ROBL-CRG	**Experiment title:** Photoacoustic Detection of the XAS Signal	**Experiment number**: MI-346
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Report:

XAS experiments are normally carried out by detecting transmitted X-rays. The signal is detected by gas ionization chamber detectors positioned before and after the sample. This method is limited in the concentration of the absorber because too low absorption generates a very small difference in the signals of the two ion chamber detectors.

In a photoacoustic measurement, the signal is generated by the production of transient heat after absorption of X-rays[1),2)]. This signal is directly proportional to the concentration of the absorber. Therefore, a lower detection limit for the absorber is expected. The method can be compared to Laser-Induced Photoacoustic Spectroscopy (LIPAS). Comparing this method to conventional UV-Vis spectroscopy, we have an increase in the detection limit up to three orders of magnitude [3)].

In a first run of 4 shifts we tested a normal photoacoustic design using ring piezo ceramics and piezo plates. The sample was mounted on the piezo using TESA-film. The electrical signal from the piezo was amplified by a spectroscopic amplifier to reduce the impedance and the noise of the signal and measured with an oscilloscope. The synchrotron beam was interrupted using a chopper system. A signal of this chopper was used for synchronisation of the measured signal with the frequency of the chopped beam. Unfortunately, we did not observe the prospected photoacoustic signal. This may have been caused by the following disadvantages:

i) The contact beteween the sample and the piezo ceramic detector was not stable enough. In a next run we will avoid this problem by using special contact paste.

ii) The concentration of the absorber in the sample was not adequate to the proposed signal.

iii) The used chopper did not absorb enough of the synchrotron radiation, so that the sample was excited continuosly. If this is the case, we have no interuption of the beam, which causes at least no detectable signal.

Using a frequency analyzing system, we tested the frequency of the photoacoustic signal. The measurements at the ESRF showed a very small difference signal between a run with and without an absorbing sample. The maximum of the difference in the frequency was at 30 Hz. At this frequency we can expect the photoacoustic signal. The next step is to determine the delay time between the chopped synchrotron beam and the photoacoustic signal. In laser-induced photoacoustic measurements, this delay time is about 30 µs. This may be somewhat shorter in the synchrotron based photoacoustic mesurement due to the shorter distance between the sample and the detector. A chopped cw-laser will be used to measure the signal delay.
A second experimental run with synchrotron radiation at the ESRF is planned after test measurements of the photoacoustic signals with very low chopped laser energies.

[1)] T. Masujama, Photoacoustic X-Ray Absorption Spectroscopy, in P. Hess and J. Pelzl (Eds.), Photoacoustic and Photothermal Phenomena, Springer Series in Optical Sciences 58, Springer Verl. Berlin, Heidelberg 1988, p.19

[2)] T. Masujama, X-Ray Photoacoustics for Characterization and Non-Destructive Evaluation, in J.C. Murphy et.al. (Eds.), Photoacoustic and Photothermal Phenomena II, Springer Series in Optical Sciences 62, Springer Verl. Berlin, Heidelberg 1990, p.222

[3)] G. Geipel, G. Bernhard, V. Brendler, H. Nitsche, Complex Formation between UO_2^{2+} and CO_3^{2-}: Studied by Laser-Induced Photoacoustic Spectroscopy (LIPAS), Radiochimica Acta, 82, 59 (1998).