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Bis[O-propan-2-yl (4-ethoxyphenyl)-dithiophosphonato- κ^2S,S']nickel(II)

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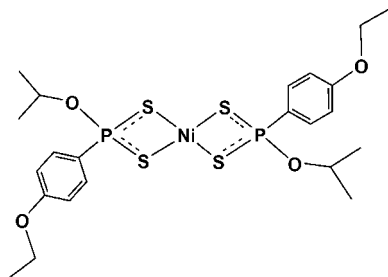
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 Key indicators: single-crystal X-ray study; $T = 173$ K; mean $\sigma(C-C) = 0.003$ Å; R factor = 0.030; wR factor = 0.065; data-to-parameter ratio = 19.9.

The title compound, $[Ni(C_{11}H_{16}O_2PS_2)_2]$, is a neutral four-coordinate mononuclear complex with a square-planar geometry. The complex lies on an inversion center. The metal atom is surrounded by two chelating isobidentate O-propan-2-yl (4-ethoxyphenyl)dithiophosphonate ligands in a *trans* configuration binding through the S-donor atoms. The Ni—S bond lengths are 2.2328 (5) and 2.2369 (5) Å, an insignificant difference to be considered anisobidentate. The Ni···P separation is 2.8224 (5) Å and the S—P bond lengths are 2.0035 (7) and 2.0053 (7) Å. The S—Ni—S (chelating) and S—Ni—S (*trans*) bond angles are 88.321 (18) and 180°. The Ni—S—P bond angles are 83.26 (2) and 83.33 (2)°, indicating a very minor distortion from ideal square-planar geometry for the Ni atom. The P atom, however, is distorted quite significantly from an ideal tetrahedral geometry, as reflected by the S—P—S and O—P—C bond angles of 101.93 (3) and 100.70 (7)°, respectively.

Related literature

For information on dithiophosphonate compounds, see: Van Zyl & Fackler (2000); Van Zyl (2010). For examples of nickel(II) dithiophosphonate complexes, see: Liu *et al.* (2004); Gray *et al.* (2004); Aragoni *et al.* (2007); Arca *et al.* (1997); Malatesta & Pizzotti (1945); Hartung (1967).



Experimental

Crystal data

$[Ni(C_{11}H_{16}O_2PS_2)_2]$
 $M_r = 609.37$
 Triclinic, $P\bar{1}$
 $a = 7.8893$ (6) Å
 $b = 8.4178$ (7) Å
 $c = 11.4825$ (10) Å
 $\alpha = 109.530$ (4)°
 $\beta = 101.959$ (4)°

$\gamma = 93.913$ (5)°
 $V = 695.22$ (10) Å³
 $Z = 1$
 Mo $K\alpha$ radiation
 $\mu = 1.14$ mm⁻¹
 $T = 173$ K
 $0.39 \times 0.26 \times 0.14$ mm

Data collection

Nonius KappaCCD diffractometer
 Absorption correction: multi-scan
 (SADABS; Sheldrick, 1996)
 $T_{min} = 0.665$, $T_{max} = 0.857$

14410 measured reflections
 3062 independent reflections
 2381 reflections with $I > 2\sigma(I)$
 $R_{int} = 0.035$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.030$
 $wR(F^2) = 0.065$
 $S = 1.02$
 3062 reflections

154 parameters
 H-atom parameters constrained
 $\Delta\rho_{max} = 0.27$ e Å⁻³
 $\Delta\rho_{min} = -0.31$ e Å⁻³

Data collection: COLLECT (Nonius, 2000); cell refinement: DENZO-SMN (Otwinowski & Minor, 1997); data reduction: DENZO-SMN; program(s) used to solve structure: SHELXS97 (Sheldrick, 2008); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: SHELXTL (Sheldrick, 2008); 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: BH2460).

