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# Peculiarities of the Crystal Structure and Packing of the Host and Guest Molecules in the $[M(4\text{-MePy})_4(NCS)_2]\cdot 0.67(4\text{-MePy})\cdot 0.33\ H_2O$ Clathrates ($M = Cu(II), Mn(II)$; 4-MePy = 4-Methylpyridine)

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**Abstract.** The crystal structures of the two clathrates with the composition $[M(4\text{-MePy})_4(NCS)_2]\cdot 0.67(4\text{-MePy})\cdot 0.33\ H_2O$ ($M = Cu(II), Mn(II)$; 4-MePy = 4-methylpyridine) have been determined. These compounds are trigonal, with the $[M(4\text{-MePy})_4(NCS)_2]$ host molecules being centrosymmetric. The parameters of the unit cells are $a = 27.365(7)$ and $27.738(6)$, $c = 11.303(9)$ and $11.250(8)$ Å, $V = 7325(2)$ and $7493(2)$ Å$^3$, space group $R\bar{3}$, $R = 0.053$ and $0.109$ for $M = Cu(II)$ and Mn(II), respectively. For $Z = 9$ $d_{calcd}$ is equal to 1.271 and 1.225 g/cm$^3$, and $d_{measd}$ is equal to 1.252(2) and 1.213(2) g/cm$^3$ for the Cu and Mn clathrates, respectively. The coordination environment of the metal atoms in these compounds is an irregular octahedron, while in the Mn compound these distortions are rather small ($Mn—N_{MePy}$ 2.30, 2.34 Å, $Mn—N_{NCS}$ 2.18 Å, and $Cu—N_{MePy}$ 2.06 Å, $Cu—N_{NCS}$ 1.98 Å and $Cu—N_{MePy}$ 2.50 Å).

The molecular packing in the structures is such that the channels of variable diameter are formed along the short cell dimension (the maximum diameter is $\cong 10$ Å, the minimum being $\cong 6$ Å) where the guest 4-MePy and $H_2O$ molecules are placed.

**Key words.** Crystal structure, clathrate, 4-methylpyridine, Cu(II), Mn(II).

## 1. Introduction

Complex compounds of the general formula $[MA_4X_2]$ [1], where M is a transition metal(II), X is an anionic ligand (halogen or pseudohalogen of the $NCS^-$ type), and A is a neutral ligand, such as a substituted pyridine, are of interest because of their unusual ability selectively to include different organic molecules resulting in the formation of clathrates $[MA_4X_2]\cdot G$. This property makes it possible to use these clathrates for the separation of a number of organic compounds by gas- and liquid-phase chromatography [2, 3].

Clathrates with the $[Ni(4\text{-MePy})_4(NCS)_2]$ host component have been investigated the most [4]; they form different structures depending on the type of the guest molecule.

In this paper the results of the X-ray diffraction study of clathrates of $[Cu(4\text{-MePy})_4(NCS)_2]\cdot 0.67(4\text{-MePy})\cdot 0.33\ H_2O$ (**I**) and $[Mn(4\text{-MePy})_4\ (NCS)_2]\cdot 0.67\ (4\text{-MePy})\cdot 0.33\ H_2O$ (**II**) are reported. These compounds have a general guest/host ratio equal to 1 : 1, but differ greatly from the clathrates investigated earlier.

We have previously reported the structure of the clathrate [Cd(4-MePy)$_4$(NCS)$_2$]·0.67(4-MePy)·0.33 $H_2O$ (**III**) [5] of a similar composition.

## 2. Experimental

Green, needle-shaped crystals of **I** were obtained by slow cooling of a saturated solution of [Cu(4-MePy)$_2$(NCS)$_2$] in 4-methylpyridine at 50 °C [6].

The copper analysis using complexometric titration of a 0.01 N solution by EDTA solution revealed 10.18 wt.% of Cu (10.24 wt.% of Cu was calculated for the compound **I**).

