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trans-Bis(1*H*-indole-3-carbaldehyde thiosemicarbazonato-κ^2N^1,S)nickel(II)

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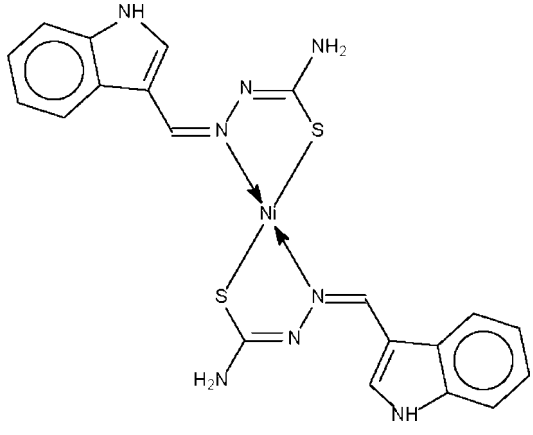
Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.003 Å; R factor = 0.034; wR factor = 0.081; data-to-parameter ratio = 15.1.

The Ni atom in the centrosymmetric title compound, $[Ni(C_{10}H_9N_4S)_2]$, is *N*,*S*-chelated by the deprotonated Schiff bases in a square-planar geometry. The –CH═N—N═C(S)—NH_2 frament is planar. Adjacent molecules are linked by hydrogen bonds between the indolyl –NH (donor) site and the double-bond ═N– (acceptor) site of an adjacent molecule, forming a layer motif.

Related literature

For the structure of the neutral Schiff base, see: Rizal *et al.* (2008). For background literature on the medicinal activity of metal complexes of the Schiff base and related compounds, see: Husain *et al.* (2007); Wilson *et al.* (2005).



Experimental

Crystal data

$[Ni(C_{10}H_9N_4S)_2]$
M_r = 493.25
Monoclinic, $P2_1/c$
a = 10.4388 (3) Å
b = 5.2604 (1) Å
c = 19.1122 (5) Å
β = 104.803 (2)°
V = 1014.66 (4) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 1.19 mm^{-1}
T = 100 (2) K
0.14 × 0.04 × 0.01 mm

Data collection

Bruker SMART APEX diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.851, T_{max} = 0.988
12357 measured reflections
2326 independent reflections
1774 reflections with $I > 2\sigma(I)$
R_{int} = 0.062

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.034
$wR(F^2)$ = 0.081
S = 1.02
2326 reflections
154 parameters
3 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.43 e Å^{-3}
$\Delta\rho_{min}$ = −0.30 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Ni1—N2	1.918 (2)	Ni1—S1	2.1669 (6)
N2—Ni1—S1	85.72 (6)	N2—Ni1—S1^{i}	94.28 (6)

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

Table 2
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1n···N3^{ii}	0.88 (3)	2.06 (2)	2.876 (3)	155 (3)

Symmetry code: (ii) $-x+1, y+\frac{1}{2}, -z+\frac{3}{2}$.

Data collection: *APEX2* (Bruker, 2007); cell refinement: *SAINT* (Bruker, 2007); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *X-SEED* (Barbour, 2001; Dolomanov *et al.*, 2003); software used to prepare material for publication: *publCIF* (Westrip, 2008).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SG2241).

References

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supplementary materials

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trans-Bis(1*H*-indole-3-carbaldehyde thiosemicarbazonato-κ^2N^1,S)nickel(II)

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Comment

A previous study reports the structure of 1*H*-indole-3-carboxaldehyde thiosemicarbazone (Rizal *et al.*, 2008). The compound in its deprotonated form can function as a bidentate chelate, and this is confirmed in the present nickel(II) derivative (Scheme I, Fig. 1). The metal center lies on a center-of-inversion in a square planar coordination geometry. Adjacent molecules are linked by hydrogen bonds between the indolyl –NH (donor) site and the double-bond =N– (acceptor) site of an adjacent molecule to form a layer motif (Fig. 2).

Experimental

Nickel acetate tetrahydrate (0.06 g,0.22 mmol) and 1*H*-indole-3-carboxaldehyde thiosemicarbazone (0.10 g, 0.44 mmol), ethanol (4 ml) and water (10 ml) were sealed in a 15-ml, Teflon-lined, Parr bomb. The bomb was heated at 383 K for 2 days. The bomb when cooled to room temperature over a day to give orange plates.

Refinement

Carbon-bound H-atoms were placed in calculated positions (C—H 0.95 Å) and were included in the refinement in the riding model approximation, with *U*(H) set to 1.2*U*(C). The nitrogen-bound H-atoms were located in a difference Fourier map, and were refined with an N–H distance restraint of 0.88±0.01 Å; their temperature factors were freely refined.

