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Bis{methyl [1-(6-acetyl-2-pyridyl)ethylidene]hydrazinecarbodithioato}nickel(II)

Mohammad Akbar Ali,[a] Aminul Huq Mirza[a] and Paul V. Bernhardt[b]*

[a]Department of Chemistry, Universiti Brunei Darussalam, Jalan Tungku Link, Brunei Darussalam, and [b]Department of Chemistry, University of Queensland, Brisbane 4072, Australia
Correspondence e-mail: p.bernhardt@uq.edu.au

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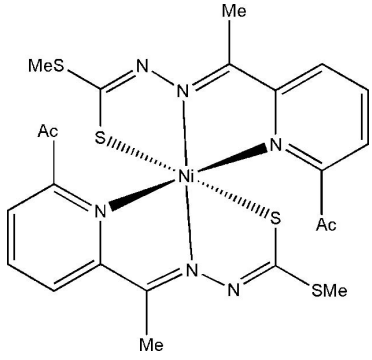
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.006 Å; R factor = 0.027; wR factor = 0.075; data-to-parameter ratio = 8.2.

The title compound, $[Ni(C_{11}H_{12}N_3OS_2)_2]$, is a six-coordinate Ni^{II} complex that is similar to other bis-tridentate N,N',S-coordinated Schiff bases, but in this case an acetyl substituent adjacent to the coordinated pyridyl N atom elongates the Ni—N bond by more than 0.06 Å relative to the complex where no substituent is present. A related 6-methyl-substituted Ni complex also shows a similar Ni—N bond elongation.

Related literature

For related literature, see: Akbar Ali, Mahbub-ul-Haque Majumder, Butcher, Jasinski & Jasinski (1997); Akbar Ali, Mirza, Keng & Butcher (2003); Akbar Ali, Mirza, Tan, Wei & Bernhardt (2004); Akbar Ali, Mirza, Voo, Tan & Bernhardt (2003); Su *et al.* (1998).



Experimental

Crystal data

$[Ni(C_{11}H_{12}N_3OS_2)_2]$
M_r = 591.42
Orthorhombic, $P2_12_12_1$
a = 10.6343 (8) Å
b = 10.8217 (7) Å
c = 22.603 (2) Å
V = 2601.2 (3) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 1.10 mm^{-1}
T = 293 (2) K
0.6 × 0.6 × 0.3 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.558, T_{max} = 0.734
2601 measured reflections
2601 independent reflections
2197 reflections with $I > 2\sigma(I)$
3 standard reflections
frequency: 120 min
intensity decay: 1%

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.027
$wR(F^2)$ = 0.075
S = 1.05
2601 reflections
316 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.36 e Å^{-3}
$\Delta\rho_{min}$ = −0.24 e Å^{-3}

Data collection: *CAD-4 EXPRESS* (Enraf–Nonius, 1994); cell refinement: *CAD-4 EXPRESS*; data reduction: *XCAD4* (Harms & Wocadlo, 1995); 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: *WinGX* (Version 1.64.02; Farrugia, 1999).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HG2264).

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supplementary materials

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Bis{methyl [1-(6-acetyl-2-pyridyl)ethylidene]hydrazinecarbodithioato}nickel(II)

M. Akbar Ali, A. H. Mirza and P. V. Bernhardt

Comment

The potentially pentadendate Schiff base ligand (I) and its close relatives have been shown to favour pentagonal bipyramidal structures when complexed with metals such Sn(IV) (Akbar Ali *et al.*, 2004), Zn(II) and Cd(II) (Akbar Ali, Mirza, Voo *et al.*, 2003). In the synthesis of (I) it has been reported that the intermediate one-armed analogue (II) is also a product of the condensation reaction between 2,6-diacetylpyridine and *S*-methyldithiocarbate (Akbar Ali, Mirza, Keng & Butcher, 2003). The Pd(II) complex of (II) was structurally characterized. Herein we report the crystal structure of the bis-ligated six-coordinate Ni^{II} complex (III).

