ESRF	**Experiment title: Microcrystal Diffraction of Molecular Sieves**	**Experiment number:** CH-417
Beamline: ID11	**Date of experiment:** from: 26-Mar-98 to: 30-Mar-98	**Date of report:** 6-Aug-98
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Report:

We have successfully used beamline ID1 1 to solve the crystal structures of a number of previously unknown materials. The mixed valence iron phosphate **(1)** was found to adopt a structure similar to the mineral ohmsteadite. The structure has a three-dimensional framework of linked PO_4 and FeO_6 polyhedra, which enclose small channels occupied by K^+ and H_2O. This structure was solved using a crystal size of 60 x 15 x 5 μm. An unusual and novel layered aluminophosphate material was also characterized; $[NH_3CH_2CH_2NH_3]_{2.5}[Al_4H(HPO_4)_4(H_2PO_4)_2(C_2O_4)_4$ (2) is the first known metal phosphate to contain organic cations within the framework of the structure. It consists of chains of corner linked AlO_6 and PO_4 polyhedra linked into sheets via bridging PO_4 groups and $-C_2O_4$ . . .HOP- hydrogen bonds to give an open framework structure enclosing the templates.

The structure of a novel layered gallium phosphate has also been solved. This structure was elucidated from a very small crystal (15 x 10 x -3 μm) The composition of GaPO-DMAP (3) was calculated to be $Ga_4(HPO_4)(PO_4)_3.H_2O.2[C_7N_2H_{10}]$. The structure of GaPO-DMAP consists of a novel basket-like cage secondary building unit constructed from five phosphate tetrahedra and five gallium-centred polyhedra, two of which are GaO_4F units while the remaining three are GaO_4 tetrahedra. The two GaO_4F units share one vertex; the fluorine atom. The fluorine atom can be described as sitting inside the basket, which is defined by (if we discount the presence of the fluorine) the equivalent of four four-ring units and a compound six ring. The fluorine atom does not sit in the exact centre of this cage, but is shifted towards the two gallium atoms with which it makes bonds.

The novel secondary building units are linked together in an end to end fashion to form columns that run parallel to the (100) direction. These columns are then linked into sheets by four rings. These layers are organic material in which the DMAP units are stacked in a "herringbone" arrangement with the pyridine hydrogen atoms hydrogen bonded to the GaPO layers through pendant oxygen atoms. Two of these oxygens are connected to phosphorus atoms, while two are part of the GaO_4F polyhedra.

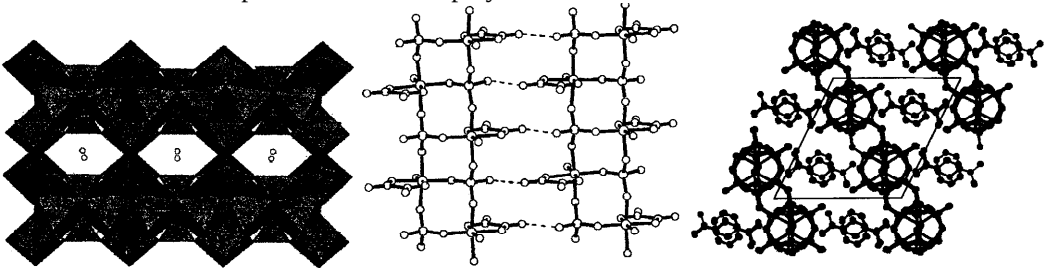

Figure The crystal structures of compounds I,2 and 3.

We also examined the templating role of the trimethyenediquinuclidinium cation in the formation of a molecular sieve with the MAPO-56 structure. The single crystal structure of this material was refined using data from ID1 1 and the template located from difference Fourier maps. This is the first time that a template molecule has been located from the diffraction data in this material, and the results are helping us towards an understanding of how templating in microporous materials occurs.

Manuscripts describind compounds 1,2 and 3 have been submitted to J. Sol. St, Chem, Chem. Comm. and J. Mater Chem. respectively. The MAPO-56 paper is in preparation