	Experiment title: Analysis of complex structures using high-resolution powder diffraction data	**Experiment number**: 01-01-639
Beamline: BM01B	**Date of experiment**: from: 28-Apr-2004 to: 30-Apr-2004	**Date of report**: 23-Aug-2004
Shifts: 6	**Local contact(s)**: Denis Testemale	*Received at ESRF:*

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Report:

High-resolution powder diffraction data were collected on three samples during this run: silver behenate, the layered aluminophosphate Phase G (from C. Marichal, Université de Haute Alsace, Mulhouse, France), and the high-silica zeolite ZSM-5.

Silver behenate is used in photographic films, and its structure is of considerable interest to the film industry. However, the indexing of the diffraction pattern (Figure 1) has

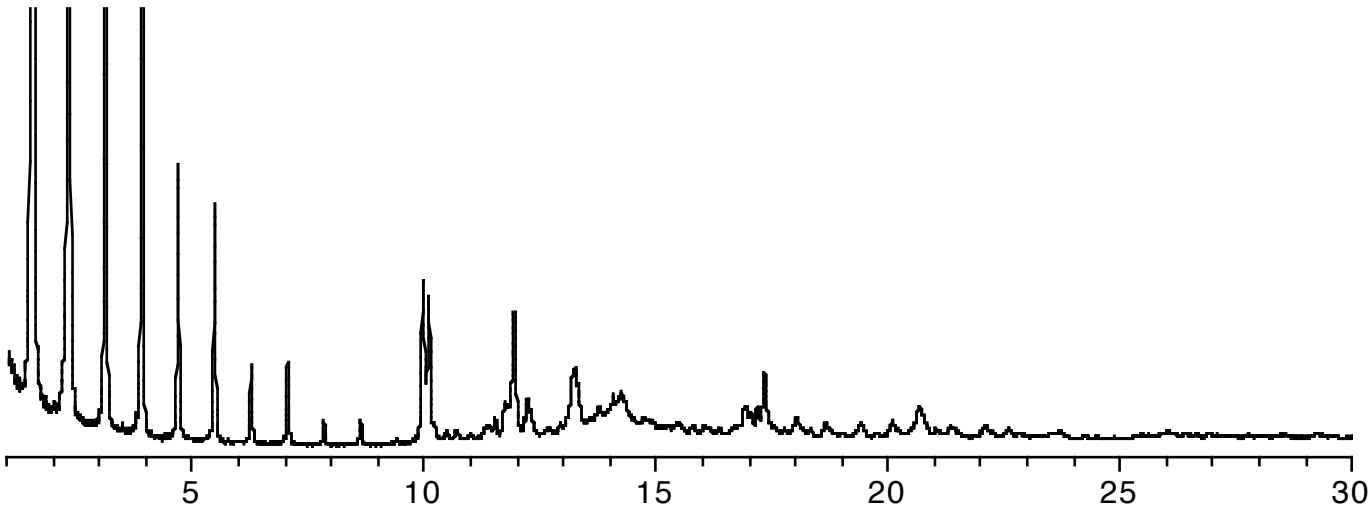

Figure 1. Diffraction pattern of silver behenate ($\lambda = 0.79948$ Å). The intensity scale has been increased by a factor of 10 to show more detail at higher angles.

proven to be surprisingly difficult. This is primarily due to the fact that the first 11 lines of the diffraction pattern are all along the same direction (i.e. higher orders of the first). The repeat distance in this direction is ca 60Å, but it has been very difficult to establish the other two lattice parameters. It was hoped that by combining very high-resolution data collected on a new sample with data collected on a textured preparation of the same sample, that the unit cell could be established. The data on the textured sample will be collected in December (Experiment 01-02-689).

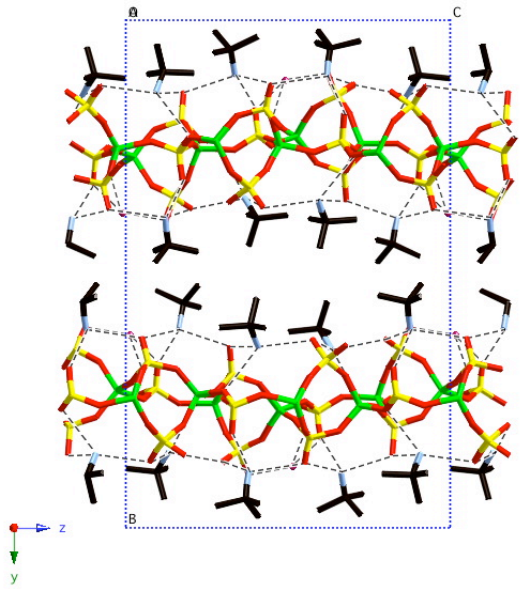

Figure 2. Layered $AlPO_4$ structure of Phase G showing the H-bonded *t*-$BuNH_3^+$ ions.

