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[*N*-(2,5-Dichlorophenylsulfonyl)dithiocarbimato(2−)-$\kappa^2 S,S'$]bis(triphenylphosphine-κP)nickel(II)

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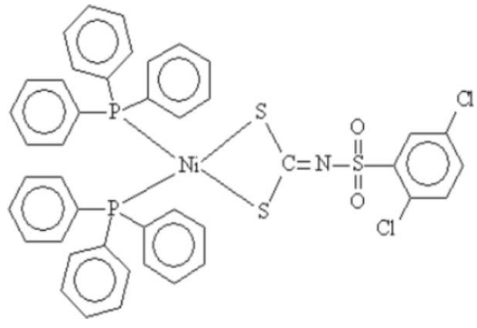
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.005 Å; R factor = 0.047; wR factor = 0.123; data-to-parameter ratio = 19.0.

In the title complex, $[Ni(C_7H_3Cl_2NO_2S_3)(C_{18}H_{15}P)_2]$, a distorted *cis*-$NiS_2P_2$ square-planar configuration around the Ni atom occurs due to the steric effect of the bulky triphenylphosphine ligands and the bidendate chelation by the two S atoms of the dithiocarbimate ligand. The crystal packing is stabilized by weak C_{ar}—H···X (X = O and S) intermolecular interactions.

Related literature

For related literature, see: Allen *et al.* (1987); Cavell *et al.* (1998); Foulds *et al.* (1994); Franca *et al.* (2006); Oliveira *et al.* (2002, 2003); Orpen *et al.* (1989); Bruno *et al.* (2002).



Experimental

Crystal data

$[Ni(C_7H_3Cl_2NO_2S_3)(C_{18}H_{15}P)_2]$
M_r = 883.43
Orthorhombic, *Pbca*
a = 18.4654 (3) Å
b = 15.2416 (2) Å
c = 28.8619 (5) Å
V = 8123.0 (2) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.88 mm^{-1}
T = 293 (2) K
0.20 × 0.18 × 0.03 mm

Data collection

Nonius KappaCCD diffractometer
Absorption correction: multi-scan (*SORTAV*; Blessing, 1995)
T_{min} = 0.844, T_{max} = 0.974
41274 measured reflections
9277 independent reflections
5597 reflections with $I > 2\sigma(I)$
R_{int} = 0.063

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.047
$wR(F^2)$ = 0.124
S = 1.01
9277 reflections
487 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.44 e Å^{-3}
$\Delta\rho_{min}$ = −0.38 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Ni—S1	2.2027 (8)	Ni—P1	2.2227 (8)
Ni—S2	2.2147 (8)	Ni—P2	2.2564 (8)
S1—Ni—S2	77.09 (3)	S2—Ni—P2	90.00 (3)
S1—Ni—P1	89.89 (3)	P1—Ni—P2	103.02 (3)

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

D—H···A	D—H	H···A	D···A	D—H···A
C36—H36···S1^{i}	0.93	2.8	3.641 (3)	151
C34—H34···O1^{ii}	0.93	2.57	3.194 (5)	125
C41—H41···O2^{iii}	0.93	2.48	3.197 (4)	134

Symmetry codes: (i) $-x-\frac{1}{2}, y-\frac{1}{2}, z$; (ii) $-x, y-\frac{1}{2}, -z+\frac{1}{2}$; (iii) $x, -y+\frac{1}{2}, z-\frac{1}{2}$.

Data collection: *COLLECT* (Enraf–Nonius, 2000); cell refinement: *SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *DENZO* (Otwinowski & Minor, 1997) and *SCALEPACK*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB2442).

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supplementary materials

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[*N*-(2,5-Dichlorophenylsulfonyl)dithiocarbimato(2-)-$\kappa^2 S,S'$]bis(triphenylphosphine-κP)nickel(II)

C. Novais, S. Guilardi, I. Machado Jr and M. R. L. Oliveira

Comment

We became interested in the syntheses and characterization of nickel(II) complexes with dithiocarbimates and phosphines due to their similarities with the dithiocarbimate compounds, which have shown catalytic activity, especially for oligomerization of olefins (Cavell *et al.*, 1998; Foulds *et al.*, 1994). As only the nickel complexes with general formulae $Ni(RSO_2N{=}CS_2)(PPh_3)_2$ (R = 2-$CH_3C_6H_4$, 4-$CH_3C_6H_4$ and 4-BrC_6H_4) have had their structures determined by X-ray diffraction techniques (Oliveira *et al.*, 2002), the title compound (I) was prepared.

As shown in Fig. 1, the structure of (I) is composed of neutral $Ni(2,5\text{-}Cl_2C_6H_3SO_2NCS_2)(Ph_3P)_2$ complex molecules. The Ni^{II} atom is coordinated by two sulfur atoms from the dithiocarbimate anion and by two phosphorus atoms of the triphenylphosphine ligands into a distorted square-planar geometry. The small S—Ni—S angle is determined by the geometry of the chelate ligand, while the opposite P—Ni—P angle is rather large, probably due to the steric effect of the large triphenylphosphine ligands (Table 1). The two Ni—S bond lenghts are not significantly different, although the two Ni—P distances are. The C—S [1.732 (3) and 1.722 (3) Å] bond lengths of the NCS_2 fragment are nearly equal and are much shorter than typical C—S single bonds [*ca* 1.81 Å]. The $C_1{=}N$ bond distance of 1.303 (3)Å is shorter than normal single C_{sp^2}—N_{sp^2} bond length [*ca* 1.35 Å] and similar to that of the double bond C═N [1.275–1.300 Å] (Allen *et al.*, 1987; Orpen *et al.*, 1989). This behavior indicates that the electron density is delocalized over the entire NCS_2 moiety. Similar behavior is observed for related nickel complexes (Oliveira *et al.*, 2002; Oliveira *et al.*, 2003; Franca *et al.*, 2006). The repulsive interaction *cis* between the $SO_2(2,5\text{-}Cl_2C_6H_3)$ group and the S_1 atom, which are in greater position in relation to the C1—N bond, is responsible for the difference between the angles S1—C1—N [132.6 (2)°] and S2—C1—N [121.8 (2)°].

