other gold complexes containing coordinated bromide ligands (Bennett, Hoskins, Kneen, Nyholm, Hitchcock, Mason, Robertson & Towl, 1971; Komiya, Huffman & Kochi, 1977; Perutz & Weisz, 1946; Strähle & Bärnighausen, 1971). The two Au—Br bonds *trans* to one another, Au—Br(1) [2·416 (1) Å] and Au—Br(2) [2·407 (1) Å] are equivalent. By comparison the Au—Br(3) bond *trans* to the phosphine is lengthened considerably [2·468 (1) Å]. This observation is expected, however, owing to the strong *trans* influence of the coordinated phosphine (Cotton & Wilkinson, 1980). An analogous observation was reported for the crystal structure of trichloro(triphenylphosphine)gold(III) (Bandoli, Clemente, Marangoni & Catalini, 1973). The $\mathrm{Au^{III}}$—P bond length of 2·328 (2) Å is quite normal for bonds of this type (Bandoli, Clemente, Marangoni & Catalini, 1973; Baker & Pauling, 1969).

The angles subtended at the gold atom are indicative of its square-planar coordination environment (see Table 2). Slight deviations from the ideal value of 90° in the P—Au—Br(1) [87·61 (7)°] and the P—Au—Br(2) [91·99 (7)°] angles are probably attributable to subtle adjustments owing to steric constraints of the phosphine ligand. The square-planar arrangement of atoms is further exemplified by the least-squares plane through the five atoms Au, Br(1), Br(2), Br(3), P, from which no atom deviates by more than 0·043 (1) Å.

There are no unusual intermolecular contacts within the crystal lattice. The closest Au–Au contact is 4·921 (1) Å while the closest intermolecular contact with Au is 3·910 (1) Å between Au and Br(2) of a molecule related by the crystallographic inversion center.

Table 2. *Bond angles* (°)

Br(1)—Au—Br(2)	176·05 (5)	Au—P—C(1)	111·0 (4)
Br(1)—Au—Br(3)	90·07 (5)	Au—P—C(2)	115·8 (5)
Br(1)—Au—P	87·61 (7)	Au—P—C(3)	108·1 (4)
Br(2)—Au—Br(3)	90·26 (5)	C(1)—P—C(2)	106·7 (6)
Br(2)—Au—P	91·99 (7)	C(1)—P—C(3)	107·4 (5)
Br(3)—Au—P	177·43 (7)	C(2)—P—C(3)	107·5 (6)
P—C(1)—C(12)	114·6 (7)	P—C(3)—C(32)	113·5 (8)
P—C(2)—C(22)	116·0 (10)		

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Structure of Tris(1,10-phenanthroline)cobalt(II) Diperchlorate Monohydrate, $[\mathrm{Co(C_{12}H_8N_2)_3](ClO_4)_2.H_2O}$

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Abstract. $M_r = 816{\cdot}48$, monoclinic, $C2/c$, $a = 36{\cdot}366\ (12)$, $b = 15{\cdot}854\ (2)$, $c = 12{\cdot}341\ (1)$ Å, $\beta = 102{\cdot}83\ (2)^\circ$, $V = 6938\ (2)$ Å^3, $Z = 8$, $D_m = 1{\cdot}547\ (5)$, $D_x = 1{\cdot}563\ (1)$ Mg m^{-3}, $\lambda(\mathrm{Mo}\ K\alpha) = 0{\cdot}71073$ Å, $\mu = 0{\cdot}74$ mm^{-1}, $F(000) = 3336$, $T = 295$ K, final $R = 0{\cdot}086$ for 3849 independent observed reflections. The metal ion is coordinated to the six N atoms of the bidentate chelates in a distorted octahedral arrangement with average Co—N length of 2·127 (14) Å and average N—Co—N within each phenanthroline of 78·1 (4)°, where the standard deviations are based on the spread in values. This coordination is different from the tetragonally octahedral coordination present in the isostructural $\mathrm{Cu^{II}}$ analogue.

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Introduction. A comparison of the cell parameters and symmetry of the title complex, given earlier by Escobar & Garland (1970), with those of $[Cu^{II}(phen)_3](ClO_4)_2$ (phen = 1,10-phenanthroline) after its structure determination by Anderson (1973), suggested that these two complexes could be isostructural. Asymmetry in metal–bidentate ligand bonds can be different even in complexes which are isostructural, *e.g.* $[M(bpy)_2(ONO)]NO_3$ with M = Cu, Zn (bpy = 2,2′-bipyridyl) (Simmons, Seff, Clifford & Hathaway, 1983). The structure determination of the title compound offers the possibility of comparing bond distortions in the coordination of Cu^{II} and Co^{II} ions in an identical crystalline environment.

