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trans-Bis(diethanolamine)bis(isothiocyanato)nickel(II)

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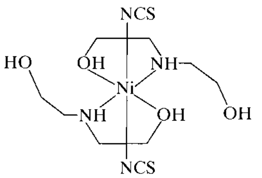
In the neutral title complex, *trans*-bis(2,2′-iminodiethanol-*N*,*O*)bis(isothiocyanato)nickel(II), $[Ni(NCS)_2(C_4H_{11}NO_2)_2]$, the isothiocyanate ions and the diethanolamine molecules act as monodentate and bidentate ligands, respectively. The Ni^{II} ion exhibits a distorted octahedral configuration with crystallographically imposed inversion symmetry and N_{NCS}—Ni—N_{amine} and N_{NCS}—Ni—O_{amine} bond angles of 88.78 (10) and 89.44 (10)°, respectively. The Ni—N bond distances are in the range 2.069 (3)–2.096 (2) Å. The molecules are linked by hydrogen bonds to form a three-dimensional infinite lattice.

Comment

As part of our study on the synthesis and spectral and thermal analysis of transition metal complexes of ethanolamines (İçbudak *et al.*, 1995; Karadag & Yilmaz, 2000), we describe here the structure of the title complex, (I), resulting from the reaction of diethanolamine with KSCN and $NiSO_4·6H_2O$.



(I)

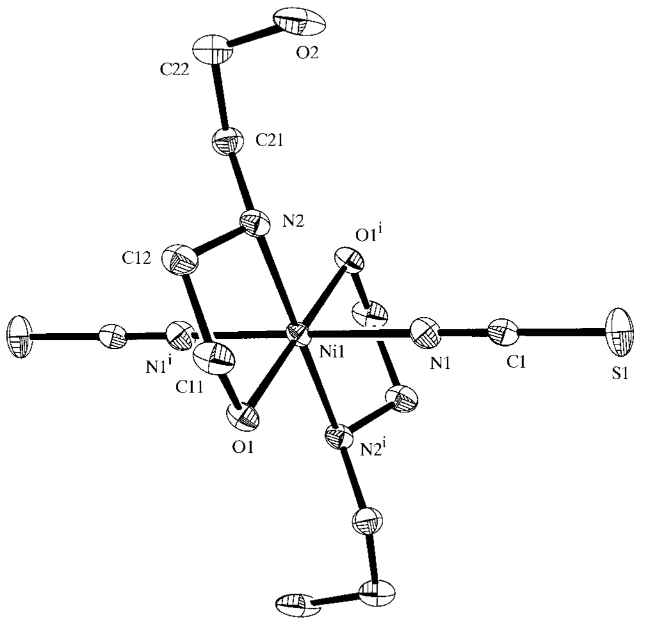


Figure 1
The structure of the title complex with the atom-numbering scheme. Displacement ellipsoids are shown at the 50% probability level. H atoms have been omitted for clarity. [Symmetry code: (i) $-x$, $1-y$, $1-z$.]

The Ni^{II} ion occupies a crystallographic inversion centre and is octahedrally coordinated by two mutually *trans* diethanolamine molecules and two mutually *trans* isocyanate ions (Fig. 1). Each diethanolamine acts as a bidentate donor through its N and one O atom (O1); the other ethanolic group containing O2 does not participate in coordination. The coordinating N and O atoms of diethanolamine are positioned in the equatorial plane of the octahedron, while the monodentate isothiocyanate ions occupy the axial positions. The Ni—N_{amine} bonds [2.096 (3) Å] are somewhat longer than the Ni—N_{NCS} bonds [2.069 (3) Å] and these Ni—N bond distances are comparable with those found in the analogous monoethanolamine Ni^{II} complex [2.085 (5) and 2.065 (5) Å, respectively; Hursthouse *et al.*, 1990]. The Ni—O distances in the title complex are 2.076 (2) Å and are comparable with those in the related di- and triethanolamine complexes of Ni^{II} reported by Hursthouse *et al.* (1990) and İçbudak *et al.* (1995) of 2.094 (4) and 2.068 (1) Å, respectively.

The N_{NCS}—Ni—N_{amine}, N_{NCS}—Ni—O and N_{amine}—Ni—O bond angles of 91.22 (10), 89.44 (10) and 83.16 (9)°, respectively, indicate significant distortion from ideally octahedral coordination geometry around the Ni^{II} atom. The NCS groups are almost linear, but significant bending is observed in the

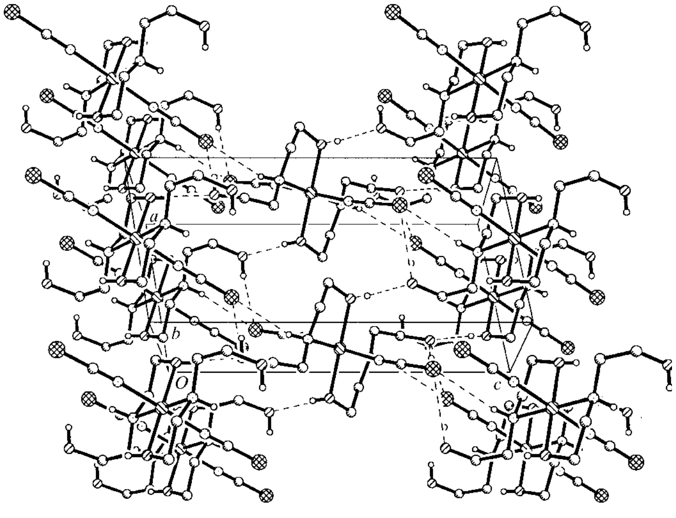


Figure 2
A view along the *b* direction showing the hydrogen bonding. H atoms not involved in hydrogen bonding have been omitted.

