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Structure of $pzB(pz)_3Mo(CO)_2(\eta^2\text{-}OCNMe_2)$

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Abstract. Dicarbonyl(dimethylaminocarbonyl)(1-pyrazolyl)tris(pyrazolylborane)molybdenum, $C_{17}H_{18}BMoN_9O_3$, $M_r = 503{\cdot}14$, monoclinic, $C2/c$, $a = 24{\cdot}568$ (3), $b = 9{\cdot}089$ (2), $c = 18{\cdot}616$ (2) Å, $\beta = 95{\cdot}03$ (1)°, $V = 4141$ (2) Å^3, $Z = 8$, $D_x = 1{\cdot}61$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71073 Å, $\mu = 6{\cdot}6$ cm^{-1}, $F(000) = 2032$, $T = 294$ K, final $R = 0{\cdot}029$ for 2967 unique observed reflections. Crystals of the title compound $pzB(pz)_3Mo(CO)_2(\eta^2\text{-}OCNMe_2)$ (I) (pz = 1-pyrazolyl) were isolated in low yield from the reaction of an excess of diethylamine with $[pzB(pz)_3(CO)_2\text{-}Mo(\eta^2\text{-}SMeCNMe_2)]BF_4$. An attempt to achieve a rational synthesis of the complex *via* the reaction of Me_2NCOCl with $[pzB(pz)_3Mo(CO)_3]^-$ was not successful. Compound (I) is isostructural (but not isomorphous) with $pzB(pz)_3Mo(CO)_2(\eta^2\text{-}SCNMe_2)$ (II) [Desmond, Lalor, O'Sullivan & Ferguson (1990). *J. Organomet. Chem.* **381**, C33–C37] with the sulfur atom replaced by an oxygen in (I). As in (II) the $\eta^2\text{-}OCN(CH_3)_2$ is situated between two carbonyl ligands and one pyrazolyl ring, but close to the C5—O5 group; the orientation of the dihapto ligand is specified by two torsion angles N21—Mo—O1—C1 [−75·7 (2)°] and C5—Mo—O1—C1 [33·9 (2)°]; the corresponding values in (II) are −74·9 (2) and 42·3 (2)°, respectively. The conformation of the two molecules differs only in the orientation of the uncomplexed pyrazolyl ring (N41–C45), presumably due to differences in crystal packing. The coordination about the Mo atom is distorted octahedral with the tridentate ligand [Mo—N11 2·181 (3), Mo—N21 2·294 (3), Mo—N31 2·218 (3) Å] and two carbonyl groups [Mo—C4 1·933 (4), Mo—C5 1·970 (3) Å] occupying five sites and the $\eta^2\text{-}OCN(CH_3)_2$ ligand in the sixth position [Mo—O1 2·207 (2), Mo—C1 2·050 (3) Å]. The packing is due mainly to van der Waals interactions and all intermolecular contacts agree with those predicted from radii-sum rules.

Experimental. Three-dimensional intensity data were collected on an Enraf–Nonius CAD-4 diffractometer using a dark-red needle-shaped crystal 0·18 × 0·11 × 0·38 mm and graphite-monochromated Mo $K\alpha$ radiation; lattice parameters were refined using 25 reflections in the range $8 < \theta < 20°$; reflections were measured using $\omega/2\theta$ scan ($2 < 2\theta < 54°$); ω-scan width $(0{\cdot}6 + 0{\cdot}35\tan\theta)°$; range of hkl: h 0 to 31, k 0 to 8, l −17 to 17; 4928 reflections were measured of which only 3026 had $I \geq 3\sigma I$ and were labelled observed. Three reflections (10,0,$\bar{4}$, 408 and 134) were measured periodically throughout the data collection and showed less than 2% variation. After averaging equivalent reflections ($R_{int} = 0{\cdot}009$), 2967 reflections were retained and used in the analysis. Lorentz, polarization and absorption corrections [Gaussian integration; Coppens, Leiserowitz & Rabinovich (1965)] were applied to the data; range of transmission coefficients 0·890 to 0·934. Structure was solved by the heavy-atom method; a Fourier map calculated on the basis of the position of the Mo atom revealed the entire molecule. Structural

