Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

[1,4-Bis(diphenylphosphino)butane-κ^2P,P'](dithiocarbonato-κ^2S,S')nickel(II)

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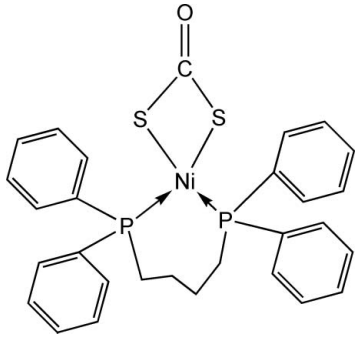
Received 8 June 2007; accepted 12 June 2007

Key indicators: single-crystal X-ray study; T = 294 K; mean σ(C–C) = 0.004 Å; R factor = 0.038; wR factor = 0.097; data-to-parameter ratio = 21.5.

The Ni atom in the title complex, $[Ni(COS_2)(C_{28}H_{28}P_2)]$, adopts a square-planar coordination geometry defined by two S atoms from the dithiocarbonate ligand and two P atoms from the diphosphine ligand. Molecules are self-assembled into a supramolecular array based upon weak intermolecular C—H···O and C—H···π contacts.

Related literature

For related literature, see: Haiduc *et al.* (2003); Perpiñán *et al.* (1987); Tenorio *et al.* (1996); Tiekink & Haiduc (2005); Trávnicek *et al.* (1996).



Experimental

Crystal data

$[Ni(COS_2)(C_{28}H_{28}P_2)]$
M_r = 577.28
Triclinic, $P\overline{1}$
a = 10.1860 (5) Å
b = 10.6926 (6) Å
c = 14.5447 (8) Å
α = 71.906 (1)°
β = 82.710 (1)°
γ = 64.272 (1)°
V = 1356.46 (13) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 1.01 mm^{-1}
T = 294 (2) K
0.40 × 0.35 × 0.24 mm

Data collection

Bruker SMART APEX CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2001)
T_{min} = 0.685, T_{max} = 1
(expected range = 0.538–0.785)
16944 measured reflections
6807 independent reflections
5803 reflections with $I > 2\sigma(I)$
R_{int} = 0.029

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.038
$wR(F^2)$ = 0.097
S = 1.02
6807 reflections
316 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.50 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Ni1—S1	2.1993 (5)	Ni1—P1	2.1922 (5)
Ni1—S2	2.2042 (5)	Ni1—P2	2.2000 (5)
S1—Ni1—S2	79.23 (2)	S2—Ni1—P1	170.74 (2)
S1—Ni1—P1	92.18 (2)	S2—Ni1—P2	91.74 (2)
S1—Ni1—P2	170.09 (2)	P1—Ni1—P2	96.563 (19)

Table 2
Hydrogen-bond geometry (Å, °).

*Cg*1 is the centroid of the C18–C23 ring.

D—H···A	D—H	H···A	D···A	D—H···A
C2—H2A···O1^{i}	0.97	2.43	3.366 (3)	163
C3—H3A···Cg1^{ii}	0.97	2.95	3.886 (3)	164

Symmetry codes: (i) $x+1, y, z$; (ii) $-x+1, -y, -z+1$.

Data collection: *SMART-NT* (Bruker, 2001); cell refinement: *SAINT-Plus NT* (Bruker, 2001); data reduction: *SAINT-Plus NT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* (Farrugia, 1997) and *DIAMOND* (Brandenburg, 1999); software used to prepare material for publication: *SHELXTL* (Sheldrick, 2000).

The authors thank the National Council for University Research, Bucharest, for CEEX grant No. CEx05-D11-42.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: TK2161).

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  doi:10.1107/S1600536807028760

supplementary materials

Acta Cryst. (2007). E**63**, m2168 [**doi:10.1107/S1600536807028760**]

[1,4-Bis(diphenylphosphino)butane-κ^2P,P'](dithiocarbonato-κ^2S,S')nickel(II)