Thermogravimetric analysis of the clathrate **I** indicated that two 4-methylpyridine ligands were removed along with the guest in the course of the thermal dissociation [7], giving [Cu(4-MePy)$_2$(NCS)$_2$] as the decomposition product.

Despite the use of dry reagents (4-methylpyridine and [Cu(4-MePy)$_2$(NCS)$_2$] containing 0.04 wt.% $H_2O$) we have obtained a stoichiometric (in accordance with the formula) amount of water in clathrate **I**. This process is analogous to the process by which water is concentrated in clathrate **III**, which has been described earlier [5, 8].

The transparent, colourless, needle-shaped crystals of **II** were obtained by slow crystallization of a saturated solution of [Mn(4-MePy)$_4$(NCS)$_2$]·$y$(4-MePy) in 4-methylpyridine at 50 °C. (This compound, with $y \cong 0.6$, was obtained while trying to synthesize the [Mn(4-MePy)$_4$(NCS)$_2$] host by adding the equivalent quantity of 4-methylpyridine to the aqueous solution containing $Mn^{2+}$ and $SCN^-$ ions.)

The analysis of the clathrate **II** for manganese and water (by the Karl Fisher method) gave the following values: 9.04 wt.% of Mn (8.88 wt.% of Mn was calculated by using formula **II**), 0.82 wt.% of $H_2O$ (0.97 wt.% of $H_2O$ calculated). The thermal dissociation of clathrate **II** is similar to the dissociation of clathrate **I** removing 4-methylpyridine into the gas phase [9].

The crystals of clathrates **I** and **II** are unstable in air and were protected by a varnish prepared by dissolving celluloid in 4-methylpyridine.

The experimental data are shown in Table I.

The structures were initially solved in the triclinic system with unit cell parameters of: $a = 11.303(7)$, $b = 16.236(9)$, $c = 16.443(8)$ Å, $\alpha = 114.74(4)$, $\beta = 103.40(5)$, $\gamma = 103.48(4)°$, $V = 2441(2)$ Å$^3$, $Z = 3$, $d_{\text{calcd}} = 1.266$ g/cm$^3$, $d_{\text{measd}} = 1.252(2)$ g/cm$^3$, 3783 $I_{hkl}$, $R = 0.0945$ for **I**, and $a = 11.250(6)$, $b = 16.443(8)$, $c = 16.441(8)$ Å, $\alpha = 114.94(3)$, $\beta = 103.21(3)$, $\gamma = 103.16(3)°$, $V = 2497(2)$Å$^3$, $Z = 3$, $d_{\text{calcd}} = 1.220$ g/cm$^3$, $d_{\text{measd}} = 1.213(2)$ g/cm$^3$, 2950 $I_{hkl}$, $R = 0.136$ for **II**. The referees suggested the trigonal system as being more appropriate and the unit cell has been transformed using the matrix.

$$\begin{vmatrix} 1 & 2 & 1 \\ -1 & -1 & -2 \\ -1 & 0 & 0 \end{vmatrix}.$$

Accordingly the unit cell dimensions and the $I_{hkl}$ arrays have been transformed.

The structures have been solved by the heavy atom method, the coordinates of the Cu and Mn atoms have been defined from the Patterson function three-

Table I. Crystal data and experimental parameters for the clathrate structures **I** and **II**.

| Clathrate<br>Formula | **I**<br>$[Cu(4\text{-}MePy)_4(NCS)_2]\cdot G^*$ | **II**<br>$[Mn(4\text{-}MePy)_4(NCS)_2]\cdot G^*$ |
|---|---|---|
| Space group | $R\bar{3}$ | $R\bar{3}$ |
| $a$, Å | 27.356(7) | 27.738(6) |
| $c$, Å | 11.303(9) | 11.250(8) |
| $V$, $Å^3$ | 7325(2) | 7493(2) |
| $Z$ | 9 | 9 |
| $D_{measd}$, $g/cm^3$ | 1.252(2) | 1.213(2) |
| $d_{calcd}$, $g/cm^3$ | 1.271 | 1.225 |
| $t$, °C | 25 | 25 |
| Radiation | $MoK_\alpha$ | $MoK_\alpha$ |
| Diffractometer | Syntex $\mathrm{P2_1}$ | Syntex $P2_1$ |
| Scanning | $\theta/2\theta$ | $\theta/2\theta$ |
| $2\theta_{max}$, deg | 40 | 40 |
| Measured reflections | 4864 | 4941 |
| Observed reflections ($I > 3\sigma I$) | 1226 | 1072 |
| $R$ | 0.053 | 0.109 |

