Rozenberg & Lubovskaya, 1980). Furthermore, the S(1)—C(8) and S(3)—C(9) bonds are slightly longer than S(1)—C(2) and S(3)—C(2) as predicted theoretically (Haddon, 1975). The $Cu_2Cl_6^{2-}$ anion has two types of Cu—Cl bond, 2·224 (1) and 2·297 (1) Å, which are comparable with those [2·267 (3) and 2·314 (3) Å] of $K_2Cu_2Cl_6$ (Willett, Dwiggins, Kruh & Rundle, 1963).

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Bis(3-aminopropanol-*O*,*N*)bis(isothiocyanato)nickel(II), $[Ni(NCS)_2(C_3H_9NO)_2]$

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Abstract. $M_r = 325{\cdot}1$, monoclinic, $P2_1/n$, $a = 8{\cdot}869\ (2)$, $b = 8{\cdot}857\ (3)$, $c = 9{\cdot}163\ (3)$ Å, $\beta = 107{\cdot}05\ (4)°$, $V = 688{\cdot}2\ (7)$ Å^3, $Z = 2$, $D_x = 1{\cdot}569$ Mg m^{-3}, $\lambda(\text{Mo } K\alpha) = 0{\cdot}71073$ Å, $\mu = 1{\cdot}69$ mm^{-1}, $F(000) = 340$, $T = 300$ K. Final $R = 0{\cdot}026$ for 1474 observed reflections. The complex is centrosymmetric with Ni^{II} octahedrally coordinated by N of the NCS^- ions, and by O and N of the 3-aminopropanol bidentate ligands. The moiety Ni—N(1)—C(3)—C(2)—C(1)—O forms a chair conformation flattened at the Ni side. There is an OH···S internal hydrogen bond of 3·25 Å.

Introduction. In pursuance of our studies of metal complexes of aminoalcohols, we have prepared a series of novel nickel complexes using various such ligands. The title compound was prepared using a procedure in which nickel(II) acetate was treated with hot 3-aminopropanol and sodium thiocyanate (dissolved in methanol) was added to the resultant bluish-green viscous liquid. Blue crystals of the title compound separated out after a few days. Crystallization was from hot methanol. Elemental analysis confirmed the identity of the complex (found: Ni 18·09, S 19·85, H 5·55, N 17·30, C 29·44; calculated for $C_8H_{18}N_4NiO_2S_2$: Ni 18·06, S 19·73, H 5·58, N 17·23, C 29·56%).

We undertook the X-ray analysis in order to confirm low-temperature magnetic-measurement results which revealed the presence of a six-membered ring (in the nickel coordination) in the chair form and to establish the possibility of internal hydrogen bonding.

Experimental. Crystal 0·2 × 0·1 × 0·15 mm. Cell dimensions determined from setting angles of 25 high-order reflections. Enraf–Nonius CAD-4 diffractometer, graphite-monochromatized Mo radiation, ω/θ scan, range $0 < \theta \le 27°$, $0 \le h \le 11$, $0 \le k \le 11$, $-11 \le l \le 11$. Three intensity- and orientation-control reflections measured every 120 min and 200 reflections respectively, no significant drift noted. Intensities not corrected for absorption because of small crystal size and low linear absorption coefficient. 1735 independent

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reflections measured, 1474 observed with $I \geq 2\sigma(I)$. Structure solved by Patterson and heavy-atom Fourier analysis, H positions found from a difference Fourier map. Least-squares refinement on F's in which each individual reflection was given a weight based on counting statistics. All atom positions including those of H refined, isotropic temperature factors of H atoms fixed to 4 Å^2; $R = 0{\cdot}026$, $R_w = 0{\cdot}031$, $S = 5{\cdot}6$. Extinction parameter refined as $0{\cdot}58\ (4) \times 10^{-6}$ mm. Max. peak in final difference Fourier map 0·3 e Å^{-3}. Max. least-squares parameter shift $0{\cdot}02\sigma$. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974). Computer calculations all with Enraf–Nonius CAD-4 *SDP* (Frenz, 1978).

Discussion. Relevant atomic parameters* are listed in Table 1; the numbering of the atoms is given in Fig. 1.

