

**P08.07.58***Acta Cryst.* (2008). A64, C436**Atomic displacement parameters based on quantum mechanical treatment of anharmonic oscillator**

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Gram-Charlier expansion (GCE) and one-particle potential generalized by Tanaka & Marumo (GOPP) sometime produce probability density function (PDF) with negative area which is not accessible physically. We derived atomic displacement parameters (ADPs) based on PDF without negative area, using quantum mechanical treatment of anharmonic oscillator. The ADPs including anharmonic vibrations of atoms is formulated taking the anharmonic potential as a perturbation to the harmonic potential in the 3D harmonic oscillator case. Since the first order correction to wave function of the atomic vibration is expected to be a linear combination of the wave functions of the harmonic oscillator, which is expressed in terms of Gauss-Hermite polynomials multiplied by Gaussian. In order to show it clearly the PDF functions assumed in GOPP, GCE and the present study are summarized below: GOPP  $\text{Pdf}_{\text{harm}}(a + \sum d_{ijk} u_{ijk} + \sum e_{ijkl} u_{ijkl})$ , GCE  $\text{Pdf}_{\text{harm}}(1 + \sum d_{ijk} \text{He}(u)_{ijk} + \sum e_{ijkl} \text{He}(u)_{ijkl})$ , Present  $\text{Pdf}_{\text{harm}}(a + \sum b_i H(u)_i + \sum c_{ij} H(u)_{ij} + \sum d_{ijk} H(u)_{ijk} + \dots)^2$ . Where He or H denotes Chebychev-Hermite or Gauss-Hermite polynomials. The three methods commonly assumed that each atom vibrates independently from others. Since the squares of the wave function is the PDF function of the atom, PDF function never be negative in the present study, while in the other methods it easily becomes negative. Since the formalism of the present study is based on the quantum mechanical treatment of vibration, it is expected to represent the ADPs of X-ray diffraction more properly than GCE. Since recent development of neutron diffraction like J-PARC project will increase the opportunity to get more reliable ADPs, the ADP formalism becomes more important and can be compared on more reliable experimental facts.

Keywords: probability density function, atomic displacement parameters, anharmonic oscillator

**P08.08.59***Acta Cryst.* (2008). A64, C436**Amoxicillin trihydrate - potassium clavulanate solid solution**

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Amoxicillin trihydrate and potassium-clavulanate for several decades have been combined in a drug dosage form to maintain amoxicillin's antibiotic potency. In fact, the high variability quality and pharmacokinetic profile of their dosage forms often found. This phenomenon was predicted base on their solid state intermolecular interaction. Then, the investigation of this interaction were performed by crystallography analyzes, such as thermal analysis, Powder X-Ray Diffractometer, Scanning Electron Microscope, Fourier Transformed Infra Red, and Neutron Magnetic Resonance. A series of amoxicillin-clavulanate molar fractions were treated by freeze drying, co-ground by solvent dropping, and co-heating. Crystallographic data proved that amoxicillin-clavulanate arranged a non-stoichiometrical co-crystal, known as solid solution, with the lowest energy interaction

of 1:1 molar ratio. The result of FTIR and NMR analysis depicted that the bonding was occurred between amine of amoxicillin with the carboxylic of clavulanate and also hydrates of amoxicillin with amine and carboxylic of clavulanate. Furthermore a salt reaction arranged after freeze drying. The solid solution showed different physico-chemical properties and antibiotic potency compare to each single component and their physical mixture counterparts.

Keywords: intermolecular interaction, solid solution, amoxicillin-clavulanate

**P08.08.60***Acta Cryst.* (2008). A64, C436**Cohesion forces in halomethane crystals**

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Methanes are the simplest carbon compounds and therefore their structures are of fundamental interest. The methane molecules interact by van der Waals forces, and owing to the small size and bulky shape the close packing of molecules is not severely restricted by close packing in the crystal structures [1]. In this respect methanes can be considered as models for the aggregation of molecular fragments in many organic substances. An analogous reasoning applies to halogenated methanes, however their structures additionally reveal the basic aggregation patterns involving halogen...halogen and CH...halogen contacts. Along with hydrogen bonds, halogen...halogen interactions are considered to be the strongest of cohesion forces in molecular crystals, but their role for molecular aggregation is still disputable. Apart from the evidence of molecular attraction to the halogen atoms, there are also arguments for their repulsion. Hence we have studied a series of halomethanes [2-4]. Isostructural relations and phase transitions of halomethanes have been rationalized by the competing patterns of CH...halogen hydrogen bonds and halogen...halogen intermolecular interactions. The obtained results shed new light on the nature of these intermolecular interactions and on the values of van der Waals radii in molecular crystals [5].