The structure of (III) reveals a distorted octahedral coordination geometry (Fig. 1). Each ligand deprotonates at N1a/b and the C1a/b-N1/a/b bonds approach double bond order due to the preferred ene-thiolate form of the coordinated monoanionic ligand (Table 2). The ligands each bind as a tridentate *N,N,S* chelate and the planar nature of the ligand enforces a meridional coordination mode. The shortest bond lengths are the central Ni—N bonds as expected. A point of interest is the rather long Ni—N3a/b bonds in comparison with the homologous complex (IV) (av. 2.12 Å) which lacks any substituents on the 6-positions of its pyridyl rings (Su *et al.*, 1998). In this case the Ni—N bond lengths are more than 0.06 Å longer than found in the structure of (IV). Steric interactions between the non-coordinating acetyl groups and the adjacent ligand result in this bond elongation. The structure of the 6-methyl substituted analog (V) also exhibits a similar lengthening of the Ni—N_{pyr} coordinate bonds (Akbar Ali *et al.*, 1997). It is also notable that these coordinate bond distortions are localized at the pyridyl groups as the remaining Ni—N and Ni—S coordinate bonds are the same within experimental error as those reported for (IV).

In conclusion, the absence of a second chelating arm in the ligand (II) leads to a preferred bis-tridentate coordination mode in complex with Ni^{II} and the 6-acetyl substituents on the pyridyl rings play a role in lengthening the Ni—N_{pyr} bonds substantially.

Experimental

To a solution of nickel(II) chloride hexahydrate (0.03 g; 1.25 *x* 10^{-4} mol) in boiling methanol (20 ml), conc. HCl (*ca* 0.3 ml) was added. The solution was mixed with a hot solution of the Schiff base, 2,6-diacetylpyridinebis(*S*-methyldithiocarbazonate, (I)) (0.09 g; 2.5 *x* 10^{-4} mol) in methanol (50 ml) and the mixture was heated on a water bath for about one minute. On standing, the reaction mixture deposited crystals of the complex which were filtered off, washed with methanol and dried in a desiccator over anhydrous silica gel. Hydrolysis of (I) to (II) during the reaction was confirmed by isolation of its Ni^{II} complex (III).

Refinement

All H atoms were treated as riding with C—H distances ranging from 0.93 to 0.96 Å and U_{iso}(H) values equal to 1.5 (methyl H atoms) or 1.2 (all other atoms) times U_{eq} of the parent atom. Only a unique (octant) data set was measured for the orthorhombic system so the R_{int} value is undetermined. Also the Flack parameter is only nominal given the lack of Friedel pairs in the data set.

Figures

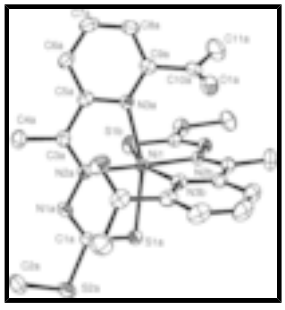

Fig. 1. *ORTEP-3* plot of (III) (30% probability ellipsoids). H-atoms have been omitted for clarity. Atom labelling on ligand B follows that shown on ligand A.

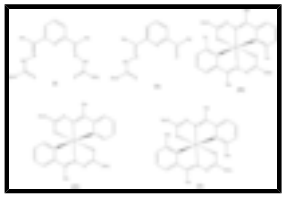

Fig. 2. The structures of compounds (I)–(V).

Bis{methyl [1-(6-acetyl-2-pyridyl)ethylidene]hydrazinecarbodithioato}nickel(II)

Crystal data

$[Ni(C_{11}H_{12}N_3O_1S_2)_2]$	F_{000} = 1224
M_r = 591.42	D_x = 1.51 Mg m^{-3}
Orthorhombic, $P2_12_12_1$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: P 2ac 2ab	Cell parameters from 25 reflections
a = 10.6343 (8) Å	θ = 11.5–13.5º
b = 10.8217 (7) Å	μ = 1.10 mm^{-1}
c = 22.603 (2) Å	T = 293 (2) K
V = 2601.2 (3) Å^3	Prism, brown
Z = 4	0.6 × 0.6 × 0.3 mm

