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trans-Bis(*O*,*O*′-diethyl dithiophosphato-*S*,*S*′)bis(4-methylpyridine)nickel(II)

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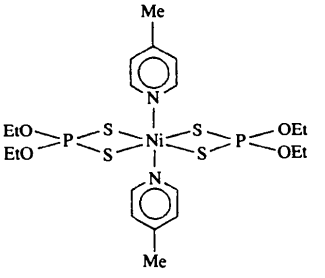
Abstract

The Ni atom in *trans*-bis(*O*,*O*′-diethyl dithiophosphato-*S*,*S*′)bis(4-methylpyridine)nickel(II), [Ni{$(C_2H_5O)_2PS_2$}$_2$(C_6H_7N)$_2$], has slightly distorted octahedral coordination. It lies in the plane formed by the four S atoms of the two chelating diethyl dithiophosphates; the two 4-methylpyridine ligands occupy axial sites. The Ni—S bond lengths are 2.488 (1) and 2.498 (1) Å and the Ni—N2 bond lengths are 114 (4) Å.

Comment

Dialkyl dithiophosphate complexes of transition metals have received increasing attention in recent years owing to their extensive applications in lubrication engineering and in the plastics industry (So, Lin, Huang Gibbs & Chang Terny, 1993; Mikhailov, Kokhanov, Kazaryan, Matreeva & Kozodoi, 1970). In addition to their syntheses and various physicochemical investigations, the crystal structures of many of these complexes and their adducts with nitrogen bases have been reported (McConnell & Kastalsky, 1967; Ooi & Fernando, 1970; Huang, Xiong, Dong & You, 1995). In a continuation of our investigation of the reaction of (diethyl dithiophosphate)nickel(II) with neutral nitrogen bases we determined the crystal structure of *trans*-[Ni{$(C_2H_5O)_2PS_2$}$_2$(C_6H_7N)$_2$], (I).



(I)

The Ni atom in (I) is coordinated to four S atoms and two *trans* N atoms. The dihedral angle between the plane of Ni, C(1), C(2), C(3), C(4), C(5), N and C(6) and that of Ni, S(1), S(2) and P is 88.66 (7)°. In agreement with the data for [Ni{$(C_2H_5O)_2PS_2$}$_2$(C_5H_5N)$_2$] (Ooi & Fernando, 1970), the Ni—S bond distances are 2.488 (1) and 2.498 (1) Å, the Ni—N bond distances are 2.114 (4) Å, and the S(1)—Ni—S(2) and S(1)—Ni—N bond angles are 81.30 (5) and 90.5 (1)°, respectively.

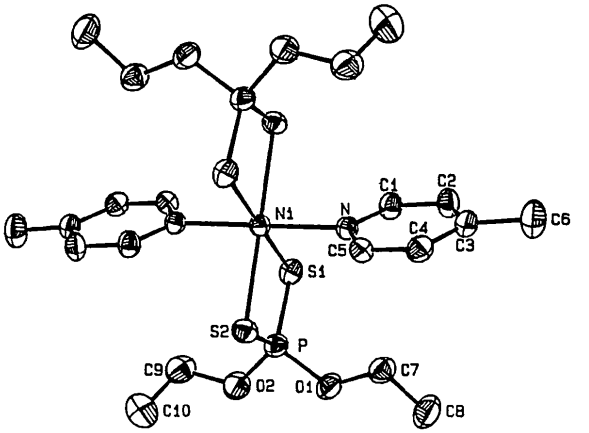


Fig. 1. Molecular structure showing 30% probability displacement ellipsoids. H atoms are omitted for clarity.

Experimental

Bis(*O*,*O*′-diethyl dithiophosphato)nickel(II) was dissolved in ethanol and 4-methylpyridine in $CHCl_3$ solution was added

dropwise until the colour changed from purple to green. Crystals were obtained by evaporation at room temperature.