M. V. Câmpian, I. Haiduc and R. F. Semeniuc

Comment

The chemistry of nickel xanthates has been extensively investigated, resulting in a large number of crystal structures reported in the literature (Tiekink & Haiduc, 2005). In several cases, the reaction of nickel xanthates with dithiophosphinoethanes, *via* dealkylation of the xanthate ligand leads to formation of nickel dithiocarbonato complexes. Here, we report the crystal structure of one such complex, [Ni(S_2CO)(dppb)] (**I**), where dppb is $Ph_2P(CH_2)_4PPh_2$, where dealkylation of the xanthate ion took place. A few complexes of the type [Ni(S_2CO)*L*] [*L* = dppe = diphenylphosphinoethane (Trávnicek *et al.*, 1996, Perpiñán *et al.*, 1987, Haiduc *et al.*, 2003), and *L* = dippe = diisopropylphosphinoethane (Tenorio *et al.*, 1996)] are described in the literature, three of them characterized by X-Ray crystallography. The formation of dithiocarbonato complexes is sensitive to solvent used, molar ratio, reaction times and starting materials. The complex [Ni(S_2CO)(dppe)] was obtained as an orange-red solid, starting from [Ni(S_2COR)$_2$] (*R* = Me, Et, Cy) with an excess of dppe in acetone/$CHCl_3$ with long reaction times (Perpiñán *et al.*, 1987) or starting from [Ni(S_2COR)$_2$] (*R* = ^{i}Pr, Me, Et, MeOEt) in methylethylketone (Haiduc *et al.*, 2003) or $CHCl_3$ (Trávnicek *et al.*, 1996) in a 1:1 molar ratio as orange crystals. The formation of a nickel dithiocarbonato complex also occurs when the complex [$NiBr_2$(dippe)] is reacted with two equivalents of alkylxanthates (Tenorio *et al.*, 1996) in acetone.

The nickel atom in **I**, Figure 1, adopts a distorded square planar coordination geometry, defined by two sulfur atoms from the dithiocarbonato ligand and two phosphorous atoms from dppb, with the metal deviation from S_2P_2 plane being −0.078 (2) Å. The dithiocarbonato ligand is more planar with an average deviation from the S_2CO plane of 0.003 (2) Å. The Ni—S bond lengths in **I** are essentially equivalent (Table 1).

Complex molecules of (I) are self-assembled into a supramolecular array *via* two types of contacts. One contact is defined by C—H···O interactions along the *a*-axis. The contacts between adiacent molecules are C2a—H2a···O1 = 2.43 Å, C2a···O1 = 3.366 (3) Å, with the angle at H2a = 163° for symmetry code: 1 + *x*, *y*, *z*. The other interactions are of the type C—H···π, with C3—H3a···C*g*1 [C*g*1 is the centroid of ring C18—C23 at (1 − *x*, −*y*, 1 − *z*)] = 2.95 Å, and an angle of 164° at H3a. A view of the crystal packing is shown in Fig. 2.

Experimental

Complex **I** was prepared by two different methods:

1- To a solution of $NiCl_2$ (0.118 g, 0.002 mol) in ethanol was added dppb (0.213 g, 0.002 mol). The cream precipitate which formed after 15 minutes was dried. A dichloromethane solution of resulting [$NiCl_2$(dppb)] complex was treated with $KS_2COCH_2C_6H_5$ (0.111 g, 0.002 mol). The colour of solution changed to orange. Evaporation of the solution produced orange-red crystals.

2- To a solution of [Ni($S_2COCH_2C_6H_5)_2$] in dichloromethane was added dppb in molar rations 1:1 and 2:1. The colour of the solutions changed from green-brown to orange. In both cases, slow evaporation of the solutions produced orange-red crystals.

The crystals investigated in the present work were obtained by method 2.

IR (KBr): 1700 and 1606 ν(C=O) cm^{-1}. *M*.p. 485 K (dec.).

Refinement

Hydrogen atoms were placed in geometrically idealized positions and included as riding atoms with C—H = 0.93 − 0.97 Å, and with U_{iso}(H) = 1.2U_{iso}(C).

Figures

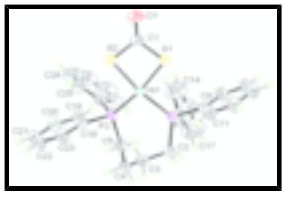

Fig. 1. The structures of **I** showing displacement ellipsoids at the 50% probability level and the atom-numbering scheme.

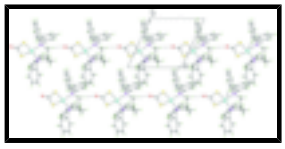

Fig. 2. Supramolecular array in **I**. Dashed lines indicate C—H···O contacts. Colour code: Ni cyan, S yellow, O red, P purple, C grey and H green.

