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(2,2′-Bipyridyl-κ^2N,N')bis(*O*,*O*′-diisopropyl dithiophosphato-κ^2S,S')nickel(II)

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Key indicators

Single-crystal X-ray study
T = 120 K
Mean σ(C–C) = 0.005 Å
R factor = 0.043
wR factor = 0.133
Data-to-parameter ratio = 22.1

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

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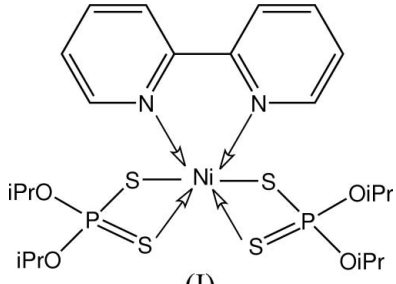
The monomeric title compound, $[Ni(C_6H_{14}O_2PS_2)_2(C_{10}H_8N_2)]$, has the Ni atom within a distorted octahedral *cis*-N_2S_4 geometry. The crystal structure is stabilized by C—H···S interactions, leading to the formation of a linear chain.

Comment

In continuation of our interest in the structural chemistry of bipyridine adducts of nickel(II) dithiophosphates, with general formula $Ni[S_2P(OR)_2]_2$(2,2′-bipyridine) (Berdugo & Tiekink, 2006), the title complex, where *R* = ^{i}Pr, (I), was investigated. The distorted octahedral geometry in (I) (Fig. 1) is based on a *cis*-N_2S_4 donor set and is in agreement with those found in related structures, namely *R* = Me (Arora *et al.*, 1977), *R* = ^{n}Bu [You *et al.*, 1986; see Hu (1999) for space group revision] and *R* = ^{i}Bu (Berdugo & Tiekink, 2006). The Ni—S distances (Table 1) lie in the relatively narrow range 2.4548 (9) (Ni—S1) to 2.4964 (9) Å (Ni—S4) and the P—S distances follow the expected trends in that the shorter bond is always associated with the less tightly bound S atom. Distortions from the ideal octahedral geometry may be attributed to the acute chelate angles. The 2,2′-bipyridine molecule features a small twist about the central C—C bond (Table 1).



(I)

The most prominent intermolecular contact in the structure is of the type $C_{aromatic}$—H···S [H16···S3^i = 2.70 Å, C16···S3^i = 3.514 (4) Å and C16—H16···S3^i = 144°; symmetry code: (i) 1 + *x*, *y*, *z*]. These interactions lead to the formation of a linear chain as illustrated in Fig. 2. There are intramolecular C—H···π interactions of note involving the methine C1/H1 and C10/H10 atoms with the ring centroids of the N1- and N2-pyridine rings, respectively, with distances and angles of 2.75 Å and 109°, and 2.78 Å and 108°, respectively. In the recently determined structure of the *R* = ^{i}Bu analogue (Berdugo & Tiekink, 2006), related C—H···π contacts were present, but owing to the greater reach of the isobutyl ligand, these interactions were intermolecular and served to stabilize the chain mediated by C—H···S interactions.

Experimental

The title compound was prepared by refluxing the parent nickel dithiophosphate with 2,2′-bipyridine (Acros Organics) following a literature procedure (Lai *et al.*, 2004). Green crystals were isolated by the slow evaporation of a $CHCl_3$ solution of the compound; m.p. 463 K (decompostion). IR (KBr disk): ν(C—O) 1174, ν(P—O) 954, $\nu(P-S)_{asymm}$ 657, $\nu(P-S)_{sym}$ 535 cm^{-1}.

Crystal data

$[Ni(C_6H_{14}O_2PS_2)_2(C_{10}H_8N_2)]$
M_r = 641.42
Monoclinic, $P2_1/n$
a = 9.1585 (3) Å
b = 30.6703 (12) Å
c = 11.6407 (4) Å
β = 110.808 (1)°
V = 3056.53 (19) Å^3
Z = 4
D_x = 1.394 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 1.04 mm^{-1}
T = 120 (2) K
Rod, green
0.48 × 0.06 × 0.03 mm

