

***trans*-Bis{2-[(3-cyclohexylaminopropyl-imino)methyl]phenolato}dithiocyanato-nickel(II)**

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Key indicators

Single-crystal X-ray study

$T = 298\text{ K}$

Mean $\sigma(\text{C}-\text{C}) = 0.007\text{ \AA}$

R factor = 0.045

wR factor = 0.145

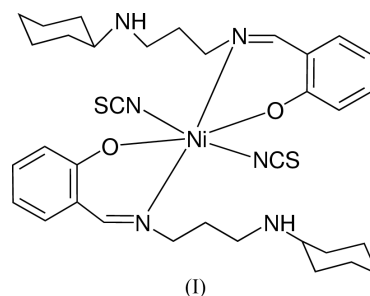
Data-to-parameter ratio = 14.8

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

In the title compound, $[\text{Ni}(\text{C}_{16}\text{H}_{23}\text{N}_2\text{O})_2(\text{NCS})_2]$, a mononuclear complex possessing C_i symmetry, the Ni^{II} atom is coordinated by six atoms from two Schiff bases and two thiocyanate anions. In the crystal structure, molecules are held together by intermolecular $\text{N}-\text{H}\cdots\text{S}$ hydrogen bonds, which link the molecules along the b axis to form one-dimensional chains.

Comment

The title compound, (I), is a discrete electronically neutral centrosymmetric mononuclear nickel(II) complex (Fig. 1). The central Ni atom is in an octahedral geometry and coordinates to two Schiff base ligands and two thiocyanate groups. The Schiff base acts as a bidentate ligand and ligates to atom Ni1 through the phenolate O and imine N atoms. The thiocyanate anion is a monodentate ligand and coordinates to the metal *via* the N atom. The three diagonal angles for the nickel(II) octahedron are all 180° , from symmetry, and all other angles around atom Ni1 are close to 90° , varying from $87.27(14)$ to $92.73(14)^\circ$, which indicates a slightly distorted octahedral geometry around the Ni atom. The Ni1–O1 (phenolate O atom) bond length of $2.030(3)\text{ \AA}$ is comparable to the value of $2.013(5)\text{ \AA}$ observed in a salicylaldehyde nickel(II) complex (Steward *et al.*, 1961). The Ni1–N1 bond distance of $2.078(3)\text{ \AA}$ (imine N atom) is close to the value of $2.068(3)\text{ \AA}$ observed in the complex $[\text{Ni}_2(\text{tp})(\text{pren})_4(\text{Him})_2](\text{ClO}_4)_2$ (Zhu *et al.*, 2001), (where tp is terephthalate, pren is 1,3-diaminopropane and Him is imidazole). The NCS angle of $178.7(4)^\circ$ in the coordinated thiocyanate anions is comparable to that observed in other nickel(II) complexes with NCS^- anions.



In the crystal structure, the molecules are connected by intermolecular $\text{N}2-\text{H}2\cdots\text{S}1^i$ hydrogen bonds (see Fig. 2 and Table 1) to form columns parallel to the b axis (Fig. 3). As expected, the cyclohexyl groups in the complex adopt chair conformations to minimize steric effects.

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Experimental

N-cyclohexyl-1,3-diaminopropane and salicylaldehyde were available commercially and were used without further purification. *N*-cyclohexyl-1,3-diaminopropane (2.0 mmol, 312 mg) and salicylaldehyde (2.0 mmol, 244 mg) were dissolved in methanol (10 ml). The mixture

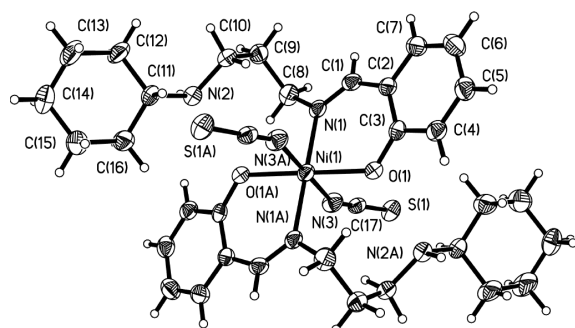


Figure 1
The structure of (I), showing 50% probability displacement ellipsoids and the atom-numbering scheme. [Symmetry code: (A) $1 - x, 1 - y, -z$.]

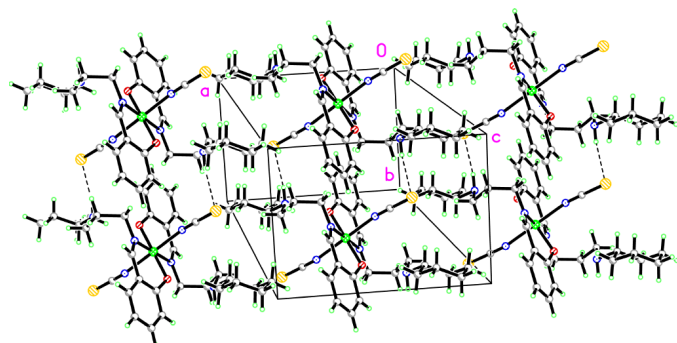


Figure 2
The crystal packing of (I), showing the $N-H \cdots S$ hydrogen-bonding interactions as dashed lines.