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supplementary materials

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Bis[O-propan-2-yl (4-ethoxyphenyl)dithiophosphonato- κ^2 S,S']nickel(II)

Shirveen Sewpersad and Werner E. Van Zyl

Comment

The phosphor-1,1,-dithiolate class of compounds is the heavier and softer congener of the more popular phosphonate derivatives. It contains the S₂P functionality as a common feature and several sub-categories are known which include the dithiophosphato [S₂P(OR')₂]⁻, (typically, R' = alkyl), dithiophosphinato [S₂PR₂]⁻ (R = alkyl or aryl), and dithiophosphonato [S₂PR(OR')]⁻, (typically, R = aryl or ferrocenyl, R' = alkyl) monoanionic ligands. The latter may be described as a hybrid of the former two, and are also much less developed. Amongst all metals involved in the coordination chemistry of dithiophosphonato ligands, however, nickel(II) is by far the best represented (Aragoni *et al.*, 2007; Arca *et al.*, 1997; Liu *et al.*, 2004; Gray *et al.*, 2004), with the first example dating back to 1945 (Malatesta & Pizzotti, 1945) whilst the first X-ray structural report of a nickel(II) dithiophosphonate complex reported more than 2 decades later (Hartung, 1967). The complex in the present study was formed from the reaction between NiCl₂·6H₂O and the ammonium salt of [S₂P(OⁱPr)(4-C₆H₄OEt)] (molar ratio 1:2) in an aqueous/methanolic solution, the NH₄Cl by-product was dissolved and the precipitated product filtered off and washed with water. General and convenient methods to prepare dithiophosphonate salt derivatives have been reported (Van Zyl & Fackler, 2000; Van Zyl, 2010).

Experimental

A colorless methanol (40 ml) solution of NH₄[S₂P(OⁱPr)(4-C₆H₄OEt)] (982 mg, 3.347 mmol) was prepared. A second green solution of NiCl₂·6H₂O (399 mg, 1.679 mmol) in deionized water (20 ml) was prepared, and added to the colorless solution with stirring over a period of 5 min. This resulted in a purple precipitate indicating the formation of the title complex. The precipitate was collected by vacuum filtration, washed with water (3 × 10 ml) and allowed to dry under vacuum for a period of 3 hrs, yielding a dry, free-flowing purple powder. Purple crystals suitable for X-ray analysis were grown by the slow diffusion of hexane into a dichloromethane solution of the title complex. Yield: 761 mg, 75%. *M.p.* 168–169°C.

³¹P NMR (CDCl₃): δ (p.p.m.): 97.96. ¹H NMR (CDCl₃): δ (p.p.m.): 7.96 (2H, dd, *o*-ArH), 6.95 (2H, dd, *m*-ArH), 5.19 (1H, d quart, OCH), 4.06 (2H, quart, ArOCH₂), 1.42 (3H, t, ArOCH₂CH₃), 1.38 (6H, d, CH₃). ¹³C NMR (CDCl₃): δ (p.p.m.): 162.22 (*p*-ArC), 131.71 (*m*-ArC), 128.04 (Ar—C₁), 114.44 (*o*-ArC), 72.10 (CH), 63.76(ArOCH₂), 24.30 (CH₃), 14.67 (ArOCH₂CH₃).

Refinement

All hydrogen atoms were found in the difference electron density maps and were placed in idealized positions and refined with geometrical constraints, with C—H bond lengths in the range 0.95–1.00 Å. The structure was refined to *R* factor of 0.0303.

Computing details

Data collection: *COLLECT* (Nonius, 2000); cell refinement: *DENZO-SMN* (Otwinowski & Minor, 1997); data reduction: *DENZO-SMN* (Otwinowski & Minor, 1997); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXL97* (Sheldrick, 2008).

