Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Bis(*O*,*O*′-diisopropyl dithiophosphato-κ^2S,S')(1,10-phenanthroline)nickel(II)

Erick Berdugo and Edward R. T. Tiekink*

Department of Chemistry, University of Texas at San Antonio, One UTSA Circle, San Antonio, Texas 78249-0698, USA
Correspondence e-mail: edward.tiekink@utsa.edu

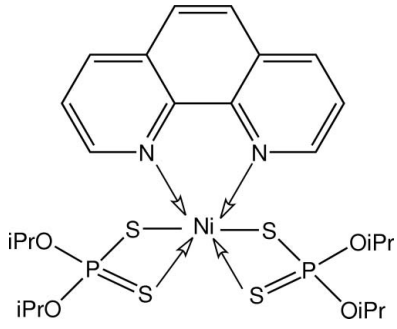
Received 3 October 2007; accepted 4 October 2007

Key indicators: single-crystal X-ray study; T = 173 K; mean σ(C–C) = 0.006 Å; R factor = 0.057; wR factor = 0.118; data-to-parameter ratio = 18.7.

The Ni atom in the monomeric title compound, $[Ni(C_6H_{14}O_2PS_2)_2(C_{12}H_8N_2)]$, is within a distorted octahedral N_2S_4 geometry, defined by two chelating dithiophosphate ligands and a chelating 1,10-phenanthroline ligand.

Related literature

For related literature, see Berdugo & Tiekink (2006); Berdugo *et al.* (2006, 2007); Hoskins & Tiekink (1985); Lai *et al.* (2004). For related structures, see Shetty & Fernando (1970); Craig *et al.* (1971); Hao *et al.* (2001).



Experimental

Crystal data

$[Ni(C_6H_{14}O_2PS_2)_2(C_{12}H_8N_2)]$
M_r = 665.44
Monoclinic, $P2_1/c$
a = 14.4115 (17) Å
b = 11.3618 (11) Å
c = 18.582 (2) Å
β = 96.829 (3)°
V = 3021.0 (6) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 1.06 mm^{-1}
T = 173 (2) K
0.12 × 0.10 × 0.09 mm

Data collection

Rigaku AFC12κ/SATURN724 diffractometer
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
T_{min} = 0.787, T_{max} = 1
(expected range = 0.716–0.909)
30553 measured reflections
6248 independent reflections
5770 reflections with $I > 2\sigma(I)$
R_{int} = 0.047

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.057
$wR(F^2)$ = 0.118
S = 1.17
6248 reflections
334 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.67 e Å^{-3}
$\Delta\rho_{min}$ = −0.39 e Å^{-3}

Data collection: *CrystalClear* (Rigaku, 2005); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; program(s) used to solve structure: *PATTY* in *DIRDIF* (Beurskens *et al.*, 1992); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPII* (Johnson, 1976); software used to prepare material for publication: *SHELXL97*.

The authors are grateful to the NIH/NIGMS MBRS–RISE program (grant No. GM60655) for support. Cheminova is also thanked for the gift of the dithiophosphate ligand used in this study.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HG2309).

References

Berdugo, E., Tiekink, E. R. T., Wardell, J. L. & Wardell, S. M. S. V. (2006). *Acta Cryst.* E**62**, m2693–m2694.
Berdugo, E., Tiekink, E. R. T., Wardell, J. L. & Wardell, S. M. S. V. (2007). *Acta Cryst.* E**63**, m764–m766.
Berdugo, E. & Tiekink, E. R. T. (2006). *Acta Cryst.* E**62**, m2218–m2220.
Beurskens, P. T., Admiraal, G., Beurskens, G., Bosman, W. P., García-Granda, S., Gould, R. O., Smits, J. M. M. & Smykalla, C. (1992). *The DIRDIF Program System.* Technical Report of the Crystallography Laboratory, University of Nijmegen, The Netherlands.
Craig, D. C., Pallister, E. T. & Stephenson, N. C. (1971). *Acta Cryst.* B**27**, 1163–1168.
Hao, Q.-L., Fun, H.-K., Chantrapromma, S., Razak, I. A., Jian, F.-F., Yang, X.-J., Lu, L.-D. & Wang, X. (2001). *Acta Cryst.* C**57**, 717–718.
Higashi, T. (1995). *ABSCOR*. Rigaku Corporation, Tokyo, Japan.
Hoskins, B. F. & Tiekink, E. R. T. (1985). *Acta Cryst.* C**41**, 322–324.
Johnson, C. K. (1976). *ORTEPII*. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.
Lai, C. S., Liu, S. & Tiekink, E. R. T. (2004). *CrystEngComm*, **6**, 221–226.
Rigaku (2005). *CrystalClear*. Rigaku Americas Corporation, The Woodlands, Texas, USA.
Sheldrick, G. M. (1997). *SHELXL97*. University of Göttingen, Germany.
Shetty, P. S. & Fernando, Q. (1970). *J. Am. Chem. Soc.* **92**, 3964–3969.