Data collection

Bruker–Nonius KappaCCD diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)
T_{min} = 0.793, T_{max} = 1
29206 measured reflections
6997 independent reflections
5541 reflections with $I > 2\sigma(I)$
R_{int} = 0.072
θ_{max} = 27.5°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.043
$wR(F^2)$ = 0.133
S = 1.05
6997 reflections
316 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0675P)^2 + 3.279P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.002
$\Delta\rho_{max}$ = 0.55 e Å^{-3}
$\Delta\rho_{min}$ = −0.49 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Ni—S1	2.4548 (9)	Ni—N2	2.088 (3)
Ni—S2	2.4840 (8)	S1—P1	1.9907 (11)
Ni—S3	2.4839 (9)	S2—P1	1.9929 (11)
Ni—S4	2.4964 (9)	S3—P2	1.9790 (11)
Ni—N1	2.071 (2)	S4—P2	1.9890 (11)
S1—Ni—S2	81.50 (3)	S3—Ni—S4	81.48 (3)
S1—Ni—S4	174.11 (3)	S3—Ni—N2	165.22 (8)
S2—Ni—N1	167.00 (8)	N1—Ni—N2	78.82 (10)
N1—C17—C18—N2	−6.2 (4)		

H atoms were included in the riding-model approximation with C—H distances = 0.95–1.00 Å, and with U_{iso}(H) = 1.5U_{eq}(methyl C) and U_{iso}(H) = 1.2U_{eq}(remaining C).

Data collection: *COLLECT* (Hooft, 1998); cell refinement: *DENZO* (Otwinowski & Minor, 1997) and *COLLECT*; data reduction: *DENZO* and *COLLECT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPII* (Johnson, 1976) and *DIAMOND* (Brandenburg, 2006); software used to prepare material for publication: *SHELXL97*.

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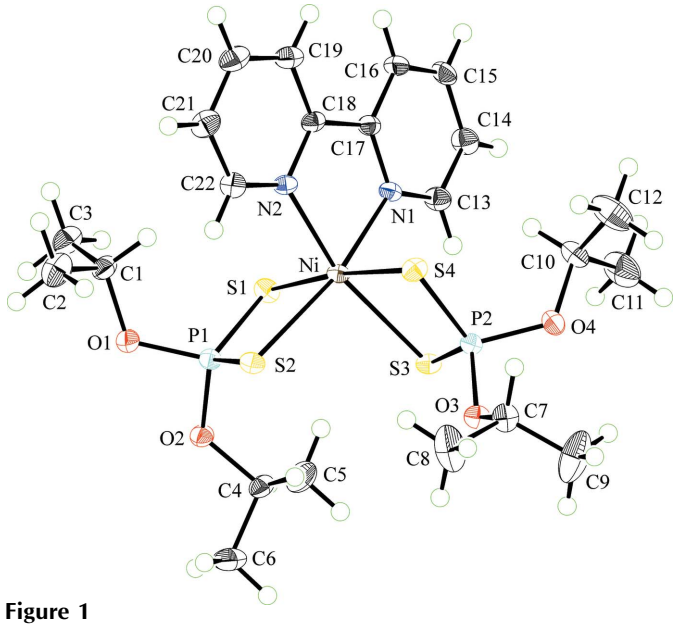

Figure 1
Molecular structure and crystallographic numbering scheme for (I). Displacement ellipsoids are shown at the 50% probability level.

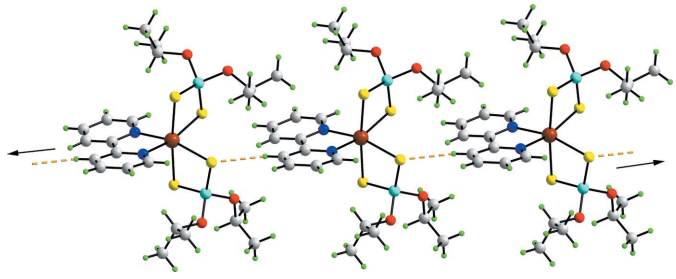

Figure 2
The chain in (I), running parallel to *a*, mediated by C—H···S interactions, shown as dashed orange lines. Colour code: Ni (brown), S (yellow), P (light blue), O (red), N (blue), C (grey) and H (green).

Cheminova is thanked for the gift of the dithiophosphate ligand used in this study. The authors also thank the EPSRC X-ray Crystallographic Service, University of Southampton, England, for the data collection.