$^*G = 0.67(4\text{-}MePy)\cdot 0.33\ H_2O$

dimensional distribution $P(uvw)$ independently of compound **III**, because the isostructural nature of the compounds was not evident. The final positions of all the atoms were found in the space group $R\bar{3}$ and refined by full-matrix least-squares calculations with isotropic and anisotropic factors using YANX [10]. The positions of the guest molecules are given in an isotropic approximation and have not been refined. The final coordinates and temperature factors are shown in Tables II and III.

## 3. Description and Analysis of the Structures

Compounds **I**, **II** and **III** are isostructural in the arrangement of both the host and the guest molecules, provided that the unit cell of **III** is converted according to the

$$\begin{vmatrix} -1 & -2 & 0 \\ 2 & 1 & 1 \\ 0 & 0 & 1 \end{vmatrix}$$

matrix and taking account of the fact that in [5] the $b$-dimension was erroneously given as 16.942 Å instead of the correct value of 16.492 Å. However, it is noteworthy that the $NCS^-$ and 4-MePy ligand orientations are different in structures **I** and **II** (Figure 1).

In compounds **I** and **II** the metal atom occupies the centre of inversion and has a distorted octahedral environment of N atoms of the (4-MePy)-rings and the N atoms of the NCS groups. The distortion of the octahedron in the Mn clathrate is rather small ($Mn—N_{MePy}$ bonds are 2.30, 2.34 Å and $Mn—N_{NCS}$ bonds are 2.18 Å) while the copper coordination polyhedra in clathrate **I** is an elongated bipyramid

Table II. Fractional atomic coordinates ( $\times 10^4$) with e.s.d.s in parentheses and equivalent temperature factors $U^*_{eq}$ ( $\times 10^3$) of the non-hydrogen atoms for the [Cu(4-MePy)$_4$(NCS)$_2$]·0.67 (4-MePy)·0.33 $H_2O$ clathrate.

| | $x$ | $y$ | $z$ | $U_{eq}$ (Å$^2$) |
|---|---|---|---|---|
| *Host* | | | | |
| Cu | 5000 | 0 | 0 | 63.3(8) |
| S(1) | 5832(1) | 1129(1) | −3304(2) | 125(2) |
| N(1) | 5291(2) | 584(3) | −1248(6) | 70(4) |
| N(2) | 5809(2) | 169(3) | 346(5) | 60(4) |
| N(3) | 4880(3) | −732(3) | −1480(6) | 72(5) |
| C(1) | 5518(3) | 819(3) | −2100(7) | 64(5) |
| C(2) | 6245(2) | 690(3) | 273(6) | 67(6) |
| C(3) | 6791(3) | 812(4) | 444(7) | 72(6) |
| C(4) | 6905(3) | 381(4) | 690(7) | 80(6) |
| C(5) | 6454(4) | −158(4) | 771(7) | 79(6) |
| C(6) | 5919(3) | −244(3) | 593(7) | 71(6) |
| C(7) | 7502(4) | 498(5) | 850(9) | 123(9) |
| C(8) | 4772(3) | −1251(4) | −1224(8) | 78(6) |
| C(9) | 4696(3) | −1645(4) | −2080(9) | 84(7) |
| C(10) | 4720(3) | −1514(4) | −3260(8) | 84(7) |
| C(11) | 4824(4) | −977(4) | −3522(8) | 92(7) |
| C(12) | 4904(4) | −609(4) | −2614(8) | 86(7) |
| C(13) | 4635(5) | −1929(4) | −4230(9) | 128(9) |
| *Guest* | | | | |
| N(4) | 3454 | 7185 | −247 | |
| C(14) | 3650 | 7578 | 673 | |
| C(15) | 4023 | 7379 | 1070 | |
| C(16) | 3489 | 6484 | −820 | |
| C(17) | 3557 | 6236 | 305 | |
| C(18) | 4084 | 6828 | 737 | |
| C(19) | 4358 | 6636 | 1238 | |
| O | 0 | 0 | 0 | |

