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Bis{2′-[(5-chloro-1*H*-3-indolyl)methylene]-2-(1*H*-3-indolyl)acetohydrazido-κ^2N,O}nickel(II) dimethyl sulfoxide disolvate

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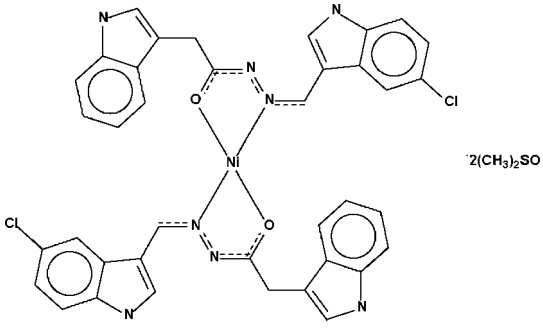
Key indicators: single-crystal X-ray study; T = 139 K; mean σ(C–C) = 0.008 Å; R factor = 0.049; wR factor = 0.198; data-to-parameter ratio = 16.8.

The Ni atom in the title compound, $[Ni(C_{19}H_{14}ClN_4O)_2]\cdot 2C_2H_6OS$, which lies on a centre of inversion, is *N*,*O*-chelated by the two monoanionic ligands in a square-planar geometry. The asymmetric unit also contains a dimethyl sulfoxide solvent molecule. The donor amino sites of the metal-bearing molecule interact with the solvent molecules to furnish a hydrogen-bonded layer structure.

Related literature

For the structure of 2′-[(5-chloro-1*H*-indol-3-yl)methylene]-2-(1*H*-indol-3-yl)acetohydrazide, see Ali *et al.* (2007).



Experimental

Crystal data

$[Ni(C_{19}H_{14}ClN_4O)_2]\cdot 2C_2H_6OS$
M_r = 914.55
Monoclinic, $P2_1/c$
a = 11.659 (1) Å
b = 10.219 (1) Å
c = 17.585 (2) Å
β = 102.272 (3)°
V = 2047.2 (3) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.76 mm^{-1}
T = 139 (2) K
0.42 × 0.05 × 0.02 mm

Data collection

Bruker APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.776, T_{max} = 0.985
13220 measured reflections
4683 independent reflections
2216 reflections with $I > 2\sigma(I)$
R_{int} = 0.108

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.049
$wR(F^2)$ = 0.198
S = 0.99
4683 reflections
278 parameters
2 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.50 e Å^{-3}
$\Delta\rho_{min}$ = −0.89 e Å^{-3}

Table 1
Selected bond lengths (Å).

Ni1—O1	1.845 (4)	Ni1—N2	1.861 (4)

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1*N*···O2	0.88 (4)	1.90 (5)	2.766 (6)	167 (6)
N4—H4*N*···O2^{i}	0.88 (3)	2.05 (3)	2.887 (6)	158 (6)

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

Data collection: *APEX2* (Bruker, 2005); cell refinement: *SAINT* (Bruker, 2005); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *X-SEED* (Barbour, 2001); software used to prepare material for publication: *publCIF* (Westrip, 2007).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SJ2417).

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supplementary materials

Acta Cryst. (2007). E**63**, m3033 [**doi:10.1107/S1600536807056966**]

Bis{2'-[(5-chloro-1*H*-3-indolyl)methylene]-2-(1*H*-3-indolyl)acetohydrazido-κ^2N,O}nickel(II) dimethyl sulfoxide disolvate

H. M. Ali, S. J. Nazzatush, M. R. Rizal and S. W. Ng

Experimental

2'-[(5-Chloro-1*H*-indol-3-yl)methylene]-2-(1*H*-indol-3-yl)acetohydrazide was synthesized by using a reported procedure (Ali *et al.*, 2007). This compound (1 g, 2.85 mmol) and nickel acetate tetrahydrate (0.35 g, 1.42 mmol) were heated in ethanol (50 ml) for 5 h. The product that separated was collected and recrystallized from DMSO.

Refinement

Carbon-bound H atoms were placed at calculated positions (C–H 0.95–0.99 Å), and were included in the refinement in the riding model approximation with U(H) set to 1.2–1.5U_{eq}(C). The amino hydrogen atom was located in a difference Fouier map, and was refined with a distance restraint of N–H 0.88±0.01 Å.

