Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å²)*

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

	x	y	z	U_{eq}
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 (Å, °)*

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: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *SHELXTL/PC*. Software used to prepare material for publication: *SHELXL*93. Geometrical calculations: *PARST* (Nardelli, 1983).

The authors would like to thank the Malaysian Government and the Universiti Sains Malaysia for research grant R&D No. 123-3417-2201, and the State Science and Technology Commission and the National Nature Science Foundation of China for a Major Key Research Project. H-KF would like to thank Drs D. Bertlemen and C. Champana of Siemens for the use of the CCD SMART System at Madison, Wisconsin, USA. KS thanks the Universiti Sains Malaysia for a Visiting Postdoctoral Research Fellowship.

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). *SHELXL*93. *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.

Acta Cryst. (1996). C**52**, 1152–1154

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*

(*Received 13 November 1995; accepted 13 December 1995*)

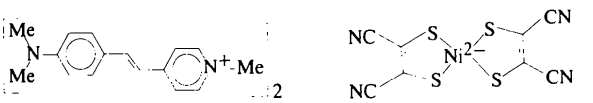
Abstract

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.

Comment

Salts of metal dithiolate complex anions, $[M(mnt)_2]^{n-}$ [M = Ni, Zn, *etc.*; mnt = maleonitriledithiolate(2−)], with a variety of cations are found to possess interesting magnetic, electrical and optical properties (Manoharan, Noordik, de Boer & Keijzers, 1981; Clemenson, 1990). As part of our work on the synthesis and characterization of such complexes (Shan, Zhang, You, Fun & Sivakumar, 1995), we report the structure determination of the title compound, (I).



(I)

Bond lengths and angles in the anion and cations are normal. The Ni atom has square-planar coordination and the NiS_4 chromophore is planar. Unlike other complexes of this type where the Ni atom occupies an inversion centre which relates pairs of adjacent cations, the asymmetric unit comprises an anion and two adjacent cations which have their longest molecular axes roughly parallel (Fig. 1). The anion bisects the angle between the planes of the independent cations, with a dihedral angle of 76.41 (4)° between the planes of the anion and cation A and an angle of 76.88 (3)° between those of the anion and cation B; the dihedral angle between the planes of cations A and B is 153.29 (5)°. There are no unusually short interionic contacts in this structure.

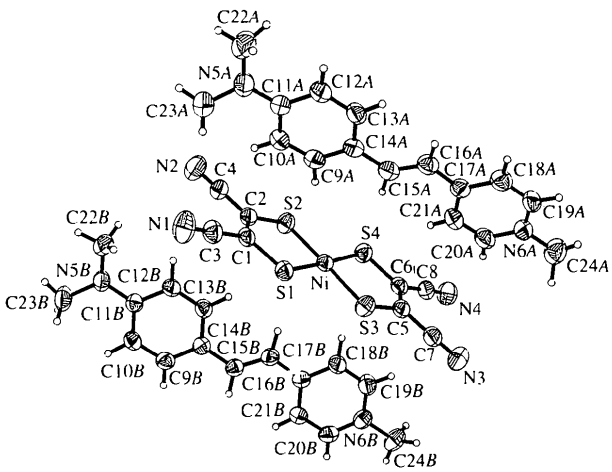


Fig. 1. A 50% displacement ellipsoid plot of (I) showing the atomic numbering scheme.

Experimental

The title salt was synthesized by the addition of an alcoholic solution of the pyridinium cation (as its iodide salt) to a solution of $(NBu_4)_2[Ni(mnt)_2]$. Single crystals were obtained by recrystallization from dimethylformamide.

Crystal data

$(C_{16}H_{19}N_2)_2[Ni(C_4N_2S_2)_2]$
M_r = 817.73
Triclinic
$P\bar{1}$
a = 7.438 (1) Å
b = 15.332 (2) Å
c = 17.722 (3) Å
α = 103.36 (1)°
β = 92.14 (1)°
γ = 101.70 (1)°
V = 1917.9 (5) Å^3
Z = 2
D_x = 1.416 Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
λ = 0.71073 Å
Cell parameters from 25 reflections
θ = 8–25°
μ = 0.765 mm^{-1}
T = 293 (2) K
Plate
0.60 × 0.42 × 0.12 mm
Black

Data collection

Siemens $P4$ diffractometer
$\theta/2\theta$ scans
Absorption correction:
ψ scans (*XSCANS*; Siemens, 1994)
T_{min} = 0.823, T_{max} = 0.977
9967 measured reflections
8120 independent reflections
5642 observed reflections [$I > 2\sigma(I)$]

R_{int} = 0.021
θ_{max} = 27.5°
$h = -1 \rightarrow 9$
$k = -19 \rightarrow 19$
$l = -23 \rightarrow 23$
3 standard reflections monitored every 97 reflections
intensity decay: <3%

Refinement

Refinement on F^2
$R(F)$ = 0.0368
$wR(F^2)$ = 0.1059
S = 0.934
8120 reflections
630 parameters
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0620P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max}$ = −0.001
$\Delta\rho_{max}$ = 0.38 e Å^{-3}
$\Delta\rho_{min}$ = −0.26 e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