$^*U_{eq} = 1/3\ (U_{11} + U_{22} + U_{33})$

(coordination number = 6(4 + 2)), one of the Cu—N$_{MePy}$ bond lengths being equal to 2.50 Å. The other 4-MePy ligand and the NCS$^-$ group lie in the equatorial plane of the bipyramid, the Cu—N$_{NCS}$ and Cu—N$_{MePy}$ bond lengths being similar (the Cu—N$_{MePy}$ bond length is 2.06 Å and Cu—N$_{NCS}$ bond length is 1.98 Å). The bond lengths and angles for the [Cu(4-MePy)$_4$(NCS)$_2$] and [Mn(4-MePy)$_4$(NCS)$_2$] host molecules are given in Tables IV and V.

Thus, an increase of the metal-ligand bond length is observed depending on the size of the central atom in the order Cu, Mn and Cd

| | Cu | Mn | Cd |
|---|---|---|---|
| M—N$_{CS}$ (Å) | 1.98 | 2.18 | 2.28 |
| M—N$_{MePy}$ (Å) | 2.06 | 2.30 | 2.38 |
| M—N$_{MePy}$ (Å) | 2.50 | 2.34 | 2.49 |

Table III. Fractional atomic coordinates ($\times 10^4$) with e.s.d.s in parentheses and equivalent temperature factors $U_{eq}$ ($\times 10^3$) of the non-hydrogen atoms for the $[Mn(4\text{-}MePy)_4(NCS)_2]\cdot 0.67(4\text{-}MePy)\cdot 0.33\ H_2O$ clathrate.

| | $x$ | $y$ | $z$ | $U_{eq}$ (Å) |
|---|---|---|---|---|
| *Host* | | | | |
| Mn | 5000 | 0 | 0 | 73(3) |
| S(1) | 4668(4) | −1194(3) | 3365(7) | 164(7) |
| N(1) | 4680(6) | −648(7) | 1350(9) | 80(10) |
| N(2) | 4298(7) | 191(8) | 380(10) | 80(10) |
| N(3) | 4447(6) | −665(7) | −1420(10) | 70(10) |
| C(1) | 4668(7) | −881(8) | 2210(20) | 70(10) |
| C(2) | 3790(10) | −210(10) | 660(20) | 100(20) |
| C(3) | 3350(9) | −140(10) | 860(20) | 90(20) |
| C(4) | 3430(10) | 390(10) | 750(20) | 110(20) |
| C(5) | 3950(10) | 830(10) | 440(20) | 90(20) |
| C(6) | 4363(8) | 697(8) | 270(20) | 80(10) |
| C(7) | 2950(10) | 500(10) | 900(30) | 160(30) |
| C(8) | 4050(10) | −1180(10) | −1150(20) | 90(2) |
| C(9) | 3739(8) | −1571(8) | −1960(20) | 80(20) |
| C(10) | 3813(8) | −1460(10) | −3160(20) | 80(20) |
| C(11) | 4220(10) | −930(10) | −3410(20) | 90(20) |
| C(12) | 4524(7) | −554(8) | −2590(20) | 60(10) |
| C(13) | 3470(10) | −1890(10) | −4060(20) | 110(20) |
| O | 0 | 0 | 0 | |
| N(4) | 3310(20) | 6900(20) | 4160(30) | |

This tendency is violated only for the Cu clathrate because of the octahedral distortion characteristic of copper(II) complexes.

