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Studies in Werner Clathrates.
Part 6. Structures of Two Novel Polymeric Inclusion Compounds: Poly(bis(isothiocyanato)di(2-aminopyridine)nickel(II))·diethylether and Di(aqua bis(isothiocyanato)3-aminopyridine μ-3-aminopyridine nickel(II))·water

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Abstract

The X-ray crystal structures of $Ni(NCS)_2(2\text{-}NH_2\text{-}C_5H_4N)_2 \cdot C_4H_{10}O$ (I) and $(Ni(NCS)_2(3\text{-}NH_2C_5H_4\text{-}N)_2(H_2O))_2 \cdot 2H_2O$ (II) have been elucidated. In both compounds the Ni(II) atom is pseudooctahedrally coordinated.

Compound I crystallizes in polymer chains in which the thiocyanate groups are bridging. The diethylether guest molecules are located in channels running parallel to the [101] direction.

Compound II is a dimeric structure in which an amino group on one of the 3-substituted pyridine ligands is a bidentate ligand by its co-coordination to the neighbouring nickel atom. The dimers are linked into chains by hydrogen bonding with the 'guest' water molecule which is situated between them.

Introduction

There is a wide variety of inclusion compounds called Werner Clathrates which have an enclosing framework that is made up of coordination compounds represented by the general formula MX_2B_n where n is 4 (most common) or 2, M stands for a divalent cation (Fe, Co, Ni, Cu, Zn, Cd, Mn, Mg, Cr), X denotes anionic ligands (NCS^-, NCO^-, CN^-, NO_3^-, NO_2^-, Cl^-, Br^-, I^-) and B represents electrically neutral ligand-substituted pyridines, α-arylalkylamine, isoquinoline or piperidines.

The majority of these complexes that are reported in the literature are derived from extensive experimental studies performed by Schaeffer and co-workers [2] (who first applied their clathrating ability to the separation of various aromatic compounds from petroleum fractions) and from the investigations by de Radzitzky and co-workers [3].

The most versatile complex thus far investigated has been $Ni(NCS)_2(4\text{-Mepy})_4$ which can entrap a wide variety of 'guest' molecules in cavities of the channel, layer or cage type. Its crystal structures with a range of 'guest' molecules, their physicochemical behaviour and intermolecular 'host–guest' interactions have been reviewed by Lipkowski [4].

We have extended the study of Werner clathrates by studying 'host' complexes of the type: (a) $Ni(NCS)_2(4\text{-Rpy})_4$ where R = ethyl, vinyl or phenyl [1, 5, 6]; (b) $Ni(NCS)_2(4\text{-Mepy})_4(4\text{-Phpy})_2$ [7]; (c) $Ni(NCS)_2(3,5\text{-diMepy})_4$ and $Ni(NCS)_2(3\text{-Mepy})_4$ [6]. In all cases variation of the type of substituent and its position on the pyridine ring results in large differences in the clathrating ability of these complexes.

The $Ni(NCS)_2B_4$ molecules ususally have irregular octahedral coordination and are 'overcrowded' in their central region, *i.e.* there are non-bonded repulsive interactions between the ligands which lead to a limited number of stable conformations. The most common one is a 'propeller' conformation (e.g. in the $Ni(NCS)_2(4\text{-Mepy})_4$ molecule the 4 pyridine rings are twisted by 43°–55° from the coplanar arrangement) [4].

Inclusion compound formation from two-base complexes of the type $Ni(NCS)_2B_2$ has not been as extensively studied. This work was stimulated by a previous study [8] on the dependence of the stereochemistry of $Ni(NCS)_2(q\text{-Rpy})_2$ complexes on various positions of a substituent (q = 2, 3 or 4) on the pyridine ligand (py), as well as on the nature of the substituent (R = Me, Et, NH_2, Cl, Br and CN). The authors reported association of solvent molecules when $2\text{-}NH_2py$ was used as the neutral ligand.

