

O2—Co—O1	83.6 (2)	N1—Co—N3	92.5 (2)
O2—Co—N1	174.6 (2)	N2—Co—N3	91.7 (2)
O1—Co—N1	91.0 (2)	O2—Co—N4	88.4 (2)
O2—Co—N2	91.6 (2)	O1—Co—N4	88.1 (2)
O1—Co—N2	175.1 (3)	N1—Co—N4	90.9 (2)
N1—Co—N2	93.8 (3)	N2—Co—N4	92.6 (2)
O2—Co—N3	87.8 (2)	N3—Co—N4	174.4 (2)
O1—Co—N3	87.3 (2)		

The structure was solved by direct methods and refined by full-matrix least-squares methods on F^2 . Atoms C8 and C9 were found to be disordered over two positions (C8, C8' and C9, C9'); initially their occupancies were refined, then later fixed at 0.7 (C8 and C9) and 0.3 (C8' and C9'). The disordered non-H atoms in the salpron ligand and the atoms of the solvent pyridine molecule were refined isotropically whereas the remaining non-H atoms were refined anisotropically. All the H atoms were generated geometrically and allowed to ride on their respective parent atoms, but not refined. The H atoms attached to C8 and C9 have occupancies of 0.7 and those at C8' and C9' have occupancies of 0.3. The H atoms at C10 have two positions (with 0.7 and 0.3 occupancies) corresponding to the disordered atoms C8 and C9.

Data collection: XSCANS (Siemens, 1994). Cell refinement: XSCANS. Data reduction: XSCANS. 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.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: AS1219). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

- Calligaris, M., Nardin, G. & Randaccio, L. (1972). *Coord. Chem. Rev.* **7**, 385–403.
- Crumbly, A. L. & Bosolo, F. (1970). *J. Am. Chem. Soc.* **92**, 55–65.
- Elerman, Y., Svoboda, I. & Fuess, H. (1991). *Z. Kristallogr.* **196**, 309–311.
- Flack, H. D. (1983). *Acta Cryst.* **A39**, 876–881.
- Floriani, C. & Fachinetti, G. (1974). *J. Chem. Soc. Chem. Commun.* pp. 615–616.
- Pahor, N. B., Calligaris, M., Delise, P., Dodic, G., Nardin, G. & Randaccio, L. (1976). *J. Chem. Soc. Dalton Trans.* pp. 2478–2483.
- Sheldrick, G. M. (1990). *SHELXTL/PC User's 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.
- Shi, X.-H., You, X.-Z., Li, C., Song, B.-L., Li, T.-H. & Huang, X.-Y. (1995). *Acta Cryst.* **C51**, 206–207.
- Siemens (1994). *XSCANS User's Manual*. Version 2.1. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.

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Bis(1,4-dimethylpyridinium) Bis(maleonitriledithiolato)nickelate(II)

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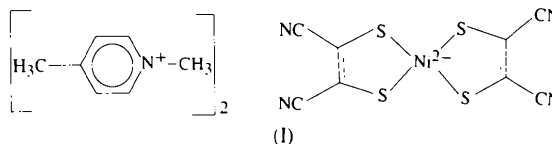
(Received 8 September 1995; accepted 18 October 1995)

Abstract

The [Ni(S₂C₄N₂)₂]²⁻ anion and CH₃C₅H₄NCH₃⁺ cation are individually planar and their planes define a dihedral angle of 126.09 (6)°. Thick layers of anions and cations interconnected by C—H···N hydrogen bonds run parallel to the (110) planes.

Comment

1,2-Dithiolato ligands, R₂C₂S₂²⁻, are capable of forming a large variety of complexes with transition metal ions in various oxidation states. The planar coordination of the metal atom to the S atoms of the ligand permits columnar ionic packing in the solid state, thereby giving rise to interesting magnetic, electrical and optical properties (Manoharan, Noordik, de Boer & Keijzers, 1981; Clemenson, 1990). The title compound, (I), the 1-methyl-4-methylpyridinium salt of the anion [Ni(mnt)₂]²⁻, mnt = maleonitriledithiolate, has been synthesized to investigate its electrical and magnetic properties. The crystal structure analysis was carried out to elucidate its solid-state structural features.



A displacement ellipsoid plot of the complex with the atom-numbering scheme is shown in Fig. 1. The bond lengths and angles observed in both the anion and cation are normal. Distances in the anion agree, within experimental error, with those reported for other [Ni(mnt)₂]²⁻ complexes (Mahadevan, Seshasayee, Radha & Manoharan, 1984). The Ni atom occupies an inversion centre, as

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is usual for $[\text{Ni}(\text{mnt})_2]^{2-}$ complexes, so that the NiS_4 chromophore is strictly planar and nearly square planar, with Ni—S distances of 2.166 (1) and 2.176 (1) Å, and a bite angle of 92.36 (2)°. The mnt ligand is essentially planar and the Ni atom deviates by 0.116 (1) Å from the plane. Parallel pairs of the planar cations are stacked with a perpendicular distance between them of 3.55 (6) Å and they make an angle of 126.09 (6)° with the plane of the anion.

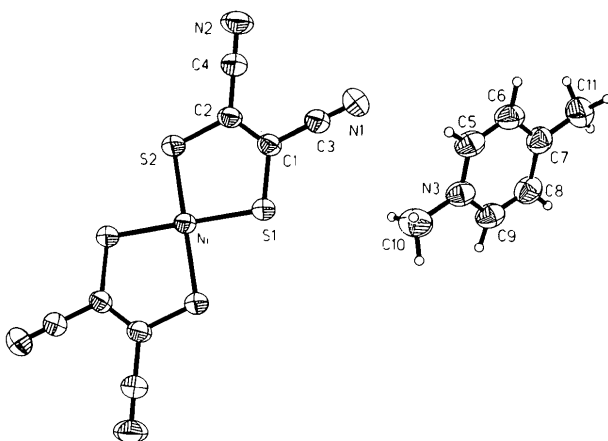


Fig. 1. A 50% probability displacement ellipsoid plot with atom-numbering scheme.