Data collection

Enraf–Nonius CAD-4 diffractometer	R_{int} = 0
Monochromator: graphite	θ_{max} = 25.0º
T = 293(2) K	θ_{min} = 1.8º

non–profiled ω/2θ scans	$h = 0 \rightarrow 12$
Absorption correction: ψ scan (North *et al.*, 1968)	$k = 0 \rightarrow 12$
$T_{min} = 0.558$, $T_{max} = 0.734$	$l = 0 \rightarrow 26$
2601 measured reflections	3 standard reflections
2601 independent reflections	every 120 min
2197 reflections with $I > 2\sigma(I)$	intensity decay: 1%

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.027$	$w = 1/[\sigma^2(F_o^2) + (0.0488P)^2 + 0.1306P]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.075$	$(\Delta/\sigma)_{max} < 0.001$
$S = 1.05$	$\Delta\rho_{max} = 0.36$ e Å^{-3}
2601 reflections	$\Delta\rho_{min} = -0.23$ e Å^{-3}
316 parameters	Extinction correction: none
Primary atom site location: structure-invariant direct methods	Absolute structure: Flack (1983), with how many Friedel pairs?
Secondary atom site location: difference Fourier map	Flack parameter: 0.032 (17)

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 $F^2 > 2$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}
Ni1	0.73584 (4)	0.93441 (4)	0.12063 (2)	0.03305 (14)
C1A	0.7000 (4)	0.7464 (4)	0.21782 (18)	0.0388 (9)
C1B	0.5642 (4)	0.9426 (4)	0.01415 (17)	0.0409 (9)
C2A	0.6984 (6)	0.6758 (5)	0.33646 (19)	0.0660 (15)
H2A1	0.6993	0.6094	0.3646	0.099*
H2A2	0.6235	0.7241	0.3417	0.099*
H2A3	0.7709	0.7273	0.3424	0.099*
C2B	0.5101 (5)	0.9472 (6)	−0.10626 (19)	0.0710 (15)
H2B1	0.447	0.9499	−0.1366	0.107*
H2B2	0.5595	0.8735	−0.1107	0.107*

