

Crystal Structure of Nickel- and Cobalt-Diazidotetrapyridinemetall(II)-2(Pyridine) Clathrates

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Abstract. $[\text{MPy}_4(\text{N}_3)_2] \cdot 2\text{Py}$ ($\text{M} = \text{Ni}, \text{Co}$) are clathrates where two of six pyridine species are included in the crystal structure of the host complex without chemical bonding. The clathrates are isostructural, orthorhombic, space group $Pnna$ (52), $Z = 4$. For the Ni compound $a = 14.958(5)$, $b = 11.909(5)$, $c = 17.517(7)$ Å, $V = 3120(2)$ Å³, $\mu = 12.25$ cm⁻¹, final $R = 0.057$. The Cocompound is of very similar structure ($a = 14.980(3)$, $b = 11.980(2)$, $c = 17.350(5)$ Å, $V = 3114(1)$ Å³, $\mu = 46.50$ cm⁻¹). The metal cation in the host molecule is surrounded by six donor nitrogens of two terminal azido-groups in *trans*-positions and four pyridine ligands. The complex molecule lies on a twofold axis parallel to a .

Key words: Crystal structure, Werner clathrate, azide, pyridine, Ni(II), Co(II).

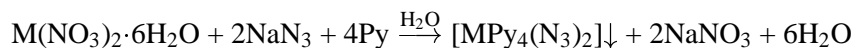
1. Introduction

The clathrate nature of compounds of the type $[\text{MPy}_4\text{X}_2] \cdot 2\text{Py}$ ($\text{M} = \text{metal(II)}$, $\text{Py} = \text{Pyridine}$, $\text{X} = \text{monovalent anionic ligand}$) was recently suggested [1, 2] and proved chemically by replacing the guest pyridine with other organic components [3]. Structurally, these compounds can be described as a new type of Werner clathrates, the clathrates first developed by Schaeffer *et al.* [4] and studied extensively in the 1960–1970s [5]. Recently, a number of structures of compounds of this new type have been studied, with variation of both the metal cation and the anionic ligand: $[\text{MPy}_4\text{Br}_2] \cdot 2\text{Py}$ ($\text{M} = \text{Mg}$ [6], Co , Ni [2], Cu [2, 7, 8]), $[\text{MPy}_4\text{I}_2] \cdot 2\text{Py}$ ($\text{M} = \text{Co}$ [2], Cu [9]), $[\text{NiPy}_4(\text{NCS})_2] \cdot 2\text{Py}$ [10], $[\text{NiPy}_4(\text{HCOO})_2] \cdot 2\text{Py}$ [2], $[\text{MPy}_4(\text{NO}_3)_2] \cdot 2\text{Py}$ ($\text{M} = \text{Co}$ [2], Ni [2, 11], Cu [2, 12, 13], Zn , Cd [2]). The present communication extends this series to novel clathrates with $\text{X} = \text{azide}$. This allows consideration of the peculiarities of the host framework and the molecular packing resulting from the specific geometry and coordination mode of the azido ligand.

2. Experimental

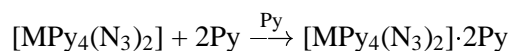
2.1. PREPARATIONS

All reagents used were of reagent-grade quality or better. The $[\text{MPy}_4(\text{N}_3)_2]$ host complexes were obtained according to a slight modification of Schaeffer's method [4]:



The respective metal nitrate (0.01 mole) and 0.02 mole of sodium azide were dissolved in a minimal quantity of water. A slight excess of pyridine (ca. 0.043 mole) was gradually poured into the resultant solution. After the reaction was complete the malachite-coloured (Ni) or lilac (Co) powder precipitate was separated by filtration, washed twice with a little water and air-dried.

Crystalline clathrates were obtained by direct crystallization of the respective host complexes from pure pyridine:



The large emerald (Ni) or red (Co) clathrate crystals resembled truncated octahedra or prisms; they were unstable in air.