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Crystal data

[Ni(COS_2)($C_{28}H_{28}P_2$)]	*Z* = 2
M_r = 577.28	F_{000} = 600
Triclinic, *P*$\bar{1}$	D_x = 1.413 Mg m^{-3}
Hall symbol: -P 1	Mo *K*α radiation λ = 0.71073 Å
a = 10.1860 (5) Å	Cell parameters from 6076 reflections
b = 10.6926 (6) Å	θ = 2.2–28.3º
c = 14.5447 (8) Å	μ = 1.01 mm^{-1}
α = 71.906 (1)º	*T* = 294 (2) K
β = 82.710 (1)º	Block, orange-red
γ = 64.272 (1)º	0.40 × 0.35 × 0.24 mm
V = 1356.46 (13) $Å^3$	

Data collection

Bruker SMART APEX CCD diffractometer	6807 independent reflections

Radiation source: fine-focus sealed tube	5803 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.029$
$T = 294(2)$ K	$\theta_{max} = 28.5°$
ω scans	$\theta_{min} = 1.5°$
Absorption correction: multi-scan (SADABS; Bruker, 2001)	$h = -13\rightarrow13$
$T_{min} = 0.685$, $T_{max} = 1$	$k = -14\rightarrow14$
16944 measured reflections	$l = -19\rightarrow19$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.038$	H-atom parameters constrained
$wR(F^2) = 0.097$	$w = 1/[\sigma^2(F_o^2) + (0.0507P)^2 + 0.2402P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.02$	$(\Delta/\sigma)_{max} = 0.001$
6807 reflections	$\Delta\rho_{max} = 0.50$ e Å^{-3}
316 parameters	$\Delta\rho_{min} = -0.21$ e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit *S* are based on F^2, conventional *R*-factors *R* are based on *F*, with *F* set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on *F*, and *R*- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
Ni1	0.08348 (2)	0.33684 (2)	0.277861 (16)	0.03010 (8)
P1	0.19635 (5)	0.46982 (5)	0.28031 (4)	0.03368 (11)
P2	0.27605 (5)	0.15608 (5)	0.24566 (4)	0.03491 (12)
S1	−0.13030 (5)	0.50629 (5)	0.29704 (4)	0.04401 (13)
S2	−0.06149 (5)	0.23653 (5)	0.26890 (4)	0.04244 (13)
C1	−0.2082 (2)	0.3967 (2)	0.27981 (15)	0.0412 (4)
C2	0.3590 (2)	0.3826 (2)	0.35932 (16)	0.0452 (5)
H2A	0.4368	0.4036	0.3221	0.054*
H2B	0.3365	0.4268	0.4116	0.054*
C3	0.4157 (2)	0.2194 (2)	0.40317 (17)	0.0505 (5)

