



	<b>Experiment title:</b> Intergrowth and stacking faults in microporous catalysts	<b>Experiment number:</b> 01-01- 760
<b>Beamline:</b> BM01B	<b>Date of experiment:</b> from: 7/5/08 to: 12/5/08	<b>Date of report:</b> 22/1/09
<b>Shifts:</b> 9	<b>Local contact(s):</b> Wouter Van Beek	<i>Received at ESRF:</i>

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### **Report:**

The structure of methanol adsorbed in SAPO-34 has been determined from high resolution XRD data collected at station BM01B of the SNBL. Powder XRD data were collected at roomtemperature and 100K on a sample of SAPO.-34 which had been treated with methanol. The structure was determined by starting a Rietveld refinement with the SAPO-34 framework model and calculating difference Fourier maps once a good fit had been achieved. From these maps carbon and oxygen positions were obtained and gradually added to the refinement with occupancy factors for each site being determined. Restraints were used on the C-O bond lengths and a reasonable refinement was obtained ( $R_{wp} = 0.0671$ ,  $RF^2 = 0.0694$ ,  $\chi^2 = 3.183$ )- however it was not possible to remove the restraints and so we decided to replace the freely refined carbon and oxygen atoms with methanol rigid bodies. The rigid bodies were initially placed in the positions indicated by the previous refinement and allowed to rotate about their centres of gravity. A final refinement (against the 100K dataset) was obtained which converged with a small amount of Marquadt damping (damping factor 1.48) to  $R_{wp} = 0.0690$ ,  $RF^2 = 0.0722$ ,  $\chi^2 = 3.329$ . This fit is statistically slightly worse than the original refinement but we feel that it represents a more meaningful model. Visually the fits are indistinguishable (figure 1).

The Formula of SAPO-34 with adsorbed methanol is calculated as  $(AlP_{0.95}Si_{0.05}O_4)_2 \cdot (CH_3OH)_{1.75}$ , and the structure is shown in figure 2.

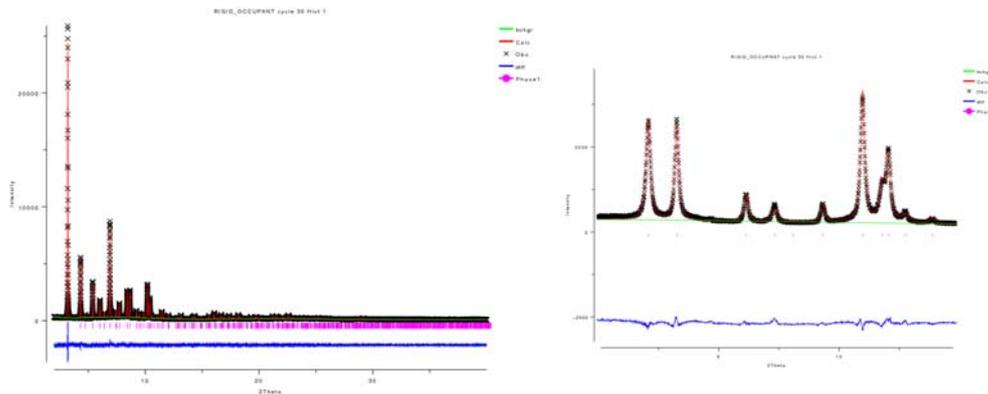


Figure 1. Plots of the Rietveld refinement of SAPO-34 with adsorbed methanol.

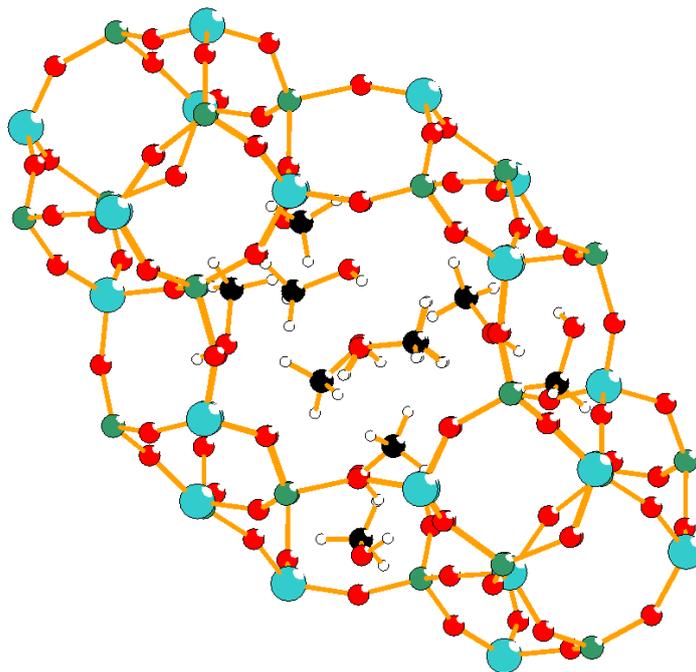


Figure 2. The structure of SAPO-34 with adsorbed methanol. Phosphorus/silicon = green spheres; aluminium = blue; oxygen = red; carbon= black; hydrogen = white.