2.2. CRYSTALLOGRAPHY

For an X-ray diffraction analysis, a crystal fragment of conventional dimensions (Table I) was chipped off under the mother liquor layer and fixed inside a sealed Lindemann glass capillary tube in a droplet of pyridine. Twenty-five reflections chosen randomly in the $10\text{--}20^\circ$ θ -range were used for determination, and 25 more in the $20\text{--}30^\circ$ θ -range for verification and refinement of the unit cell constants. These values, along with salient experimental details, data collection and structure refinement parameters are given in Table I.

During data collection, three reference reflections were periodically monitored to control changes in crystal orientation and to allow correction of data for crystal decay; their intensity decrease did not exceed 10%. Lorentz-polarization corrections were also applied. For the Ni-compound no correction was made for absorption, while for the Co-compound spherical correction was applied (the crystal used was of rather regular shape, thus allowing for such an approximation).

The structure of $[\text{NiPy}_4(\text{N}_3)_2] \cdot 2\text{Py}$ was solved by the heavy-atom method using the SHELXS-86 program system [14]. The original *Pbnn* space group was reduced to the standard *Pnna*, with the data transformed using the matrix $(0\ 1\ 0\ 1\ 0\ 0\ 0\ 0\ -1)$.

The refinement was performed using SHELXL-93 [15] and anisotropic thermal parameters for non-hydrogen atoms. All data with positive intensities were

Table I. Data collection and structure analysis parameters.

Clathrate	[NiPy ₄ (N ₃) ₂]-2Py	[CoPy ₄ (N ₃) ₂]-2Py
Empirical formula	C ₃₀ H ₃₀ N ₁₂ Ni	C ₃₀ H ₃₀ N ₁₂ Co
Formula weight	617.4	617.6
Crystal system	Orthorhombic	Orthorhombic
Space group	<i>Pnna</i> (52)	<i>Pnna</i> (52)
Unit cell parameters:		
<i>a</i> , Å	14.958(5)	14.980(3)
<i>b</i> , Å	11.909(5)	11.980(2)
<i>c</i> , Å	17.517(7)	17.350(5)
<i>V</i> , Å ³	3120(2)	3114(1)
<i>Z</i>	4	4
<i>F</i> (000)	1288	1284
Calculated density, g cm ⁻³	1.314	1.317
Crystal size, mm	0.4 × 0.5 × 0.5	0.3 × 0.3 × 0.35
Diffractometer	CAD4	KM4
Radiation (monochromator)	CuK _α (graphite)	CuK _α (graphite)
Absorption coefficient, cm ⁻¹	12.25	46.50
Scan parameters:		
mode	$\omega/2\theta$	$\omega/2\theta$
θ -range, deg	2–78	2–78
width ($\Delta\omega$), deg	2.0 + 0.14tan θ	1.6 + 0.2tan θ
speed (ω), deg/min.	2.0(min) – 5.5(max)	2.4(min) – 9.0(max)
background time	1/3 of measuring time	1/4 of measuring time
Orientation control reflections	3 after every 100 refl.	3 after every 100 refl.
Intensity control reflections	3 after each 1 hour	3 after every 100 refl.
<i>h</i> , <i>k</i> , <i>l</i> -range	0/18, 0/14, 0/22	0/18, 0/15, –22/0
Number of reflections:		
unique-measured	3166	3298
unique-observed (<i>l</i> > 2 σ _{<i>l</i>})	1670	728
Number of refined parameters	207	67
Final <i>R</i> -value*	0.057	0.105
(observed data)		
<i>R</i> _w **	0.138	0.240
(observed data)	(<i>k</i> = 0.068)	(<i>k</i> = 0.164)
Residual extrema in final difference		
map, e Å ⁻³	–0.24/+0.29	–0.49/+0.34

$$* R = \sum ||F_o| - |F_c|| / \sum |F_o|.$$

$$** R_w^2 = \sum [w(F_o^2 - F_c^2)^2] / \sqrt{\sum [w(F_o^2)^2]}; w = 1/[\sigma^2(F_o^2) + (k \cdot P)^2], \text{ where } P = (\max(F_o^2) + 2 \cdot F_c^2)/3.$$

involved. Hydrogen atoms were included at calculated positions ($d_{C-H} = 0.93$ Å) and ‘riding’ on corresponding carbon atoms. They were refined isotropically with thermal factors 1.2 times greater than those for adjacent carbon atoms. The azide

ligand was found to be disordered over two positions; atoms of the second orientation (refined site occupation factor of 17.9(6) %), N5' and N6' were refined isotropically with the thermal factor arbitrarily set at 0.1 Å².