Ni—N—C(—S) linkage [Ni—N1—C1 173.4 (3)°]. The NCS, NH and free OH groups are involved in intermolecular hydrogen bonding. The H atoms of the NH and uncoordinated OH groups form hydrogen bonds with the S atoms of adjacent molecules, and the H atoms of coordinated OH groups also participate in hydrogen bonding with the H atom of the free OH groups of other molecules. The individual molecules are thus linked by hydrogen bonds to form a three-dimensional infinite network, part of which is shown in Fig. 2. Similar hydrogen bonding has been reported for other ethanolamine compounds (Yilmaz *et al.*, 1996, 1997).

Experimental

Solid KSCN (1.94 g, 20 mmol) was added slowly with continuous stirring to a solution of $NiSO_4{\cdot}6H_2O$ (2.63 g, 10 mmol) in distilled water (15 ml). Diethanolamine (2.10 g, 20 mmol) was dissolved in distilled water (20 ml) and the two solutions were mixed and stirred for 2 h. A pale-green polycrystalline precipitate was filtered off and blue crystals suitable for X-ray diffraction analysis were obtained by slow evaporation from the resulting solution at room temperature.

Crystal data

$[Ni(NCS)_2(C_4H_{11}NO_2)_2]$
$M_r = 385.15$
Monoclinic, $P2_1/n$
$a = 6.8157\ (8)$ Å
$b = 8.3508\ (10)$ Å
$c = 14.725\ (2)$ Å
$\beta = 100.119\ (10)°$
$V = 825.08\ (18)$ Å^3
$Z = 2$
$D_x = 1.550$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 62 reflections
$\theta = 4.0$–$12.5°$
$\mu = 1.447$ mm^{-1}
$T = 173\ (2)$ K
Square prism, blue
0.30 × 0.16 × 0.14 mm

Data collection

Siemens *P*4 diffractometer
ω scans
Absorption correction: ψ scan (*XEMP*; Siemens, 1994)
$T_{\min} = 0.773$, $T_{\max} = 0.798$
1568 measured reflections
1568 independent reflections
1072 reflections with $I > 2\sigma(I)$
$\theta_{\max} = 27.50°$
$h = -7 \rightarrow 7$
$k = -10 \rightarrow 10$
$l = -18 \rightarrow 18$
3 standard reflections every 247 reflections intensity decay: none

Table 1
Selected geometric parameters (Å, °).

Ni1—N1	2.069 (3)	N2—C12	1.480 (4)
Ni1—N2	2.096 (2)	N1—C1	1.159 (4)
Ni1—O1	2.076 (2)	C21—C22	1.518 (4)
S1—C1	1.647 (3)	C12—C11	1.522 (4)
O1—C11	1.438 (4)	C22—O2	1.432 (4)
N2—C21	1.476 (4)		
N1—Ni1—N2	88.78 (10)	Ni1—N2—C12	106.34 (18)
N1—Ni1—O1	89.44 (10)	Ni1—N1—C1	173.4 (3)
N2—Ni1—O1	83.16 (9)	N1—C1—S1	178.5 (3)
Ni1—O1—C11	109.89 (18)	O2—C22—C21	110.0 (3)
C21—N2—C12	114.5 (3)	O1—C11—C12	110.0 (3)
Ni1—N2—C21	116.5 (2)		

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.034$
$wR(F^2) = 0.071$
$S = 0.929$
1568 reflections
110 parameters
H atom treatment: see below
$w = 1/[\sigma^2(F_o^2) + (0.0316P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.35$ e Å^{-3}
$\Delta\rho_{\min} = -0.36$ e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···O2^{i}	0.72 (4)	1.93 (4)	2.651 (3)	174 (5)
N2—H2···S1^{ii}	0.88 (3)	2.81 (3)	3.630 (3)	156 (3)
O2—H3···S1^{ii}	0.83 (4)	2.45 (4)	3.225 (3)	156 (4)

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

H atoms bonded to O and N atoms were identified from difference syntheses and were refined freely. Other H atoms were included using a riding model (Sheldrick, 1997). The crystal is a non-merohedral twin. The twin matrix was determined as $(100/0\bar{1}0/\bar{\tfrac{3}{4}}0\bar{1})$ and the refined value of −0.7592 rather than −0.75 was derived from *XPREP* using the equation $c(\text{new}) = (2c\cos\beta)/a - c(\text{old})$ to obtain the exact cell parameters. Reflections with $h = 4$ and 8 show partial overlapping and were therefore omitted. Reflections with $h = 0$ were split into two components with a refined *BASF* value of 0.216 (4). The resulting data set was read using *HKLF*5 (Sheldrick, 1997).

Data collection: *XSCANS* (Fait, 1991); cell refinement: *XSCANS*; data reduction: *XSCANS*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1990); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *XP* (Siemens, 1994); software used to prepare material for publication: *SHELXL*97.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: BM1413). Services for accessing these data are described at the back of the journal.

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