The torsion angle C1—N—S3—C2 describing the conformation of the ligands along the N—S3 bond is 85.1 (3)°. The C_{ar}—C_{ar} bond lengths of the triphenylphosphine aromatic rings involved in the intermolecular interactions are shorter than typical values described in the literature[1.372–1.388 Å] (Allen *et al.*, 1987).

In the crystal structure of (I), the molecules are linked through C—H···O and C—H···S interactions (Table 2 and Fig. 2).

Experimental

The potassium dithiocarbimate dihydrate was prepared according to Franca *et al.*,(2006). The title compound was prepared in 30:20 ml e thanol:water mixture from nickel chloride hexahydrate (1.0 mmol), triphenylphosphine (2.0 mmol) and potassium 2,5-dichlorophenylsulfonyldithiocarbimate dihydrate (1.0 mmol) analogously as described in the literature for similar compounds (Oliveira *et al.*, 2002). Suitable crystals were obtained after slow evaporation of a solution of the compound in dichloromethane/ethanol (2:3 *v*/*v*). Elemental analysis: Found (calculated)%: C 56.77 (58.46), H 3.81 (3.76), N 1.54 (1.59) and Ni 6.79 (6.64). *M*.p. 175.0–176.5°C. IR (most important bands) (cm^{-1}): 1449 ν(C═N); 1312 $\nu_{ass}(SO_2)$; 1153 $\nu_{sym}(SO_2)$; 939 $\nu_{ass}(CS_2)$ and 370 ν(NiS).

Refinement

All the H atoms were geometrically placed (C—H = 0.93 Å) and refined as riding with U_{iso}(H) = 1.2 U_{eq}(C).

Figures

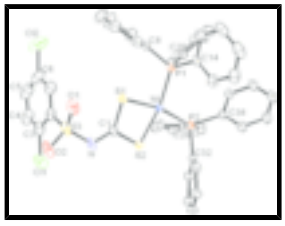

Fig. 1. The molecular structure of (I). Displacement ellipsoids are drawn at the 30% probability level. H atoms have been omitted for clarity.

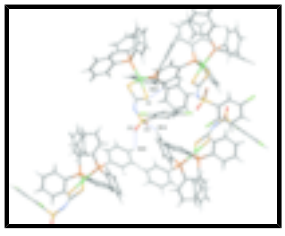

Fig. 2. View of the C—H···S and C—H···O intermolecular interactions (broken lines) in (I).

[*N*-(2,5-Dichlorophenylsulfonyl)dithiocarbimato(2-)- κ^2*S*,*S*']bis(triphenylphosphine-κ*P*)nickel(II)

Crystal data

$[Ni(C_7H_3Cl_2NO_2S_3)(C_{18}H_{15}P)_2]$	F_{000} = 3632
M_r = 883.43	D_x = 1.445 Mg m^{-3}
Orthorhombic, *Pbca*	Mo *K*α radiation λ = 0.71073 Å
Hall symbol: -P 2ac 2ab	Cell parameters from 41144 reflections
a = 18.4654 (3) Å	θ = 2.9–27.5º
b = 15.2416 (2) Å	μ = 0.88 mm^{-1}
c = 28.8619 (5) Å	*T* = 293 (2) K
V = 8123.0 (2) Å^3	Prism, brown
Z = 8	0.20 × 0.18 × 0.03 mm

Data collection

Nonius KappaCCD diffractometer	R_{int} = 0.063
CCD rotation images, thick slices scans	θ_{max} = 27.5º
Absorption correction: multi-scan (SORTAV; Blessing, 1995)	θ_{min} = 2.9º
T_{min} = 0.844, T_{max} = 0.974	*h* = −23→22
41274 measured reflections	*k* = −17→19
9277 independent reflections	*l* = −28→37
5597 reflections with $I > 2\sigma(I)$	

Refinement

Refinement on F^2	H-atom parameters constrained
Least-squares matrix: full	$w = 1/[\sigma^2(F_o^2) + (0.0606P)^2 + 0.6249P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)] = 0.047$	$(\Delta/\sigma)_{max} = 0.031$
$wR(F^2) = 0.124$	$\Delta\rho_{max} = 0.44$ e Å^{-3}
$S = 1.02$	$\Delta\rho_{min} = -0.38$ e Å^{-3}
9277 reflections	Extinction correction: none
487 parameters	

