ESRF	**Experiment title:** Single Crystal Diffraction Data for the aluminophosphate VPI-5	**Experiment number:** CH-22
Beamline: IDH : BL2	**Date of Experiment:** from: 13.10.94. to: 16.10.94	**Date of Report:** 31.7.95.
Shifts: 12	**Local contact(s):** Dr A Kvick	*Received at ESRF:*

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Report:

The aluminophosphate VPI5, $AL_3(PO_4)_3 .nH_2O$, is a large pore molecular seive compound. Its structure was established in detail by McCusker, Baerlocher, Jahn and Bulow, (1991, Zeolites, 11, 308-313) from high quality synchrotrons radiation powder diffraction data. It is hexagonal, with a= 18.96, c= 8.11 A. The structure refinement was successful because they postulated a structure in space group P63, not in P63cm which had been assigned to earlier models. In 1993 A. Dunlop at Edinburgh University Chemistry Department prepared some crystals of VPI-5 which, though small, *ca* 30x5x5 µm, appeared under the microscope to be of much higher quality than any we had previously seen, clear with good faces. The objective of this experiment was to obtain diffraction data from these crystals to confirm the P63 structure and refine the structural parameters further.

Five crystals of VPI-5 from the preparation were examined on the beamline, using Fuji image plates to record diffraction patterns and a Molecular Dynamics Image Plate Scanner. Only one crystal showed diffraction spots, and these were very small and sharp, and nearly lost in the heavy background. Data collection was attempted for this

crystal, with crystal-detector distance 150 mm, wavelength 0.60 A, beam monitor current between 0.17 and 0.10 mA. 20 oscillation ranges were used in order to minimise the background; the backstop was moved close to the crystal, *ca* 10 mm, again to reduce background. 22 exposures were recorded (300 s, 5 oscillations for the first fifteen, 450 s, 8 oscillations for the others), covering 440 of rotation about the spindle. The needle axis was *ca* 150 from the spindle direction. A similar data set was then recorded at 145 K, on the same crystal, using the Oxford Cryostream cooler.

Although some small sharp diffraction spots could be seen on each image at the time of the ESRF visit, it was not until later that they could be properly assessed after applying a good background subtraction routine. 30-50 spots were present on each image, out to a resolution of *ca* 1 A, but they could not be indexed as a single crystal pattern. It was eventually concluded that the sample represented 2-5 separate crystals, and the diffraction patterns could not yield any new structural information.

The experiment was therefore not successful in its primary purpose. However i) a single crystal diffraction pattern for crystals of this composition and substantially smaller than 30x5x5 μm has been seen, ii) methods of reducing background scattering sufficiently to record such patterns have been explored, and iii) a few images were recorded for each of several larger, but still small crystals ($AlPO_4$-14, 30X10X10 μm, hydroxyzincite 60x60x5 μm, rosasite 20X20X10 μm) and could certainly have been processed if data collection on these compounds had been the objective.