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supporting information

Acta Cryst. (2006). E**62**, m2693–m2694 [https://doi.org/10.1107/S1600536806037913]

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$c = 11.6407$ (4) Å
$\beta = 110.808$ (1)°
$V = 3056.53$ (19) Å^3
$Z = 4$
$F(000) = 1344$
$D_x = 1.394$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71069$ Å
Cell parameters from 6812 reflections
$\theta = 2.9$–27.5°
$\mu = 1.04$ mm^{-1}
$T = 120$ K
Rod, green
0.48 × 0.06 × 0.03 mm

Data collection

Bruker–Nonius KappaCD diffractometer
Radiation source: Bruker–Nonius FR591 rotating anode
Graphite monochromator
Detector resolution: 9.091 pixels mm^{-1}
φ and ω scans
Absorption correction: multi-scan (SADABS; Sheldrick, 2003)
$T_{min} = 0.793$, $T_{max} = 1$
29206 measured reflections
6997 independent reflections
5541 reflections with $I > 2\sigma(I)$
$R_{int} = 0.072$
$\theta_{max} = 27.5°$, $\theta_{min} = 3.1°$
$h = -11\rightarrow8$
$k = -39\rightarrow37$
$l = -15\rightarrow14$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.043$
$wR(F^2) = 0.133$
$S = 1.05$
6997 reflections
316 parameters
0 restraints
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
$w = 1/[\sigma^2(F_o^2) + (0.0675P)^2 + 3.279P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.002$
$\Delta\rho_{max} = 0.55$ e Å^{-3}
$\Delta\rho_{min} = -0.49$ e Å^{-3}

Special details

Experimental. IR (KBr disk): ν(C—O) 1174, ν(P—O) 954, ν(P—S)$_{asymm}$ 657, ν(P—S)$_{sym}$ 535 cm^{-1}.
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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
Ni	0.32652 (4)	0.878909 (13)	0.11621 (4)	0.01529 (12)
S1	0.37235 (9)	0.94928 (3)	0.22367 (8)	0.02038 (18)
S2	0.22284 (9)	0.86035 (3)	0.28044 (7)	0.01862 (17)
S3	0.06932 (9)	0.90159 (2)	−0.03429 (7)	0.01779 (17)
S4	0.25069 (9)	0.80864 (3)	0.00226 (8)	0.02103 (18)
P1	0.28068 (9)	0.92140 (3)	0.33698 (7)	0.01687 (18)
P2	0.05795 (9)	0.84093 (3)	−0.09480 (8)	0.01730 (18)
O1	0.3930 (2)	0.92490 (8)	0.4760 (2)	0.0226 (5)
O2	0.1408 (2)	0.94779 (7)	0.3539 (2)	0.0203 (5)
O3	−0.0975 (2)	0.81962 (7)	−0.0903 (2)	0.0204 (5)
O4	0.0249 (3)	0.83740 (8)	−0.2381 (2)	0.0230 (5)
N1	0.4483 (3)	0.90291 (8)	0.0102 (2)	0.0168 (5)
N2	0.5464 (3)	0.85112 (8)	0.2038 (2)	0.0172 (5)
C1	0.5577 (4)	0.91228 (11)	0.5106 (3)	0.0232 (7)
H1	0.5775	0.9019	0.4360	0.028*
C2	0.5883 (5)	0.87564 (12)	0.6019 (4)	0.0345 (9)
H2A	0.5222	0.8507	0.5637	0.052*
H2B	0.5643	0.8853	0.6735	0.052*
H2C	0.6984	0.8671	0.6281	0.052*
C3	0.6544 (4)	0.95263 (11)	0.5606 (3)	0.0284 (8)
H3A	0.6287	0.9751	0.4965	0.043*
H3B	0.7656	0.9453	0.5861	0.043*
H3C	0.6315	0.9636	0.6314	0.043*
C4	−0.0137 (4)	0.94983 (11)	0.2568 (3)	0.0215 (7)
H4	−0.0307	0.9229	0.2052	0.026*
C5	−0.0227 (4)	0.98933 (12)	0.1769 (4)	0.0326 (8)
H5A	0.0556	0.9868	0.1378	0.049*
H5B	−0.0026	1.0157	0.2277	0.049*
H5C	−0.1271	0.9911	0.1135	0.049*
C6	−0.1306 (4)	0.95096 (15)	0.3207 (4)	0.0386 (10)
H6A	−0.1194	0.9247	0.3709	0.058*
H6B	−0.2364	0.9522	0.2592	0.058*
H6C	−0.1120	0.9768	0.3735	0.058*
C7	−0.1345 (4)	0.77380 (11)	−0.1276 (3)	0.0239 (7)
H7	−0.0447	0.7611	−0.1460	0.029*
C8	−0.1507 (6)	0.74988 (14)	−0.0225 (4)	0.0483 (12)
H8A	−0.0535	0.7524	0.0487	0.072*
H8B	−0.1722	0.7191	−0.0443	0.072*
H8C	−0.2370	0.7623	−0.0021	0.072*
C9	−0.2755 (6)	0.77242 (14)	−0.2421 (4)	0.0554 (13)