The structure of [CoPy₄(N₃)₂]-2Py appeared to be very similar to the structure of the Ni-compound. The intensity data were of rather poor quality, however. For this reason the refinement was performed with the use of a very limited set of parameters (anisotropically for the Co and isotropically for all the remaining atoms; the geometry of the pyridines was taken unchanged from the Ni compound and kept rigid during structure refinement). The same kind of azide-group disordering was found. Atoms of the first orientation (78(1) %), N5 and N6, as well as atoms of the second orientation (22(1)%), N5' and N6', were refined with the fixed thermal factor 0.1 Å².

The resulting fractional atomic coordinates, bond lengths and angles for the Ni-clathrate are listed in Tables II–IV. The lists of coordinates of hydrogen atoms, anisotropic thermal parameters and F_0/F_c tables and all material for the Co-compound are available upon request from the author (J.L.).

3. Results and Discussion

3.1. GENERAL FEATURES

The results obtained completely support the supramolecular character of the compounds and allow their identification as Werner type clathrates. The structures are isostructural; the main architectural motif is analogous with that of other known clathrates of the general formula [MPy₄X₂]-2Guest. These compounds consist of isolated *trans*-[MPy₄(N₃)₂] host molecules and two crystallographically inequivalent pyridine guest species; the nearest contacts between atoms of neighbouring molecules are greater than the respective sums of their van der Waals radii. The formula [MPy₄(N₃)₂]-2Py adequately reflects the presence in each structure of two kinds of molecules with a molar ratio of 1 : 2.

3.2. THE [NiPy₄(N₃)₂]-2Py CLATHRATE

The structure of the [NiPy₄(N₃)₂] host complex revealed in the clathrate is depicted in Figure 1. The molecule has the central nickel atom in a distorted octahedral configuration with the terminal azido-ligands in the *trans*-positions (which are disordered over two orientations) and the pyridine moieties in the propeller conformation. The interplanar angles of 52–60° between the planes of the pyridine rings and the equatorial plane of the complex (Table V), suggest that this conformation type is the best from the viewpoint of intramolecular steric hindrances [17] and is the most frequently found [5]. The Ni—N_{Py} distances are within 2.14–2.16 Å. This is longer than in the similar propeller conformation of the [NiPy₄(NCS)₂] complex found in its clathrates with triphenylmethane (2.11–2.13 Å) [18] and pyridine (2.12 Å) [10], and longer than the values of 2.08–2.14 Å found in a similar clathrate

Table II. Fractional atomic coordinates ($\times 10^4$) and equivalent isotropic displacement parameters U_{iso} ($\text{\AA}^2 \times 10^3$) for the $[\text{NiPy}_4(\text{N}_3)_2]\cdot 2\text{Py}$ clathrate ($U_{\text{iso}} = 1/3(U_{11} + U_{22} + U_{33})$).