H2B3	0.5636	1.0183	−0.1096	0.107*
C3A	0.6694 (4)	1.0590 (4)	0.22846 (16)	0.0376 (9)
C3B	0.8810 (4)	0.9067 (4)	0.01403 (19)	0.0424 (10)
C4A	0.6422 (5)	1.0822 (4)	0.29212 (17)	0.0531 (11)
H4A1	0.6389	1.0049	0.3129	0.08*
H4A2	0.5629	1.1237	0.2958	0.08*
H4A3	0.7074	1.1329	0.3087	0.08*
C4B	0.9165 (5)	0.8986 (5)	−0.05007 (18)	0.0612 (14)
H4B1	0.8424	0.9058	−0.074	0.092*
H4B2	0.9563	0.8205	−0.0576	0.092*
H4B3	0.9737	0.9643	−0.0596	0.092*
C5A	0.6787 (4)	1.1619 (4)	0.18649 (19)	0.0380 (9)
C5B	0.9791 (4)	0.8978 (4)	0.06058 (19)	0.0400 (10)
C6A	0.6311 (4)	1.2788 (4)	0.1985 (2)	0.0475 (11)
H6A	0.5937	1.2954	0.2349	0.057*
C6B	1.1035 (4)	0.8744 (5)	0.0466 (2)	0.0545 (12)
H6B	1.1279	0.8658	0.0073	0.065*
C7A	0.6397 (5)	1.3698 (4)	0.1562 (2)	0.0573 (12)
H7A	0.6052	1.4474	0.1631	0.069*
C7B	1.1902 (4)	0.8640 (5)	0.0910 (2)	0.0605 (13)
H7B	1.2741	0.8479	0.0825	0.073*
C8A	0.6995 (5)	1.3451 (4)	0.1041 (2)	0.0542 (12)
H8A	0.7058	1.4055	0.0749	0.065*
C8B	1.1505 (4)	0.8780 (4)	0.1483 (2)	0.0578 (13)
H8B	1.2074	0.8697	0.1793	0.069*
C9A	0.7504 (4)	1.2290 (4)	0.09532 (18)	0.0442 (9)
C9B	1.0268 (4)	0.9041 (4)	0.15966 (19)	0.0432 (10)
C10A	0.8309 (6)	1.2056 (4)	0.0410 (2)	0.0630 (14)
C10B	0.9866 (4)	0.9276 (4)	0.22341 (19)	0.0481 (10)
C11A	0.7768 (8)	1.2393 (6)	−0.0167 (2)	0.103 (3)
H11D	0.8364	1.2215	−0.0475	0.155*
H11E	0.7571	1.3258	−0.017	0.155*
H11F	0.7014	1.1924	−0.0233	0.155*
C11B	1.0123 (6)	0.8259 (5)	0.2656 (2)	0.0741 (16)
H11A	0.9843	0.8494	0.3044	0.111*
H11B	1.101	0.8094	0.2666	0.111*
H11C	0.9683	0.7529	0.2532	0.111*
N1A	0.6847 (3)	0.8516 (3)	0.24452 (15)	0.0412 (8)
N1B	0.6739 (3)	0.9282 (3)	−0.00883 (13)	0.0403 (7)
N2A	0.6903 (3)	0.9504 (3)	0.20621 (13)	0.0355 (7)
N2B	0.7690 (3)	0.9214 (3)	0.03321 (13)	0.0361 (7)
N3A	0.7383 (3)	1.1368 (3)	0.13456 (13)	0.0353 (7)
N3B	0.9405 (3)	0.9159 (3)	0.11689 (15)	0.0378 (7)
S1A	0.71898 (12)	0.71710 (10)	0.14389 (5)	0.0465 (3)
S1B	0.52284 (9)	0.95535 (12)	0.08710 (4)	0.0471 (3)
S2A	0.70145 (14)	0.61389 (11)	0.26300 (5)	0.0592 (3)
S2B	0.43594 (11)	0.94696 (14)	−0.03511 (5)	0.0573 (3)
O1A	0.9365 (4)	1.1685 (4)	0.04757 (19)	0.0833 (13)
O1B	0.9389 (3)	1.0233 (3)	0.23694 (15)	0.0592 (9)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Ni1	0.0334 (2)	0.0345 (2)	0.0312 (2)	−0.0014 (2)	0.0013 (2)	−0.0021 (2)
C1A	0.036 (2)	0.037 (2)	0.044 (2)	−0.0014 (18)	−0.0042 (18)	0.0084 (18)
