ESRF	**Experiment title:** X-ray Absorption Spectroscopy of $Me_3(VO_4)_2$ (Me: Ca, Sr, Ba) at High Pressure and Temperature	**Experiment number:** CH361
Beamline: ID24	**Date of experiment:** from: 29 Oct 97 to: 2 Nov 97	**Date of report:**
Shifts: 12	**Local contact(s):** Dr Sakura Pascarelli	***Received at ESRF:***

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Report:

The goal of our project was to perform high pressure XAS experiments at the vanadium K-edge at less than 7 keV. In this case, the strong absorption of diamonds at 5 keV does not allow for the use of a conventional geometry of the experimental set up (a beam path through the diamonds). We thus proposed that the transmission of the beam would be measured through the beryllium gaskets placed between the diamonds. The spot size of the beam should then be of about the same size as the gap between the culets of the diamonds (about 30-40 μm). We indicated that the energy dispersive EXAFS ID24 station at ESRF would be a good place to carry out such experiments because of the X-ray beam size of 15 μm FWHM at the focal point. Our experiments also required a special vacuum chamber to hold the cell in a non-standard setting, to measure the pressure, and to evacuate all the air from the X-ray path between the monochromator and detector. It should be noted that high pressure classical XAS experiments utilizing beryllium as a gasket material were successful [Kaindl et al., ***Phys. Rev. B, 38,*** 10174 (1988); Hu et al., ***Phys. Rev. B, 49, 39*** (1994)].

We decided to start our experiments with collecting XAS reference spectra of pure V_2O_5 inside the gasketed diamond cell (diamonds with the 600 μm diameter culets, the 200 μm sample chamber) without any pressure to compare them with the ones already published [Nabavi et al., J. ***Phys. Chem. So/ids,*** **51,** 5 (1990)] and to optimize the alignment of the experimental set up. However, during the entire

allocated time to our work (12 shifts), we were not able to obtain any XAS spectra of V_2O_5. Most of the time we spent trying to align the ceil in respect to the X-ray beam. While we were able to observe the X-ray beam intensity decrease due to the diamond and beryllium gasket absorption, there was no evidence for the K-edge absorption spectra of vanadium. We believe that our failure was due to the experimental set up. In the future attempts to carry out the XAS experiments with Be gaskets, a better design of the diamond cell would be required. We used a membrane cell with two slots cut at its side for the X-ray beam to pass through the cell. We found that the slots were just too small to freely orient the cell in the vacuum chamber.

These initial experiments at the EXAFS ID24 station at ESRF gave us invaluable experience in designing this new type of *in situ* high pressure experiment on light element XAS in the diamond anvil vell with Be gaskets. We plan follow-up experiments at both ESRF and LURE as we continue to compare the advantages of classical and energy dispersive XAS for carrying out such studies. Proposals to both LURE and ESRF are being prepared to carry out these experiments, in the context of establishment of our program in solid state chemistry at high pressure.