Crystal data

$[Ni(C_4H_{10}O_2PS_2)_2(C_6H_7N)_2]$
$M_r = 615.39$
Triclinic
$P\bar{1}$
$a = 8.459\ (2)$ Å
$b = 9.132\ (3)$ Å
$c = 9.903\ (2)$ Å
$\alpha = 95.22\ (2)°$
$\beta = 84.14\ (2)°$
$\gamma = 104.72\ (2)°$
$V = 734.3\ (3)$ Å^3
$Z = 1$
$D_x = 1.39$ Mg m^{-3}
Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 8.74–14.56°$
$\mu = 1.070$ mm^{-1}
$T = 296$ K
Column
0.25 × 0.15 × 0.15 mm
Green

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: ψ scans (*TEXSAN*; Molecular Structure Corporation, 1985)
$T_{min} = 0.9372$, $T_{max} = 1.00$
2768 measured reflections
2575 independent reflections
2079 observed reflections $[I > 3\sigma(I)]$
$R_{int} = 0.0086$
$\theta_{max} = 25°$
$h = 0 \rightarrow 10$
$k = -10 \rightarrow 10$
$l = -11 \rightarrow 11$
3 standard reflections monitored every 300 reflections
intensity decay: 3.8%

Refinement

Refinement on F
$R = 0.052$
$wR = 0.065$
$S = 1.56$
2079 reflections
152 parameters
H-atom parameters not refined
$w = 1/\sigma^2(F)$
$(\Delta/\sigma)_{max} = 0.01$
$\Delta\rho_{max} = 0.54$ e Å^{-3}
$\Delta\rho_{min} = -0.44$ e Å^{-3}
Extinction correction: none
Atomic scattering factors from Cromer & Waber (1974)

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

$B_{eq} = (8\pi^2/3)\Sigma_i\Sigma_j U_{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j$.

	x	y	z	B_{eq}
Ni	1	0	0	3.33 (3)
S(1)	1.0003 (2)	0.1856 (1)	0.2015 (1)	4.44 (5)
S(2)	0.7122 (1)	−0.0972 (1)	0.0952 (1)	4.30 (5)
P	0.7674 (2)	0.0739 (2)	0.2337 (1)	4.40 (5)
O(1)	0.6471 (4)	0.1823 (4)	0.2447 (3)	5.6 (2)
O(2)	0.7204 (5)	0.0227 (4)	0.3845 (3)	6.1 (2)
N	0.9219 (4)	0.1442 (4)	−0.1200 (3)	3.6 (1)
C(1)	1.0020 (6)	0.2883 (5)	−0.1324 (5)	4.4 (2)
C(2)	0.9608 (6)	0.3814 (5)	−0.2172 (5)	5.0 (2)
C(3)	0.8293 (6)	0.3253 (5)	−0.2973 (5)	4.6 (2)
C(4)	0.7449 (6)	0.1770 (6)	−0.2814 (5)	4.5 (2)
C(5)	0.7923 (5)	0.0906 (5)	−0.1947 (4)	4.0 (2)
C(6)	0.7849 (8)	0.4208 (7)	−0.3959 (7)	7.0 (3)
C(7)	0.6371 (7)	0.2648 (7)	0.1305 (6)	5.6 (2)
C(8)	0.5368 (9)	0.3755 (8)	0.1745 (7)	7.8 (4)
C(9)	0.7948 (8)	−0.0871 (8)	0.4318 (6)	6.8 (3)
C(10)	0.671 (1)	−0.219 (1)	0.471 (1)	10.2 (5)

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

Ni—N	2.114 (4)	Ni—N^i	2.114 (4)
Ni—S(1)	2.498 (1)	Ni—S(1^i)	2.498 (1)
Ni—S(2)	2.488 (1)	Ni—S(2^i)	2.488 (1)
P—S(1)	1.983 (2)	P—O(2)	1.587 (4)
O(1)—C(7)	1.436 (6)	O(2)—C(9)	1.444 (7)
N—Ni—N^i	180.00	S(1)—Ni—S(2)	81.30 (5)
N—Ni—S(1)	90.5 (1)	N^i—Ni—S(2)	90.1 (1)
N—Ni—S(2)	89.9 (1)	S(1)—Ni—N^i	89.5 (1)
S(1)—P—S(2)	110.20 (8)	O(1)—P—O(2)	94.7 (2)
O(1)—P—S(1)	112.2 (2)	O(2)—P—S(2)	112.6 (2)

Symmetry code: (i) $2 - x, -y, -z$.

Data were collected using *CONTROL* software (Molecular Structure Corporation, 1988). The structure was solved by direct methods using *MITHRIL* (Gilmore, 1983); the heavy atom Ni was located in an E map and the remaining non-H atoms were located using *DIRDIF* (Beurskens, 1984). H atoms were placed in geometrically calculated positions (C—H = 0.95 Å) and were not included in the refinement. The structure was refined by full-matrix least-squares techniques with anisotropic displacement parameters for all atoms. Calculations were performed on a VAX3100 computer using the *TEXSAN* (Molecular Structure Corporation, 1985) program package.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, complete geometry and least-squares-planes data have been deposited with the IUCr (Reference: MU1176). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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