SNBL	**Experiment title:** Analysis of complex structures using high-resolution powder diffraction data	**Experiment number**: 01-01-830
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Report:

High-resolution powder diffraction datasets were collected on four samples during this experimental session: the zeolites SSZ-52 [1] and SSZ-82 [2] and two metal-organic framework (MOF) materials. The latter are Ag^+-ion-exchanged versions of $NaLa(PO_3H)_2CH$-C_6H_4-$CH(PO_3H)_2{\cdot}4H_2O$ ($NaLa(H_4L)$), which exhibits an exceptional selectivity for monovalent cations [3]. These two modified MOF's appear to be significantly different from their Na parent and exhibit unexpected I_2 adsorption properties. One of the patterns has been indexed ($Ag_3La(H_2L)$: a = 8.810 Å, b = 22.269 Å, c = 10.059 Å, β = 97.2°), but the second $AgNa_{0.4}La(H_{3.6}L)$ is proving to be difficult. A second phase may be present in that sample. SSZ-52 is probably a member of the ABC-6 family of zeolite framework structures, but those data have not yet been analyzed.

The structure of calcined SSZ-82, however, has been solved and refined. Its pattern could be indexed in the space group *Pmmn* with a = 24.293 Å, b = 11.481 Å and c = 14.119 Å. Unfortunately, the electron density maps obtained from a simple application of the powder charge flipping algorithm [4] in the program *Superflip* [5] to the extracted reflection intensities could not be interpreted satisfactorily, so a newly developed approach that we have dubbed 2D-XPD [6] was applied.

This approach involves (1) phasing low-resolution (3 Å) 2-dimensional subsets (e.g. [001], [010] and [100] projections) of the full 3-dimensional data using *Superflip*, (2) extending that initial phasing to 1.5 Å resolution, and then (3) imposing those phases in the first 50 cycles of *Superflip* using the full 3-dimensional dataset. In this way, interpretable electron density maps showing the framework structure were obtained (Figure 1).

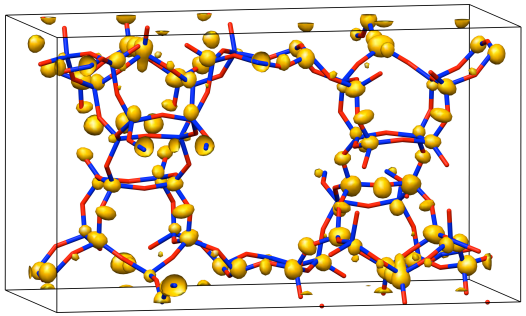

Figure 1. Electron density map for SSZ-82 obtained using the 2D-XPD approach. The final refined framework structure is overlaid for comparison.

Rietveld refinement of the structure allowed the positions of the B atoms in the framework to be identified and converged with R_F = 0.041 and R_{wp} = 0.140 (R_{exp} = 0.129) (Figure 2). Some water molecules, presumably adsorbed after the calcination of the material, were apparent in the channels. The zeolite has 11 Si/B in the asymmetric unit and a 10-/12-ring 2-dimensional channel system.

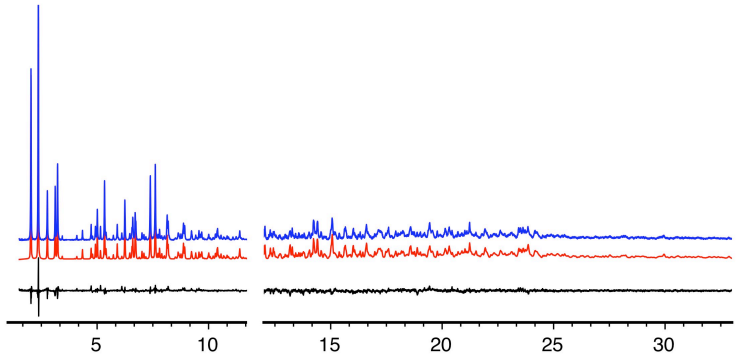

Figure 2. Observed (blue), calculated (red) and difference (black) profiles for SSZ-82. The second part of the pattern has been scaled up by a factor of 5 to show more detail.

1 G.S. Lee and S.I. Zones, US Patent 6379531, 2002.
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4 Ch. Baerlocher, L.B. McCusker and L. Palatinus, *Z. Kristallogr.* **222**, 47-53 (2007).
5 L. Palatinus and G. Chapuis, *J. Appl. Crystallogr.* **40**, 786-790 (2007).
6 D. Xie, *Ph.D. Thesis*, ETH Zurich, 2011. D. Xie, Ch. Baerlocher and L.B. McCusker, *J. Appl. Crystallogr.*, submitted.