H3A	0.4664	0.1926	0.4632	0.061*
H3B	0.3331	0.1944	0.4189	0.061*
C4	0.5177 (2)	0.1305 (3)	0.3392 (2)	0.0583 (6)
H4A	0.5465	0.0287	0.3732	0.070*
H4B	0.6050	0.1479	0.3291	0.070*
C5	0.4550 (2)	0.1611 (2)	0.24049 (17)	0.0493 (5)
H5A	0.5230	0.0903	0.2091	0.059*
H5B	0.4463	0.2560	0.2008	0.059*
C6	0.08675 (19)	0.6338 (2)	0.31755 (14)	0.0367 (4)
C7	0.0233 (2)	0.7690 (2)	0.25106 (17)	0.0500 (5)
H7	0.0401	0.7790	0.1854	0.060*
C8	−0.0650 (3)	0.8894 (2)	0.28206 (19)	0.0600 (6)
H8	−0.1064	0.9797	0.2369	0.072*
C9	−0.0918 (3)	0.8768 (3)	0.37830 (19)	0.0565 (6)
H9	−0.1512	0.9581	0.3985	0.068*
C10	−0.0306 (3)	0.7435 (3)	0.44477 (18)	0.0541 (5)
H10	−0.0495	0.7345	0.5102	0.065*
C11	0.0589 (2)	0.6225 (2)	0.41530 (16)	0.0464 (5)
H11	0.1007	0.5329	0.4611	0.056*
C12	0.2431 (2)	0.5434 (2)	0.15673 (15)	0.0417 (4)
C13	0.1534 (3)	0.5659 (3)	0.08351 (17)	0.0613 (6)
H13	0.0779	0.5365	0.0989	0.074*
C14	0.1743 (4)	0.6314 (3)	−0.0120 (2)	0.0787 (8)
H14	0.1123	0.6472	−0.0602	0.094*
C15	0.2856 (4)	0.6724 (4)	−0.0347 (2)	0.0882 (10)
H15	0.3016	0.7143	−0.0989	0.106*
C16	0.3741 (4)	0.6524 (4)	0.0360 (3)	0.1005 (12)
H16	0.4494	0.6821	0.0194	0.121*
C17	0.3541 (3)	0.5879 (3)	0.1330 (2)	0.0689 (7)
H17	0.4150	0.5754	0.1808	0.083*
C18	0.3102 (2)	−0.0233 (2)	0.32730 (14)	0.0386 (4)
C19	0.2280 (2)	−0.0386 (2)	0.41011 (14)	0.0407 (4)
H19	0.1541	0.0430	0.4236	0.049*
C20	0.2553 (2)	−0.1748 (2)	0.47298 (16)	0.0492 (5)
H20	0.2004	−0.1842	0.5288	0.059*
C21	0.3629 (3)	−0.2957 (2)	0.45310 (18)	0.0560 (6)
H21	0.3801	−0.3869	0.4951	0.067*
C22	0.4457 (3)	−0.2825 (2)	0.3713 (2)	0.0621 (6)
H22	0.5190	−0.3647	0.3582	0.074*
C23	0.4200 (2)	−0.1472 (2)	0.30866 (18)	0.0546 (6)
H23	0.4765	−0.1388	0.2535	0.066*
C24	0.2616 (2)	0.1405 (2)	0.12638 (15)	0.0475 (5)
C25	0.2061 (3)	0.0502 (3)	0.1138 (2)	0.0681 (7)
H25	0.1762	−0.0062	0.1670	0.082*
C26	0.1950 (4)	0.0438 (4)	0.0202 (3)	0.0967 (11)
H26	0.1580	−0.0172	0.0111	0.116*
C27	0.2393 (4)	0.1286 (5)	−0.0583 (3)	0.1049 (14)
H27	0.2344	0.1226	−0.1201	0.126*
C28	0.2892 (4)	0.2191 (5)	−0.0459 (2)	0.1035 (13)