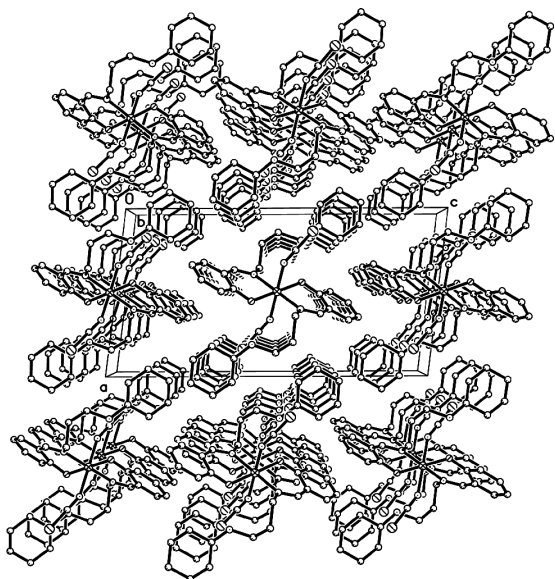


Figure 3
The crystal packing of (I), viewed along the *b* axis. H atoms have been omitted for clarity.

was stirred for *ca* 30 min to obtain a clear yellow solution of *L* (2.0 mmol), where *L* is 2-[(3-cyclohexylaminopropylimino)methyl]phenol. To the solution of *L* was added a solution of $Ni(NCS)_2$ (1.0 mmol, 175 mg) in pyridine, with stirring. After keeping the resulting solution in air for 5 d, petal-like green crystals were formed on slow evaporation of the solvents. The crystals were isolated, washed with methanol three times and dried in a vacuum desiccator using $CaCl_2$ (yield 52%). Analysis found: C 59.06, H 6.76, N 12.10%; calculated for $C_{34}H_{46}N_6NiO_2S_2$: C 58.88, H 6.68, N 12.12%.

Crystal data

$[Ni(C_{16}H_{23}N_2O)_2(NCS)_2]$
 $M_r = 693.60$
Monoclinic, $P2_1/c$
 $a = 10.906$ (7) Å
 $b = 7.796$ (5) Å
 $c = 20.721$ (13) Å
 $\beta = 97.131$ (11)°
 $V = 1748.0$ (18) Å³
 $Z = 2$

$D_x = 1.318$ Mg m⁻³
Mo $K\alpha$ radiation
Cell parameters from 1432 reflections
 $\theta = 2.6$ – 18.5°
 $\mu = 0.71$ mm⁻¹
 $T = 298$ (2) K
Petal-like, green
0.24 × 0.22 × 0.17 mm

Data collection

Bruker SMART CCD area-detector diffractometer
 φ and ω scans
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)
 $T_{min} = 0.847$, $T_{max} = 0.888$
8924 measured reflections

3085 independent reflections
1857 reflections with $I > 2\sigma(I)$
 $R_{int} = 0.050$
 $\theta_{max} = 25.0^\circ$
 $h = -12 \rightarrow 12$
 $k = -9 \rightarrow 9$
 $l = -14 \rightarrow 24$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.045$
 $wR(F^2) = 0.145$
 $S = 0.94$
3085 reflections
209 parameters

H atoms treated by a mixture of independent and constrained refinement
 $w = 1/[\sigma^2(F_o^2) + (0.0718P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{max} < 0.001$
 $\Delta\rho_{max} = 0.48$ e Å⁻³
 $\Delta\rho_{min} = -0.50$ e Å⁻³

Table 1

Hydrogen-bonding geometry (Å, °).

<i>D</i> – <i>H</i> ⋯ <i>A</i>	<i>D</i> – <i>H</i>	<i>H</i> ⋯ <i>A</i>	<i>D</i> ⋯ <i>A</i>	<i>D</i> – <i>H</i> ⋯ <i>A</i>
N2–H2⋯S1 ⁱ	0.922 (10)	2.433 (12)	3.348 (4)	171 (3)

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

The amino H atom was located from a difference Fourier synthesis and refined isotropically. All the C–H atoms were placed in idealized positions and constrained to ride on their parent atoms, with C–H distances of 0.97 Å and $U_{iso}(H)$ values equal to $1.2U_{eq}$ of the parent C atom.

Data collection: SMART (Siemens, 1996); cell refinement: SMART; data reduction: SHELXTL (Bruker, 1997); program(s) used to solve structure: SHELXS97 (Sheldrick, 1997); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: SHELXTL; software used to prepare material for publication: SHELXTL.

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