Figures

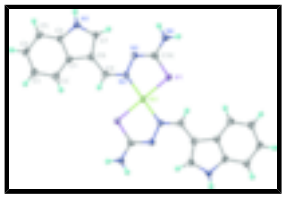

Fig. 1. Thermal ellipsoid plot of $Ni(C_{10}H_9N_4S)_2$ at the 70% probability level; hydrogen atoms are drawn as spheres of arbitrary radius. The molecule lies on a center-of-inversion. Unlabeled atoms are related to the labeled ones by this symmetry element.

Fig. 2. *OLEX* (Dolomanov *et al.*, 2003) representation of the hydrogen-bonded layer motif.

trans-Bis(1*H*-indole-3-carbaldehyde thiosemicarbazonato-κ^2N^1,S)nickel(II)

Crystal data

$[Ni(C_{10}H_9N_4S)_2]$	F_{000} = 508
M_r = 493.25	D_x = 1.614 Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation

	λ = 0.71073 Å
Hall symbol: -P 2ybc	Cell parameters from 1799 reflections
a = 10.4388 (3) Å	θ = 2.6–24.7º
b = 5.2604 (1) Å	μ = 1.19 mm^{-1}
c = 19.1122 (5) Å	*T* = 100 (2) K
β = 104.803 (2)º	Plate, orange
V = 1014.66 (4) Å^3	0.14 × 0.04 × 0.01 mm
Z = 2	

Data collection

Bruker SMART APEX diffractometer	2326 independent reflections
Radiation source: fine-focus sealed tube	1774 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.062
T = 100(2) K	θ_{max} = 27.5º
φ and ω scans	θ_{min} = 2.0º
Absorption correction: Multi-scan (SADABS; Sheldrick, 1996)	*h* = −12→13
T_{min} = 0.851, T_{max} = 0.988	*k* = −6→6
12357 measured reflections	*l* = −24→24

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.034	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2)$ = 0.081	$w = 1/[\sigma^2(F_o^2) + (0.0362P)^2 + 0.5143P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.02	$(\Delta/\sigma)_{max}$ = 0.001
2326 reflections	$\Delta\rho_{max}$ = 0.43 e Å^{-3}
154 parameters	$\Delta\rho_{min}$ = −0.30 e Å^{-3}
3 restraints	Extinction correction: none
Primary atom site location: structure-invariant direct methods	

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

	x	*y*	*z*	U_{iso}*/U_{eq}
Ni1	0.5000	0.5000	0.5000	0.01261 (12)
S1	0.33444 (6)	0.74950 (12)	0.45463 (3)	0.01747 (15)
N1	0.6654 (2)	1.1528 (4)	0.78929 (11)	0.0171 (5)
H1N	0.637 (3)	1.262 (5)	0.8165 (14)	0.045 (10)*
N2	0.52205 (19)	0.6972 (4)	0.58664 (10)	0.0143 (4)
N3	0.42554 (19)	0.8723 (4)	0.59419 (10)	0.0153 (4)
N4	0.2345 (2)	1.0700 (4)	0.53342 (12)	0.0205 (5)

