Absorption correction: refined from ΔF (*DIFABS*; Walker & Stuart, 1983)	$\theta_{max} = 25°$
$T_{min} = 0.960$, $T_{max} = 0.996$	$h = 0 \rightarrow 11$
2890 measured reflections	$k = -20 \rightarrow 0$
2741 independent reflections	$l = -21 \rightarrow 21$
	3 standard reflections monitored every 300 reflections
	intensity decay: 1.9%

Refinement

Refinement on F	$w = 1/\sigma^2(F)$
$R = 0.064$	$(\Delta/\sigma)_{max} = 0.11$
$wR = 0.074$	$\Delta\rho_{max} = 0.67$ e Å^{-3}
$S = 1.71$	$\Delta\rho_{min} = -0.49$ e Å^{-3}
1920 reflections	Extinction correction: none
195 parameters	Atomic scattering factors from Cromer & Waber (1974)
H-atom parameters not refined	

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

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

	x	y	z	B_{eq}
Ni	1/2	0.23077 (5)	1/4	2.73 (4)
S	0.1432 (2)	0.1871 (1)	0.0482 (1)	5.7 (1)
F(1)	0.2787 (6)	0.4204 (2)	0.3860 (3)	8.4 (3)
F(2)	0.3840 (6)	0.4777 (2)	0.3080 (3)	10.6 (3)
F(3)	0.1609 (6)	0.4640 (3)	0.2943 (3)	10.8 (3)
O(1)	0.3287 (4)	0.2291 (2)	0.1740 (2)	3.4 (1)
O(2)	0.4057 (4)	0.3172 (2)	0.3046 (2)	3.5 (2)
N	0.5988 (4)	0.1501 (2)	0.1863 (2)	3.2 (2)
C(1)	0.2311 (5)	0.2775 (3)	0.1654 (3)	3.2 (2)
C(2)	0.2167 (6)	0.3417 (3)	0.2102 (3)	3.5 (2)
C(3)	0.3047 (6)	0.3557 (3)	0.2739 (3)	3.1 (2)
C(4)	0.2824 (7)	0.4293 (3)	0.3149 (4)	4.4 (3)
C(5)	0.1271 (6)	0.2648 (3)	0.1011 (3)	3.6 (2)
C(6)	0.0043 (9)	0.2123 (5)	−0.0107 (4)	5.9 (4)
C(7)	−0.0589 (8)	0.2750 (5)	0.0062 (4)	6.5 (4)
C(8)	0.0007 (6)	0.3120 (3)	0.0737 (3)	3.5 (2)
C(9)	0.7386 (6)	0.1516 (3)	0.1796 (3)	4.2 (3)
C(10)	0.8070 (7)	0.1001 (4)	0.1379 (4)	4.9 (3)
C(11)	0.7297 (7)	0.0425 (3)	0.1005 (3)	4.4 (3)
C(12)	0.5833 (7)	0.0420 (3)	0.1065 (5)	4.3 (3)
C(13)	0.5230 (6)	0.0951 (3)	0.1492 (3)	3.8 (3)
C(14)	0.797 (1)	−0.0154 (4)	0.0553 (4)	7.1 (4)

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

Ni—O(1)	2.022 (3)	O(1)—C(1)	1.251 (6)
Ni—O(2)	2.058 (4)	O(2)—C(3)	1.253 (6)
Ni—N	2.097 (4)		
O(1)—Ni—O(2)	89.3 (1)	O(1)—Ni—O(1^i)	178.3 (2)
O(1^i)—Ni—O(2)	91.9 (1)	O(2)—Ni—N	174.5 (2)
N^i—Ni—O(1^i)	88.8 (1)	O(2)—Ni—O(2^i)	84.6 (2)
N—Ni—N^i	94.7 (2)	O(2)—Ni—N^i	90.4 (2)
N^i—Ni—O(1)	90.1 (2)		

Symmetry code: (i) $1 - x, y, \frac{1}{2} - z$.

Data were collected using *CONTROL* software (Molecular Structure Corporation, 1988). H atoms were placed in calculated positions (C—H = 0.95 Å) and their parameters refined. The structure was refined by full-matrix least-squares techniques with anisotropic displacement parameters for all non-H atoms. All calculations were performed on a VAX3100 computer using the *TEXSAN* (Molecular Structure Corporation, 1985) program package.