Atom	x/a	y/b	z/c	U_{iso}
<i>HOST:</i>				
Ni	193(1)	2500	2500	54(1)
N(1)	151(2)	2452(3)	3733(2)	57(1)
C(11)	506(3)	3268(3)	4162(2)	62(1)
C(12)	443(3)	3284(4)	4952(3)	74(1)
C(13)	9(3)	2425(4)	5320(3)	77(1)
C(14)	-360(3)	1586(4)	4889(3)	76(1)
C(15)	-274(3)	1624(4)	4103(2)	67(1)
N(2)	-1237(3)	2500	2500	57(1)
C(21)	-1709(3)	1729(3)	2103(2)	59(1)
C(22)	-2628(3)	1703(3)	2086(2)	62(1)
C(23)	-3106(4)	2500	2500	64(1)
N(3)	1625(3)	2500	2500	55(1)
C(31)	2097(3)	3278(3)	2117(2)	61(1)
C(32)	3021(3)	3306(4)	2102(2)	69(1)
C(33)	3489(4)	2500	2500	70(2)
N(4)	260(3)	743(3)	2504(3)	79(1)
First orientation of the azide ligand (82.1(6) %)				
N(5)	3(3)	-19(4)	2179(3)	56(1)
N(6)	-229(3)	-769(4)	-1798(3)	91(2)
Second orientation of the azide ligand (17.9(6) %):				
N(5')	19(21)	61(43)	2735(23)	100
N(6')	-227(16)	-769(26)	2905(16)	100
<i>GUEST 1:</i>				
N(1G)	7500	0	485(3)	77(2)
C(11G)	7068(3)	763(4)	82(3)	82(1)
C(12G)	7040(3)	801(4)	-702(3)	79(1)
C(13G)	7500	0	1096(4)	79(2)
<i>GUEST 2:</i>				
N(2G)	2500	0	4486(3)	76(2)
C(21G)	2172(3)	845(4)	4887(3)	82(1)
C(22G)	2150(4)	885(5)	5669(3)	91(2)
C(23G)	2500	0	6064(4)	100(2)

$[\text{NiPy}_4(\text{NO}_3)_2]\cdot 2\text{Py}$ [11]. The Ni—N_{azide} distance of 2.10 Å is also significantly longer than the Ni—N_{CS} distances found in the $[\text{NiPy}_4(\text{NCS})_2]$ molecule (2.05 [18] and 2.03 Å [10]).

Table III. Bond lengths (Å) (involving nonhydrogen atoms) found in the $[\text{NiPy}_4(\text{N}_3)_2]\cdot 2\text{Py}$ clathrate.

Ni—N(1)	2.161(3)
Ni—N(2)	2.139(4)
Ni—N(3)	2.142(4)
Ni—N(4)	2.095(4)
N(1)—C(11)	1.339(5)
C(11)—C(12)	1.387(5)
C(12)—C(13)	1.372(6)
C(13)—C(14)	1.369(6)
C(14)—C(15)	1.383(5)
N(1)—C(15)	1.340(5)
N(2)—C(21)	1.351(4)
C(21)—C(22)	1.376(5)
C(22)—C(23)	1.392(5)
N(3)—C(31)	1.344(4)
C(31)—C(32)	1.382(5)
C(32)—C(33)	1.377(5)
N(4)—N(5)	1.138(6)
N(5)—N(6)	1.168(6)
N(4)—N(5')	0.98(5)
N(5')—N(6')	1.10(4)
N(1G)—C(11G)	1.319(5)
C(11G)—C(12G)	1.375(6)
C(12G)—C(13G)	1.364(5)
N(2G)—C(21G)	1.322(5)
C(21G)—C(22G)	1.372(6)
C(22G)—C(23G)	1.366(6)

As shown in [18], the coordination bond parameters of this type of molecules are strongly dependent upon the packing peculiarities present in a given phase. In the majority of $[\text{MPy}_4\text{X}_2]\cdot 2\text{Py}$ clathrates ($\text{X} = \text{Br}, \text{I}, \text{NO}_2, \text{NO}_3, \text{HCOO}$) which crystallize in the *Ccca* space group [2, 6–9, 11] the molecule of a host complex is located on the 222 symmetry point. The introduction of the longer azido-ligand leads to overall distortion of the structure and decreases its symmetry. The displacement of the molecule from the 222 symmetry site results, in turn, in more distortion of the coordination octahedron and general elongation of all metal–ligand bonds in the title $[\text{NiPy}_4(\text{N}_3)_2]\cdot 2\text{Py}$ clathrate. The host molecule in the structure lies on a twofold axis coinciding with the N(2)—Ni—N(3) bonds, parallel to the *a*-direction. The symmetry decrease is also manifested in the inequivalency of the two guest pyridines (they lie on twofold axes parallel to *c*). Comparison of analogous projections of the title compound $[\text{NiPy}_4(\text{N}_3)_2]\cdot 2\text{Py}$ and the known $[\text{NiPy}_4(\text{NO}_3)_2]\cdot 2\text{Py}$ clathrate structures (Figure 2a,b) makes the above discussion more evident.

