compounds can give a better insight into the properties of hydrogen bonds and of other, weaker, non-covalent interactions in these systems. This can, in turn, be helpful for getting a better understanding of the conformational changes induced by temperature, pressure, or chemicals in the biopolymers built from amino acids (peptides). In the contribution we shall illustrate this by the results of recent X-ray single-crystal and X-ray powder diffraction, Raman and IR-spectroscopy studies at variable temperatures and pressures, as well as of the DSC and adiabatic calorimetry studies from 5K to the decomposition temperatures.

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Keywords: hydrogen bonds, high pressures, low temperatures

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Neutron Diffraction Structure of the β-Cyclodextrin Ibuprofen Complex at 15K
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The structure of the inclusion complex of β-cyclodextrin (β-CD) with ibuprofen has been determined as part of a study of β-CD complexes with non steroidal anti-inflammatory drug molecules and similar organic compounds. Ibuprofen is a hydrophobic molecule but becomes soluble in water by complexation with β-CD. This complex forms dimers in the crystalline state. Very often β-CD complexes crystallize as dimers linked head to head by hydrogen bonds between secondary hydroxyls. These dimers form infinite two dimensional layers in a C2 unit cell. The extended crystal structure is built up by linking together the layers in different packing modes. As well as the substantial pharmaceutical interest of describing the interaction between the drug and the CD molecule in the crystalline complex, one of our goals was to investigate how the nature of the guest and the solvent molecules influences the packing mode in the crystal, how the hydrogen bonding interactions are important in the supramolecular structure, and how order-disorder phenomena observed in analogous compounds can be explained. In these studies, we have used X-ray and neutron diffraction data, as well as X-ray diffuse scattering patterns. The results of the X-ray diffuse scattering analyses will not be described here. Here we report the first neutron diffraction structure of a dimeric β-CD complex (at 15K) and the comparison with the Synchrotron X-ray structure (at 300K).
Keywords: β-cyclodextrin-ibuprofen, neutron diffraction, order-disorder

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Electrostatic Properties of Two Precursors of Potent HIV-1 Integrase Inhibitors
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New AIDS therapy developments focus on the integrase inhibition in order to block the virus replication. Quinoline derivatives are potent drugs in this novel chemotherapy [1]. These molecules are formed by a quinoline moiety connected to a hydroxylated aromatic ring through a spacer fragment. This latter plays an important role in both inhibition and toxicity of the drugs. We have carried out the study of electrostatic properties of the two main precursors. These properties are derived experimentally from high-resolution X-ray diffraction experiments and from quantum mechanics calculations at Hartree-Fock level. The topological features of the electron density of precursors are carefully analyzed. The atomic charges and the electrostatic potential are discussed to highlight the correlation between the drug activity and the electronic structure.

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Keywords: electron density, electrostatic properties, drug structure-activity relationships

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Crystal Structures of Potential Sweeteners. The Kier Glucophore Geometry
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We compare molecular geometry and interactions of new potential sweeteners which were designed using chemical modification of a known sweet compound [1] and bioisosteric replacement. We determined crystal structures of three arylsulfonylalcanoic acids and one bioisoster containing a tetrazole instead of the carboxylic group. Unfortunately, last of them occured to be bitter. However, it is not very surprising since the sweet and bitter tastes are strongly related.

According to the geometrical model of glucophore given by Kier, there are three fundamental fragments of a sweet compound which interact with a sweet taste receptor [2]. A sweetener should contain a donor and an acceptor of hydrogen bond and a fragment which can be involved in hydrophobic interactions [3]. Distances between those fragments define a glucophore. However, the geometry of our sweet compounds in the crystalline state do not agree with the Kier model.

We observed a pair of very strong hydrogen bonds in sweet compounds building a dimer *via* inversion centre whereas in tetrazole the dimeric structure does not occur. That can explain why the bioisoster is bitter.

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Keywords: sweetener, dimer, hydrogen bond

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Chloroquine Derivatives. Conformation and Intermolecular Interactions
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Crystal structure of hydroxychloroquine (OHClQ) sulfate has been determined: a=10.5437(3)Å, b=8.8532(2)Å, c=22.0923(8)Å, α=90°, β=101.426(1)°, γ=90°, $P2_1/c$, Z=4, in order to compare its conformation and intermolecular interactions to those in the crystalline chloroquine (ClQ) phosphate [1] and quinine salicylate (QSal) monohydrate [2].

Molecular conformations of OHClQ and ClQ are comparable in both salts; the differences between corresponding torsion angles are not greater than 10°. Each of the nitrogen atoms is a proton donor in the intermolecular hydrogen bonds with the oxygen atoms of sulfate or phosphate anions. While the parameters of the N1-H1···O and N3-H3···O bonds are similar, the distance N···O within the bond N2-H2···O is much shorter in the case of OHClQ. The -OH group of OHClQ forms an additional H-bond with the oxygen atom of SO_4^{2-}.