Figures

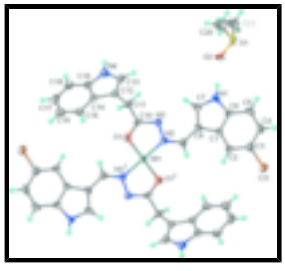


Fig. 1. Thermal ellipsoid plot of $(C_{19}H_{14}ClN_4OS)_2Ni\cdot 2DMSO$. Displacement ellipsoids are drawn at the 50% probability level, and H atoms are shown as spheres of arbitrary radii. [Symmetry code (i): $1-x, 1-y, 1-z$.]

Bis{2'-[(5-chloro-1*H*-3-indolyl)methylene]-2-(1*H*-3- indolyl)acetohydrazido-κ^2N,O}nickel(II) dimethyl sulfoxide disolvate

Crystal data

$[Ni(C_{19}H_{14}ClN_4O)_2]\cdot 2C_2H_6OS$	F_{000} = 948
M_r = 914.55	D_x = 1.484 Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2ybc	Cell parameters from 1360 reflections
a = 11.659 (1) Å	θ = 4.4–22.5°
b = 10.219 (1) Å	μ = 0.76 mm^{-1}
c = 17.585 (2) Å	T = 139 (2) K
β = 102.272 (3)°	Block, red
V = 2047.2 (3) Å^3	0.42 × 0.05 × 0.02 mm

$Z = 2$

Data collection

Bruker APEXII CCD diffractometer	4683 independent reflections
Radiation source: medium-focus sealed tube	2216 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.108$
$T = 139(2)$ K	$\theta_{max} = 27.5^{o}$
φ and ω scans	$\theta_{min} = 1.8^{o}$
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	$h = -15\rightarrow14$
$T_{min} = 0.776$, $T_{max} = 0.985$	$k = -13\rightarrow10$
13220 measured reflections	$l = -22\rightarrow22$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.049$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.198$	$w = 1/[\sigma^2(F_o^2) + (0.076P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 0.99$	$(\Delta/\sigma)_{max} = 0.001$
4683 reflections	$\Delta\rho_{max} = 0.50$ e Å^{-3}
278 parameters	$\Delta\rho_{min} = -0.89$ e Å^{-3}
2 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	0.0207 (3)
Cl1	0.77582 (14)	−0.23758 (13)	0.53762 (9)	0.0329 (4)
S1	0.21730 (14)	−0.18848 (14)	0.10978 (9)	0.0305 (4)
O1	0.3902 (3)	0.5808 (3)	0.4234 (2)	0.0240 (9)
O2	0.2384 (3)	−0.0989 (4)	0.1797 (2)	0.0315 (10)
N1	0.4210 (4)	−0.0021 (5)	0.2925 (3)	0.0263 (11)
H1N	0.369 (4)	−0.029 (6)	0.252 (2)	0.05 (2)*
N2	0.4846 (4)	0.3641 (4)	0.4281 (3)	0.0214 (10)
N3	0.4110 (4)	0.3933 (4)	0.3554 (3)	0.0242 (11)
N4	−0.0266 (5)	0.5368 (5)	0.3064 (3)	0.0289 (12)
H4N	−0.096 (2)	0.499 (5)	0.297 (4)	0.040 (19)*
C1	0.5565 (5)	0.0105 (5)	0.4043 (3)	0.0207 (12)
C2	0.6428 (5)	−0.0401 (5)	0.4652 (3)	0.0230 (13)
H2	0.6787	0.0131	0.5079	0.028*