H4N1	0.240 (3)	1.177 (5)	0.5691 (12)	0.042 (10)*
H4N2	0.184 (3)	1.121 (6)	0.4919 (10)	0.043 (10)*
C1	0.7786 (2)	0.8186 (5)	0.76257 (12)	0.0154 (5)
C2	0.8845 (2)	0.6471 (5)	0.77478 (13)	0.0182 (5)
H2	0.8869	0.5159	0.7410	0.022*
C3	0.9859 (2)	0.6731 (5)	0.83732 (13)	0.0195 (5)
H3	1.0588	0.5590	0.8460	0.023*
C4	0.9830 (2)	0.8646 (5)	0.88809 (13)	0.0190 (5)
H4	1.0538	0.8766	0.9306	0.023*
C5	0.8795 (2)	1.0361 (5)	0.87760 (12)	0.0178 (5)
H5	0.8774	1.1659	0.9118	0.021*
C6	0.7782 (2)	1.0092 (5)	0.81421 (12)	0.0159 (5)
C7	0.5945 (2)	1.0621 (5)	0.72458 (12)	0.0166 (5)
H7	0.5132	1.1309	0.6969	0.020*
C8	0.6586 (2)	0.8537 (5)	0.70493 (12)	0.0166 (5)
C9	0.6276 (2)	0.6972 (5)	0.64112 (12)	0.0163 (5)
H9	0.6935	0.5761	0.6381	0.020*
C10	0.3354 (2)	0.9073 (5)	0.53374 (13)	0.0159 (5)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Ni1	0.0129 (2)	0.0143 (2)	0.0104 (2)	0.00022 (19)	0.00260 (16)	−0.00047 (18)
S1	0.0180 (3)	0.0207 (3)	0.0124 (3)	0.0042 (3)	0.0014 (2)	−0.0010 (2)
N1	0.0177 (11)	0.0189 (11)	0.0140 (10)	0.0007 (9)	0.0029 (8)	−0.0039 (9)
N2	0.0161 (10)	0.0141 (10)	0.0127 (9)	0.0014 (8)	0.0036 (8)	−0.0004 (8)
N3	0.0155 (11)	0.0171 (11)	0.0137 (10)	0.0021 (9)	0.0047 (8)	−0.0002 (8)
N4	0.0225 (12)	0.0210 (12)	0.0172 (11)	0.0080 (9)	0.0035 (9)	−0.0025 (9)
C1	0.0156 (12)	0.0156 (12)	0.0151 (11)	−0.0034 (10)	0.0042 (10)	0.0002 (9)
C2	0.0190 (13)	0.0192 (13)	0.0177 (12)	−0.0019 (10)	0.0068 (10)	−0.0025 (10)
C3	0.0153 (13)	0.0225 (14)	0.0205 (12)	0.0005 (11)	0.0040 (10)	0.0028 (11)
C4	0.0166 (13)	0.0245 (14)	0.0148 (11)	−0.0037 (11)	0.0018 (10)	−0.0005 (10)
C5	0.0196 (13)	0.0207 (14)	0.0127 (11)	−0.0040 (11)	0.0034 (10)	−0.0005 (10)
C6	0.0172 (12)	0.0167 (12)	0.0151 (11)	−0.0011 (11)	0.0063 (9)	0.0013 (10)
C7	0.0166 (12)	0.0190 (14)	0.0137 (11)	−0.0016 (10)	0.0030 (10)	−0.0003 (9)
C8	0.0191 (13)	0.0177 (13)	0.0135 (11)	−0.0022 (10)	0.0051 (10)	0.0001 (10)
C9	0.0175 (12)	0.0176 (13)	0.0145 (11)	0.0008 (10)	0.0055 (10)	0.0003 (10)
C10	0.0180 (13)	0.0140 (12)	0.0182 (12)	−0.0033 (10)	0.0092 (10)	0.0007 (10)

Geometric parameters (Å, °)

Ni1—N2^{i}	1.919 (2)	C1—C6	1.408 (3)
Ni1—N2	1.918 (2)	C1—C8	1.453 (3)
Ni1—S1^{i}	2.1669 (6)	C2—C3	1.386 (3)
Ni1—S1	2.1669 (6)	C2—H2	0.9500
S1—C10	1.723 (2)	C3—C4	1.404 (4)
N1—C7	1.355 (3)	C3—H3	0.9500
N1—C6	1.377 (3)	C4—C5	1.382 (4)