H9A	−0.2554	0.7889	−0.3069	0.083*
H9B	−0.3644	0.7853	−0.2261	0.083*
H9C	−0.2996	0.7421	−0.2684	0.083*
C10	0.1447 (4)	0.85094 (12)	−0.2873 (3)	0.0276 (8)
H10	0.2396	0.8612	−0.2186	0.033*
C11	0.0795 (6)	0.88761 (15)	−0.3760 (4)	0.0466 (11)
H11A	0.0551	0.9123	−0.3326	0.070*
H11B	−0.0159	0.8779	−0.4415	0.070*
H11C	0.1567	0.8966	−0.4119	0.070*
C12	0.1854 (6)	0.81163 (15)	−0.3467 (5)	0.0529 (13)
H12A	0.2272	0.7888	−0.2849	0.079*
H12B	0.2640	0.8196	−0.3821	0.079*
H12C	0.0914	0.8009	−0.4118	0.079*
C13	0.3972 (4)	0.93320 (11)	−0.0782 (3)	0.0228 (7)
H13	0.2924	0.9431	−0.1011	0.027*
C14	0.4908 (4)	0.95061 (11)	−0.1373 (3)	0.0257 (7)
H14	0.4517	0.9724	−0.1986	0.031*
C15	0.6422 (4)	0.93569 (12)	−0.1056 (3)	0.0258 (7)
H15	0.7090	0.9469	−0.1449	0.031*
C16	0.6945 (4)	0.90436 (11)	−0.0163 (3)	0.0230 (7)
H16	0.7978	0.8933	0.0055	0.028*
C17	0.5968 (3)	0.88875 (10)	0.0423 (3)	0.0176 (6)
C18	0.6483 (3)	0.85807 (10)	0.1460 (3)	0.0179 (6)
C19	0.7945 (4)	0.83808 (11)	0.1853 (3)	0.0250 (7)
H19	0.8646	0.8430	0.1430	0.030*
C20	0.8359 (4)	0.81109 (12)	0.2863 (4)	0.0295 (8)
H20	0.9358	0.7976	0.3152	0.035*
C21	0.7305 (4)	0.80370 (11)	0.3458 (3)	0.0273 (8)
H21	0.7562	0.7850	0.4152	0.033*
C22	0.5873 (4)	0.82442 (11)	0.3009 (3)	0.0221 (7)
H22	0.5146	0.8195	0.3408	0.027*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Ni	0.0146 (2)	0.0137 (2)	0.0195 (2)	0.00014 (14)	0.00844 (16)	0.00082 (15)
S1	0.0245 (4)	0.0158 (4)	0.0238 (4)	−0.0041 (3)	0.0121 (3)	−0.0013 (3)
S2	0.0197 (4)	0.0148 (4)	0.0240 (4)	−0.0006 (3)	0.0110 (3)	0.0014 (3)
S3	0.0165 (4)	0.0139 (4)	0.0244 (4)	0.0003 (3)	0.0091 (3)	0.0002 (3)
S4	0.0200 (4)	0.0159 (4)	0.0258 (4)	0.0032 (3)	0.0064 (3)	−0.0018 (3)
P1	0.0172 (4)	0.0155 (4)	0.0186 (4)	0.0007 (3)	0.0073 (3)	0.0003 (3)
P2	0.0172 (4)	0.0169 (4)	0.0188 (4)	−0.0004 (3)	0.0077 (3)	−0.0009 (3)
O1	0.0198 (11)	0.0268 (13)	0.0205 (12)	0.0024 (9)	0.0064 (9)	−0.0020 (10)
O2	0.0182 (11)	0.0204 (12)	0.0217 (12)	0.0038 (9)	0.0065 (9)	−0.0018 (9)
O3	0.0209 (11)	0.0156 (11)	0.0260 (12)	−0.0024 (9)	0.0099 (10)	−0.0023 (9)
O4	0.0251 (12)	0.0269 (13)	0.0194 (12)	−0.0007 (10)	0.0106 (10)	−0.0013 (10)
N1	0.0162 (12)	0.0172 (13)	0.0196 (13)	−0.0013 (10)	0.0094 (11)	−0.0012 (11)
N2	0.0147 (12)	0.0151 (13)	0.0223 (14)	0.0017 (10)	0.0074 (11)	0.0003 (11)