Root-mean-square planes have been constructed for the host complexes in structures **I** and **II**. These planes cross over the metal atoms, the atoms of the separate (4-MePy) rings and of the NCS groups. The atom deviations from the proper planes do not exceed 0.04 Å. The plane angle of the (4-MePy) rings is 83° (**I**) and 84° (**II**), and the MNCS plane forms with the planes of the (4-MePy) rings angles of 65° and 34° (**I**), and 66° and 31° (**II**), respectively.

In addition, the torsion angles of the $N_{NCS}MN_{MePy}\ C_{MePy}$ type ($\varphi$) have been calculated in structures **I** and **II** to describe a complex stability with allowance for the non-valent interactions of two neighbouring (4-MePy) rings [11]. The location of the two rings of the Cu complex is determined by the angles 35.7° and 168.3°, respectively. The corresponding values for the Mn complex are 35.8° and 13.1°, respectively. Thus, the torsion angles in structures **I** and **II** correspond to the minimum non-bonded interaction energy of the 4-MePy ligands for the $[Ni(4\text{-}MePy)_4(NCS)_2$ host [11].

The $(2\bar{1}0)$ plane section of the channel for compound **I** is shown in Figure 3. The channel section has a maximum diameter of $\cong 10$ Å at $z \cong 0$, and a minimum diameter of $\cong 6$ Å at $z \cong 1/3$. The channel shape requires that the guest molecules lie along the channel. The statistical distribution of the (4-MePy) molecules

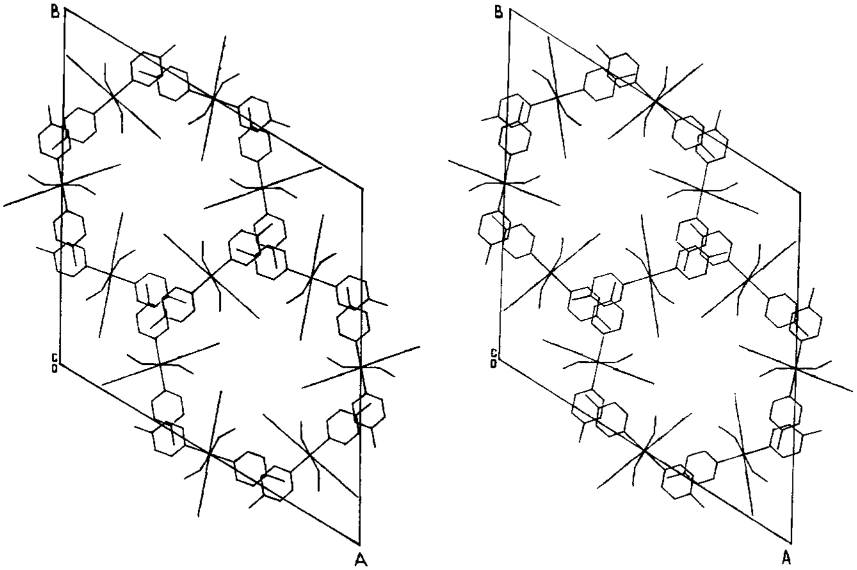

Fig. 1. Projections of the $[Cu(4\text{-}MePy)_4(NCS)_2]\cdot 0.67(4\text{-}MePy)\cdot 0.33\ H_2O$ (a, left) and $[Mn(4\text{-}MePy)_4(NCS)_2]\cdot 0.67(4\text{-}MePy)\cdot 0.33\ H_2O$ (b, right) crystal structures on the (001) plane.

