

**Experiment title:**

Determination of Molecular Motion in Aspartame in the Crystal

Experiment number:

01-02-352

Beamline:

BM1A

Date of experiment:

from: 21.11.01 to: 27.11.01

Date of report:

27.2.02

Shifts:

16

Local contact(s):

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Two shifts were lost at the beginning of the experiment because of machine problems.

When we started checking crystals on the 6-circle diffractometer, we found that the few crystals of aspartame we had were not good enough to be measured on the Kuma.

During the second day of experiment we decided to use the following beamtime to complete the project on Glycyl-L-Alanin (CH-1004) for which in the last experimental session we were not able to measure the very low temperature data-sets essential to the ADP-analysis.

In order to make full use of the short time left, we also decided to change the instrument and switch to the MAR345 image plate system. The parallel optic usually used for the Kuma diffractometer was kept, in order to reach a short wavelength and therefore a high resolution on the MAR plate.

The Helijet was used to measure three additional data-sets on the Gly-Ala crystals at 15, 30 and 60K.

The liquid-Nitrogen cryostream has been used to measure an additional data set at 100K, to allow a comparison between the different techniques of data-collection on the same compound (diffractometer equipped with point-detector v. image plate system).

Problems occurred as soon as we started ramping down in temperature with the Helijet (a later inspection of the Helijet head revealed a vacuum leak).

Few cycles of cooling-down/warming-up, while pumping the vacuum, were necessary to reach 15K and stabilize the temperature there. We had also ice formation all around the sample, for which we had to enter the hutch from time to time and clean out the ice on the sample holder.

Preliminary processing of the 15K data showed that the diffraction patterns are noisy, most likely due to ice formation during data collection. Internal consistency is also not excellent (R_{int} is only 0.1635 and R_{sigma} is 0.0661) but this could be due to the software used for processing that did not account properly for the area of the image plate obscured by the shadow of the cryostream.

A first attempt to use these 15K data in the ADP's analysis, together with the data already collected at 100, 160 and 220K (see report on experiment CH-1004) showed that:

the frequency associated with the libration an axis parallel to the crystallographic b-axis is still undetermined ($250(131)\text{cm}^{-1}$)

the variances on the values of the temperature-independent contributions to ADP's are reduced compared to those obtained with only the datasets at 100, 160 and 220K but still of the same order of magnitude of the tensors components themselves

an agreement factor of only 15% between calculated and observed ADP's, is a clear indication that additional molecular deformations on the top of the usual rigid-body librational and translational descriptors need to be included in the model of motion.

A new treatment of the data with a new software package, including also the 30 and 60K sets, and the elaboration of a more general model of motion of Glycyl-L-Alanine in the crystal are still in progress.