ESRF	**Experiment title:** Ordering in metal trichloride melts	**Experiment number:** HS2269
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Report:
The aim of this experiment was to investigate the local atomic structure of several salts (in liquid and solid phase) belonging to the metal halides group: YCl_3, $SbCl_3$, $InCl_3$. In order to carry out the experiment great care was needed during samples manipulation due to their strong sensitivity to moisture. A novel type of quartz cell has been designed and manufactured which guarantees the sample confinement in vacuum environment and which is suitable for high temperature X-ray measurements in transmission geometry. The cell is made by two quartz flat windows (20mm × 15 mm, thickness 0.5 mm) soldered close to their borders and a hollow quartz cylinder (fig. 1). The triangular shape of the lateral surfaces of the cell gives higher rigidity to the structure and it permits to tune the sample absorption varying the vertical position of the cell (fig. 2). Due to its small thickness, the cell can be easily positioned inside a standard C crucible matching perfectly with the L'Aquila-Camerino oven geometry. Three quartz cells have been filled, in inert (Ar) atmosphere, with commercial high quality metal trichlorides YCl_3, $SbCl_3$, $InCl_3$ (Aldrich) and then accurately sealed under vacuum. In order to balance the vapor pressure inside the quartz cells at high temperature, the L'Aquila-Camerino oven has been filled with 100 mbar of N_2 during the measurements. The continuous monitoring of the temperature has been obtained with an Inconel insulated thermocouple (Thermocoax) inserted between the crucible and the cell, very close to the x-ray beam. A set of XAS spectra from $25°C$ up to the samples melting temperatures have been collected at the Y, In, Sb K-edge energies (17038 eV, 27940 eV, 30491 eV) exploiting the high photon flux provided by the bending magnet X-ray source.

The excellent temperature stability along the whole sample provided by the L'Aquila-Camerino furnace and the good X-ray transmission coefficient of the quartz cells (T = 50% at about 19 KeV) have made possible to obtain high quality data also at high temperature. The sample melting has been verified performing several ESXD scans with the powerful multichannel detector available at BM-29. In figure 3 and 4 four XANES spectra are shown related to the $SbCl_3$ and YCl_3 samples in the solid (red solid lines) and liquid (blue dot-dashed lines) phases. As expected, after melting the local environment in these salts doesn't seem to change drastically as confirmed by the similarities between the solid and liquid spectra, which are particularly evident for the $SbCl_3$ case. These preliminary results are promising, showing that a reliable determination of the local structure for metal-halides is feasible with the experimental techniques developed at BM-29. The collected XAS data are currently under treatment and a more advanced quartz cell has been developed in order to increase the structural cell rigidity and obtain a higher X-ray transmission.

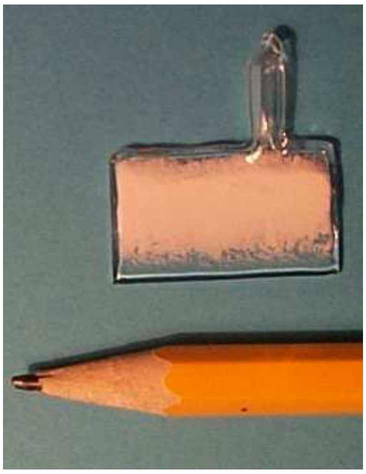

fig. 1: the quartz cell filled with a powder

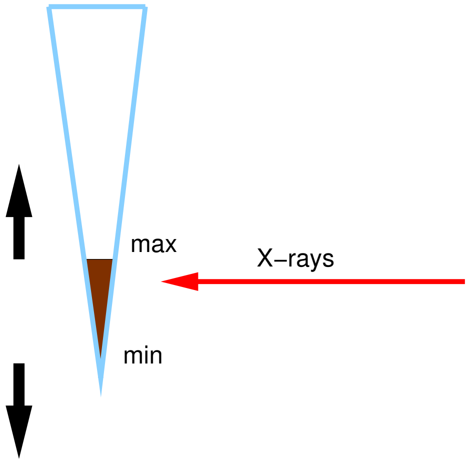

fig. 2: quartz cell: side view

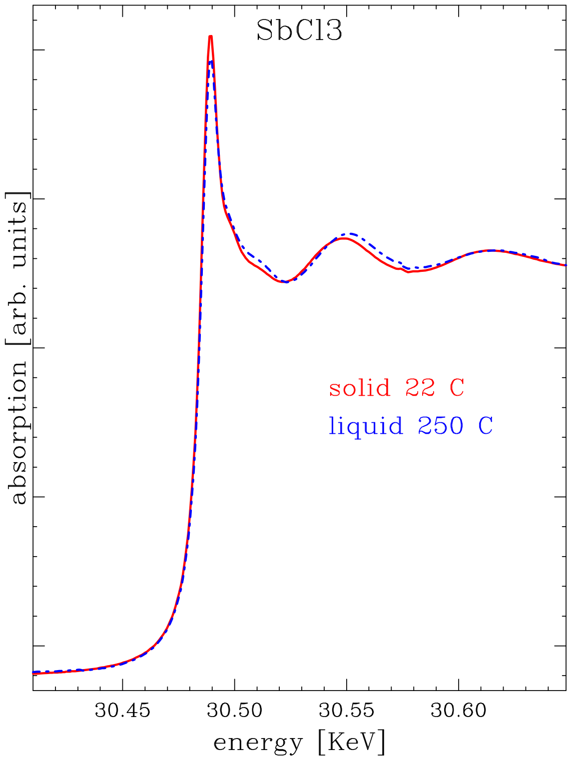

fig. 3: XANES of solid and liquid $SbCl_3$

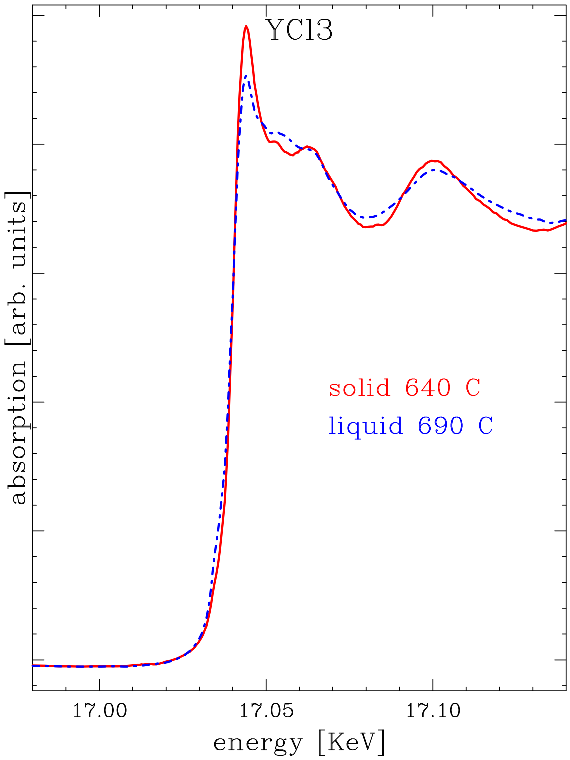

fig. 4: XANES of solid and liquid YCl_3