  doi:10.1107/S1600536807048659

supplementary materials

Acta Cryst. (2007). E**63**, m2688 [**doi:10.1107/S1600536807048659**]

Bis(*O*,*O*'-diisopropyl dithiophosphato-$\kappa^2 S,S'$)(1,10-phenanthroline)nickel(II)

E. Berdugo and E. R. T. Tiekink

Comment

Nickel(II) dithiophosphates, $Ni(S_2P(OR)_2)_2$, are well known to form stable adducts with pyridine-type ligands (Berdugo & Tiekink, 2006; Berdugo *et al.*, 2006, 2007). In {$Ni[S_2P(O^iPr)_2]_2$(1,10-phenanthroline)} (I), Fig. 1, the Ni atom exists within a N_2S_4 donor set defined by a two chelating dithiophosphate ligands and a chelating 1,10-phenanthroline ligand. The dithiophosphate ligands chelate in the symmetric mode with the Ni—S distances lying in the relatively narrow range of 2.4674 (11) to 2.5075 (11) Å. The Ni—N distances are equal within experimental error, *i.e.* 2.098 (3) and 2.100 (3) Å. The major distortions from the ideal octahedral geometry are related to the acute chelate angles that range from 79.75 (12)° for N1—Ni—N2 to 81.80 (4)° for S1—Ni—S2. The structure conforms closely to other structures of the general formula $[Ni(S_2P(OR)_2)_2$(1,10-phenanthroline)], *R* = Me (Shetty & Fernando, 1970), *R* = Et (Craig *et al.*, 1971) and *R* = *p*-tol (Hao *et al.*, 2001).

Experimental

The title compound was prepared by warming (338 K) the parent nickel dithiophosphate (Hoskins & Tiekink, 1985) with 1,10-phenanthroline (Acros Organics) following a literature procedure in $CHCl_3$ (40 ml) (Lai *et al.*, 2004). Green crystals were isolated by the slow evaporation (4 days) of a 1:7:2 methanol/ethanol/$CHCl_3$ solution of the compound; m.p. 569 K. IR (KBr disk): ν(C—O) 1174, ν(P—O) 956, $\nu(P—S)_{asymm}$ 660, $\nu(P—S)_{sym}$ 554 cm^{-1}.

Refinement

The C-bound H atoms were included in the riding-model approximation with C—H distances = 0.95 to 0.98 Å, and with U_{iso}(methyl-H) = 1.5U_{eq}(methyl-C) and U_{iso}(H) = 1.2U_{eq}(remaining-C).

Figures

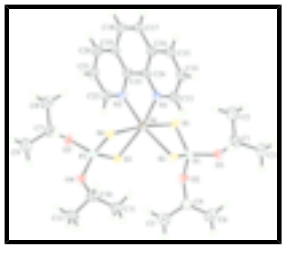


Fig. 1. Molecular structure and crystallographic numbering scheme for (I). Displacement ellipsoids are shown at the 50% probability level.

Bis(*O*,*O*'-diisopropyl dithiophosphato-κ^2*S*,*S*')(1,10-phenanthroline)nickel(II)

Crystal data

$[Ni(C_6H_{14}O_2PS_2)_2(C_{12}H_8N_2)]$	F_{000} = 1392
M_r = 665.44	D_x = 1.463 Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation λ = 0.71070 Å
Hall symbol: -P 2ybc	Cell parameters from 11263 reflections
a = 14.4115 (17) Å	θ = 2.3–30.4º
b = 11.3618 (11) Å	μ = 1.06 mm^{-1}
c = 18.582 (2) Å	T = 173 (2) K
β = 96.829 (3)º	Block, green
V = 3021.0 (6) Å^3	0.12 × 0.10 × 0.09 mm
Z = 4	

Data collection

Rigaku AFC12κ/SATURN724 diffractometer	6248 independent reflections
Radiation source: fine-focus sealed tube	5770 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.047
T = 173(2) K	θ_{max} = 26.5º
ω scans	θ_{min} = 2.1º
Absorption correction: multi-scan (ABSCOR; Higashi, 1995)	h = −17→18
T_{min} = 0.787, T_{max} = 1	k = −14→14
30553 measured reflections	l = −23→23