N1—H1n	0.88 (3)	C4—H4	0.9500
N2—C9	1.309 (3)	C5—C6	1.397 (3)
N2—N3	1.399 (3)	C5—H5	0.9500
N3—C10	1.303 (3)	C7—C8	1.385 (3)
N4—C10	1.355 (3)	C7—H7	0.9500
N4—H4n1	0.88 (3)	C8—C9	1.438 (3)
N4—H4n2	0.88 (3)	C9—H9	0.9500
C1—C2	1.400 (3)		
$N2^{i}$—Ni1—N2	180.000 (1)	C2—C3—H3	119.3
$N2^{i}$—Ni1—$S1^{i}$	85.72 (6)	C4—C3—H3	119.3
N2—Ni1—S1	85.72 (6)	C5—C4—C3	121.5 (2)
N2—Ni1—$S1^{i}$	94.28 (6)	C5—C4—H4	119.3
$N2^{i}$—Ni1—S1	94.28 (6)	C3—C4—H4	119.3
$S1^{i}$—Ni1—S1	180.0	C4—C5—C6	116.8 (2)
C10—S1—Ni1	96.63 (9)	C4—C5—H5	121.6
C7—N1—C6	110.0 (2)	C6—C5—H5	121.6
C7—N1—H1N	126 (2)	N1—C6—C5	129.5 (2)
C6—N1—H1N	123 (2)	N1—C6—C1	107.7 (2)
C9—N2—N3	113.60 (19)	C5—C6—C1	122.9 (2)
C9—N2—Ni1	125.30 (17)	N1—C7—C8	109.7 (2)
N3—N2—Ni1	120.96 (14)	N1—C7—H7	125.1
C10—N3—N2	112.16 (19)	C8—C7—H7	125.1
C10—N4—H4N1	121 (2)	C7—C8—C9	131.6 (2)
C10—N4—H4N2	119 (2)	C7—C8—C1	106.1 (2)
H4N1—N4—H4N2	114 (3)	C9—C8—C1	122.2 (2)
C2—C1—C6	119.1 (2)	N2—C9—C8	129.5 (2)
C2—C1—C8	134.4 (2)	N2—C9—H9	115.3
C6—C1—C8	106.5 (2)	C8—C9—H9	115.3
C3—C2—C1	118.5 (2)	N3—C10—N4	118.5 (2)
C3—C2—H2	120.8	N3—C10—S1	123.44 (19)
C1—C2—H2	120.8	N4—C10—S1	118.03 (18)
C2—C3—C4	121.3 (2)		
$N2^{i}$—Ni1—S1—C10	172.73 (10)	C8—C1—C6—N1	−0.4 (3)
N2—Ni1—S1—C10	−7.27 (10)	C2—C1—C6—C5	0.1 (4)
$S1^{i}$—Ni1—N2—C9	15.3 (2)	C8—C1—C6—C5	−179.6 (2)
S1—Ni1—N2—C9	−164.7 (2)	C6—N1—C7—C8	0.3 (3)
$S1^{i}$—Ni1—N2—N3	−169.40 (16)	N1—C7—C8—C9	−177.4 (2)
S1—Ni1—N2—N3	10.60 (16)	N1—C7—C8—C1	−0.5 (3)
C9—N2—N3—C10	166.4 (2)	C2—C1—C8—C7	−179.1 (3)
Ni1—N2—N3—C10	−9.4 (3)	C6—C1—C8—C7	0.6 (3)
C6—C1—C2—C3	−0.5 (4)	C2—C1—C8—C9	−1.8 (4)
C8—C1—C2—C3	179.2 (3)	C6—C1—C8—C9	177.8 (2)
C1—C2—C3—C4	0.6 (4)	N3—N2—C9—C8	−2.0 (4)
C2—C3—C4—C5	−0.4 (4)	Ni1—N2—C9—C8	173.7 (2)
C3—C4—C5—C6	0.1 (4)	C7—C8—C9—N2	−7.0 (5)
C7—N1—C6—C5	179.2 (2)	C1—C8—C9—N2	176.5 (2)
C7—N1—C6—C1	0.1 (3)	N2—N3—C10—N4	179.1 (2)

C4—C5—C6—N1	−178.9 (2)	N2—N3—C10—S1	1.4 (3)
C4—C5—C6—C1	0.1 (4)	Ni1—S1—C10—N3	5.4 (2)
C2—C1—C6—N1	179.3 (2)	Ni1—S1—C10—N4	−172.37 (19)

Symmetry codes: (i) −*x*+1, −*y*+1, −*z*+1.

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1n···N3^{ii}	0.88 (3)	2.06 (2)	2.876 (3)	155 (3)

Symmetry codes: (ii) −*x*+1, *y*+1/2, −*z*+3/2.

Fig. 1

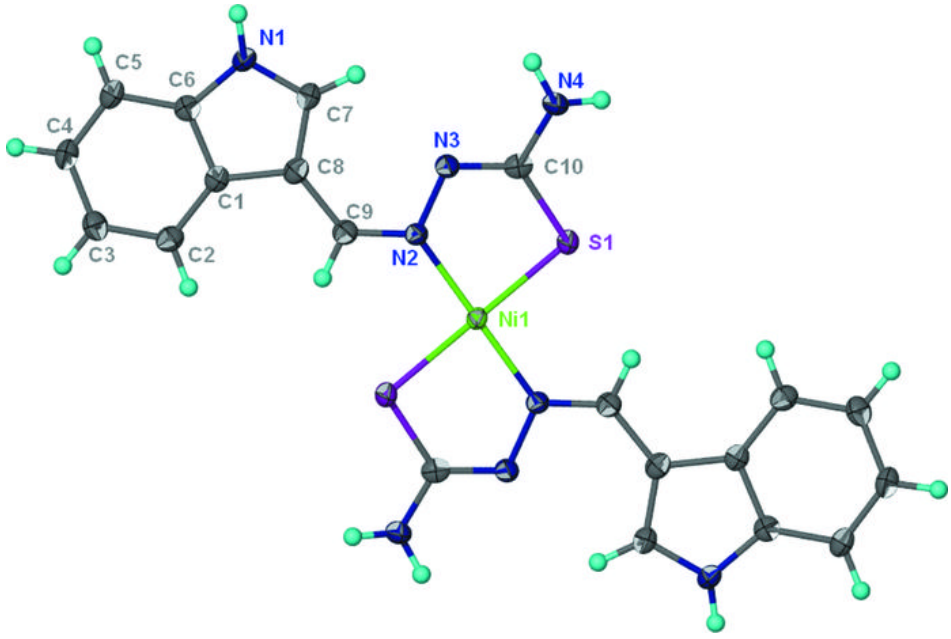

Fig. 2