

We developed a combined imaging and dynamic light scattering (DLS) system for routine measurements in droplets of multi-well plates as well as in gel tubes used for protein crystallization. The system is of high value for rapid identification of good crystallization conditions. Today automated methods to crystallize macromolecules are widely used and can easily generate thousands of crystallization droplets. Nevertheless the evaluation of crystallization experiments to find optimal growth conditions remains a bottleneck. Therefore we have investigated methods to improve the process of evaluating results and finding crystal growth conditions. One method is DLS, the second is the use of combined white/UV illumination for determination of whether crystal-like objects are biomolecular and identification of crystals in crystallisation set-ups. Up to now, it has been impossible to determine the particle size directly in protein solution droplets because of size and configuration constraints. We have developed a CCD camera-based imaging instrument and combined a laser source and a detector to perform DLS measurements in situ. The plate-screening system allows to monitor and evaluate the entire crystallization process in an automated way. For example the stages of nucleation and the progress of crystal growth without disrupting the course of equilibration can be analyzed. The data provide information to understand in greater detail the process of crystal initiation and growth and will allow further optimisation, thereby leading to better crystals. Finally we will also describe a method to support the identification of protein crystals, exploiting the fact that most proteins and other biomolecules fluoresce when illuminated with UV light.

Keywords: crystallization process, dynamic light scattering, instrumentation and software

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Watching photo-induced dynamics with picosecond time-resolved X-ray diffraction

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Picosecond time-resolved X-ray experiments using synchrotron radiation sources are becoming general and powerful tools to explore structural dynamics of condensed matters in material and biological sciences. The beam line NW14A is a newly constructed undulator beam line for 100-ps time-resolved X-ray experiments at the Photon Factory Advanced Ring, KEK [1]. This beam line was designed to conduct a wide variety of time-resolved X-ray measurements, such as time-resolved X-ray diffraction, scattering and absorption. The beam line has been operational for two years, and current status of the beam line and scientific activities utilizing shock-induced lattice deformation [2] will be presented.

Reference:

- [1] Nozawa et al. (2007) *J. Synchrotron Rad.* 14, 313.
[2] Ichiyanagi et al. (2007) *Appl. Phys. Lett.* 91, 231918.

Keywords: time-resolved diffraction, synchrotron X-ray instrumentation, structural dynamics

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Photo-crystallographic studies of dimerisation processes: From picoseconds to hours transformation

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Though photo-induced solid state reactions are known since over hundred years, the structural mechanism underlying photo-induced solid state reactions have not been explored yet to a sufficient stage. In the following contribution we will present photo-crystallographic studies on [2+2] photodimerisation reactions of cinnamic acid derivatives. Common for all the investigated systems is the homogeneous character of the reaction (single crystal to single crystal transformation). We will report on high resolution photo-crystallographic studies / electron density studies monitoring the structural mechanism of the seconds to hours photodimerisation processes - and how to speed up these processes which can then only be investigated by ultrafast time-resolved crystallography (picosecond time resolution).

- [1] G. Busse, Th. Tschentscher, A. Plech, M. Wulff, B. Frederichs & S. Techert, *Faraday Discuss.* 122 105117 (2002)
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[3] J. Davaasambuu, G. Busse, S. Techert, *J. Phys. Chem. A* 110 (Juergen Troe Festschrift), 3261-3265 (2006).

Keywords: photodimerization, time-resolved crystallography, organic crystals

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Picosecond and femtosecond X-ray absorption studies of the photoinduced spin change in Fe-complexes

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Because of close-lying low spin (LS) singlet and high spin (HS) quintet states, molecular FeII-complexes are particularly interesting in relation to the spin cross-over (SCO) phenomenon, where conversion from the LS ground state to the HS excited state (or the reverse) can be induced either by temperature, pressure or light. Several steady-state and ultrafast studies of the light-induced SCO have been carried out by optical spectroscopies, concluding that population is funneled to the HS state in < 1 ps, through intermediate singlet and triplet metal-centred (MC) states upon excitation into the singlet metal-to-ligand-charge-transfer state. However, the pathways of this cascade remain unclear, in part because the intermediate and final states are optically silent. In addition, depending on the ligand, the lifetime of the HS state spans several decades, and the origin of this large variation is unclear. In [FeII(bpy)₃]²⁺ at room temperature, the HS lifetime is 660 ps. Using X-ray absorption spectroscopy (XAS) studies with 50-100 ps resolution, we found that the Fe-N bond elongates by 0.2 Å in the HS state. This elongation is similar in Fe-complexes having much longer HS lifetimes, leading us to conclude that the structure of the HS spin does not determine its lifetime, but rather it is its energetics that does. Our results show important spectral changes between the LS and HS states in the X-ray Absorption Near-Edge Structure (XANES) of the Fe K-edge.