H28	0.3160	0.2776	−0.0995	0.124*
C29	0.3014 (3)	0.2270 (3)	0.04526 (19)	0.0734 (8)
H29	0.3364	0.2906	0.0526	0.088*
O1	−0.33527 (16)	0.4258 (2)	0.27567 (15)	0.0696 (5)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Ni1	0.02394 (12)	0.02904 (13)	0.03664 (13)	−0.01032 (9)	0.00049 (8)	−0.00970 (9)
P1	0.0275 (2)	0.0324 (2)	0.0412 (3)	−0.01285 (18)	−0.00091 (18)	−0.00970 (19)
P2	0.0288 (2)	0.0326 (2)	0.0407 (3)	−0.01038 (18)	0.00403 (18)	−0.01192 (19)
S1	0.0282 (2)	0.0381 (3)	0.0661 (3)	−0.01034 (19)	0.0024 (2)	−0.0213 (2)
S2	0.0338 (2)	0.0407 (3)	0.0592 (3)	−0.0182 (2)	0.0016 (2)	−0.0192 (2)
C1	0.0298 (9)	0.0465 (11)	0.0481 (11)	−0.0166 (8)	0.0018 (8)	−0.0140 (9)
C2	0.0343 (9)	0.0436 (11)	0.0592 (13)	−0.0145 (8)	−0.0108 (9)	−0.0151 (9)
C3	0.0425 (11)	0.0496 (12)	0.0558 (13)	−0.0184 (9)	−0.0172 (9)	−0.0049 (10)
C4	0.0321 (10)	0.0457 (12)	0.0901 (18)	−0.0085 (9)	−0.0163 (11)	−0.0153 (12)
C5	0.0294 (9)	0.0464 (12)	0.0704 (15)	−0.0142 (8)	0.0104 (9)	−0.0207 (10)
C6	0.0334 (9)	0.0332 (9)	0.0461 (10)	−0.0159 (7)	−0.0013 (7)	−0.0114 (8)
C7	0.0576 (13)	0.0377 (11)	0.0494 (12)	−0.0142 (10)	−0.0059 (10)	−0.0113 (9)
C8	0.0675 (15)	0.0356 (11)	0.0658 (15)	−0.0083 (10)	−0.0146 (12)	−0.0134 (10)
C9	0.0517 (12)	0.0475 (12)	0.0709 (16)	−0.0097 (10)	−0.0062 (11)	−0.0313 (11)
C10	0.0546 (13)	0.0563 (14)	0.0524 (13)	−0.0196 (11)	0.0044 (10)	−0.0235 (11)
C11	0.0472 (11)	0.0416 (11)	0.0475 (12)	−0.0181 (9)	0.0018 (9)	−0.0103 (9)
C12	0.0380 (9)	0.0354 (10)	0.0468 (11)	−0.0136 (8)	0.0058 (8)	−0.0101 (8)
C13	0.0653 (15)	0.0685 (16)	0.0486 (13)	−0.0325 (13)	−0.0027 (11)	−0.0069 (11)
C14	0.093 (2)	0.081 (2)	0.0474 (14)	−0.0309 (17)	−0.0022 (14)	−0.0062 (13)
C15	0.088 (2)	0.085 (2)	0.0576 (17)	−0.0255 (18)	0.0222 (16)	0.0002 (15)
C16	0.080 (2)	0.116 (3)	0.093 (2)	−0.058 (2)	0.0311 (19)	0.000 (2)
C17	0.0552 (14)	0.0801 (18)	0.0725 (17)	−0.0403 (14)	0.0092 (12)	−0.0088 (14)
C18	0.0360 (9)	0.0305 (9)	0.0461 (11)	−0.0098 (7)	−0.0020 (8)	−0.0116 (8)
C19	0.0389 (10)	0.0363 (10)	0.0440 (11)	−0.0121 (8)	−0.0008 (8)	−0.0125 (8)
C20	0.0513 (12)	0.0445 (12)	0.0476 (12)	−0.0207 (10)	−0.0042 (9)	−0.0049 (9)
C21	0.0614 (14)	0.0354 (11)	0.0648 (15)	−0.0176 (10)	−0.0128 (11)	−0.0043 (10)
C22	0.0583 (14)	0.0345 (11)	0.0785 (17)	−0.0025 (10)	−0.0045 (12)	−0.0195 (11)
C23	0.0510 (12)	0.0402 (11)	0.0616 (14)	−0.0086 (9)	0.0100 (10)	−0.0193 (10)
C24	0.0406 (10)	0.0488 (12)	0.0432 (11)	−0.0065 (9)	0.0021 (8)	−0.0188 (9)
C25	0.0665 (16)	0.0746 (18)	0.0691 (17)	−0.0239 (14)	−0.0034 (13)	−0.0351 (14)
C26	0.082 (2)	0.114 (3)	0.104 (3)	−0.018 (2)	−0.017 (2)	−0.070 (2)
C27	0.083 (2)	0.149 (4)	0.064 (2)	−0.011 (2)	−0.0089 (17)	−0.057 (2)
C28	0.101 (3)	0.140 (4)	0.0462 (16)	−0.032 (3)	0.0029 (16)	−0.0237 (19)
C29	0.0768 (18)	0.085 (2)	0.0487 (14)	−0.0296 (16)	0.0097 (12)	−0.0163 (13)
O1	0.0308 (7)	0.0726 (12)	0.1132 (15)	−0.0208 (8)	0.0029 (8)	−0.0388 (11)

Geometric parameters (Å, °)

Ni1—S1	2.1993 (5)	C11—H11	0.9300
Ni1—S2	2.2042 (5)	C12—C17	1.376 (3)
Ni1—P1	2.1922 (5)	C12—C13	1.386 (3)

