SNBL	**Experiment title:** Using texture to unravel the relative intensities of overlapping reflections in a zeolite powder diffraction pattern	**Experiment number:** 01-01-146
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Report:

Samples of three polycrystalline materials of unknown structure, UTD-1F, IM-5 and $HAlF_4$, were examined during this experiment. Because the samples were of different thicknesses (and therefore had different absorption properties), intensity calibration curves were measured for two samples of untextured zeolite A with the appropriate dimensions. For each of the high-silica zeolites, UTD-1F and IM-5, 7 pole figures were measured to establish the texture, and then full diffraction patterns were collected at five different sample orientations (chosen to give maximum contrast). For $HAlF_4$, 6 pole figures and 4 full diffraction patterns were measured.

The texture of UTD-1F could be established from the pole figure data, and a set of near-single-crystal reflection intensities could be extracted from the five diffraction patterns (see Figure 1). The structure was determined from these intensities in the space group $P2_1/c$ (a=14.9633 Å, b=8.4704 Å, c=30.0098 Å, β=102.7°) using direct methods. All 16 Si atoms and 17 of the 32 O atoms were found on the initial E-map. Difference

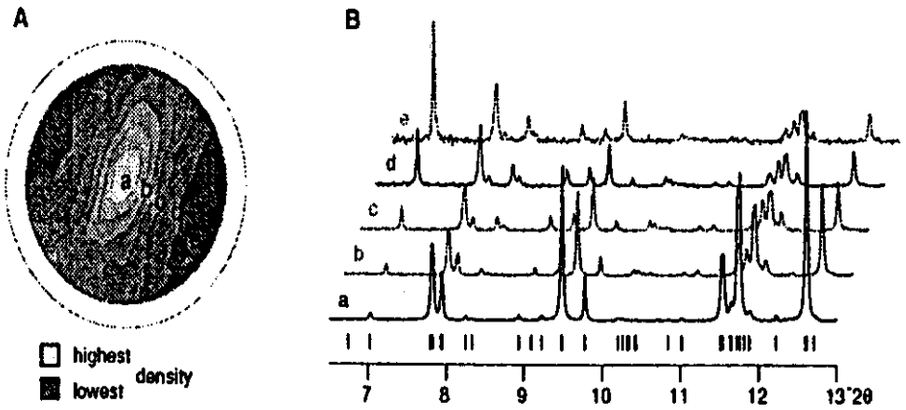

Figure 1. (A) Pole figure data for the $10\bar{2}$ reflection and (B) small sections of the five diffraction patterns collected at the sample orientations indicated in the pole figure (*Science* (1999) 284, 477-479).

Fourier maps then revealed the positions of the rest of the framework O atoms and those of the $Co(Cp^*)_2$ complex in the 14-ring channel. Subsequent Rietveld refinement showed the true space group to be non-centrosymmetric (*Pc*) and refinement of the 349 positional parameters converged satisfactorily with $R_{wp} = 0.134$ and $R_I = 0.041$ (see Figure 2). The final structure, with 117 atoms in the asymmetric unit, displays no evidence of faulting and has a fully ordered arrangement of the Co complex.

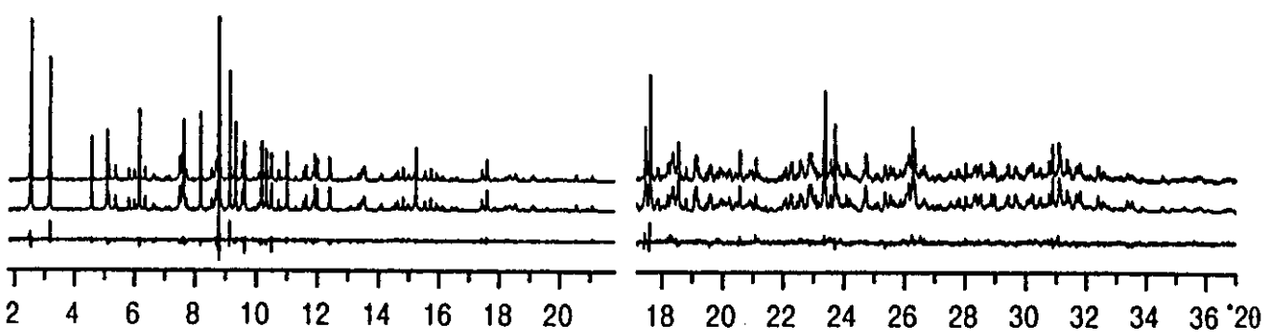

Figure 2. Observed (top), calculated (middle) and difference (bottom) profiles ($\lambda = 0.69796$ Å) for UTD-1F. To show more detail, the highest peak has been cut at ca 60% of its full height, and the scale for the second 2θ region has been increased by a factor of 5 (*J. Am. Chem. Soc.* (1999) 121, 6242-6247).

The data from the IM-5 sample allowed the impurity peaks in the diffraction pattern to be identified unambiguously, and this finally made a correct indexing of the peaks from the sample possible (orthorhombic, $a = 14.277$ Å, $b = 57.369$ Å, $c = 20.107$ Å). Although the texture could be well described, the size of the unit cell (16'000 Å^3) requires that more full diffraction patterns be collected before a reliable deconvolution can be obtained.

For the $HAlF_4$ sample, the texture could also be described and a set of reflection intensities were extracted. A partial structure could be obtained, but the chemical analysis appears to be incorrect, so further analysis of the diffraction data has been temporarily suspended.