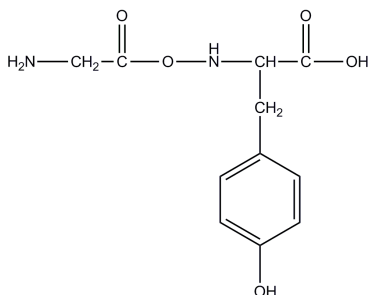


from powder diffraction data.

Glycyl-L-tyrosine is known to crystallize as dihydrate from an aqueous-methanol solution [1]. Recently, we obtained the trihydrate from an aqueous solution, and carried out single-crystal structure analysis [2]. Humidity- and temperature-dependent phase transitions of both hydrates were examined by powder X-ray diffraction analysis. The dihydrate is stable at room temperature in the relative humidity (rh) range from 80% to 0% and the structural transition proceeds around 50 °C at 0% rh. On the other hand, the trihydrate loses a part of crystal water at 0% rh, and further dehydrates around 80 °C.

At the first step, we have successfully determined the crystal structure of the dihydrate from powder diffraction data. The structure coincides with that determined by single-crystal structure analysis. In addition the cell parameters of the partly dehydrated state of the trihydrate at 0% rh have been determined and structure analysis from powder diffraction data is in progress.



Gly-L-Tyr

[1] P.M. Cotrait et al., *Acta Cryst. B* **1974**, *30*, 1024-1028. [2] T. Kiyotani et al., *CrSJ Annual Meeting 2010*, **2010**, OB-II-03.

Keywords: Glycyl-L-tyrosine, powder diffraction, hydrate

MS10.P08

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Ab-initio Structure determination of a new phosphodiesterase enzyme inhibitor

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Single crystal X-ray diffraction analysis is the usual way in the determination of structure of crystalline solids. Nowadays, however, many materials such as pharmaceutical compounds cannot be easily crystallized and exist only as powders. In these cases powder diffraction arises as an alternative. Synchrotron radiation is the most powerful source of X-rays currently available for powder diffraction experiments. Narrow peaks, accurate peak positions and excellent signal to background ratios means that synchrotron data are ideal for structure solution [1].

In this study, we focused on structure determination of 6-(4-(4-methylpiperazin-1-yl)-4-oxobutoxy)-4-methylquinolin-2(1H)-one as a phosphodiesterase (PDE) enzymes inhibitor which improve cardiac contractility and may be used in congestive heart failure (CHF) which is a major cause of death in patients with heart disease [2].

[1] J.K. Harper, J.A. Doebbler, E. Jacques, D.M. Grant, R.B. Von Dreele, *J. Am. Chem. Soc.* **2010**, *132*, 2928-2937. [2] S.M. Seyedi, Z. Jafari, N. Attaran, H. Sadeghian, M.R. Saberi, M.M. Riazi, *Bioorg. Med. Chem.* **2009**, *17*, 1614-1617.

Keywords: Synchrotron, Powder Diffraction, Enzyme Inhibitor.

MS10.P09

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Powder structures of two pharmaceutically interesting alkylaminobisphosphonates

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Bisphosphonates are chemically stable molecules having resistance to enzymatic hydrolysis due to their P-C-P backbone.¹ At first they were widely utilized by industry as detergents, corrosion inhibitors and tartar preventer in tooth paste. More recently their therapeutic properties were discovered in form of ability to inhibit bone resorption, thereby enabling their use for treating various bone diseases (osteoporosis, skeletal metastases from solid tumours, hypercalcaemia of malignancy, multiple myeloma and Paget's disease). Bisphosphonates are widely studied (mostly in health-related aspects), as nearly 6000 articles have appeared since year 2000 alone, of which only around 50 deals with their structural chemistry. Therefore in this study, *ab initio* powder structure determination of two zwitterionic, alkylamino-bisphosphonates having amino group in the ω position of the carbon chain, will be presented and compared with the existing crystal structures found in the literature. The presented compounds were synthesized according to the method reported by Kieczykowski *et al.*²

[1] K.R. Williams, *J. Chem. Educ.*, **2004**, *81* (10), p 1406, [2] G. Kieczykowski, R. Jobson, D. Melillo, D. Reinhold, V. Grenda, I. Shinkai, *J. Org. Chem.*, **1995**, *60*, 8310.

Keywords: alkylaminobisphosphonates, X-ray powder diffraction, pharmaceutical

MS10.P10

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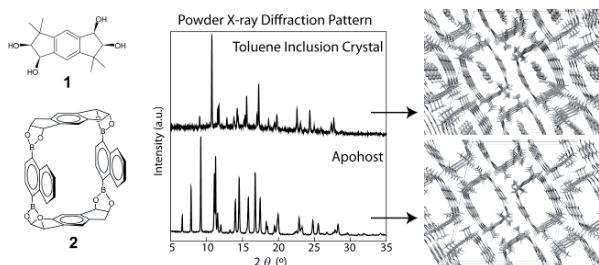
Crystal Structural change by guest sorption/release processes of the macrocyclic boronic ester

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Macrocyclic compounds attract many interest because they can be used as a host molecule to absorb and store several types of guest molecules. Recently, diboronic acid and racemic tetrol (**1**) are found to form a self-assembled macrocyclic boronic ester in the presence of appropriate guest molecules.[1] Among this type of compounds, the toluene inclusion crystal of the macrocyclic boronic ester (**2**) was found to form one dimensional stacking of **2** along the *b*-axis with an infinite one dimensional toluene channel. It is interesting to explore the crystal structure of the guest-free apohost, in order to investigate whether the crystal can retain its one dimensional tunnel structure, which has enough size to absorb guest molecules, after the guest release. However, **2** tends to incorporate guest molecules during the recrystallization processes and the apohost crystal can only be obtained by guest release process, such as heating of the sample, which usually results to form micro-crystalline powders. Obviously, the crystal structure determination from powder X-ray diffraction data is an essential tool to establish the crystal structure of the apohost. In this

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.



[1] N. Iwasawa, H. Takahagi, *J. Am. Chem. Soc.*, **2007**, *129*, 7754-7755

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

MS11.P01

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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.

[1] P. Pernot et al, *Journal of Physics: Conference Series* **2010**, 247 [2] J.

Santiago, F. Dupeux, A. Round, R. Antoni, S-Y. Park, M. Jamin, S.R. Cutler, P.L. Rodriguez, J.A. Márquez, *Nature* **2009**, *462*, 665-668.

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

MS11.P02

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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 μ l 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

MS12.P01

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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