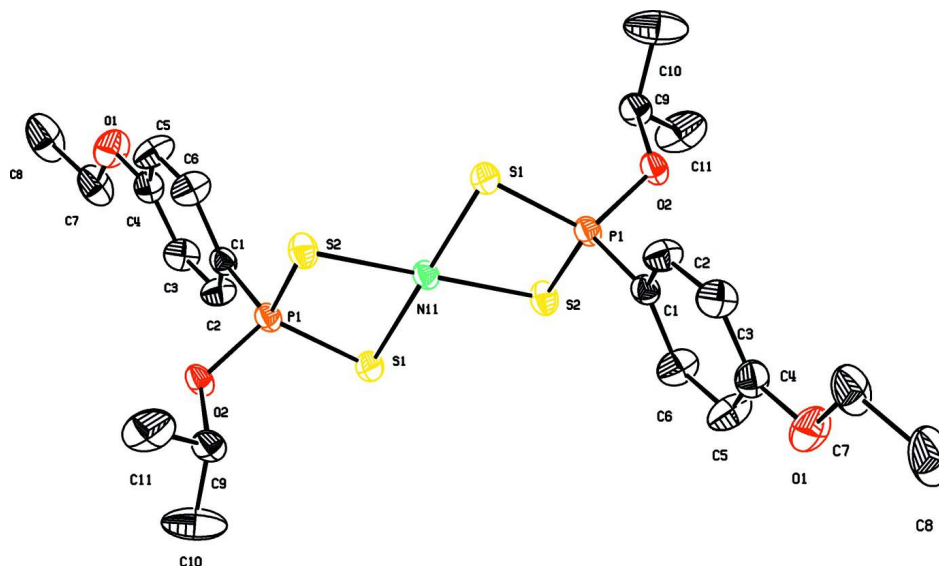


Figure 1

The molecular structure of the title complex, shown with 50% probability displacement ellipsoids.

Bis[O-propan-2-yl (4-ethoxyphenyl)dithiophosphonato- κ^2S,S']nickel(II)

Crystal data

$[\text{Ni}(\text{C}_{11}\text{H}_{16}\text{O}_2\text{PS}_2)_2]$

$M_r = 609.37$

Triclinic, $P\bar{1}$

Hall symbol: -P 1

$a = 7.8893$ (6) Å

$b = 8.4178$ (7) Å

$c = 11.4825$ (10) Å

$\alpha = 109.530$ (4)°

$\beta = 101.959$ (4)°

$\gamma = 93.913$ (5)°

$V = 695.22$ (10) Å³

$Z = 1$

$F(000) = 318$

$D_x = 1.455$ Mg m⁻³

Melting point: 441 K

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 14410 reflections

$\theta = 2.6\text{--}27.5^\circ$

$\mu = 1.14$ mm⁻¹

$T = 173$ K

Block, purple

$0.39 \times 0.26 \times 0.14$ mm

Data collection

Nonius KappaCCD

diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

1.2° φ scans and ω scans

Absorption correction: multi-scan

(*SADABS*; Sheldrick, 1996)

$T_{\min} = 0.665$, $T_{\max} = 0.857$

14410 measured reflections

3062 independent reflections

2381 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.035$

$\theta_{\max} = 27.5^\circ$, $\theta_{\min} = 2.6^\circ$

$h = 0 \rightarrow 10$

$k = -10 \rightarrow 10$

$l = -14 \rightarrow 14$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.030$

$wR(F^2) = 0.065$

$S = 1.02$

3062 reflections

154 parameters

0 restraints

0 constraints

Primary atom site location: structure-invariant
direct methods

Secondary atom site location: difference Fourier
map

Hydrogen site location: inferred from
neighbouring sites

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.028P)^2 + 0.2462P]$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} < 0.001$