C1	0.0177 (15)	0.0245 (18)	0.0238 (17)	0.0033 (13)	0.0029 (13)	−0.0029 (14)
C2	0.036 (2)	0.024 (2)	0.036 (2)	0.0046 (16)	0.0035 (17)	0.0017 (16)
C3	0.0244 (17)	0.0228 (18)	0.034 (2)	0.0002 (14)	0.0051 (15)	−0.0043 (15)
C4	0.0165 (15)	0.0210 (17)	0.0241 (17)	0.0035 (13)	0.0037 (13)	0.0001 (13)
C5	0.0281 (18)	0.027 (2)	0.036 (2)	0.0039 (15)	0.0025 (16)	0.0071 (16)
C6	0.0221 (18)	0.053 (3)	0.043 (2)	0.0071 (17)	0.0151 (17)	0.004 (2)
C7	0.0295 (17)	0.0152 (16)	0.0268 (18)	−0.0036 (13)	0.0097 (15)	−0.0007 (13)
C8	0.080 (3)	0.031 (2)	0.035 (2)	−0.020 (2)	0.021 (2)	0.0006 (18)
C9	0.068 (3)	0.028 (2)	0.043 (3)	−0.014 (2)	−0.013 (2)	0.0027 (19)
C10	0.0333 (19)	0.0288 (19)	0.0274 (19)	0.0023 (15)	0.0190 (16)	−0.0004 (15)
C11	0.060 (3)	0.046 (3)	0.045 (3)	0.009 (2)	0.032 (2)	0.021 (2)
C12	0.071 (3)	0.044 (3)	0.062 (3)	0.008 (2)	0.046 (3)	−0.009 (2)
C13	0.0229 (16)	0.0212 (17)	0.0265 (18)	0.0003 (13)	0.0116 (14)	0.0042 (14)
C14	0.0285 (18)	0.0242 (18)	0.0272 (18)	−0.0025 (14)	0.0133 (15)	0.0059 (14)
C15	0.0250 (17)	0.035 (2)	0.0228 (17)	−0.0074 (15)	0.0153 (14)	−0.0012 (15)
C16	0.0161 (15)	0.0306 (19)	0.0247 (17)	−0.0023 (13)	0.0100 (13)	−0.0025 (14)
C17	0.0158 (14)	0.0176 (16)	0.0206 (16)	−0.0020 (12)	0.0078 (12)	−0.0054 (12)
C18	0.0145 (14)	0.0153 (16)	0.0232 (16)	−0.0030 (12)	0.0058 (12)	−0.0058 (13)
C19	0.0193 (16)	0.0239 (18)	0.0328 (19)	0.0005 (13)	0.0106 (14)	−0.0030 (15)
C20	0.0210 (16)	0.0271 (19)	0.037 (2)	0.0044 (14)	0.0063 (15)	0.0001 (16)
C21	0.0255 (17)	0.0212 (18)	0.032 (2)	0.0054 (14)	0.0064 (15)	0.0079 (15)
C22	0.0245 (16)	0.0174 (16)	0.0250 (17)	0.0001 (13)	0.0094 (14)	0.0020 (13)

Geometric parameters (Å, º)