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.057	H-atom parameters constrained
$wR(F^2)$ = 0.118	$w = 1/[\sigma^2(F_o^2) + (0.0251P)^2 + 8.8715P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.17	$(\Delta/\sigma)_{max}$ = 0.001
6248 reflections	$\Delta\rho_{max}$ = 0.67 e Å^{-3}
334 parameters	$\Delta\rho_{min}$ = −0.39 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}
Ni	0.23701 (3)	0.47852 (4)	0.80759 (3)	0.02291 (13)
S1	0.07915 (7)	0.39132 (9)	0.79641 (6)	0.0325 (2)
S2	0.26773 (7)	0.32161 (9)	0.90167 (5)	0.0279 (2)
S3	0.19746 (7)	0.63322 (8)	0.89438 (5)	0.0285 (2)
S4	0.39635 (7)	0.56769 (8)	0.83575 (5)	0.0277 (2)
P1	0.12922 (7)	0.30853 (9)	0.88634 (5)	0.0258 (2)
P2	0.32526 (7)	0.68842 (8)	0.88466 (5)	0.0255 (2)
O1	0.0927 (2)	0.1774 (2)	0.88923 (16)	0.0350 (7)
O2	0.08171 (18)	0.3637 (2)	0.95129 (14)	0.0308 (6)
O3	0.31619 (19)	0.8101 (2)	0.84164 (15)	0.0295 (6)
O4	0.38178 (19)	0.7324 (2)	0.95864 (15)	0.0307 (6)
N1	0.2881 (2)	0.3751 (3)	0.72725 (16)	0.0239 (6)
N2	0.2021 (2)	0.5858 (3)	0.71649 (16)	0.0241 (6)
C1	0.1298 (4)	0.0758 (4)	0.8559 (3)	0.0469 (12)
H1	0.1964	0.0654	0.8772	0.056*
C2	0.1269 (4)	0.0811 (5)	0.7766 (3)	0.0593 (15)
H2A	0.1649	0.1474	0.7634	0.089*
H2B	0.0621	0.0919	0.7548	0.089*
H2C	0.1516	0.0077	0.7589	0.089*
C3	0.0733 (4)	−0.0264 (4)	0.8809 (3)	0.0594 (15)
H3A	0.0766	−0.0254	0.9338	0.089*
H3B	0.0989	−0.1008	0.8652	0.089*
H3C	0.0080	−0.0189	0.8596	0.089*
C4	0.1173 (3)	0.3375 (4)	1.0269 (2)	0.0327 (9)
H4	0.1819	0.3048	1.0284	0.039*
C5	0.1224 (4)	0.4531 (4)	1.0668 (3)	0.0509 (12)
H5A	0.1632	0.5073	1.0442	0.076*
H5B	0.1477	0.4402	1.1175	0.076*
H5C	0.0596	0.4872	1.0647	0.076*
C6	0.0566 (4)	0.2475 (5)	1.0577 (3)	0.0633 (16)
H6A	0.0568	0.1746	1.0295	0.095*
H6B	−0.0074	0.2776	1.0554	0.095*
H6C	0.0809	0.2316	1.1083	0.095*

