ESRF	Experiment title: Self-Assembled Supramolecular Architectures	Experiment number: CH-714
Beamline: ID11	**Date of experiment:** from: 15/7/99 to: 20/7/99	**Date of report:** 25-Aug-99
Shifts: 15	**Local contact(s):** **Dr. Gavin Vaughan**	*Received at ESRF:* 0 3 SEP. 1999

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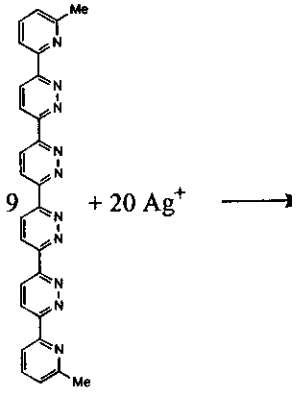
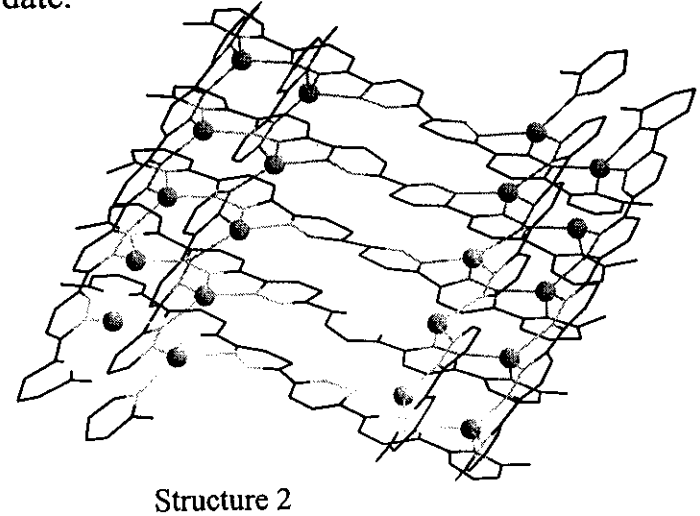
Report:

We have successfully used beamline ID11 to solve crystal structures of a number of previously unknown compounds. The experiment consisted of X-ray diffraction data collection on monocrystals that are extremely sensitive to solvent loss and are thermally unstable. Data sets were measured at 250K on crystals of typical dimension 50 x 20 x 20 μm at 25KeV with the Bruker "Smart" CCD Camera area detector system. The resulting monocrystal from the self-assembly of 20 Ag^+ ions with 9 equivalents of ligand (**I**) has been characterized to be a 5x4 grid type structure (**2**) (see Figure 1). This is the largest grid structure of its type known to date.



Ligand (I)

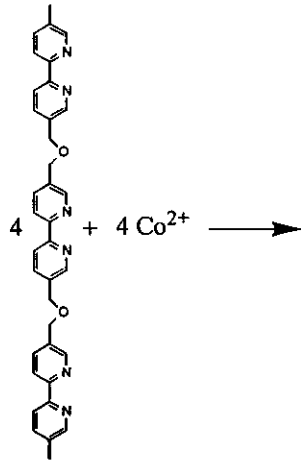


Structure 2

Figure 1

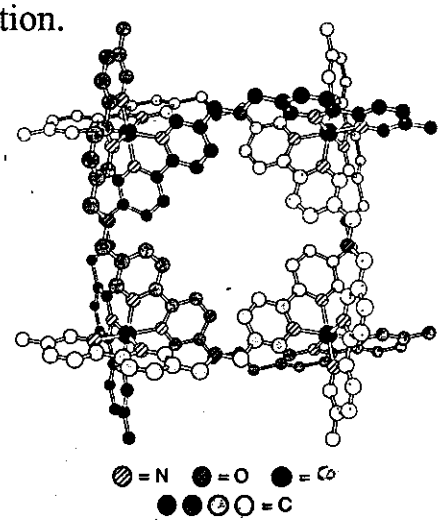
The central ligand (**a**) had undergone a 180° rotation about the central C-C bond and thus structure (**2**) shows a conformation type of 'two-over and two-under' with respect to positioning of the Ag^+ rows in the grid. Each silver ion has a distorted tetrahedral geometry with Ag–N bond lengths of 2.20–2.35Å. The results are very recent and the structure is still being refined. The manuscript describing compound (**2**) has been prepared and is awaiting the crystal structure refinement.

The elongated tris-bipyridine (**3**) was found to yield a tetrameric circular helicate (**4**) with Co^{2+} ions. The data collection was carried out on a yellow monocrystal of size 50 x 20 x 15μm. Initial result shows the compound to have the structure shown in Figure 2. The four Co^{2+} centres to have an octahedral geometry with Co–N distance of 2.13-2.16Å. The manuscript describing compound (**4**) is in preparation.



Tris-bipyridine (3)

Figure 2



Tetrameric Circular Helicate (4)

Data was also collected on a very unstable crystal sample that is expected to have the structure (**5**) shown in Figure 3. The structure is expected to form a combination of a 2x2 grid with a double helix at each corner (a "grid-helix" system). The data set was weak and we ran out of beam time. Additional data collections would be required to confirm the unique structure below.