Table IV. Bond angles (deg.) found in the $[\text{NiPy}_4(\text{N}_3)_2] \cdot 2\text{Py}$ clathrate.

N(1)—N(1)—N(1)#1	176.7(2)
N(1)—Ni—N(2)	88.33(8)
N(1)—Ni—N(3)	91.67(8)
N(1)—Ni—N(4)	88.4(2)
N(1)—Ni—N(4)#1	91.8(2)
N(2)—Ni—N(3)	180
N(2)—Ni—N(4)	92.7(1)
N(3)—Ni—N(4)	87.3(1)
N(4)—Ni—N(4)#1	174.5(2)
Ni—N(1)—C(11)	122.0(3)
C(15)—N(1)—Ni	121.1(3)
C(15)—N(1)—C(11)	116.8(4)
N(1)—C(11)—C(12)	122.9(4)
C(11)—C(12)—C(13)	119.4(4)
C(12)—C(13)—C(14)	118.4(4)
C(13)—C(14)—C(15)	119.2(4)
N(1)—C(15)—C(14)	123.3(4)
C(21)—N(2)—Ni	121.5(2)
C(21)—N(2)—Ni	121.5(2)
C(21)—N(2)—C(21)	117.0(5)
N(2)—C(21)—C(22)	123.3(4)
C(21)—C(22)—C(23)	119.1(4)
C(22)—C(23)—C(22)	118.2(5)
C(31)—N(3)—Ni	121.7(2)
C(31)—N(3)—Ni	121.7(2)
C(31)—N(3)—C(31)	116.6(5)
N(3)—C(31)—C(32)	123.4(4)
C(31)—C(32)—C(33)	118.8(4)
C(32)—C(33)—C(32)	118.9(6)
N(5)—N(4)—Ni	141.1(5)
N(4)—N(5)—N(6)	175.0(8)
N(5')—N(4)—Ni	145(2)
N(4)—N(5')—N(6')	171(5)
C(G11)—N(G1)—C(G11)	115.3(6)
N(G1)—C(G11)—C(G12)	124.9(5)
C(G13)—C(G12)—C(G11)	117.8(5)
C(G12)—C(G13)—C(G12)	119.2(6)
C(G21)—N(G2)—C(G21)	115.8(6)
N(G2)—C(G21)—C(G22)	124.5(5)
C(G23)—C(G22)—C(G21)	118.0(6)
C(G22)—C(G23)—C(G22)	119.1(7)