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Table 1. *Positional and thermal parameters with their e.s.d's*

$$B_{eq} = (4/3)[a^2B(1,1) + b^2B(2,2) + c^2B(3,3) + ab(\cos\gamma)B(1,2) + ac(\cos\beta)B(1,3) + bc(\cos\alpha)B(2,3)].$$

	x	y	z	B_{eq} (Å^2)
Mo	0·14012 (1)	0·19838 (3)	0·12740 (1)	2·299 (4)
C1	0·2087 (1)	0·1643 (4)	0·1963 (2)	2·69 (6)
O1	0·18188 (9)	0·2433 (3)	0·2350 (1)	3·21 (5)
N1	0·2587 (1)	0·1139 (3)	0·2129 (2)	3·20 (6)
C2	0·2886 (2)	0·1551 (5)	0·2810 (2)	4·8 (1)
C3	0·2884 (2)	0·0322 (4)	0·1620 (2)	3·88 (8)
C4	0·0904 (1)	0·0708 (4)	0·1730 (2)	3·39 (7)
O4	0·0621 (1)	−0·0112 (3)	0·1999 (2)	5·66 (7)
C5	0·1594 (1)	−0·0010 (4)	0·0967 (2)	2·95 (7)
O5	0·1653 (1)	−0·1230 (3)	0·0826 (2)	4·43 (6)
N11	0·0985 (1)	0·2151 (3)	0·0193 (1)	2·80 (5)
N12	0·0942 (1)	0·3478 (3)	−0·0148 (1)	2·42 (5)
C13	0·0724 (1)	0·3281 (4)	−0·0829 (2)	3·35 (7)
C14	0·0612 (2)	0·1807 (4)	−0·0927 (2)	4·18 (8)
C15	0·0779 (2)	0·1158 (4)	−0·0280 (2)	3·73 (8)
N21	0·1965 (1)	0·3642 (3)	0·0780 (2)	2·77 (5)
N22	0·17728 (9)	0·4824 (3)	0·0383 (1)	2·39 (5)
C23	0·2191 (1)	0·5711 (4)	0·0247 (2)	2·84 (6)
C24	0·2665 (1)	0·5105 (4)	0·0556 (2)	3·44 (7)
C25	0·2508 (1)	0·3831 (4)	0·0872 (2)	3·35 (7)
N31	0·0924 (1)	0·4016 (3)	0·1420 (1)	2·68 (5)
N32	0·0895 (1)	0·5143 (3)	0·0932 (1)	2·39 (5)
C33	0·0706 (1)	0·6349 (4)	0·1248 (2)	2·95 (6)
C34	0·0559 (1)	0·6006 (4)	0·1938 (2)	3·43 (7)
C35	0·0734 (1)	0·4550 (4)	0·2019 (2)	3·28 (7)
N41	0·0973 (1)	0·6196 (3)	−0·0293 (1)	2·61 (5)
N42	0·1303 (1)	0·6724 (3)	−0·0789 (1)	3·12 (6)
C43	0·0984 (2)	0·7577 (4)	−0·1224 (2)	3·42 (7)
C44	0·0452 (2)	0·7600 (4)	−0·1034 (2)	3·59 (7)
C45	0·0458 (1)	0·6713 (4)	−0·0449 (2)	3·38 (7)
B	0·1152 (1)	0·4950 (4)	0·0210 (2)	2·36 (6)

parameters for the non-H atoms were refined using full-matrix least-squares methods with anisotropic thermal parameters. All H atoms were clearly visible in a difference Fourier map computed at an intermediate stage of the refinement; they were positioned geometrically (C—H 0·95 Å) and included as riding atoms with $B_{iso} = 4{\cdot}0$ Å^2 (but not refined) in subsequent refinement calculations. Refinement of the structure, with and without absorption correction, yielded the same R factors and dimensions which were the same to within 1σ. At convergence, the residuals were $R = 0{\cdot}029$ and $wR = 0{\cdot}036$ for the 2967 observations; the weighting scheme used was $w = 1/[\sigma^2F_o + 0{\cdot}05F_o^2]$; $S = 1{\cdot}13$; $(\Delta/\sigma)_{max} < 0{\cdot}02$ for all refined atoms in last cycle; density in final difference map was $\pm 0{\cdot}37$ e Å^{-3}; scattering factors were from *International Tables for X-ray Crystallography* (1974, Vol. IV). Programs used were *NRCVAX-PC* version (Gabe, Le Page, Charland, Lee & White, 1989), *ORTEP*II (Johnson, 1976) and *SDP-Plus* (B. A. Frenz & Associates, Inc., 1984). Final fractional coordinates are in Table 1* and details of molecular