This work was supported by a grant for a Major Project from the State Science and Technology Commission, and the National Science Foundation of China, as well as the State Key Laboratory of Tribology of Tsinghua University.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, least-squares-planes data and complete geometry have been deposited with the IUCr (Reference: MU1173). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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The Adduct of Bis(*O*,*O*′-diethyl dithiophosphato)nickel(II) with 3-Aminopyridine

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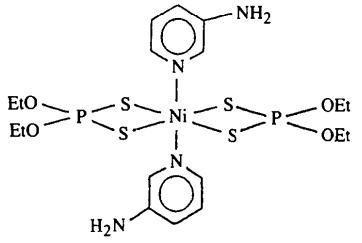
Abstract

The coordination compound *trans*-bis(3-aminopyridine)-bis(*O*,*O*′-diethyl dithiophosphato-*S*,*S*′)nickel(II), [Ni{$(C_2H_5O)_2PS_2$}$_2$($C_5H_6N_2$)$_2$], displays distorted octahedral geometry around the central Ni atom. In the

complex, two *O,O'*-diethyl dithiophosphate ions act as bidentate ligands with their S atoms coordinated to Ni. Each forms a four-membered chelate ring in the equatorial plane. The pyridine N atoms of the other two ligands are axially coordinated to the Ni atom. The Ni—S bond distances are 2.491 (2) and 2.505 (3) Å and the Ni—N bond distances are 2.103 (3) Å.

Comment

Recently, adducts of bis(*O,O'*-dialkyl dithiophosphato)-nickel(II) with neutral nitrogen ligands have attracted attention (Ooi & Fernando, 1967; Liu *et al.*, 1991; Huang, Xiong, Dong & You, 1995; You, Xiong, Dong & Huang, 1994). As part of our work on the reactions of bis(*O,O'*-dialkyl dithiophosphato)nickel(II) with neutral nitrogen bases, we report here the crystal structure of the title complex, (I) (Nidtp-3Apy; dtp = *O,O'*-diethyl dithiophosphate), which exhibits a slightly distorted *trans*-octahedral configuration.



(I)

In the adduct (I), $—NH_2$ does not take part in the coordination of the Ni atom; the same observation was made for Nidtp-4Apy by You *et al.* (1994). The Ni—S and Ni—N bond lengths do not change significantly compared with those of Nidtp.4Apy (You *et al.*, 1994). Similarly, no apparent differences are observed in the S(1)—Ni—S(2) and S(1)—Ni—N bond angles in the two structures, despite the NH_2 groups being in different positions. In addition, the dihedral angle between the least-squares plane of Ni, S(1), S(2) and P and that of N(1), C(1), C(2), C(3), C(4), C(5) and N(2) is 85.7 (1)°, *i.e.* the planes are almost perpendicular.

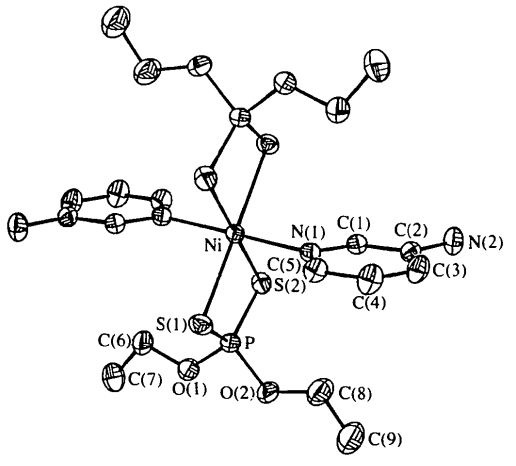


Fig. 1. Molecular structure showing 30% probability displacement ellipsoids. H atoms have been omitted for clarity.

Experimental

$Ni(dtp)_2$ was dissolved in ethanol and 3-aminopyridine in $CHCl_3$ solution was added dropwise until the colour changed from purple to green. The solution evaporated at room temperature to give a green prismatic crystal.

Crystal data

$[Ni(C_4H_{10}O_2PS_2)_2(C_5H_6N_2)_2]$
$M_r = 617.36$
Monoclinic
$P2_1/c$
$a = 8.142\ (9)$ Å
$b = 17.617\ (8)$ Å
$c = 10.479\ (4)$ Å
$\beta = 107.23\ (7)°$
$V = 1436\ (3)$ Å^3
$Z = 2$
$D_x = 1.43$ Mg m^{-3}
Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 13.95–15.32°$
$\mu = 1.096$ mm^{-1}
$T = 296$ K
Prism
0.55 × 0.35 × 0.22 mm
Green

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: ψ scans (Molecular Structure Corporation, 1985)
$T_{min} = 0.5880$, $T_{max} = 1.000$
2769 measured reflections
2624 independent reflections
2130 observed reflections $[I > 3\sigma(I)]$
$R_{int} = 0.0058$
$\theta_{max} = 25°$
$h = -9 \rightarrow 9$
$k = 0 \rightarrow 20$
$l = 0 \rightarrow 12$
3 standard reflections monitored every 300 reflections
intensity decay: 0.5%

