Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å^2)*

$$U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}
Ni	1	0	1/2	0.03644 (12)
S1	0.97031 (6)	0.23678 (5)	0.41892 (5)	0.04808 (14)
S2	0.72190 (6)	0.07596 (5)	0.67155 (5)	0.04832 (14)
N1	0.6415 (3)	0.6505 (2)	0.5154 (2)	0.0659 (5)
N2	0.3210 (3)	0.4437 (2)	0.8404 (2)	0.0682 (5)
N3	0.9320 (2)	0.7717 (2)	0.1689 (2)	0.0494 (4)
C1	0.7515 (2)	0.3483 (2)	0.5421 (2)	0.0412 (3)
C2	0.6434 (2)	0.2793 (2)	0.6528 (2)	0.0410 (3)
C3	0.6897 (3)	0.5163 (2)	0.5272 (2)	0.0477 (4)
C4	0.4637 (2)	0.3708 (2)	0.7567 (2)	0.0481 (4)
C5	0.8114 (3)	0.8859 (3)	0.2822 (2)	0.0526 (4)
C6	0.6943 (3)	1.0181 (3)	0.2604 (2)	0.0559 (5)
C7	0.6958 (3)	1.0387 (2)	0.1214 (2)	0.0526 (4)
C8	0.8225 (3)	0.9194 (3)	0.0066 (2)	0.0570 (5)
C9	0.9379 (3)	0.7887 (3)	0.0324 (2)	0.0545 (5)
C10	1.0538 (4)	0.6269 (4)	0.1965 (3)	0.0666 (6)
C11	0.5653 (4)	1.1819 (3)	0.0969 (4)	0.0740 (7)

Table 2. *Selected geometric parameters ($\text{Å}, °$)*

Ni—S2	2.1659 (6)	C1—C2	1.362 (3)
Ni—S1	2.1762 (5)	C1—C3	1.430 (2)
S1—C1	1.730 (2)	C2—C4	1.429 (2)
S2—C2	1.735 (2)	C5—C6	1.359 (3)
N1—C3	1.140 (3)	C6—C7	1.385 (3)
N2—C4	1.139 (2)	C7—C8	1.392 (3)
N3—C9	1.344 (3)	C7—C11	1.493 (3)
N3—C5	1.349 (2)	C8—C9	1.360 (3)
N3—C10	1.475 (3)		
S2—Ni—S1	92.36 (2)	C2—C1—S1	121.06 (13)
C1—S1—Ni	102.81 (7)	C1—C2—S2	120.39 (13)
C2—S2—Ni	103.21 (6)		

The structure was solved by direct methods and refined by full-matrix least squares. All the H atoms were located from difference maps and refined isotropically.

Data collection: *XSCANS* (Siemens, 1994). Cell refinement: *XSCANS*. Data reduction: *XSCANS*. Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1990a). Program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993). Molecular graphics: *SHELXTL/PC* (Sheldrick, 1990b). Software used to prepare material for publication: *SHELXL93*. Geometric calculations: *PARST* (Nardelli, 1983).

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: MU1222). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

Clemenson, P. I. (1990). *Coord. Chem. Rev.* **106**, 171–203.
Hove, M. J., Hoffman, B. M. & Ibers, J. A. (1972). *J. Chem. Phys.* **56**, 3490–3502.

Mahadevan, C., Seshasayee, M., Radha, A. & Manoharan, P. T. (1984). *Acta Cryst.* C40, 2032–2034.
Manoharan, P. T., Noordik, J. H., de Boer, E. & Keijzers, C. P. (1981). *J. Chem. Phys.* **74**, 1980–1989.
Nardelli, M. (1983). *Comput. Chem.* **7**, 95–98.
Sheldrick, G. M. (1990a). *Acta Cryst.* A46, 467–473.
Sheldrick, G. M. (1990b). *SHELXTL/PC Users Manual.* Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
Sheldrick, G. M. (1993). *SHELXL93. Program for the Refinement of Crystal Structures.* University of Göttingen, Germany.
Siemens (1994). *XSCANS Users Manual.* Version 2.1. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.

cis-Bis(2-methyl-8-quinolinolato-N,O)-dioxomolybdenum(VI)

Hoong-Kun Fun,[a]* Kandasamy Sivakumar,[a]† Jing-Yang Niu,[b] Jing-Ping Wang[b] and Xiao-Zeng You[b]

[a]*X-ray Crystallography Laboratory, School of Physics, Universiti Sains Malaysia, 11800 USM, Penang, Malaysia, and [b]Coordination Chemistry Institute, State Key Laboratory of Coordination Chemistry, Centre for Advanced Studies in Science and Technology of Microstructures, Nanjing University, Nanjing 210093, People's Republic of China. E-mail: hkfun@usm.my*

Abstract

In [Mo($C_{10}H_8NO$)$_2$(O)$_2$], the metal coordination is octahedral. The oxo ligands are *cis* with respect to each other and *trans* with respect to the quinolinolato N donor atoms. The Mo—O_{oxo} distances are shorter than the Mo—$O_{quinolinolato}$ bond lengths. One of the oxo ligands is involved in C—H$\cdots$O hydrogen bonding.

