SNBL	**Experiment title:** Analysis of complex structures using high-resolution powder diffraction data	**Experiment number**: 01-01-122
Beamline: BM01	**Date of experiment**: from: 25-Mar-98 to: 30-Mar-98	**Date of report**: 25-Sep-98
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Report:

High-resolution powder diffraction data were collected on five samples: three aluminophosphate molecular sieves, one zincophosphate and one high-silica zeolite. In all cases, a 1mm capillary sample and a pre-detector Si 111 analyzer crystal were used for the data collection. The zincophosphate is probably a layered material, but its structure has not yet been determined, and the data for the high-silica zeolite, SSZ-44, were collected for use in conjunction with those collected on a textured sample (experiment 01-01-122).

Two of the aluminophosphates were produced in a series of hydrothermal syntheses exploring the effect of incorporating organometallic complexes into the synthesis mixture. With $Ni(en)_3$, a mixture of two novel phases, one blue and the other white, was obtained. The two phases were separated by eye under the microscope, and then examined separately.

The white phase is monoclinic (a = 9.631, b = 9.440, c = 14.576 Å, □=98.22°), and appears to be a layered compound, but its structure has not yet been fully elucidated. The blue phase is triclinic (a = 9.0854, b = 9.3327, c = 8.9363 Å, □ = 86.27°, □=101.26°, □= 92.72°), and proved to have a framework structure closely related to that of chabasite, but with some linkages interrupted to accommodate a $Ni(en)_2$ complex in the cage (Figure 1).

Refinement of that structure is nearing completion (R_{wp} = 0.134, R_{exp} = 0.110, R_{exp} = 0.110, and R_F = 0.052). A portion of the powder pattern showing the high quality of the data and the current Rietveld fit is shown in Figure 2.

The other aluminophosphate examined was the pure $AlPO_4$ analog of the silicoaluminophosphate SAPO-40. ^{31}P MAS NMR spectroscopy indicated that the symmetry of this material must be lower than that of SAPO-40 (*Pccn*, a = 21.9443, b = 13.6911, c = 14.2486 Å) even

Figure 1. The $Ni(en)_2$ complex in the chabasite-like cage connected to the framework via 2 O-bridges.

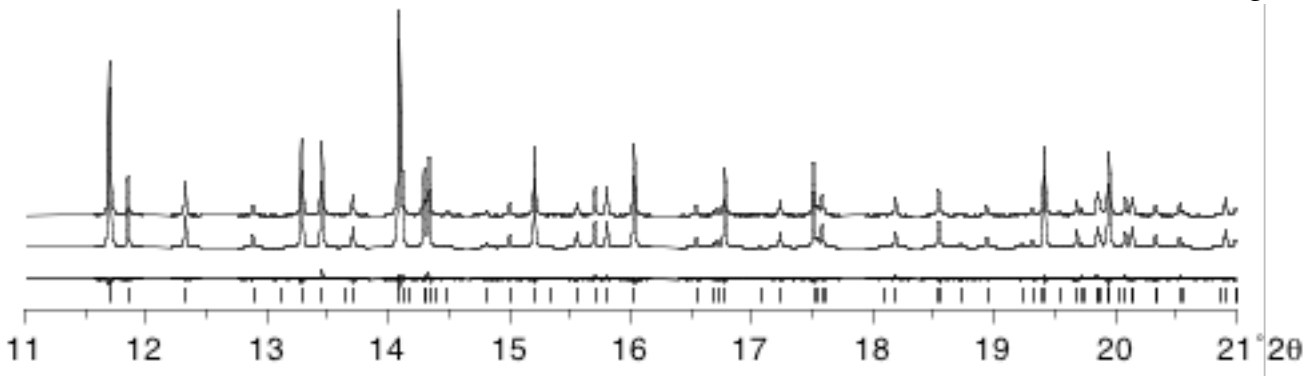

Figure 2. Observed (top), calculated (middle) and difference (bottom) profiles for a portion of the diffraction pattern of the blue $AlPO_4$ phase showing the high resolution of the data and the quality of the Rietveld fit.

though the unit cell dimensions are similar. Analysis of the diffraction data showed that the center of symmetry has been lost ($Pc2_1n$), with the consequence that there are 62 non-H atoms in the asymmetric unit (186 positional parameters). This is the largest structure yet refined using the Rietveld method, so high-resolution data were essential. The refinement was complicated by the fact that the lattice parameters changed slightly during the course of the data collection (a similar change was also found for SAPO-40), but the refinement has now converged with R_{wp} = 0.163, R_{exp} = 0.120, and R_F = 0.066. A portion of the Rietveld plot is shown below.

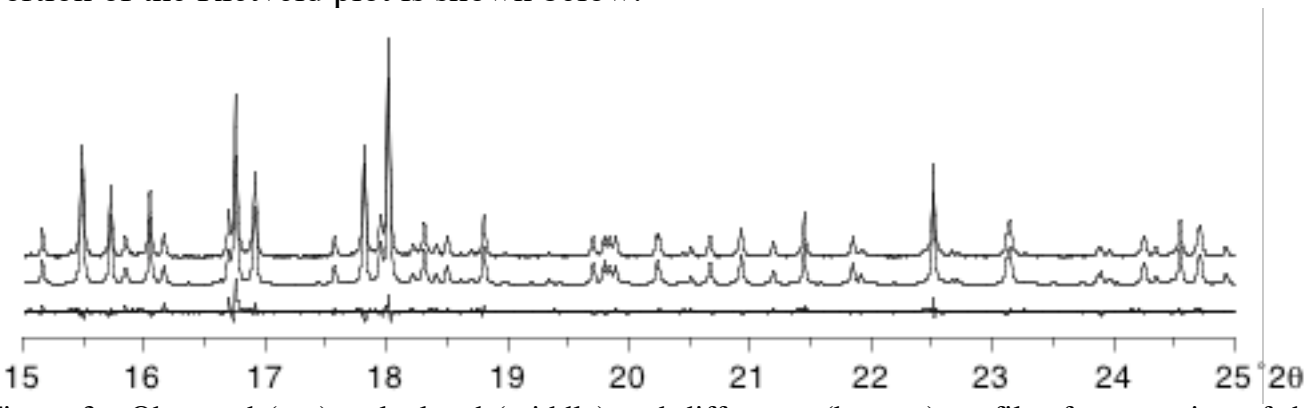

Figure 3. Observed (top), calculated (middle) and difference (bottom) profiles for a portion of the diffraction pattern of $AlPO_4$-40.