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.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
Ni	−0.108821 (19)	0.29799 (2)	0.164898 (11)	0.03513 (11)
P2	−0.17650 (4)	0.20227 (5)	0.12389 (2)	0.03769 (18)
S1	−0.05827 (4)	0.38162 (5)	0.21807 (2)	0.04220 (19)
S2	−0.16167 (5)	0.25340 (5)	0.22963 (2)	0.0505 (2)
P1	−0.04277 (4)	0.36360 (5)	0.11063 (2)	0.03681 (18)
S3	−0.07421 (5)	0.41888 (6)	0.33031 (3)	0.0564 (2)
Cl1	−0.23725 (6)	0.44726 (8)	0.37366 (4)	0.0924 (3)
Cl2	−0.09475 (9)	0.74289 (9)	0.25675 (7)	0.1463 (6)
O2	−0.07530 (15)	0.39752 (17)	0.37835 (7)	0.0786 (8)
O1	−0.00420 (13)	0.43555 (16)	0.31015 (8)	0.0709 (7)
N	−0.12051 (14)	0.34355 (16)	0.30359 (8)	0.0505 (7)
C26	−0.26532 (16)	0.24552 (17)	0.10822 (9)	0.0419 (7)
C37	−0.26867 (18)	0.0864 (2)	0.17204 (10)	0.0519 (8)
H37	−0.3068	0.1161	0.158	0.062*
C14	0.03409 (16)	0.29937 (19)	0.09022 (9)	0.0416 (7)
C27	−0.29528 (18)	0.31181 (18)	0.13457 (11)	0.0505 (8)
H27	−0.2686	0.3369	0.1585	0.061*
C1	−0.11252 (16)	0.33135 (18)	0.25921 (9)	0.0399 (7)
C15	0.03301 (18)	0.2090 (2)	0.09561 (11)	0.0534 (8)
H15	−0.006	0.182	0.1102	0.064*
C8	−0.00162 (17)	0.46596 (18)	0.13038 (9)	0.0411 (7)
C32	−0.19791 (17)	0.10864 (18)	0.16150 (9)	0.0416 (7)
C39	−0.14815 (18)	0.1978 (2)	0.02842 (10)	0.0517 (8)
H39	−0.1675	0.2542	0.0281	0.062*
C38	−0.14443 (16)	0.15119 (18)	0.07007 (9)	0.0411 (7)

C9	0.06137 (17)	0.4641 (2)	0.15675 (10)	0.0507 (8)
H9	0.085	0.4111	0.1617	0.061*
C25	−0.16342 (18)	0.42726 (18)	0.06566 (11)	0.0519 (8)
H25	−0.183	0.4295	0.0953	0.062*
C43	−0.11539 (18)	0.06701 (19)	0.06948 (10)	0.0525 (8)
H43	−0.1124	0.0349	0.0968	0.063*
C41	−0.0948 (2)	0.0775 (2)	−0.01238 (12)	0.0689 (11)
H41	−0.0782	0.0529	−0.0399	0.083*
C36	−0.2827 (2)	0.0201 (2)	0.20334 (12)	0.0667 (10)
H36	−0.3303	0.0057	0.2105	0.08*
C20	−0.09327 (16)	0.39802 (18)	0.05934 (9)	0.0417 (7)
C35	−0.2269 (3)	−0.0250 (2)	0.22402 (11)	0.0677 (11)
H35	−0.2367	−0.0699	0.2449	0.081*
C21	−0.06526 (19)	0.3956 (2)	0.01444 (10)	0.0583 (9)
H21	−0.018	0.3767	0.0094	0.07*
C34	−0.1570 (2)	−0.0035 (2)	0.21386 (10)	0.0603 (9)
H34	−0.1192	−0.0342	0.2276	0.072*
C33	−0.14217 (19)	0.06362 (19)	0.18324 (10)	0.0495 (8)
H33	−0.0943	0.0788	0.1771	0.059*
C40	−0.1235 (2)	0.1611 (2)	−0.01208 (11)	0.0641 (10)
H40	−0.1261	0.1928	−0.0395	0.077*
C3	−0.1939 (2)	0.5287 (2)	0.34238 (12)	0.0669 (10)
C29	−0.40438 (19)	0.3065 (2)	0.09024 (13)	0.0628 (9)
H29	−0.4507	0.3275	0.084	0.075*
C13	−0.03587 (19)	0.5454 (2)	0.12431 (12)	0.0593 (9)
H13	−0.078	0.5486	0.1068	0.071*
C12	−0.0077 (3)	0.6202 (2)	0.14415 (15)	0.0856 (13)
H12	−0.0315	0.6734	0.1402	0.103*
C28	−0.36505 (19)	0.3412 (2)	0.12564 (13)	0.0606 (9)
H28	−0.3851	0.385	0.144	0.073*
C31	−0.30672 (18)	0.2095 (2)	0.07258 (11)	0.0581 (9)
H31	−0.2878	0.1643	0.0546	0.07*
C10	0.0891 (2)	0.5400 (3)	0.17565 (11)	0.0621 (10)
H10	0.1318	0.5379	0.1927	0.075*
C24	−0.2048 (2)	0.4534 (2)	0.02786 (15)	0.0700 (10)
H24	−0.2519	0.4734	0.0323	0.084*
C17	0.1492 (2)	0.1978 (3)	0.05903 (13)	0.0802 (12)
H17	0.1876	0.1637	0.0484	0.096*
C19	0.09431 (18)	0.3380 (2)	0.06974 (11)	0.0574 (9)
H19	0.0961	0.3986	0.0663	0.069*
C4	−0.2293 (2)	0.6077 (3)	0.33782 (15)	0.0851 (13)
H4	−0.2738	0.6167	0.3523	0.102*
C2	−0.12607 (18)	0.5160 (2)	0.32230 (11)	0.0547 (8)
C42	−0.0909 (2)	0.0307 (2)	0.02816 (11)	0.0643 (10)
H42	−0.0716	−0.0257	0.028	0.077*
C30	−0.37536 (19)	0.2404 (2)	0.06382 (12)	0.0656 (10)
H30	−0.4023	0.2163	0.0398	0.079*
C22	−0.1073 (3)	0.4210 (3)	−0.02223 (12)	0.0775 (12)
H22	−0.0884	0.4185	−0.0521	0.093*

