SNBL	**Experiment title:** Analysis of complex structures using high-resolution powder diffraction data	**Experiment number**: 01-01-265
Beamline: BM01B	**Date of experiment**: from: 16-Dec-2002 to: 19-Dec-2002	**Date of report**: 31-Mar-2003
Shifts: 9	**Local contact(s)**: Hermann Emerich	*Received at UNIL:*

Names and affiliations of applicants (* indicates experimentalists):

Christian Bärlocher, Lab. für Kristallographie, ETHZ, Zürich
Lynne McCusker, Lab. für Kristallographie, ETHZ, Zürich
*Sinisa Prokic, Lab. für Kristallographie, ETHZ, Zürich
*Fabian Gramm, Lab. für Kristallographie, ETHZ, Zürich
*Jose Luis Jorda, Lab. für Kristallographie, ETHZ, Zürich

Report:

High-resolution powder diffraction data were collected on three microporous materials during this run: the aluminosilicate CAS-1 (from Chun Feng Xue, Taiyuan, China), the clathrasil RFS-105 (from A. Corma, Valencia, Spain), and the aluminophosphate IST-1 (from F. Ribeiro, Lisbon, Portugal). All have novel and unknown framework structures.

The CAS-1 data could be indexed on a monoclinic unit cell (a = 24.1677 Å, b = 7.0205 Å, c = 6.4858 Å, □ = 95.24°) and will be used in combination with data we have collected on a textured sample (experiment CH-1318).

Although the pattern of the clathrasil RFS-105 is of high quality, indexing has proven to be a problem. We are currently investigating the possibility that an unidentified impurity is present in the sample.

The data for the aluminophosphate IST-1 are of excellent quality. The pattern was indexed on an orthorhombic unit cell (a = 9.615 Å, b = 8.669 Å, c = 16.220 Å), and the systematic absences indicated that the most probable space groups were either *Pcam* (standard setting *Pbcm*) or $Pca2_1$. Although ^{27}Al MAS NMR data indicated the presence of 4-, 5- and 6-coordinate Al, it was hoped that the framework could still be regarded as a 4-connected net and that these Al species had simply incorporated additional ligands into

their coordination spheres. This would mean that the program *focus*, which was written specifically for determining zeolite (3-dimensional 4-connected) framework structures from powder diffraction data [1,2], could be applied. Reflection intensities were extracted from the pattern and structure solution was attempted in both space groups. A framework structure consistent with alternating Al and P atoms was found in the non-centrosymmetric space group $Pca2_1$. Subsequent difference Fourier maps allowed an oxygen species bridging between two Al of the framework and two crystallographically distinct methylamine species to be found. One of the latter is coordinated directly to an Al atom while the other is in the channels of the framework. The final structure (Figure 1) is consistent not only with the ^{27}Al MAS NMR results (3 Al sites with 3 different coordination numbers), but also with ^{31}P (3 P sites) and ^{13}C (2 different methylamine species) data. Rietveld refinement of this model converged with the error indices R_F = 0.042 and R_{wp} = 0.125 (R_{exp} = 0.043) (Figure 2).

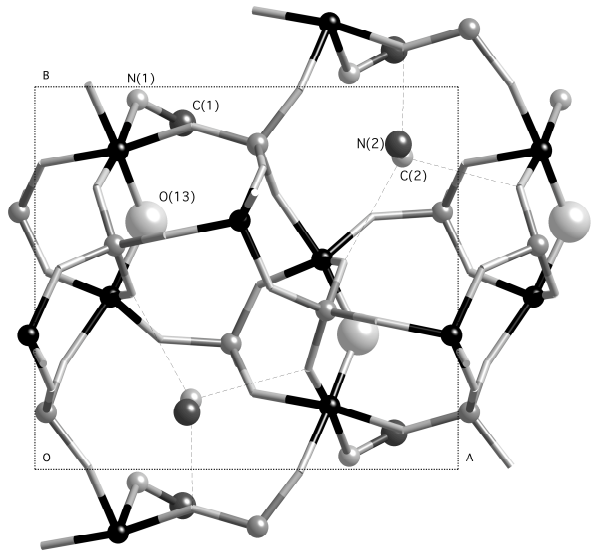

Figure 1. The structure of IST-1.

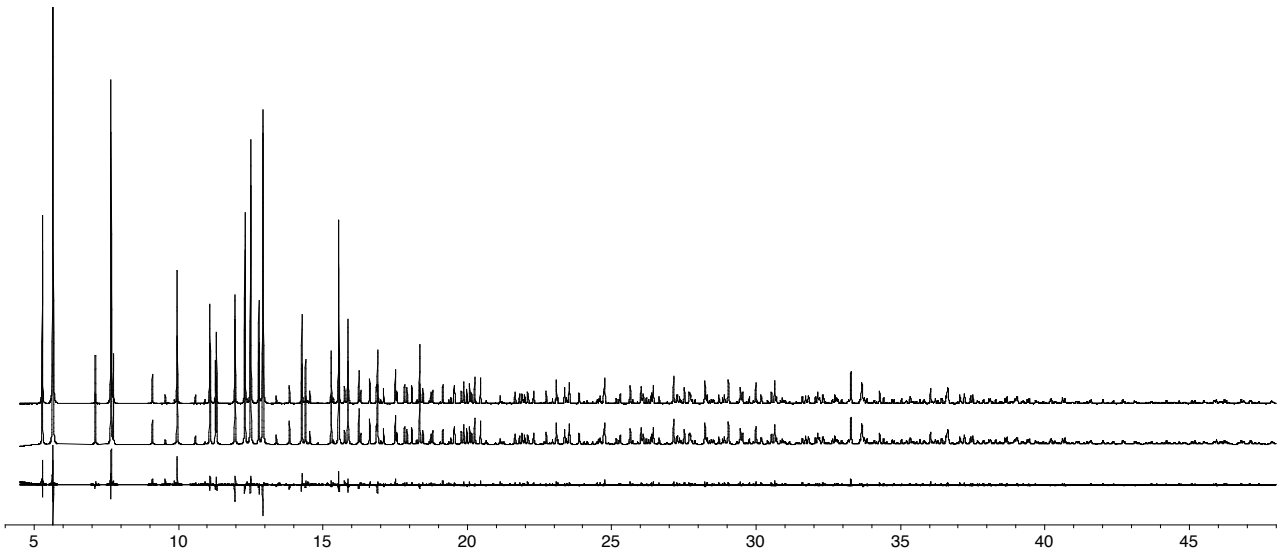

Figure 2. The observed (top), calculated (middle) and difference (bottom) profiles for the Rietveld refinement of the as synthesized form of IST-1.

[1] Grosse-Kunstleve, R.W., McCusker, L.B. and Baerlocher, Ch., *J. Appl. Crystallogr.*, **30**, 985-995 (1997)
[2] Grosse-Kunstleve, R.W., McCusker, L.B. and Baerlocher, Ch., *J. Appl. Crystallogr.*, **32**, 536-542 (1999)