The comparison of the hydrogen bonds formed by OHClQ and ClQ with those of quininium anion in QSal shows that these antimalarial molecules may interact with their putative receptor in a similar way.

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Keywords: antimalarials, chloroquine, intermolecular interactions

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Synthesis and Structural Characterization of 3-(4-fluorophenyl)-2-(α-naphtyl)-1,3-thiazolidin-4-one

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One of the richest sources of diversity for the medicinal chemist is small heterocyclic rings, which in addition to often exhibiting biological activity, may serve as rigid scaffolds for further display of functionalities. Thiazolidine derivatives belong to an important family of these heterocyclic compounds. Substituted thiazolidines display diverse biological activities such as tuberculostatic, fungicidal, pesticidal, herbicidal, antidiabetic, anti-inflammatory and bactericidal. The biological significance of this kind of compounds urged us to study of the synthesis and physicochemical properties of some 3-aryl-2-(α-naphtyl)-4-thiazolidinones due to their possible biological activities. As part of our ongoing research program aiming at the search of structural chemistry and substituted thiazolidinone synthesis from accessible aldimines, we used α-naphtaldimines in the preparation of new series of 3-aryl-2-(α-naphthyl)-1,3-thiazolidin-4-ones.

The compound 3-(4-fluorophenyl)-2-(α-naphtyl)-1,3-thiazolidin-4-one crystallizes in a monoclinic cell with the cell parameters a = 10.6097(5), b = 10.8356(5) c = 13.2278(6)Å and β = 101.0850(10)°, Space group $P2_1/c$ [No 14], V = 1492.33Å^3 and Z= 2.

Keywords: thiazolidinone, structural characterization, single crystal

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Structural Analysis of the N-terminal Domain of PriA from *E. coli*

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PriA, a DEXH-type DNA helicase, is essential for restoration of stalled replication forks and is a candidate sensor protein that recognizes arrested replication forks in bacteria [1]. The N-terminal domain binds to a free 3' terminus through the putative 3'-terminus recognition pocket [2]. We analyzed the interaction between N-terminal minimum binding domain of PriA[1-105] and oligonucleotides by using the single molecule fluorescence detection system MF20 (Olympus, Tokyo). The results indicated that PriA[1-105] recognized only the 3' terminal nucleotide portion of oligonucleotides.

We determined the crystal structure of the N-terminal domain of PriA to reveal the structural basis of the 3' terminal nucleotide recognition. Single crystals of native and SeMet PriA[1-105] were grown in hanging drops with a reservoir solution consisting of 0.1 M sodium citrate pH 3.6-3.8 and 0.15-0.35 M ammonium sulfate. Data sets were collected on BL38B1 at the SPring8 and PF-BL6A at the KEK. Native crystal diffracted to 2.8 Å and belongs to space group *R32*, with unit cell parameters, a=b=111Å, c=260Å.

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Keywords: DNA recognition, arrested replication fork, free 3'-terminus

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Crystal Structure Determination of a Valinium Hybrid Compound

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In recent years, organic-inorganic hybrid materials have attracted considerable attention as prefered materials in nonlinear optics (NLO), such as second harmonic generation (SHG) and optical bistability, owing to their large optical nonlinearities [1]. L-valinium hydrogenphosphite, results from our systematic investigation of organic-inorganic hybrid materials obtained by interaction between various phosphoric oxyacids and amino acids [2],[3].

As part of our continuing interest in this field, we report here the crystal structure of L-Valinium hydrogenphosphite [$C_5H_{12}NO_2^+$, $H_2PO_3^-$], it can be described as a stacking of l-valinium and hydrogenphosphite ions. The stability of such an arrangement results from a network of hydrogen bonds, which maintain the cohesion of the organic-inorganic layers in the crystal. The asymmetric unit contains two valinium residues and two hydrogenphosphite ions, one of which is disordered.

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Keywords: valinium, hydrogen-bonding, disorder

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SAXS Studies of Nucleation of Glycine from its Supersaturated Solution

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The early stages of the process of crystallization, especially that of small molecules from their supersaturated solution is not yet fully understood. In an effort to understand the process of nucleation and crystallization of such molecules, small angle x-ray scattering (SAXS) has been used to study the crystallization of the amino acid glycine from its supersaturated aqueous solution. The scattering data was analyzed using the Unified Fit Model which helps in studying complex systems that may contain multiple levels of related structural features. The results suggest that glycine molecules exist as dimers in supersaturated solution. The structure factor and the form factor obey power-law behaviour that indicates the presence of fractals in the solution. A transformation from mass fractal structure to surface fractal structure is observed during the crystallization process, which could be the signature of a two-step nucleation process.

Keywords: SAXS, nucleation, fractals