* Lists of calculated hydrogen coordinates, anisotropic thermal parameters, torsion angles, a difference map showing the hydrogens of the uncomplexed pz ring, and observed and calculated structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54337 (38 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 2. *Bond lengths* (Å) *and bond angles* (°)

Mo	C1	2·050 (3)	N21	N22	1·365 (4)
Mo	O1	2·207 (2)	N21	C25	1·341 (4)
Mo	C4	1·933 (4)	N22	C23	1·347 (4)
Mo	C5	1·970 (3)	N22	B	1·536 (4)
Mo	N11	2·181 (3)	C23	C24	1·368 (4)
Mo	N21	2·294 (3)	C24	C25	1·369 (5)
Mo	N31	2·218 (3)	N31	N32	1·366 (4)
C1	O1	1·246 (4)	N31	C35	1·337 (4)
C1	N1	1·321 (4)	N32	C33	1·346 (4)
N1	C2	1·458 (5)	N32	B	1·544 (4)
N1	C3	1·450 (5)	C33	C34	1·370 (5)
C4	O4	1·163 (5)	C34	C35	1·370 (5)
C5	O5	1·152 (4)	N41	C42	1·369 (4)
N11	N12	1·362 (4)	N41	C45	1·356 (4)
N11	C15	1·330 (4)	N41	B	1·509 (4)
N12	C13	1·345 (4)	N42	C43	1·328 (4)
N12	B	1·562 (4)	C43	C44	1·383 (6)
C13	C14	1·377 (5)	C44	C45	1·355 (5)
C14	C15	1·371 (5)			

C1	Mo	O1	33·8 (1)	C13	N12	B	127·2 (3)
C1	Mo	C4	98·7 (1)	N12	C13	C14	108·3 (3)
C1	Mo	C5	80·6 (1)	C13	C14	C15	105·1 (3)
C1	Mo	N11	151·8 (1)	N11	C15	C14	110·9 (3)
C1	Mo	N21	81·8 (1)	Mo	N21	N22	122·8 (2)
C1	Mo	N31	117·6 (1)	Mo	N21	C25	131·5 (2)
O1	Mo	C4	88·5 (1)	N22	N21	C25	105·2 (3)
O1	Mo	C5	109·2 (1)	N21	N22	C23	109·8 (2)
O1	Mo	N11	165·3 (1)	N21	N22	B	117·6 (2)
O1	Mo	N21	89·60 (9)	C23	N22	B	132·5 (3)
O1	Mo	N31	86·78 (9)	N22	C23	C24	108·3 (3)
C4	Mo	C5	75·7 (2)	C23	C24	C25	105·1 (3)
C4	Mo	N11	100·6 (1)	N21	C25	C24	111·5 (3)
C4	Mo	N21	175·7 (1)	Mo	N31	N32	122·7 (2)
C4	Mo	N31	94·9 (1)	Mo	N31	C35	129·1 (2)
C5	Mo	N11	84·4 (1)	N32	N31	C35	106·3 (3)
C5	Mo	N21	108·6 (1)	N31	N32	C33	108·6 (3)
C5	Mo	N31	160·9 (1)	N31	N32	B	119·5 (3)
N11	Mo	N21	80·54 (9)	C33	N32	B	131·0 (3)
N11	Mo	N31	81·04 (9)	N32	C33	C34	109·1 (3)
N21	Mo	N31	81·2 (1)	C33	C34	C35	104·9 (3)
Mo	C1	O1	80·0 (2)	N31	C35	C34	111·0 (3)
Mo	C1	N1	153·5 (3)	N42	N41	C45	109·4 (3)
O1	C1	N1	126·3 (3)	N42	N41	B	121·6 (3)
Mo	O1	C1	66·2 (2)	C45	N41	B	127·4 (3)
C1	N1	C2	120·1 (3)	N41	N42	C43	105·2 (3)
C1	N1	C3	122·4 (3)	N42	C43	C44	111·9 (3)
C2	N1	C3	117·0 (3)	C43	C44	C45	104·7 (3)
Mo	C4	O4	176·9 (3)	N41	C45	C44	108·8 (3)
Mo	C5	O5	171·9 (3)	N12	B	N22	108·1 (3)
Mo	N11	N12	120·2 (2)	N12	B	N32	108·9 (3)
Mo	N11	C15	133·1 (2)	N12	B	N41	108·1 (2)
N12	N11	C15	106·4 (3)	N22	B	N32	107·5 (2)
N11	N12	C13	109·2 (3)	N22	B	N41	114·6 (3)
N11	N12	B	123·4 (2)	N32	B	N41	109·6 (3)

