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Chlorido(1-pyrrolidinecarbodithioato-κ^2S,S')(triphenylphosphine-κP)nickel(II) chloroform hemisolvate

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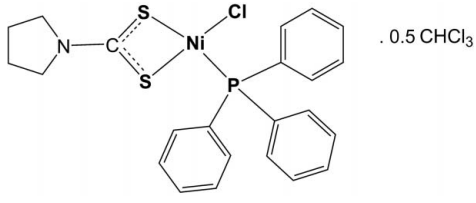
Key indicators: single-crystal X-ray study; T = 295 K; mean σ(C–C) = 0.005 Å; disorder in solvent or counterion; R factor = 0.058; wR factor = 0.119; data-to-parameter ratio = 21.2.

In the crystal structure of the title complex, $[Ni\{S_2CN(CH_2)_4\}Cl(C_{18}H_{15}P)]\cdot 0.5CHCl_3$, the Ni atom is coordinated by a bidentate dithiocarbamate, one chloride and triphenylphosphine in a square-planar arrangement. The chloroform solvent molecule interacts with the complex through a weak C—H···S hydrogen bond. The solvent molecule is disordered equally over two inversion-related sites.

Related literature

For related literature, see: Allen (2002); Garton *et al.* (1963); Kropidłowska *et al.* (2007); Pastorek *et al.* (1996, 1999); Venanzi (1958).



Experimental

Crystal data

$[Ni(C_5H_8NS_2)Cl(C_{18}H_{15}P)]\cdot 0.5CHCl_3$
M_r = 1124.72
Triclinic, $P\overline{1}$
a = 9.713 (2) Å
b = 10.076 (2) Å
c = 14.509 (3) Å
α = 90.37 (2)°
β = 91.21 (2)°
γ = 117.39 (2)°
V = 1260.3 (5) Å^3
Z = 1
Mo $K\alpha$ radiation
μ = 1.28 mm^{-1}
T = 295 (2) K
0.25 × 0.22 × 0.18 mm

Data collection

Kuma KM-4 with CCD area detector diffractometer
Absorption correction: analytical (face-indexed) (*SHELXTL*; Sheldrick, 1990)
T_{min} = 0.741, T_{max} = 0.799
16395 measured reflections
6060 independent reflections
3417 reflections with $I > 2\sigma(I)$
R_{int} = 0.050

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.058
$wR(F^2)$ = 0.119
S = 1.01
6060 reflections
286 parameters
6 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.48 e Å^{-3}
$\Delta\rho_{min}$ = −0.51 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C37—H37···S1	0.96	2.40	3.280 (1)	151.6

Data collection: *CrysAlisCCD* (Oxford Diffraction, 2005); cell refinement: *CrysAlisCCD*; data reduction: *CrysAlisRED* (Oxford Diffraction, 2005); 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: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: IM2018).

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supplementary materials

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Chlorido(1-pyrrolidinecarbodithioato-$\kappa^2 S,S'$)(triphenylphosphine-κP)nickel(II) chloroform hemisolvate

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Comment

The 1-Pyrrolidinecarbodithioato-group is one of the most frequently used sulfur donor ligands and structural data for nearly 100 complexes are stored in the Cambridge Structural Database (CSD-2007, Allen 2002). Recently, we reported the synthesis of the tris(1-pyrrolidinylcarbodithioato-*S,S'*)-cobalt(III) complex obtained as chloroform disolvate $[Co(S_2CN(CH_2)_4)_3] \times 2CHCl_3$ (I) (Kropidłowska *et al.* 2007). The most notable feature of its structure was the apparent interaction of $CHCl_3$ with a sulfur atom from the ligand (Cl_3C—H···S).

In the present paper we describe the structure of a nickel(II) complex - chlorido(1-pyrrolidinecarbodithioato-*S,S'*)(triphenylphosphine)nickel(II) (**I**) which was isolated as a chloroform hemisolvate. It can be regarded as a dithiocarbamate complex with a four-coordinated metal(II) ion within a square planar, heterogeneous $[NiClS_2P]$ coordination sphere. There are only small deviations from planarity, not exceeding 0.07 Å. The molecular structure of (**I**) with atom numbering scheme is shown in Fig.1. The solvating chloroform molecule is equally disordered over two sites related by an inversion center. Again, the interaction of $CHCl_3$ with the complex seems to be present (Cl_3C—H···S distance equals 2.4 Å) and one can suppose that weak C—H···S hydrogen bond is formed, somewhat reinforcing the structure (Fig. 2). Short Cl1···S2 (3.341 Å) as well as some short C_{ar}H···Cl (*ca* 2.93 Å) contacts may be also noticed.