$\Delta\rho_{\max} = 0.27 \text{ e } \text{\AA}^{-3}$

$\Delta\rho_{\min} = -0.31 \text{ e } \text{\AA}^{-3}$

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

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
Ni1	0.5000	0.5000	0.0000	0.02229 (10)
S1	0.64099 (6)	0.75272 (6)	0.02356 (5)	0.02729 (13)
S2	0.58144 (7)	0.38847 (6)	-0.18193 (5)	0.02842 (13)
P1	0.64598 (6)	0.63370 (6)	-0.15849 (5)	0.02271 (12)
O1	0.19743 (19)	0.8621 (2)	-0.53641 (14)	0.0416 (4)
O2	0.82744 (15)	0.67472 (17)	-0.18857 (12)	0.0266 (3)
C1	0.5013 (2)	0.7025 (2)	-0.26799 (17)	0.0231 (4)
C2	0.4982 (3)	0.8752 (3)	-0.2422 (2)	0.0317 (5)
H2	0.5664	0.9551	-0.1634	0.038*
C3	0.3977 (3)	0.9335 (3)	-0.3289 (2)	0.0336 (5)
H3	0.3976	1.0523	-0.3099	0.040*
C4	0.2974 (2)	0.8179 (3)	-0.44330 (19)	0.0308 (5)
C5	0.2947 (3)	0.6447 (3)	-0.4682 (2)	0.0405 (6)
H5	0.2227	0.5647	-0.5455	0.049*
C6	0.3960 (3)	0.5880 (3)	-0.3815 (2)	0.0360 (5)
H6	0.3937	0.4690	-0.3996	0.043*
C7	0.2176 (3)	1.0381 (3)	-0.5253 (2)	0.0463 (6)
H7A	0.3430	1.0834	-0.5079	0.056*
H7B	0.1696	1.1073	-0.4548	0.056*
C8	0.1178 (3)	1.0441 (4)	-0.6510 (3)	0.0654 (8)
H8A	0.1653	0.9734	-0.7200	0.098*
H8B	0.1299	1.1621	-0.6482	0.098*
H8C	-0.0063	1.0008	-0.6662	0.098*
C9	0.9927 (2)	0.6387 (3)	-0.11992 (19)	0.0302 (5)
H9	0.9693	0.6009	-0.0502	0.036*
C10	1.1185 (3)	0.8017 (3)	-0.0629 (3)	0.0646 (8)
H10A	1.1334	0.8447	-0.1302	0.097*
H10B	1.2319	0.7811	-0.0211	0.097*
H10C	1.0725	0.8863	0.0001	0.097*
C11	1.0541 (3)	0.4983 (3)	-0.2128 (2)	0.0567 (7)
H11A	0.9642	0.3971	-0.2481	0.085*
H11B	1.1628	0.4711	-0.1695	0.085*
H11C	1.0758	0.5343	-0.2819	0.085*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Ni1	0.02098 (18)	0.0252 (2)	0.0246 (2)	0.00292 (14)	0.00633 (14)	0.01367 (16)
S1	0.0299 (3)	0.0276 (3)	0.0257 (3)	0.0000 (2)	0.0069 (2)	0.0119 (2)
S2	0.0343 (3)	0.0258 (3)	0.0305 (3)	0.0050 (2)	0.0128 (2)	0.0138 (2)
P1	0.0197 (2)	0.0268 (3)	0.0260 (3)	0.00378 (19)	0.0065 (2)	0.0145 (2)
O1	0.0440 (9)	0.0502 (10)	0.0364 (9)	0.0170 (7)	0.0024 (7)	0.0253 (8)
O2	0.0170 (6)	0.0397 (8)	0.0333 (8)	0.0077 (6)	0.0074 (6)	0.0248 (7)
C1	0.0184 (9)	0.0282 (11)	0.0257 (10)	0.0047 (8)	0.0065 (8)	0.0125 (9)
C2	0.0288 (11)	0.0299 (12)	0.0315 (12)	0.0026 (9)	-0.0009 (9)	0.0100 (10)
C3	0.0329 (11)	0.0284 (11)	0.0413 (13)	0.0073 (9)	0.0041 (10)	0.0170 (10)
C4	0.0259 (10)	0.0406 (13)	0.0323 (12)	0.0111 (9)	0.0076 (9)	0.0198 (10)
C5	0.0466 (14)	0.0352 (13)	0.0292 (12)	0.0069 (10)	-0.0062 (10)	0.0073 (10)
C6	0.0407 (12)	0.0283 (11)	0.0355 (12)	0.0090 (9)	0.0008 (10)	0.0112 (10)
C7	0.0392 (13)	0.0601 (16)	0.0630 (16)	0.0159 (11)	0.0154 (12)	0.0483 (14)
C8	0.0461 (15)	0.107 (2)	0.082 (2)	0.0262 (15)	0.0198 (14)	0.0779 (19)
C9	0.0194 (10)	0.0415 (12)	0.0363 (12)	0.0080 (9)	0.0029 (8)	0.0240 (10)
C10	0.0313 (13)	0.0465 (15)	0.097 (2)	0.0041 (11)	-0.0166 (14)	0.0217 (16)
C11	0.0352 (13)	0.0701 (18)	0.0548 (17)	0.0292 (12)	-0.0002 (12)	0.0123 (14)