C18	0.1515 (2)	0.2872 (3)	0.05452 (12)	0.0764 (11)
H18	0.1917	0.3138	0.0411	0.092*
C7	−0.0958 (2)	0.5822 (3)	0.29587 (12)	0.0699 (10)
H7	−0.051	0.5741	0.2818	0.084*
C16	0.0905 (2)	0.1588 (2)	0.07918 (13)	0.0722 (11)
H16	0.0889	0.0981	0.0819	0.087*
C5	−0.1980 (3)	0.6738 (3)	0.31137 (17)	0.0919 (14)
H5	−0.2218	0.7272	0.3079	0.11*
C6	−0.1327 (3)	0.6609 (3)	0.29047 (16)	0.0864 (13)
C23	−0.1763 (3)	0.4497 (3)	−0.01572 (14)	0.0790 (12)
H23	−0.2041	0.4669	−0.041	0.095*
C11	0.0547 (3)	0.6171 (3)	0.16952 (14)	0.0816 (12)
H11	0.0734	0.6682	0.1825	0.098*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Ni	0.0387 (2)	0.0379 (2)	0.02878 (18)	−0.00171 (16)	0.00288 (15)	−0.00028 (14)
P2	0.0394 (4)	0.0402 (4)	0.0334 (4)	−0.0018 (3)	0.0043 (3)	−0.0045 (3)
S1	0.0436 (5)	0.0507 (4)	0.0323 (4)	−0.0052 (4)	0.0008 (3)	−0.0028 (3)
S2	0.0643 (6)	0.0518 (5)	0.0354 (4)	−0.0143 (4)	0.0103 (4)	−0.0024 (3)
P1	0.0373 (4)	0.0401 (4)	0.0330 (4)	−0.0005 (3)	0.0014 (3)	0.0018 (3)
S3	0.0527 (5)	0.0816 (6)	0.0348 (4)	0.0103 (4)	−0.0072 (4)	−0.0117 (4)
Cl1	0.0748 (7)	0.1148 (9)	0.0875 (7)	0.0109 (6)	0.0260 (6)	−0.0013 (6)
Cl2	0.1631 (15)	0.0837 (9)	0.1921 (15)	−0.0407 (9)	−0.0270 (12)	0.0282 (10)
O2	0.0895 (19)	0.116 (2)	0.0306 (11)	0.0289 (17)	−0.0115 (12)	−0.0063 (12)
O1	0.0446 (15)	0.1049 (19)	0.0631 (14)	0.0028 (13)	−0.0065 (12)	−0.0231 (14)
N	0.0566 (17)	0.0619 (16)	0.0329 (13)	0.0060 (14)	0.0042 (12)	−0.0008 (12)
C26	0.0423 (18)	0.0439 (16)	0.0394 (15)	−0.0005 (14)	0.0034 (14)	−0.0007 (13)
C37	0.051 (2)	0.0528 (18)	0.0520 (18)	−0.0115 (16)	0.0086 (16)	−0.0057 (15)
C14	0.0406 (18)	0.0492 (17)	0.0351 (14)	0.0010 (14)	0.0006 (13)	−0.0009 (13)
C27	0.051 (2)	0.0460 (17)	0.0545 (19)	0.0027 (16)	0.0006 (15)	−0.0080 (15)
C1	0.0403 (17)	0.0435 (15)	0.0357 (15)	0.0092 (13)	0.0023 (12)	0.0006 (13)
C15	0.053 (2)	0.0528 (19)	0.0541 (19)	0.0094 (16)	−0.0067 (16)	0.0001 (15)
C8	0.0439 (18)	0.0463 (16)	0.0331 (14)	−0.0062 (15)	0.0044 (13)	0.0025 (12)
C32	0.050 (2)	0.0405 (15)	0.0348 (15)	−0.0060 (15)	0.0064 (13)	−0.0075 (13)
C39	0.064 (2)	0.0492 (18)	0.0423 (17)	−0.0053 (16)	0.0049 (16)	−0.0046 (14)
C38	0.0388 (17)	0.0446 (17)	0.0399 (16)	−0.0074 (14)	0.0059 (13)	−0.0060 (13)
C9	0.046 (2)	0.060 (2)	0.0462 (18)	−0.0042 (16)	0.0019 (15)	0.0051 (15)
C25	0.054 (2)	0.0465 (17)	0.0556 (19)	0.0008 (16)	−0.0089 (16)	0.0033 (15)
C43	0.062 (2)	0.0534 (19)	0.0424 (17)	0.0034 (16)	0.0089 (15)	−0.0024 (15)
C41	0.087 (3)	0.073 (2)	0.046 (2)	0.005 (2)	0.0177 (19)	−0.0136 (18)
C36	0.072 (3)	0.061 (2)	0.067 (2)	−0.025 (2)	0.023 (2)	−0.0069 (19)
C20	0.0435 (19)	0.0435 (16)	0.0380 (15)	−0.0059 (14)	−0.0032 (13)	0.0049 (13)
C35	0.104 (3)	0.0481 (19)	0.051 (2)	−0.021 (2)	0.022 (2)	0.0010 (16)
C21	0.058 (2)	0.076 (2)	0.0411 (17)	−0.0096 (19)	−0.0001 (15)	0.0096 (16)
C34	0.092 (3)	0.0468 (18)	0.0424 (17)	0.0018 (19)	0.0033 (18)	0.0024 (15)
C33	0.053 (2)	0.0535 (18)	0.0419 (16)	−0.0006 (16)	0.0078 (15)	−0.0048 (15)