Table 1. *Positional parameters in fractions of the cell edges and isotropic temperature factors* (Å^2)

The e.s.d.'s given in parentheses refer to the last significant digit. Isotropic temperature factors are calculated from anisotropic temperature parameters assuming equal volume of the 50% probability region according to Lipson & Cochran (1968): $B_{\rm iso} = 8\pi^2(U^{\circ}_{11}U^{\circ}_{22}U^{\circ}_{33})^{1/3}$. All anisotropic thermal parameters were physically acceptable.

	x	y	z	$B_{\rm iso}$
Ni	0·0000	0·0000	0·0000	2·19
S	−0·3076 (1)	0·4251 (1)	0·0674 (1)	3·42
O	−0·1624 (2)	0·0165 (2)	−0·2178 (2)	3·01
N(1)	0·1571 (2)	0·1420 (3)	−0·0641 (2)	2·94
N(2)	−0·1046 (2)	0·1856 (3)	0·0655 (2)	3·05
C(1)	0·3304 (3)	−0·0199 (3)	0·2407 (3)	3·14
C(2)	0·3860 (3)	0·1107 (3)	0·1671 (3)	3·18
C(3)	0·3295 (3)	0·1131 (3)	−0·0051 (3)	3·26
C(4)	−0·1870 (3)	0·2845 (3)	0·0679 (2)	2·50

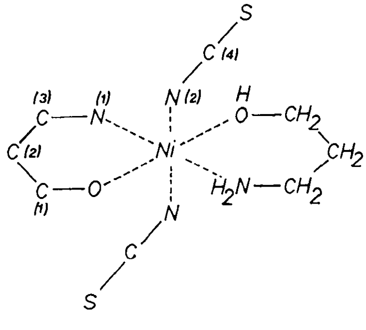

Fig. 1. Structural formula and numbering of the atoms.

Table 2. *Bonded distances* (Å) *and angles* (°) *in the title compound with e.s.d.'s in parentheses*

Ni coordination			
Ni–O	2·096 (1)	O–Ni–N(1)	91·09 (4)
Ni–N(1)	2·085 (1)	O–Ni–N(2)	91·20 (3)
Ni–N(2)	2·062 (1)	N(1)–Ni–N(2)	90·26 (4)
Isothiocyanato anion			
C(4)–S	1·640 (1)	S–C(4)–N(2)	178·7 (1)
C(4)–N(2)	1·145 (1)		
3-Aminopropanol			
O–C(1)	1·443 (1)	O–C(1)–C(2)	112·5 (1)
C(1)–C(2)	1·494 (2)	C(1)–C(2)–C(3)	115·6 (1)
C(2)–C(3)	1·509 (2)	N(1)–C(3)–C(2)	111·9 (1)
C(3)–N(1)	1·488 (1)	Ni–O–C(1)	122·1 (1)
		Ni–N(1)–C(3)	119·6 (1)
Torsion angles (°) in the Ni–N(1)–C(3)–C(2)–C(1)–O ring			
C(3)–N(1)–Ni–O	36·9 (5)	O–C(1)–C(2)–C(3)	−66·8 (5)
N(1)–Ni–O–C(1)	−36·3 (5)	C(1)–C(2)–C(3)–N(1)	68·7 (5)
Ni–O–C(1)–C(2)	53·8 (5)	C(2)–C(3)–N(1)–Ni	−56·7 (5)

The complex is centrosymmetric with the Ni^{2+} ion at a crystallographic inversion center. Ni is octahedrally coordinated by the N atoms of two NCS$^-$ ions and the N and O atoms of two 3-aminopropanol molecules acting as bidentate ligands. The Ni coordination as well as bond lengths and valence angles in the isothiocyanato and 3-aminopropanol ligands are presented in Table 2. The OH distance is 0·80 (1) Å, NH bond lengths are 0·80 (1) and 0·87 (1) Å, and CH distances range from 0·92 (1) to 1·12 (1) Å. All bonded distances are near the expected values.

The six-membered ring in the equatorial plane formed by Ni—N(1)—C(3)—C(2)—C(1)—O has a chair conformation. Cremer & Pople (1975) ring-puckering parameters, with e.s.d.'s according to Norrestam (1981), are: $q_1 = -0{\cdot}540\ (4)$, $q_2 = 0{\cdot}114\ (4)$, $Q = 0{\cdot}552\ (4)$ Å, $\varphi_2 = 346\ (2)$ and $\theta_2 = 168\ (1)$°. Individual ring torsion angles are given in Table 2, showing a flattening at the Ni side and a puckering at the C side of the ring. The NCS$^-$ ions are almost linearly coordinated to the Ni.

The structure contains an internal hydrogen bond OH···S with an O···S distance of 3·25 Å, an H···S distance of 2·50 Å, and an O—H···S angle of 153°.

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* Lists of structure factors, H-atom positions and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 39828 (8 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.