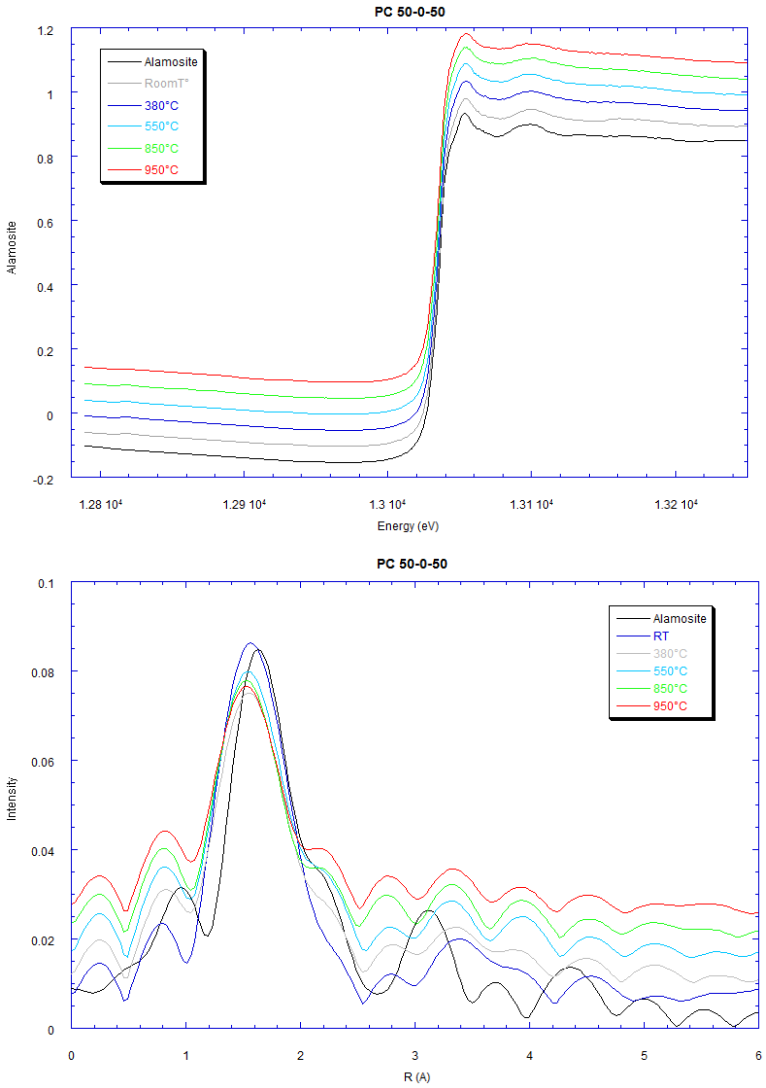
Report about the experiment MA-2009 performed on the beamline BM 26A

At the BM26A beamline, we performed XAS spectroscopy (XANES and EXAFS) experiments. We collected spectra on several samples of lead-based glasses and crystals at room temperature and high temperatures (only for glasses). We studied four different compositions of lead-based glasses and three lead-based crystals (minium, alamosite and plattnerite). The glass samples were previously synthesized and quenched at the Laboratooire Géomatériaux et Environnement, Université Paris Est. We used a heating wire system to perform in-situ acquisitions at high temperatures.

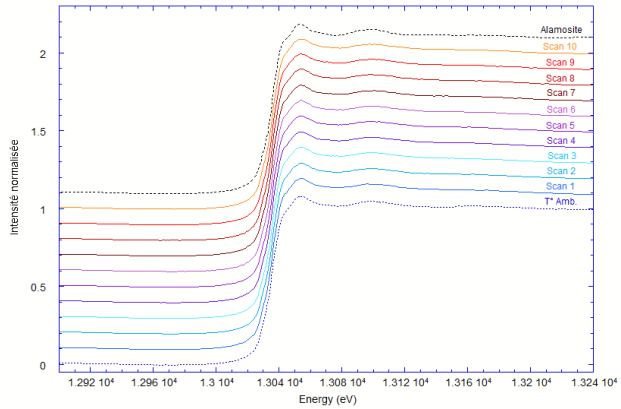
1) The first glass studied is an alamosite-typed glass ($PbSiO_3$). This composition melts at 760°C, and then we performed four other experiments on this sample at room temperature, 380°C (under the glass transition temperature), 850°C and 950°C (melt acquisitions) and 550°C (nucleation). Several acquisitions have been made for each temperature.