The structures of two similar hemisolvated nickel(II) complexes have been reported previously: homologous bromido(1-pyrrolidinylcarbodithioato-*S,S'*)(triphenylphosphine)nickel(II) (refcode TIZJAN, Pastorek *et al.*, 1996) and closely related thiazolidinedithiocarbamate with nickel(II) bonded to triphenylphosphine and chloride ligands (refcode GOZBUS, Pastorek *et al.*, 1999). However, in both cases, the Cl_3C—H···S distance is greater than 2.7 Å and even speculations about the existence of any weak hydrogen bond seem doubtful.

Experimental

Nickel chloride, $NiCl_2 \times 6H_2O$ (0.594 g, 0.0025 mol, purchased from POCh) was dissolved in 50 ml of methanol/water (10/1, *v*/*v*) and this solution was added dropwise to the ammonium salt of pyrrolidinecarbodithioic acid $C_4H_8NCS_2NH_4$ (0.82 g, 0.005 mol, Fluka) dissolved in methanol/water. This mixture was stirred vigorously under argon atmosphere for *app.* 20 minutes, then filtered and the filtrate left for crystallization at 5°C. After a week the green crystalline product $Ni(S_2CNC_4H_8)_2$ was collected. It was further dissolved (0.199 g, 0.00057 mol) in 10 ml of chloroform and mixed with solution of equimolar amount of $NiCl_2(PPh_3)_2$ (0.37 g) prepared as described in the literature (Venanzi, 1958; Garton *et al.* 1963). The mixture which turned into deep violet color, was stirred for 10 minutes and then filtered. To this solution 10 ml of Et_2O was added. After dwo days purple crystals were collected and washed with several portions of ether.

Refinement

All H atoms were positioned geometrically and treated as riding with U_{iso}(H) values of 1.5U_{eq} of the C joined directly the H or 1.2U_{eq} of the C joined H for aromatic. The solvated molecule of $CHCl_3$ is statistically disordered (occupation factor of 1/2).

Figures

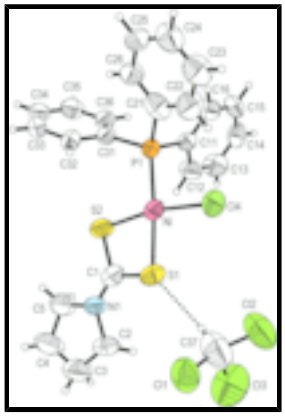


Fig. 1. Molecular structure and atom-numbering scheme for (**I**) with displacement ellipsoids drawn at 50% probability level. The chloroform molecule is disordered - only one set is shown. Broken line denotes the assumed C—H···S hydrogen bond. Color codes: Ni pink-red, N blue, S yellow, Cl green, P orange, C gray. H atoms are shown as small white rings of arbitrary size.

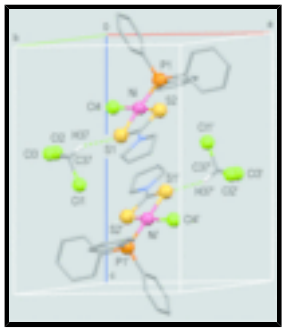


Fig. 2. Schematic drawing of the crystal packing of molecules of (**I**) showing the C—H···S interactions. Besides chloroform, all other H atoms have been omitted for clarity.