C40	0.086 (3)	0.064 (2)	0.0420 (18)	0.002 (2)	0.0125 (17)	0.0024 (16)
C3	0.058 (2)	0.078 (2)	0.065 (2)	0.004 (2)	−0.0110 (18)	−0.0194 (19)
C29	0.047 (2)	0.073 (2)	0.069 (2)	0.0107 (18)	0.0001 (18)	0.0132 (19)
C13	0.058 (2)	0.0492 (19)	0.071 (2)	0.0044 (17)	−0.0144 (18)	−0.0059 (17)
C12	0.101 (3)	0.046 (2)	0.110 (3)	0.006 (2)	−0.029 (3)	−0.016 (2)
C28	0.054 (2)	0.0497 (19)	0.078 (2)	0.0084 (17)	0.0094 (19)	−0.0071 (18)
C31	0.049 (2)	0.073 (2)	0.0522 (19)	0.0042 (18)	−0.0030 (16)	−0.0184 (17)
C10	0.058 (2)	0.079 (3)	0.0494 (19)	−0.024 (2)	−0.0110 (17)	0.0016 (18)
C24	0.055 (2)	0.060 (2)	0.095 (3)	−0.0007 (18)	−0.025 (2)	0.013 (2)
C17	0.071 (3)	0.108 (3)	0.061 (2)	0.042 (3)	0.005 (2)	−0.007 (2)
C19	0.056 (2)	0.063 (2)	0.0533 (19)	0.0029 (18)	0.0134 (16)	0.0000 (16)
C4	0.064 (3)	0.087 (3)	0.105 (3)	0.010 (3)	−0.025 (2)	−0.028 (3)
C2	0.049 (2)	0.066 (2)	0.0495 (18)	−0.0003 (17)	−0.0147 (16)	−0.0180 (17)
C42	0.083 (3)	0.056 (2)	0.053 (2)	0.0124 (19)	0.0149 (18)	−0.0098 (17)
C30	0.047 (2)	0.096 (3)	0.054 (2)	0.000 (2)	−0.0100 (16)	−0.009 (2)
C22	0.095 (3)	0.094 (3)	0.044 (2)	−0.021 (3)	−0.013 (2)	0.0176 (19)
C18	0.057 (3)	0.115 (3)	0.057 (2)	0.015 (2)	0.0179 (18)	0.000 (2)
C7	0.066 (3)	0.081 (3)	0.063 (2)	−0.012 (2)	−0.0185 (19)	−0.010 (2)
C16	0.081 (3)	0.064 (2)	0.071 (2)	0.033 (2)	−0.011 (2)	−0.0080 (19)
C5	0.084 (4)	0.076 (3)	0.116 (4)	0.010 (3)	−0.047 (3)	−0.022 (3)
C6	0.095 (4)	0.064 (3)	0.100 (3)	−0.017 (3)	−0.039 (3)	−0.004 (2)
C23	0.082 (3)	0.086 (3)	0.069 (3)	−0.016 (2)	−0.031 (2)	0.028 (2)
C11	0.100 (3)	0.060 (2)	0.085 (3)	−0.025 (2)	−0.019 (2)	−0.013 (2)

Geometric parameters (Å, °)

Ni—S1	2.2027 (8)	C36—C35	1.374 (5)
Ni—S2	2.2147 (8)	C36—H36	0.93
Ni—P1	2.2227 (8)	C20—C21	1.396 (4)
Ni—P2	2.2564 (8)	C35—C34	1.363 (5)
P2—C26	1.824 (3)	C35—H35	0.93
P2—C38	1.836 (3)	C21—C22	1.369 (5)
P2—C32	1.836 (3)	C21—H21	0.93
S1—C1	1.732 (3)	C34—C33	1.379 (4)
S2—C1	1.722 (3)	C34—H34	0.93
P1—C14	1.822 (3)	C33—H33	0.93
P1—C8	1.827 (3)	C40—H40	0.93
P1—C20	1.827 (3)	C3—C4	1.376 (5)
S3—O2	1.425 (2)	C3—C2	1.394 (5)
S3—O1	1.440 (3)	C29—C28	1.360 (5)
S3—N	1.626 (3)	C29—C30	1.373 (5)
S3—C2	1.779 (3)	C29—H29	0.93
Cl1—C3	1.731 (4)	C13—C12	1.377 (5)
Cl2—C6	1.732 (5)	C13—H13	0.93
N—C1	1.303 (3)	C12—C11	1.366 (6)
C26—C27	1.380 (4)	C12—H12	0.93
C26—C31	1.394 (4)	C28—H28	0.93
C37—C36	1.380 (4)	C31—C30	1.376 (5)
C37—C32	1.384 (4)	C31—H31	0.93