Ni1—P2	2.2000 (5)	C13—C14	1.384 (3)
P1—C12	1.818 (2)	C13—H13	0.9300
P1—C6	1.831 (2)	C14—C15	1.354 (5)
P1—C2	1.8472 (19)	C14—H14	0.9300
P2—C18	1.8225 (19)	C15—C16	1.358 (5)
P2—C24	1.824 (2)	C15—H15	0.9300
P2—C5	1.838 (2)	C16—C17	1.397 (4)
S1—C1	1.762 (2)	C16—H16	0.9300
S2—C1	1.754 (2)	C17—H17	0.9300
C1—O1	1.198 (2)	C18—C19	1.385 (3)
C2—C3	1.520 (3)	C18—C23	1.393 (3)
C2—H2A	0.9700	C19—C20	1.386 (3)
C2—H2B	0.9700	C19—H19	0.9300
C3—C4	1.516 (3)	C20—C21	1.370 (3)
C3—H3A	0.9700	C20—H20	0.9300
C3—H3B	0.9700	C21—C22	1.375 (3)
C4—C5	1.528 (3)	C21—H21	0.9300
C4—H4A	0.9700	C22—C23	1.380 (3)
C4—H4B	0.9700	C22—H22	0.9300
C5—H5A	0.9700	C23—H23	0.9300
C5—H5B	0.9700	C24—C25	1.376 (4)
C6—C7	1.386 (3)	C24—C29	1.393 (3)
C6—C11	1.392 (3)	C25—C26	1.405 (4)
C7—C8	1.386 (3)	C25—H25	0.9300
C7—H7	0.9300	C26—C27	1.383 (6)
C8—C9	1.367 (3)	C26—H26	0.9300
C8—H8	0.9300	C27—C28	1.337 (6)
C9—C10	1.371 (3)	C27—H27	0.9300
C9—H9	0.9300	C28—C29	1.379 (4)
C10—C11	1.382 (3)	C28—H28	0.9300
C10—H10	0.9300	C29—H29	0.9300
S1—Ni1—S2	79.23 (2)	C9—C10—H10	119.7
S1—Ni1—P1	92.18 (2)	C11—C10—H10	119.7
S1—Ni1—P2	170.09 (2)	C10—C11—C6	120.5 (2)
S2—Ni1—P1	170.74 (2)	C10—C11—H11	119.8
S2—Ni1—P2	91.74 (2)	C6—C11—H11	119.8
P1—Ni1—P2	96.563 (19)	C17—C12—C13	118.7 (2)
C12—P1—C6	102.21 (9)	C17—C12—P1	123.75 (19)
C12—P1—C2	109.93 (10)	C13—C12—P1	117.32 (16)
C6—P1—C2	102.47 (10)	C14—C13—C12	121.2 (2)
C12—P1—Ni1	108.16 (7)	C14—C13—H13	119.4
C6—P1—Ni1	115.82 (6)	C12—C13—H13	119.4
C2—P1—Ni1	117.17 (7)	C15—C14—C13	119.5 (3)
C18—P2—C24	104.29 (10)	C15—C14—H14	120.2
C18—P2—C5	102.64 (9)	C13—C14—H14	120.2
C24—P2—C5	102.18 (10)	C14—C15—C16	120.2 (3)
C18—P2—Ni1	115.87 (6)	C14—C15—H15	119.9
C24—P2—Ni1	111.50 (7)	C16—C15—H15	119.9
C5—P2—Ni1	118.56 (7)	C15—C16—C17	121.3 (3)