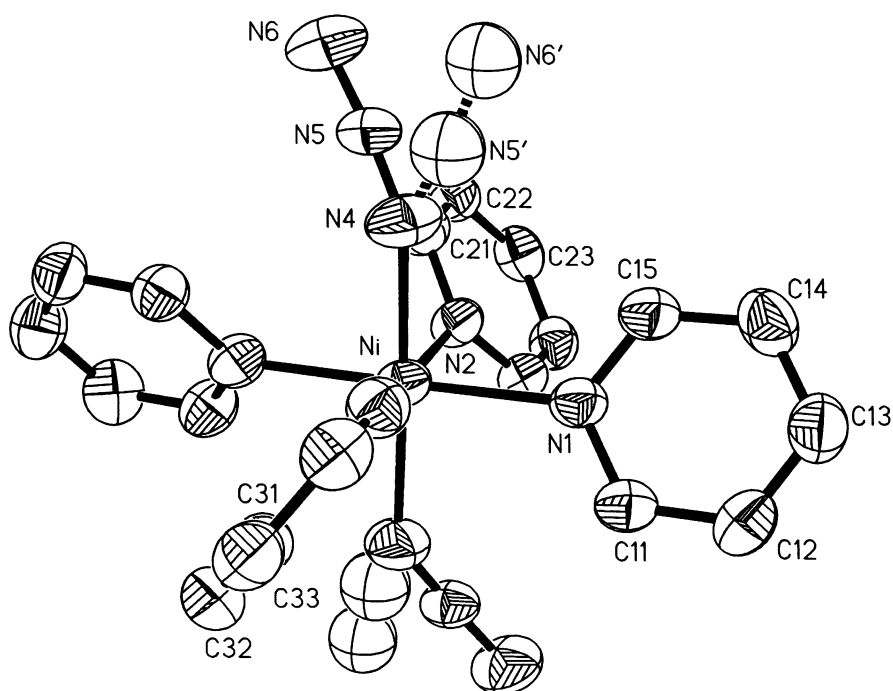


Figure 1. ORTEP projection [16] and the numbering scheme of the host and guest molecules in the $[\text{NiPy}_4(\text{N}_3)_2] \cdot 2\text{Py}$ clathrate. Ellipsoids are drawn at the 50% probability level; hydrogen atoms are omitted.

Table V. Comparison of the geometry of the host molecules found in the title clathrates.

		$[\text{NiPy}_4(\text{N}_3)_2] \cdot 2\text{Py}$	$[\text{CoPy}_4(\text{N}_3)_2] \cdot 2\text{Py}$
Distance, Å:	M-N1(Py1)	2.161(3)	2.201(5)
	M-N2(Py2)	2.139(4)	2.168(7)
	M-N3(Py3)	2.142(4)	2.189(7)
	M-N4(azide)	2.095(4)	2.084(1)
Angle, deg:	M-N4-NS	141.1(5)	143(2)
	N4-N5-N6	175.0(8)	171(3)
	M-N4-N5'	145(2)	140(4)
	M-N5'-N6'	171(5)	155(10)
Interplanar angle, deg:	equatorial (N1, N2, N3, M) – Py1	59.3(1)	59.9(3)
	equatorial – Py2	54.3(2)	54.0(8)
	equatorial – Py3	52.6(2)	54.8(8)
	equatorial – guest 1	54.4(2)	58.5(9)
	equatorial – guest 2	63.8(2)	63.2(9)
	(M, N4, N5, N6) – (M, N4, N5', N6')	83(1)	88(3)