C37—H37	0.93	C10—C11	1.348 (5)
C14—C15	1.386 (4)	C10—H10	0.93
C14—C19	1.390 (4)	C24—C23	1.365 (5)
C27—C28	1.388 (5)	C24—H24	0.93
C27—H27	0.93	C17—C16	1.367 (6)
C15—C16	1.391 (5)	C17—C18	1.369 (5)
C15—H15	0.93	C17—H17	0.93
C8—C13	1.378 (4)	C19—C18	1.381 (5)
C8—C9	1.390 (4)	C19—H19	0.93
C32—C33	1.387 (4)	C4—C5	1.391 (6)
C39—C40	1.374 (4)	C4—H4	0.93
C39—C38	1.398 (4)	C2—C7	1.382 (5)
C39—H39	0.93	C42—H42	0.93
C38—C43	1.391 (4)	C30—H30	0.93
C9—C10	1.377 (4)	C22—C23	1.360 (6)
C9—H9	0.93	C22—H22	0.93
C25—C20	1.382 (4)	C18—H18	0.93
C25—C24	1.390 (4)	C7—C6	1.387 (5)
C25—H25	0.93	C7—H7	0.93
C43—C42	1.390 (4)	C16—H16	0.93
C43—H43	0.93	C5—C6	1.363 (6)
C41—C42	1.372 (5)	C5—H5	0.93
C41—C40	1.380 (5)	C23—H23	0.93
C41—H41	0.93	C11—H11	0.93
S1—Ni—S2	77.09 (3)	C22—C21—C20	120.0 (4)
S1—Ni—P1	89.89 (3)	C22—C21—H21	120
S2—Ni—P1	166.98 (3)	C20—C21—H21	120
S1—Ni—P2	167.01 (3)	C35—C34—C33	120.3 (4)
S2—Ni—P2	90.00 (3)	C35—C34—H34	119.9
P1—Ni—P2	103.02 (3)	C33—C34—H34	119.9
C26—P2—C38	103.50 (13)	C34—C33—C32	120.6 (3)
C26—P2—C32	103.53 (14)	C34—C33—H33	119.7
C38—P2—C32	103.90 (12)	C32—C33—H33	119.7
C26—P2—Ni	113.22 (9)	C39—C40—C41	120.6 (3)
C38—P2—Ni	122.64 (10)	C39—C40—H40	119.7
C32—P2—Ni	108.16 (9)	C41—C40—H40	119.7
C1—S1—Ni	88.66 (10)	C4—C3—C2	120.6 (4)
C1—S2—Ni	88.53 (10)	C4—C3—Cl1	117.2 (3)
C14—P1—C8	103.64 (14)	C2—C3—Cl1	122.2 (3)
C14—P1—C20	106.85 (13)	C28—C29—C30	119.6 (3)
C8—P1—C20	102.70 (13)	C28—C29—H29	120.2
C14—P1—Ni	114.43 (9)	C30—C29—H29	120.2
C8—P1—Ni	113.12 (9)	C12—C13—C8	120.1 (3)
C20—P1—Ni	114.85 (10)	C12—C13—H13	120
O2—S3—O1	116.51 (15)	C8—C13—H13	120
O2—S3—N	107.03 (15)	C11—C12—C13	120.9 (4)
O1—S3—N	113.89 (13)	C11—C12—H12	119.6
O2—S3—C2	108.00 (15)	C13—C12—H12	119.6
O1—S3—C2	106.50 (16)	C29—C28—C27	120.6 (3)

N—S3—C2	104.08 (14)	C29—C28—H28	119.7
C1—N—S3	120.5 (2)	C27—C28—H28	119.7
C27—C26—C31	118.4 (3)	C30—C31—C26	120.4 (3)
C27—C26—P2	119.2 (2)	C30—C31—H31	119.8
C31—C26—P2	122.2 (2)	C26—C31—H31	119.8
C36—C37—C32	120.0 (3)	C11—C10—C9	120.4 (3)
C36—C37—H37	120	C11—C10—H10	119.8
C32—C37—H37	120	C9—C10—H10	119.8
C15—C14—C19	118.7 (3)	C23—C24—C25	119.9 (4)
C15—C14—P1	119.1 (2)	C23—C24—H24	120
C19—C14—P1	122.2 (2)	C25—C24—H24	120
C26—C27—C28	120.4 (3)	C16—C17—C18	119.8 (4)
C26—C27—H27	119.8	C16—C17—H17	120.1
C28—C27—H27	119.8	C18—C17—H17	120.1
N—C1—S2	121.8 (2)	C18—C19—C14	120.6 (3)
N—C1—S1	132.6 (2)	C18—C19—H19	119.7
S2—C1—S1	105.68 (15)	C14—C19—H19	119.7
C14—C15—C16	119.8 (3)	C3—C4—C5	119.3 (4)
C14—C15—H15	120.1	C3—C4—H4	120.4
C16—C15—H15	120.1	C5—C4—H4	120.4
C13—C8—C9	118.1 (3)	C7—C2—C3	119.4 (3)
C13—C8—P1	121.4 (2)	C7—C2—S3	117.5 (3)
C9—C8—P1	120.1 (2)	C3—C2—S3	123.1 (3)
C37—C32—C33	118.7 (3)	C41—C42—C43	120.5 (3)
C37—C32—P2	121.6 (2)	C41—C42—H42	119.7
C33—C32—P2	119.5 (2)	C43—C42—H42	119.7
C40—C39—C38	120.5 (3)	C29—C30—C31	120.6 (3)
C40—C39—H39	119.8	C29—C30—H30	119.7
C38—C39—H39	119.7	C31—C30—H30	119.7
C43—C38—C39	118.5 (3)	C23—C22—C21	121.0 (4)
C43—C38—P2	121.7 (2)	C23—C22—H22	119.5
C39—C38—P2	119.7 (2)	C21—C22—H22	119.5
C10—C9—C8	120.7 (3)	C17—C18—C19	120.3 (4)
C10—C9—H9	119.6	C17—C18—H18	119.9
C8—C9—H9	119.6	C19—C18—H18	119.9
C20—C25—C24	120.3 (3)	C2—C7—C6	119.6 (4)
C20—C25—H25	119.9	C2—C7—H7	120.2
C24—C25—H25	119.9	C6—C7—H7	120.2
C42—C43—C38	120.2 (3)	C17—C16—C15	120.8 (3)
C42—C43—H43	119.9	C17—C16—H16	119.6
C38—C43—H43	119.9	C15—C16—H16	119.6
C42—C41—C40	119.6 (3)	C6—C5—C4	120.4 (4)
C42—C41—H41	120.2	C6—C5—H5	119.8
C40—C41—H41	120.2	C4—C5—H5	119.8
C35—C36—C37	120.6 (3)	C5—C6—C7	120.6 (4)
C35—C36—H36	119.7	C5—C6—Cl2	120.1 (4)
C37—C36—H36	119.7	C7—C6—Cl2	119.2 (4)
C25—C20—C21	118.6 (3)	C22—C23—C24	120.1 (4)
C25—C20—P1	117.7 (2)	C22—C23—H23	119.9