Table IV. Bond lengths (Å) and angles (deg) for the host $[Cu(4\text{-}MePy)_4(NCS)_2]$ molecule.

| | | | |
|---|---|---|---|
| Cu—N(1) | 1.975(7) | Cu—N(2) | 2.060(5) |
| Cu—N(3) | 2.503(6) | S—C(1) | 1.606(9) |
| N(1)—C(1) | 1.153(8) | N(2)—C(2) | 1.326(8) |
| N(2)—C(6) | 1.334(8) | N(3)—C(8) | 1.329(9) |
| N(3)—C(12) | 1.318(9) | C(2)—C(3) | 1.37(1) |
| C(3)—C(4) | 1.39(1) | C(4)—C(5) | 1.37(1) |
| C(4)—C(7) | 1.51(1) | C(5)—C(6) | 1.38(1) |
| C(8)—C(9) | 1.38(1) | C(9)—C(10) | 1.38(1) |
| C(10)—C(11) | 1.38(1) | C(10)—C(13) | 1.51(1) |
| C(11)—C(12) | 1.38(1) | | |
| N(1)—Cu—N(3) | 90.5(2) | Cu—N(1)—C(1) | 159.3(6) |
| N(1)—Cu—N(2) | 90.0(2) | Cu—N(2)—C(6) | 121.4(5) |
| N(2)—Cu—N(3) | 90.1(2) | Cu—N(3)—C(8) | 125.4(6) |
| Cu—N(2)—C(2) | 121.1(5) | C(8)—N(3)—C(12) | 116.2(7) |
| Cu—N(3)—C(12) | 118.4(5) | N(3)—C(8)—C(9) | 123.1(7) |
| S—C(1)—N(1) | 178.2(7) | C(9)—C(10)—C(11) | 116.3(8) |
| C(2)—N(2)—C(6) | 117.5(6) | C(8)—C(9)—C(10) | 120.4(8) |
| N(2)—C(2)—C(3) | 122.5(7) | C(9)—C(10)—C(13) | 122.6(9) |
| C(3)—C(4)—C(5) | 117.3(7) | C(10)—C(11)—C(12) | 119.4(8) |
| C(5)—C(4)—C(7) | 121.2(9) | N(3)—C(12)—C(11) | 124.7(8) |
| C(2)—C(3)—C(4) | 120.0(7) | C(11)—C(10)—C(13) | 121.1(9) |
| C(3)—C(4)—C(7) | 121.4(9) | | |
| C(4)—C(5)—C(6) | 118.9(8) | | |
| N(2)—C(6)—C(5) | 123.8(7) | | |

Table V. Bond lengths (Å) and angles (deg) for the host $[Mn(4\text{-}MePy)_4(NCS)_2]$ molecule.

| | | | |
|---|---|---|---|
| Mn—N(1) | 2.18(2) | Mn—N(2) | 2.30(1) |
| Mn—N(3) | 2.34(1) | S—C(1) | 1.57(2) |
| N(1)—C(1) | 1.15(2) | N(2)—C(2) | 1.33(2) |
| N(2)—C(6) | 1.33(2) | N(3) – C(8) | 1.32(2) |
| N(3)—C(12) | 1.34(2) | C(2)—C(3) | 1.35(2) |
| C(3)—C(4) | 1.38(3) | C(4)—C(5) | 1.39(3) |
| C(4)—C(7) | 1.53(3) | C(5)—C(6) | 1.38(2) |
| C(8)—C(9) | 1.36(2) | C(9)—C(10) | 1.38(3) |
| C(10)—C(11) | 1.36(3) | C(10)—C(13) | 1.48(3) |
| C(11)—C(12) | 1.34(2) | | |
| N(1)—Mn—N(3) | 89.5(5) | Mn—N(1)—C(1) | 159(2) |
| N(1)—Mn—N(2) | 91.0(6) | Mn—N(2)—C(6) | 123(1) |
| N(2)—Mn—N(3) | 90.7(5) | Mn—N(3)—C(8) | 123(1) |
| Mn—N(2)—C(2) | 121(1) | C(8)—N(3)—C(12) | 115(2) |
| Mn—N(3)—C(12) | 122(1) | N(3)—C(8)—C(9) | 124(2) |
| S—C(1)—N(1) | 179(2) | C(9)—C(10)—C(11) | 113(2) |
| C(2)—N(2)—C(6) | 116(2) | C(8)—C(9)—C(10) | 121(2) |
| N(2)—C(2)—C(3) | 125(2) | C(9)—C(10)—C(13) | 122(2) |
| C(3)—C(4)—C(5) | 121(2) | C(10)—C(11)—C(12) | 124(2) |
| C(5)—C(4)—C(7) | 118(2) | N(3)—C(12)—C(11) | 122(2) |
| C(2)—C(3)—C(4) | 118(2) | C(11)—C(10)—C(13) | 125(2) |
| C(3)—C(4)—C(7) | 121(2) | | |
| C(4)—C(5)—C(6) | 115(2) | | |
| N(2)—C(6)—C(5) | 125(2) | | |

