ESRF	**Experiment title:** Investigation of the rattling motion of Potassium	**Experiment number**: he3957
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Shifts: 18	**Local contact(s)**: Simon Brown	*Received at ESRF:*

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Report:

The purpose of the experiment was to investigate forbidden reflections in the pyrochlore compound KOs_2O_6. This compound is interesting because it hosts rattling potassium ions in large cages. At low temperature it becomes superconductor and it is believed that the superconductivity is related to the rattling motion of the Potassium ions. We hoped to measured the Thermal Motion Induced Resonant Scattering at the Potassium K edge and witness some effect across the superconducting transition and another less understood phase transition.

We mounted 3 small crystals provided by our collaborator Junichi Yamaura (Figure 1), and measured 2 of them. They were single grains of good crystallographic quality, as seen by the sharp rocking curves on allowed reflections.

However, we did not manage to find any forbidden reflection. We could not observe the fluorescence either. The reason could be the high absorption of x-rays at such low energy (3.6 keV), despite the care taken to reduce the air path and the windows, combined with the intrinsic weakness of the signal, and the strong self-absorption due to the heavy Osmium atoms.

After 3 days of search without success, we decided to switch to another project, closely related in topic.

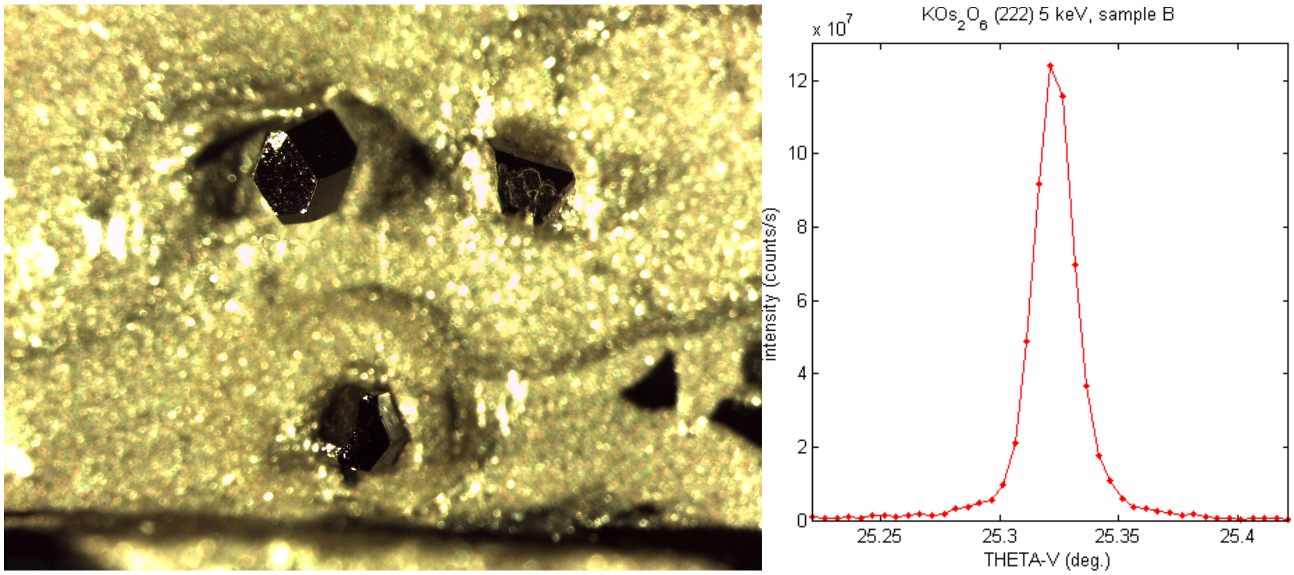

Figure 1: Left: photo of the KOs2O6 crystals mounted on the diffractometer. The larger one is about 0.5 mm. Right: rocking curve on the (222) allowed reflection of the larger crystal. FWHM is ~0.02°.

During the 3 remaining days, we investigated forbidden reflections in KH_2PO_4 (KDP) at the Potassium K edge. In this material, the temperature dependence of the forbidden reflections could be related not only to thermal motion, but also to quantum tunnelling of the protons between two equilibrium positions either side of a higher symmetry position. We already knew from previous measurements that the resonant scattering in KDP is particularly strong.