C3	0.6737 (5)	−0.1689 (5)	0.4610 (3)	0.0262 (14)
C4	0.6230 (5)	−0.2500 (5)	0.3983 (3)	0.0276 (14)
H4	0.6474	−0.3386	0.3976	0.033*
C5	0.5385 (5)	−0.2023 (5)	0.3380 (3)	0.0276 (14)
H5	0.5041	−0.2560	0.2950	0.033*
C6	0.5055 (5)	−0.0719 (5)	0.3425 (3)	0.0230 (13)
C7	0.4171 (5)	0.1210 (5)	0.3197 (3)	0.0240 (13)
H7	0.3657	0.1877	0.2950	0.029*
C8	0.4989 (5)	0.1359 (5)	0.3887 (3)	0.0237 (13)
C9	0.5233 (5)	0.2449 (5)	0.4401 (3)	0.0226 (13)
H9	0.5739	0.2285	0.4891	0.027*
C10	0.3674 (5)	0.5089 (5)	0.3609 (3)	0.0223 (12)
C11	0.2836 (5)	0.5632 (5)	0.2906 (3)	0.0243 (13)
H11A	0.3127	0.6495	0.2774	0.029*
H11B	0.2820	0.5041	0.2458	0.029*
C12	0.1612 (5)	0.5787 (5)	0.3033 (3)	0.0224 (13)
C13	0.0737 (5)	0.4888 (5)	0.2867 (3)	0.0252 (13)
H13	0.0806	0.4049	0.2648	0.030*
C14	0.1126 (5)	0.6907 (5)	0.3346 (3)	0.0211 (12)
C15	0.1571 (5)	0.8133 (5)	0.3635 (3)	0.0267 (14)
H15	0.2351	0.8379	0.3620	0.032*
C16	0.0856 (6)	0.8971 (6)	0.3938 (3)	0.0331 (16)
H16	0.1152	0.9799	0.4134	0.040*
C17	−0.0294 (6)	0.8624 (5)	0.3964 (3)	0.0315 (15)
H17	−0.0761	0.9212	0.4187	0.038*
C18	−0.0766 (6)	0.7439 (5)	0.3671 (3)	0.0291 (14)
H18	−0.1554	0.7208	0.3675	0.035*
C19	−0.0038 (5)	0.6605 (5)	0.3371 (3)	0.0233 (13)
C20	0.1682 (6)	−0.0854 (6)	0.0285 (4)	0.0445 (18)
H20A	0.2345	−0.0345	0.0182	0.067*
H20B	0.1080	−0.0257	0.0396	0.067*
H20C	0.1349	−0.1388	−0.0171	0.067*
C21	0.0809 (6)	−0.2677 (6)	0.1102 (4)	0.0408 (17)
H21A	0.0888	−0.3214	0.1572	0.061*
H21B	0.0593	−0.3236	0.0641	0.061*
H21C	0.0198	−0.2017	0.1095	0.061*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Ni1	0.0197 (6)	0.0166 (5)	0.0232 (6)	0.0004 (4)	−0.0016 (4)	−0.0016 (4)
Cl1	0.0278 (9)	0.0262 (8)	0.0419 (9)	0.0053 (6)	0.0011 (7)	0.0021 (6)
S1	0.0267 (9)	0.0282 (8)	0.0343 (9)	0.0030 (7)	0.0017 (7)	−0.0093 (7)
O1	0.024 (2)	0.0199 (19)	0.025 (2)	0.0006 (17)	−0.0017 (18)	0.0009 (16)
O2	0.023 (2)	0.037 (2)	0.029 (2)	−0.0041 (19)	−0.0044 (19)	−0.0139 (18)
N1	0.026 (3)	0.026 (3)	0.024 (3)	−0.004 (2)	−0.002 (2)	−0.007 (2)
N2	0.016 (3)	0.023 (2)	0.025 (3)	−0.002 (2)	0.003 (2)	−0.0009 (19)
N3	0.025 (3)	0.021 (2)	0.023 (3)	0.001 (2)	−0.003 (2)	0.0013 (19)

N4	0.023 (3)	0.027 (3)	0.036 (3)	−0.006 (2)	0.002 (2)	−0.001 (2)
C1	0.019 (3)	0.018 (3)	0.025 (3)	−0.004 (2)	0.004 (2)	−0.005 (2)
C2	0.018 (3)	0.025 (3)	0.025 (3)	−0.005 (2)	0.000 (3)	−0.005 (2)
C3	0.023 (3)	0.023 (3)	0.033 (3)	0.000 (2)	0.006 (3)	0.001 (2)
C4	0.028 (4)	0.017 (3)	0.041 (4)	−0.001 (3)	0.013 (3)	−0.006 (3)
C5	0.024 (3)	0.025 (3)	0.033 (3)	−0.009 (3)	0.005 (3)	−0.010 (3)
C6	0.015 (3)	0.027 (3)	0.024 (3)	−0.004 (2)	−0.001 (3)	0.000 (2)
C7	0.026 (3)	0.022 (3)	0.023 (3)	−0.006 (2)	0.001 (3)	−0.001 (2)
C8	0.025 (3)	0.021 (3)	0.023 (3)	−0.002 (2)	0.001 (3)	−0.004 (2)
C9	0.022 (3)	0.019 (3)	0.025 (3)	−0.002 (2)	0.003 (3)	0.000 (2)
C10	0.017 (3)	0.027 (3)	0.021 (3)	−0.004 (2)	−0.001 (2)	0.002 (2)
C11	0.024 (3)	0.025 (3)	0.021 (3)	0.003 (3)	−0.003 (3)	0.000 (2)
C12	0.023 (3)	0.022 (3)	0.019 (3)	0.000 (2)	−0.002 (3)	0.004 (2)
C13	0.023 (3)	0.024 (3)	0.027 (3)	0.001 (3)	0.002 (3)	0.002 (2)
C14	0.021 (3)	0.021 (3)	0.018 (3)	−0.002 (2)	−0.001 (2)	0.005 (2)
C15	0.026 (3)	0.024 (3)	0.026 (3)	−0.003 (3)	−0.003 (3)	0.004 (2)
C16	0.041 (4)	0.022 (3)	0.032 (4)	−0.002 (3)	−0.002 (3)	0.001 (3)
C17	0.034 (4)	0.028 (3)	0.033 (4)	0.010 (3)	0.007 (3)	0.003 (3)
C18	0.024 (3)	0.031 (3)	0.031 (3)	0.002 (3)	0.003 (3)	0.007 (3)
C19	0.026 (3)	0.020 (3)	0.022 (3)	−0.002 (2)	0.001 (3)	0.006 (2)
C20	0.058 (5)	0.044 (4)	0.030 (4)	−0.012 (4)	0.007 (4)	−0.003 (3)
C21	0.042 (4)	0.040 (4)	0.036 (4)	−0.017 (3)	−0.001 (3)	−0.001 (3)