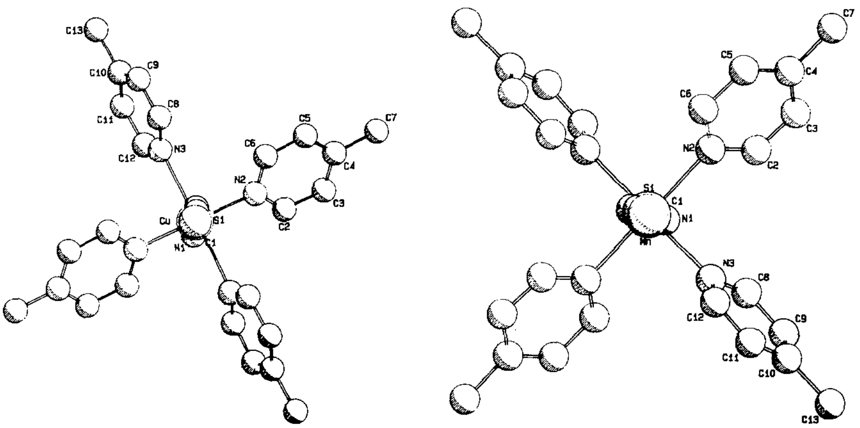

Fig. 2. Molecules of the host in $[Cu(4\text{-}MePy)_4(NCS)_2]\cdot 0.67(4\text{-}MePy)\cdot 0.33\ H_2O$ (a, left) and $[Mn(4\text{-}MePy)_4(NCS)_2]\cdot 0.67(4\text{-}MePy)\cdot 0.33\ H_2O$ (b, right).

observed in the channel cavities, the maximum dimensions of which allow the guest molecules to rotate around the three-fold axis with the $H_2O$ molecules in the centre of the inversion.

The crystals of compound **II** were of a bad quality, and therefore it was possible to localize unambiguously only the positions of the $O_{H_2O}$ atom and the N atoms of the (4-MePy)-rings.

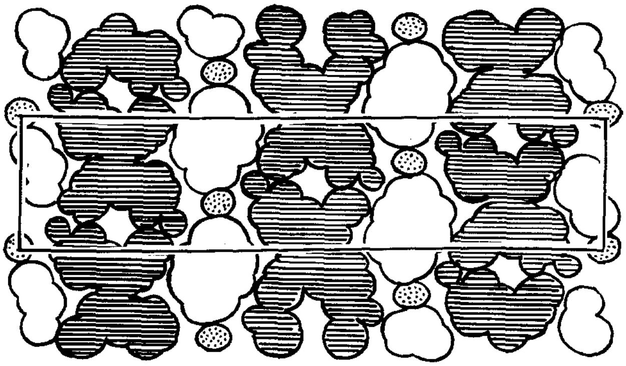

Fig. 3. The channel cross-section of the $(2\bar{1}0)$ plane for the clathrate $[Cu(4\text{-}MePy)_4(NCS)_2]\cdot 0.67(4\text{-}MePy)\cdot 0.33\,H_2O$.

We have not been able to check the transformations in clathrate **III** from the space group $P1$ to $R\bar{3}$ due to lack of samples and contemporary records of $I_{hkl}$.

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