Comment

Molybdenum and its complexes have been found to be useful in both biological and industrial applications (Amos & Sawyer, 1974). 2-Methyl-8-quinolinol is an important reagent in analytical and industrial chemistry. The structures of its complexes with gallium (Shiro & Fernand, 1971), aluminium (Kushi & Fernand, 1970) and palladium (Prout & Wheeler, 1966) have been reported. The properties of the 2-methyl-8-quinolinol complex with molybdenum have been studied in solution (Howie, Bosserman & Sawyer, 1980; Amos & Sawyer, 1974; Miki, Masano & Iwasaki, 1993), but the crystal structure of the title compound, (I), has not, to our knowledge, been reported.

† On leave from the Department of Physics, Anna University, Madras 600 025, India.

(I)
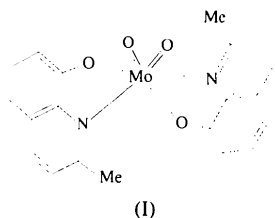
A displacement ellipsoid plot of the title complex is shown in Fig. 1. The Mo—O_{oxo} bond distances are 1.721 (2) and 1.727 (2) Å, and those involving the O atoms of the 2-methyl-8-quinolinolato ligands are 1.9717 (14) and 1.9878 (14) Å. The Mo—N distances [Mo—N1 2.411 (2) and Mo—N2 2.492 (2) Å] differ significantly and are longer than the Mo—N bonds found in the Ga, Al and Pd complexes of 2-methyl-8-quinolinol. The M—O distances in the Ga and Al complexes are longer than those found here, which
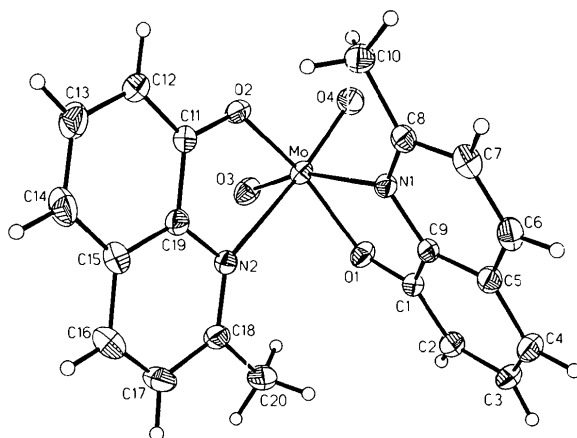

Fig. 1. A displacement ellipsoid plot (50% probability) of the title molecule with the atomic numbering scheme.
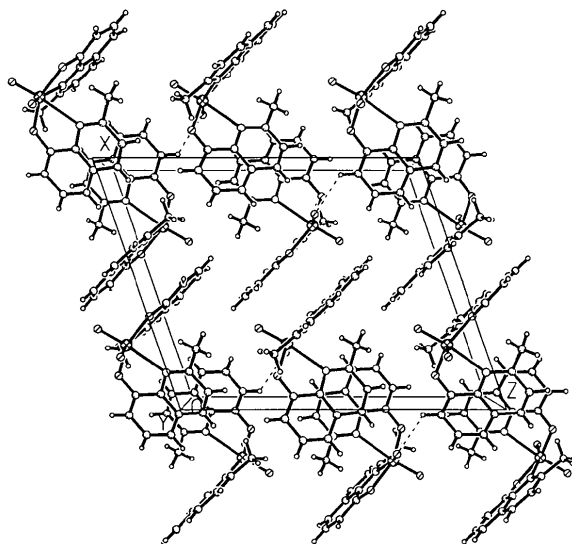

Fig. 2. The packing of the title molecules viewed down the b axis.

are in turn longer than the M—O distances found in the Pd complex. The N—M—O bite angles of the 2-methyl-8-quinolinolato ligand are 84.2, 82.8 and 84.0°, respectively, in the Ga, Al and Pd complexes, reducing to 72.73 (6) and 73.74 (6)° in the Mo complex. The two ligands are nearly planar and the dihedral angle between them is 98.21 (4)°. One of the two oxo groups (O3) is involved in three possible C—H$\cdots$O contacts, whereas the other oxo group (O4) is not involved in any such contacts: C7$\cdots$O3^i 3.313 (3) Å and C7—H7$\cdots$O3^i 148 (2)°, C10$\cdots$O3^i 3.308 (3) Å and C10—H10C$\cdots$O3^i 135 (2)°, and C12—O3^{ii} 3.173 (3) Å and C12—H12$\cdots$O3^{ii} 126 (2)° [symmetry codes: (i) x, $1 + y$, z; (ii) $2 - x$, $\frac{1}{2} + y$, $\frac{1}{2} - z$].