Ni—S1	2.4548 (9)	C6—H6B	0.9800
Ni—S2	2.4840 (8)	C6—H6C	0.9800
Ni—S3	2.4839 (9)	C7—C8	1.479 (5)
Ni—S4	2.4964 (9)	C7—C9	1.491 (5)
Ni—N1	2.071 (2)	C7—H7	1.0000
Ni—N2	2.088 (3)	C8—H8A	0.9800
S1—P1	1.9907 (11)	C8—H8B	0.9800
S2—P1	1.9929 (11)	C8—H8C	0.9800
S3—P2	1.9790 (11)	C9—H9A	0.9800
S4—P2	1.9890 (11)	C9—H9B	0.9800
P1—O1	1.583 (2)	C9—H9C	0.9800
P1—O2	1.586 (2)	C10—C11	1.500 (5)
P2—O3	1.584 (2)	C10—C12	1.500 (5)
P2—O4	1.590 (2)	C10—H10	1.0000
O1—C1	1.469 (4)	C11—H11A	0.9800
O2—C4	1.465 (4)	C11—H11B	0.9800
O3—C7	1.474 (4)	C11—H11C	0.9800
O4—C10	1.467 (4)	C12—H12A	0.9800
N1—C13	1.341 (4)	C12—H12B	0.9800
N1—C17	1.348 (4)	C12—H12C	0.9800
N2—C22	1.337 (4)	C13—C14	1.384 (4)
N2—C18	1.347 (4)	C13—H13	0.9500
C1—C2	1.503 (5)	C14—C15	1.379 (5)

C1—C3	1.512 (5)	C14—H14	0.9500
C1—H1	1.0000	C15—C16	1.371 (5)
C2—H2A	0.9800	C15—H15	0.9500
C2—H2B	0.9800	C16—C17	1.389 (4)
C2—H2C	0.9800	C16—H16	0.9500
C3—H3A	0.9800	C17—C18	1.471 (5)
C3—H3B	0.9800	C18—C19	1.394 (4)
C3—H3C	0.9800	C19—C20	1.376 (5)
C4—C6	1.505 (5)	C19—H19	0.9500
C4—C5	1.511 (5)	C20—C21	1.392 (5)
C4—H4	1.0000	C20—H20	0.9500
C5—H5A	0.9800	C21—C22	1.382 (5)
C5—H5B	0.9800	C21—H21	0.9500
C5—H5C	0.9800	C22—H22	0.9500
C6—H6A	0.9800		

N1—Ni—S1	87.73 (7)	C4—C6—H6B	109.5
N2—Ni—S1	98.58 (8)	H6A—C6—H6B	109.5
N1—Ni—S3	93.25 (8)	C4—C6—H6C	109.5
S1—Ni—S3	93.49 (3)	H6A—C6—H6C	109.5
N2—Ni—S2	95.54 (7)	H6B—C6—H6C	109.5
S1—Ni—S2	81.50 (3)	O3—C7—C8	108.0 (3)
S1—Ni—S4	174.11 (3)	O3—C7—C9	109.0 (3)
S2—Ni—N1	167.00 (8)	C8—C7—C9	114.6 (4)
S3—Ni—S4	81.48 (3)	O3—C7—H7	108.4
S3—Ni—N2	165.22 (8)	C8—C7—H7	108.4
S3—Ni—S2	94.64 (3)	C9—C7—H7	108.4
N1—Ni—S4	95.58 (8)	C7—C8—H8A	109.5
N2—Ni—S4	86.86 (7)	C7—C8—H8B	109.5
S2—Ni—S4	95.79 (3)	H8A—C8—H8B	109.5
N1—Ni—N2	78.82 (10)	C7—C8—H8C	109.5
P1—S1—Ni	85.64 (4)	H8A—C8—H8C	109.5
P1—S2—Ni	84.81 (4)	H8B—C8—H8C	109.5
P2—S3—Ni	84.52 (4)	C7—C9—H9A	109.5
P2—S4—Ni	83.99 (4)	C7—C9—H9B	109.5
O1—P1—O2	95.66 (12)	H9A—C9—H9B	109.5
O1—P1—S1	112.00 (9)	C7—C9—H9C	109.5
O2—P1—S1	114.35 (10)	H9A—C9—H9C	109.5
O1—P1—S2	113.66 (10)	H9B—C9—H9C	109.5
O2—P1—S2	112.89 (9)	O4—C10—C11	108.1 (3)
S1—P1—S2	108.05 (5)	O4—C10—C12	107.1 (3)
O3—P2—O4	99.69 (12)	C11—C10—C12	112.9 (4)
O3—P2—S3	108.09 (9)	O4—C10—H10	109.5
O4—P2—S3	113.81 (10)	C11—C10—H10	109.5
O3—P2—S4	113.74 (10)	C12—C10—H10	109.5
O4—P2—S4	111.21 (9)	C10—C11—H11A	109.5
S3—P2—S4	110.00 (5)	C10—C11—H11B	109.5
C1—O1—P1	119.56 (19)	H11A—C11—H11B	109.5