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

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

	x	y	z	U_{eq}
Ni	0.20145 (4)	0.01576 (2)	0.253946 (14)	0.03621 (9)
S1	0.40326 (8)	0.14430 (4)	0.29683 (3)	0.04304 (14)
S2	−0.02857 (8)	0.08258 (4)	0.28050 (3)	0.04676 (14)
S3	0.42859 (8)	−0.05420 (4)	0.23103 (4)	0.04805 (14)
S4	−0.00249 (8)	−0.11001 (4)	0.20687 (3)	0.04729 (15)
N1	0.4206 (4)	0.3955 (2)	0.3673 (2)	0.0787 (7)
N2	−0.1187 (4)	0.3180 (2)	0.3392 (2)	0.0741 (7)
N3	0.5056 (4)	−0.2927 (2)	0.16451 (14)	0.0676 (6)
N4	−0.0363 (3)	−0.3631 (2)	0.14397 (13)	0.0647 (6)
C1	0.2669 (3)	0.22441 (14)	0.31856 (11)	0.0397 (5)
C2	0.0807 (3)	0.19764 (15)	0.31075 (12)	0.0406 (5)
C3	0.3545 (4)	0.3193 (2)	0.34546 (14)	0.0518 (6)
C4	−0.0317 (4)	0.2636 (2)	0.32670 (13)	0.0484 (5)
C5	0.3159 (3)	−0.16793 (15)	0.19670 (11)	0.0412 (5)
C6	0.1291 (3)	−0.19216 (15)	0.18767 (11)	0.0412 (5)
C7	0.4225 (4)	−0.2366 (2)	0.17914 (13)	0.0480 (5)
C8	0.0361 (4)	−0.2868 (2)	0.16318 (12)	0.0468 (5)
N5A	0.2297 (4)	0.4324 (2)	0.10566 (12)	0.0677 (7)
N6A	0.2770 (3)	−0.28008 (14)	−0.09130 (11)	0.0490 (5)
C9A	0.1974 (4)	0.1937 (2)	0.11535 (13)	0.0505 (6)

C10*A*	0.1969 (4)	0.2853 (2)	0.13686 (13)	0.0508 (6)
C11*A*	0.2325 (3)	0.3410 (2)	0.08395 (13)	0.0474 (5)
C12*A*	0.2670 (4)	0.2979 (2)	0.00840 (13)	0.0500 (6)
C13*A*	0.2684 (3)	0.2058 (2)	−0.01183 (13)	0.0474 (5)
C14*A*	0.2343 (3)	0.1507 (2)	0.04111 (12)	0.0450 (5)
C15*A*	0.2346 (3)	0.0532 (2)	0.02316 (14)	0.0484 (5)
C16*A*	0.2683 (4)	0.0018 (2)	−0.04342 (14)	0.0502 (6)
C17*A*	0.2714 (3)	−0.0947 (2)	−0.05712 (12)	0.0438 (5)
C18*A*	0.3172 (4)	−0.1407 (2)	−0.12901 (14)	0.0519 (6)
C19*A*	0.3195 (4)	−0.2309 (2)	−0.14436 (14)	0.0528 (6)
C20*A*	0.2346 (4)	−0.2376 (2)	−0.02067 (14)	0.0543 (6)
C21*A*	0.2298 (4)	−0.1480 (2)	−0.00238 (14)	0.0521 (6)
C22*A*	0.2578 (7)	0.4893 (3)	0.0511 (2)	0.0779 (10)
C23*A*	0.2434 (7)	0.4809 (3)	0.1864 (2)	0.0784 (10)
C24*A*	0.2762 (6)	−0.3789 (2)	−0.1088 (2)	0.0718 (8)
N5*B*	0.1694 (3)	0.40742 (13)	0.61611 (11)	0.0531 (5)
N6*B*	0.3070 (3)	−0.29303 (13)	0.43527 (11)	0.0456 (4)
C9*B*	0.2612 (3)	0.1888 (2)	0.64186 (12)	0.0438 (5)
C10*B*	0.2402 (3)	0.2780 (2)	0.65959 (12)	0.0453 (5)
C11*B*	0.1963 (3)	0.31897 (15)	0.60023 (12)	0.0405 (5)
C12*B*	0.1766 (3)	0.2652 (2)	0.52332 (12)	0.0419 (5)
C13*B*	0.1969 (3)	0.1766 (2)	0.50690 (12)	0.0422 (5)
C14*B*	0.2389 (3)	0.13488 (15)	0.56573 (12)	0.0394 (5)
C15*B*	0.2602 (3)	0.0403 (2)	0.55064 (12)	0.0416 (5)
C16*B*	0.2395 (3)	−0.0195 (2)	0.48172 (13)	0.0421 (5)
C17*B*	0.2638 (3)	−0.11269 (15)	0.46800 (12)	0.0391 (4)
C18*B*	0.2369 (3)	−0.1682 (2)	0.39248 (12)	0.0466 (5)
C19*B*	0.2591 (4)	−0.2563 (2)	0.37714 (14)	0.0509 (6)
C20*B*	0.3333 (3)	−0.2416 (2)	0.50888 (14)	0.0507 (6)
C21*B*	0.3116 (4)	−0.1540 (2)	0.52662 (13)	0.0487 (6)
C22*B*	0.1659 (5)	0.4539 (2)	0.5536 (2)	0.0570 (7)
C23*B*	0.2061 (6)	0.4648 (2)	0.6948 (2)	0.0652 (8)
C24*B*	0.3320 (5)	−0.3885 (2)	0.4166 (2)	0.0620 (7)