C7	0.4010 (3)	0.8641 (4)	0.8187 (2)	0.0336 (9)
H7	0.4556	0.8107	0.8317	0.040*
C8	0.3835 (4)	0.8768 (5)	0.7377 (3)	0.0561 (14)
H8A	0.3746	0.7988	0.7154	0.084*
H8B	0.4373	0.9155	0.7200	0.084*
H8C	0.3273	0.9245	0.7247	0.084*
C9	0.4175 (4)	0.9787 (4)	0.8586 (3)	0.0528 (13)
H9A	0.4308	0.9633	0.9107	0.079*
H9B	0.3616	1.0281	0.8495	0.079*
H9C	0.4707	1.0194	0.8416	0.079*
C10	0.4348 (3)	0.6570 (4)	1.0117 (2)	0.0333 (9)
H10	0.4832	0.6141	0.9876	0.040*
C11	0.3739 (3)	0.5684 (4)	1.0438 (2)	0.0370 (10)
H11A	0.3453	0.5159	1.0056	0.056*
H11B	0.3248	0.6095	1.0661	0.056*
H11C	0.4120	0.5220	1.0808	0.056*
C12	0.4834 (3)	0.7399 (4)	1.0680 (2)	0.0414 (11)
H12A	0.5227	0.7949	1.0445	0.062*
H12B	0.5224	0.6943	1.1048	0.062*
H12C	0.4365	0.7840	1.0910	0.062*
C13	0.3303 (3)	0.2713 (3)	0.7337 (2)	0.0281 (8)
H13	0.3377	0.2342	0.7799	0.034*
C14	0.3642 (3)	0.2141 (4)	0.6759 (2)	0.0339 (9)
H14	0.3938	0.1396	0.6826	0.041*
C15	0.3541 (3)	0.2673 (4)	0.6086 (2)	0.0348 (9)
H15	0.3756	0.2287	0.5684	0.042*
C16	0.3124 (3)	0.3781 (4)	0.5999 (2)	0.0294 (8)
C17	0.3004 (3)	0.4407 (4)	0.5331 (2)	0.0364 (10)
H17	0.3221	0.4066	0.4914	0.044*
C18	0.2589 (3)	0.5477 (4)	0.5277 (2)	0.0368 (10)
H18	0.2539	0.5885	0.4828	0.044*
C19	0.2221 (3)	0.6008 (3)	0.5885 (2)	0.0289 (8)
C20	0.1742 (3)	0.7084 (4)	0.5857 (2)	0.0330 (9)
H20	0.1650	0.7516	0.5416	0.040*
C21	0.1407 (3)	0.7509 (4)	0.6463 (2)	0.0332 (9)
H21	0.1074	0.8232	0.6447	0.040*
C22	0.1561 (3)	0.6863 (3)	0.7115 (2)	0.0298 (8)
H22	0.1325	0.7163	0.7534	0.036*
C23	0.2340 (2)	0.5408 (3)	0.6556 (2)	0.0243 (8)
C24	0.2792 (3)	0.4293 (3)	0.6611 (2)	0.0247 (8)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Ni	0.0266 (3)	0.0216 (2)	0.0211 (2)	0.00100 (18)	0.00519 (19)	0.00161 (18)
S1	0.0291 (5)	0.0380 (6)	0.0295 (5)	−0.0018 (4)	0.0000 (4)	0.0077 (4)
S2	0.0263 (5)	0.0313 (5)	0.0265 (5)	0.0023 (4)	0.0044 (4)	0.0068 (4)
S3	0.0307 (5)	0.0288 (5)	0.0274 (5)	0.0008 (4)	0.0091 (4)	−0.0011 (4)

S4	0.0278 (5)	0.0250 (5)	0.0312 (5)	−0.0001 (4)	0.0076 (4)	−0.0039 (4)
P1	0.0283 (5)	0.0246 (5)	0.0250 (5)	−0.0037 (4)	0.0047 (4)	0.0017 (4)
P2	0.0313 (5)	0.0224 (5)	0.0232 (5)	0.0003 (4)	0.0053 (4)	−0.0005 (4)
