ESRF

ESRF	**Experiment title:** Investigations on the dynamics of [2+2] photo-polymerisations	**Experiment number**: CH 1550
Beamline:	**Date of experiment**: from: 03/10/03 to: 07/10/03	**Date of report**: 31/08/04
Shifts: 15	**Local contact(s)**: M. Wulff	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

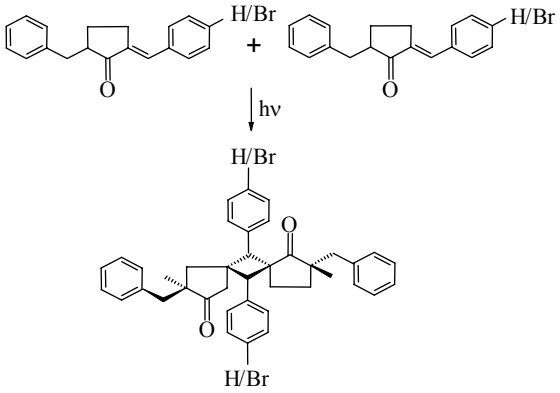
* Simone Techert, MPibpc – Structural dynamics of (Bio)chemical Systems, 37077 Goettingen, Germany

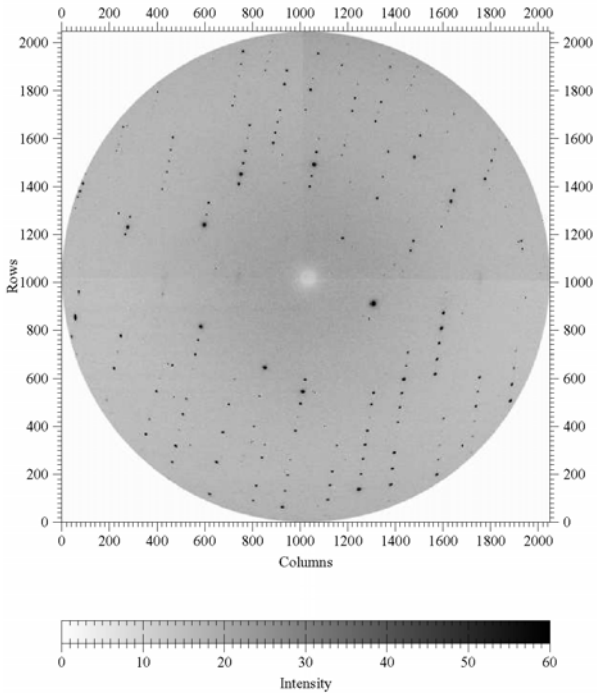
* Jav Davaasambuu, MPibpc – Structural dynamics of (Bio)chemical Systems, 37077 Goettingen, Germany

Report:

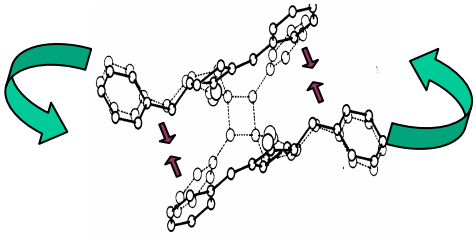
In experiment No **CH-1550** we investigated the topochemcial [2+2] photoreaction of the spiro-compound 2-benzyl-5-benzylidenecyclopentanone (BBCP) to its dimeric product state. As method, picosecond time-resolved x-ray oscillation diffractometry was applied (at ID09B). From a technical point of view, the run was very succesfull. We could follow the photo-induced transformation in the crystals on the long (hours) and on the short (picoseconds) time scale. Figure 1 summarises the proposed dimerisation process and a typical single crystal diffraction pattern (under the experimental conditions of **CH-1550** (see description in Figure 1). Figure 2, left shows the intensity changes on the picosecond time scale, which essentially coincide with results found for the dimerisation investigated in **CH-1227** for p-formyl-trans-cinnamic acid. In Figure 2, right, the cell parameter changes as a function of the over-all transformation time of the crystal are presented. The crystal data were indexed with the XDS software. The monomer (reactant) and dimer (product) states were already refined using the SHELX97 software and we are currently developing a software which enables the refinement of the third unknown intermediate structure which has been found on the picosecond time scale. The data were already presented at several conferences and will be published as soon as the data refinement is finished – a publication is already in preparation.