Experimental

The title compound was synthesized by the reaction of $H_3PMo_{12}O_{40}.nH_2O$ with 2-methyl-8-quinolinol in alcohol solution. Single crystals suitable for X-ray analysis were obtained from an acetonitrile/water solution by slow evaporation.

Crystal data

[Mo(C$_{10}$H$_8$NO)$_2$(O)$_2$]	Mo $K\alpha$ radiation
$M_r = 444.29$	$\lambda = 0.71073$ Å
Monoclinic	Cell parameters from 6199
$P2_1/c$	reflections
$a = 13.3698$ (2) Å	$\theta = 1.62–26.30°$
$b = 8.5803$ (1) Å	$\mu = 0.761$ mm^{-1}
$c = 16.5573$ (1) Å	$T = 153$ (2) K
$\beta = 109.459$ (1)°	Prism
$V = 1790.91$ (4) Å^3	$0.40 \times 0.40 \times 0.30$ mm
$Z = 4$	Yellow
$D_x = 1.648$ Mg m^{-3}	
D_m not measured	

Data collection

Siemens SMART area-detector diffractometer	3246 observed reflections $[I > 2\sigma(I)]$
ω scans	$R_{int} = 0.0191$
Absorption correction: none	$\theta_{max} = 26.30°$
	$h = -16 \rightarrow 14$
6199 measured reflections	$k = -10 \rightarrow 4$
3301 independent reflections	$l = -20 \rightarrow 19$

Refinement

Refinement on F^2	$\Delta\rho_{max} = 0.31$ e Å^{-3}
$R(F) = 0.0229$	$\Delta\rho_{min} = -0.33$ e Å^{-3}
$wR(F^2) = 0.0556$	Extinction correction:
$S = 1.143$	SHELXL93 (Sheldrick,
3301 reflections	1993)
309 parameters	Extinction coefficient:
All H-atom parameters refined	0.0036 (3)
$w = 1/[\sigma^2(F_o^2) + (0.0066P)^2 + 2.1247P]$	Atomic scattering factors from International Tables
where $P = (F_o^2 + 2F_c^2)/3$	for Crystallography (1992, Vol. C, Tables 4.2.6.8 and
$(\Delta/\sigma)_{max} < 0.001$	6.1.1.4)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters ($\mathring{A}^2$)*

$$U_{eq} = (1/3)\Sigma_i\Sigma_jU_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}
Mo	0.746823 (13)	0.15256 (2)	0.106914 (10)	0.01862 (8)
N1	0.68030 (13)	0.3970 (2)	0.03812 (10)	0.0185 (3)
N2	0.85932 (14)	0.1726 (2)	0.01384 (11)	0.0204 (4)
O1	0.62936 (11)	0.0997 (2)	0.00005 (9)	0.0237 (3)
O2	0.87571 (11)	0.2691 (2)	0.17166 (9)	0.0228 (3)
O3	0.78954 (13)	−0.0384 (2)	0.12405 (10)	0.0284 (4)
O4	0.68206 (12)	0.1876 (2)	0.17916 (10)	0.0276 (3)
C1	0.5771 (2)	0.2072 (3)	−0.05969 (13)	0.0217 (4)
C2	0.4984 (2)	0.1664 (3)	−0.13632 (14)	0.0274 (5)
C3	0.4472 (2)	0.2864 (3)	−0.19557 (14)	0.0316 (5)
C4	0.4707 (2)	0.4421 (3)	−0.17812 (14)	0.0299 (5)
C5	0.5494 (2)	0.4872 (3)	−0.09871 (13)	0.0233 (4)
C6	0.5746 (2)	0.6437 (3)	−0.07057 (15)	0.0258 (5)
C7	0.6469 (2)	0.6727 (3)	0.00931 (15)	0.0242 (4)
C8	0.6996 (2)	0.5466 (2)	0.06402 (13)	0.0203 (4)
C9	0.6038 (2)	0.3676 (2)	−0.04086 (13)	0.0202 (4)
C10	0.7764 (2)	0.5817 (3)	0.15304 (14)	0.0255 (5)
C11	0.9569 (2)	0.3074 (3)	0.14243 (13)	0.0222 (4)
C12	1.0443 (2)	0.3932 (3)	0.19175 (15)	0.0283 (5)
C13	1.1273 (2)	0.4286 (3)	0.1587 (2)	0.0336 (5)
C14	1.1232 (2)	0.3780 (3)	0.0786 (2)	0.0330 (5)
C15	1.0344 (2)	0.2887 (3)	0.02683 (14)	0.0260 (5)
C16	1.0244 (2)	0.2245 (3)	−0.0552 (2)	0.0331 (5)
C17	0.9361 (2)	0.1383 (3)	−0.09833 (15)	0.0325 (5)
C18	0.8534 (2)	0.1125 (3)	−0.06299 (13)	0.0246 (5)
C19	0.9498 (2)	0.2563 (2)	0.05844 (13)	0.0211 (4)
C20	0.7595 (2)	0.0141 (3)	−0.1129 (2)	0.0351 (6)