Geometric parameters (\AA , $^\circ$)

Ni1—S2	2.2328 (5)	C4—C5	1.387 (3)
Ni1—S2 ⁱ	2.2328 (5)	C5—C6	1.378 (3)
Ni1—S1 ⁱ	2.2369 (5)	C5—H5	0.9500
Ni1—S1	2.2369 (5)	C6—H6	0.9500
Ni1—P1	2.8224 (5)	C7—C8	1.513 (3)
Ni1—P1 ⁱ	2.8224 (5)	C7—H7A	0.9900
S1—P1	2.0035 (7)	C7—H7B	0.9900
S2—P1	2.0053 (7)	C8—H8A	0.9800
P1—O2	1.5828 (12)	C8—H8B	0.9800
P1—C1	1.7894 (18)	C8—H8C	0.9800
O1—C4	1.364 (2)	C9—C11	1.489 (3)
O1—C7	1.438 (3)	C9—C10	1.497 (3)
O2—C9	1.484 (2)	C9—H9	1.0000
C1—C6	1.385 (3)	C10—H10A	0.9800
C1—C2	1.386 (3)	C10—H10B	0.9800
C2—C3	1.383 (3)	C10—H10C	0.9800
C2—H2	0.9500	C11—H11A	0.9800
C3—C4	1.381 (3)	C11—H11B	0.9800
C3—H3	0.9500	C11—H11C	0.9800
S2—Ni1—S2 ⁱ	180.0	O1—C4—C5	116.20 (19)
S2—Ni1—S1 ⁱ	91.679 (18)	C3—C4—C5	119.59 (18)
S2 ⁱ —Ni1—S1 ⁱ	88.321 (18)	C6—C5—C4	120.3 (2)
S2—Ni1—S1	88.321 (18)	C6—C5—H5	119.8
S2 ⁱ —Ni1—S1	91.679 (18)	C4—C5—H5	119.8
S1 ⁱ —Ni1—S1	180.0	C5—C6—C1	120.7 (2)
S2—Ni1—P1	44.885 (16)	C5—C6—H6	119.6

