SHORT-FORMAT PAPERS

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Structure of Bis(4-chlorophenolato)[hydrotris(3,5-dimethyl-1-pyrazolyl)borato]-oxomolybdenum(V)*

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Abstract. $[Mo(C_{15}H_{22}BN_6)(C_6H_4ClO)_2O]$, $M_r = 664.24$, triclinic, $P\bar{1}$, $a = 10.585$ (2), $b = 12.068$ (3), $c = 13.728$ (3) Å, $\alpha = 88.01$, $\beta = 69.94$, $\gamma = 65.78°$, $V = 1490.8$ Å^3, $Z = 2$, $D_x = 1.48$ g cm^{-3}, Mo $K\alpha$, $\lambda = 0.71073$ Å, $\mu = 6.5$ cm^{-1}, $F(000) = 678$, $T = 296$ (1) K, $R = 0.034$, $wR = 0.049$ for 4634 observed independent reflections with $F^2 > 3\sigma(F^2)$. This is the second structurally characterized mononuclear monooxo transition-metal complex containing the phenolato ligand. The molecule exhibits a distorted octahedral coordination geometry, and the Mo atom is ligated by a terminal O atom, two *p*-chloro-substituted phenolato groups, and a tridentate facially coordinated hydrotris(3,5-dimethylpyrazolyl)borate ligand (*L*).

Experimental. The title compound, $LMoO(OC_6H_4Cl)_2$, was synthesized and purified using procedures similar to that of previous literature (Cleland *et al.*, 1987), and crystallized by slow evaporation from a saturated benzene solution at room temperature (3 weeks). A dark ruby-red pyramidal-shaped crystal of approximate dimensions 0.55 × 0.30 × 0.22 mm was mounted on a glass fiber with epoxy. The measurements were performed on an automated four-circle Syntex $P2_1$ diffractometer with graphite-monochromated Mo $K\alpha$ radiation. No absorption correction was applied. ω scans of several intense reflections were measured and the width at half-height was 0.25°. Lattice parameters were obtained from the setting angles of 25 reflections in the range $20 < 2\theta < 30°$. No systematic absences were observed and the space group was determined to be $P\bar{1}$. Data were collected in θ–2θ scan mode with $2\theta_{\max} = 50°$; $(\sin\theta_{\max})/\lambda = 0.595$ Å^{-1}. The scan rate varied from 2 to 8° min^{-1} depending upon the intensity of the reflection. The data covered the index range $0 \le h \le 13$, $-15 \le k \le 15$, $-17 \le l \le 17$. Three standard reflections ($2\bar{4}\bar{2}$, $31\bar{4}$ and 542) were monitored and showed 1.0% decay in intensity; no decay correction was applied. Lorentz and polarization corrections were applied. A total of 5587 reflections were collected. $R_{\rm int} = 0.7\%$ for averaged reflections leading to 5287 unique reflections. Of these, 4634 with $F_o^2 > 3\sigma(F_o^2)$ were used in refinement. The structure was solved using the Patterson method to locate the Mo atom, followed by successive difference Fourier syntheses. Full-matrix least squares, based on F's, using anisotropic thermal parameters, were used to refine all non-H atoms, H atoms were included at calculated positions, with fixed thermal parameters and

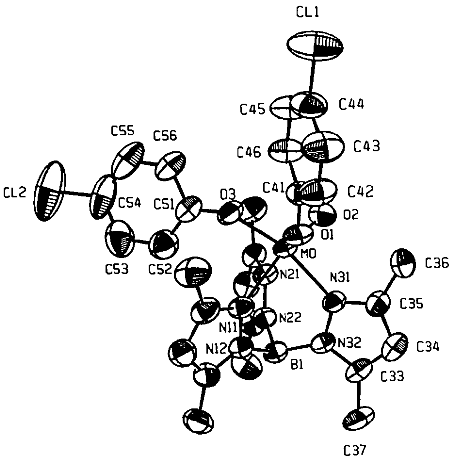

Fig. 1. The structure of $[HB(C_5H_7N_2)_3]MoO(OC_6H_4Cl)_2$. The numbering of the atoms in the pyrazole ring containing N11 and N21 parallels that shown for the ring containing N31.

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Table 1. *Positional and equivalent isotropic thermal parameters*

$U_{eq} = 1/3 \times$ (trace of the orthogonalized U_{ij} matrix).

