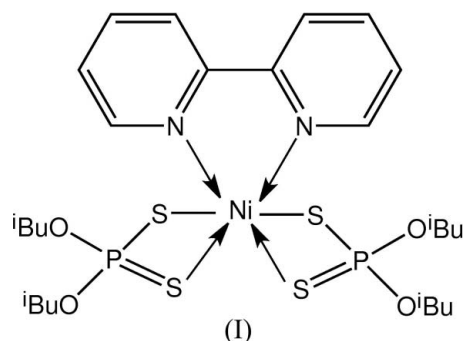


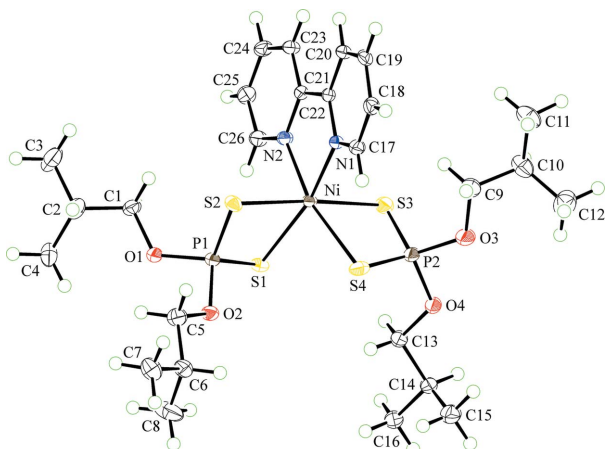
(2,2'-Bipyridyl)bis(O,O'-diisobutyl  
dithiophosphato- $\kappa^2S,S'$ )nickel(II)Erick Berdugo and Edward R. T.  
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## Key indicators

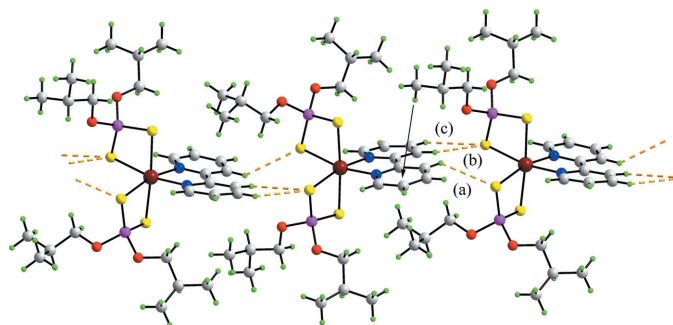
Single-crystal X-ray study  
 $T = 103\text{ K}$   
Mean  $\sigma(\text{C}-\text{C}) = 0.004\text{ \AA}$   
 $R$  factor = 0.045  
 $wR$  factor = 0.101  
Data-to-parameter ratio = 19.5For details of how these key indicators were  
automatically derived from the article, see  
<http://journals.iucr.org/e>.In the monomeric title compound,  $[\text{Ni}(\text{C}_8\text{H}_{18}\text{O}_2\text{PS}_2)_2(\text{C}_{10}\text{H}_8\text{N}_2)]$ , the Ni atom exhibits distorted octahedral  $\text{N}_2\text{S}_4$  coordination. The crystal structure contains intermolecular  $\text{C}-\text{H}\cdots\pi$  and  $\text{C}-\text{H}\cdots\text{S}$  interactions.Received 29 July 2006  
Accepted 12 August 2006

