	Experiment title: **Tracking the speciation of Br in magmas at depth**	**Experiment number**: HC776
Beamline: BM23	**Date of experiment**: from: 30/04/2013 to: 06/05/2013	**Date of report**:
Shifts: **18**	**Local contact(s)**: Innokenty Kantor	*Received at ESRF:*

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Abstract:

Halogens are minor volatiles in the Earth's mantle and crust, but they have significant and specific influences on magmatic and degassing processes. They also provide insights about subsurface magma movement and eruption likelihood in subduction-related volcanism. Their speciation in silicate melts affects volatile exsolution, rheology, and the thermodynamic properties of the melts but still remains relatively unknown. A few studies have explored halogen speciation at room conditions, i.e. in glasses but no firm conclusion has yet been reached. Furthermore, halogen speciation remains unexplored at high pressures and temperatures.

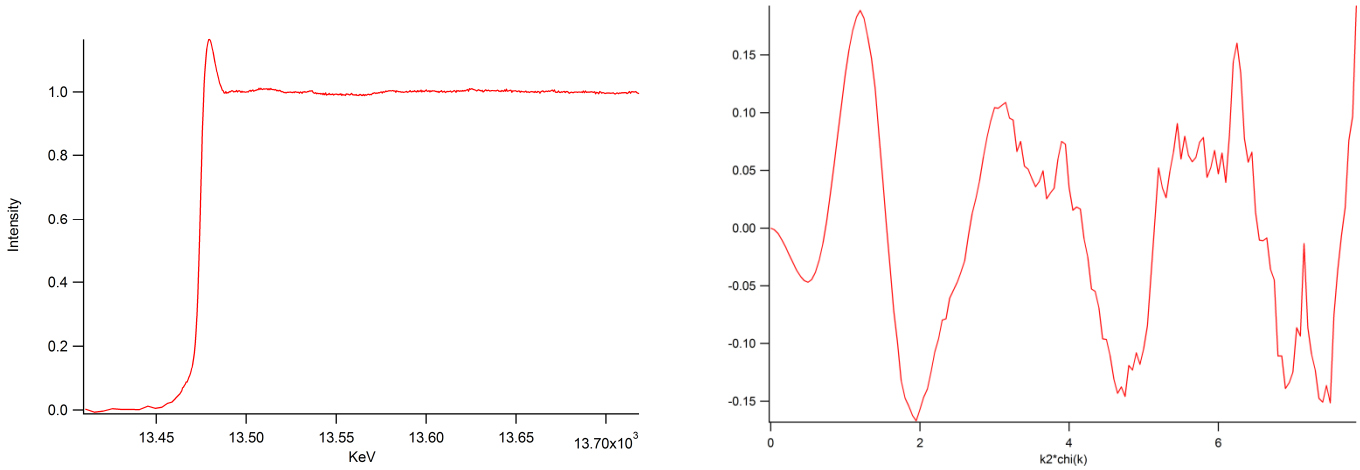
In this work we investigated the speciation of Br in subduction-related melt (hydrous haplogranite melt) up to 1200°C and 7 GPa using X-ray absorption spectroscopy (XANES and EXAFS) at the Br K-edge. High P-T conditions were generated by the Paris-Edinburgh press. The use of nanocrystalline diamond capsules enabled us to avoid glitches in the EXAFS spectra. The results provided valuable information on Br speciation and its evolution with pressure. It gave insights into solubility mechanisms for halogens in magmas at depth and on their degassing from the melt. In addition, we were able to identify quench effects on the atomic environment of Br by comparison of high P-T in-situ spectra and ex-situ spectra recorded on quenched samples.

Experimental conditions:

High P-T conditions up to 7 GPa-1200 K, have been generated by the Paris-Edinburgh press that we regularly use for X-ray diffraction on melts at ESRF. The experimental cell-assembly consisted of a boron-epoxy gasket (7 mm diameter), a graphite heater, and a diamond capsule capped by Pt-Rh discs to preserve the chemical integrity of the sample. We used nanocrystalline diamond capsules (from T. Irifune) as the glitches from either a single or even polycristalline crystal would have prevented a proper collection of the EXAFS signal (see report HD563). Talc powder was packed on the external sides of the Pt-Rh caps (i.e. away from the sample) to control the oxygen fugacity. The intensity of the beam before and after the sample was measured by ion chambers in order to collect the EXAFS signal. P was measured from the X-ray diffraction signal of a Pt foil inserted on the side of the diamond capsule. The X-ray diffraction signal was collected from a MAR CCD detector. T was read from the power curve, as we now have a good database for this cell assembly.
The sample was a hydrous granite containing ~3000ppm Br (calculated from the EXAFS absorption jump). EXAFS spectra were collected above the liquidus at the Br K-edge (13.47 keV), at different P steps (2 GPa, 5 GPa and 7 GPa) and on the quenched sample back to room conditions. We also recorded spectra on Br oxide standards (KBrO3 and NaBrO3 bromates) in order to identify Br speciation by comparison.

Results:

To obtain a good signal to noise ratio, 5 to 9 EXAFS spectra were recorded for a given P/T condition. The figures below show the EXAFS spectrum recorded at 5 GPa-1200°C, plotted as a function of energy (left) and its k2-weighted EXAFS oscillations (right). The data are of good quality and will enable us to determine Br speciation and local environment.



Figures: EXAFS spectrum recorded at 5 GPa-1200C, plotted as a function of energy (left) and k2-weighted EXAFS oscillations (right)

With the set of EXAFS spectra recorded at different P/T conditions, we will get valuable information on Br speciation and its evolution with pressure. Careful EXAFS and XANES analyses combined with XANES calculations are being undertaken at the moment to constrain the local environment of Br at high pressure.