



	<b>Experiment title:</b> Development of a Gas Flow Thermostat Sample Cell with Control of Relative Humidity and Equipment for the	<b>Experiment number:</b> MI-483
<b>Beamline:</b> BM01A	<b>Date of experiment:</b> from: 30.05.01 07:00 to: 05.06.01 07:00	<b>Date of report:</b> 29.08.01
<b>Shifts:</b> 18	<b>Local contact(s):</b> Silvia Capelli, SNBL	<i>Received at ESRF:</i>
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### Report:

The purpose of the experiment is described in the title. The instrument for our experiment, a six-circle diffractometer, was not in proper working conditions following a commissioning period of new control programs. The first two days of our beamtime was used by SNBL staff to realign the instrument and redetermine zero-points. When we took over it was still not possible to obtain an orientation matrix or determine unit-cell parameters of acceptable quality for a test crystal or for our own sample crystals. Because of limitations in the existing (old) software we could neither centre the narrow intensity profiles from small crystals of rather high perfection which is a requirement for our experiment, nor could we carry out a proper data collection even with a larger specimen with broader profiles due to the imprecise description of the crystal orientation.

The magnitude of the latter problem is illustrated in the following: Even with more than 40 reflections for the calculation of an orientation matrix (OM) for the CaF<sub>2</sub> test crystal reflections at intermediate  $2\theta$  (40 - 50°) were as much as 0.12° off the calculated  $2\theta$  value in some sectors of reciprocal space. The OM for our sample crystal was of similar quality or less, typical FWHM-values as determined from  $\omega$  scans were about 0.003 - 0.004°. As a rule of thumb one needs some 5 - 6 steps over the peak FWHM in order to do a decent intensity collection. Assuming a typical FWHM = 0.0035° the step length should be no more than

about  $6 \cdot 10^{-4} \text{ }^\circ$ . The current programs have a maximum 124 steps for an  $\omega$  or  $\omega/2\theta$  scan. With this restriction a step length of  $6 \cdot 10^{-4} \text{ }^\circ$  permits a total maximum  $\omega$  scan of about  $0.075^\circ$  which is obviously completely inadequate in view of the indeterminacy of the crystal orientation!

We also want to include that there were frequent crashes of the PEP unit which further slowed down our work.

Because of the conditions described above, we did not achieve very much in terms of testing our sample cell. Nevertheless, some unsatisfactory details were found and will be corrected or changed. We do not blame the SNBL staff for the outcome as they took over problems created by the people responsible for the commissioning.

Under the circumstances we want to resubmit our application in the coming proposal round. In view of what happened it is also reasonable, that we, if granted beamtime in the next period, cover all our expenses for travel, food and accomodation in Grenoble.