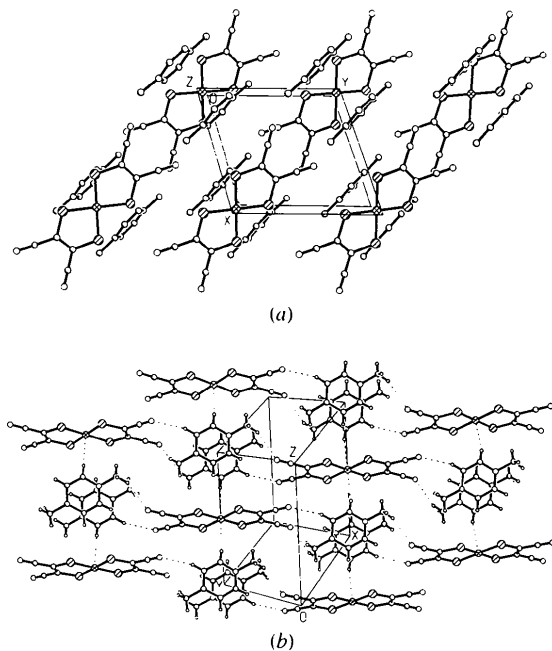


Fig. 2 (a) Packing of the molecules in the crystal, viewed down the *c* axis, showing the formation of thick layers of cations and anions parallel to the (110) planes; H atoms are omitted for clarity. (b) View of a single thick layer perpendicular to its mean plane; C—H...N hydrogen bonds and CH...Ni short contacts are drawn as dashed lines.

Fig. 2(a) shows the packing of thick layers of anions and cations running parallel to the (110) planes. In each layer the ions are knitted together by two C—H...N hydrogen bonds and by a short CH...Ni contact, as shown in Fig. 2(b) [C6...N1ⁱ 3.419 (2) Å and C6—H6...N1ⁱ 163 (2)°, C10...N2ⁱⁱ 3.432 (3) Å and C10—H...N2ⁱⁱ 153 (3)°, C5...Ni^{iii,iv} 3.528 (3) Å; symmetry codes: (i) 1 - *x*, 2 - *y*, 1 - *z*; (ii) 1 + *x*, *y*, *z* - 1; (iii) *x*, 1 + *y*, *z*; (iv) 2 - *x*, 1 - *y*, 1 - *z*]. The packing arrangement in this structure is similar to that of $[\text{TMPD}]_2^+[\text{Ni}(\text{mnt})_2]^{2-}$ (TMPD = tetramethyl-*p*-phenylenediamine (Hove, Hoffman & Ibers, 1972).

Experimental

The title complex was synthesized by adding an alcoholic solution of the pyridinium cation (as the iodide salt) to the anionic dithiolenic complex $[\text{Ni}(\text{mnt})_2]^{2-}$ in solution (as the NBu_4^+ salt). Single crystals were obtained by recrystallization from dimethylformamide.

Crystal data

$(\text{C}_7\text{H}_{10}\text{N})_2[\text{Ni}(\text{C}_4\text{N}_2\text{S}_2)_2]$

$M_r = 555.39$

Triclinic

$P\bar{1}$

$a = 8.534 (1) \text{ \AA}$

$b = 8.765 (1) \text{ \AA}$

$c = 9.975 (1) \text{ \AA}$

$\alpha = 87.56 (1)^\circ$

$\beta = 64.89 (1)^\circ$

$\gamma = 72.26 (1)^\circ$

$V = 640.23 (12) \text{ \AA}^3$

$Z = 1$

$D_x = 1.440 \text{ Mg m}^{-3}$

Mo $K\alpha$ radiation

$\lambda = 0.71073 \text{ \AA}$

Cell parameters from 25

reflections

$\theta = 8\text{--}25^\circ$

$\mu = 1.106 \text{ mm}^{-1}$

$T = 293 (2) \text{ K}$

Prism

$0.58 \times 0.42 \times 0.36 \text{ mm}$

Brown-red

Data collection

Siemens P4 diffractometer

$\theta/2\theta$ scans

Absorption correction:

none

3509 measured reflections

2913 independent reflections

2678 observed reflections

$[I > 2\sigma(I)]$

$R_{\text{int}} = 0.0127$

$\theta_{\text{max}} = 27.5^\circ$

$h = -1 \rightarrow 10$

$k = -11 \rightarrow 11$

$l = -12 \rightarrow 12$

3 standard reflections

monitored every 97

reflections

intensity decay: <3%

Refinement

Refinement on F^2

$R[F^2 > 2\sigma(F^2)] = 0.0327$

$wR(F^2) = 0.0984$

$S = 1.077$

2913 reflections

191 parameters

All H-atom parameters

refined

$w = 1/[\sigma^2(F_o^2) + (0.0677P)^2 + 0.0513P]$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\text{max}} = 0.001$

$\Delta\rho_{\text{max}} = 0.34 \text{ e \AA}^{-3}$

$\Delta\rho_{\text{min}} = -0.60 \text{ e \AA}^{-3}$

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 (Å²)
$$U_{eq} = (1/3) \sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j$$

	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 (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).

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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.

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

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Abstract

In [Mo(C₁₀H₈NO)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···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.

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