Refinement

Refinement on F
$R = 0.040$
$wR = 0.051$
$S = 1.42$
2130 reflections
152 parameters
H-atom parameters not refined
$w = 1/\sigma^2(F)$
$(\Delta/\sigma)_{max} = 0.02$
$\Delta\rho_{max} = 0.34$ e Å^{-3}
$\Delta\rho_{min} = -0.32$ e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV, Tables 2.2A and 2.31)

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	1	2.90 (2)
S(1)	0.9305 (1)	−0.08006 (5)	0.79500 (9)	4.28 (4)
S(2)	0.7319 (1)	−0.06119 (4)	1.0218 (1)	4.03 (4)

P	0.7327 (1)	−0.11240 (4)	0.8548 (1)	3.99 (4)
O(1)	0.7209 (3)	−0.2022 (1)	0.8647 (3)	5.2 (1)
O(2)	0.5536 (3)	−0.1054 (1)	0.7429 (3)	5.7 (1)
N(1)	0.8518 (3)	0.0886 (1)	0.8885 (2)	3.3 (1)
N(2)	0.5255 (4)	0.2240 (2)	0.9188 (3)	4.9 (1)
C(1)	0.7336 (4)	0.1254 (2)	0.9302 (3)	3.2 (1)
C(2)	0.6419 (4)	0.1877 (2)	0.8667 (3)	3.6 (1)
C(3)	0.6773 (5)	0.2130 (2)	0.7521 (4)	4.8 (2)
C(4)	0.7946 (5)	0.1750 (2)	0.7068 (4)	5.1 (2)
C(5)	0.8806 (4)	0.1135 (2)	0.7756 (3)	4.2 (1)
C(6)	0.8567 (5)	−0.2455 (2)	0.9560 (5)	6.0 (2)
C(7)	0.8140 (6)	−0.3272 (2)	0.9377 (5)	6.6 (2)
C(8)	0.4778 (6)	−0.0334 (3)	0.6971 (5)	7.4 (2)
C(9)	0.3026 (7)	−0.0426 (3)	0.6156 (6)	9.1 (3)

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

Ni—N(1)	2.103 (3)	Ni—N(1^{i})	2.103 (3)
Ni—S(1)	2.491 (2)	Ni—S(1^{i})	2.491 (2)
Ni—S(2)	2.505 (3)	Ni—S(2^{i})	2.505 (3)
P—S(1)	1.977 (2)	P—O(2)	1.582 (3)
O(1)—C(6)	1.446 (5)	O(2)—C(8)	1.430 (5)
N(1)—Ni—N(1^{i})	180.00	S(1)—Ni—S(2)	81.58 (7)
N(1)—Ni—S(1)	89.71 (9)	N(1^{i})—Ni—S(2)	90.1 (1)
N(1)—Ni—S(2)	89.9 (1)	S(1)—Ni—N(1^{i})	90.29 (9)
S(1)—P—S(2)	111.52 (8)	O(1)—P—O(2)	93.8 (2)
O(1)—P—S(1)	112.2 (1)	O(2)—P—S(2)	112.2 (1)

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

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 non-H atoms. Calculations were performed on a VAX3100 computer using the *TEXSAN* (Molecular Structure Corporation, 1985) program package.

This work was supported by a grant for a Major Project from the State Science and Technology Commission, and the National Science Foundation of China, as well as the State Key Laboratory of Tribology of Tsinghua University.

Lists of structure factors, torsion angles, least-squares-planes data, anisotropic displacement parameters, H-atom coordinates and bond distances and angles involving H atoms have been deposited with the IUCr (Reference: AB1261). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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The Adduct of Bis(*O*,*O*′-dibutyl dithiophosphato)nickel(II) with Isoquinoline

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Abstract

In the title compound, $[Ni\{(C_4H_9O)_2PS_2\}_2(C_9H_7N)_2]$, the metal atom is coordinated in a distorted octahedral arrangement with the central Ni atom lying in the plane formed by the four S atoms and with the two isoquinoline moieties in *trans* positions. The Ni—S bond distances are 2.491 (3) and 2.498 (2) Å and the Ni—N distances are 2.103 (4) Å.

Comment

In recent years, adducts of nickel(II) dialkyl dithiophosphate with neutral nitrogen bases have received increasing attention, partly because of the reactivity of a variety of nitrogen bases with nickel(II) dialkyl dithiophosphate in solution (Liu *et al.*, 1991; You, Xiong, Dong & Huang, 1994; You *et al.*, 1991). Furthermore, the amines in lubricating oil have a great influence on the properties of metal dialkyl dithiophosphate additives (Shiomi, Tokashiki, Tomizawa