Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

(2,2′-Bipyridyl-κ^2N,N')bis(*O*,*O*′-diisopropyl dithiophosphato-κ^2S,S')nickel(II)

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Key indicators

Single-crystal X-ray study
T = 120 K
Mean σ(C–C) = 0.005 Å
R factor = 0.043
wR factor = 0.133
Data-to-parameter ratio = 22.1

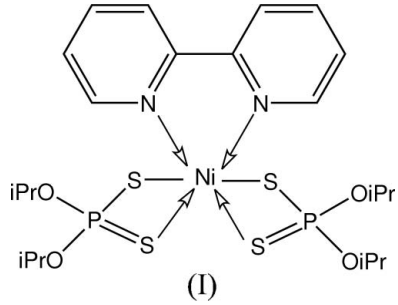
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 15 September 2006
Accepted 18 September 2006

The monomeric title compound, $[Ni(C_6H_{14}O_2PS_2)_2(C_{10}H_8N_2)]$, has the Ni atom within a distorted octahedral *cis*-N_2S_4 geometry. The crystal structure is stabilized by C—H···S interactions, leading to the formation of a linear chain.

Comment

In continuation of our interest in the structural chemistry of bipyridine adducts of nickel(II) dithiophosphates, with general formula $Ni[S_2P(OR)_2]_2$(2,2′-bipyridine) (Berdugo & Tiekink, 2006), the title complex, where *R* = ^{i}Pr, (I), was investigated. The distorted octahedral geometry in (I) (Fig. 1) is based on a *cis*-N_2S_4 donor set and is in agreement with those found in related structures, namely *R* = Me (Arora *et al.*, 1977), *R* = ^{n}Bu [You *et al.*, 1986; see Hu (1999) for space group revision] and *R* = ^{i}Bu (Berdugo & Tiekink, 2006). The Ni—S distances (Table 1) lie in the relatively narrow range 2.4548 (9) (Ni—S1) to 2.4964 (9) Å (Ni—S4) and the P—S distances follow the expected trends in that the shorter bond is always associated with the less tightly bound S atom. Distortions from the ideal octahedral geometry may be attributed to the acute chelate angles. The 2,2′-bipyridine molecule features a small twist about the central C—C bond (Table 1).



(I)

The most prominent intermolecular contact in the structure is of the type $C_{aromatic}$—H···S [H16···S3^i = 2.70 Å, C16···S3^i = 3.514 (4) Å and C16—H16···S3^i = 144°; symmetry code: (i) 1 + *x*, *y*, *z*]. These interactions lead to the formation of a linear chain as illustrated in Fig. 2. There are intramolecular C—H···π interactions of note involving the methine C1/H1 and C10/H10 atoms with the ring centroids of the N1- and N2-pyridine rings, respectively, with distances and angles of 2.75 Å and 109°, and 2.78 Å and 108°, respectively. In the recently determined structure of the *R* = ^{i}Bu analogue (Berdugo & Tiekink, 2006), related C—H···π contacts were present, but owing to the greater reach of the isobutyl ligand, these interactions were intermolecular and served to stabilize the chain mediated by C—H···S interactions.

Experimental

The title compound was prepared by refluxing the parent nickel dithiophosphate with 2,2′-bipyridine (Acros Organics) following a literature procedure (Lai *et al.*, 2004). Green crystals were isolated by the slow evaporation of a $CHCl_3$ solution of the compound; m.p. 463 K (decompostion). IR (KBr disk): ν(C—O) 1174, ν(P—O) 954, $\nu(P-S)_{asymm}$ 657, $\nu(P-S)_{sym}$ 535 cm^{-1}.

Crystal data

$[Ni(C_6H_{14}O_2PS_2)_2(C_{10}H_8N_2)]$
M_r = 641.42
Monoclinic, $P2_1/n$
a = 9.1585 (3) Å
b = 30.6703 (12) Å
c = 11.6407 (4) Å
β = 110.808 (1)°
V = 3056.53 (19) Å^3
Z = 4
D_x = 1.394 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 1.04 mm^{-1}
T = 120 (2) K
Rod, green
0.48 × 0.06 × 0.03 mm