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Keywords: intermolecular interactions, high-pressure structures, halogens

**P08.08.61***Acta Cryst.* (2008). A64, C436-437**Structural studies of CLIC protein complexes**Juanita Mintono Phang<sup>1</sup>, Stephen J Harrop<sup>1,2</sup>, Krystyna E Wilk<sup>1</sup>, Anthony Duff<sup>3</sup>, Samuel N Breit<sup>2</sup>, Paul MG Curmi<sup>1,2</sup><sup>1</sup>University of New South Wales, Biophysics, School of Physics, University of New South Wales, Sydney, New South Wales, 2052,

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Chloride Intracellular Channel (CLIC) proteins are recently discovered members of the chloride ion channels family that have the unusual property of existing as both soluble proteins and integral membrane channels. There are 6 members in the human CLIC family (CLIC1-6). Most of CLICs exist in cells as both soluble and integral membrane proteins. Although the specific cellular function of these CLIC proteins remains unclear, CLICs have been associated with some cellular functions such as cell cycle regulation and maintenance of intracellular membranes. The mechanism of CLIC insertion into the membrane remains unclear, however previous studies suggest that this process may involve structural changes and/or protein-protein interactions. CLIC proteins are believed to form a complex with the N-terminus FERM domain (band Four-point-one, Ezrin, Radixin, Moesin domain) of the ERM (Ezrin-Radixin-Moesin) proteins. ERM is a member of protein 4.1 superfamily which is characterised by the ~300kDa FERM domain. This superfamily mediates the cross-linking between the cytoskeleton and the plasma membrane. Current studies using both blue native polyacrylamide gel electrophoresis and Western Blots indicate that CLIC1 forms a complex with the FERM domain of Ezrin. Further crystallographic studies of these complexes are necessary to reveal the nature of the complex.

Keywords: CLIC, FERM domain, macromolecular complexes

## P08.08.62

*Acta Cryst.* (2008). A64, C437

### Interaction of sols on a dispersion containing only the counterions dissociated from the surface

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The electrical potential for the case of two identical, planar parallel sols immersed in a salt-free medium, where the ionic species is the counterions come solely from that dissociated from the surfaces, is evaluated. We show that in a salt-free dispersion if the separation distance between two sols is sufficiently far, the electrical repulsive force dominates, that is, the total energy is positive and does not have a secondary minimum, which is not the case for a dispersion where both coions and counterions are present. Also, the conditions used to calculate the critical coagulation concentration in the classic DLVO (Derjaguin, Landau, Verwey, and Overbeek) theory become inappropriate and Derjaguin approximation is inapplicable. We show that if the surface charge density exceeds ca.  $0.04 \text{ C/m}^2$ , the stability of a salt-free dispersion remains essentially the same. If the surface charge density is sufficiently high, the maximum separation distance between two particles below which coagulation occurs is in the range [0,1 nm] and [1,7 nm] for the case where Hamaker constant are  $10^{-20} \text{ J}$  and  $10^{-19} \text{ J}$ , respectively.

Keywords: salt-free dispersion, Poisson-Boltzmann equation, stability

## P08.08.63

*Acta Cryst.* (2008). A64, C437

### The origin of polar ordering in high pressure phases of chloroform and bromoform

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The majority of materials crystallize in centrosymmetric or nonpolar space groups. One of the examples are chloroform ( $\text{CHCl}_3$ , CF) and bromoform ( $\text{CHBr}_3$ , BF). Despite their pyramidal molecular structure all their low-temperature (LT) crystalline phases are centrosymmetric. This contrasts with most of crystals built of pyramidal molecules or ions, like a prototypical pyroelectric  $\alpha\text{-LiIO}_3$ . Therefore we undertook to perform an X-ray diffraction study of pressure-frozen CF and BF. Only one LT phase of CF was reported (space group  $Pnma$ ) [1], while BF is known to exist in 3 phases: a disordered one ( $P6_3/m$ ), formed just below melting point, which transforms on cooling further to ordered  $P-1$  or  $P-3$  polymorphs [2]. Due to the less attractive character of Cl...Cl interactions the structure of CF is distinct from the BF phases, in which the molecules are arranged in bipolar sheets interacting by a cooperative triangular motif of Br...Br bridges. We revealed that CF crystallizes at 0.62 GPa yielding a LT phase, which transforms between 0.62 and 0.75 GPa to a polar  $P6_3$  phase. Isochorically frozen BF forms isostructural polar crystals already at the freezing pressure of ca. 0.1 GPa. It can be shown that X...X and H...X interactions in the structures of CF and BF can be rationalized in terms of electrostatic forces and molecular electrostatic potential mapped onto molecular surfaces. The polar crystals of CF and BF contribute to a better understanding of the structural mechanism responsible for polar symmetries of materials, required for technological applications [3].

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Keywords: polarity, halogens, high-pressure polymorphism

## P08.08.64

*Acta Cryst.* (2008). A64, C437-438

### Construction of $2_1$ helical assemblies of fluorescent molecules and the study on their properties

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$2_1$  helical assemblies are often found and general in crystals. Conventionally, it has been assumed that right- or left- handedness of  $2_1$  helical assemblies cannot be determined on the basis of mathematical viewpoint, because the two-fold screw axis operation includes just 180 degrees rotation and translation. We had, however, been aware of that those can be chiral when the objects consisting of themselves are not spherically-symmetric. Recently, we have suggested that it is possible to define the handedness of  $2_1$  helical aggregates observed in crystals on the basis of the molecular tilt against the  $2_1$  axis. In this study, we prepared the crystals in which fluorescent molecules form  $2_1$  helical assemblies. Namely, crystallization of 9-fluorenone-2-carboxylic acid (9F2CA) and chiral amines yielded homo-chiral  $2_1$  helical structures (Figure). We determined the handedness of the  $2_1$  helical assemblies on the basis