O1	0.0437 (17)	0.0279 (15)	0.0349 (16)	−0.0065 (12)	0.0112 (13)	−0.0006 (12)
O2	0.0291 (14)	0.0380 (16)	0.0260 (14)	0.0010 (12)	0.0059 (11)	0.0023 (12)
O3	0.0329 (14)	0.0237 (13)	0.0326 (15)	0.0012 (11)	0.0073 (12)	0.0044 (11)
O4	0.0393 (15)	0.0249 (14)	0.0272 (14)	0.0010 (12)	0.0008 (12)	−0.0025 (11)
N1	0.0252 (15)	0.0241 (16)	0.0224 (16)	0.0006 (12)	0.0029 (12)	0.0001 (12)
N2	0.0251 (16)	0.0261 (16)	0.0213 (15)	0.0016 (13)	0.0039 (12)	0.0018 (13)
C1	0.063 (3)	0.032 (2)	0.048 (3)	−0.004 (2)	0.018 (2)	−0.006 (2)
C2	0.068 (4)	0.065 (4)	0.048 (3)	−0.025 (3)	0.018 (3)	−0.017 (3)
C3	0.092 (4)	0.036 (3)	0.051 (3)	−0.024 (3)	0.011 (3)	0.000 (2)
C4	0.034 (2)	0.038 (2)	0.027 (2)	0.0034 (18)	0.0068 (17)	0.0028 (17)
C5	0.067 (3)	0.046 (3)	0.039 (3)	−0.004 (2)	0.006 (2)	−0.007 (2)
C6	0.089 (4)	0.064 (4)	0.041 (3)	−0.023 (3)	0.026 (3)	0.004 (3)
C7	0.036 (2)	0.029 (2)	0.037 (2)	−0.0024 (17)	0.0099 (18)	0.0031 (18)
C8	0.067 (3)	0.064 (3)	0.039 (3)	−0.017 (3)	0.016 (3)	0.009 (2)
C9	0.062 (3)	0.036 (3)	0.063 (3)	−0.014 (2)	0.015 (3)	−0.009 (2)
C10	0.036 (2)	0.035 (2)	0.028 (2)	0.0071 (18)	0.0023 (17)	0.0012 (17)
C11	0.041 (2)	0.036 (2)	0.032 (2)	0.0004 (19)	−0.0039 (18)	0.0046 (18)
C12	0.037 (2)	0.052 (3)	0.034 (2)	−0.006 (2)	−0.0028 (19)	−0.006 (2)
C13	0.030 (2)	0.0223 (18)	0.031 (2)	0.0022 (15)	0.0009 (16)	−0.0008 (16)
C14	0.036 (2)	0.027 (2)	0.040 (2)	0.0017 (17)	0.0108 (19)	−0.0024 (18)
C15	0.038 (2)	0.034 (2)	0.034 (2)	−0.0048 (18)	0.0116 (18)	−0.0107 (18)
C16	0.029 (2)	0.034 (2)	0.027 (2)	−0.0064 (16)	0.0094 (16)	−0.0063 (17)
C17	0.045 (2)	0.042 (2)	0.024 (2)	−0.007 (2)	0.0113 (18)	−0.0049 (18)
C18	0.049 (3)	0.037 (2)	0.024 (2)	−0.011 (2)	0.0056 (18)	0.0033 (17)
C19	0.033 (2)	0.029 (2)	0.0244 (19)	−0.0080 (16)	0.0025 (16)	0.0002 (16)
C20	0.036 (2)	0.032 (2)	0.030 (2)	−0.0012 (17)	0.0005 (17)	0.0104 (17)
C21	0.034 (2)	0.027 (2)	0.038 (2)	0.0026 (16)	0.0012 (18)	0.0078 (17)
C22	0.032 (2)	0.027 (2)	0.032 (2)	0.0008 (16)	0.0068 (17)	0.0028 (16)
C23	0.0196 (17)	0.0293 (19)	0.0239 (19)	−0.0040 (14)	0.0018 (14)	0.0031 (15)
C24	0.0242 (18)	0.0262 (19)	0.0240 (19)	−0.0055 (15)	0.0039 (15)	−0.0022 (15)