Experimental. Yellow–brown crystals, thick platelets, synthesized from stoichiometric amounts of $Co(ClO_4)_2$ and phenanthroline in ethanol; density measured by flotation using carbon tetrachloride and dioxane; crystal 0·50 × 0·20 × 0·18 mm, Nonius CAD-4 diffractometer, graphite-monochromatized Mo $K\alpha$ radiation; unit-cell parameters by least-squares fit from 43 reflections with $5 < 2\theta < 41°$; two standard reflections monitored after every 50 reflections showed random intensity fluctuations <2·4% from the mean; 3926 independent reflections with $2\theta < 46{\cdot}2°$, range of hkl: h −39→38, k 0→17, l 0→10, 3849 observed reflections with $|F_o| > 2\sigma(F_o)$, Lp corrections applied, absorption ignored; $R_{\rm int} = 0{\cdot}013$ for 261 pairs of equivalent reflections. Starting coordinates taken from structure of $[Cu^{II}(phen)_3](ClO_4)_2$ and refined by least squares, anisotropic full-matrix in stages, with the Co atom and one phenanthroline at a time, the perchlorate ions and water oxygen refined separately; function minimized $\sum w(|F_o| - |F_c|)^2$ with $w = w_1w_2$ such that $w_1 = 1$ if $\sin\theta > 0{\cdot}30$ otherwise $w_1 = \sin\theta/0{\cdot}30$, and $w_2 = 1$ if $|F_o| < 100$ otherwise $w_2 = 100/|F_o|$; H atoms included in fixed theoretical positions (C—H = 1·00 Å) with isotropic temperature factors of the parent C atoms; final $R = 0{\cdot}086$, $S = 3{\cdot}2$ for 3849 reflections and 552 parameters refined; max. and mean shift-to-error ratios in final cycle 0·007 and 0·002 for the cation, 3·50 and 0·86 for the perchlorate anions; highest residuals in final difference Fourier synthesis of 0·55 e Å^{-3} in the vicinity of the Co atom and next highest residuals about the perchlorate anions. Scattering factors for non-H atoms from Cromer & Mann (1968) and for H from Stewart, Davidson & Simpson (1965); real and imaginary parts of dispersion corrections for Co and Cl from *International Tables for X-ray Crystallography* (1974); computations with *XRAY76* (Stewart, Machin, Dickinson, Ammon, Heck & Flack, 1976) and *NRC22* (Ahmed, Hall, Pippy & Huber, 1973) for mean planes on an IBM 370/145 computer at the Computer Centre of the Universidad de Chile.

Table 1. *Fractional coordinates* (×10^4) *and equivalent isotropic thermal parameters* (Å^2 ×10^3) *for non*-H *atoms, with e.s.d.'s in parentheses* (*pp* = *occupancy*)