Molecular structures have largely been determined by magnetic and spectral measurements [8, 9]; however X-ray structure analyses have been carried out on $Ni(NCS)_2(2,5\text{-diMepy})_2$ [10], $Ni(NCS)_2(piperidine)_2 \cdot C_6H_6$ [11] and $Ni(NCS)_2$(3-methyl isoquinoline)$_2$ [12].

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In mononuclear $Ni(NCS)_2(2\text{-}Rpy)_2$ structures the substituent in the 2-position shows steric interaction with the thiocyanate groups resulting in destabilization of the structure. However stabilization can be achieved by the lengthening of the Ni–N(py) bond and by the twisting of the pyridine ring from the equatorial plane, as supported by X-ray structure analysis results for the $Ni(NCS)_2(2,5\text{-}diMepy)_2$ complex [10]. The 2-substituent of the twisted pyridine ring in this case makes the interaction in axial positions more difficult, contrary to substituents in positions 3 or 4.

Experimental Procedure and Structure Solutions

The 'host' complexes $Ni(NCS)_2(2\text{-}NH_2py)_2$ and $Ni(NCS)_2(3\text{-}NH_2py)H_2O$ were prepared in solution by reacting an aqueous nickel isothiocyanate solution with a stoichiometric quantity of the respective substituted amino pyridine. The resulting green solutions were filtered and left standing to evaporate.

After approximately 4 months dark green composite aggregates of tiny crystals grew in the aqueous solution of the 'host' of **I**. Some of these aggregates were removed from the mother liquor and dried with tissue paper then washed several times with diethylether before being left standing in this solvent (no evaporation was allowed to take place). The light green needle shaped single crystals which grew on the surface of the aggregates analysed for **I**.

Small turquoise single crystals grew in the aqueous 'host' solution of **II**, these crystals analysed for compound **II**.

The densities of single crystals of both compounds were obtained using a linear density column containing water and KI solution in the range 1.00 to 1.63 g cm^{-3}. The column was calibrated with oil droplets of predetermined densities.

Microanalysis was then carried out on the crystals in order to ascertain % C, % H and % N. The results are reported in Table I.

Crystal data and the experimental details of the intensity data collection are also listed in Table I. For both structural determinations a single crystal of suitable size (see Table I for crystal dimensions of both compounds) was mounted on a glass fibre. The crystal was then covered by a thin layer of transparent glue to prevent any deterioration in the atmosphere. Initial cell parameters for both compounds were established photographically using standard single crystal techniques. These were then refined by least-squares analysis of 24 reflections measured in the range $16° < \theta < 17°$ on a Nonius CAD4 diffractometer with graphite monochromated Mo Kα radiation ($\lambda = 0.7107$ Å). The intensity data collections were carried out at 294 K, scans were in the $\omega - 2\theta$ mode with a final acceptance limit of 20σ at 20° min^{-1} in ω and a maximum recording time of 40 s. In each case the intensity variation of three standard reflections was monitored every hour to check crystal stability and recentering was carried out every 100 measured reflections. All intensities were corrected both for Lorentz and polarization factors as well as for absorption [13].

Both structures were solved in $P2_1/n$ unique axis *b* (No. 14 cell choice 2 with the origin at $\bar{1}$) by the heavy atom method and subsequent difference Fourier syntheses, with least-squares refinement of *F* magnitudes, using the SHELX 76 [16] program system.

In both cases the final model employed anisotropic parameters for Ni and S only and isotropic ones for all other atom types. Aromatic hydrogen atoms in both structures were subjected to constrained refinement, riding at 1.08 Å from their parent carbon atoms and in **II** they had a common temperature factor. In **I** each aromatic hydrogen has a temperature factor that is fixed at 1.3 times that of its parent carbon; 'guest' and amino hydrogens were omitted.

In **II**, 'guest' and amino hydrogens were placed in calculated positions (1.08 Å from the parent O or N atom), each type with a common temperature factor.