We managed to measure the spectra of the (002) and (222) forbidden reflections at 2 different azimuths as a function of the temperature between 10 K and 320 K (Figure 2). The structural transition at ~123 K is responsible for the sudden change of shape and intensity (the lower temperature structure allows E1E1 terms, not the higher temperature one), and above 123 K there is a smoother but clear temperature dependence. Analysis is now in progress to fit the data, taking into account both the thermal motion and the quantum tunnelling.

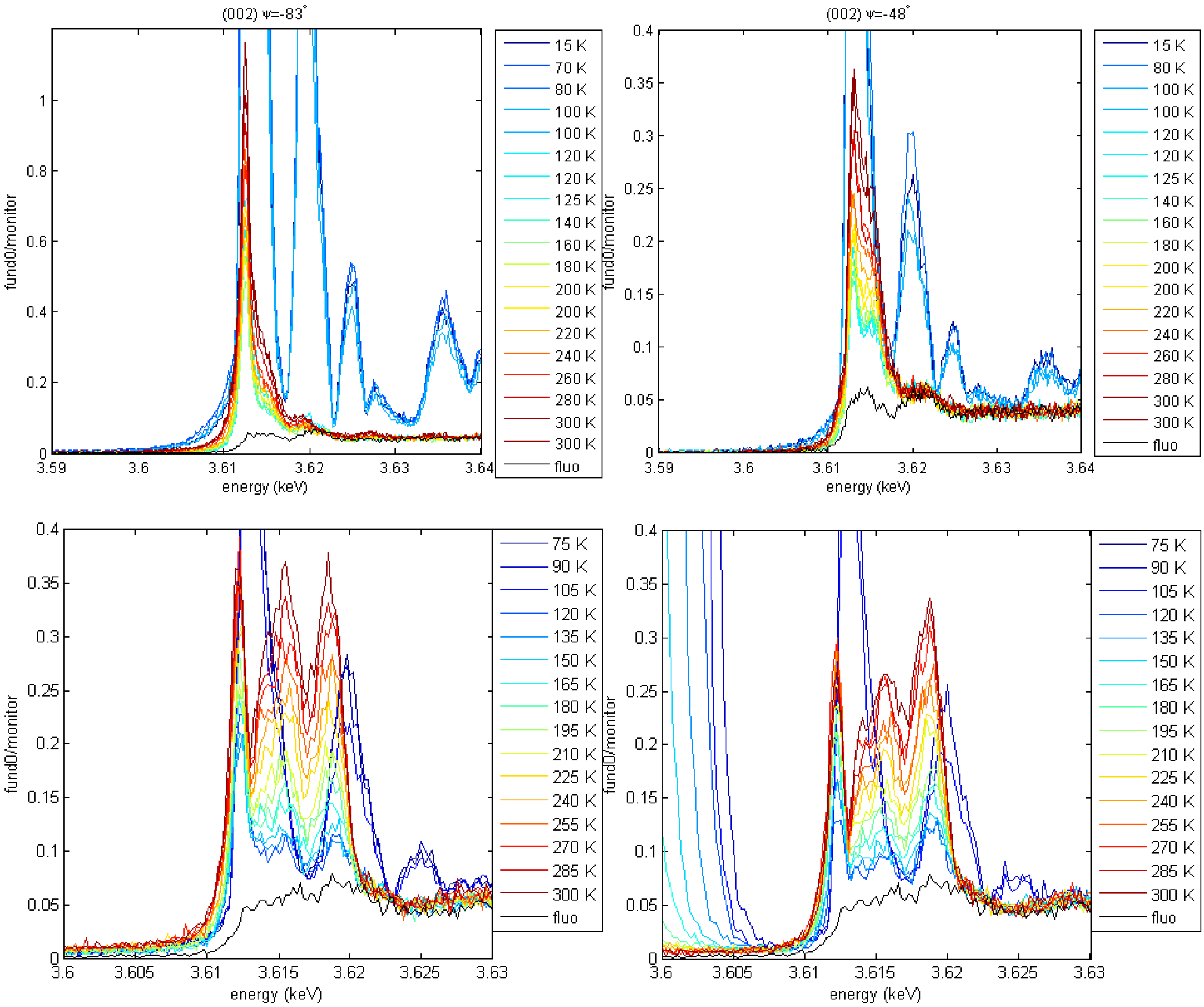

Figure 2: Spectra of the forbidden reflections of KDP as a function of temperature. Top row: (002) (left: azimuth -83°; right: azimuth -48°). Bottom row: (222) (left: azimuth -60°; right: azimuth -47°). Azimuthal reference: (100) for (002) and (001) for (222).