C1B	0.046 (2)	0.033 (2)	0.044 (2)	0.001 (2)	−0.0068 (18)	−0.003 (2)
C2A	0.092 (4)	0.063 (3)	0.043 (3)	−0.003 (3)	0.003 (3)	0.012 (2)
C2B	0.075 (3)	0.096 (4)	0.042 (2)	−0.009 (3)	−0.010 (2)	0.009 (3)
C3A	0.0350 (19)	0.040 (2)	0.0376 (19)	−0.003 (2)	0.0012 (16)	−0.0049 (19)
C3B	0.046 (2)	0.038 (2)	0.044 (2)	−0.0020 (19)	0.0095 (19)	−0.0023 (19)
C4A	0.068 (3)	0.052 (3)	0.039 (2)	−0.004 (3)	0.007 (2)	−0.007 (2)
C4B	0.057 (3)	0.084 (4)	0.043 (2)	0.004 (3)	0.015 (2)	−0.004 (2)
C5A	0.035 (2)	0.034 (2)	0.045 (2)	−0.0054 (17)	−0.006 (2)	−0.0063 (18)
C5B	0.038 (2)	0.033 (2)	0.049 (2)	−0.0027 (17)	0.0031 (19)	−0.0017 (18)
C6A	0.049 (3)	0.039 (2)	0.054 (3)	0.002 (2)	0.006 (2)	−0.013 (2)
C6B	0.037 (2)	0.053 (3)	0.073 (3)	−0.004 (2)	0.012 (2)	−0.006 (3)
C7A	0.061 (3)	0.032 (2)	0.079 (3)	0.005 (2)	0.002 (3)	−0.004 (2)
C7B	0.028 (2)	0.058 (3)	0.095 (4)	0.002 (2)	0.004 (2)	−0.011 (3)
C8A	0.062 (3)	0.033 (2)	0.067 (3)	0.000 (2)	0.005 (2)	0.010 (2)
C8B	0.034 (2)	0.053 (3)	0.086 (3)	0.002 (2)	−0.016 (2)	−0.010 (3)
C9A	0.045 (2)	0.040 (2)	0.048 (2)	−0.005 (2)	−0.001 (2)	0.0049 (18)
C9B	0.038 (2)	0.034 (2)	0.058 (3)	−0.0058 (18)	−0.006 (2)	−0.0061 (19)
C10A	0.097 (4)	0.033 (2)	0.060 (3)	−0.009 (3)	0.023 (3)	0.007 (2)
C10B	0.039 (2)	0.051 (3)	0.054 (2)	−0.004 (2)	−0.0140 (19)	−0.011 (2)
C11A	0.172 (8)	0.086 (4)	0.052 (3)	−0.007 (5)	0.020 (4)	0.009 (3)
C11B	0.083 (4)	0.074 (4)	0.065 (4)	0.011 (3)	−0.013 (3)	0.006 (3)
N1A	0.0463 (19)	0.0378 (19)	0.0394 (18)	−0.0045 (16)	0.0008 (16)	0.0074 (16)
N1B	0.0421 (18)	0.0453 (19)	0.0336 (16)	0.0015 (18)	−0.0038 (14)	0.0003 (17)
N2A	0.0364 (17)	0.0354 (18)	0.0347 (16)	−0.0019 (15)	−0.0012 (13)	0.0003 (14)
N2B	0.0364 (16)	0.0369 (17)	0.0351 (15)	0.0005 (17)	0.0048 (15)	−0.0003 (14)
N3A	0.0354 (17)	0.0336 (16)	0.0367 (17)	0.0007 (15)	0.0030 (15)	0.0003 (13)
N3B	0.0317 (15)	0.0347 (18)	0.0470 (18)	−0.0014 (14)	−0.0029 (15)	−0.0028 (17)
S1A	0.0627 (7)	0.0349 (5)	0.0417 (5)	−0.0032 (5)	0.0036 (5)	−0.0027 (4)
S1B	0.0327 (5)	0.0656 (8)	0.0428 (5)	−0.0014 (5)	0.0003 (4)	−0.0091 (6)
S2A	0.0878 (9)	0.0398 (6)	0.0500 (7)	−0.0036 (6)	−0.0048 (6)	0.0104 (5)
S2B	0.0481 (6)	0.0739 (9)	0.0499 (6)	0.0051 (7)	−0.0147 (5)	−0.0001 (7)
O1A	0.086 (3)	0.065 (3)	0.098 (3)	0.009 (2)	0.048 (3)	0.013 (2)
O1B	0.058 (2)	0.0513 (19)	0.069 (2)	0.0038 (17)	−0.0120 (17)	−0.0159 (17)