Table 2. *Selected geometric parameters (Å, °)*

Anion			
Ni—S4	2.1658 (7)	N2—C4	1.143 (3)
Ni—S1	2.1766 (7)	N3—C7	1.146 (3)
Ni—S2	2.1787 (7)	N4—C8	1.150 (3)
Ni—S3	2.1805 (7)	C1—C2	1.355 (3)
S1—C1	1.732 (2)	C1—C3	1.429 (3)
S2—C2	1.735 (2)	C2—C4	1.424 (3)
S3—C5	1.730 (2)	C5—C6	1.357 (3)
S4—C6	1.728 (2)	C5—C7	1.428 (3)
N1—C3	1.140 (3)	C6—C8	1.432 (3)
S4—Ni—S1	177.48 (2)	S4—Ni—S3	92.22 (3)
S4—Ni—S2	86.92 (3)	S1—Ni—S3	88.64 (3)
S1—Ni—S2	92.31 (3)	S2—Ni—S3	177.64 (2)
	Cation *A*		Cation *B*
N5—C11	1.370 (3)		1.377 (3)
N5—C22	1.440 (4)		1.450 (3)
N5—C23	1.442 (4)		1.450 (3)
N6—C19	1.344 (3)		1.352 (3)
N6—C20	1.351 (3)		1.343 (3)
N6—C24	1.473 (4)		1.477 (3)
C9—C10	1.369 (3)		1.374 (3)
C9—C14	1.393 (3)		1.394 (3)
C10—C11	1.405 (3)		1.404 (3)
C11—C12	1.407 (3)		1.406 (3)
C12—C13	1.376 (3)		1.363 (3)
C13—C14	1.399 (3)		1.399 (3)
C14—C15	1.455 (3)		1.454 (3)
C15—C16	1.322 (3)		1.329 (3)
C16—C17	1.447 (3)		1.443 (3)
C17—C18	1.398 (3)		1.393 (3)
C17—C21	1.411 (3)		1.405 (3)
C18—C19	1.349 (4)		1.359 (3)
C20—C21	1.346 (4)		1.351 (3)

Data collection: *XSCANS* (Siemens, 1994). Cell refinement: *XSCANS*. Data reduction: *XSCANS*. Program(s) used to solve structure: *SHELXTL/PC* (Sheldrick, 1990) by direct methods. Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *SHELXTL/PC*. Software used to prepare material for publication: *SHELXL*93. Geometrical calculations: *PARST* (Nardelli, 1983).

The authors would like to thank the Malaysian Government and the Universiti Sains Malaysia for research grant R&D No. 123-3417-2201, and the State Science and Technology Commission and the National Nature Science Foundation of China for a Major Key Research Project. KS thanks the Universiti Sains Malaysia for a Visiting Postdoctoral Research Fellowship.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: MU1233). 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.
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.
Shan, B.-Z., Zhang, X.-M., You, X.-Z., Fun, H.-K. & Sivakumar, K. (1995). *Acta Cryst.* C**52**. In the press.
Sheldrick, G. M. (1990). *SHELXTL/PC. Structure Determination Software Programs*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
Sheldrick, G. M. (1993). *SHELXL*93. *Program for the Refinement of Crystal Structures*. University of Göttingen, Germany.
Siemens (1994). *XSCANS. X-ray Single Crystal Analysis System*. Version 2.1. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.

Acta Cryst. (1996). C**52**, 1154–1156

Bis(*N*-benzyl-2-hydroxy-1-naphthaldiminato)nickel(II)

YALCIN ELERMAN,[a] MEHMET KABAK[a] AND M. NAWAZ TAHIR[b]

[a]*Department of Engineering Physics, Faculty of Sciences, University of Ankara, 06100 Besevler, Ankara, Turkey, and* [b]*Department of Engineering Physics, Faculty of Engineering, Hacettepe University, 06532 Beytepe, Ankara, Turkey. E-mail: auffdek-e@servis.net.tr*

(*Received 27 April 1995; accepted 27 November 1995*)

Abstract

The crystal structure of the title compound, bis-[1-(benzyliminomethyl)-2-naphtholato-*N*,*O*]nickel(II), $[Ni(C_{18}H_{14}NO)_2]$, has been determined. Two bidentate Schiff base ligands coordinate to the Ni atom in a square-planar arrangement.