Geometric parameters (Å, °)

Ni—S1	2.4674 (11)	C6—H6C	0.9800
Ni—S2	2.4992 (11)	C7—C9	1.504 (6)
Ni—S3	2.4967 (11)	C7—C8	1.504 (6)
Ni—S4	2.5075 (11)	C7—H7	1.0000
Ni—N2	2.098 (3)	C8—H8A	0.9800
Ni—N1	2.100 (3)	C8—H8B	0.9800
S1—P1	1.9777 (14)	C8—H8C	0.9800
S2—P1	1.9880 (14)	C9—H9A	0.9800
S3—P2	1.9741 (14)	C9—H9B	0.9800
S4—P2	1.9947 (14)	C9—H9C	0.9800
P1—O2	1.586 (3)	C10—C11	1.505 (6)
P1—O1	1.584 (3)	C10—C12	1.515 (6)
P2—O3	1.594 (3)	C10—H10	1.0000

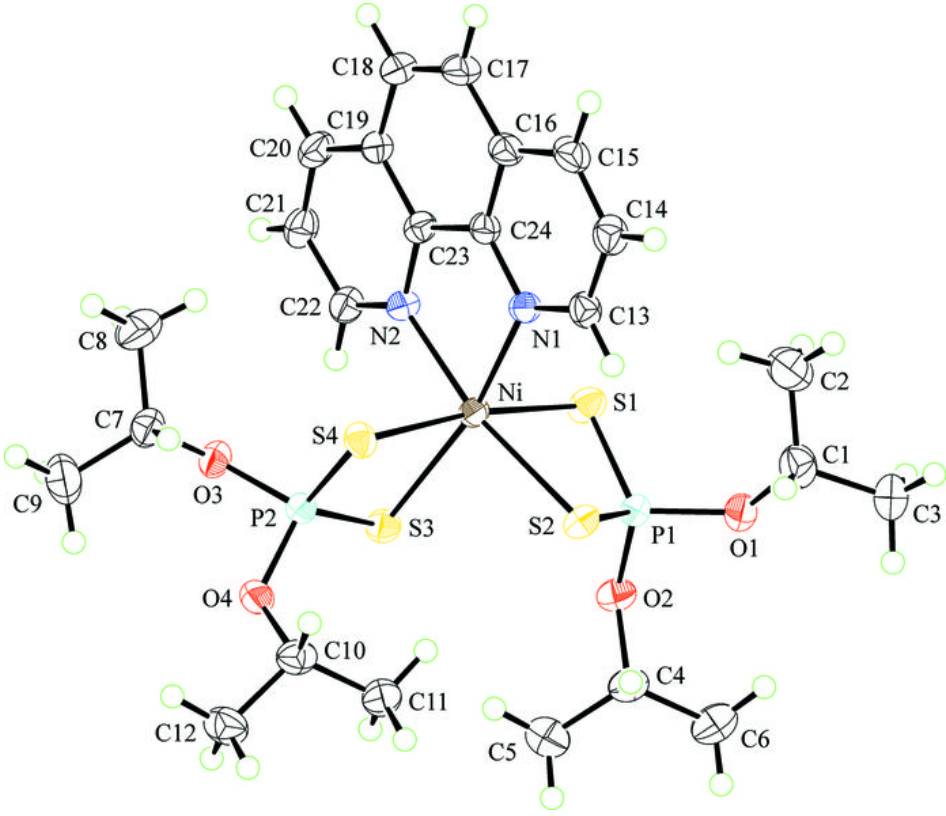
P2—O4	1.593 (3)	C11—H11A	0.9800
O1—C1	1.442 (5)	C11—H11B	0.9800
O2—C4	1.468 (5)	C11—H11C	0.9800
O3—C7	1.475 (5)	C12—H12A	0.9800
O4—C10	1.453 (5)	C12—H12B	0.9800
N1—C13	1.326 (5)	C12—H12C	0.9800
N1—C24	1.367 (5)	C13—C14	1.393 (6)
N2—C22	1.318 (5)	C13—H13	0.9500
N2—C23	1.369 (5)	C14—C15	1.380 (6)
C1—C2	1.470 (7)	C14—H14	0.9500
C1—C3	1.523 (7)	C15—C16	1.397 (6)
C1—H1	1.0000	C15—H15	0.9500
C2—H2A	0.9800	C16—C24	1.410 (5)
C2—H2B	0.9800	C16—C17	1.424 (6)
C2—H2C	0.9800	C17—C18	1.354 (6)
C3—H3A	0.9800	C17—H17	0.9500
C3—H3B	0.9800	C18—C19	1.437 (6)
C3—H3C	0.9800	C18—H18	0.9500
C4—C6	1.501 (6)	C19—C20	1.402 (6)
C4—C5	1.507 (6)	C19—C23	1.413 (5)
C4—H4	1.0000	C20—C21	1.366 (6)
C5—H5A	0.9800	C20—H20	0.9500
C5—H5B	0.9800	C21—C22	1.412 (6)
C5—H5C	0.9800	C21—H21	0.9500
C6—H6A	0.9800	C22—H22	0.9500
C6—H6B	0.9800	C23—C24	1.423 (5)
N2—Ni—N1	79.75 (12)	H6A—C6—H6C	109.5
N2—Ni—S1	92.01 (9)	H6B—C6—H6C	109.5
N1—Ni—S1	96.48 (9)	O3—C7—C9	107.8 (4)
N2—Ni—S3	93.50 (9)	O3—C7—C8	106.5 (4)
N1—Ni—S3	168.26 (9)	C9—C7—C8	114.1 (4)
S1—Ni—S3	93.33 (4)	O3—C7—H7	109.5
N2—Ni—S2	169.83 (9)	C9—C7—H7	109.5
N1—Ni—S2	92.88 (9)	C8—C7—H7	109.5
S1—Ni—S2	81.80 (4)	C7—C8—H8A	109.5
S3—Ni—S2	94.90 (4)	C7—C8—H8B	109.5
N2—Ni—S4	93.41 (9)	H8A—C8—H8B	109.5
N1—Ni—S4	89.14 (9)	C7—C8—H8C	109.5
S1—Ni—S4	172.81 (4)	H8A—C8—H8C	109.5
S3—Ni—S4	81.63 (3)	H8B—C8—H8C	109.5
S2—Ni—S4	93.48 (4)	C7—C9—H9A	109.5
P1—S1—Ni	83.09 (5)	C7—C9—H9B	109.5
P1—S2—Ni	82.06 (4)	H9A—C9—H9B	109.5
P2—S3—Ni	82.96 (4)	C7—C9—H9C	109.5
P2—S4—Ni	82.28 (5)	H9A—C9—H9C	109.5
O2—P1—O1	99.81 (15)	H9B—C9—H9C	109.5
O2—P1—S1	107.94 (12)	O4—C10—C11	112.3 (3)
O1—P1—S1	112.75 (12)	O4—C10—C12	105.3 (3)
O2—P1—S2	112.24 (12)	C11—C10—C12	112.7 (4)