Geometric parameters (Å, °)

Ni1—N2A	2.002 (3)	C5A—N3A	1.361 (5)
Ni1—N2B	2.012 (3)	C5A—C6A	1.390 (6)
Ni1—N3B	2.188 (3)	C5B—N3B	1.351 (5)
Ni1—N3A	2.213 (3)	C5B—C6B	1.383 (6)
Ni1—S1B	2.399 (1)	C6A—C7A	1.375 (6)
Ni1—S1A	2.416 (1)	C6A—H6A	0.93
C1A—N1A	1.299 (5)	C6B—C7B	1.368 (7)

C1A—S1A	1.713 (4)	C6B—H6B	0.93
C1A—S2A	1.760 (4)	C7A—C8A	1.366 (6)
C1B—N1B	1.286 (5)	C7A—H7A	0.93
C1B—S1B	1.712 (4)	C7B—C8B	1.370 (7)
C1B—S2B	1.761 (4)	C7B—H7B	0.93
C2A—S2A	1.791 (5)	C8A—C9A	1.382 (6)
C2A—H2A1	0.96	C8A—H8A	0.93
C2A—H2A2	0.96	C8B—C9B	1.371 (6)
C2A—H2A3	0.96	C8B—H8B	0.93
C2B—S2B	1.791 (5)	C9A—N3A	1.342 (5)
C2B—H2B1	0.96	C9A—C10A	1.518 (7)
C2B—H2B2	0.96	C9B—N3B	1.338 (5)
C2B—H2B3	0.96	C9B—C10B	1.524 (6)
C3A—N2A	1.297 (5)	C10A—O1A	1.201 (7)
C3A—C5A	1.466 (6)	C10A—C11A	1.473 (8)
C3A—C4A	1.489 (5)	C10B—O1B	1.193 (5)
C3B—N2B	1.277 (5)	C10B—C11B	1.482 (6)
C3B—C5B	1.485 (6)	C11A—H11D	0.96
C3B—C4B	1.500 (5)	C11A—H11E	0.96
C4A—H4A1	0.96	C11A—H11F	0.96
C4A—H4A2	0.96	C11B—H11A	0.96
C4A—H4A3	0.96	C11B—H11B	0.96
C4B—H4B1	0.96	C11B—H11C	0.96
C4B—H4B2	0.96	N1A—N2A	1.377 (4)
C4B—H4B3	0.96	N1B—N2B	1.390 (4)
N2A—Ni1—N2B	176.0 (1)	C5A—C6A—H6A	120.3
N2A—Ni1—N3B	106.6 (1)	C7B—C6B—C5B	119.4 (5)
N2B—Ni1—N3B	77.4 (1)	C7B—C6B—H6B	120.3
N2A—Ni1—N3A	77.3 (1)	C5B—C6B—H6B	120.3
N2B—Ni1—N3A	102.0 (1)	C8A—C7A—C6A	119.4 (4)
N3B—Ni1—N3A	94.8 (1)	C8A—C7A—H7A	120.3
N2A—Ni1—S1B	93.92 (9)	C6A—C7A—H7A	120.3
N2B—Ni1—S1B	82.07 (9)	C6B—C7B—C8B	118.5 (4)
N3B—Ni1—S1B	159.4 (1)	C6B—C7B—H7B	120.7
N3A—Ni1—S1B	87.85 (9)	C8B—C7B—H7B	120.7
N2A—Ni1—S1A	81.7 (1)	C7A—C8A—C9A	118.9 (4)
N2B—Ni1—S1A	99.12 (9)	C7A—C8A—H8A	120.5
N3B—Ni1—S1A	89.61 (9)	C9A—C8A—H8A	120.5
N3A—Ni1—S1A	158.93 (8)	C7B—C8B—C9B	119.7 (4)
S1B—Ni1—S1A	95.20 (5)	C7B—C8B—H8B	120.2
N1A—C1A—S1A	129.0 (3)	C9B—C8B—H8B	120.2
N1A—C1A—S2A	116.5 (3)	N3A—C9A—C8A	123.0 (4)
S1A—C1A—S2A	114.5 (2)	N3A—C9A—C10A	117.7 (4)
N1B—C1B—S1B	129.2 (3)	C8A—C9A—C10A	119.3 (4)
N1B—C1B—S2B	116.8 (3)	N3B—C9B—C8B	122.8 (4)
S1B—C1B—S2B	114.1 (2)	N3B—C9B—C10B	118.4 (3)
S2A—C2A—H2A1	109.5	C8B—C9B—C10B	118.7 (4)
S2A—C2A—H2A2	109.5	O1A—C10A—C11A	123.9 (6)
H2A1—C2A—H2A2	109.5	O1A—C10A—C9A	118.9 (5)

