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LOW-TEMPERATURE PHASE TRANSITION AND CRYSTAL STRUCTURES OF TWO BICYCLIC ORGANIC MOLECULES

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The crystal structures of two small organic molecules 7-oxabicyclo[2.2.1]heptane ($C_6H_{10}O$) and norcamphor ($C_7H_{10}O$) have been investigated using synchrotron X-ray powder diffraction at BM16 at the ESRF. The temperature dependence of the 7-oxabicyclo[2.2.1]heptane and norcamphor diffraction pattern reveals different solid-state phase transitions. For the 7-oxabicyclo[2.2.1]heptane phase I is between 251 K and 234 K, phase II 234 K-192 K, phase III 191 K-185 K, phase IV below 182 K. For the norcamphor we observed phases between 288 K and 165 K, 164 K-154 K, and below 146 K. The crystal structures in the low-temperature phases of the 7-oxabicyclo[2.2.1]heptane have been studied at 50 K and 185 K. At 50 K the compound is found to be monoclinic, space group $P2_1/a$, with a = 24.041 Å, b = 10.259 Å, c = 13.637 Å, β = 102.52° and Z = 24. The crystal structures in the low-temperature phases of the Norcamphor have been studied at 35 K and 185K. At 35K the compound is found to be monoclinic, space group $C2_1/c$, with a = 18.443Å, b = 12.285 Å, c = 13.685 Å, β= 90.96° and Z = 16.

Keywords: AB INITIO POWDER DIFFRACTION SOLUTION, MOLECULAR COMPOUNDS, PHASE TRANSITION

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THE STRUCTURE OF ENDOTHELIAL PROTEIN C RECEPTOR IDENTIFIES THE BINDING SITES FOR PROTEIN C AND REVEALS A BOUND LIPID MOLECULE

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Endothelial protein C receptor (EPCR) binds to protein C (PC) and facilitates PC activation by membrane associated thrombin-thrombomodulin complex. The 3-D structure of EPCR is similar to the structures of MHC class1 molecules. In EPCR the counterpart of the MHC/CD1 binding site, created by a1 and a2 domains and b-pleated sheet is extremely hydrophobic. A lipid is bound within this groove in EPCR. The fatty acid chains are nearly covered whereas the headgroup of lipid is solvent exposed. The vitamin K dependent Gla domain of protein C binds near the end of the groove and does not contact the lipid. The structure illustrates a unique ligand binding site for protein C on this class of proteins plus a novel lipid moiety. Together with the ability to bind proteinase 3 EPCR appears to serve as a link between inflammation and coagulation.

Keywords: COAGULATION, EPCR, PC

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SUB-PICOSECOND X-RAY SOURCES AT SLAC, AND THEIR CRYSTALLOGRAPHIC APPLICATIONS

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The high-energy electron linear accelerator at the Stanford Linear Accelerator Center (SLAC) is being modified to serve as a source of very bright, sub-picosecond pulses of hard x-rays. Within the next year, the Sub-Picosecond Photon Source (SPPS) will begin producing conventional synchrotron radiation from electron pulses with FWHM < 100 fs. After a few years, this source will be displaced by the much more elaborate Linac Coherent Light Source (LCLS), an x-ray free-electron laser producing extremely intense, bright pulses with duration of a few hundred fs or less. Combining the time resolution of ultrafast lasers with the atomic resolution of x-rays, these sources will provide unique capabilities for performing experiments of interest to chemists, biologists, and condensed-matter physicists.

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APPLICATION OF SINGULAR VALUE DECOMPOSITION TO TIME-RESOLVED X-RAY DATA; SIMULATIONS AND EXPERIMENTS

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The applicability of singular value decomposition (SVD) to the analysis of time-resolved X-ray data was successfully demonstrated using simulated time-dependent difference maps. Random noise of varying levels in the difference structure factor amplitudes, different extents of reaction-initiation and different numbers of time-points were used to cover a range of realistic experimental conditions. A method to reduce the random noise in the difference maps was developed and called SVD-flattening. The identification and the fit of a kinetic mechanism were successful and the time-independent structures of the intermediates could be recovered. These SVD-driven methods were applied to 15 experimental time-dependent difference maps, which were calculated with the help of time-dependent Laue structure amplitudes collected from crystals of Photoactive Yellow Protein (PYP) after a single (7ns) laser flash at beamline 14ID-B, BioCARS, APS, USA. After SVD-flattening the largely improved maps were subjected to SVD and the significant right singular vectors (rSV) inspected. Three relaxation times were determined. Candidate mechanisms, which account for the number of relaxation times were fit to the rSV. From this, the time-independent difference maps of the intermediates were extracted. The atomic structures of three intermediates at times from 5 \ms to 100 ms were determined. The results are used for an explicit description of the photocycle of PYP at longer times.

Keywords: TIME-RESOLVED CRYSTALLOGRAPHY CHEMICAL KINETIC MECHANIS PHOTO ACTIVE YELLOW PROTEIN