The best model for refinement of the diethylether 'guest' in **I** was obtained by allowing a rigid molecular model (whose parameters had been obtained by a previous X-ray study [17]) in which all atoms had *SOF* values of 1.0 to match the difference electron density map. The common isotropic temperature factor for the 'guest' atoms was as expected higher than those obtained for non-hydrogen 'host' atoms. A final difference electron density map, calculated after the last full-matrix least-squares refinement, yielded 2 peaks, with one electron per cubic angstrom in the vicinity of the 'guest' molecule, which can be accounted for as imperfect modelling of the 'guest' molecule.

The maximum peak height in the final difference Fourier synthesis of **II** corresponds to less than 1 electron $Å^{-3}$.

Final refinement parameters are listed in Table I, and fractional atomic coordinates are given in Tables II and III. All complex neutral atom scattering factors for hydrogen were taken from Stewart *et al.* [18], and for all other atoms from Cromer and Mann [15], with dispersion corrections from Cromer and Liberman [14].

Molecular parameters (tables of bond lengths, angles and torsion angles) were obtained from the program PARST [19] and drawings from PLUTO [20]. See 'Supplementary Material'. All computations were carried out on a Sperry 1100 computer.

TABLE I. Crystal Data and Experimental and Refinement Parameters for Compounds I and II

	Compound I	Compound II
Crystal data		
Microanalysis % C, % H, % N		
determined	42.1, 4.7, 18.6	36.3, 4.0, 21.2
calculated	42.4, 4.9, 18.5	36.1, 4.0, 21.1
Molecular formula	$C_{12}H_{12}N_6NiS_2 \cdot C_4H_{10}O$	$[C_{12}H_{14}N_6NiOS_2 \cdot H_2O]_2$
Molecular weight (g mol^{-1})	421.23	798.28
Space group	$P2_1/n$	$P2_1/n$
a (Å)	9.582(1)	12.041(6)
b (Å)	16.838(2)	9.765(1)
c (Å)	13.275(2)	14.535(5)
β (°)	105.76(1)	94.81(4)
V ($Å^3$)	2061.20	1702.91
Z	4	4 (for 1/2 the dimer)
Host:guest ratio	1:1	1:1
D_m (g cm^3)	1.48	1.60
D_c (g cm^{-3})	1.46	1.56
μ (Mo Kα) (cm^{-1})	10.99	13.25
$F(000)$	944	824
Data collection		
Crystal dimensions (mm)	0.13 × 0.16 × 0.32	0.22 × 0.22 × 0.25
Scan width (ω) (°)	$(0.44 + 0.35 \tan \theta)$	$(0.93 + 0.35 \tan \theta)$
Aperture width (mm)	$(1.12 + 1.05 \tan \theta)$	$(1.10 + 1.05 \tan \theta)$
Vertical aperture length (mm)	4	4
Range scanned (°)	$1 < \theta < 25$	$1 < \theta < 25$
Stability standard reflections (%)	1.50	1.29
Number reflections collected	3928	3332
Number of 'observed' reflections with $I_{rel} > 2\sigma I_{rel}$	2258	2173
Average transmission (%)	96.15	96.52
Final refinement		
Number of variables	132	134
$R = \Sigma \lVert F_o \rvert - \lvert F_c \rVert / \Sigma \lvert F_o \rvert$	0.0989	0.0463
$R_w = \Sigma w^{1/2} \lVert F_o \rvert - \lvert F_c \rVert / \Sigma w^{1/2} \lvert F_o \rvert$	0.0989	0.0461
Weighting scheme	unity	$(\sigma^2 F)^{-1}$

Discussion

In both compounds I and II each nickel atom occupies a general position and is coordinated to six donor ligands in such a way that they form an irregular octahedron. Figure 1 illustrates the host portion of the asymmetric unit, with atomic nomenclature, of I. A perspective view of the asymmetric unit of the dimer of II, with atomic nomenclature is shown in Fig. 2. The environment of the nickel atom, in each compound including the isothiocyanate ligands, is illustrated in Fig. 3.

