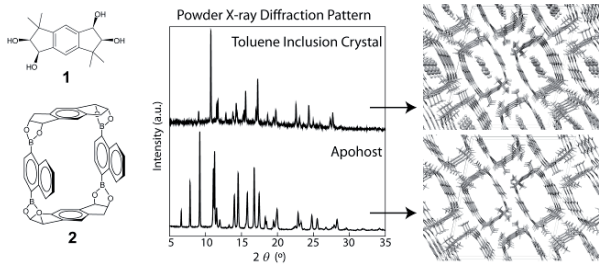
study, the apohost of **2** was determined from the laboratory powder X-ray diffraction data and the structural change by guest sorption and release processes were investigated.

The powder X-ray diffraction data of the toluene inclusion crystal of **2** and the apohost crystal of **2**, which was obtained by heating of the toluene inclusion crystal, are significantly different as shown in the figure. However, interestingly, the apohost structure, determined from the laboratory powder X-ray diffraction data, was found to retain its crystal packing even after the guest release. The apohost has one dimensional stacking of **2** along the *b*-axis forming the one dimensional guest free tunnel. This tunnel is expected to absorb the guest molecules easily and, in fact, the apohost crystal readily absorbs the toluene molecules, when the toluene vapor was applied to the solid apohost, and it transforms into the toluene inclusion crystal within 20 min.



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Keywords: ab-initio powder structure determination, solid-state transformation, macrocyclic compound

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Highly automated synchrotron beamline dedicated to SAXS on proteins in solution

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Introduction of the new generation sample changer on the recently rebuilt BioSAXS ID14-3 beamline at the ESRF together with the implementation of 1M Pilatus detector have sensibly boosted its high-throughput capacities. The sample changer developed in collaboration between the ESRF and the EMBL (Grenoble and Hamburg outstations) automates the entire cycle of sample loading-unloading-cell cleaning and can hold up to several hundred of samples in micro-plates, eppendorf strips (PCR) or tubes. Thermal control, smart pipetting and sample positioning together with other features allow to run completely automated data collection without any user intervention through the dedicated beamline software, BsxCuBE. The user just needs to enter sample information (name, concentration, location in sample changer, etc.) and collection parameters (exposure time, temperature, flow during exposure, etc.). Afterwards the script performs data collection in the most economical and safe manner, processes 1D curves and filters them according to the radiation damage. It is followed by automated processing pipeline (developed by EMBL Hamburg) which analyses 1D curves and gives structural properties of the proteins (molecular sizes and *ab-initio* models). Reliable and simple-to-use sample environment together with robust software allow to perform easily and efficiently the SAXS experiments even by non-experienced users.

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Keywords: SAXS-1, Automation-2, Biomacromolecules-3

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An automated sample environment for biological solution scattering experiments at 3rd generation synchrotrons

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Small-angle X-ray scattering for macromolecules in solution is widely used in structural biology to complement high-resolution structure determination obtained by x-ray crystallography or NMR. In the context of third-generation synchrotron sources, this increasing interest leads to developments of automated sample environments. Based on the background acquired at the EMBL Hamburg X33 beamline, a collaboration has been established between the EMBL and the ESRF to develop a system able to expose automatically to x-rays micro-volumes of solution with minimum time overhead. The key concept to reach this objective was to minimize length of the fluidic path, using an architecture where the exposure cell is connected to a fixed pipetting needle with very short tubing, and where the sample wells are moved to the needle. With this system, several hundred samples stored at controlled temperature in strip wells or in SBS Microplates can be automatically loaded for exposure to x-rays in a vacuum mounted and temperature controlled glass capillary. Volumes of solution down to 5 µl can be reliably exposed to an x-rays beam in flow mode. After exposure, the fluid path is cleaned and dried automatically in less than 20 seconds. Several thousand experiments have been carried out at the ESRF ID14-3 beamline, the EMBL Doris X33 beamline and the EMBL@PETRA-III BioSAXS beamline. The liquid handling features of the machine associated with an in-line spectrometer allows for sample concentration measurement, dilutions or additions. Efficient use of the small and intense x-ray beams available at 3rd generation synchrotron is now possible using this new automated sample environment, in particular when associated with the last generation of fast counting silicon pixel detectors.

Keywords: SAXS, high throughput, sample changer

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Structural changes in DL-serine under hydrostatic pressure up to 4.3 GPa

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The studies of molecular crystals at non-ambient conditions (low temperatures and high pressures) help to understand intermolecular interactions and their role in the formation of crystal structures and

in their response to external actions. Crystals of amino acids are of special interest in this respect because the structure-forming units in these crystals are similar to those in the biopolymers and can be used as biomimetics. The data on response of intermolecular interactions to changes in temperature and pressure in these systems may be considered also for studying properties of complicated biomolecules such as proteins. In addition, crystalline amino acids are of interest for their ferroelectric, piezoelectrical, and nonlinear optical properties [1, 2].