S2A—C2A—H2A3	109.5	C11A—C10A—C9A	117.0 (5)
H2A1—C2A—H2A3	109.5	O1B—C10B—C11B	123.9 (4)
H2A2—C2A—H2A3	109.5	O1B—C10B—C9B	120.4 (4)
S2B—C2B—H2B1	109.5	C11B—C10B—C9B	115.7 (4)
S2B—C2B—H2B2	109.5	C10A—C11A—H11D	109.5
H2B1—C2B—H2B2	109.5	C10A—C11A—H11E	109.5
S2B—C2B—H2B3	109.5	H11D—C11A—H11E	109.5
H2B1—C2B—H2B3	109.5	C10A—C11A—H11F	109.5
H2B2—C2B—H2B3	109.5	H11D—C11A—H11F	109.5
N2A—C3A—C5A	115.2 (3)	H11E—C11A—H11F	109.5
N2A—C3A—C4A	124.1 (4)	C10B—C11B—H11A	109.5
C5A—C3A—C4A	120.7 (4)	C10B—C11B—H11B	109.5
N2B—C3B—C5B	115.0 (4)	H11A—C11B—H11B	109.5
N2B—C3B—C4B	124.8 (4)	C10B—C11B—H11C	109.5
C5B—C3B—C4B	120.2 (4)	H11A—C11B—H11C	109.5
C3A—C4A—H4A1	109.5	H11B—C11B—H11C	109.5
C3A—C4A—H4A2	109.5	C1A—N1A—N2A	112.5 (3)
H4A1—C4A—H4A2	109.5	C1B—N1B—N2B	113.0 (3)
C3A—C4A—H4A3	109.5	C3A—N2A—N1A	116.9 (3)
H4A1—C4A—H4A3	109.5	C3A—N2A—Ni1	119.6 (3)
H4A2—C4A—H4A3	109.5	N1A—N2A—Ni1	123.4 (2)
C3B—C4B—H4B1	109.5	C3B—N2B—N1B	117.0 (3)
C3B—C4B—H4B2	109.5	C3B—N2B—Ni1	120.4 (3)
H4B1—C4B—H4B2	109.5	N1B—N2B—Ni1	122.7 (2)
C3B—C4B—H4B3	109.5	C9A—N3A—C5A	117.8 (3)
H4B1—C4B—H4B3	109.5	C9A—N3A—Ni1	130.1 (3)
H4B2—C4B—H4B3	109.5	C5A—N3A—Ni1	108.4 (2)
N3A—C5A—C6A	121.3 (4)	C9B—N3B—C5B	117.3 (3)
N3A—C5A—C3A	115.9 (3)	C9B—N3B—Ni1	131.5 (3)
C6A—C5A—C3A	122.7 (4)	C5B—N3B—Ni1	110.6 (3)
N3B—C5B—C6B	122.2 (4)	C1A—S1A—Ni1	92.36 (14)
N3B—C5B—C3B	116.4 (3)	C1B—S1B—Ni1	93.10 (14)
C6B—C5B—C3B	121.4 (4)	C1A—S2A—C2A	103.5 (2)
C7A—C6A—C5A	119.4 (4)	C1B—S2B—C2B	103.1 (2)
C7A—C6A—H6A	120.3		