C21—C20—P1	123.7 (2)	C24—C23—H23	119.9
C34—C35—C36	119.8 (3)	C10—C11—C12	119.8 (3)
C34—C35—H35	120.1	C10—C11—H11	120.1
C36—C35—H35	120.1	C12—C11—H11	120.1
S1—Ni—P2—C26	78.94 (17)	C26—P2—C38—C39	48.7 (3)
S2—Ni—P2—C26	85.25 (10)	C32—P2—C38—C39	156.6 (2)
P1—Ni—P2—C26	−94.30 (10)	Ni—P2—C38—C39	−80.7 (3)
S1—Ni—P2—C38	−155.86 (16)	C13—C8—C9—C10	−0.9 (4)
S2—Ni—P2—C38	−149.54 (12)	P1—C8—C9—C10	−173.6 (2)
P1—Ni—P2—C38	30.90 (12)	C39—C38—C43—C42	−0.1 (5)
S1—Ni—P2—C32	−35.17 (19)	P2—C38—C43—C42	−179.3 (3)
S2—Ni—P2—C32	−28.86 (11)	C32—C37—C36—C35	−0.6 (5)
P1—Ni—P2—C32	151.58 (10)	C24—C25—C20—C21	0.1 (4)
S2—Ni—S1—C1	−1.55 (9)	C24—C25—C20—P1	−179.3 (2)
P1—Ni—S1—C1	178.34 (9)	C14—P1—C20—C25	163.2 (2)
P2—Ni—S1—C1	4.93 (18)	C8—P1—C20—C25	−88.1 (2)
S1—Ni—S2—C1	1.56 (10)	Ni—P1—C20—C25	35.1 (3)
P1—Ni—S2—C1	1.09 (19)	C14—P1—C20—C21	−16.1 (3)
P2—Ni—S2—C1	−176.99 (10)	C8—P1—C20—C21	92.6 (3)
S1—Ni—P1—C14	103.14 (10)	Ni—P1—C20—C21	−144.2 (2)
S2—Ni—P1—C14	103.59 (18)	C37—C36—C35—C34	0.6 (5)
P2—Ni—P1—C14	−78.38 (10)	C25—C20—C21—C22	−0.6 (5)
S1—Ni—P1—C8	−15.26 (11)	P1—C20—C21—C22	178.7 (3)
S2—Ni—P1—C8	−14.8 (2)	C36—C35—C34—C33	0.5 (5)
P2—Ni—P1—C8	163.23 (11)	C35—C34—C33—C32	−1.6 (4)
S1—Ni—P1—C20	−132.72 (10)	C37—C32—C33—C34	1.6 (4)
S2—Ni—P1—C20	−132.27 (17)	P2—C32—C33—C34	176.0 (2)
P2—Ni—P1—C20	45.76 (11)	C38—C39—C40—C41	0.2 (5)
O2—S3—N—C1	−160.7 (2)	C42—C41—C40—C39	−0.1 (6)
O1—S3—N—C1	−30.4 (3)	C9—C8—C13—C12	−0.1 (5)
C2—S3—N—C1	85.1 (3)	P1—C8—C13—C12	172.5 (3)
C38—P2—C26—C27	−159.0 (2)	C8—C13—C12—C11	0.9 (6)
C32—P2—C26—C27	92.9 (2)	C30—C29—C28—C27	1.5 (5)
Ni—P2—C26—C27	−24.0 (3)	C26—C27—C28—C29	−1.4 (5)
C38—P2—C26—C31	26.1 (3)	C27—C26—C31—C30	0.8 (5)
C32—P2—C26—C31	−82.1 (3)	P2—C26—C31—C30	175.8 (3)
Ni—P2—C26—C31	161.0 (2)	C8—C9—C10—C11	1.2 (5)
C8—P1—C14—C15	146.1 (2)	C20—C25—C24—C23	0.4 (5)
C20—P1—C14—C15	−105.8 (2)	C15—C14—C19—C18	0.8 (5)
Ni—P1—C14—C15	22.5 (3)	P1—C14—C19—C18	−179.8 (3)
C8—P1—C14—C19	−33.3 (3)	C2—C3—C4—C5	2.5 (6)
C20—P1—C14—C19	74.8 (3)	Cl1—C3—C4—C5	−177.4 (3)
Ni—P1—C14—C19	−156.9 (2)	C4—C3—C2—C7	−2.9 (5)
C31—C26—C27—C28	0.2 (4)	Cl1—C3—C2—C7	176.9 (2)
P2—C26—C27—C28	−175.0 (2)	C4—C3—C2—S3	175.2 (3)
S3—N—C1—S2	−178.96 (15)	Cl1—C3—C2—S3	−5.0 (4)
S3—N—C1—S1	0.3 (4)	O2—S3—C2—C7	132.2 (3)
Ni—S2—C1—N	177.4 (2)	O1—S3—C2—C7	6.4 (3)
Ni—S2—C1—S1	−2.01 (12)	N—S3—C2—C7	−114.3 (2)