Geometric parameters (Å, °)

Ni1—O1	1.845 (4)	C7—C8	1.382 (7)
Ni1—O1^{i}	1.845 (4)	C7—H7	0.9500
Ni1—N2^{i}	1.861 (4)	C8—C9	1.425 (7)
Ni1—N2	1.861 (4)	C9—H9	0.9500
Cl1—C3	1.745 (6)	C10—C11	1.510 (7)
S1—O2	1.510 (4)	C11—C12	1.499 (8)
S1—C20	1.769 (6)	C11—H11A	0.9900
S1—C21	1.786 (6)	C11—H11B	0.9900
O1—C10	1.301 (6)	C12—C13	1.358 (7)
N1—C7	1.351 (6)	C12—C14	1.438 (7)
N1—C6	1.372 (7)	C13—H13	0.9500
N1—H1N	0.88 (4)	C14—C19	1.402 (8)
N2—C9	1.300 (6)	C14—C15	1.408 (7)
N2—N3	1.413 (6)	C15—C16	1.379 (8)
N3—C10	1.297 (6)	C15—H15	0.9500
N4—C19	1.378 (7)	C16—C17	1.398 (8)
N4—C13	1.378 (7)	C16—H16	0.9500
N4—H4N	0.88 (3)	C17—C18	1.383 (8)
C1—C2	1.403 (7)	C17—H17	0.9500
C1—C6	1.403 (7)	C18—C19	1.384 (8)
C1—C8	1.446 (7)	C18—H18	0.9500
C2—C3	1.370 (7)	C20—H20A	0.9800
C2—H2	0.9500	C20—H20B	0.9800
C3—C4	1.405 (8)	C20—H20C	0.9800