Single crystal data collected on a microcrystal of the aluminophosphate Phase G ($P2_1/c$; a = 9.8788 Å, b = 26.1522 Å, c = 16.7060 Å, β = 90.408°) were of sufficient quality to allow the approximate aluminophosphate layered structure to be determined (Figure 2), but a reliable refinement with these data proved to be unsatisfactory. Consequently, high-resolution powder diffraction data were collected for this purpose. Fourier analysis revealed the positions of the three crystallographically distinct *t*-butyl ammonium ions, and Rietveld refinement (Figure 3) of the 78 parameter structure then proceeded smoothly, converging with the *R*-values $R_F = 0.079$, $R_{wp} = 0.152$ ($R_{exp} = 0.052$).

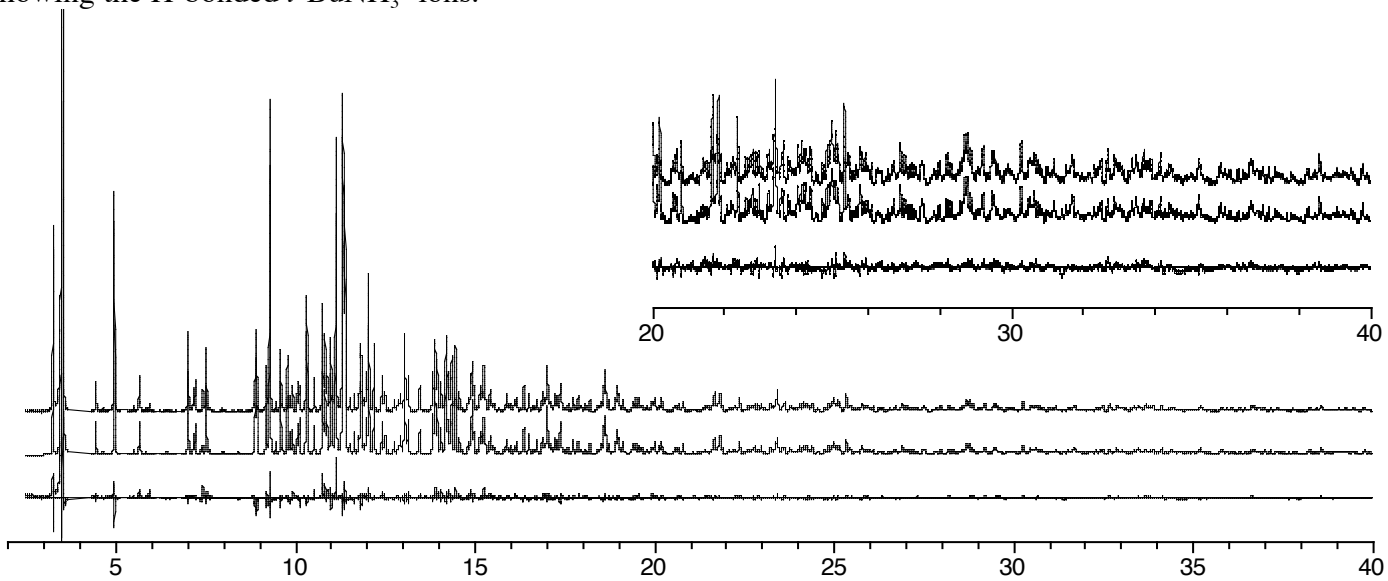

Figure 3. Observed (top), calculated (middle) and difference (bottom) profiles for the Rietveld refinement of Phase G. The scale of the second half of the pattern has been scaled up by a factor of 5 to show more detail.

The high-resolution data on the complex zeolite ZSM-5 (*Pnma*; a = 20.022 Å, b = 19.899 Å, c = 13.383 Å) are being used in conjunction with an electron crystallography project. It is hoped that by combining information from powder and electron diffraction a more single-crystal-like set of intensities can be obtained.