	pp	*x*	*y*	*z*	U_{eq}*
(*A*) Complex cation atoms					
Co		3794·4 (2)	2804·8 (5)	1264·5 (7)	51·5 (3)
N(11)		3530 (2)	3711 (4)	2107 (5)	60 (2)
N(12)		3943 (1)	3963 (3)	581 (5)	54 (2)
N(21)		4002 (1)	2034 (3)	142 (5)	53 (2)
N(22)		3310 (1)	2699 (4)	−76 (6)	61 (2)
N(31)		3679 (2)	1757 (4)	2193 (5)	61 (2)
N(32)		4268 (1)	2807 (4)	2651 (5)	56 (2)
C(11)		3326 (2)	3577 (6)	2837 (8)	77 (3)
C(12)		3159 (3)	4244 (9)	3320 (8)	94 (4)
C(13)		3221 (3)	5049 (8)	3035 (9)	96 (4)
C(14)		3441 (2)	5222 (6)	2282 (8)	78 (4)
C(15)		3522 (4)	6043 (7)	1951 (11)	107 (5)
C(16)		3727 (4)	6170 (6)	1212 (12)	108 (5)
C(17)		3885 (2)	5479 (5)	713 (8)	78 (4)
C(18)		4097 (3)	5564 (6)	−84 (10)	89 (4)
C(19)		4232 (2)	4877 (6)	−511 (8)	83 (4)
C(110)		4147 (2)	4077 (5)	−171 (6)	63 (3)
C(111)		3814 (2)	4647 (4)	1016 (6)	58 (3)
C(112)		3593 (2)	4521 (5)	1817 (6)	60 (3)
C(21)		4343 (2)	1688 (5)	271 (7)	64 (3)
C(22)		4444 (3)	1227 (5)	−572 (9)	80 (4)
C(23)		4199 (3)	1133 (5)	−1548 (9)	86 (4)
C(24)		3838 (3)	1480 (5)	−1718 (7)	71 (3)
C(25)		3551 (4)	1391 (6)	−2722 (8)	95 (5)
C(26)		3207 (3)	1707 (7)	2798 (9)	97 (4)
C(27)		3108 (2)	2150 (6)	−1933 (8)	77 (3)
C(28)		2752 (3)	2529 (8)	−1999 (10)	98 (4)
C(29)		2689 (2)	2961 (8)	−1141 (10)	95 (4)
C(210)		2969 (2)	3048 (5)	−183 (7)	70 (3)
C(211)		3380 (2)	2267 (4)	−950 (6)	57 (3)
C(212)		3751 (2)	1916 (4)	−846 (6)	58 (3)
C(31)		3384 (2)	1244 (5)	1933 (7)	71 (3)
C(32)		3309 (3)	633 (6)	2669 (10)	90 (4)
C(33)		3552 (3)	539 (6)	3672 (9)	91 (4)
C(34)		3875 (3)	1056 (6)	3955 (8)	79 (4)
C(35)		4152 (3)	1002 (7)	4969 (8)	94 (4)
C(36)		4446 (3)	1533 (8)	5226 (8)	94 (4)
C(37)		4495 (2)	2170 (6)	4457 (6)	71 (3)
C(38)		4785 (2)	2782 (7)	4686 (8)	84 (4)
C(39)		4809 (2)	3365 (7)	3904 (8)	83 (4)
C(310)		4548 (2)	3361 (5)	2896 (7)	67 (3)
C(311)		4239 (2)	2235 (5)	3435 (6)	56 (2)
C(312)		3928 (2)	1653 (5)	3179 (6)	61 (3)
(*B*) Perchlorate and water atoms					
Cl(1)		2396 (1)	5171 (2)	410 (2)	103 (1)
O(11)		2358 (5)	4570 (15)	1103 (17)	323 (14)
O(12)		2461 (3)	5829 (10)	1027 (20)	319 (13)
O(13)		2685 (5)	4934 (8)	5 (15)	252 (10)
O(14)		2061 (4)	5249 (11)	−226 (10)	235 (8)
Cl(2)		5000	5871 (2)	2500	88 (1)
O(21)		4905 (5)	5414 (13)	1628 (14)	307 (11)
O(22)		5292 (4)	6380 (10)	2491 (18)	271 (11)
Cl(3)†	0·80	5000	439 (2)	2500	93 (1)
Cl(3′)	0·20	5000	401 (2)	2500	93 (1)
O(31)	0·70	5000	1335 (7)	2500	97 (5)
O(32)	0·35	4635 (10)	178 (15)	1876 (24)	173 (13)
O(33)	0·35	5288 (16)	208 (17)	2099 (48)	294 (26)
O(34)	0·35	5017 (7)	204 (15)	3617 (17)	123 (9)
O(31′)	0·20	5000	−495 (24)	2500	86 (16)
O(32′)	0·10	4729 (30)	732 (62)	1582 (114)	270 (72)
O(33′)	0·10	5360 (24)	732 (54)	2439 (70)	148 (44)
O(34′)	0·10	4893 (16)	726 (48)	3413 (62)	63 (32)
O(35)	0·10	4838 (37)	970 (135)	3195 (93)	224 (105)
O(36)	0·10	5252 (42)	−62 (62)	3187 (93)	268 (67)
O(1)		2076 (7)	2509 (13)	5532 (14)	328 (13)

* $U_{eq} = \frac{1}{3}\sum_i\sum_j U_{ij}a^*_ia^*_j(\mathbf{a}_i.\mathbf{a}_j)$.

† Parameters for the Cl(3) perchlorate represent a model in which three possible orientations are given with different weights (0·70, 0·20, and 0·10). Primes are used to designate an alternative Cl(3) perchlorate with the Cl—O bond inverted along the twofold axis. O(35) and O(36), bonded to Cl(3), correspond to a perchlorate oriented with its twofold axis along the symmetry axis of the site.