C1—S1—Ni1	87.28 (7)	C15—C16—H16	119.4
C1—S2—Ni1	87.33 (7)	C17—C16—H16	119.4
O1—C1—S2	126.94 (18)	C12—C17—C16	119.0 (3)
O1—C1—S1	127.09 (18)	C12—C17—H17	120.5
S2—C1—S1	105.97 (10)	C16—C17—H17	120.5
C3—C2—P1	116.19 (14)	C19—C18—C23	118.72 (18)
C3—C2—H2A	108.2	C19—C18—P2	120.30 (14)
P1—C2—H2A	108.2	C23—C18—P2	120.98 (16)
C3—C2—H2B	108.2	C18—C19—C20	120.33 (19)
P1—C2—H2B	108.2	C18—C19—H19	119.8
H2A—C2—H2B	107.4	C20—C19—H19	119.8
C4—C3—C2	115.0 (2)	C21—C20—C19	120.2 (2)
C4—C3—H3A	108.5	C21—C20—H20	119.9
C2—C3—H3A	108.5	C19—C20—H20	119.9
C4—C3—H3B	108.5	C20—C21—C22	120.3 (2)
C2—C3—H3B	108.5	C20—C21—H21	119.9
H3A—C3—H3B	107.5	C22—C21—H21	119.9
C3—C4—C5	115.25 (17)	C21—C22—C23	120.0 (2)
C3—C4—H4A	108.5	C21—C22—H22	120.0
C5—C4—H4A	108.5	C23—C22—H22	120.0
C3—C4—H4B	108.5	C22—C23—C18	120.5 (2)
C5—C4—H4B	108.5	C22—C23—H23	119.7
H4A—C4—H4B	107.5	C18—C23—H23	119.7
C4—C5—P2	114.22 (15)	C25—C24—C29	119.0 (2)
C4—C5—H5A	108.7	C25—C24—P2	121.76 (19)
P2—C5—H5A	108.7	C29—C24—P2	119.2 (2)
C4—C5—H5B	108.7	C24—C25—C26	119.6 (3)
P2—C5—H5B	108.7	C24—C25—H25	120.2
H5A—C5—H5B	107.6	C26—C25—H25	120.2
C7—C6—C11	118.40 (19)	C27—C26—C25	119.7 (4)
C7—C6—P1	122.11 (16)	C27—C26—H26	120.1
C11—C6—P1	119.39 (15)	C25—C26—H26	120.1
C8—C7—C6	120.3 (2)	C28—C27—C26	120.4 (3)
C8—C7—H7	119.9	C28—C27—H27	119.8
C6—C7—H7	119.9	C26—C27—H27	119.8
C9—C8—C7	120.8 (2)	C27—C28—C29	120.9 (4)
C9—C8—H8	119.6	C27—C28—H28	119.5
C7—C8—H8	119.6	C29—C28—H28	119.5
C8—C9—C10	119.5 (2)	C28—C29—C24	120.4 (3)
C8—C9—H9	120.2	C28—C29—H29	119.8
C10—C9—H9	120.2	C24—C29—H29	119.8
C9—C10—C11	120.5 (2)		
S1—Ni1—P1—C12	−103.16 (7)	C6—P1—C12—C17	80.7 (2)
P2—Ni1—P1—C12	72.16 (7)	C2—P1—C12—C17	−27.5 (2)
S1—Ni1—P1—C6	10.79 (7)	Ni1—P1—C12—C17	−156.61 (19)
P2—Ni1—P1—C6	−173.89 (7)	C6—P1—C12—C13	−93.94 (19)
S1—Ni1—P1—C2	131.98 (9)	C2—P1—C12—C13	157.80 (18)
P2—Ni1—P1—C2	−52.70 (9)	Ni1—P1—C12—C13	28.74 (19)
P1—Ni1—P2—C18	127.04 (7)	C17—C12—C13—C14	0.2 (4)