Table 2. *Selected geometric parameters ($\mathring{A}$, °)*

Mo—O4	1.721 (2)	Mo—O1	1.9878 (14)
Mo—O3	1.727 (2)	Mo—N1	2.411 (2)
Mo—O2	1.9717 (14)	Mo—N2	2.492 (2)
O4—Mo—O3	105.49 (8)	O2—Mo—N1	86.20 (6)
O4—Mo—O2	94.84 (7)	O1—Mo—N1	73.74 (6)
O3—Mo—O2	102.15 (7)	O4—Mo—N2	164.77 (7)
O4—Mo—O1	103.14 (7)	O3—Mo—N2	86.12 (7)
O3—Mo—O1	92.86 (7)	O2—Mo—N2	72.73 (6)
O2—Mo—O1	152.61 (6)	O1—Mo—N2	85.75 (6)
O4—Mo—N1	89.11 (7)	N1—Mo—N2	81.46 (6)
O3—Mo—N1	162.29 (7)		

Data were collected using a Siemens CCD SMART System fitted with a low-temperature attachment.

Data collection: *ASTRO* (Siemens, 1995). Cell refinement: *SAINT* (Siemens, 1995). Data reduction: *SAINT*. Program(s) used to solve structure: *SHELXTL/PC* (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993). Molecular graphics: *SHELXTL/PC*. Software used to prepare material for publication: *SHELXL93*. Geometrical calculations: *PARST* (Nardelli, 1983).

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: MU1231). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

Amos, L. W. & Sawyer, D. T. (1974). *Inorg. Chem.* **13**, 78–83.
Howie, J. K., Bosserman, P. & Sawyer, D. T. (1980). *Inorg. Chem.* **19**, 2293–2296.
Kushi, Y. & Fernand, Q. (1970). *J. Am. Chem. Soc.* **92**, 91–96.
Miki, E., Masano, H. & Iwasaki, H. (1993). *Inorg. Chim. Acta*, **205**, 129–136.
Nardelli, M. (1983). *Comput. Chem.* **7**, 95–98.
Prout, C. K. & Wheeler, A. G. (1966). *J. Chem. Soc.* pp. 1286–1290.
Sheldrick, G. M. (1990). *SHELXTL/PC. Structure Determination Software Programs.* Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
Sheldrick, G. M. (1993). *SHELXL93. Program for the Refinement of Crystal Structures.* University of Göttingen, Germany.
Shiro, M. & Fernand, Q. (1971). *Anal. Chem.* **43**, 1222–1230.
Siemens (1995). *ASTRO* and *SAINT. Data Collection and Processing Software for the SMART System.* Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.

Bis[4-(4-dimethylaminostyryl)-N-methylpyridinium] Bis[maleonitriledithiolato(2–)-S,S']nickelate(II)

Hoong-Kun Fun,[a]* Kandasamy Sivakumar,[a]† Bao-Zhen Shan[b] and Xiao-Zeng You[b]

[a]*X-ray Crystallography Laboratory, School of Physics, Universiti Sains Malaysia, 11800 USM, Penang, Malaysia,* and [b]*Coordination Chemistry Institute, State Key Laboratory of Coordination Chemistry, Centre for Advanced Studies in Science and Technology of Microstructures, Nanjing University, Nanjing 210093, People's Republic of China. E-mail: hkfun@usm.my*

Abstract

In the title salt, $(C_{16}H_{19}N_2)_2[Ni(C_4N_2S_2)_2]$, the bis-[maleonitriledithiolato(2−)-*S,S'*]nickelate(II) anion and the two 4-(4-dimethylaminostyryl)-*N*-methylpyridinium cations are individually planar. The plane of the anion bisects the angle between the planes of the two independent cations.

† On leave from the Department of Physics, Anna University, Madras 600 025, India.