C4—O2—P1	121.8 (2)	C10—C11—H11C	109.5
C7—O3—P2	119.92 (19)	H11A—C11—H11C	109.5
C10—O4—P2	120.1 (2)	H11B—C11—H11C	109.5
C13—N1—C17	118.6 (3)	C10—C12—H12A	109.5
C13—N1—Ni	126.0 (2)	C10—C12—H12B	109.5
C17—N1—Ni	115.1 (2)	H12A—C12—H12B	109.5
C22—N2—C18	118.9 (3)	C10—C12—H12C	109.5
C22—N2—Ni	126.5 (2)	H12A—C12—H12C	109.5
C18—N2—Ni	114.2 (2)	H12B—C12—H12C	109.5
O1—C1—C2	107.8 (3)	N1—C13—C14	122.6 (3)
O1—C1—C3	107.1 (3)	N1—C13—H13	118.7
C2—C1—C3	113.8 (3)	C14—C13—H13	118.7
O1—C1—H1	109.3	C15—C14—C13	118.7 (3)
C2—C1—H1	109.3	C15—C14—H14	120.6
C3—C1—H1	109.3	C13—C14—H14	120.6
C1—C2—H2A	109.5	C16—C15—C14	118.9 (3)
C1—C2—H2B	109.5	C16—C15—H15	120.5
H2A—C2—H2B	109.5	C14—C15—H15	120.5
C1—C2—H2C	109.5	C15—C16—C17	120.0 (3)
H2A—C2—H2C	109.5	C15—C16—H16	120.0
H2B—C2—H2C	109.5	C17—C16—H16	120.0
C1—C3—H3A	109.5	N1—C17—C16	121.0 (3)
C1—C3—H3B	109.5	N1—C17—C18	115.5 (3)
H3A—C3—H3B	109.5	C16—C17—C18	123.3 (3)
C1—C3—H3C	109.5	N2—C18—C19	121.5 (3)
H3A—C3—H3C	109.5	N2—C18—C17	115.8 (3)
H3B—C3—H3C	109.5	C19—C18—C17	122.8 (3)
O2—C4—C6	106.3 (3)	C20—C19—C18	119.1 (3)
O2—C4—C5	109.6 (3)	C20—C19—H19	120.5
C6—C4—C5	113.1 (3)	C18—C19—H19	120.5
O2—C4—H4	109.2	C19—C20—C21	119.6 (3)
C6—C4—H4	109.2	C19—C20—H20	120.2
C5—C4—H4	109.2	C21—C20—H20	120.2
C4—C5—H5A	109.5	C22—C21—C20	118.0 (3)
C4—C5—H5B	109.5	C22—C21—H21	121.0
H5A—C5—H5B	109.5	C20—C21—H21	121.0
C4—C5—H5C	109.5	N2—C22—C21	123.0 (3)
H5A—C5—H5C	109.5	N2—C22—H22	118.5
H5B—C5—H5C	109.5	C21—C22—H22	118.5
C4—C6—H6A	109.5		

N1—Ni—S1—P1	−172.58 (8)	S3—Ni—N1—C17	−164.8 (2)
N2—Ni—S1—P1	−94.24 (8)	S2—Ni—N1—C17	67.9 (4)
S3—Ni—S1—P1	94.31 (4)	S4—Ni—N1—C17	−83.0 (2)
S2—Ni—S1—P1	0.13 (4)	N1—Ni—N2—C22	−178.8 (3)
N1—Ni—S2—P1	34.2 (3)	S1—Ni—N2—C22	95.3 (3)
N2—Ni—S2—P1	97.76 (8)	S3—Ni—N2—C22	−120.3 (3)
S1—Ni—S2—P1	−0.13 (4)	S2—Ni—N2—C22	13.1 (3)

