ESRF	**Experiment title:** Ultra Small Angle Scattering on precursors of zeolites and molecular sieves and clays	**Experiment number:** SC 138
Beamline: ID2/BL4	**Date of experiment:** from: 12-04-1996 to: 14-04-1996	**Date of report:** 15-08-1996
Shifts: 9	**Local contact(s):** Dr. O. Diat, Dr. P. Bösecke	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

* P.P.E.A. de Moor, Eindhoven University of Technology
* T.P.M. Beelen, Eindhoven University of Technology
* K. Jacobs, Katholieke Universiteit Leuven

Report:

During experiment SC 138 the crystallization of zeolites, molecular sieves and clay was investigated *in situ* at station ID2/BL4 (P. Bösecke, O. Diat), using a Bonse-Hart camera and a special rotating and controlled-heating synthesis cell. The scanned range (d = 2000-20 run) showed a satisfactory overlap with the SAXS-range (d = 50-2.5 run) measured in Daresbury. Experiments have been performed on two types of molecular sieves/zeolites:

A. Silicalite-1

In situ syntheses of silicalite-1 have been performed from a clear solution for three Si/OH ratios. The USAXS spectra showed good agreement with SAXS results [1] in the overlapping range and confirmed the influence of the Si/OH ratio on the formation of precursor gel particles in the crystallization of silicalite-1 from a clear solution. With USAXS we could now prove that crystals were formed with a fractal surface (D_s = 2.3-2.4) and sizes up to 550 nn [2,3].

B. Zeolite L

The influence of aging at room temperature of the synthesis mixture on the (trans)-formations of the gel phase was investigated. The spectra showed that fractal aggregates with a size up to 3 micrometers and a mass-fractal dimension of 2.0 were formed in the fresh synthesis mixture. The size of the primary particles was about 9 nm. After heating to reaction temperature (160°C) a densification of the aggregates was observed (for preliminary publication see [4]). This densification may be identified with the formation of crystals.

Preliminary USAXS experiments on the *in situ* preparation of clay, showed for Li-hectorite a slope of approximately -2 in the log I vs. log Q plot, corresponding with the formation of sheets or large disks. The growth of these sheets and the intensity of the USAXS signal may be representative for the growth of Li-hectorite.

[1] P.P.E.A. de Moor, T.P.M. Beelen, R.A. van Santen, submitted to Microporous Materials, 1996.

[2] P.P.E.A. de Moor, T.P.M. Beelen, R.A. van Santen, submitted to Journal of Applied Crystallography, edition proceeding “X International Conference on Small Angle Scattering”, 1996.

[3] P.P.E.A. de Moor, T.P.M. Beelen, O. Diat, P. Bösecke, R.A. van Santen, in preparation.

[4] O. Diat, P. Bösecke, J. Lambard, P.P.E.A. de Moor, submitted to Journal of Applied Crystallography, edition proceeding “X International Conference on Small Angle Scattering”, 1996.