Fig. 1

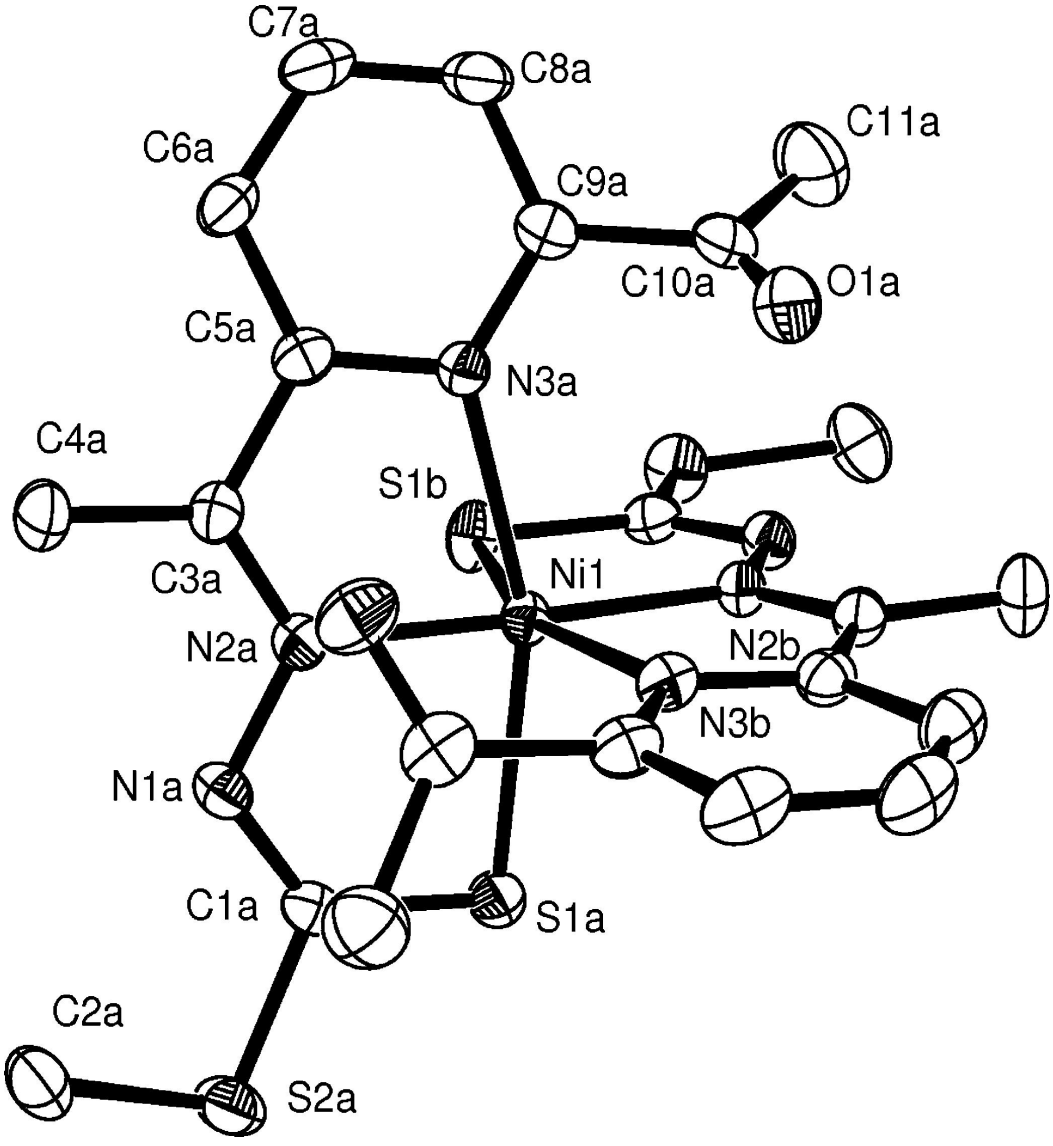
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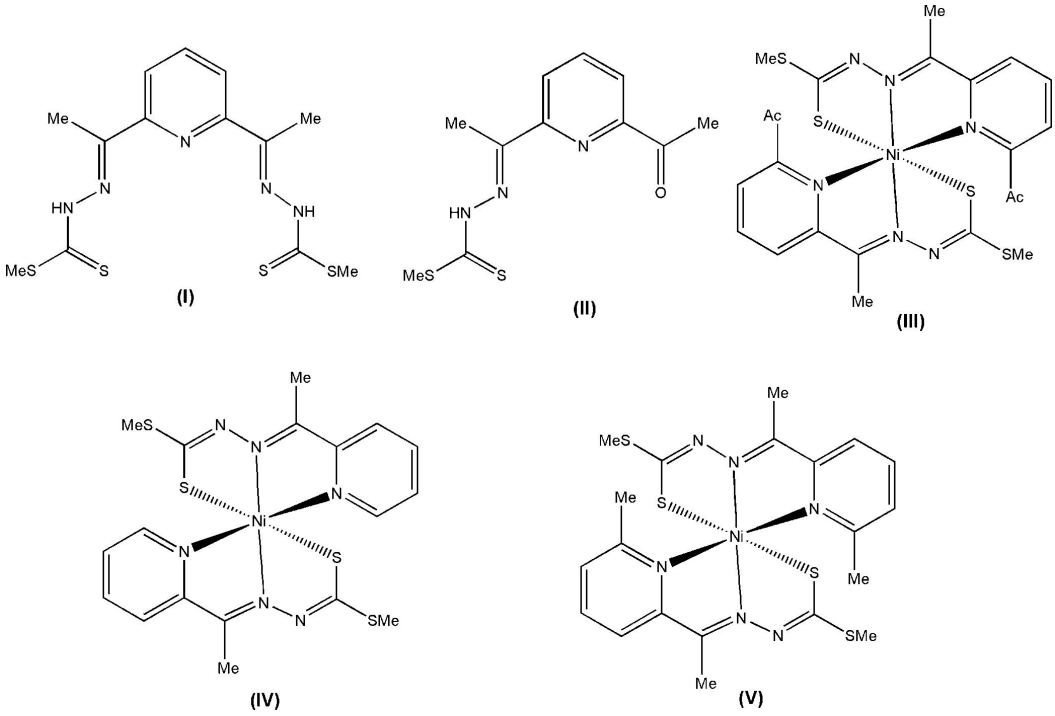
Fig. 2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