## Comment

Nickel(II) dithiophosphates of the general formula  $\text{Ni}(\text{S}_2\text{P}(\text{OR})_2)_2$ , where  $R$  is an organic substituent group, are well known to form stable adducts with pyridine-type ligands, with over 20 such structures currently present in the Cambridge Structural Database (Version 5.27, with January 2006 and May 2006 updates; Allen, 2002). Two of these structures include 2,2'-bipyridine, with  $R = \text{Me}$  (Arora *et al.*, 1977) and  $R = \text{}^t\text{Bu}$  [You *et al.*, 1986; see Hu (1999) for space-group revision]. The title compound, (I), with  $R = \text{}^i\text{Bu}$  (Fig. 1), is the third such structure. The study of (I) was motivated by analogous structural investigations of the Zn and Cd systems (Lai *et al.*, 2004; Lai & Tiekink, 2004; Chen *et al.*, 2006).The Ni atom in (I) lies within an  $\text{N}_2\text{S}_4$  donor set defined by the three chelating ligands. The coordination geometry is distorted octahedral (Table 1), with the greatest distortions from regular geometry being ascribed to the acute chelate angles. The dithiophosphate ligands chelate the Ni centre with essentially equivalent Ni–S bond distances in the case of the S1/S2 ligand, but with slight asymmetry in the case of the S3/S4 ligand, which exhibits the shortest and longest of the four Ni–S distances (Table 1).The most notable intermolecular interactions in the crystal structure of (I) are of the types  $\text{C}-\text{H}\cdots\pi$  and  $\text{C}-\text{H}\cdots\text{S}$ . The distance between the C16–H16B atoms and the ring centroid of (N1/C17–C21)<sup>i</sup> is 2.74 Å [symmetry code:  $\frac{1}{2} - x, -\frac{1}{2} + y, \frac{1}{2} - z$ ], with an angle at H16B of 162°. Referring to the labels (a), (b) and (c) in Fig. 2, the H23 $\cdots$ S4<sup>i</sup>, H19 $\cdots$ S1<sup>i</sup> and H20 $\cdots$ S1<sup>i</sup> distances are 3.05, 3.01 and 3.00 Å, respectively. The above-mentioned interactions can be considered to link the



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



**Figure 2**  
The chain in the structure of (I), running parallel to *b*, mediated by C—H... $\pi$  (arrow) and C—H...S interactions (dashed lines). Colour code: Ni atoms are coloured brown, S atoms yellow, P atoms pink, O atoms red, N atoms blue, C atoms grey and H atoms green.

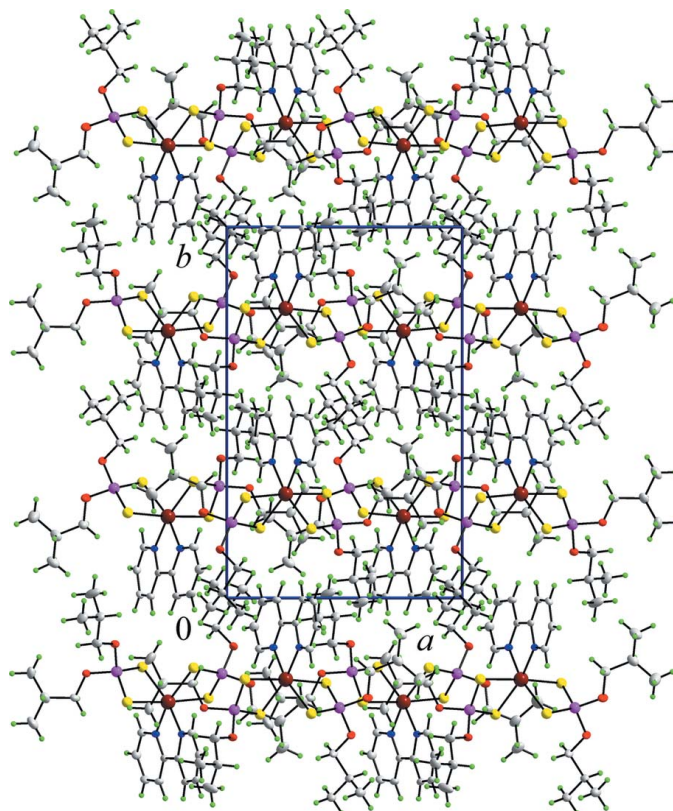
molecules into chains running parallel to the *b* axis (Fig. 2). Further C—H...S interactions exist between these chains, so that the methylene atom H9A and methyl atom H12B of one <sup>i</sup>Bu group approach atoms S1 and S3 of one molecule in a neighbouring chain, with H9A...S1<sup>ii</sup> = 2.94 Å and H12B...S3<sup>ii</sup> = 2.95 Å [symmetry code: (ii)  $\frac{1}{2} + x, \frac{1}{2} - y, \frac{1}{2} + z$ ]. A view of the overall crystal packing is shown in Fig. 3.