Ni—S1—C1—N	−177.3 (3)	O2—S3—C2—C3	−45.9 (3)
Ni—S1—C1—S2	2.02 (12)	O1—S3—C2—C3	−171.8 (3)
C19—C14—C15—C16	−2.2 (5)	N—S3—C2—C3	67.6 (3)
P1—C14—C15—C16	178.4 (2)	C40—C41—C42—C43	−0.1 (6)
C14—P1—C8—C13	143.4 (3)	C38—C43—C42—C41	0.2 (5)
C20—P1—C8—C13	32.3 (3)	C28—C29—C30—C31	−0.6 (5)
Ni—P1—C8—C13	−92.1 (2)	C26—C31—C30—C29	−0.6 (5)
C14—P1—C8—C9	−44.1 (3)	C20—C21—C22—C23	0.7 (6)
C20—P1—C8—C9	−155.2 (2)	C16—C17—C18—C19	−0.8 (6)
Ni—P1—C8—C9	80.4 (2)	C14—C19—C18—C17	0.6 (6)
C36—C37—C32—C33	−0.5 (4)	C3—C2—C7—C6	1.3 (5)
C36—C37—C32—P2	−174.8 (2)	S3—C2—C7—C6	−176.9 (3)
C26—P2—C32—C37	−0.5 (3)	C18—C17—C16—C15	−0.6 (6)
C38—P2—C32—C37	−108.4 (2)	C14—C15—C16—C17	2.1 (5)
Ni—P2—C32—C37	119.9 (2)	C3—C4—C5—C6	−0.5 (6)
C26—P2—C32—C33	−174.7 (2)	C4—C5—C6—C7	−1.1 (6)
C38—P2—C32—C33	77.4 (2)	C4—C5—C6—Cl2	178.8 (3)
Ni—P2—C32—C33	−54.4 (2)	C2—C7—C6—C5	0.7 (6)
C40—C39—C38—C43	−0.1 (5)	C2—C7—C6—Cl2	−179.3 (3)
C40—C39—C38—P2	179.1 (3)	C21—C22—C23—C24	−0.2 (6)
C26—P2—C38—C43	−132.1 (3)	C25—C24—C23—C22	−0.3 (6)
C32—P2—C38—C43	−24.2 (3)	C9—C10—C11—C12	−0.4 (6)
Ni—P2—C38—C43	98.5 (2)	C13—C12—C11—C10	−0.6 (7)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C36—H36···S1^{i}	0.93	2.8	3.641 (3)	151
C34—H34···O1^{ii}	0.93	2.57	3.194 (5)	125
C41—H41···O2^{iii}	0.93	2.48	3.197 (4)	134

Symmetry codes: (i) −*x*−1/2, *y*−1/2, *z*; (ii) −*x*, *y*−1/2, −*z*+1/2; (iii) *x*, −*y*+1/2, *z*−1/2.

Fig. 1

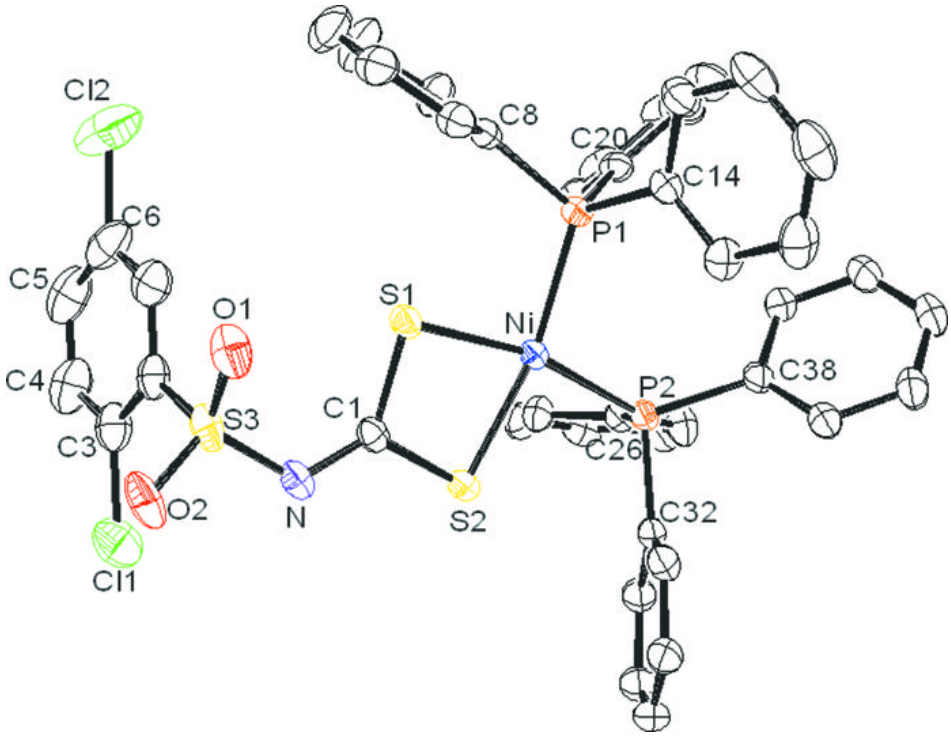
Cl2
C8
C20
C14
C6
P1
C5
S1
Ni
O1
C4
P2
C38
S3
C1
C3
C26
O2
N
S2
C32
Cl1

Fig. 2