

indexing programs: ITO, TREOR90, DICVOL, KOHL, TAUP, FJZN, and LZON. The atomic coordinates obtained by Monte Carlo methods were used to initialize the Rietveld refinements, which were performed using the FULLPROF program [2].

In both cases, NA ligand "organizes" the packing by strong amide-amide hydrogen bonds forming a 3D network with four complexes around each other.

[1] T. Roisnel and J. Rodríguez-Carvajal, *Mater. Sci. Forum.* **2001**, *118*, 378 [2] J. Rodríguez-Carvajal, FULLPROF, V. 1.9c. LLB, CEA/Saclay, France, **2001**.

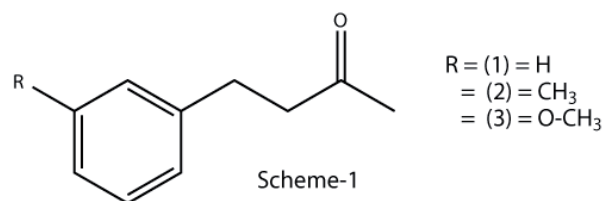
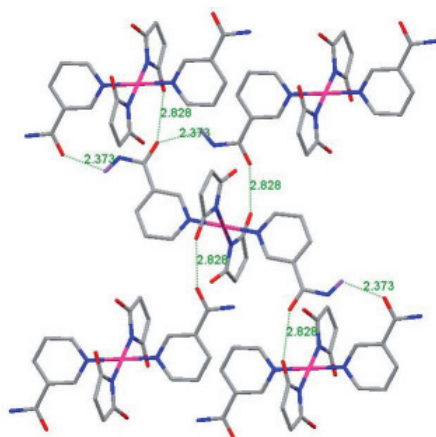
Keywords: synchrotron, rietveld, palladium

MS10.P05

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Crystal structure from X-ray powder diffraction data with $Z'=2$
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Phenylpropionic acid derivatives exhibit a strong binding ability to peroxidases, which catalyze the oxidation of a number of organic and inorganic substrates. Structural studies of phenylpropionic acid derivatives carried out by our group revealed that the crystal structures are predominantly built up by carboxylic acid dimers forming $R_2^2(8)$ synthons. In the course of our ongoing program of structural characterization of organic compounds from X-ray powder diffraction data, we came across 3-phenylpropionic acid (**1**), 3-(3-methylphenyl)propanoic acid (**2**) and 3-(3-methoxyphenyl)propanoic acid (**3**) [scheme 1]. Crystal structures of three compounds have been solved from laboratory X-ray powder diffraction data using direct-space approaches and refined by Rietveld method using the program GSAS package with an EXPGUI interface. Hirshfeld surface analysis of compounds were also performed to visualize and discriminate the features of molecular interactions in the compounds. The essential difference between the 3-phenylpropionic acid (**1**) and the compounds **2** and **3** is the presence of different substituent at the 2 positions (methyl group in **2** and methoxy in **3**). The asymmetric unit of compounds **1-3** contains two molecules ($Z'=2$). The carboxylic acid group in **1-3** forms O-H...O hydrogen bonded dimer with O1...O2 distances of 2.683(5) - 2.902(3) Å in an $R_2^2(8)$ graph-set motif. The interconnection of $R_2^2(8)$ rings via C-H...O hydrogen bonds generates a ladder-like one dimensional architecture based on fused $R_2^2(14)$ and $R_4^4(20)$ synthons in **1**. Pairs of molecules forming $R_2^2(8)$ rings are further connected through intermolecular C-H...O hydrogen bonds to generate three dimensional structures in compound **2**. In **3**, however, the interconnection of $R_2^2(8)$ rings via C-H...O hydrogen bonds generates a two dimensional architecture based on edge fused $R_4^4(32)R_2^2(14)$ synthon. Although crystal structures of molecular compounds can now be accomplished from X-ray powder diffraction data, structure solution of compounds with $Z'>1$ is not very common.



Keywords: Powder diffraction, Z prime>1, Hirshfeld surface.

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TALP: A L.S. structure solution program of molecular compounds from powder data

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In recent years, global optimization methods are increasingly important in crystal structure determination of molecular compounds from powder diffraction data. Most procedures make extensive use of simulated annealing or other more or less exotic optimization algorithms [1]. Here a general easy-to-implement method based on the well-known L.S. algorithm is presented which allows solving crystal structures of a wide variety of relatively complex compounds. TALP structure solution program is based on a global optimization technique which combines a random/incremental iteration strategy for positioning the molecule with an ultra-fast Rietveld refinement. The geometry of the molecule is fixed by restraints. Unlike other structure solution programs, atom coordinates but not torsion angles are refined. Torsion angles are used only to create initial pseudo-random models.

TALP has been tested on powder diffraction data of molecular compounds of different complexity. For simple compounds with cell volumes up to 1400 Å³ and 4 torsion angles, the solution is obtained straightforwardly. More complex structures with e.g. cell volumes up to 3500 Å³, up to 8 torsion angles, and two independent molecules in the unit cell, have been also solved but longer time is required. Although TALP is focused on organic compounds, also coordination compounds have been successfully solved.

[1] W.I.F. David, K. Shankland, *Acta Cryst.* **2007**, *A64*, 52-64.

Keywords: Powder diffraction, molecular compounds, *ab initio* structure solution

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Crystal structures and humidity-dependent phase transitions of Gly-L-Tyr hydrates

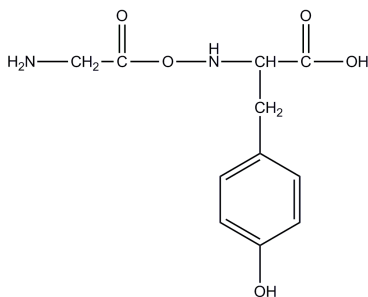
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Oligopeptides are one of the physiologically active substances and used as medicines, sweeteners, and food additives. They frequently crystallize as hydrates, and crystal structural transformations are induced by hydration and dehydration processes. Under such backgrounds, we aim to carry out *ab initio* crystal structure determination of oligopeptides

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

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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.

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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