well with those found in other octahedrally coordinated tris(phenanthroline)–metal complexes (Zalkin, Templeton & Ueki, 1973) and in molecular phenanthroline (Nishigaki, Yoshioka & Nakatsu, 1978). They exhibit somewhat different planarities with maximum deviations of the atoms from their least-squares planes of 0·031 (11), 0·035 (9) and 0·064 (10) Å in phen (1), phen (2) and phen (3), respectively, and dihedral angles 1–2 = 92·4, 1–3 = 97·2 and 2–3 = 97·2° ($\sigma < 1\cdot0°$).

Co—N bond lengths and N—Co—N bite angles fall within the expected range. The main difference from the Cu^{II} analogue lies essentially in the coordination bond geometry. The Cu^{II} complex exhibits two long and four short Cu—N bonds giving rise to a tetragonally distorted octahedral coordination which was considered typical of a static Jahn–Teller distortion. The substantially longer axial bond lengths have a mean of 2·328 (10) Å while the four equatorial short bonds have a mean of 2·037 (30) Å (standard deviations based on spread in values). The lengthening of one of the bonds in two of the ligands produces a decrease in the corresponding N—Cu—N bite angles with a mean of 76·9 (4)° while the ligand with no bond distortion has a bite angle of 81·6 (5)°. In the Co^{II} complex the coordinating ligands exhibit similar, though not equal (errors considered), bond distances and bite angles (Table 2).

The packing of the structure is mainly achieved by Coulomb and van der Waals interactions with the uncoordinated perchlorate anions and water molecules occupying the cavities between the complex cations. The particular orientation of Cl(3) perchlorate, incompatible with the site symmetry, may be attributed to strong geometrical restrictions imposed by the cavity. There are some very close proximities of the perchlorate O atoms to the surrounding phenanthroline C atoms, many of them *via* H atoms. The water molecule appears to be hydrogen-bonded to Cl(1) perchlorate with an estimated distance O(H)···O(12) = 2·98 (3) Å.

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μ-Oxo-bis{[hydrotris(3,5-dimethyl-1-pyrazolyl)borato]dioxomolybdenum(VI)}, $Mo_2(C_{15}H_{22}BN_6)_2O_5$

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Abstract. $M_r = 866$, monoclinic, $C2/c$, $a = 28\cdot253$ (11), $b = 9\cdot550$ (2), $c = 15\cdot761$ (5) Å, $\beta = 115\cdot78$ (3)°, $V = 3829\cdot3$ Å^3, $Z = 4$, $D_m = 1\cdot51$ (1), $D_x = 1\cdot50$ g cm^{-3}, Mo $K\alpha$, $\lambda = 0\cdot71073$ Å, $\mu = 6\cdot310$ cm^{-1}, $F(000) = 1132$, room temperature, $R = 0\cdot028$, $wR = 0\cdot038$ for 2576 reflections. The molecule contains an $O_2MoOMoO_2$ core with the bridging O atom lying on a crystallographic twofold axis. The Mo—O—Mo angle is 167·1 (2)°. The tridentate ligand is coordinated through the N atoms. The Mo—N distance *trans* to the bridging O atom is significantly shorter than those *trans* to terminal O atoms.

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Introduction. We have been investigating the synthesis and properties of sterically hindered molybdenum compounds as models for the active sites of certain molybdenum-containing enzymes. The bulky tridentate ligand, hydrotris(3,5-dimethyl-1-pyrazolyl)borate, stabilizes monomeric Mo compounds of oxidation states IV, V and VI, and constrains the co-ligands to *fac* stereochemistry. During the course of our investigations, we have also isolated a binuclear $\mathrm{Mo^{VI}}$ complex containing an $\mathrm{O_2MoOMoO_2}$ core. Such compounds are still relatively rare.

Experimental. Crystal 0·3 × 0·1 × 0·3 mm, sealed in epoxy, D_m by flotation ($\mathrm{ZnBr_2}$ and $\mathrm{H_2O}$), mirror-plane symmetry element observed on *b*-axis X-ray photograph, indicating *C*2/*c*, 25 reflections for measuring lattice parameters, Syntex $P2_1$ four-circle diffractometer, graphite-monochromated Mo $K\alpha$, θ–2θ scans, $2\theta_{\max} = 50°$, $-31 \le h \le 30$, $0 \le k \le 11$, $0 \le l \le 17$, standard reflections 800, 040, 006, no change; 3355 reflections measured, 2576 with $I > 3\sigma(I)$ used in refinement; coordinates of Mo readily obtained from Patterson calculations, 25 non-hydrogen atoms from difference Fourier map, full-matrix least squares on *F*, all non-hydrogen atoms anisotropic, no hydrogen atoms located, H atoms calculated and introduced at idealized positions (C—H = 0·95 Å) with isotropic temperature factor 1·5 times that of atom to which it is bonded; $R = 0{\cdot}028$, $wR = 0{\cdot}038$, $w^{-2} = \sigma^2(F_o) + (cF_o)^2$, $c = 0{\cdot}03$, $\Delta/\sigma = 0{\cdot}09$, max. height in difference map 1·221 e Å^{-3}; scattering factors for non-hydrogen atoms from Cromer & Waber (1974), *Structure Determination Package* (Frenz, 1978) for structure determination and refinement.*