	x	y	z	U_{eq} (Å^2)
Mo	0.20857 (2)	0.19198 (2)	0.32306 (2)	0.044
O1	0.2812 (2)	0.0159 (2)	0.2851 (1)	0.055
O2	0.0287 (2)	0.2277 (2)	0.2886 (2)	0.059
O3	0.2170 (2)	0.2221 (2)	0.1821 (1)	0.062
N11	0.4588 (2)	0.1511 (2)	0.2636 (2)	0.048
N12	0.5077 (2)	0.2123 (2)	0.3144 (2)	0.046
N21	0.1827 (2)	0.3759 (2)	0.3573 (2)	0.046
N22	0.2718 (2)	0.3973 (2)	0.3998 (2)	0.045
N31	0.2584 (2)	0.1565 (2)	0.4640 (2)	0.047
N32	0.3382 (2)	0.2078 (2)	0.4911 (2)	0.045
C13	0.6468 (3)	0.1942 (3)	0.2565 (2)	0.055
C14	0.6901 (3)	0.1205 (3)	0.1668 (3)	0.061
C15	0.5720 (3)	0.0940 (3)	0.1733 (2)	0.057
C16	0.5647 (4)	0.0164 (4)	0.0962 (3)	0.079
C17	0.7313 (3)	0.2483 (3)	0.2918 (3)	0.075
C23	0.2298 (3)	0.5193 (2)	0.4136 (2)	0.052
C24	0.1112 (3)	0.5761 (3)	0.3822 (3)	0.057
C25	0.0838 (3)	0.4853 (3)	0.3477 (2)	0.049
C26	−0.0341 (3)	0.4997 (3)	0.3053 (3)	0.063
C27	0.3082 (3)	0.5747 (3)	0.4543 (3)	0.075
C33	0.3524 (3)	0.1697 (3)	0.5816 (2)	0.053
C34	0.2797 (3)	0.0964 (3)	0.6144 (2)	0.062
C35	0.2219 (3)	0.0899 (3)	0.5392 (2)	0.054
C36	0.1310 (3)	0.0238 (3)	0.5370 (3)	0.082
C37	0.4360 (3)	0.2047 (4)	0.6337 (2)	0.083
C41	0.2352 (3)	−0.0476 (2)	0.2365 (2)	0.048
C42	0.2955 (4)	−0.1733 (3)	0.2326 (3)	0.084
C43	0.2484 (4)	−0.2422 (3)	0.1874 (3)	0.093
C44	0.1442 (3)	−0.1857 (3)	0.1446 (3)	0.080
C45	0.0848 (4)	−0.0630 (3)	0.1463 (3)	0.085
C46	0.1298 (3)	0.0077 (3)	0.1917 (3)	0.068
Cl1	0.0828 (1)	−0.2719 (1)	0.0881 (1)	0.152
C51	0.2474 (3)	0.2885 (3)	0.1057 (2)	0.054
C52	0.3472 (4)	0.3373 (3)	0.0938 (3)	0.085
C53	0.3794 (5)	0.4018 (4)	0.0081 (4)	0.104
C54	0.3079 (6)	0.4146 (4)	−0.0616 (3)	0.091
C55	0.2063 (5)	0.3696 (4)	−0.0482 (3)	0.097
C56	0.1752 (4)	0.3077 (4)	0.0339 (3)	0.078
Cl2	0.3525 (3)	0.4925 (1)	−0.1681 (1)	0.166
B1	0.3992 (3)	0.2896 (3)	0.4196 (2)	0.046

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

Numbers in parentheses are the estimated standard deviations in the least significant figure.

Mo—O1	1.959 (3)	Mo—N21	2.170 (3)
Mo—O2	1.673 (3)	Mo—N31	2.160 (3)
Mo—O3	1.934 (3)	O1—C41	1.354 (5)
Mo—N11	2.324 (4)	O3—C51	1.330 (6)

O1—Mo—O2	99.6 (2)	O1—Mo—N31	90.1 (1)
O2—Mo—O3	103.3 (2)	O2—Mo—N11	168.9 (1)
O1—Mo—O3	90.7 (1)	O2—Mo—N21	93.1 (1)
Mo—O1—C41	131.5 (3)	O2—Mo—N31	91.7 (1)
Mo—O3—C51	145.3 (3)	O3—Mo—N11	86.8 (1)
O1—Mo—N11	84.6 (1)	O3—Mo—N21	90.5 (1)
O1—Mo—N21	166.6 (1)	O3—Mo—N31	164.6 (1)

constrained to ride on the atom to which they are bonded. The refinement (361 parameters) converged to $R = 0.034$, $wR = 0.049$ and maximum shift $<0.005\sigma$. The weighting scheme used was $w = 4(F_o^2)/\sigma^2(F_o^2)$. The residual electron density showed no special features; min. and max. values were -0.64 and $0.52\ \text{e Å}^{-3}$, respectively. Atomic scattering factors were taken from Cromer & Waber (1974). Anomalous-dispersion effects were included in F_c and the values for f' and f'' were those of Cromer (1974). All calculations were performed on a VAX computer using the programs *SDP* (Frenz, 1978) and *ORTEP* (Johnson, 1965). Positional and equivalent isotropic thermal parameters are listed in Table 1 and selected interatomic distances and bond angles in Table 2.* Fig. 1 shows the molecular structures.

Related literature. The title compound is part of a series of LMoO(X)(Y) compounds (Chang & Enemark, 1991) with similar steric and geometric properties that have been synthesized to investigate the role of inductive effects of the ligand ring substituents on the properties of the molybdenum center (Chang, 1991). Other crystallographically characterized members of this series include $L\text{MoO}(\text{SC}_6\text{H}_5)_2$ (Cleland *et al.*, 1987) and $L\text{MoO}(\text{OC}_6\text{H}_5)_2$ (Kipke, Cleland, Roberts & Enemark, 1989).

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* Lists of structure factors, anisotropic thermal parameters, H-atom parameters, bond distances, bond angles, torsional angles and best least-squares planes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54776 (32 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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