Chlorido(1-pyrrolidinecarbodithioato-κ^2*S*,*S*')(triphenylphosphine- κ*P*)nickel(II) chloroform hemisolvate

Crystal data

$[Ni(C_5H_8NS_2)Cl(C_{18}H_{15}P)]\cdot 0.5CHCl_3$	$Z = 1$
$M_r = 1124.72$	$F_{000} = 578$
	$D_x = 1.482$ Mg m^{-3}
Triclinic, $P\overline{1}$	$D_m = 1.48$ Mg m^{-3}
	D_m measured by floatation
	Mo $K\alpha$ radiation
Hall symbol: -P 1	λ = 0.71073 Å
$a = 9.713$ (2) Å	Cell parameters from 876 reflections
$b = 10.076$ (2) Å	θ = 2.7–28.0º
$c = 14.509$ (3) Å	μ = 1.28 mm^{-1}
α = 90.37 (2)º	$T = 295$ (2) K

$\beta = 91.21\ (2)^{\circ}$	Paralellepiped, purple
$\gamma = 117.39\ (2)^{\circ}$	0.25 × 0.22 × 0.18 mm
$V = 1260.3\ (5)\ Å^3$	

Data collection

Kuma KM-4 with CCD area detector diffractometer	6060 independent reflections
Radiation source: fine-focus sealed tube	3417 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.050$
Detector resolution: 1024x1024 with blocks 2x2 pixels mm^{-1}	$\theta_{max} = 28.0^{\circ}$
$T = 295(2)$ K	$\theta_{min} = 2.7^{\circ}$
ω scans	$h = -12 \rightarrow 12$
Absorption correction: analytical (face-indexed) (SHELXTL; Sheldrick, 1990)	$k = -13 \rightarrow 10$
$T_{min} = 0.741$, $T_{max} = 0.799$	$l = -18 \rightarrow 19$
16395 measured reflections	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.058$	H-atom parameters constrained
$wR(F^2) = 0.119$	$w = 1/[\sigma^2(F_o^2) + (0.0351P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.01$	$(\Delta/\sigma)_{max} = 0.006$
6060 reflections	$\Delta\rho_{max} = 0.48$ e $Å^{-3}$
286 parameters	$\Delta\rho_{min} = -0.51$ e $Å^{-3}$
6 restraints	Extinction correction: none
Primary atom site location: structure-invariant direct methods	

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}	Occ. (<1)
Cl4	0.11616 (10)	0.13720 (11)	0.28665 (7)	0.0926 (4)	
Ni	0.36198 (4)	0.29476 (4)	0.28472 (3)	0.05004 (11)	