Discussion. Atomic coordinates and isotropic thermal parameters are listed in Table 1 and selected interatomic distances and bond angles in Table 2. O(3) is the bridging atom lying on the twofold axis, while O(1) and O(2) are the terminal atoms. Table 3 compares the distances and bond angles found in the $\mathrm{O_2MoOMoO_2}$ core of $\mathrm{Mo_2O_5[HB(C_5H_7N_2)_3]_2}$ to those found in other complexes containing the same core. Fig. 1 shows the molecular structure.

The terminal and bridging oxo groups adopt *fac* stereochemistry. In the known oxo-bridged dinuclear $\mathrm{Mo^{VI}}$ complexes which contain no other bridging ligands, the Mo—O—Mo angle ranges from strictly 180° (Cotton, Morehouse & Wood, 1964) to 136° (Stomberg, 1968) (Table 3). The present structure has

Table 1. *Atomic coordinates and equivalent isotropic thermal parameters*

Isotropic equivalent thermal parameters are defined as $[U(1,1) + U(2,2) + U(3,3)]/3$.

	x	y	z	$U_{\rm eq}$(Å^2 × 10^3)
Mo	0·06748 (1)	0·26039 (3)	0·25440 (2)	31·6 (2)
N(11)	0·07525 (9)	0·0191 (3)	0·2698 (2)	31 (2)
N(21)	0·09662 (9)	0·2461 (3)	0·4159 (2)	38 (2)
N(31)	0·15300 (9)	0·2287 (3)	0·3069 (2)	35 (2)
O(1)	0·07689 (9)	0·4365 (3)	0·2676 (2)	51 (2)
O(2)	0·05773 (9)	0·2340 (2)	0·1416 (2)	46 (2)
O(3)	0·000	0·2382 (3)	0·250	41 (2)
N(12)	0·11423 (9)	−0·0472 (3)	0·3455 (2)	34 (2)
C(11)	0·1089 (1)	−0·1871 (3)	0·3343 (2)	38 (2)
C(12)	0·1460 (2)	−0·2869 (4)	0·4042 (3)	63 (2)
C(13)	0·0660 (1)	−0·2126 (3)	0·2505 (2)	39 (2)
C(14)	0·0458 (1)	−0·0829 (3)	0·2114 (2)	33 (2)
C(15)	0·0010 (1)	−0·0543 (4)	0·1187 (2)	41 (2)
N(22)	0·12645 (9)	0·1350 (3)	0·4675 (2)	39 (2)
C(21)	0·1281 (1)	0·1358 (4)	0·5542 (2)	52 (2)
C(23)	0·1003 (1)	0·2516 (5)	0·5591 (2)	59 (2)
C(22)	0·1531 (2)	0·0231 (5)	0·6241 (3)	73 (2)
C(24)	0·0814 (1)	0·3174 (4)	0·4730 (2)	46 (2)
C(25)	0·0493 (1)	0·4486 (4)	0·4442 (3)	64 (2)
N(32)	0·18178 (9)	0·1383 (3)	0·3785 (2)	32 (2)
C(31)	0·2322 (1)	0·1412 (4)	0·3924 (2)	38 (2)
C(32)	0·2745 (1)	0·0571 (4)	0·4660 (3)	53 (2)
C(33)	0·2358 (1)	0·2352 (4)	0·3296 (2)	50 (2)
C(34)	0·1860 (1)	0·2881 (4)	0·2774 (2)	43 (2)
C(35)	0·1685 (2)	0·3956 (5)	0·2012 (3)	68 (2)
B	0·1549 (1)	0·0434 (4)	0·4236 (3)	37 (2)

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

Mo—N(11)	2·318 (2)	Mo—O(1)	1·701 (2)
Mo—N(21)	2·314 (2)	Mo—O(3)	1·889 (1)
Mo—N(31)	2·208 (2)	Mo—O(2)	1·696 (2)
N(11)—Mo—N(21)	81·59 (7)	N(31)—Mo—O(1)	90·14 (9)
N(11)—Mo—N(31)	77·64 (7)	N(31)—Mo—O(3)	157·02 (7)
N(11)—Mo—O(1)	165·34 (9)	N(31)—Mo—O(2)	91·42 (9)
N(11)—Mo—O(3)	86·57 (9)	O(1)—Mo—O(3)	102·5 (1)
N(11)—Mo—O(2)	85·83 (8)	O(1)—Mo—O(2)	102·7 (1)
N(21)—Mo—N(31)	76·82 (8)	O(3)—Mo—O(2)	104·11 (7)
N(21)—Mo—O(1)	87·82 (9)		
N(21)—Mo—O(3)	84·50 (5)		
N(21)—Mo—O(2)	164·32 (8)		
Mo—O(3)—Mo$^{\rm i}$	167·1 (2)		

Numbers in parentheses are estimated standard deviations in the least significant digits. Symmetry operation for equivalent atoms: (i) $-x, y, -\frac{1}{2} - z$.