C4—C5	1.374 (8)	C21—H21A	0.9800
C4—H4	0.9500	C21—H21B	0.9800
C5—C6	1.395 (7)	C21—H21C	0.9800
C5—H5	0.9500		
O1—Ni1—O1^{i}	180.000 (1)	C8—C9—H9	116.1
O1—Ni1—N2^{i}	96.52 (17)	N3—C10—O1	124.4 (5)
O1^{i}—Ni1—N2^{i}	83.48 (17)	N3—C10—C11	118.0 (5)
O1—Ni1—N2	83.48 (17)	O1—C10—C11	117.6 (5)
O1^{i}—Ni1—N2	96.52 (17)	C12—C11—C10	112.9 (5)
N2^{i}—Ni1—N2	180.000 (1)	C12—C11—H11A	109.0
O2—S1—C20	105.4 (3)	C10—C11—H11A	109.0
O2—S1—C21	105.2 (3)	C12—C11—H11B	109.0
C20—S1—C21	97.9 (3)	C10—C11—H11B	109.0
C10—O1—Ni1	110.4 (3)	H11A—C11—H11B	107.8
C7—N1—C6	109.3 (5)	C13—C12—C14	106.4 (5)
C7—N1—H1N	121 (4)	C13—C12—C11	126.3 (5)
C6—N1—H1N	129 (4)	C14—C12—C11	127.3 (5)
C9—N2—N3	117.9 (4)	C12—C13—N4	110.1 (5)
C9—N2—Ni1	127.5 (4)	C12—C13—H13	124.9
N3—N2—Ni1	114.2 (3)	N4—C13—H13	124.9
C10—N3—N2	107.2 (4)	C19—C14—C15	118.0 (5)
C19—N4—C13	108.8 (5)	C19—C14—C12	107.4 (5)
C19—N4—H4N	125 (4)	C15—C14—C12	134.6 (5)
C13—N4—H4N	126 (4)	C16—C15—C14	119.1 (6)
C2—C1—C6	119.1 (5)	C16—C15—H15	120.5
C2—C1—C8	134.5 (5)	C14—C15—H15	120.5
C6—C1—C8	106.3 (5)	C15—C16—C17	121.2 (6)
C3—C2—C1	117.8 (5)	C15—C16—H16	119.4
C3—C2—H2	121.1	C17—C16—H16	119.4
C1—C2—H2	121.1	C18—C17—C16	121.2 (6)
C2—C3—C4	122.5 (5)	C18—C17—H17	119.4
C2—C3—Cl1	119.3 (4)	C16—C17—H17	119.4
C4—C3—Cl1	118.2 (4)	C17—C18—C19	117.0 (6)
C5—C4—C3	120.6 (5)	C17—C18—H18	121.5
C5—C4—H4	119.7	C19—C18—H18	121.5
C3—C4—H4	119.7	N4—C19—C18	129.2 (6)
C4—C5—C6	117.1 (5)	N4—C19—C14	107.3 (5)
C4—C5—H5	121.4	C18—C19—C14	123.5 (5)
C6—C5—H5	121.4	S1—C20—H20A	109.5
N1—C6—C5	129.0 (5)	S1—C20—H20B	109.5
N1—C6—C1	108.2 (5)	H20A—C20—H20B	109.5
C5—C6—C1	122.8 (5)	S1—C20—H20C	109.5
N1—C7—C8	110.2 (5)	H20A—C20—H20C	109.5
N1—C7—H7	124.9	H20B—C20—H20C	109.5
C8—C7—H7	124.9	S1—C21—H21A	109.5
C7—C8—C9	130.6 (5)	S1—C21—H21B	109.5
C7—C8—C1	105.9 (5)	H21A—C21—H21B	109.5
C9—C8—C1	123.4 (5)	S1—C21—H21C	109.5

N2—C9—C8	127.8 (5)	H21A—C21—H21C	109.5
N2—C9—H9	116.1	H21B—C21—H21C	109.5
N2^{i}—Ni1—O1—C10	175.5 (4)	Ni1—N2—C9—C8	170.9 (5)
N2—Ni1—O1—C10	−4.5 (4)	C7—C8—C9—N2	−10.9 (11)
O1—Ni1—N2—C9	−167.7 (5)	C1—C8—C9—N2	173.1 (6)
O1^{i}—Ni1—N2—C9	12.3 (5)	N2—N3—C10—O1	0.0 (7)
O1—Ni1—N2—N3	4.7 (3)	N2—N3—C10—C11	−179.2 (4)
O1^{i}—Ni1—N2—N3	−175.3 (3)	Ni1—O1—C10—N3	3.8 (7)
C9—N2—N3—C10	169.3 (5)	Ni1—O1—C10—C11	−177.0 (4)
Ni1—N2—N3—C10	−3.8 (5)	N3—C10—C11—C12	113.6 (6)
C6—C1—C2—C3	0.3 (8)	O1—C10—C11—C12	−65.7 (6)
C8—C1—C2—C3	177.8 (6)	C10—C11—C12—C13	−91.7 (7)
C1—C2—C3—C4	0.5 (9)	C10—C11—C12—C14	88.7 (7)
C1—C2—C3—Cl1	−177.1 (4)	C14—C12—C13—N4	−0.6 (6)
C2—C3—C4—C5	−0.4 (9)	C11—C12—C13—N4	179.8 (5)
Cl1—C3—C4—C5	177.3 (5)	C19—N4—C13—C12	−0.5 (6)
C3—C4—C5—C6	−0.6 (9)	C13—C12—C14—C19	1.4 (6)
C7—N1—C6—C5	179.2 (6)	C11—C12—C14—C19	−178.9 (5)
C7—N1—C6—C1	0.5 (7)	C13—C12—C14—C15	178.9 (6)
C4—C5—C6—N1	−177.1 (6)	C11—C12—C14—C15	−1.4 (10)
C4—C5—C6—C1	1.4 (9)	C19—C14—C15—C16	1.0 (8)
C2—C1—C6—N1	177.5 (5)	C12—C14—C15—C16	−176.3 (6)
C8—C1—C6—N1	−0.7 (6)	C14—C15—C16—C17	0.0 (8)
C2—C1—C6—C5	−1.3 (9)	C15—C16—C17—C18	−1.4 (9)
C8—C1—C6—C5	−179.4 (5)	C16—C17—C18—C19	1.6 (8)
C6—N1—C7—C8	−0.2 (7)	C13—N4—C19—C18	−177.5 (5)
N1—C7—C8—C9	−176.8 (6)	C13—N4—C19—C14	1.4 (6)
N1—C7—C8—C1	−0.2 (7)	C17—C18—C19—N4	178.2 (5)
C2—C1—C8—C7	−177.2 (6)	C17—C18—C19—C14	−0.5 (8)
C6—C1—C8—C7	0.6 (6)	C15—C14—C19—N4	−179.7 (5)
C2—C1—C8—C9	−0.3 (10)	C12—C14—C19—N4	−1.7 (6)
C6—C1—C8—C9	177.4 (5)	C15—C14—C19—C18	−0.8 (8)
N3—N2—C9—C8	−1.3 (9)	C12—C14—C19—C18	177.3 (5)