Compound I is a polymeric structure with the thiocyanate groups forming bridges between the nickel atoms. In an asymmetric unit the nickel atom is coordinated to two isothiocyanate sulfur atoms, which are *cis* to each other, and to two pyridine nitrogen atoms of the 2-aminopyridine base ligands as illustrated in Fig. 1. Each nickel atom is also coordinated to two isothiocyanate nitrogen atoms of the neighbouring asymmetric unit. The Ni–N bond distances for the isothiocyanate ligands are approximately 0.09 Å shorter than for the pyridine ligands (see Fig. 3 for exact lengths) as found in the traditional $Ni(NCS)_2B_4$ Werner complexes [4–8]. The Ni–S bond distances are in the expected range, which is shorter than those found in a similar type of complex [21]. The angles subtended at the nickel atom vary from 82.0° to 94.6° (Fig. 3), giving it a distorted octahedral configuration. This irregular octahedral arrangement (obtained in spite of the presence of 2-substituted pyridine ligands) forms infinite chains, as shown in Fig. 4, running parallel to the crystallographic a axis of the monoclinic unit cell which is different from the structure suggested previously by Jamnicky and

TABLE II. Fractional Atomic Coordinates ($\times 10^4$) and Thermal Parameters ($\times 10^3$) of Compound **I** (e.s.d.s in parentheses)

	x	y	z	U_{iso}
Ni(1)	2062(2)	184(1)	−1150(1)	a
S(1)	2082(4)	−1024(2)	89(3)	a
C(1)	725(13)	−785(7)	565(10)	41(3)
S(2)	3284(4)	1001(2)	551(3)	a
C(2)	4870(13)	581(7)	929(9)	41(2)
N(1)	−227(11)	−613(6)	870(8)	44(2)
N(2)	5988(11)	277(7)	1188(8)	47(2)
N(11)	2133(12)	1180(7)	−2120(9)	51(2)
C(12)	3202(20)	1651(11)	−2158(14)	83(5)
C(13)	3011(21)	2364(11)	−2874(14)	82(5)
C(14)	1739(23)	2506(13)	−3387(16)	97(6)
C(15)	747(24)	2042(10)	−3322(14)	106(1)
C(16)	920(20)	1447(10)	−2771(11)	101(1)
N(12)	4495(19)	1492(11)	−1617(13)	107(5)
N(21)	845(12)	−503(7)	−2428(8)	50(3)
C(22)	1165(19)	−606(10)	−3364(13)	65(2)
C(23)	211(22)	−1039(14)	−4169(17)	85(6)
C(24)	−1047(24)	−1349(14)	−4009(17)	103(1)
C(25)	−1320(20)	−1284(11)	−3038(14)	80(5)
C(26)	−367(15)	−841(8)	−2293(12)	57(3)
N(22)	2293(14)	−296(8)	−3500(10)	68(3)
CG(11)	670(23)	2984(12)	−845(14)	108(1)
CG(12)	861(23)	3074(10)	240(14)	108(1)
OG(11)	1600(14)	3830(8)	301(10)	108(1)
CG(13)	1269(23)	3765(10)	1309(13)	108(1)
CG(14)	1354(23)	4605(10)	1466(15)	108(1)

[a] Anisotropic atoms have thermal parameters of the form: $T = \exp\{-2\pi^2(U_{11}h^2a^{*2} + U_{22}k^2b^{*2} + U_{33}l^2c^{*2} + 2U_{23}klb^*c^* + 2U_{13}hla^*c^* + 2U_{12}hka^*b^*) \times 10^3\}$, with the following parameters. Ni(1): U_{11}, 38(1); U_{22}, 48(1); U_{33}, 44(1); U_{23}, 4(1); U_{13}, 16(1); U_{12}, 8(1). S(1): U_{11}, 52(2); U_{22}, 61(2); U_{33}, 61(2); U_{23}, 5(2); U_{13}, 28(1); U_{12}, 18(1). S(2): U_{11}, 49(2); U_{22}, 63(2); U_{33}, 64(2); U_{23}, −9(2); U_{13}, 11(1); U_{12}, 18(1).

