ESRF	**Experiment title:** Time-resolved in situ XAS/FTIR/MS mechanistic study on low-temperature oxidation of CH_4 CO and NO over ceria supported Pt catalysts	**Experiment number**: CH2975
Beamline: ID24	**Date of experiment**: from: 100121 to: 100126	**Date of report**: 100825
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Report:

We have carried out time-resolved *in situ* dispersive XAS measurements of supported Pt catalysts operating at transient conditions. Both alumina and ceria supported catalysts were studied in this session. It turned out that the technically relevant ceria used in our sample preparation resulted in catalysts that are very difficult to study with XAS in the energy range relevant for Pt L_{III} XANES. Due to the difficulties connected to the ceria and some other obstacles related to the beam line set-up, we changed direction to study instead alumina supported platinum catalyst. On this sample we made a number of experiments that complement earlier experiments (SI-1799) for which one scientific paper recently was published in *J. Phys. Chem. C*. Also, due to temporary problems with the FTIR, we focused on using XAS/MS in this session.

The overall goal with the complementary experiments was to understand the connection between the chemical state of the supported Pt crystallites and the activity for methane oxidation. Also the influence of hydrogen and carbon monoxide, products that may form under partial methane oxidation conditions, on the Pt L_{III} XANES spectra for our sample was studied. Various temperature programmed experiments and step- and pulse response experiments were carried out. At the time of writing this report, we are working on the detailed evaluation of the Pt L_{III} XANES and mass spectrometry data. For evaluation of the XANES data we use a method that is based on analysis of the area under the white-line (WLA). This method has previously been developed for qualitative analysis by our group. The WLA has been found sensitive also to small changes in the absorption spectrum and thus suitable for studies of the mentioned catalytic reactions. During this session we aimed at improving the WLA method so that quantitative results can be obtained. The results will be presented in scientific papers during the fall 2010/2011. Also the reults will be combined with results from SI-1799 and possible also complementary FTIR/MS exeriments that will be carried out at Chalmers during 2010/2011.

The included figure shows the Pt L_{III} XANES data from a temperature programmed methane oxidation experiment in oxygen excess over Pt/Al_2O_3. The sample is rather rapidly oxidised at a certain temperature and then futher oxidised as temperture is increased. This platinum oxidation can be connected to changes in activity (not shown). The type of experiment complement previous studies (SI-1799, *J. Phys. Chem. C 2010*) where it was concluded that the surface O:Pt ratio is crucial for the methane oxidation activity.

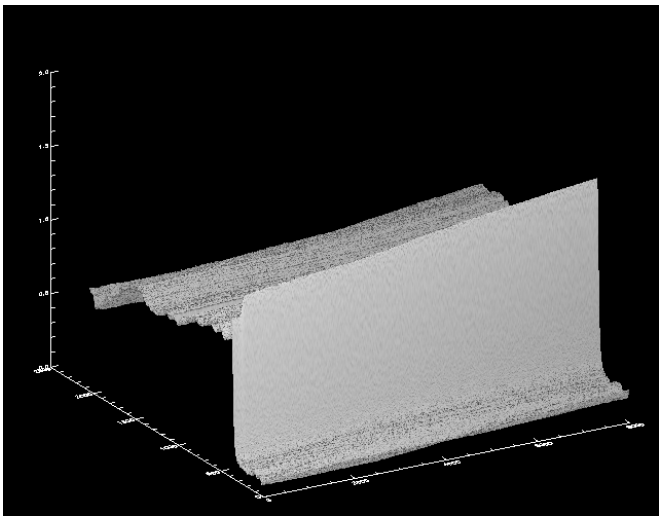

Figure 1. Three-dimensional representation of Pt L_{III} XANES spectra during temperature programmed oxidation of 1000 vol.-ppm CH_4 in oxygen excess over Pt/Al_2O_3 catalyst.