ESRF	**Experiment title:** Zeolite structure analysis using high resolution powder diffraction data	**Experiment number:** HC-324
Beamline: BM16	**Date of experiment:** from: 2-Oct-96 to: 9-Oct-96	**Date of report:** 7-Feb-97
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Report:

High resolution powder diffraction data were collected on four aluminophosphate molecular sieves: the dehydrated form of the very large pore molecular sieve VPI-5, the partially rehydrated forms of $AlPO_4$-18 and $CoAlPO_4$-CHA, and a molecular sieve with an unknown framework structure.

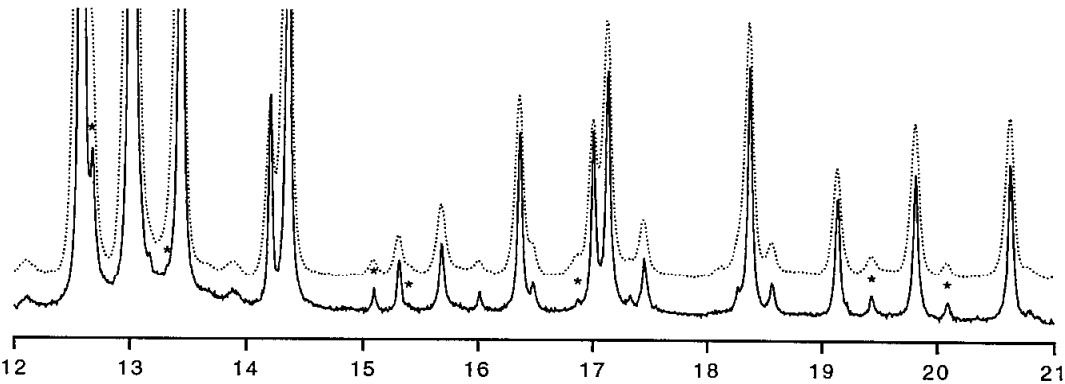

Figure 1. Comparison between laboratory (dotted line, converted to λ=0.92032 Å) and ESRF (solid line) data for dehydrated VPI-5. Asterisks indicate peaks that cannot be indexed on a hexagonal unit cell.

The high quality of the data for dehydrated VPI-5 (both resolution and statistics) has allowed small deviations from hexagonal symmetry to be seen clearly and has made Rietveld refinement of the structure in the monoclinic space group ***Cm*** possible (R, = 0.062, R_{wp} = 0.098, R_{exp} = 0.047). A comparison between laboratory data and ESRF data for a small section of this pattern is shown in Figure 1. The asterisks indicate peaks that require the lower symmetry. A section of the observed, calculated and difference profiles for the refinement (166 positional parameters) are shown in Figure 2.

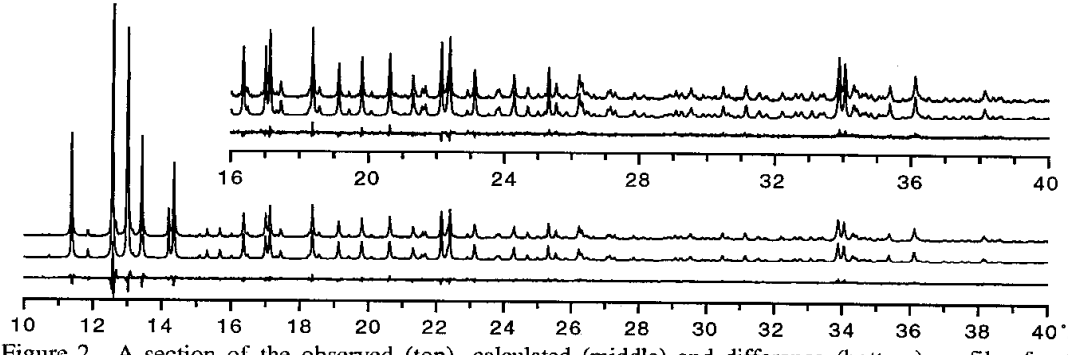

Figure 2. A section of the observed (top), calculated (middle) and difference (bottom) profiles for the Rietveld refinement of the structure of dehydrated VPI-5. The inset has been scaled up by a factor of 2.5.

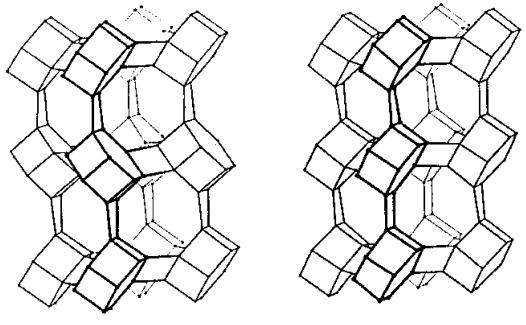
Figure **3. AEI** (left) and CHA (right)

The framework structures of $AlPO_4$-18 and $AlPO_4$-CHA are closely related (Figure 3), and both materials show interesting (and not fully understood) symmetry changes upon rehydration. In both cases, the unit cells become triclinic, so high-resolution data is essential for a successful structure refinement. The key appears to lie in the identification of the framework Al atoms that expand their coordination spheres to include one or two of the resorbed water molecules. This produces a significant distortion of the double 6-rings and results in a destruction of the symmetry. Initial refinement attempts look promising, but the presence of strong pseudo-symmetry makes this a slow process. Refinement of the $AlPO_4$-18 structure is further hampered by the presence of a small amount of an unidentified impurity.

Attempts to solve the structure of the fourth aluminophosphate molecular sieve have not yet been successful. Even with the high resolution of the synchrotron data, the choice of unit cell and space group remains ambiguous, so all possibilities need to be explored.