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Redetermination of bis(*O*,*O*′-dicyclohexyl phosphorodithioato)nickel(II)

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Key indicators

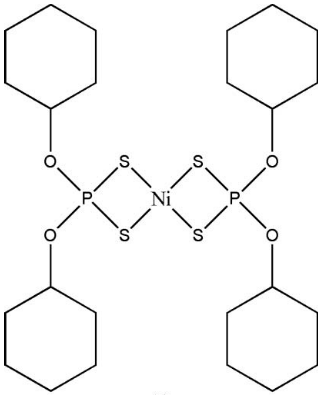
Single-crystal X-ray study
T = 296 K
Mean σ(C–C) = 0.005 Å
R factor = 0.032
wR factor = 0.084
Data-to-parameter ratio = 23.2

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

The structure of the title compound, $[Ni(C_{12}H_{22}O_2PS_2)_2]$, has been determined previously [Chichang *et al.* (1987). *Chin. J. Struct. Chem.* **6**, 186], but the current determination provides a significant increase in the precision of the geometric parameters. The Ni^{II} atom lies on a crystallographic center of symmetry in a distorted square-planar coodination environment.

Comment

Organodithio derivatives of phosphorus have been widely studied for many years (Larson, 2004; Gray *et al.*, 2004; Ivanov *et al.*, 2000). The corresponding acids and metal complexes of phosphoroditihoates (Dtph) have been shown to have many industrial and agricultural applications (Gray *et al.*, 2003, 2004). For example, coordination compounds of transition metals with these ligands are used in agriculture as fungicides; they also function as antioxidants and corrosion inhibitors (Ivanov *et al.*, 2000). Sodium or potassium salts of *O*,*O*′-dialkyl phosphorodithioates are used as selective collector reagents of sulfide minerals (Gray *et al.*, 2003, 2004; Ivanov *et al.*, 2000; Larson, 2004). These complexes can be mono-, di- or polynuclear, the choice being mainly determined by the ability of the Dtph ligands to perform terminal, bridging or mixed structural functions (El-Khaldy *et al.*, 2003). Nickel complexes of Dtph ligands are mononuclear, while zinc complexes of the ligands are dinuclear (Gray *et al.*, 2003, 2004; Ivanov *et al.*, 2000). The structure of the title compound, (I), was reported previously by Chichang *et al.* (1987). The current room-temperature structure determination, however, provides a significant increase in the precision of the geometric parameters.



(I)

Selected bond distances and angles of (I) are listed and compared with those of the previously reported structure, (II)

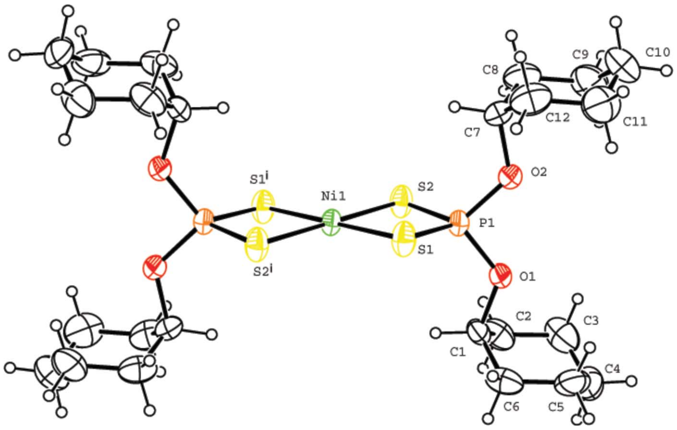


Figure 1
An *ORTEP-3* (Farrugia, 1997) drawing of the title complex, showing the atom-numbering scheme and ellipsoids drawn at the 30% probability level [symmetry code: (i) $1 - x, 1 - y, 1 - z$].

(Chichang *et al.*, 1987), in Table 1, along with those of two nickel complexes of related dialkyl phosphorodithioate ligands, (III) and (IV) (Gray *et al.*, 2003, 2004), and a similar zinc complex, (V) (Ivanov *et al.*, 2000). The title mononuclear nickel complex is shown in Fig. 1, where the Ni atom resides on a crystallographic inversion center and is coordinated by four S atoms in a distorted square-planar geometry with symmetric NiS_2P rings and Ni—S distances of 2.2192 (5) and 2.2202 (5) Å. These distances are similar to those in (II), (III) and (IV), but shorter than the Zn—S distances in (V). In (I), the fairly short P—Ni contacts [2.8470 (5) Å] reflect the presence of possible electronic interaction of the P and Ni centers as a result of the short distance imposed by the four-membered chelate ring.

