

**Experiment title:**

Improving the reference model system for the study of diffraction phenomena in finite crystals – charge density study of squaric acid

Experiment**number:**

01-02-308

Beamline:

BM01A

Date of experiment:

from: 22/06/01, 07:00 to: 25/06/01, 23:00

Date of report:

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Shifts:

11

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Report:

The wavelength was calibrated to 0.7000(2) Å using the CaF₂ - standard crystal. Three crystals were tested on the diffractometer before a suitable (*i.e.* perfect enough) specimen was found. It seems as if "fresh" crystals mounted on site are superior to pre-mounted in this respect.

The beam size of the sample was 0.7 × 0.7 mm² with detector slit size of 0.6° × 0.6°. In addition, Huber anti-scatter slits were used, and also different (50, 100 and 150 μm) Cu-foils to attenuate the incoming beam. The peaks were narrow - typically in the order of FWHM ~ 0.006°. An orientation matrix was obtained after some difficulty (the pseudo tetragonal symmetry of the title compound represents a challenge in this respect).

The scan width was set to 0.14°. Some peaks did however migrate significantly within this scan-window, which caused "cut-off" problems in correctly determining the background. The reason for this migration remains somewhat uncertain, but is probably related to sentring/alignment procedures.

Approximately 10 500 reflections were collected on the perfect crystal. $(\sin \theta / \lambda)_{max} = 1.32 \text{ \AA}^{-1}$. Some pre-calculated three-beam cases were also checked (on purpose - in order to avoid serious intensity perturbations). In this respect it would have been very useful to have a software, in data collection mode, that enabled directly to measure reflections (from file) at a specific ψ -value. Some system-halts were encountered in connection with measuring the standard reflections.

Based on the measurement one weak (*i.e.* "kinematical") reflection in both the horizontal and vertical plane, the polarization factor was determined to 0.98.

The data-reduction program used (partly on-site) was `xd_red` (1.0) (Mathiesen, R.H. *J. Appl. Cryst.* (2001) **34**; Accepted).

A preliminary analysis indicates an $R_{merge}(obs) = 0.0342$ of equivalent reflections. Further analysis and refinement of the data set are in progress.

After completing the data-collection a test was made in order to increase the mosaicity of the sample. It was shock-cooled by immersing it several times into liquid Nitrogen. This was successful: the crystal remained on the glass-spike, and the reflections were easy to re-find. The profiles now were significantly broader (an increase in FWHM of about 3 times), but still homogeneous - *e.g.*: For reflection $0\bar{2}0$, $FWHM_{Perfect} = 0.0079^\circ$ and $FWHM_{Imperfect} = 0.0203^\circ$; whereas for reflection $\bar{2}\bar{1}1$, $FWHM_{Perfect} = 0.0047^\circ$ and $FWHM_{Imperfect} = 0.0135^\circ$.

A small number of reflections (about 130) were measured for the imperfect crystal state. The preliminary analysis indicates that a larger set should have been collected. A substantial fraction of the (also moderately strong) reflections was still influenced by coherent scattering effects.

It now thus appears very attractive to collect two full data sets on the same sample: one dynamical (perfect crystal) and one kinematical (imperfect crystal, after quenching).