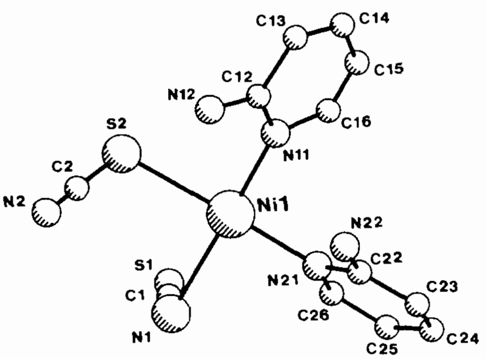

Fig. 1. Perspective view of the portion of a 'host' molecule that is in an asymmetric unit of **I** with atomic nomenclature.

TABLE III. Fractional Atomic Coordinates ($\times 10^4$) and Thermal Parameters ($\times 10^3$) of Compound **II** (e.s.d.s in parentheses)

	x	y	z	U_{iso}
Ni(1)	897(0)	6159(1)	1724(1)	a
N(1)	891(3)	4769(4)	2762(3)	35(0)
C(1)	932(4)	4255(5)	3478(3)	26(0)
S(1)	1025(1)	3521(1)	4483(1)	a
N(2)	1003(3)	7404(4)	613(3)	35(0)
C(2)	972(4)	7995(5)	−71(3)	33(2)
S(2)	908(1)	8820(2)	−1052(1)	a
N(11)	−850(3)	6168(4)	1571(3)	32(9)
C(12)	−1389(4)	7330(5)	1301(3)	32(1)
C(13)	−2538(4)	7444(5)	1199(4)	40(3)
N(131)	−3015(4)	8629(6)	802(4)	60(4)
C(14)	−3150(5)	6296(6)	1419(4)	51(5)
C(15)	−2613(5)	5123(6)	1684(4)	56(6)
C(16)	−1460(5)	5060(6)	1749(4)	46(4)
N(231)	2718(3)	6183(4)	1992(3)	28(9)
0(1)	961(3)	4518(4)	760(3)	45(9)
N(21)	902(3)	7833(4)	2649(2)	26(9)
C(22)	1472(3)	8992(4)	2510(3)	25(0)
C(23)	1649(4)	9992(5)	3175(3)	27(0)
C(24)	1227(4)	9794(5)	4027(3)	35(2)
C(25)	606(4)	8639(5)	4153(3)	37(2)
C(26)	453(4)	7688(5)	3464(3)	33(1)
OG(1)	3218(3)	6796(4)	4009(3)	50(0)

[a] Anisotropic atoms have thermal parameters of the form: $T = \exp\{-2\pi^2(U_{11}h^2a^{*2} + U_{22}k^2b^{*2} + U_{33}l^2c^{*2} + 2U_{23}klb^*c^* + 2U_{13}hla^*c^* + 2U_{12}hka^*b^*) \times 10^3\}$, with the following parameters. Ni(1): U_{11}, 28(1); U_{22}, 22(1); U_{33}, 27(1); U_{23}, 2(1); U_{13}, −1(1); U_{12}, −1(1). S(1): U_{11}, 51(1); U_{22}, 42(1); U_{33}, 35(1); U_{23}, 8(1); U_{13}, 9(1); U_{12}, 2(1). S(2): U_{11}, 77(1); U_{22}, 72(1); U_{33}, 48(1); U_{23}, 31(1); U_{13}, −9(1); U_{12}, −24(1).

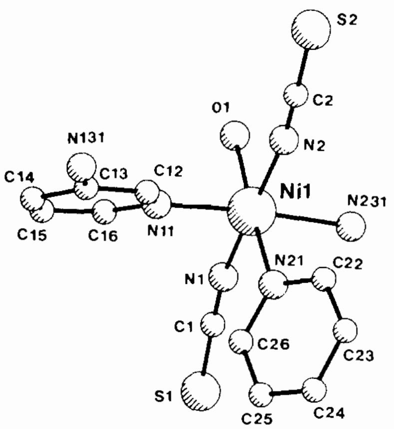

Fig. 2. Perspective view of the portion of a 'host' molecule that is in an asymmetric unit of **II** with atomic nomenclature.