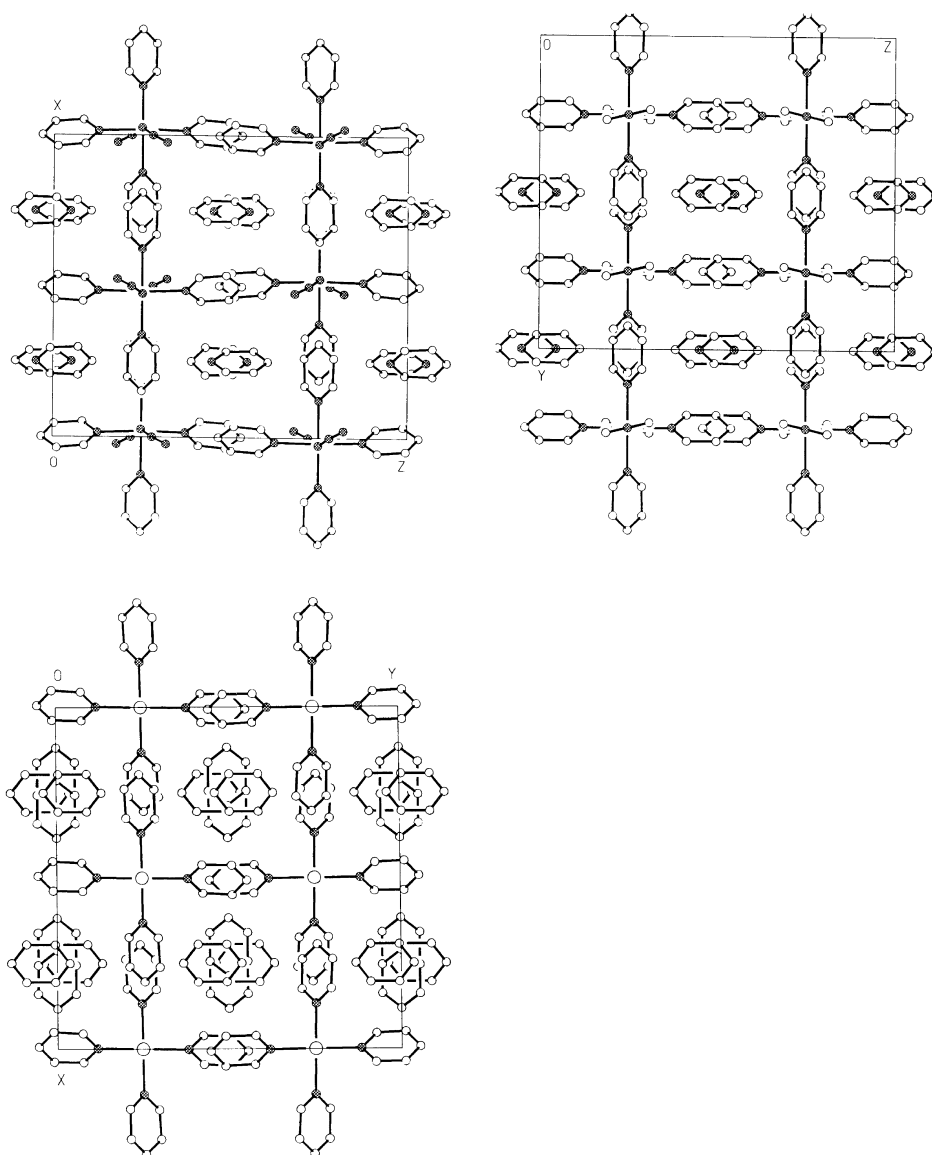


Figure 2. Projections of the studied and known clathrates along the channel axis directions: (a) $[\text{NiPy}_4(\text{N}_3)_2] \cdot 2\text{Py}$ (this work); (b) $\text{NiPy}_4(\text{NO}_3)_2 \cdot 2\text{Py}$ [11]; (c) $[\text{NiPy}_4(\text{NCS})_2] \cdot 2\text{Py}$ [10]. For clarity nitrogen atoms are hatched; hydrogen atoms are omitted.

In the $[\text{CuPy}_4(\text{NO}_3)_2] \cdot 2\text{Py}$ clathrate [12, 13] the same structure distortion occurs, but this is due to substantial elongation of the Cu–nitrate bond (2.42 Å), which appears to be equivalent (from the point of view of non-bonded interactions in the clathrates) in this case to the elongation of the X-ligand itself. The clathrate also crystallizes in the $Pnma$ space group.

In contrast, the introduction of the NCS ligand leads to an increase in symmetry and to remarkable shortening of coordination bonds. In this case the possibility of the NCS ligand being constrained by crystal symmetry to an axial position results in minimization of host the intramolecular ligand repulsions in the tetragonal $[\text{NiPy}_4(\text{NCS})_2] \cdot 2\text{Py}$ structure [10] (Figure 2c).

As seen from Figure 2, the packing motif in the title compound, $[\text{NiPy}_4(\text{N}_3)_2] \cdot 2\text{Py}$, is very similar to that in other known $[\text{MPy}_4\text{X}_2] \cdot 2\text{Py}$ clathrates, in spite of the substantially different crystallographic parameters of these three compounds. The guest species are located in channels which run parallel to the axial direction of the host complex molecules. Throughout the series, the general features of this clathrate matrix do not change qualitatively.

3.3. THE $[\text{CoPy}_4(\text{N}_3)_2] \cdot 2\text{Py}$ CLATHRATE

The general structural features of $[\text{CoPy}_4(\text{N}_3)_2] \cdot 2\text{Py}$ are very similar to those of its Ni-counterpart with obvious differences in the coordination bond lengths; the comparison of respective data is given in Table V. Thermal motion or disordering of the X-ligand is frequently found in the compounds of the type $[\text{MPy}_4\text{X}_2] \cdot 2\text{Py}$. This is observed, for example, for nitrate ligand in $[\text{NiPy}_4(\text{NO}_3)_2] \cdot 2\text{Py}$ [11] and this seems to be responsible for the polymorphous phase transition observed in the clathrate $[\text{CdPy}_4(\text{NO}_3)_2] \cdot 2\text{Py}$ [19].

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