MLF Experimental Report	提出日 Date of Report
課題番号 Project No. 実験課題名 Title of experiment Structure of liquid SnI_4 実験責任者名 Name of principal investigator Kazuhiro Fuchizaki 所属 Affiliation Department of Physics, Ehime University	装置責任者 Name of responsible person Toshiya Otomo 装置名 Name of Instrument/(BL No.) NOVA/BL21 実施日 Date of Experiment March 14–15

試料、実験方法、利用の結果得られた主なデータ、考察、結論等を、記述して下さい。（適宜、図表添付のこと）
Please report your samples, experimental method and results, discussion and conclusions. Please add figures and tables for better explanation.

1. 試料 Name of sample(s) and chemical formula, or compositions including physical form.

tin tetraiodide (SnI_4)
a molecular crystalline solid with the space symmetry Pa3 composed of molecules with the point symmetry Td at ambient conditions

NOTE Because SnI_4 is a hazardous substance, a careful treatment is required upon sampling.

2. 実験方法及び結果 （実験がうまくいかなかった場合、その理由を記述してください。）
Experimental method and results. If you failed to conduct experiment as planned, please describe reasons.

Experimental Method

Sample preparation

A silica tube with dimensions of 5.5 mm outer diameter (4.5 mm inner diameter) and 65 mm length was loaded with a powdered sample that has been grounded from polycrystalline samples. The sample actually loaded was about 30 mm in length. The end of the tube was sealed with care by glasswork. The sealing was confirmed by no SnI_4 gas leaked out even after the sample was boiled by heating the tube. This confirmation was made twice. Another sample with almost equal quantity was prepared in exactly the same way as a reserve. These two samples were registered prior to the experiment.

Measurement

One of the tubes containing the sample was held in an electric furnace fixed in a vessel. The vessel, including the furnace, was then evacuated for about an hour to attain 10^{-3} Pa. The furnace was gradually heated, while the vessel was kept evacuated. It took about three hours to reach and be held at 160 degrees centigrade.

2. 実験方法及び結果（つづき） Experimental method and results (continued)

Although the fluctuations of temperature are rather large (146.3 degrees at minimum, and 178.1 degrees at maximum), the first scan was started. The scan was, however, aborted due to some troubles occurred in the heater system. The network connection between the heater and the controller was also lost. After fixing these problems, the scan at 160 degrees (nominal temperature) was restarted. It took about 2 hours for this first scan. Another three scans were done at temperatures, 200, 250, and 300 degrees. It took about four to five hours for each scan.

After these sample measurements, a blank experiment, in which only a silica tube is measured, was originally planned. It is necessary to subtract the scattered intensity from the container to obtain the intensity from the sample itself. However, the blank experiment was not carried out because of the reason mentioned below.

Experimental Results

Because we have to go through many complicated procedures to obtain the structure factor, and it takes several months to carry out the whole process, we looked into the raw intensity profile stored in a single counter after all the sample measurements were done. Contrary to our expectations, there was no quantitative change in the profiles against the variation in temperature. Moreover, the profile seems to resemble the one obtainable from vitreous silica. At this moment, we all suspected that what we measured were not a sample but only a container.

To confirm this unpleasant expectation, we had to break the vacuum to take the tube out from the furnace. Quite unfortunately, no sample was retained. The sample completely leaked out, leaving many faint horizontal stripes on the inner wall of the tube. The latter fact implied that the leakage of the sample took place intermittently after it melted. The problem was when the leakage happened. If this happened during the measurements, the leakage of the sample necessarily brought about a quite troublesome situation of contamination. Fortunately, radiation measurement showed no indication of contamination.

We tabulated chronologically all the events during the experiment by reviewing a log kept by each researcher, and noticed that there was a sudden drop in a degree of vacuum at early stage of the vacuum drawing process during heating the sample beyond the melting point. Some of the researchers noticed this event at that time, but they thought that this was caused by diffusion of boron nitride, a substance used as a lubricant upon fixing the tube to the furnace. Perhaps, this deterioration in the degree of vacuum was brought about by diffusion of the sample that leaked as a gas. The leakage was probably completed before the temperature reached 160 degrees at which the first scan began. If this was the case, then the sample was not irradiated at all. This may be the reason why no contamination was detected.

Examination of the Failure to Prevent Recurrence

No microcracks were detected on close inspection of the tube surface using a microscope. However, this fact did not deny a possibility of a presence of even smaller cracks associated with deterioration; the tube was not a new one. Another cause of the leakage was due to incompleteness of the sealing. Considering rather a high saturated vapor pressure of the sample, sampling in vacuo should have been desired.