ESRF	**Experiment title:** **Phase transition in optically active $Rb_2S_2O_6$**	**Experiment number:** CH-424
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Preliminary Report:

A lmm diameter capillary was filled with optically active $Rb_2S_2O_6$ and mounted on a goniometer on the X-Ray-powder diffractometer. The wavelength used was 0.85 A, for which the absorption of rubidium is low. A high quality data set was taken at room temperature (297K) for 6 hours, with 2θ ranging from 0° to 78°, at 1°/minute every 100 miliseconds. The low angle data (28 ranging from 0° to 40°, was scanned one time and the high angle data (from 40° on) was scanned three times. As it was known that the transition is accompanied by a temperature hysteresis of $\cong$ 30K in the optical activity measured along

the optic axis, the temperature was lowered slowly and short runs, of ten minutes each, in a 2θ range from $5°$ to $20°$, where taken at a 10K intervals starting at 297K. This interval in 28 was chosen because at small angles the room temperature pattern shows fewer reflections and consequently was easier to follow the modifications expected in the X-ray pattern. In fact, at 240K some new reflections started to appear and raised in intensity, so that at 230K splitting of other reflections was also observed. The most striking feature was the existence of a new very strong reflection close to the already existing (111). At 230k the room temperature and the low temperature phases coexist at almost 50% each. Further decrease in temperature leads to an increase of the low temperature phase reflections and the disappearing of some room temperature ones. At 220K the reflections (101), (200),(102), (21l), (301), (202) which were already weak at room tempearture are now missing. A high quality data set was taken at 230K for 6 hours, with 28 ranging from $0°$ to $78°$, at 1^{o}/minute every 100 miliseconds. Just like was done for the room tempertaure run, the low angle data (20 ranging from $0°$ to $40°$, was scanned one time and the high angle data (from $40°$ on) was scanned three times. Far away from the transition (at 200K) a high quality data set was also taken within the conditions used for the other two long runs. To cover the temperature hysteresis the sample was warmed slowly in a similar way to the cooling process, short runs being taken at 10K. The existence of a hysteresis is confirmed as is at 265 K that the room temperature and low temperature phases coexist again at almost 50% each. The phase transition was observed to be reversible. It was also found that the transition is accompained by a pronounced collapse of the c lattice parameter from 6.42 to 6.19 Å which is clearly shown by the changing in the position of some reflections towards higher 28 values.

The Room Temperature structure has already been refined from the BM16 measurements with a χ^2 of 6 before refinement of thermal vibration parameters or the application of absorption corrections. The refined positional parameters agree, to within the standard deviations, with the previous values obtained by us from a single crystal study and we therefore expect an equally good result for the low temperature structure.