Data collection

Bruker–Nonius KappaCCD diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)
T_{min} = 0.793, T_{max} = 1
29206 measured reflections
6997 independent reflections
5541 reflections with $I > 2\sigma(I)$
R_{int} = 0.072
θ_{max} = 27.5°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.043
$wR(F^2)$ = 0.133
S = 1.05
6997 reflections
316 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0675P)^2 + 3.279P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.002
$\Delta\rho_{max}$ = 0.55 e Å^{-3}
$\Delta\rho_{min}$ = −0.49 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Ni—S1	2.4548 (9)	Ni—N2	2.088 (3)
Ni—S2	2.4840 (8)	S1—P1	1.9907 (11)
Ni—S3	2.4839 (9)	S2—P1	1.9929 (11)
Ni—S4	2.4964 (9)	S3—P2	1.9790 (11)
Ni—N1	2.071 (2)	S4—P2	1.9890 (11)
S1—Ni—S2	81.50 (3)	S3—Ni—S4	81.48 (3)
S1—Ni—S4	174.11 (3)	S3—Ni—N2	165.22 (8)
S2—Ni—N1	167.00 (8)	N1—Ni—N2	78.82 (10)
N1—C17—C18—N2	−6.2 (4)		

H atoms were included in the riding-model approximation with C—H distances = 0.95–1.00 Å, and with U_{iso}(H) = 1.5U_{eq}(methyl C) and U_{iso}(H) = 1.2U_{eq}(remaining C).

Data collection: *COLLECT* (Hooft, 1998); cell refinement: *DENZO* (Otwinowski & Minor, 1997) and *COLLECT*; data reduction: *DENZO* and *COLLECT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPII* (Johnson, 1976) and *DIAMOND* (Brandenburg, 2006); software used to prepare material for publication: *SHELXL97*.

This work was supported by the departmental research grant AX-0026 from The Robert A. Welch Foundation.

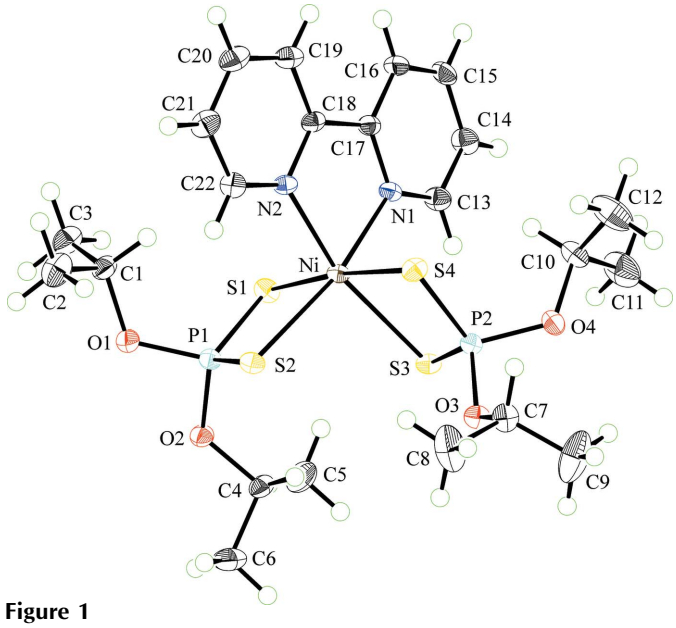


Figure 1
Molecular structure and crystallographic numbering scheme for (I). Displacement ellipsoids are shown at the 50% probability level.

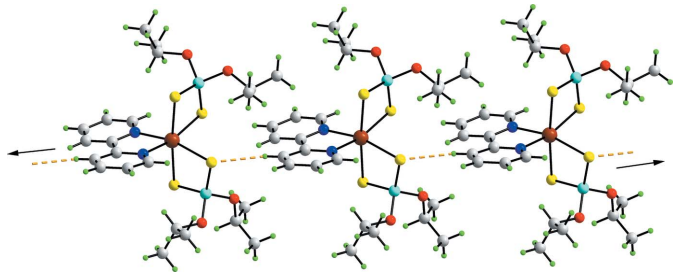


Figure 2
The chain in (I), running parallel to *a*, mediated by C—H···S interactions, shown as dashed orange lines. Colour code: Ni (brown), S (yellow), P (light blue), O (red), N (blue), C (grey) and H (green).

Cheminova is thanked for the gift of the dithiophosphate ligand used in this study. The authors also thank the EPSRC X-ray Crystallographic Service, University of Southampton, England, for the data collection.

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