## Experimental

The title compound was prepared by refluxing the parent nickel dithiophosphate with 2,2'-bipyridine (Acros Organics), following the literature procedure of Lai *et al.* (2004). Green crystals of (I) were isolated by slow evaporation of a CHCl<sub>3</sub> solution of the compound [m.p. 421 K (decomposition)]. IR (KBr disk, cm<sup>-1</sup>):  $\nu(\text{C—O})$  1165,  $\nu(\text{P—O})$  947,  $\nu(\text{P—S})_{\text{asym}}$  673,  $\nu(\text{P—S})_{\text{sym}}$  560.

### Crystal data

[Ni(C <sub>8</sub> H <sub>18</sub> O <sub>2</sub> PS <sub>2</sub> ) <sub>2</sub> (C <sub>10</sub> H <sub>8</sub> N <sub>2</sub> )]	<i>Z</i> = 4
<i>M<sub>r</sub></i> = 697.52	<i>D<sub>x</sub></i> = 1.391 Mg m <sup>-3</sup>
Monoclinic, <i>P</i> 2 <sub>1</sub> / <i>n</i>	Mo <i>K</i> $\alpha$ radiation
<i>a</i> = 12.102 (2) Å	$\mu$ = 0.96 mm <sup>-1</sup>
<i>b</i> = 18.526 (3) Å	<i>T</i> = 103 (2) K
<i>c</i> = 15.307 (3) Å	Plate, green
$\beta$ = 103.954 (5)°	0.20 × 0.14 × 0.05 mm
<i>V</i> = 3330.4 (10) Å <sup>3</sup>	



**Figure 3**  
A view of the structure of (I), along *c*. Colour code as for Fig. 2.

### Data collection

Rigaku AFC-12K/Saturn224 CCD	61525 measured reflections
area-detector diffractometer	6854 independent reflections
$\omega$ scans	6441 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan	<i>R</i> <sub>int</sub> = 0.042
( <i>ABSCOR</i> ; Higashi, 1995)	$\theta_{\text{max}}$ = 26.5°
<i>T</i> <sub>min</sub> = 0.831, <i>T</i> <sub>max</sub> = 1.000	

### Refinement

Refinement on <i>F</i> <sup>2</sup>	$w = 1/[\sigma^2(F_o^2) + (0.038P)^2 + 5.4602P]$
$R[F^2 > 2\sigma(F^2)] = 0.045$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.101$	$(\Delta\sigma)_{\text{max}} = 0.001$
<i>S</i> = 1.09	$\Delta\rho_{\text{max}} = 1.19 \text{ e } \text{Å}^{-3}$
6854 reflections	$\Delta\rho_{\text{min}} = -0.79 \text{ e } \text{Å}^{-3}$
352 parameters	
H-atom parameters constrained	

**Table 1**

Selected geometric parameters (Å, °).

Ni—S1	2.4888 (7)	Ni—S4	2.5148 (7)
Ni—S2	2.4784 (8)	Ni—N1	2.067 (2)
Ni—S3	2.4715 (8)	Ni—N2	2.080 (2)
S1—Ni—S2	81.92 (2)	S2—Ni—N1	87.78 (6)
S1—Ni—S3	94.52 (3)	S2—Ni—N2	94.51 (6)
S1—Ni—S4	92.64 (3)	S3—Ni—S4	80.90 (3)
S1—Ni—N1	168.19 (6)	S3—Ni—N1	96.00 (6)
S1—Ni—N2	96.02 (6)	S3—Ni—N2	88.31 (6)
S2—Ni—S3	175.67 (2)	S4—Ni—N1	94.30 (6)
S2—Ni—S4	96.74 (3)	S4—Ni—N2	166.66 (6)

H atoms bound to C atoms were included in the riding-model approximation, with C–H distances in the range 0.95–1.00 Å, and with  $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{methyl C})$ , or  $1.2U_{\text{eq}}(\text{C})$  for all other H atoms. The maximum residual electron-density peak of  $1.19 \text{ e } \text{Å}^{-3}$  is located between the Ni and P2 atoms.

Data collection: *CrystalClear* (Rigaku, 2005); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; program(s) used to solve structure: *PATY* in *DIRDIF92* (Beurskens *et al.*, 1992); 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*.

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