Table 2. *Intramolecular distances* (Å) *and bond angles* (°), *with e.s.d.'s in parentheses*

(*A*) Metal coordination

Co–N(11)	2·124 (7)	Co–N(21)	2·109 (6)	Co–N(31)	2·111 (6)
Co–N(12)	2·138 (6)	Co–N(22)	2·138 (5)	Co–N(32)	2·142 (5)
N(11)–Co–N(12)	78·1 (2)	N(21)–Co–N(22)	78·5 (2)	N(31)–Co–N(32)	77·8 (2)
N(11)–Co–N(21)	168·6 (3)	N(12)–Co–N(31)	170·7 (2)	N(22)–Co–N(32)	175·1 (3)
N(11)–Co–N(22)	92·7 (2)	N(12)–Co–N(21)	94·6 (2)	N(21)–Co–N(31)	92·5 (2)
N(11)–Co–N(31)	95·8 (2)	N(12)–Co–N(22)	89·9 (2)	N(21)–Co–N(32)	100·6 (2)
N(11)–Co–N(32)	88·8 (2)	N(12)–Co–N(32)	94·9 (2)	N(22)–Co–N(31)	97·5 (2)

Table 2 (*cont.*)

(*B*) Phenanthroline molecules [m = 1,2,3 for phen(1), (2), (3) respectively]

	$m = 1$	$m = 2$	$m = 3$	Mean*
N(m1)–C(m1)	1·306 (12)	1·331 (9)	1·328 (9)	1·322 (14)
N(m1)–C(m12)	1·366 (10)	1·364 (8)	1·354 (9)	1·361 (6)
N(m2)–C(m10)	1·324 (11)	1·336 (9)	1·328 (9)	1·329 (6)
N(m2)–C(m11)	1·340 (9)	1·348 (10)	1·347 (10)	1·345 (4)
C(m1)–C(m2)	1·415 (16)	1·385 (14)	1·396 (14)	1·399 (21)
C(m2)–C(m3)	1·355 (18)	1·337 (14)	1·358 (15)	1·350 (11)
C(m3)–C(m4)	1·382 (16)	1·395 (14)	1·412 (14)	1·396 (15)
C(m4)–C(m5)	1·413 (15)	1·440 (13)	1·422 (13)	1·425 (14)
C(m4)–C(m12)	1·417 (12)	1·374 (12)	1·390 (12)	1·394 (22)
C(m5)–C(m6)	1·316 (22)	1·329 (18)	1·341 (16)	1·329 (13)
C(m6)–C(m7)	1·438 (16)	1·390 (15)	1·424 (14)	1·417 (25)
C(m7)–C(m8)	1·382 (16)	1·413 (13)	1·414 (13)	1·403 (18)
C(m7)–C(m11)	1·412 (11)	1·397 (10)	1·395 (9)	1·401 (9)
C(m8)–C(m9)	1·350 (14)	1·324 (18)	1·352 (15)	1·342 (16)
C(m9)–C(m10)	1·392 (13)	1·385 (12)	1·387 (11)	1·388 (4)
C(m11)–C(m12)	1·419 (12)	1·437 (10)	1·438 (10)	1·431 (11)
C(m1)–N(m1)–C(m12)	119·2 (7)	118·1 (6)	119·0 (7)	118·8 (6)
C(m10)–N(m2)–C(m11)	118·1 (6)	117·9 (6)	117·5 (6)	117·8 (3)
N(m1)–C(m1)–C(m2)	122·0 (9)	121·5 (7)	122·2 (7)	121·9 (4)
C(m1)–C(m2)–C(m3)	119·0 (10)	120·1 (9)	119·2 (9)	119·8 (7)
C(m2)–C(m3)–C(m4)	121·0 (11)	120·4 (10)	119·9 (10)	120·4 (6)
C(m3)–C(m4)–C(m12)	116·9 (9)	117·0 (8)	117·3 (8)	117·1 (2)
C(m5)–C(m4)–C(m12)	118·7 (10)	118·2 (9)	117·9 (8)	118·3 (4)
C(m4)–C(m5)–C(m6)	121·8 (11)	121·0 (10)	122·8 (10)	121·9 (9)
C(m5)–C(m6)–C(m7)	121·5 (10)	122·4 (9)	119·9 (8)	121·3 (13)
C(m6)–C(m7)–C(m11)	118·9 (9)	118·7 (8)	119·8 (8)	119·1 (6)
C(m8)–C(m7)–C(m11)	116·3 (9)	117·2 (9)	116·6 (8)	116·7 (5)
C(m7)–C(m8)–C(m9)	120·5 (9)	119·5 (9)	119·3 (7)	119·8 (6)
C(m8)–C(m9)–C(m10)	119·5 (9)	120·7 (8)	119·8 (8)	120·0 (6)
N(m2)–C(m10)–C(m9)	122·3 (8)	122·2 (9)	122·9 (8)	122·5 (4)
N(m2)–C(m11)–C(m7)	123·3 (8)	122·6 (7)	123·7 (6)	123·2 (6)
C(m7)–C(m11)–C(m12)	118·8 (7)	119·7 (7)	119·1 (7)	119·2 (5)
N(m1)–C(m12)–C(m4)	121·9 (8)	122·9 (7)	122·4 (7)	122·4 (5)
C(m4)–C(m12)–C(m11)	120·2 (7)	120·0 (7)	120·4 (6)	120·2 (2)