Experimental

The synthetic route of the ligand was derived from the literature (Rudzinski & Fernando, 1978; Gianini *et al.*, 2000). The title complex was prepared by reacting aqueous solutions of $NiCl_2 \cdot 6H_2O$ (0.005 mol, 1.18 g) and the ammonium salt of the *O*,*O*′-dicyclohexyl phosphorodithioate ligand (0.01 mol, 2.94 g). The resulting violet solid was recrystallized from chloroform.

Crystal data

$[Ni(C_{12}H_{22}O_2PS_2)_2]$
$M_r = 645.50$
Monoclinic, $P2_1/n$
$a = 11.7991$ (6) Å
$b = 9.3550$ (5) Å
$c = 14.2138$ (8) Å
$\beta = 96.764$ (5)°
$V = 1558.01$ (14) Å^3
$Z = 2$

$D_x = 1.376$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 20380 reflections
$\theta = 2.1$–27.9°
$\mu = 1.02$ mm^{-1}
$T = 296$ (2) K
Hexagonal-shaped plate, violet
0.58 × 0.48 × 0.07 mm

Data collection

Stoe IPDS-II diffractometer
ω scans
Absorption correction: integration (*X-RED32*; Stoe & Cie, 2002)
$T_{min} = 0.589$, $T_{max} = 0.932$
20380 measured reflections
3706 independent reflections

2749 reflections with $I > 2\sigma(I)$
$R_{int} = 0.048$
$\theta_{max} = 27.9°$
$h = -15 \rightarrow 15$
$k = -12 \rightarrow 12$
$l = -18 \rightarrow 18$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.032$
$wR(F^2) = 0.084$
$S = 1.04$
3706 reflections
160 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0384P)^2 + 0.2064P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.23$ e Å^{-3}
$\Delta\rho_{min} = -0.22$ e Å^{-3}

Table 1
Selected geometric parameters for complexes (I)–(V).

Distances (Å)	(I)	(II)	(III)	(IV)	(V)[b]
Ni—S1	2.2192 (5)	2.221 (3)	2.2330 (6)	2.2298 (6)	2.411 (2)
Ni—S2	2.2202 (5)	2.217 (3)	2.2413 (6)	2.2327 (6)	2.369 (2)
P1—S1	1.9879 (8)	1.982 (4)	2.0043 (8)	2.0042 (8)	1.955 (3)
P1—S2	1.9917 (8)	1.992 (4)	2.0048 (7)	2.0082 (8)	1.992 (3)
P1—O1	1.5566 (14)	1.565 (6)	1.7904 (18)[a]	1.792 (2)[a]	1.569 (5)
P1—O2	1.5610 (14)	1.568 (7)	1.5856 (13)	1.5855 (15)	1.572 (6)
Angles (°)					
S1—Ni—S2	88.11 (2)	88.01 (10)	88.87 (2)	88.50 (2)	85.62 (8)
S1—P1—S2	101.74 (3)	101.75 (16)	102.77 (3)	101.80 (3)	109.12 (13)
P1—S1—Ni	84.99 (2)	85.02 (13)	82.38 (2)	83.43 (2)	81.99 (10)
P1—S2—Ni	84.87 (2)	84.91 (13)	82.58 (3)	83.26 (2)	83.14 (9)
O1—P1—S2	115.07 (7)	114.0 (3)	112.57 (7)[a]	114.18 (8)[a]	113.4(2)
O2—P1—S1	115.34 (7)	115.4 (3)	113.59 (6)	114.90 (6)	113.4(2)
O1—P1—O2	95.56 (7)	95.9 (3)	101.82 (7)[a]	100.02 (9)[a]	96.4 (3)

Notes: (*a*) In (III) and (IV), atom O1 is replaced by C1; (*b*) the terminal bonding of the ligands in the dimeric Zn complex is used for comparison.

All H atoms were placed in calculated positions, with C—H distances in the range 0.97–0.98 Å and $U_{iso}(H) = 1.2U_{eq}(C)$.

Data collection: *X-AREA* (Stoe & Cie, 2002); cell refinement: *X-AREA*; data reduction: *X-RED32* (Stoe & Cie, 2002); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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