S1	0.33333 (9)	0.44183 (9)	0.38849 (6)	0.0619 (3)	
S2	0.59132 (9)	0.48808 (9)	0.28702 (6)	0.0633 (3)	
C1	0.5177 (3)	0.5698 (3)	0.36361 (19)	0.0538 (9)	
N1	0.5905 (3)	0.7057 (3)	0.39337 (17)	0.0562 (8)	
C2	0.5209 (4)	0.7713 (3)	0.4563 (2)	0.0702 (11)	
H2A	0.4920	0.7164	0.5133	0.105*	
H2B	0.4297	0.7717	0.4280	0.105*	
C3	0.6481 (4)	0.9290 (4)	0.4737 (3)	0.1076 (17)	
H3A	0.6210	1.0002	0.4442	0.161*	
H3B	0.6629	0.9515	0.5393	0.161*	
C4	0.7866 (5)	0.9375 (5)	0.4359 (4)	0.109 (2)	
H4A	0.8544	0.9355	0.4853	0.163*	
H4B	0.8408	1.0309	0.4037	0.163*	
C5	0.7491 (4)	0.8119 (4)	0.3719 (3)	0.0829 (13)	
H5A	0.7556	0.8435	0.3084	0.124*	
H5B	0.8189	0.7684	0.3818	0.124*	
P1	0.41303 (8)	0.16922 (8)	0.17730 (5)	0.0439 (2)	
C11	0.3175 (3)	0.1762 (3)	0.07035 (19)	0.0436 (8)	
C12	0.3251 (3)	0.3130 (3)	0.0462 (2)	0.0630 (10)	
H12	0.3753	0.3959	0.0853	0.076*	
C13	0.2575 (4)	0.3253 (4)	−0.0365 (2)	0.0738 (11)	
H13	0.2654	0.4174	−0.0533	0.089*	
C14	0.1803 (3)	0.2046 (4)	−0.0929 (2)	0.0673 (11)	
H14	0.1322	0.2133	−0.1470	0.081*	
C15	0.1729 (3)	0.0681 (3)	−0.0701 (2)	0.0623 (11)	
H15	0.1223	−0.0143	−0.1096	0.075*	
C16	0.2407 (3)	0.0552 (3)	0.0110 (2)	0.0588 (10)	
H16	0.2348	−0.0367	0.0262	0.071*	
C21	0.3574 (3)	−0.0297 (3)	0.1936 (2)	0.0686 (9)	
C22	0.2114 (4)	−0.1258 (4)	0.2214 (3)	0.0904 (14)	
H22	0.1417	−0.0890	0.2340	0.109*	
C23	0.1652 (5)	−0.2791 (4)	0.2311 (3)	0.0955 (17)	
H23	0.0650	−0.3442	0.2483	0.127*	
C24	0.2698 (4)	−0.3304 (4)	0.2147 (3)	0.0820 (13)	
H24	0.2417	−0.4311	0.2227	0.098*	
C25	0.4138 (4)	−0.2375 (3)	0.1870 (2)	0.0745 (11)	
H25	0.4831	−0.2748	0.1746	0.089*	
C26	0.4585 (4)	−0.0872 (3)	0.1769 (2)	0.0614 (10)	
H26	0.5586	−0.0239	0.1586	0.074*	
C31	0.6157 (3)	0.2440 (3)	0.14972 (19)	0.0426 (8)	
C32	0.7206 (3)	0.2612 (3)	0.2213 (2)	0.0549 (9)	
H32	0.6872	0.2443	0.2817	0.066*	
C33	0.8749 (3)	0.3035 (3)	0.2029 (3)	0.0695 (11)	

