ESRF	**Experiment title:** Analysis of complex structures using high-resolution powder diffraction data	**Experiment number:** CH-430
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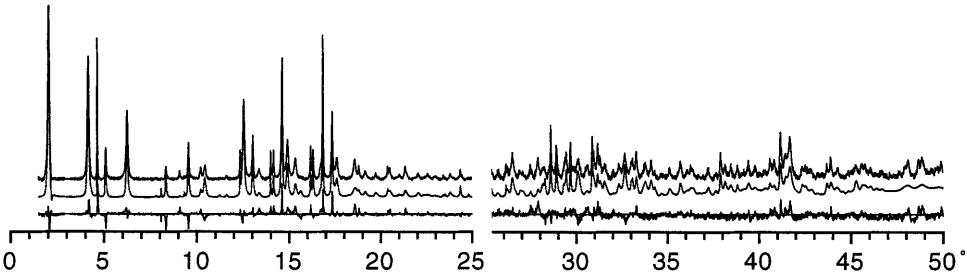
Report:

High resolution powder diffraction data were collected on four samples: the layered precursor to the high-silica zeolite ITQ-1 **(MWW** topology), the calcined form of the aluminosilicate IM-5 of unknown structure, a post-synthesis modification of the zeolite EMC-2 **(EMT** topology), and the polypeptide sa322.

ITQ-1(P): The data for ITQ-l(P) were of sufficiently high quality that the layer structure of this precursor could be elucidated, and a number of points regarding its transformation to the fully 4-connected, 3-dimensional framework of ITQ-1 upon calcination clarified. Rietveld refinement was combined with molecular simulation to locate the hexamethyleneimine (HMI) molecules and the *N,N,N*-trimethyl-(1 -adamantyl) ammonium ($TMADA^+$) ions used in the synthesis. In each unit cell, there are two $TMADA^+$ ions and three HMI molecules within the layer, and four HMI molecules between the layers. Both the powder data and the molecular simulations indicate that the HMI molecules between the layers are disordered. The layers differ slightly from the

proposed structure in that some Si-OH groups thought to be at the surface of the layer are missing and that anionic 3-ring silicate species appear to lie between two $TMADA^+$ ions nestled in adjacent layers. The structure refinement is still in progress (see profile below). Current R-values are $R_F = 0.082$ and $R_{wp} = 0.241$ ($R_{exp} = 0.127$).



Observed (top), calculated (middle) and difference (bottom) profiles for the present state of the refinement of ITQ-l(P). The second half of the profile has been scaled up by a factor of 5 to show more detail.

IM-5: The IM-5 sample proved to contain several unidentified impurities. With the help of electron diffraction micrographs, it was possible to obtain a preliminary indexing of the high-resolution powder diffraction pattern. The unit cell appears to be orthorhombic with a = 28.60, b = 19.98 and c = 11.38 A, but there are still some small peak position discrepancies, which cannot be attributed to the presence of impurities. Attempts to synthesize a purer sample for structure determination are in progress.

EMC-2: It was hoped that synchrotron data would facilitate the interpretation of the electron density within the cages of the EMC-2 zeolite in which the photosensitizer tris(4-methoxyphenyl)methylium had been encapsulated. Data analysis to date has been hindered by a puzzle that we have not yet been able to solve. The main electron density appears to lie within the medium-sized cage (not the more accessible large one as expected), but its shape and size do not conform to that expected for the cation.

Polypeptide: The high-resolution data for the polypeptide sa322 have allowed the unit cell to be confirmed (a = 11.19, ***b*** = 10.17, c = 61.01 A) and the most likely space group ($P222_1$) to be established. Unfortunately, the counting statistics are less than optimal although the data collection time was increased considerably. Nonetheless, structure solution using a structure envelope mask in combination with a Monte Carlo direct space approach is in progress.