S2 ⁱ —Ni1—P1	135.115 (17)	C1—C6—H6	119.6
S1 ⁱ —Ni1—P1	135.174 (16)	O1—C7—C8	106.7 (2)
S1—Ni1—P1	44.827 (16)	O1—C7—H7A	110.4
S2—Ni1—P1 ⁱ	135.115 (16)	C8—C7—H7A	110.4
S2 ⁱ —Ni1—P1 ⁱ	44.885 (16)	O1—C7—H7B	110.4
S1 ⁱ —Ni1—P1 ⁱ	44.827 (16)	C8—C7—H7B	110.4
S1—Ni1—P1 ⁱ	135.173 (16)	H7A—C7—H7B	108.6
P1—Ni1—P1 ⁱ	180.0	C7—C8—H8A	109.5
P1—S1—Ni1	83.26 (2)	C7—C8—H8B	109.5
P1—S2—Ni1	83.33 (2)	H8A—C8—H8B	109.5
O2—P1—C1	100.70 (7)	C7—C8—H8C	109.5
O2—P1—S1	113.76 (6)	H8A—C8—H8C	109.5
C1—P1—S1	113.38 (7)	H8B—C8—H8C	109.5
O2—P1—S2	114.16 (6)	O2—C9—C11	107.84 (16)
C1—P1—S2	113.47 (7)	O2—C9—C10	107.22 (17)
S1—P1—S2	101.93 (3)	C11—C9—C10	113.9 (2)
O2—P1—Ni1	141.40 (5)	O2—C9—H9	109.3
C1—P1—Ni1	117.90 (6)	C11—C9—H9	109.3
S1—P1—Ni1	51.913 (17)	C10—C9—H9	109.3
S2—P1—Ni1	51.790 (18)	C9—C10—H10A	109.5
C4—O1—C7	118.46 (17)	C9—C10—H10B	109.5
C9—O2—P1	121.36 (11)	H10A—C10—H10B	109.5
C6—C1—C2	118.40 (18)	C9—C10—H10C	109.5
C6—C1—P1	121.83 (15)	H10A—C10—H10C	109.5
C2—C1—P1	119.72 (15)	H10B—C10—H10C	109.5
C3—C2—C1	121.31 (19)	C9—C11—H11A	109.5
C3—C2—H2	119.3	C9—C11—H11B	109.5
C1—C2—H2	119.3	H11A—C11—H11B	109.5
C4—C3—C2	119.60 (19)	C9—C11—H11C	109.5
C4—C3—H3	120.2	H11A—C11—H11C	109.5
C2—C3—H3	120.2	H11B—C11—H11C	109.5
O1—C4—C3	124.21 (19)		
S2—Ni1—S1—P1	12.58 (2)	S1—P1—O2—C9	58.46 (15)
S2 ⁱ —Ni1—S1—P1	-167.42 (2)	S2—P1—O2—C9	-57.97 (15)
P1 ⁱ —Ni1—S1—P1	180.0	Ni1—P1—O2—C9	0.38 (19)
S1 ⁱ —Ni1—S2—P1	167.43 (2)	O2—P1—C1—C6	101.89 (17)
S1—Ni1—S2—P1	-12.57 (2)	S1—P1—C1—C6	-136.21 (15)
P1 ⁱ —Ni1—S2—P1	180.0	S2—P1—C1—C6	-20.53 (18)
Ni1—S1—P1—O2	-137.71 (6)	Ni1—P1—C1—C6	-78.31 (17)
Ni1—S1—P1—C1	107.99 (7)	O2—P1—C1—C2	-75.50 (16)
Ni1—S1—P1—S2	-14.35 (3)	S1—P1—C1—C2	46.41 (17)
Ni1—S2—P1—O2	137.47 (6)	S2—P1—C1—C2	162.08 (14)
Ni1—S2—P1—C1	-107.90 (7)	Ni1—P1—C1—C2	104.30 (15)
Ni1—S2—P1—S1	14.37 (3)	C6—C1—C2—C3	-2.3 (3)
S2—Ni1—P1—O2	-81.30 (9)	P1—C1—C2—C3	175.20 (15)
S2 ⁱ —Ni1—P1—O2	98.70 (9)	C1—C2—C3—C4	0.4 (3)
S1 ⁱ —Ni1—P1—O2	-99.27 (9)	C7—O1—C4—C3	10.4 (3)
S1—Ni1—P1—O2	80.73 (9)	C7—O1—C4—C5	-169.75 (19)

S2—Ni1—P1—C1	99.02 (8)	C2—C3—C4—O1	-178.27 (18)
S2 ⁱ —Ni1—P1—C1	-80.98 (8)	C2—C3—C4—C5	1.9 (3)
S1 ⁱ —Ni1—P1—C1	81.05 (8)	O1—C4—C5—C6	177.9 (2)
S1—Ni1—P1—C1	-98.95 (8)	C3—C4—C5—C6	-2.2 (3)
S2—Ni1—P1—S1	-162.03 (3)	C4—C5—C6—C1	0.3 (3)
S2 ⁱ —Ni1—P1—S1	17.97 (3)	C2—C1—C6—C5	1.9 (3)
S1 ⁱ —Ni1—P1—S1	180.000 (1)	P1—C1—C6—C5	-175.49 (17)
S2 ⁱ —Ni1—P1—S2	180.0	C4—O1—C7—C8	169.83 (18)
S1 ⁱ —Ni1—P1—S2	-17.97 (3)	P1—O2—C9—C11	112.06 (18)
S1—Ni1—P1—S2	162.03 (3)	P1—O2—C9—C10	-124.87 (18)
C1—P1—O2—C9	-179.91 (14)		

Symmetry code: (i) $-x+1, -y+1, -z$.