H33	0.9440	0.3112	0.2505	0.083*	
C34	0.9246 (4)	0.3338 (3)	0.1144 (2)	0.0686 (11)	
H34	1.0283	0.3636	0.1026	0.082*	
C35	0.8257 (4)	0.3211 (3)	0.0435 (2)	0.0664 (11)	
H35	0.8612	0.3434	−0.0163	0.080*	
C36	0.67116 (16)	0.27453 (16)	0.06166 (10)	0.0518 (9)	
H36	0.6026	0.2635	0.0130	0.062*	
Cl1	0.12808 (9)	0.52365 (14)	0.58149 (7)	0.1235 (10)	0.50
Cl2	−0.11177 (9)	0.34498 (14)	0.45802 (7)	0.1810 (16)	0.50
Cl3	0.02592 (9)	0.65936 (14)	0.44795 (7)	0.1659 (18)	0.50
C37	0.05796 (9)	0.50814 (14)	0.46966 (7)	0.143 (5)	0.50
H37	0.1324	0.5067	0.4278	0.207*	0.50

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl4	0.0502 (5)	0.1054 (7)	0.1103 (8)	0.0253 (5)	0.0148 (5)	−0.0215 (6)
Ni	0.04922 (18)	0.0602 (2)	0.0474 (2)	0.03066 (15)	0.00490 (18)	0.00314 (17)
S1	0.0667 (4)	0.0665 (5)	0.0618 (5)	0.0378 (4)	0.0182 (4)	0.0040 (4)
S2	0.0594 (4)	0.0589 (4)	0.0717 (6)	0.0267 (4)	0.0177 (4)	−0.0071 (4)
C1	0.0638 (16)	0.0658 (17)	0.0437 (17)	0.0397 (13)	0.0075 (15)	0.0105 (14)
N1	0.0593 (13)	0.0519 (13)	0.0588 (16)	0.0264 (11)	0.0103 (13)	−0.0072 (12)
C2	0.089 (2)	0.0697 (18)	0.066 (2)	0.0492 (15)	0.0069 (19)	−0.0092 (17)
C3	0.099 (3)	0.077 (2)	0.137 (4)	0.033 (2)	0.009 (3)	−0.040 (3)
C4	0.097 (3)	0.088 (3)	0.100 (5)	0.021 (3)	0.011 (4)	−0.041 (3)
C5	0.074 (2)	0.069 (2)	0.093 (3)	0.0221 (19)	0.018 (2)	−0.015 (2)
P1	0.0406 (4)	0.0461 (4)	0.0462 (5)	0.0211 (3)	0.0014 (4)	0.0024 (4)
C11	0.0477 (13)	0.0398 (13)	0.0473 (17)	0.0234 (11)	0.0050 (14)	0.0080 (13)
C12	0.0791 (19)	0.0551 (17)	0.061 (2)	0.0369 (15)	−0.0110 (18)	−0.0067 (16)
C13	0.092 (2)	0.0659 (19)	0.073 (2)	0.0442 (17)	−0.004 (2)	0.0112 (18)
C14	0.0557 (17)	0.088 (2)	0.060 (2)	0.0352 (16)	−0.0135 (17)	0.0069 (18)
C15	0.0595 (19)	0.0636 (19)	0.0527 (19)	0.0196 (16)	−0.0157 (17)	−0.0104 (16)
C16	0.0646 (18)	0.0550 (17)	0.057 (2)	0.0276 (14)	0.0001 (17)	−0.0002 (15)
C21	0.0743 (15)	0.0629 (15)	0.0697 (18)	0.0135 (12)	−0.0012 (15)	−0.0007 (14)
C22	0.067 (2)	0.065 (2)	0.128 (3)	0.0201 (19)	0.024 (2)	0.017 (2)
C23	0.083 (3)	0.062 (2)	0.118 (4)	0.013 (2)	0.017 (3)	0.020 (3)
C24	0.090 (3)	0.0528 (19)	0.092 (3)	0.0238 (18)	−0.019 (2)	0.0194 (19)
C25	0.0838 (19)	0.0535 (17)	0.100 (3)	0.0443 (14)	−0.022 (2)	0.0027 (18)
C26	0.0631 (19)	0.0474 (17)	0.071 (2)	0.0230 (15)	0.0043 (18)	0.0076 (16)
C31	0.0401 (13)	0.0333 (13)	0.0523 (18)	0.0149 (11)	0.0055 (14)	0.0033 (13)
C32	0.0430 (14)	0.0623 (17)	0.062 (2)	0.0268 (13)	0.0016 (16)	−0.0041 (16)
C33	0.0470 (17)	0.068 (2)	0.090 (3)	0.0244 (15)	−0.0150 (19)	−0.0247 (19)
C34	0.0484 (17)	0.0573 (18)	0.093 (3)	0.0183 (14)	0.0086 (19)	−0.0264 (19)
C35	0.0627 (19)	0.0535 (18)	0.073 (2)	0.0175 (16)	0.0182 (19)	−0.0055 (17)
C36	0.0501 (16)	0.0440 (15)	0.0590 (19)	0.0196 (13)	0.0050 (16)	0.0014 (14)
Cl1	0.0964 (16)	0.181 (2)	0.0792 (15)	0.0526 (16)	−0.0068 (13)	−0.0183 (15)
Cl2	0.125 (2)	0.195 (3)	0.172 (3)	0.033 (2)	−0.032 (2)	−0.089 (2)
Cl3	0.176 (3)	0.165 (3)	0.176 (5)	0.105 (2)	0.013 (3)	0.074 (3)

C37	0.146 (9)	0.103 (6)	0.173 (10)	0.055 (6)	−0.073 (7)	−0.021 (6)

Geometric parameters (Å, °)