O1—P1—S2	113.45 (12)	O4—C10—H10	108.8
S1—P1—S2	110.16 (6)	C11—C10—H10	108.8
O3—P2—O4	99.43 (15)	C12—C10—H10	108.8
O3—P2—S3	107.43 (11)	C10—C11—H11A	109.5
O4—P2—S3	114.06 (12)	C10—C11—H11B	109.5
O3—P2—S4	112.56 (11)	H11A—C11—H11B	109.5
O4—P2—S4	111.79 (12)	C10—C11—H11C	109.5
S3—P2—S4	111.01 (6)	H11A—C11—H11C	109.5
C1—O1—P1	126.6 (3)	H11B—C11—H11C	109.5
C4—O2—P1	121.0 (2)	C10—C12—H12A	109.5
C7—O3—P2	119.0 (2)	C10—C12—H12B	109.5
C10—O4—P2	125.1 (2)	H12A—C12—H12B	109.5
C13—N1—C24	118.3 (3)	C10—C12—H12C	109.5
C13—N1—Ni	129.0 (3)	H12A—C12—H12C	109.5
C24—N1—Ni	112.6 (2)	H12B—C12—H12C	109.5
C22—N2—C23	118.7 (3)	N1—C13—C14	123.0 (4)
C22—N2—Ni	129.0 (3)	N1—C13—H13	118.5
C23—N2—Ni	112.2 (2)	C14—C13—H13	118.5
O1—C1—C2	115.4 (4)	C15—C14—C13	119.1 (4)
O1—C1—C3	103.9 (4)	C15—C14—H14	120.5
C2—C1—C3	112.6 (4)	C13—C14—H14	120.5
O1—C1—H1	108.2	C14—C15—C16	119.8 (4)
C2—C1—H1	108.2	C14—C15—H15	120.1
C3—C1—H1	108.2	C16—C15—H15	120.1
C1—C2—H2A	109.5	C15—C16—C24	117.5 (4)
C1—C2—H2B	109.5	C15—C16—C17	123.8 (4)
H2A—C2—H2B	109.5	C24—C16—C17	118.7 (4)
C1—C2—H2C	109.5	C18—C17—C16	121.3 (4)
H2A—C2—H2C	109.5	C18—C17—H17	119.4
H2B—C2—H2C	109.5	C16—C17—H17	119.4
C1—C3—H3A	109.5	C17—C18—C19	121.3 (4)
C1—C3—H3B	109.5	C17—C18—H18	119.3
H3A—C3—H3B	109.5	C19—C18—H18	119.3
C1—C3—H3C	109.5	C20—C19—C23	117.5 (4)
H3A—C3—H3C	109.5	C20—C19—C18	124.2 (4)
H3B—C3—H3C	109.5	C23—C19—C18	118.3 (4)
O2—C4—C6	110.5 (4)	C21—C20—C19	119.8 (4)
O2—C4—C5	106.6 (3)	C21—C20—H20	120.1
C6—C4—C5	113.8 (4)	C19—C20—H20	120.1
O2—C4—H4	108.6	C20—C21—C22	119.3 (4)
C6—C4—H4	108.6	C20—C21—H21	120.3
C5—C4—H4	108.6	C22—C21—H21	120.3
C4—C5—H5A	109.5	N2—C22—C21	122.5 (4)
C4—C5—H5B	109.5	N2—C22—H22	118.8
H5A—C5—H5B	109.5	C21—C22—H22	118.8
C4—C5—H5C	109.5	N2—C23—C19	122.1 (3)
H5A—C5—H5C	109.5	N2—C23—C24	118.0 (3)
H5B—C5—H5C	109.5	C19—C23—C24	119.9 (3)
C4—C6—H6A	109.5	N1—C24—C16	122.3 (3)