S2—Ni1—P2—C18	−57.06 (8)	P1—C12—C13—C14	175.1 (2)
P1—Ni1—P2—C24	−113.90 (8)	C12—C13—C14—C15	1.0 (5)
S2—Ni1—P2—C24	62.00 (8)	C13—C14—C15—C16	−1.5 (5)
P1—Ni1—P2—C5	4.30 (9)	C14—C15—C16—C17	0.8 (6)
S2—Ni1—P2—C5	−179.80 (9)	C13—C12—C17—C16	−0.9 (4)
P1—Ni1—S1—C1	173.66 (7)	P1—C12—C17—C16	−175.5 (2)
S2—Ni1—S1—C1	−2.87 (7)	C15—C16—C17—C12	0.4 (5)
S1—Ni1—S2—C1	2.88 (7)	C24—P2—C18—C19	−130.28 (17)
P2—Ni1—S2—C1	−173.00 (7)	C5—P2—C18—C19	123.45 (17)
Ni1—S2—C1—O1	175.8 (2)	Ni1—P2—C18—C19	−7.34 (19)
Ni1—S2—C1—S1	−3.67 (9)	C24—P2—C18—C23	50.6 (2)
Ni1—S1—C1—O1	−175.8 (2)	C5—P2—C18—C23	−55.7 (2)
Ni1—S1—C1—S2	3.68 (9)	Ni1—P2—C18—C23	173.53 (16)
C12—P1—C2—C3	−114.16 (18)	C23—C18—C19—C20	0.1 (3)
C6—P1—C2—C3	137.74 (17)	P2—C18—C19—C20	−179.03 (16)
Ni1—P1—C2—C3	9.8 (2)	C18—C19—C20—C21	−0.7 (3)
P1—C2—C3—C4	84.9 (2)	C19—C20—C21—C22	0.8 (4)
C2—C3—C4—C5	−57.5 (3)	C20—C21—C22—C23	−0.3 (4)
C3—C4—C5—P2	−50.8 (2)	C21—C22—C23—C18	−0.2 (4)
C18—P2—C5—C4	−55.40 (18)	C19—C18—C23—C22	0.3 (3)
C24—P2—C5—C4	−163.29 (16)	P2—C18—C23—C22	179.49 (19)
Ni1—P2—C5—C4	73.73 (17)	C18—P2—C24—C25	32.2 (2)
C12—P1—C6—C7	18.83 (19)	C5—P2—C24—C25	138.8 (2)
C2—P1—C6—C7	132.71 (17)	Ni1—P2—C24—C25	−93.57 (19)
Ni1—P1—C6—C7	−98.49 (16)	C18—P2—C24—C29	−150.84 (19)
C12—P1—C6—C11	−164.81 (16)	C5—P2—C24—C29	−44.2 (2)
C2—P1—C6—C11	−50.93 (17)	Ni1—P2—C24—C29	83.41 (19)
Ni1—P1—C6—C11	77.87 (16)	C29—C24—C25—C26	2.0 (4)
C11—C6—C7—C8	0.3 (3)	P2—C24—C25—C26	178.9 (2)
P1—C6—C7—C8	176.71 (18)	C24—C25—C26—C27	−0.2 (5)
C6—C7—C8—C9	−0.5 (4)	C25—C26—C27—C28	−1.7 (5)
C7—C8—C9—C10	0.0 (4)	C26—C27—C28—C29	1.8 (6)
C8—C9—C10—C11	0.7 (4)	C27—C28—C29—C24	−0.1 (5)
C9—C10—C11—C6	−0.8 (3)	C25—C24—C29—C28	−1.8 (4)
C7—C6—C11—C10	0.3 (3)	P2—C24—C29—C28	−178.9 (2)
P1—C6—C11—C10	−176.18 (17)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C2—H2A···O1^{i}	0.97	2.43	3.366 (3)	163
C3—H3A···Cg1^{ii}	0.97	2.95	?	164

Symmetry codes: (i) *x*+1, *y*, *z*; (ii) −*x*+1, −*y*, −*z*+1.

Fig. 1

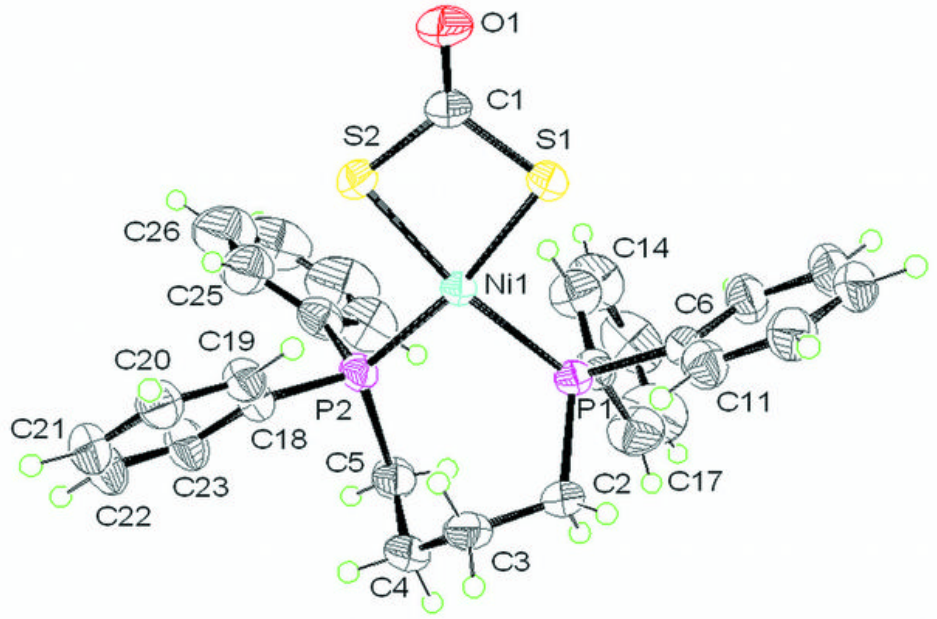
O1
C1
S2
S1
C26
C25
Ni1
C14
C6
C19
C20
P2
P1
C11
C21
C18
C5
C23
C2
C17
C22
C3
C4

Fig. 2