hydrogen bonds system (intra- and intermolecular ones) and crystal packing are also discussed. The crystal packing are stabilized by π-π-interactions between benzene rings and/or triazole heterocycles. The packing coefficient and solvent accessible potential area in crystal were also analyzed.
Keywords: triazoles, X-ray structure, tautomerism

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An Investigation of Interactions in Barakol Complexes
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Barakol has various interactions that contribute to its biological activities. This work presents the first successful crystal structure determination of anhydrobarakol and its analogues. Anhydrobarakol and anhydrobarakol hydrochloride were crystallized in a monoclinic system, space group $P2_1/c$ and $P2_1/n$, $Z = 4$ with unit cell parameters a = 13.2280(7), b= 6.8738(2) , c = 19.7879(9) Å, β = 127.013(2)° and a = 12.2547(2), b = 8.051(2) , c = 12.8133(2) Å, β = 99.514(1)°, respectively. The novel 1:1 molecular complexes of barakol and carboxylic acid (phthalic acid and 3-hydroxybenzoic acid) were synthesized and charactherized by spectroscopic and X-ray crystallographic techniques. Electrostatic effects, electron delocalization, and intermolecular interactions in the barakol ring system were investigated. The X-ray crystallographic studies revealed that the barakol-phthalate complex exists in an ion-pair complex. The formation of barakol-phthalate ion-pair complex is stabilized by the complementary of ion-ion interaction,π-π interaction and hydrogen bonding. The barakol-3-hydroxybenzoic acid complex is a π-π molecular complex. The co-crystallization of barakol- 3-hydroxybenzoic acid complex is solely stabilized by π-π interactions. The spectroscopic studies including IR, ^{1}H-NMR and UV-visible are consistent with the results from the X-ray analysis.
Keywords: barakol, X-ray diffraction, spectroscopic method

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Amino Acids at High Pressure
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The use of pressure to perform structural studies has been of important use to many areas of research, from Physics to Geochemistry. However, pressure studies have become a new notable tool in Chemistry and Biology to study the structure of small compounds. The main reason for this is the necessity of a better understanding of different processes, which happen even at extreme conditions of pressure, such as the existence of life in the deep ocean. Thus, small molecules may play important roles in these biological processes and therefore, a good knowledge of their structural features could be essential to explain how they happen.

In this work we are exploring the behaviour of amino acids structures at high pressure. Changes in pressure have been known to induce conformational changes in small molecules. We are trying to extend this research to the larger amino acids.

We have been working, principally, with L-glutamine, L-asparagine monohydrate, L- glutamic acid and L-aspartic acid. It was found that by applying pressure the cell parameters were reduced but no structural rearrangement was found up to pressures of 50-60 kbar. *Ab initio* computational studies were then performed to establish a possible relationship between the energetics of the hydrogen bonding with their compressibility.
Keywords: high-pressure crystallography, amino acids, *ab initio* calculations

P.05.05.4
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Structural Studies on ST1481, Gimatecan, a 7-substituted Camptothecin with Anti-tumor Activity
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Camptothecins are a class of of active antitumor agents that target the nuclear enzyme DNA topoisomerase I, inhibiting single strand religation. Several camptothecin derivatives are in clinical trials [1]. The substitution of position 7 by lipophilic side chains seems to be important as it increases cytotoxic potency, helps in the drug delivery and in stabilizing the DNA-topoisomerase I cleavable complex that forms in ST1481 presence and is is as part of its mechanism of action. Of 44 compounds synthesized [1] the most potent derivative contains a $CH{=}NOC(CH_3)_3$ substituent and its X-ray crystal structure has been determined. The unit cell parameters are space group: $\boldsymbol{P2_1}$, a = 12.131(8)Å, b = 6.712(5)Å, c = 13.817(8)Å, β = 96.05(3). This derivative (gimatecan) is orally administered, and thus, represents a significant advantage compared to other camptothecins. *Ab initio* studies have been performed using Density Functional Theory to analyze the lactone ring opening, a critical step in the interaction with topoisomerase I.

[1] Dallavalle S., Ferrari A., Biasotti B., Merlini L., Penco S., Gallo G., Marzi M., Tinti M. O., Martinelli R., Pisano C., Carminati P., Carenini N., Beretta G., Perego P., De Cesare M., Pratesi G., Zunino F., *J. Med. Chem.*, 2001, **44**, 3264.
Keywords: camptothecin, anti-tumor, topoisomerase I inhibitor

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Hydrogen Bonding and Absolute Configuration in Manzamine Alkaloids
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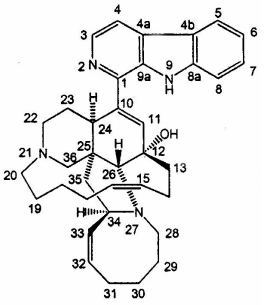
The manzamines, a class of sponge-derived alkaloids, have a complex polycyclic system with 5, 6, 8, and 13-membered rings and a β-carboline substituent. They show promising antibacterial, antitumor, and antimalarial activities, and moderate activity against HIV-1. Manzamine A (**1**) forms solvates with MeOH and acetone. Formation of the hydrochloride of 8-OH manzamine A (**2**) by N27 protonation allows absolute configuration determination. The Cl^- accepts hydrogen bonds from all four donors of one cation. Manzamine F (**3**) also has an 8-OH group and C31=O rather than C32=C33, and forms a mixed solvate with H_2O and MeCN. Ircinol A (**4**) lacks the β-carboline group, but has CH_2OH at C10, and crystallizes with Z'=3.



