



	Experiment title: Assessment of applicability of Multivariate Curve Resolution method to recover EXAFS spectra and obtain information about size of nanoparticles	Experiment number: 01-01-973
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This experiment was performed very recently, thus it is a preliminary report. The data analysis is still under progress and will be replaced with the final report in the near future.

Report:

We have successfully used complementary methods (EXAFS, PXRD and PDF) to study the formation of nanoparticles in solution. We focused on ZrO₂ synthesis as a model system to show the applicability of MCR-ALS methodology to recover EXAFS spectra in order to determine the size of newly forming nanoparticles. Fig.1 shows selected EXAFS spectra recorded in situ during reaction at 220°. We choose this system because the oxidation state of Zr does not change during reaction. What we observed instead is growth and growth driven crystal structure transformation (see Figure 2 of reference compounds). The information gained from the scattering methods (PXRD and PDF) were used to verify our hypothesis. We investigated the reactions of zirconium alkoxide in non-aqueous solvent at five temperatures between 160 and 220°C.

Moreover, we have successfully obtained preliminary results on the zinc ferrites synthesis at different reaction conditions (140-180°C) by means of XAS (XANES, EXAFS) and PXRD. The reliable PDF data could not be obtained due to the low concentration of the sample in solution, high background due to PEEK reaction container, low flux and lack of the cooling for the PDF detector.

The obtained results are highly interesting and will significantly add to the knowledge about the mechanism of crystallization in non-aqueous solution.