X	**Experiment title:** Analysis of complex structures using high-resolution powder diffraction data	**Experiment number**: 01-01-716
Beamline: BM01B	**Date of experiment**: from: 11-May-2005 to: 15-May-2005	**Date of report**: 15-Sep-2006
Shifts: 12	**Local contact(s)**: Hermann Emerich	*Received at ESRF:*

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Report:

During this experiment, high-resolution powder diffraction data were collected on five samples. The structures of three of these were unknown (a zirconium phosphate, a scandium silicate, and the high-silica zeolite IM-5). The fourth was an as-synthesized sample of another high-silica zeolite (TNU-9), whose framework structure had been solved earlier this year by combining electron microscopy with powder diffraction data [1]. In this case, the location of the structure directing agent (SDA) within the framework was of interest. The fifth sample was a layer silicate (E-401), whose structure had been determined from data collected on a textured sample [2] using a new structure determination algorithm (charge flipping) [3,4]. In this case, high-resolution data on an untextured sample were required for structure refinement.

The pattern for the zirconium phosphate could be indexed on a hexagonal unit cell (a = 14.083 Å, c = 34.228 Å), but the space group is ambiguous. The data for the scandium silicate and IM-5 are presently being used in conjunction with high-resolution electron microscopy images for structure solution. The TNU-9 structure completion will be done in collaoration with P.A. Wright's group in St. Andrews, Scotland, who have performed some computer modelling of the location of the SDA.

The space group for E-401 was not clear from the diffraction pattern, and this is one of the reasons that the structure had proven to be so difficult to solve. However, the

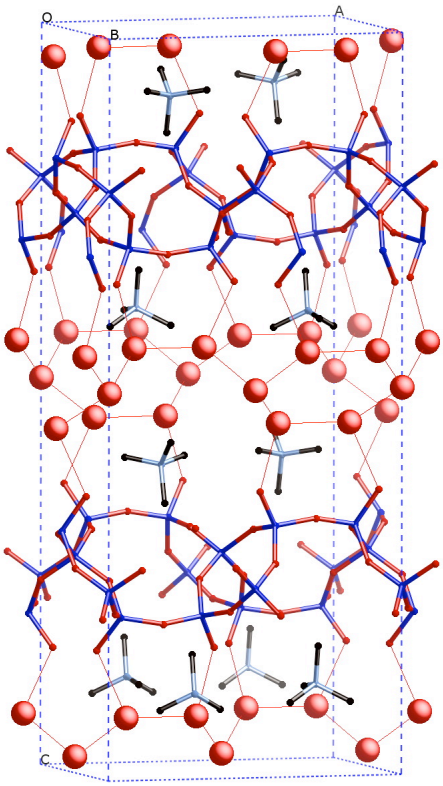

Figure 1. Structure of E-401.

combination of data from a textured sample and the charge-flipping algorithm, which works in *P*1 and is therefore less sensitive to symmetry assumptions, allowed the structure to be solved (Figure 1). The initial data were generated assuming the space group *Imma*, and these were expanded to *I*1 by the program.

The symmetry of the map turned out to be the non-centrosymmetric space group *I*2*mb*. Of the 23 non-H atoms in the asymmetric unit, 21 (including water molecule positions) could be resolved in the charge-flipping solution. Subsequent difference Fourier analysis revealed the positions of the last two C atoms, and then Rietveld refinement using the data collected on the untextured sample converged satisfactorily (R_F = 0.104, R_{wp} = 0.142, R_{exp} = 0.136, Figure 2).

The silicate layers of E-401 can be described in terms of half sodalite cages (cups) with alternating orientations. Between these layers are tetramethyl ammonium ions with one C-atom pointing towards the sodalite cup, and water molecules connect neighboring silicate layers via a H-bonding network.

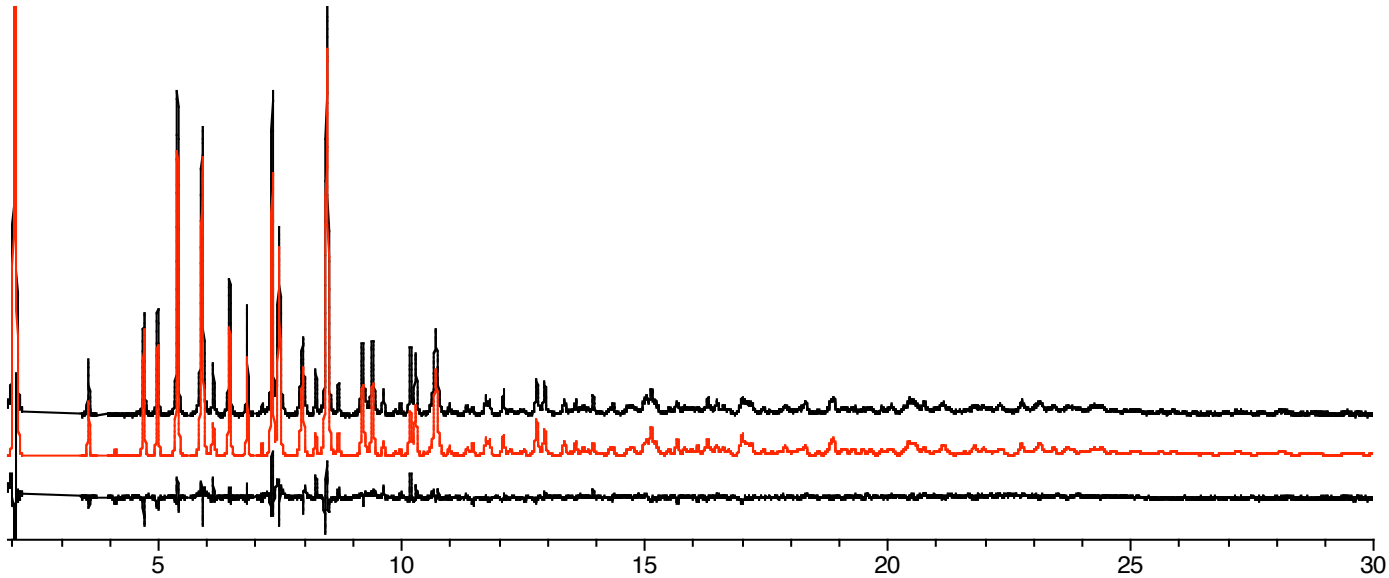

Figure 2. Observed (top), calculated (red) and difference profiles for the Rietveld refinement of E-401. The first peak has been cut off at ca 1/4 of its full intensity to show more detail. λ = 0.50067 Å.

References

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