Single Crystal – Single Crystal Transformation





- **composite diagram along [001]**



P nma: a = 31.302 Å, b = 10.784 Å, c = 8.698 Å, $\lambda_{X\text{-}ray}$ = 0.734 Å (U17), 5 mm gap, rep.frequency = 897 Hz, dist (crystal, det) = 100 mm, exp.t = 20 s, $\Delta\Phi$ = 40°, λ_{laser} = 267 nm, p_{laser} = 2-5 mW @ 897 Hz, $d_{laserspot}$ = 0.500 mm

Figure 1: The proposed dimerisation process of BBCP and a typical single crystal diffraction pattern.

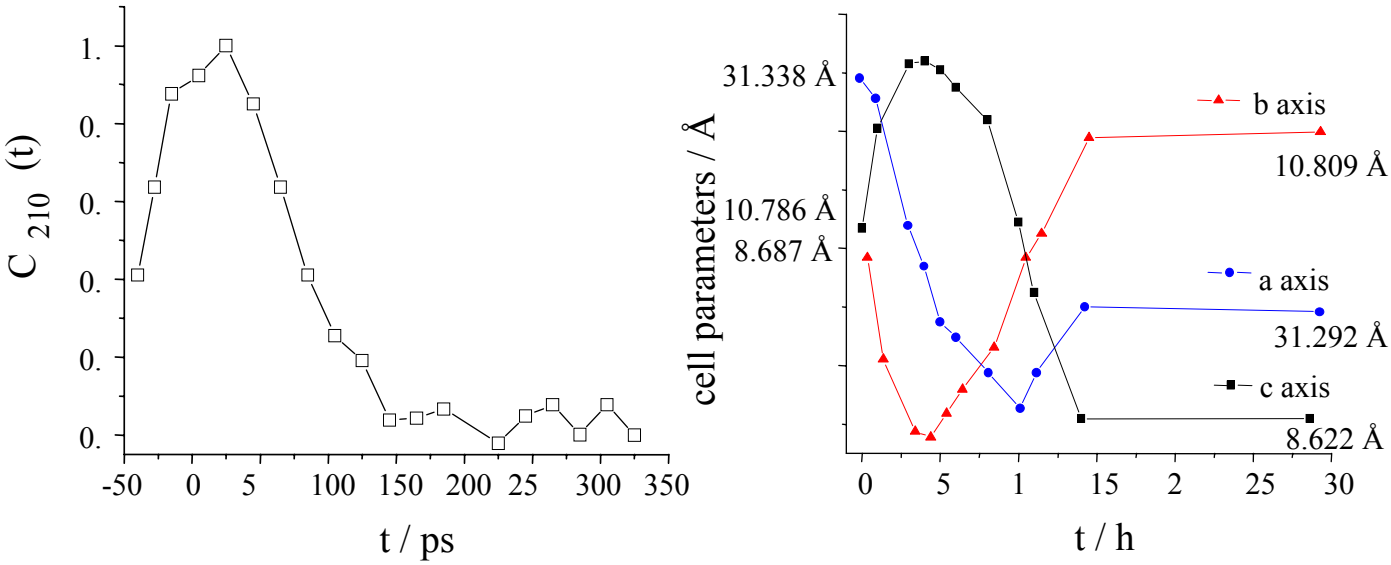


Figure 2: Left: intensity changes of the [210] Bragg diffraction peak on the picosecond time scale. Right: The cell parameter changes as a function of the over-all transformation time of the crystal.