Jona [8]. This is illustrated by the packing diagram in Fig. 5.

The non-bonded N(12)....S(2) distance of 3.48 Å suggests the possibility of a hydrogen bond, however no hydrogen atoms could be located in the final difference electron density map.

The diethylether guest molecules are located in channels running parallel to the [101] direction as

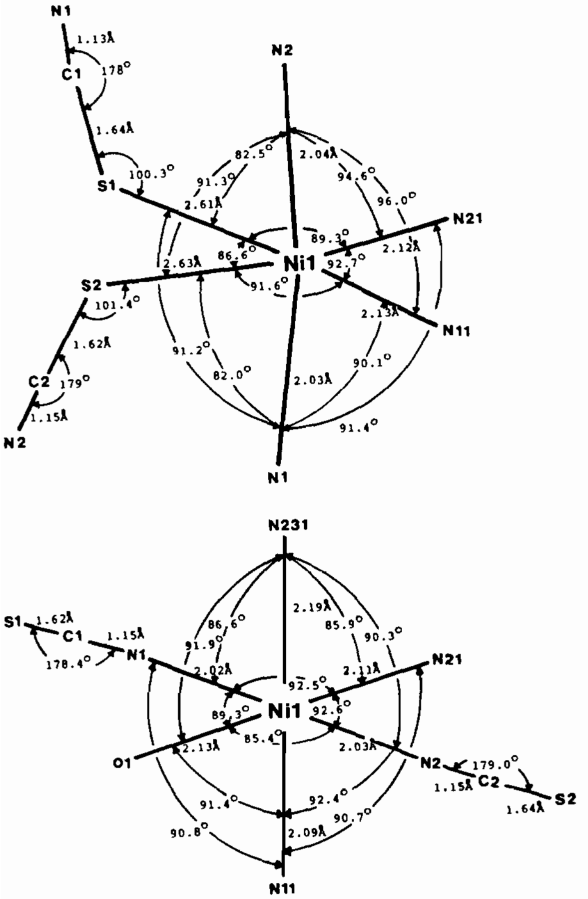

Fig. 3. The environments of the nickel atoms, including the isothiocyanate ligands, in I and II.

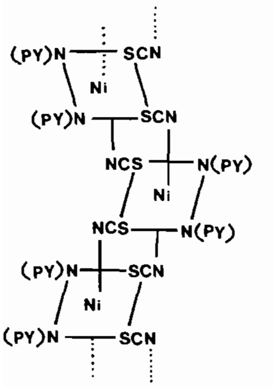

Fig. 4. A schematic diagram of the 'host' molecule chains in I.

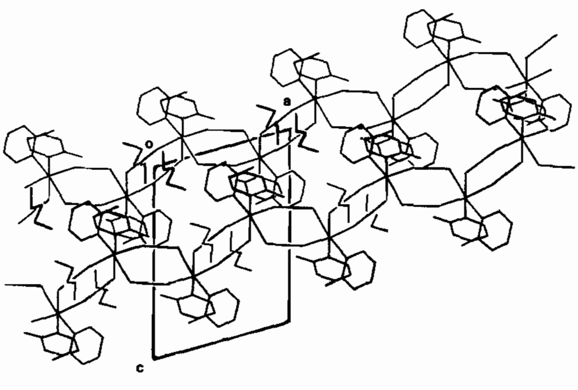

Fig. 5. An illustration of the packing of the diethylether molecules in the polymeric 'host' framework of I.