Cl4—Ni	2.1763 (11)	C14—H14	0.9300
Ni—S2	2.1820 (11)	C15—C16	1.371 (4)
Ni—P1	2.2045 (10)	C15—H15	0.9300
Ni—S1	2.2155 (11)	C16—H16	0.9300
S1—C1	1.705 (3)	C21—C22	1.368 (4)
S2—C1	1.727 (3)	C21—C26	1.375 (4)
C1—N1	1.285 (3)	C22—C23	1.408 (5)
N1—C5	1.456 (4)	C22—H22	0.9300
N1—C2	1.470 (4)	C23—C24	1.359 (6)
C2—C3	1.518 (4)	C23—H23	0.9300
C2—H2A	0.9700	C24—C25	1.347 (5)
C2—H2B	0.9700	C24—H24	0.9300
C3—C4	1.425 (6)	C25—C26	1.381 (4)
C3—H3A	0.9700	C25—H25	0.9300
C3—H3B	0.9700	C26—H26	0.9300
C4—C5	1.466 (5)	C31—C36	1.378 (3)
C4—H4A	0.9700	C31—C32	1.391 (4)
C4—H4B	0.9700	C32—C33	1.387 (4)
C5—H5A	0.9700	C32—H32	0.9300
C5—H5B	0.9700	C33—C34	1.367 (5)
P1—C31	1.811 (3)	C33—H33	0.9300
P1—C11	1.811 (3)	C34—C35	1.357 (5)
P1—C21	1.839 (3)	C34—H34	0.9300
C11—C16	1.379 (4)	C35—C36	1.385 (3)
C11—C12	1.394 (4)	C35—H35	0.9300
C12—C13	1.390 (4)	C36—H36	0.9300
C12—H12	0.9300	Cl1—C37	1.7244
C13—C14	1.353 (4)	Cl2—C37	1.7146
C13—H13	0.9300	Cl3—C37	1.7193
C14—C15	1.385 (4)	C37—H37	0.9600
Cl4—Ni—S2	167.86 (5)	C12—C13—H13	119.6
Cl4—Ni—P1	94.09 (4)	C13—C14—C15	120.1 (3)
S2—Ni—P1	95.56 (4)	C13—C14—H14	119.9
Cl4—Ni—S1	91.55 (4)	C15—C14—H14	119.9
S2—Ni—S1	78.57 (4)	C16—C15—C14	119.6 (3)
P1—Ni—S1	173.95 (3)	C16—C15—H15	120.2
C1—S1—Ni	86.07 (11)	C14—C15—H15	120.2
C1—S2—Ni	86.62 (10)	C15—C16—C11	121.2 (3)
N1—C1—S1	126.9 (3)	C15—C16—H16	119.4
N1—C1—S2	124.6 (2)	C11—C16—H16	119.4
S1—C1—S2	108.46 (16)	C22—C21—C26	118.0 (3)
C1—N1—C5	125.7 (3)	C22—C21—P1	120.4 (3)
C1—N1—C2	122.5 (2)	C26—C21—P1	121.6 (2)
C5—N1—C2	111.9 (2)	C21—C22—C23	121.2 (4)
N1—C2—C3	103.6 (3)	C21—C22—H22	119.4

N1—C2—H2A	111.0	C23—C22—H22	119.4
C3—C2—H2A	111.0	C24—C23—C22	118.5 (3)
N1—C2—H2B	111.0	C24—C23—H23	120.7
C3—C2—H2B	111.0	C22—C23—H23	120.7
H2A—C2—H2B	109.0	C25—C24—C23	121.0 (3)
C4—C3—C2	107.4 (3)	C25—C24—H24	119.5
C4—C3—H3A	110.2	C23—C24—H24	119.5
C2—C3—H3A	110.2	C24—C25—C26	120.2 (4)
C4—C3—H3B	110.2	C24—C25—H25	119.9
C2—C3—H3B	110.2	C26—C25—H25	119.9
H3A—C3—H3B	108.5	C21—C26—C25	121.0 (3)
C3—C4—C5	110.3 (3)	C21—C26—H26	119.5
C3—C4—H4A	109.6	C25—C26—H26	119.5
C5—C4—H4A	109.6	C36—C31—C32	117.9 (2)
C3—C4—H4B	109.6	C36—C31—P1	124.15 (18)
C5—C4—H4B	109.6	C32—C31—P1	117.8 (2)
H4A—C4—H4B	108.1	C33—C32—C31	120.5 (3)
N1—C5—C4	104.0 (3)	C33—C32—H32	119.8
N1—C5—H5A	110.9	C31—C32—H32	119.8
C4—C5—H5A	110.9	C34—C33—C32	119.5 (3)
N1—C5—H5B	110.9	C34—C33—H33	120.2
C4—C5—H5B	110.9	C32—C33—H33	120.2
H5A—C5—H5B	109.0	C35—C34—C33	121.4 (3)
C31—P1—C11	104.79 (13)	C35—C34—H34	119.3
C31—P1—C21	101.92 (13)	C33—C34—H34	119.3
C11—P1—C21	105.22 (13)	C34—C35—C36	118.9 (3)
C31—P1—Ni	115.11 (9)	C34—C35—H35	120.6
C11—P1—Ni	108.83 (10)	C36—C35—H35	120.6
C21—P1—Ni	119.61 (10)	C31—C36—C35	121.8 (2)
C16—C11—C12	118.6 (3)	C31—C36—H36	119.1
C16—C11—P1	124.0 (2)	C35—C36—H36	119.1
C12—C11—P1	117.4 (2)	Cl2—C37—Cl3	110.5 (2)
C13—C12—C11	119.7 (3)	Cl2—C37—Cl1	108.6 (2)
C13—C12—H12	120.2	Cl3—C37—Cl1	108.5 (2)
C11—C12—H12	120.2	Cl2—C37—H37	109.7
C14—C13—C12	120.7 (3)	Cl3—C37—H37	109.7
C14—C13—H13	119.6	Cl1—C37—H37	109.8

