ESRF	Experiment title: Electron density and thermal motion in boron nitrilotriacetate	Experiment number: CH-517
Beamline: BM01A	Date of experiment : from: 23.09.98 to: 29.09.98	Date of report : 25.01.00
Shifts: 18	Local contact(s): Kenneth Knudsen	Received at ESRF:

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Report:

The goal of the present experiment was to study the details of the electron density of boron nitrilotriacetate (NTAB) using data with very high resolution and accuracy measured with the six-circle diffractometer of SNBL (BM01A). Unfortunately, due to hardware problems during the experiment, this goal has not been met.

The crystal was mounted and centered after which the temperature was lowered using the Oxford cryostream of the beam line. Our target temperature was 100 K in order to allow direct comparison with previous laboratory data [1]. However, we could only reach 105 K – probably due to small amounts of humidity in the system. This not being a major problem we decided to perform measurements at this temperature. The diffraction profiles had noticeably widened with the lowering of temperature but not to a size leading to difficulties. The wavelength was 0.6 Å. Data were collected first in a hemisphere from 7.5° to 15° θ . The resulting 501 reflections were merged, after removing the ones for which a filter had been used (necessary as the correct value for the filter had not yet been determined), giving a merging R factor of 1.25%. This shows that the data quality was as high as we had hoped for. We continued with the hemisphere from 15° to 25° However, during the measurement of this data set, major hardware problems occurred.

The detector HV setting suddenly jumped from the set level to a higher one. This was rectified by restarting the control software and measurements continued. However, this behavior occurred several times; each time requiring that we stop the data collection and restart the software. Beyond 25° we measured only an octant in order to obtain at least some high angle data. At the end of our beam time, we had managed to measure data to θ =42.5°, but not a hemisphere as we had hoped for. We decided (having another experiment starting at the same beam line) to continue measuring to try to complete the data set. The day between the two experiments (machine dedicated time) was used to change the detector electronics and recalibrate the detector settings with a radioactive source. During this day, the temperature on the crystal started to rise, probably due to ice in the liquid N_2 . Therefore we heated the crystal to RT and dried the cryostream with dry N₂.The temperature was lowered at 200 K/h and the crystal recentered. The cell was the same as the previously found and the profiles similar in width. We started collecting data in the region where we, in the regular beam time, had only obtained an octant. Unfortunately, the φencoder of the diffractometer now gave problems, probably due to exposure to the cold nitrogen gas stream. We lowered the speed of the motor in the control software after which we could continue the data collection. The problem occurred again but could this time be solved by resetting the control computer. The measurements were interrupted by other apparent electronic problems (with the detector arm), that could each time be alleviated by rebooting the control computer.

Suddenly the beam intensity was reduced tremendously. By checking the optics of the beam line we found that the beam was now above the position of the crystal. The reason for this problem remains unknown. Therefore we decided to stop the data collection at this point, rectify the problem and start the next experiment.

In conclusion, the data collection was marred by many technical difficulties. We have not for this reason been able to collect sufficient data to perform a convincing charge density analysis even when using more time than initially foreseen and allotted. We wish to stress that the technical problems were no fault of the beam line staff and thank them for their very large effort in solving them.

References:

[1] P. Moeckli, D. Schwarzenbach, H.-B. Bürgi, J. Hauser & B. Delley *Acta Cryst*. B44, 636-645, 1988.