SNBL	**Experiment title:** Analysis of complex structures using high-resolution powder diffraction data	**Experiment number**: 01-01-265
Beamline: BM01B	**Date of experiment**: from: 3-Oct-2001 to: 4-Oct-2001	**Date of report**: 25-Oct-2002
Shifts: 6	**Local contact(s)**: Hermann Emerich	*Received at UNIL:*

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Report:

High-resolution powder diffraction data were collected on three different samples during this run. Two of these were microporous materials: an aluminophosphate AlPO-M (from Ruren Xu, Jilin University, China) and the calcined form of the aluminosilicate SUZ-4 (from Geoffrey Price, Univesity of Tulsa, USA). The third sample was an organic compound N-methyl-D-glucamine (from Procter & Gamble, USA).

We had tried to determine the structure of AlPO-M from laboratory data without success, and hoped that the higher resolution of synchrotron data would allow the structure to be solved. The synchrotron diffraction pattern could be indexed on an orthorhombic unit cell with a = 9.7461 Å, b = 29.1333 Å and c = 9.3511 Å, and the most probable space group appears to be $Pbca$. Indeed, the resolution of the data is significantly better than that of our laboratory instrument (see Figure 1), but we have still not been able to solve the structure despite considerable effort. Consequently, a textured sample of this material was prepared and texture measurements (MI-482) were performed. The software for analyzing these texture data is presently in the debugging stage, but as

soon as it is finished, structure solution will be attempted again. Should that work, we will then use the high-resolution data for the structure refinement.

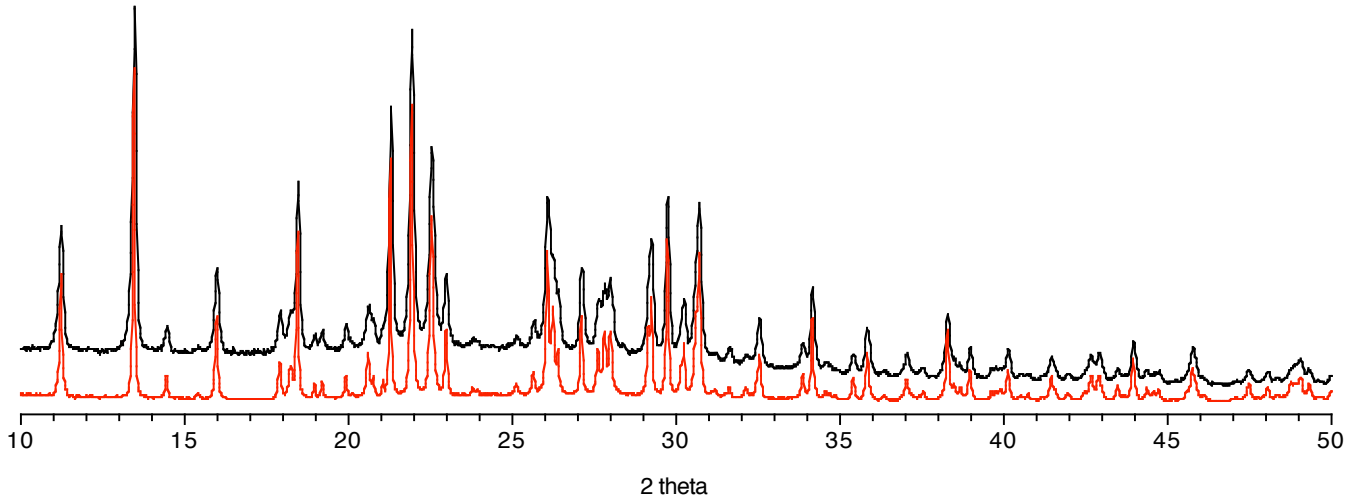

Figure 1. Comparison of the diffraction patterns collected on AlPO-M in the laboratory (black) and at SNBL (red). SNBL wavelength converted to CuKα_1 for the comparison.

The data on the calcined sample of SUZ-4 were collected to verify a framework structure proposal (Figure 2) derived from data collected on the as-synthesized form. Refinement of the structure using the original data (from the uncalcined material) was unsatisfactory, because the location of the non-framework species did not seem to make chemical sense. Therefore, the sample was calcined to eliminate any contribution from water molecules. The pattern can be indexed with an orthorhombic unit cell with a = 14.249 Å, b = 18.860 Å and c = 7.460 Å, but initial refinement in the space group *Pban* indicates that the symmetry must be lowered. Refinement in the three most likely subgroups, *Pba*2, *Pb*2*n* and *P*2*an*, is presently underway. The refinements are complicated by the presence of significant anisotropic line broadening.

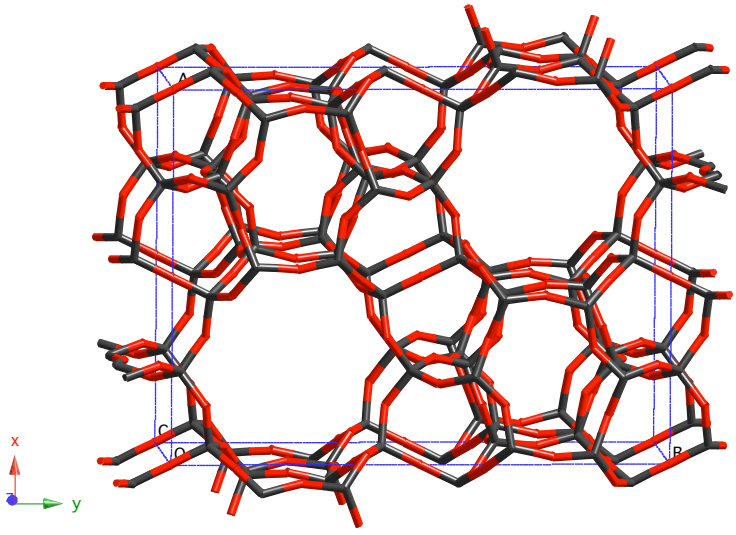

Figure 2. Framework structure proposed for the aluminosilicate SUZ-4

The structure of N-methyl-D-glucamine is known (solved from laboratory data), but the synchrotron data collected during this run will be used in conjunction with data collected on a textured sample for software development purposes.