C4—C6—H6B	109.5	N1—C24—C23	117.3 (3)
H6A—C6—H6B	109.5	C16—C24—C23	120.4 (3)
C4—C6—H6C	109.5		
N2—Ni—S1—P1	−176.93 (9)	S3—Ni—N2—C22	−13.2 (3)
N1—Ni—S1—P1	103.16 (9)	S2—Ni—N2—C22	132.5 (4)
S3—Ni—S1—P1	−83.31 (5)	S4—Ni—N2—C22	−95.0 (3)
S2—Ni—S1—P1	11.17 (5)	N1—Ni—N2—C23	−4.0 (2)
N2—Ni—S2—P1	−64.1 (5)	S1—Ni—N2—C23	−100.2 (2)
N1—Ni—S2—P1	−107.28 (9)	S3—Ni—N2—C23	166.3 (2)
S1—Ni—S2—P1	−11.14 (5)	S2—Ni—N2—C23	−48.0 (6)
S3—Ni—S2—P1	81.52 (5)	S4—Ni—N2—C23	84.5 (2)
S4—Ni—S2—P1	163.41 (5)	P1—O1—C1—C2	−60.3 (6)
N2—Ni—S3—P2	−83.39 (9)	P1—O1—C1—C3	175.9 (3)
N1—Ni—S3—P2	−29.0 (4)	P1—O2—C4—C6	−101.8 (4)
S1—Ni—S3—P2	−175.61 (5)	P1—O2—C4—C5	134.1 (3)
S2—Ni—S3—P2	102.35 (5)	P2—O3—C7—C9	−115.4 (4)
S4—Ni—S3—P2	9.55 (4)	P2—O3—C7—C8	121.8 (3)
N2—Ni—S4—P2	83.57 (9)	P2—O4—C10—C11	−62.7 (4)
N1—Ni—S4—P2	163.25 (9)	P2—O4—C10—C12	174.4 (3)
S3—Ni—S4—P2	−9.46 (4)	C24—N1—C13—C14	−1.4 (6)
S2—Ni—S4—P2	−103.91 (5)	Ni—N1—C13—C14	−177.4 (3)
Ni—S1—P1—O2	107.99 (12)	N1—C13—C14—C15	0.3 (6)
Ni—S1—P1—O1	−142.74 (13)	C13—C14—C15—C16	1.5 (6)
Ni—S1—P1—S2	−14.89 (6)	C14—C15—C16—C24	−2.1 (6)
Ni—S2—P1—O2	−105.58 (12)	C14—C15—C16—C17	178.9 (4)
Ni—S2—P1—O1	142.20 (13)	C15—C16—C17—C18	179.6 (4)
Ni—S2—P1—S1	14.73 (6)	C24—C16—C17—C18	0.5 (6)
Ni—S3—P2—O3	110.69 (12)	C16—C17—C18—C19	−2.2 (6)
Ni—S3—P2—O4	−140.14 (12)	C17—C18—C19—C20	−176.7 (4)
Ni—S3—P2—S4	−12.77 (6)	C17—C18—C19—C23	2.8 (6)
Ni—S4—P2—O3	−107.73 (12)	C23—C19—C20—C21	−0.3 (6)
Ni—S4—P2—O4	141.33 (12)	C18—C19—C20—C21	179.1 (4)
Ni—S4—P2—S3	12.73 (6)	C19—C20—C21—C22	0.9 (6)
O2—P1—O1—C1	−163.2 (4)	C23—N2—C22—C21	−1.8 (6)
S1—P1—O1—C1	82.5 (4)	Ni—N2—C22—C21	177.7 (3)
S2—P1—O1—C1	−43.6 (4)	C20—C21—C22—N2	0.2 (6)
O1—P1—O2—C4	74.1 (3)	C22—N2—C23—C19	2.3 (5)
S1—P1—O2—C4	−168.0 (3)	Ni—N2—C23—C19	−177.2 (3)
S2—P1—O2—C4	−46.4 (3)	C22—N2—C23—C24	−176.8 (3)
O4—P2—O3—C7	68.5 (3)	Ni—N2—C23—C24	3.6 (4)
S3—P2—O3—C7	−172.5 (2)	C20—C19—C23—N2	−1.3 (5)
S4—P2—O3—C7	−50.0 (3)	C18—C19—C23—N2	179.2 (3)
O3—P2—O4—C10	−160.8 (3)	C20—C19—C23—C24	177.8 (3)
S3—P2—O4—C10	85.2 (3)	C18—C19—C23—C24	−1.7 (5)
S4—P2—O4—C10	−41.8 (3)	C13—N1—C24—C16	0.7 (5)
N2—Ni—N1—C13	−180.0 (3)	Ni—N1—C24—C16	177.3 (3)
S1—Ni—N1—C13	−89.1 (3)	C13—N1—C24—C23	−179.8 (3)
S3—Ni—N1—C13	124.4 (4)	Ni—N1—C24—C23	−3.1 (4)
S2—Ni—N1—C13	−7.0 (3)	C15—C16—C24—N1	1.0 (5)

S4—Ni—N1—C13	86.4 (3)	C17—C16—C24—N1	−179.9 (4)
N2—Ni—N1—C24	3.8 (2)	C15—C16—C24—C23	−178.5 (3)
S1—Ni—N1—C24	94.7 (2)	C17—C16—C24—C23	0.6 (5)
S3—Ni—N1—C24	−51.7 (6)	N2—C23—C24—N1	−0.3 (5)
S2—Ni—N1—C24	176.8 (2)	C19—C23—C24—N1	−179.5 (3)
S4—Ni—N1—C24	−89.8 (2)	N2—C23—C24—C16	179.2 (3)
N1—Ni—N2—C22	176.5 (3)	C19—C23—C24—C16	0.1 (5)
S1—Ni—N2—C22	80.3 (3)		

Fig. 1


C18
C17
C19
C16
C20
C15
C21
C14
C24
C23
C22
N2
N1
C13
C8
Ni
S1
C2
C7
S4
C1
C9
O3
P2
S3
S2
P1
O1
C3
O4
O2
C10
C4
C11
C12
C5
C6