



	Experiment title: In Situ Single Crystal Studies of SAPO-34 in the Methanol to Olefin Process	Experiment number: CH-2922
Beamline: ID11	Date of experiment: from: 17/9/09 to: 21/9/09	Date of report: 12/08/10
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Report:

The experiment had two key goals. Firstly to establish the principle that a catalytic process such as methanol to olefin (MTO) conversion over SAPO-34 could be observed in situ using fast single crystal diffraction; and secondly to gain detailed structural information about the species inside the cages of SAPO-34 during/after the reaction. We have been successful in the first but the second currently remains unresolved.

80 datasets were collected during the experiment. Some of these are rather hard to analyse because of twinning although unit cell data have been obtained for all datasets. In situ single crystal datasets were collected with a time resolution of 27 minutes for a complete dataset during activation of the catalyst and the MTO reaction. After the reaction data were collected more slowly in order to give a better chance of resolving the structure of the species inside the pores.

Data were collected during the MTO process which show that the trends in unit cell size observed by powder XRD are also observed in single crystals (figure 1).

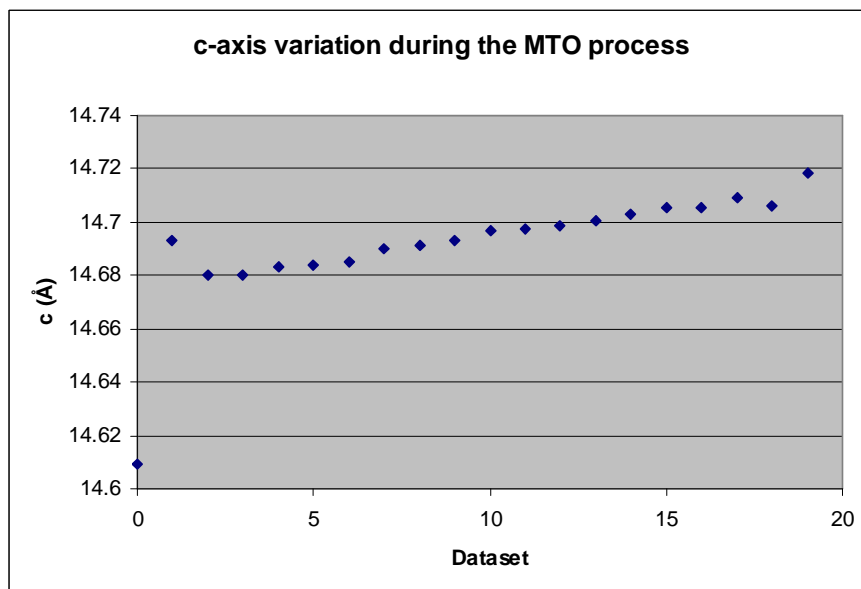


Figure 1 c-axis variation for a single crystal of SAPO-34 during the MTO process.

The variation observed in the unit cell of single crystals is smaller than for powder samples, however this is to be expected as the reactants cannot penetrate such a large percentage of the particle in a large single crystal. This results in a smaller average expansion and contributes to the difficulties in achieving our second goal.

We observe a splitting of the diffraction peaks in our data during the MTO experiment. This was initially attributed to crystal breakage caused by the coke from the reaction, however, recent high resolution synchrotron powder XRD results suggest the intriguing possibility that we are in fact seeing two or more unit cells of different sizes distinguished by different coke types. Processing of the data to account for this effect is ongoing.