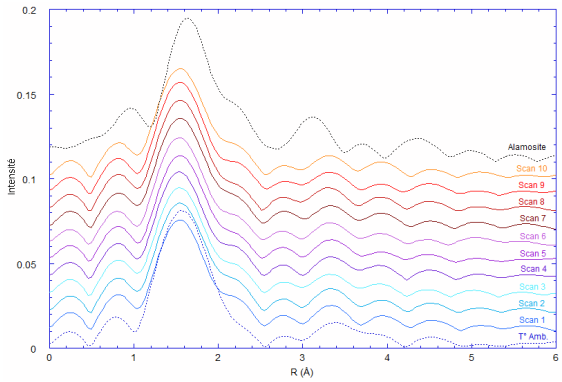


We also performed experiments on the composition $(Ca_{0.5}\ Pb_{0.5})SiO_3$, which is close to the previous one. We only replace half of lead by calcium. We made acquisitions at room

temperature, 750°C, 950°C for nucleation and 1400°C for melt. Four others compositions were also scheduled but we didn't have enough beam time to complete them.

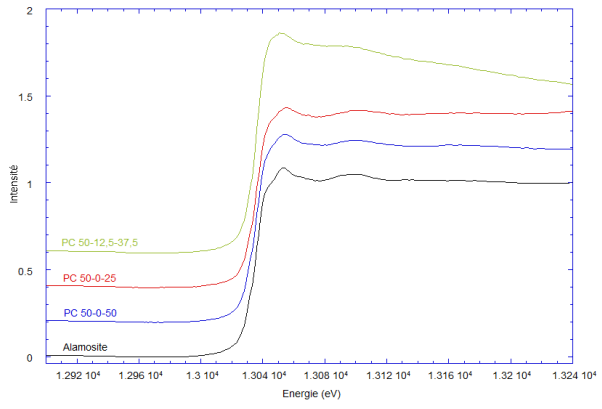
For the same alamosite-typed $PbSiO_3$ composition, we could also follow the nucleation and growth process during 10 scans of 25 minutes each, then during a bit more than 4 hours. We could observe only a slight evolution after 4 hours: longer runs are probably necessary for a better understanding of these processes.

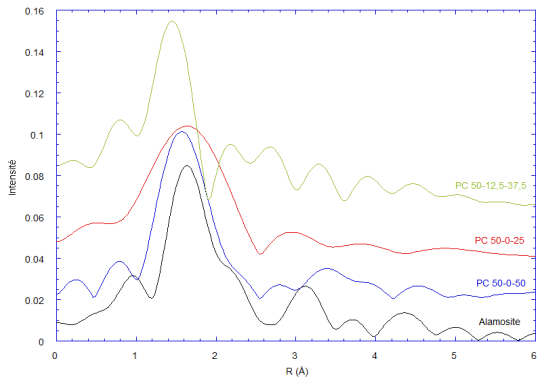




2) At ambient temperature we obtained preliminary spectra on 3 different compositions:
PC-50-0-50: SiO_2/PbO = 50/50 in mol%
PC 50-0-25: SiO_2/PbO/CaO = 50/25/25 in mol%
PC 50-12.5-37.5: SiO_2/PbO/Al_2O_3 = 50/37.5/12.5 in mol%

After the fourrier transform of the spectra, these preliminary results seem to reveal that Al could favour a better structured lead-bearing glass while Ca could have the opposite effect.





Further work is required and other compositions need to be studied for a better understanding of the effect of elements like Al and/or Ca on the local environment around lead in glasses and during the nucleation process.