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C37—H37···S1	0.96	2.40	3.280 (1)	151.6

Fig. 1

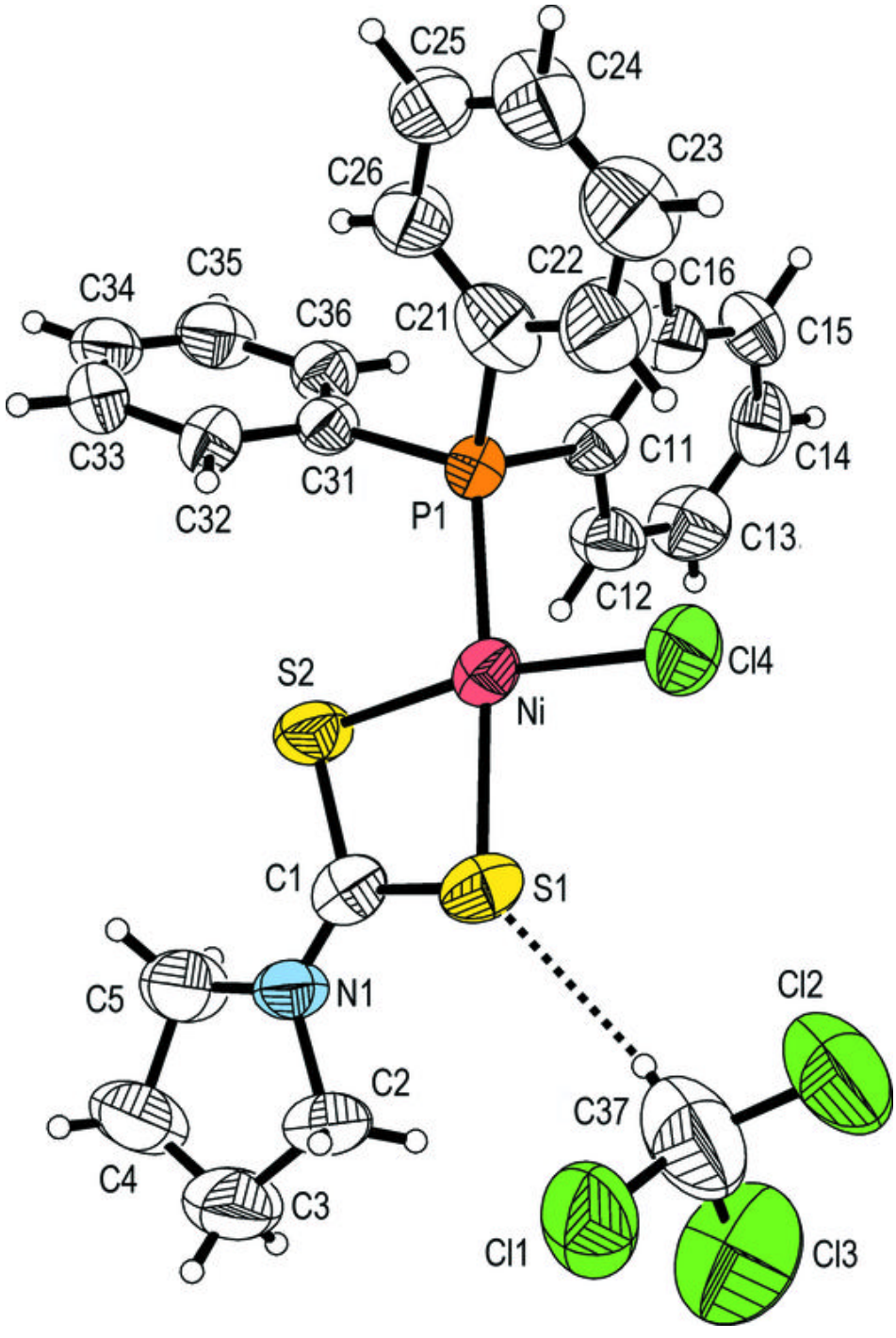
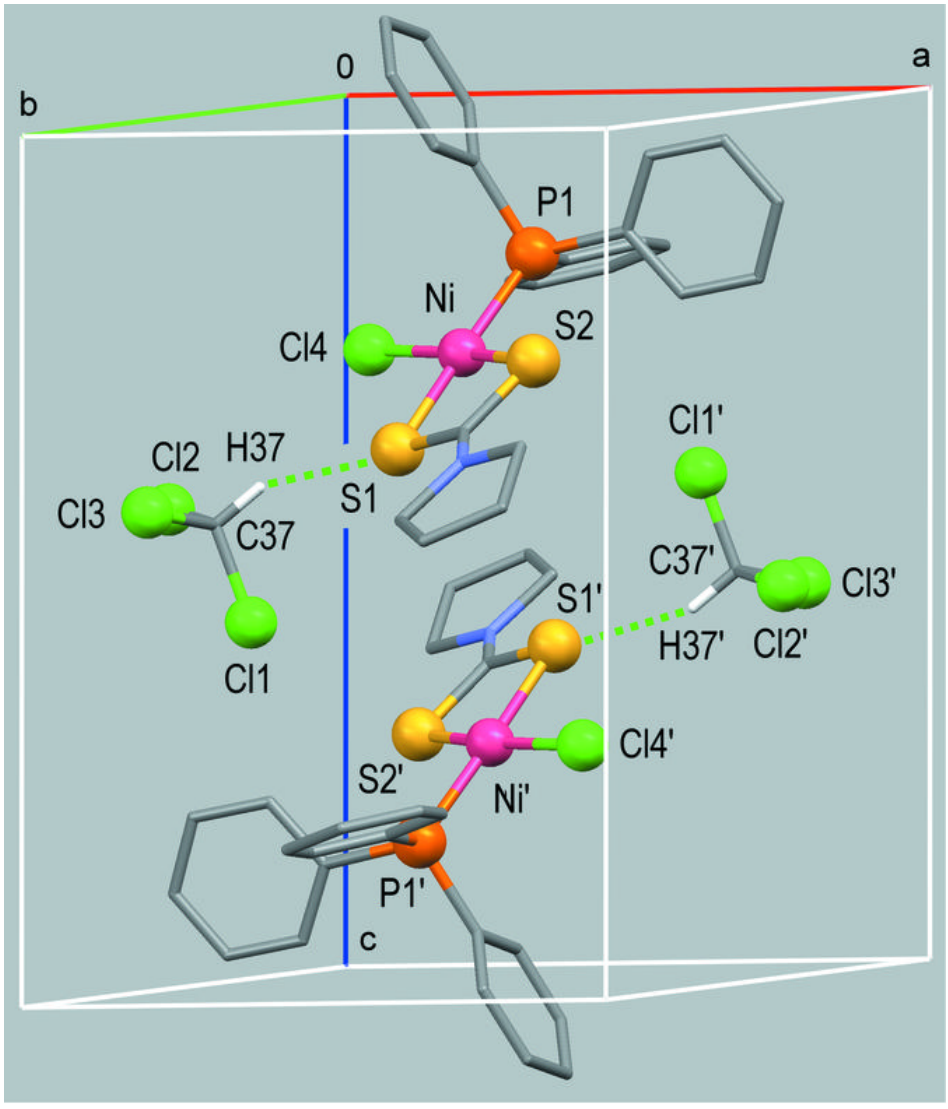

C25
C24
C23
C26
C22
C16
C35
C34
C36
C21
C15
C33
C11
C14
C31
C32
P1
C13
C12
S2
Ni
Cl4
C1
S1
C5
N1
Cl2
C2
C37
C4
C3
Cl1
Cl3

Fig. 2


0
a
b
P1
Ni
S2
Cl4
Cl2
H37
Cl3
C37
S1
Cl1
Cl1'
C37'
Cl3'
S1'
H37'
Cl2'
Cl4'
S2'
Ni'
P1'
c