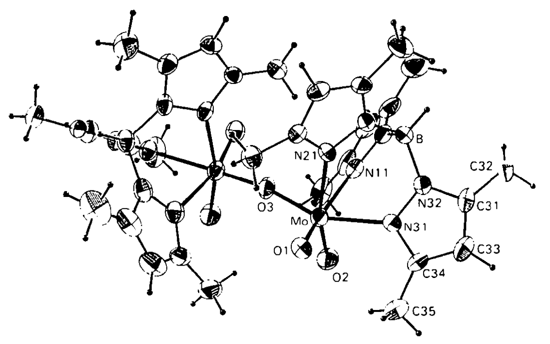

Fig. 1. The structure of $\mathrm{Mo_2O_5[HB(C_5H_7N_2)_3]_2}$. The numbering of atoms in the pyrazole rings containing N(11) and N(21) parallels that shown for the ring containing N(31).

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* Lists of structure factors, anisotropic thermal parameters, H-atom coordinates and all bond lengths and angles not involving hydrogen have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 39441 (19 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 3. *Comparison of distances and angles in* $Mo_2O_5^{2+}$ *complexes*

O*t* = terminal oxygen; O*b* = bridging oxygen.

	Mo—O—Mo (°)	Mo—O*b* (Å)	Mo—O*t* (Å)	Reference
$Mo_2O_5(C_2O_4)_2(H_2O)_2$	180	1·876	1·68, 1·70	(*a*)
$[Mo_2O_5(O_2)_4(H_2O)_2]K_2(H_2O)_2$	136·1	1·93	1·66	(*b*)
$Mo_2O_5(DMF)_4Cl_2$	171	1·90	1·68	(*c*)
$Mo_2O_5(phen)_2(NCS)_2$	162·7	1·865, 1·885	1·685, 1·688 1·691, 1·694	(*d*)
$Mo_2O_5(Hnta)_2$*	180	1·88	1·68, 1·71	(*e*)
$Mo_2O_5[(CH_3)_2NCH_2CH_2NHCH_2C(CH_3)_2S]_2$	143·8	1·929, 1·917	1·715, 1·715 1·714, 1·709	(*f*)
$Mo_2O_5(NCS)_2(Me_2SO)_4$	180	1·864	1·691, 1·686	(*g*)
$Mo_2O_5[HB(Me_2pz)_3]_2$	167·1	1·889	1·701, 1·696	(*h*)

References: (*a*) Cotton *et al.* (1964); (*b*) Stomberg (1968); (*c*) Atovmyan, Sokovova & Thachey (1970); (*d*) Viossat & Rodier (1981); (*e*) Knobler, Robinson, Wilkins & Wilson (1983); (*f*) Marabella *et al.* (1983); (*g*) Shibahara, Kuroya, Ooi & Mori (1983); (*h*) this work.

* Hnta = nitrilotriacetate(2−).

an Mo—O—Mo angle of 167·1°. Both the bridging Mo—O distance of 1·889 Å and the terminal Mo—O distances of 1·701 Å and 1·696 Å are within the range of comparable distances found in the other complexes.

The remaining *fac* positions are occupied by coordination through the chemically identical N atoms of the pyrazolylborate ligand, $HB(Me_2pz)_3^-$. Significant differences are found for the Mo—N bond distances. The Mo—N distances for the N atoms *trans* to the terminal oxygens are 2·318 and 2·314 Å, while the Mo—N distance for the N atom *trans* to the bridging oxygen is significantly shorter, at 2·208 Å. These differences in the Mo—N bond distances are similar to the differences found for the binuclear Mo^{II} compound of this trispyrazolylborate ligand, $[HB(Me_2pz)_3Mo(CO)_2]_2S$, which has sulfur as its bridging atom. There the Mo—N distances *trans* to terminal carbonyl groups are 2·247 and 2·260 Å, and the Mo—N *trans* to bridging S is 2·188 Å (Enemark, unpublished results).

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The Structure of the Monobasic Lead Salt of 2,4,6-Trinitro-1,3-benzenediol (β Polymorph), $Pb_2(OH)_2^{2+}.C_6HN_3O_8^{2-}$

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Abstract. $M_r = 691·5$, monoclinic, $P2/c$, $a = 16·606$ (4), $b = 16·635$ (8), $c = 20·462$ (7) Å, $\beta = 127·5$ (14)°, $U = 4484·4$ Å^3, $Z = 16$, $D_x = 4·10$ Mg m^{-3}, Mo $K\alpha$, $\lambda = 0·71069$ Å, $\mu = 30·24$ mm^{-1}, $F(000) = 4864$, room temperature, final $R = 0·076$ for 4479 reflections. The Pb atoms are clustered into two independent $[Pb_4(OH)_4]^{4+}$ units each with Pb atoms at the vertices of a tetrahedron and