S3—Ni—S2—P1	−92.97 (4)	S4—Ni—N2—C22	−82.4 (3)
N1—Ni—S3—P2	95.71 (8)	N1—Ni—N2—C18	−6.0 (2)
N2—Ni—S3—P2	38.8 (3)	S1—Ni—N2—C18	−92.0 (2)
S1—Ni—S3—P2	−176.37 (4)	S3—Ni—N2—C18	52.5 (4)
S2—Ni—S3—P2	−94.63 (4)	S2—Ni—N2—C18	−174.2 (2)
S4—Ni—S3—P2	0.54 (3)	S4—Ni—N2—C18	90.3 (2)
N1—Ni—S4—P2	−93.00 (8)	P1—O1—C1—C2	−119.8 (3)
N2—Ni—S4—P2	−171.44 (8)	P1—O1—C1—C3	117.4 (3)
S3—Ni—S4—P2	−0.54 (3)	P1—O2—C4—C6	145.6 (2)
S2—Ni—S4—P2	93.31 (4)	P1—O2—C4—C5	−91.9 (3)
Ni—S1—P1—O1	125.77 (10)	P2—O3—C7—C8	121.7 (3)
Ni—S1—P1—O2	−126.79 (10)	P2—O3—C7—C9	−113.2 (3)
Ni—S1—P1—S2	−0.16 (5)	P2—O4—C10—C11	−118.8 (3)
Ni—S2—P1—O1	−124.80 (10)	P2—O4—C10—C12	119.3 (3)
Ni—S2—P1—O2	127.64 (10)	C17—N1—C13—C14	−0.2 (5)
Ni—S2—P1—S1	0.16 (5)	Ni—N1—C13—C14	173.8 (3)
Ni—S3—P2—O3	124.00 (10)	N1—C13—C14—C15	1.1 (5)
Ni—S3—P2—O4	−126.27 (10)	C13—C14—C15—C16	−0.3 (5)
Ni—S3—P2—S4	−0.72 (5)	C14—C15—C16—C17	−1.3 (5)
Ni—S4—P2—O3	−120.68 (10)	C13—N1—C17—C16	−1.4 (5)
Ni—S4—P2—O4	127.73 (10)	Ni—N1—C17—C16	−176.0 (2)
Ni—S4—P2—S3	0.72 (5)	C13—N1—C17—C18	175.5 (3)
O2—P1—O1—C1	−168.6 (2)	Ni—N1—C17—C18	0.9 (3)
S1—P1—O1—C1	−49.5 (2)	C15—C16—C17—N1	2.1 (5)
S2—P1—O1—C1	73.4 (2)	C15—C16—C17—C18	−174.5 (3)
O1—P1—O2—C4	−169.9 (2)	C22—N2—C18—C19	0.0 (5)
S1—P1—O2—C4	72.9 (2)	Ni—N2—C18—C19	−173.4 (2)
S2—P1—O2—C4	−51.2 (2)	C22—N2—C18—C17	−178.4 (3)
O4—P2—O3—C7	62.4 (2)	Ni—N2—C18—C17	8.3 (3)
S3—P2—O3—C7	−178.5 (2)	N1—C17—C18—N2	−6.2 (4)
S4—P2—O3—C7	−56.0 (2)	C16—C17—C18—N2	170.6 (3)
O3—P2—O4—C10	−176.0 (2)	N1—C17—C18—C19	175.5 (3)
S3—P2—O4—C10	69.2 (2)	C16—C17—C18—C19	−7.7 (5)
S4—P2—O4—C10	−55.7 (2)	N2—C18—C19—C20	−0.8 (5)
N2—Ni—N1—C13	−171.5 (3)	C17—C18—C19—C20	177.5 (3)
S1—Ni—N1—C13	−72.3 (3)	C18—C19—C20—C21	1.1 (5)
S3—Ni—N1—C13	21.0 (3)	C19—C20—C21—C22	−0.6 (5)
S2—Ni—N1—C13	−106.3 (4)	C18—N2—C22—C21	0.5 (5)
S4—Ni—N1—C13	102.8 (3)	Ni—N2—C22—C21	173.0 (3)
N2—Ni—N1—C17	2.6 (2)	C20—C21—C22—N2	−0.2 (5)
S1—Ni—N1—C17	101.9 (2)		