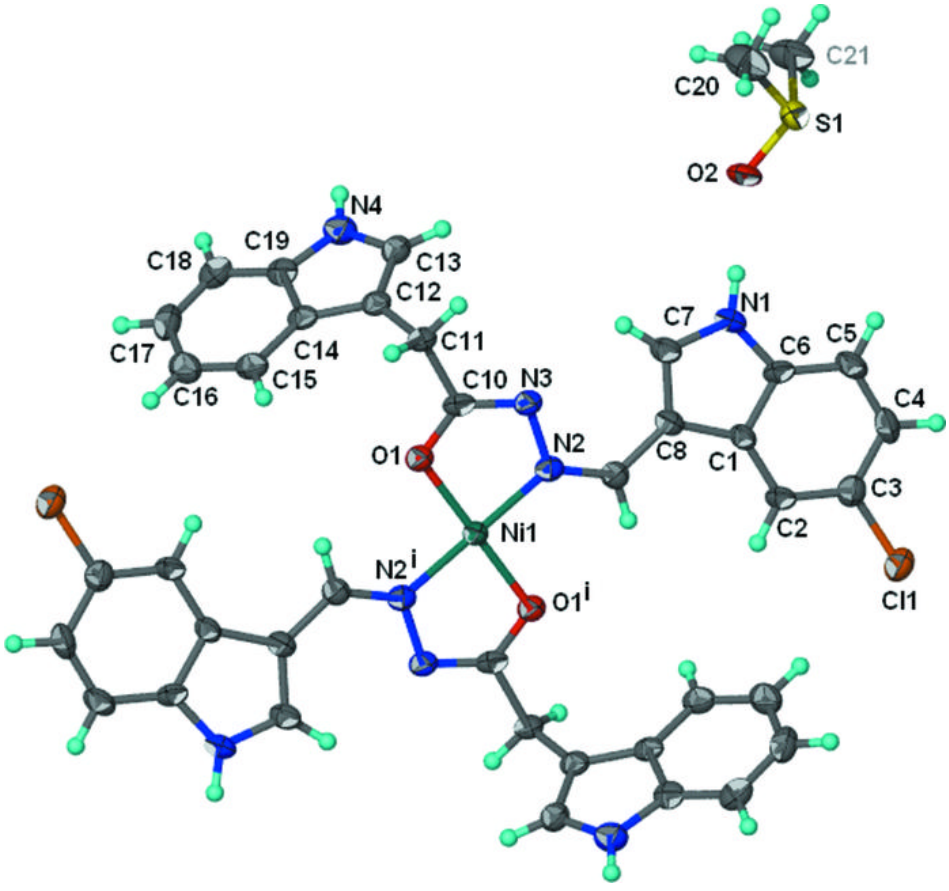
Symmetry codes: (i) −*x*+1, −*y*+1, −*z*+1.

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1N···O2	0.88 (4)	1.90 (5)	2.766 (6)	167 (6)
N4—H4N···O2^{ii}	0.88 (3)	2.05 (3)	2.887 (6)	158 (6)

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

Fig. 1


C21
C20
S1
O2
N4
C19
C18
C13
C12
C17
C14
C11
C16
C15
C10
N3
O1
N2
C7
N1
C6
C5
C4
C8
C1
C3
C2
Ni1
N2i
O1i
Cl1