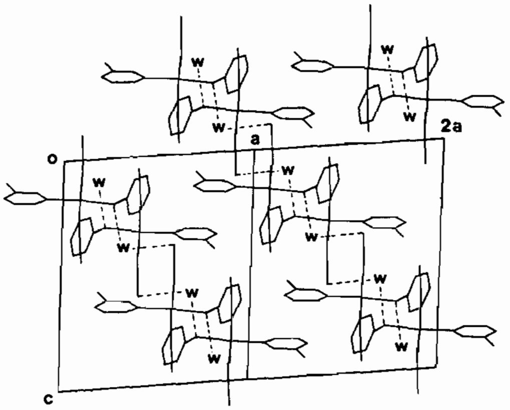

Fig. 6. A packing diagram of II with 'guest' water molecules represented as W and hydrogen bonds as dashed lines.

illustrated in Fig. 5. In each channel the guest molecules are related by two fold screw axes which run parallel to the crystallographic *b* axis.

Compound **II** is a dimeric structure in which the amino group behaves as a bidentate ligand linking the metal complexes by coordination directly to each nickel atom forming the pair. In each asymmetric unit (illustrated in Figs. 2 and 3) the irregular octahedral configuration of the nickel atom is established by its coordination to:

(a) Two isothiocyanate moieties, which are bonded *trans* to each other through their nitrogen atoms with typical Ni–N distances of 2.0 Å.

(b) Two 3-NH_2py ligands bonded *cis* to each other and through the nitrogen atom in the pyridine ring; Ni–N distances are 2.1 Å as expected.

(c) A water molecule.

(d) An amine nitrogen atom of one 3-NH_2py ligand belonging to the neighbouring asymmetric unit which forms the other half of the dimer. This Ni–N bond length is longer (see Fig. 3) than those for all the other five ligands bonded to the nickel.

A packing diagram viewed down the crystallographic *b* axis (Fig. 6) illustrates firstly how the dimers run parallel to the [101] direction and secondly how they are linked into chains by hydrogen bonding with the 'guest' water molecule (labelled W) which is situated between the dimers.

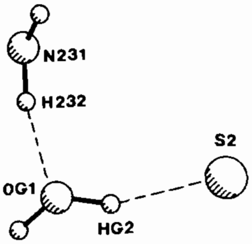

Fig. 7. An illustration of the two types of hydrogen bonds with distances and angles.

N(231)....OG(1)	3.003 Å
H(232)....OG(1)	1.952 Å
N(231)–H(232)....OG(1)	165.05°
OG(1)....S(2)	3.303 Å
HG(2)....S(2)	2.283 Å
OG(1)–HG(2)....S(2)	158.66°

There are two types of hydrogen bonds, detailed in Fig. 7, in one the 'guest' water molecule behaves as a hydrogen bond acceptor (*i.e.* N(231)–H(232).... OG(1)) and in the other as a hydrogen bond donor (*i.e.* OG(1)–HG(2)....S(2)). There is no evidence of bifurcated hydrogen bonds. The non-bonded distances of N(231)....OG(1) and OG(1)....S(2) are 3.003 and 3.303 Å respectively and are of the order of magnitude previously observed [22] for hydrogen bonds of these types. In both cases the deviation from linearity, as illustrated by the angles of 165.0° and 158.7° (see Fig. 7), is common for hydrogen bonds of this type [22].

A comparison of packing modes in the two compounds was made by calculating the packing density, expressed as the volume per non-hydrogen atom, which is 19.1 $Å^3$ for **I** and 18.5 $Å^3$ for **II**. This illustrates the greater packing efficiency of the water clathrate, **II**, as opposed to the diethylether clathrate, **I**, which is supported by the fact that the diethylether molecule in **I** is somewhat disordered and the standard deviations in positional parameters (Table II), bond lengths and angles (Supplementary Material) for the 'host' molecule are greater than those obtained for the 'host' molecule of **II** (Table III).

Two base complexes of the type $Ni(NCS)_2B_2$ have thus far not proved to be nearly as versatile in clathrate formation as the traditional $Ni(NCS)_2B_4$ Werner complexes.

Supplementary Material

Tables of bond lengths, angles and torsion angles are available from the Editor-in-Chief.

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