* Standard deviations of mean values correspond to spread in values:

$$\sigma(\bar{x}) = \left[\sum_{i=1}^{n} (x_i - \bar{x})^2/(n-1) \right]^{1/2}.$$

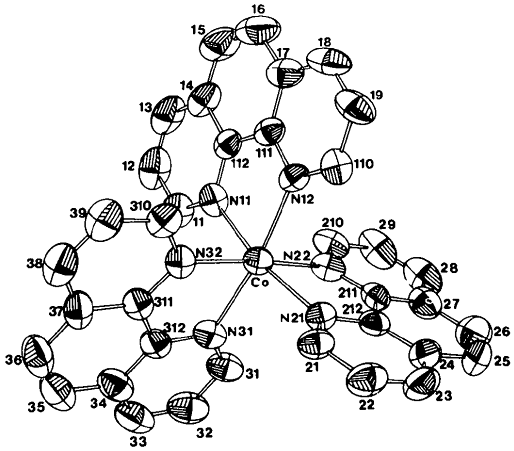

Fig. 1. The complex cation showing the coordination around the Co atom (H atoms omitted for clarity). Thermal ellipsoids are drawn at the 40% probability level.

While the Cl atoms and those of the complex cation refined well in space group $C2/c$, the O atoms of the perchlorate ions, located in difference maps, proved difficult to refine adequately due to high thermal motion and apparent disorder. There are three independent Cl atoms in the structure: one in general position 8 (*f*), and two in special positions 4 (*e*). Two of these have ordered, though highly agitated, perchlorates. One perchlorate in position 4 (*e*) is oriented with its twofold axis along the twofold axis of the site; the other perchlorate in position 4 (*e*) exhibited disorder similar to the corresponding perchlorate in the $[\mathrm{Cu^{II}(phen)_3}](\mathrm{ClO_4})_2$ complex with a Cl–O bond parallel to the twofold axis of the site. This disorder persisted when refinement was attempted in the noncentrosymmetric space group Cc. After several attempts for alternative oxygen sites in both space groups, a final model was chosen in $C2/c$, which consisted of three interlocking tetrahedral $\mathrm{ClO_4}$ groups with different populations. Because of their interdependence, these differently oriented perchlorates were refined separately.

Discussion. Atomic parameters and bond lengths and angles are listed in Tables 1 and 2.*

The analysis confirms that the present complex is isostructural with the $\mathrm{Cu^{II}}$ analogue, consisting of discrete tris(1,10-phenanthroline)cobalt(II) cations and perchlorate anions, with the water molecule [identified by O(1)] being interstitial in the structure. Fig. 1 shows the complex cation with the atom-numbering scheme. The Co atom is hexacoordinated to the N atoms of the three bidentate phenanthroline ligands in a distorted octahedral arrangement. Chemically equivalent bonds and angles in the phenanthroline molecules (Table 2) are in good agreement with each other and compare

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* Lists of structure factors, anisotropic thermal parameters, calculated H-atom parameters, perchlorate bond lengths and angles, intermolecular distances and mean planes have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 39432 (27 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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° ($\sigma < 1{\cdot}0°$).

Co—N bond lengths and N—Co—N bite angles fall within the expected range. The main difference from the $\mathrm{Cu^{II}}$ analogue lies essentially in the coordination bond geometry. The $\mathrm{Cu^{II}}$ 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 $\mathrm{Co^{II}}$ 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, $C2/c$, $a = 28{\cdot}253\ (11)$, $b = 9{\cdot}550\ (2)$, $c = 15{\cdot}761\ (5)$ Å, $\beta = 115{\cdot}78\ (3)°$, $V = 3829{\cdot}3$ Å^3, $Z = 4$, $D_m = 1{\cdot}51\ (1)$, $D_x = 1{\cdot}50$ g cm^{-3}, Mo $K\alpha$, $\lambda = 0{\cdot}71073$ Å, $\mu = 6{\cdot}310$ cm^{-1}, $F(000) = 1132$, room temperature, $R = 0{\cdot}028$, $wR = 0{\cdot}038$ 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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