ESRF	**Experiment title:** Motion of a dipeptide in the crystalline state as observed by multitemperature x-ray diffraction experiments.	**Experiment number**: CH-1004
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Report:

The primary results of a crystal structure determination are mean atomic positions and Anisotropic Displacement Parameters (ADP's). These latter represent the time and space average distribution of atoms around their mean position in the crystal and are composed of many different contributions: the mean square atomic displacements from all possible motions (libration, translation, internal vibrations), but also those coming from disorder (static or dynamic) and those reflecting systematic error either in the diffraction data or in the refinement model. A new method to derive the correlation of atomic motions from the temperature-dependence of ADP's has recently been developed[1].

Scope of this experiment was to apply the new method of analysis to the diffraction data collected at various temperature for a crystal of Glycil-L-Alanine, and consequently determine the characteristics, in terms of correlated atomic displacements and associated effective vibrational frequencies, of the molecular motions in the solid.

Data have been collected on the 6-circle Kuma diffractometer at BM1A with a point detector, in order to achive a high accuracy in the determination of the weak, high-order reflections, that give essential contributions to the ADP's. Three sets of data have been measured respectively at 100, 160 and 220K with the aid of a liquid-nitrogen cryostream, up to a resolution in $\sin\theta/\lambda$ of 0.91 Å, for a total of ~7800 reflections (~2600 uniques) for each data collection.

Unfortunately none of the data collections run smoothly from the start to the end, because of problems with the software controlling the diffractometer, consequently all of the datasets are split into pieces that need a very delicate and time-consuming scaling and merging.

An attempt to collect an additional dataset at 15K was made. We used the prototype of a liquid-helium cryo-stream from Oxford Diffraction (Helijet). The lack of a temperature controller on the cryo-stream and the fact that we had to use the manual flow-control to reach the working temperature are the main reasons for which this attempt failed. In addition, the high flow rate and the difficulty in stabilizing a laminar helium stream at crystal position using only a manual valve blew off about 8 crystals from the goniometer head during measurement. For each of those crystals we had to repeat the peak-hunting and orientation matrix refinement procedures and at the end of the allocated beamtime at this temperature we had only cell parameters.

An early processing of the three datasets collected above 100K shows that the quality of the high- resolution data is very good (R_{int} ~ 2.5%) but that the low-resolution, intense reflections needs a more accurate scaling. The intensity of the low-order reflections has, in fact, been measured using a Cu-foil filter to attenuate the signal and bring it down to a reasonable scale compared to the dynamical range of the detector, therefore if the relative filter factor used in data processing is not extremely accurate it is likely to give problems in the final scaling. An overall R_{int} of ~ 9% for the three datasets allowed anyway to refine all the structures down to ~ 4% in the spherical atom approximation.

Even if the 15K data was not available and therefore the analysis could be biased by the absence of data in the quantum regime, a preliminary analysis of the ADP's at 100, 160 and 220K has been performed.
The results, in terms of the values of the normal mode frequencies for the librational and translational modes, reflect the hydrogen bonding interactions that Glycil-L-Alanine makes with the neighboring molecules.
The highest frequency, that appears to be indeterminate with a value of ~440(680) cm^{-1}, is associated with a libration around an axis parallel to the crystallographic b-axis, such a motion is likely to deform all the hydrogen bonds in the structure and this is explain the amount of energy required to perform it. On the other end of the normal mode frequencies range, the lower value (44(1)cm^{-1}) is associated with a translation of the molecule as a whole in the direction of the crystallographic c-axis.
The temperature-independent contributions to ADP's coming from internal vibrations have also been determined in the analysis, but the large variances associated with those values are a clear indication that additional information from the very-low-temperature quantum-regime are necessary.

Additional handling of the collected data is still in progress and, because of the small dis-adventures on the experimental side, it is going to take some more time.

1. Bürgi H.B., Capelli S.C., (2000), *Acta Cryst*., **A56**, 403-412.