Keywords: marine natural products, absolute structure, hydrogen bonding

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The Structure of Protocyanin, a Complex Pigment from Blue Cornflower
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Protocyanin is a complex pigment extracted from flower petals of blue cornflower, *Centaurea cyanus*. The components of protocyanin were recently demonstrated to be anthocyanin (AN), flavone glycoside (FL), Fe^{3+}, Mg^{2+} and Ca^{2+} ions[1]. For X-ray structure determination, protocyanin was reconstructed from the components and crystallized in space group $P2_12_12_1$ with unit cell dimensions of a = 29.7, b = 49.2 and c = 78.3Å. Two protocyanin molecules are contained in an asymmetric unit. Data were collected on the beam line 6A at Photon Factory KEK to1.05 Å resolution.

The refined molecule has pseudo three-fold symmetry and four metals align along the pseudo three-fold axis in order of Ca^{2+}, Fe^{3+}, Mg^{2+} and Ca^{2+}. The four metals are coordinated to six AN and six FL molecules. The inner Fe^{3+} and Mg^{2+} ions are each coordinated to three AN's, respectively, while the outer two Ca^{2+} ions are each coordinated to three FL's. Both AN and FL molecules are self-associated with each other as AN-AN and FL-FL in pair and this hydrophobic association also exists between AN and FL molecules, building copigmentation stacks. Protocyanin is a tetra-metal (Fe^{3+}, Mg^{2+}, $2Ca^{2+}$) nuclear complex, a new type of supramolecular pigment.

[1] Takeda K., Osakabe A., Saito S., Furuyama D., Tomita A., Kojima Y., Yamadera M., Sakuta M., *Phytochemistry*, *submitted*.

Keywords: X-ray structure analysis, pigments, biological molecular complexes

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Crystal Structures of the Fungal Metabolite Oosporein

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Oosporein is a symmetrical red coloured 2,5-dihydroxybenzo-quinone derivative biosynthesized by a broad variety of soil borne fungi. The compound, being known for almost six decades, is the major secondary metabolite of the entomopathogenic fungi *Beauveria brongniartii* which is successfully applied as a biological control agent against the European cockchafer *Melolontha melolontha*. In the course of isolating and purifying pure oosporein from biological cultures we obtained a dioxane solvate and a non-solvated form which were characterized with different solid state analytical techniques including X-ray diffraction.

The molecular geometry of oosporein is x-shaped with a dihedral angle of 67.8 and 79.9° in the non-solvated form and the dioxane solvate respectively. Surprisingly the two forms crystallize in the same space group (monoclinic, C_2/c) showing a similar O-H...O network. The non-solvated form shows two dimensional O-H...O tetrameric layers which are off stacked leading to a densely packed structure. In the dioxane solvate one solvent molecule is involved in the O-H...O hydrogen bond network resembling the overall network of the anhydrous form. This pseudo-tetrameric arrangement results in a large channel along the c-axis which is occupied by highly disordered dioxane molecules.

[1] Frank R.L., Clark G.R., Coker J.N., *J. Am. Chem. Soc*., 1950, **72**, 1827.

Keywords: oosporein, natural organic molecules, crystal structure

P.05.06.1

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Xanthone Derivatives: Conformational Study and Development of Force Field

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Xanthone derivatives extracted from herbs are important components of homeopatic antibacterial, mycotoxic and cytotoxic medicines. Synthetic substituted xanthones tested against broad spectrum of biological activities revealed: antiinflammatory, cytostatic, antimycotic, and cardiovascular activities. In a series of newly synthesized substituted xanthones, two constitutional isomers, 2-methyl-2[2-(methyl)-6-xanthonoxy]-propionic acid, 2-methyl-2[4-(methyl)-6-xanthonoxy]-propionic acid, and racemic (RS)-2-[2-(methyl)-6-xanthonoxy]-propionic acid have shown differentiated antiinflammatory action.

The crystal structures of xanthone derivatives were solved using both single-crystal diffraction and HRPD data recorded with synchrotron radiation. In order to find the native, optimal structures of xanthone derivatives in their natural environment of lipid bilayer, additional force field parameters were obtained using X-ray diffraction data and *ab-initio* calculations.

Keywords: xanthone, *ab-initio* calculations, synchrotron radiation

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Diffraction and Computational Studies of Hydrogen Bonded Base Paired Systems

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Diffraction methods are often utilised to gain a greater insight hydrogen bonding interactions. In work to date the advantages of single crystal variable temperature x-ray and neutron diffraction, coupled with the imaging capabilities of Fourier difference maps, as complementary techniques for anomalous hydrogen bonding investigations have been highlighted [1]. In addition, recent advances in computational chemistry have enabled calculations to be carried out both on isolated molecules and in the periodic (i.e. crystalline) environment [2].

The main aim of this poster is to discuss the use of complementary methods to promote a better understanding of hydrogen bonding within carboxylic acid dimers and nucleic acid base paired systems. One particular system of interest is 3',5'-di-O-acetylthymidine and various techniques have been utilised to determine the presence of anomalous hydrogen behaviour. A crucial part of this has been a multiple temperature high resolution study carried out on station 9.8 at SRS, Daresbury. Alongside experimental work, recent computational work will highlight how these new methods can augment traditional experimental results. Overall it is hoped that the research presented will again highlight the importance of complementary techniques in crystallographic research.

[1] Parkin A., Harte S. M., Goeta A. E., Wilson C. C., *New J. Chem*., 2004, **28**, 718. [2] Wilson C. C., Morrison C. A., *Chem. Phys. Lett*., 2002, **362**, 85-89.

Keywords: hydrogen bonds, diffraction methods, computation

P.05.07.2

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Polymorphism of Crystalline Amino Acids. The Role of Non-covalent Interactions

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The crystals of amino acids are interesting from several points of view – as drugs, as molecular materials (e.g. piezo- and ferroelectrics), but also as biomimetics. Understanding the effects of pressure, temperature, and various chemicals on the crystal structures of these