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

proteins are 0.097 GPa^{-1} and 0.076 GPa^{-1}, respectively. It may imply that SoIPMDH, which is from land bacteria, is more sensitive to pressure than SbIPMDH from extremophile.

The SoIPMDH structure shows a dimer that presents an internal hydrophobic cavity surrounded by Pro120, Lue121, Ile125, Lue232 and Lue258 at the dimer interface. Two structurally functional water molecules appeared in the cavity at a pressure of 470 MPa, while no such water molecule could be observed under atmospheric pressure. Previous published works assume that the pressure denaturation of proteins is induced by water penetration into the hydrophobic interior of proteins. Although concrete data are lacking, several theoretical and simulation studies support this theory [2]. The present observation is a direct evidence of the fact that water molecules do penetrate into the inner cavities of proteins under high pressure.

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Keywords: high-pressure protein crystallography, high-pressure adaptation, pressure denaturation

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Automation and remote control of the MX beamlines at the canadian light source

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The Canadian Light Source (CLS) is a 2.9 GeV national synchrotron radiation facility located on the University of Saskatchewan campus in Saskatoon. The Canadian Macromolecular Crystallography Facility (CMCF) is composed of two beamlines, the small-gap in-vacuum undulator illuminated beamline 08ID-1 and a second bending magnet beamline, 08B1-1 [1]. Beamlines are equipped with very robust end-stations including on-axis visualization systems and Rayonix 300 CCD series detectors. They are each complemented with a Stanford automounter (SAM) which accepts SSRL type cassettes or universal pucks. MxDC, a beamline control system developed in-house, is integrated with a data processing module, AutoProcess, allowing full automation of data collection and data processing with minimal human intervention [2]. AutoProcess is based on XDS, Pointless, CCP4 and BEST packages. The system also allows remote control of experiments through interaction with a Laboratory Information Management System (LIMS) that was developed at the facility.

The CMCF provides service to more than 60 Principal Investigators in Canada and the United States. Up to 25% of the beam time is devoted to commercial users and the general user program is guaranteed up to 55% of the useful beam time and is run under a peer-review proposal system. The CMCF staff provides "Mail-In" crystallography service to the users with the highest scored proposals.

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New possibilities offered to crystallography by 6-axis robotic-arm based systems

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CATS and G-Rob systems were developed on protein crystallography beamline FIP-BM30A at the ESRF. CATS [1] is a high throughput reliable sample changer currently operating on the major synchrotrons worldwide (BESSY, SLS, DLS, APS, …) and commercialized by Irelec Company (www.irelec-alcen.com). G-Rob, also a 6-axis robotic arm based system, is a fully integrated device for crystallography beamlines (commercialized by Irelec) and laboratories (commercialized by NatX-ray company, www.natx-ray.com). G-Rob is an "all in one" system, since it integrates the following functions:

- sample changer,
- goniometer for frozen samples, capillaries, … [2],
- crystallization plates/micro-chips screening for *in situ* analysis of diffraction condition and data collection [3],
- beam monitoring.

G-Rob provides unique features. It is automated: thanks to its tool changer, it goes automatically from one application to another. CATS and G-Rob are also highly flexible: if a new application or a new sample format emerges in the community, a new tool can be designed to implement it. They are highly reliable systems, based on well-known, industrial quality equipments, with reduced maintenance.

Several G-Rob systems, both at synchrotrons or as laboratories in-house systems, are now available (ESRF, EPFL, …). The first system, in use on beamline FIP-BM30A, was made available to the research community in 2005 and up to now, users have expressed an unprecedented high degree of satisfaction. The crystallization plates screening capability for example appears to be a precious tool in several cases (crystals too small to be fished, or too fragile, of when there is no good cryoprotectant).

Several results obtained on FIP-BM30A are presented, such as *in situ* screening of membrane proteins, ribosome, high pressure protein diffraction, etc. Recent experiments demonstrated also the possibility of the automated structural screening for the Fragment Based Drug Design strategy: the same crystal was reproduced in presence of a library of fragments. Systematic *in situ* data collection has shown some of the fragments present in the active site, without having to manipulate the crystals individually. *In situ* data collection was also used recently to solve the structure of entire viruses (D Stuart et al.). All these experiments take advantage of a new sitting drop plate specially designed for *in situ* X-ray analysis. This plate was developed in collaboration between beamline FIP-BM30A at the ESRF (Grenoble, France) and Greiner BioOne. Movies are available on www.natx-ray.com

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Automated characterization of nanotube membranes of large Size By X-Ray scattering

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