In this work structural changes in DL-serine were studied in the pressure range 0.2 – 4.3 GPa. An Almax-Boehler diamond anvil cell was used to create hydrostatic pressure. Single-crystal X-ray diffraction and polarized Raman spectroscopy were selected for investigation of these crystals under high pressure. This combination of spectroscopy and diffraction made it possible to follow the changes in the individual hydrogen bonds with pressure in many details. The two techniques give complementary information: diffraction experiments provide data on the changes in atomic coordinates averaged over space and time; a detailed spectroscopy study can help in understanding the dynamic processes related to pressure changes (compression of individual intermolecular hydrogen bonds, rotation of molecules as a whole and of the individual molecular fragments).

Anisotropic structural compression of crystalline DL-serine was carefully studied in [3]; no phase transitions were detected up to 8.6 GPa. At the same time, as has been shown in the present contribution, the lengths of intermolecular hydrogen bonds linking amino-groups and serine side chains in the crystal structure depend non-linearly and non-monotonic on pressure in the range between 0.4 and 1.5 GPa. This is an interesting and unusual phenomenon. Another interesting observation is the rotation of carboxylic groups of serine with increasing pressure. The structure changes manifest themselves in the low-wavenumber range of the Raman spectra and can be detected by analyzing the redistribution of intensities and the shifts of appropriate modes.

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Keywords: high pressure, amino acids, intermolecular interactions

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Pressure enhancement of CH···O interactions in simple ethers

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CH···O interactions attract attention of crystallographers, chemists and biochemists, although their importance was generally accepted only in the last years of 20th century [1]. Despite their weakness (typical potential energy < 4 kJ/mol), the topology of electron density redistribution accompanying the H-bond formation is similar to that of conventional hydrogen bonds [2]. Spectroscopic studies revealed that under high pressure van der Waals CH···O interactions are enhanced and convert into hydrogen bonds [3]. We have recently supported this view with structural data.

Two simple ethers, tetrahydrofuran (THF) and its open-ring analogue diethyl ether (DE) have been chosen for the experiments to avoid blurring CH···O interactions by stronger hydrogen bonds or Coulombic forces. For THF both isochoric crystallization at 2.25, 3.26, and 3.80 GPa and isobaric freezing at ambient pressure lead to a monoclinic phase, space group *C*2/*c* [4]. The CH···O interactions are the strongest intermolecular forces in the THF molecular crystal, and the hierarchy of the CH···O distances correlates with the electrostatic potential distribution on a molecular surface and with their alignment along the lone pair direction. In THF the exposed oxygen atom is involved in six short CH···O contacts, what is not accounted for strong hydrogen bonds. In compressed THF the CH···O contacts acquire the features of hydrogen bonds, and therefore the structure is stable to at least 3.80 GPa.

At low temperature DE crystallizes in the space group $P2_12_12_1$, $Z = 8$ [5]. The structure is fairly loosely packed, and each oxygen atom of two symmetry-independent molecules is involved in three CH···O contacts shorter than 3 Å. At high pressure DE undergoes phase transitions leading to the structures with CH···O contacts resembling hydrogen bonds in THF. The energetic cost of the transformation involves conformational changes facilitating the access to oxygen atom and hence the increased number of CH···O contacts per molecule.

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Keywords: weak interactions, hydrogen bonds, high-pressure crystallography

MS.12.3

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Structure study of IPMDH from piezosensitive and piezophilic *Shewanella* species

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The organisms living in deep sea such as the Mariana Trench must be adapted to extremely high-pressure environment. For example, protein 3-isopropylmalate dehydrogenase (IPMDH) from deep-sea bacteria *Shewanella benthica* DB21MT2 (SbIPMDH) remains active under such extreme conditions, while that from land bacteria *S. oneidensis* MR-1 (SoIPMDH) becomes inactivated [1]. In order to unravel the differences existing between these two IPMDHs, we are here attempting to solve their structures by a high-pressure protein crystallography (HPPX) method using a diamond-anvil cell (DAC).

To make HPPX measurements possible at the beamline PF AR-NW12A, we have modified several equipments such as the goniometer head to accept our DAC. Using such settings, the crystal structures of SoIPMDH- and SbIPMDH-IPM complexes have been determined at about 2 Å resolution under multiple pressures and up to several hundreds MPa.

Pressure dependence of SoIPMDH- and SbIPMDH-IPM complexes is nearly uniform up to 300 MPa, and the compressibilities of the two