



	<b>Experiment title:</b> INVESTIGATION ON THE OXIDATION STATE AND LOCAL ENVIRONMENT OF METAL CATIONS IN TRANSITION METAL MIXED OXIDES CATALYSTS	<b>Experiment number:</b> CH1816
<b>Beamline:</b> BM26A	<b>Date of experiment:</b> from: 31 January 8.00 to: 1 February 8.00 6 more shifts have been allocated from 10 March 8.00 to 12 February 8.00, 2005	<b>Date of report:</b> February 25, 2005
<b>Shifts: 3</b>	<b>Local contact(s):</b> Sergey NIKITENKO	<i>Received at ESRF:</i>
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## Report:

We have enjoyed three shifts at BM26A. According to the project proposal, we recorded the XANES spectra at the V K- edge (5465 eV) and the Sb L<sub>1</sub>-edge (4698 eV) of some oxides submitted at different heat treatments. However, some problems were found to set up the experiment at the Sb L<sub>1</sub>-edge (4698 eV), mainly because of its low Energy and the background absorption of the samples. For this reason, the time remaining to carry out the planned experiments was quite short and we were re-allocated six more shifts in a short time in order to complete the experiments.

We measured the V-K edge XANES spectra of the Mo<sub>0.94</sub>V<sub>0.23</sub>Te<sub>0.23</sub>Nb<sub>0.12</sub> oxide which posses the crystalline phase M1 (see the experimental proposal). The sample was submitted to two different treatments: i) air 450 °C, ii) N<sub>2</sub> 600 °C and air 450 °C. Then, we also recorded the spectra of the sample treated as i) after its use as a catalyst in the selective oxidation of propane. The same samples were measured at the Sb L<sub>1</sub>-edge (4698 eV), but we were no able to complet the experiments at the V K-edge (sample after treatment ii) used in the catalytic test.

The results obtained show that the oxidation state of Sb changes as a function of the treatment of the sample. While in the catalyst treated in air at 450 °C Sb is as Sb<sup>5+</sup>, this is partially reduced (80 % is as Sb<sup>3+</sup> when the catalyst has been treated with N<sub>2</sub> at 600 °C in a previous step. Some Sb<sup>5+</sup> is partially reduced to Sb<sup>3+</sup> after the catalytic tests. The changes V XANES spectra are not so evident, and it appears that the

changes in the oxidation state of Sb are not accompanied by changes in the state of V, which is mainly as  $V^{4+}$ . However, more experiments are required to clear out the changes in the catalysts after the different treatments and after reaction and on a different crystalline phase, catalitically active (denoted as M1). These are planned to be carried out in the next shifts in March.

We also measured some XANES spectra at the V K-edge of the catalyst precursor (before any treatment), as we have observed that the final stage depends on the specific preparation conditions, for a catalyst of chemical composition:  $Mo_{0.94}V_{0.23}Te_{0.23}Nb_{0.12}$ . We have observed that in this case, before any treatment, the V is as  $V^{5+}$ , and the shape of the spectrum slightly varies with the preparation conditions. Again, more measurements must be done.