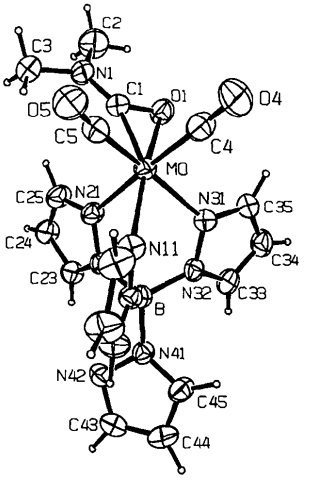

Fig. 1. A view of the $pzB(pz)_3Mo(CO)_2(\eta^2\text{-}OCNMe)_2$ molecule with our numbering scheme. Ellipsoids for non-H atoms are at the 50% level.

dimensions are in Table 2. Fig. 1 shows the molecule and our numbering scheme.

Related literature. The η^2-derivatives $HB(Me_2pz)_3$-$Mo(CO)_2(\eta^2$-$COR)$ ($R = p$-C_6H_4Me and C_6H_{11}) have been described previously (Desmond Lalor, O'Sullivan, Ferguson, Ruhl & Parvez, 1983). Curtis, Shiu & Butler (1986) have described $RBpz_3Mo(CO)_2$-(η^2-COR^1) (R = H or pz, R^1 = alkyl, aryl).

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Structure of *catena*-Poly{bis(1,1,1-trifluoro-2,4-pentanedionato-κ^2O,O')copper-μ-[(4,4'-bipyridine)-κN:$\kappa N'$]}

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Abstract. $[Cu(C_5H_4F_3O_2)_2(C_{10}H_8N_2)]$, $M_r = 525.55$, tetragonal, $P4_2/m$, $a = 8.379$ (1), $c = 15.832$ (4) Å, $V = 1111.6$ (4) Å^3, $Z = 2$, $D_x = 1.57$ g cm^{-3}, λ(Mo $K\alpha$) = 0.71073 Å, $\mu = 10.59$ cm^{-1}, $F(000) = 530$, $T = 295$ K, $R = 0.043$, $wR = 0.043$ for 658 unique observed $[I > 1.5\sigma(I)]$ reflections. Bis(1,1,1-trifluoropentane-2,4-dionato-O,O')copper(II) and 4,4'-bipyridine form a one-dimensional infinite linear structure with molecular ratio 1:1. The coordination geometry around each copper(II) atom is that of an octahedron, where the basal plane is comprised of four O atoms at a distance of 1.968 (3) Å and the axial positions are occupied by two N atoms at 2.381 (5) Å. The distance between adjacent copper(II) atoms is 11.850 (4) Å.

Experimental. The title complex was prepared by addition of 4,4'-bipyridine (0.32 g) in absolute ethanol (10 ml) to a solution of bis(1,1,1-trifluoropentane-2,4-dionato-O,O')copper(II) (1.48 g) in absolute ethanol (40 ml). After the resulting solution was stirred and refluxed for two hours, green crystals were obtained. Yield: 61%. The structure proposed is in agreement with microanalysis. $C_{20}H_{16}CuF_6N_2O_4$: Calc.: C 45.66, H 3.04, N 5.33; Found: C 45.50, H 3.15, N 5.16.

Light-green single crystals were developed from a CH_3CN–C_2H_5OH solution of the complex at room temperature. A suitable crystal, dimensions 0.40 × 0.40 × 0.50 mm, $R3M/E$ diffractometer, graphite-monochromatized Mo $K\alpha$ radiation; cell parameters from 19 reflections in 2θ range 6.86–18.34°; data collected by ω–2θ scans in 2θ range 2–45°. hkl ranges: h 0–9, k 0–9, l 0–17; 914 measured reflections, 658 unique with $[I > 1.5\sigma(I)]$, $R_{int} = 0.0103$. Corrections made for Lorentz–polarization factors, but not absorption effects. Three standard reflections monitored every 2 h, no significant variation during data collection.

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