyrazolyls do not present any significant difference from those of the monodentate.

The Mo—N(NO), Mo—C and Mo—P distances are in the expected range. The triphenylphosphine ligand presents its usual characteristics (Churchill & Chang, 1975): the coordination around the P atom is distorted tetrahedral with a mean value of 102·3° for the C—P—C angles; all phenyl rings are planar with a deviation less than 0·011 Å; the C—C distances are in the range 1·361–1·396 Å with a mean value of 1·381 Å and angles centred on C atoms bonded to P have values less than 120°.

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