

**[s9.m1.p1] Relationships between H-bonding and donor-acceptor bonds in amine-functionalized alkoxides of aluminum and gallium.** J. Zachara,<sup>1</sup> J. Lewinski,<sup>1</sup> T. Kopce,<sup>1</sup> J. Lipkowski,<sup>2</sup> E. Kolodziejczyk,<sup>2</sup> <sup>1</sup>Warsaw University of Technology, 00-664 Warsaw, Poland, <sup>2</sup>Institute of Physical Chemistry, Polish Academy of Sciences, 01-224 Warsaw, Poland

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In spite of large number contributions and advances in self-assembly of organic molecules based on noncovalent interaction, the area of organometallic compounds remains in this context largely unexplored. To date there has been no systematic attempt to exploit relationship between donor-acceptor interactions and hydrogen bonds though this relationship is of significance from material science to biochemistry. Both types of interaction are responsible for various types of self-organization and molecular recognition and without intimate understanding of cooperation between hydrogen bonds and classical donor-acceptor bonds will be difficult deduced general building principles for synthesis of self-assembly structures with well-defined properties.

In this report we present the structure investigation of the group 13 ( $M = Al, Ga, In$ ) diorganometallic chelate complexes derived from simple  $\alpha$ - and  $\beta$ -primary-amine alcohols. It will be demonstrated that group 13 amine-functionalized alkoxides may serve as an interesting model system for systematic study of interdependence between intra- and intermolecular forces resulting from donor-acceptor and hydrogen bonding interactions. The reported studies show that upon controlling of the coordination center and the chelate ligand structure *e.g.* chain size or an increased steric bulk on the alkoxide  $\alpha$ -carbon, various molecular aggregations may be constructed. The monomeric species may form either five-coordinate  $[R_2M(\mu-O,N)]_2$  dimers - in which the alkoxy oxygen is engaged in the donor-acceptor bond with the metal center - or four-coordinate hydrogen bonded polymer  $[R_2M(O,N)]_n$  containing the strong intermolecular N-H...O interactions. In addition, we show that conformational changes of *O,N*-chelate ligands can impart the morphology of a hydrogen bonded net.

**[s9.m1.p2] Ultra-high resolution structure of a new polymorphic form of bovine pancreatic trypsin inhibitor.** A. Addlagatta<sup>1</sup>, H. Czapińska<sup>2</sup>, Sz. Krzywda<sup>3</sup>, J. Otlewski<sup>2</sup>, M. Jaskólski<sup>1,3</sup> 1). Center for Biocrystallographic Research, Institute of Bioorganic Chemistry, Polish Academy of Sciences, Poznan, Poland. 2). Department of Protein Engineering, Institute of Biochemistry and Molecular Biology, University of Wrocław, 3). Dept. of Crystallography, A.Mickiewicz Univ., Poznan, Poland.

Keywords: protein crystallography, BPTI, intermolecular interactions.

Few proteins have undergone such detailed conformational analysis as bovine pancreatic trypsin inhibitor (BPTI), a 58 amino acid globular protein with three disulfide bonds, a well defined secondary structure and hydrophobic core. Several polymorphic forms have been reported for this protein and its mutants.

Recently, a room temperature, 1.42 Å resolution structure of a new tetragonal form (P4<sub>3</sub>2<sub>1</sub>2) has been reported from our laboratory for a strategic mutant with altered binding loop sequence. Many important features were observed in that structure such as: no change in the conformation of the binding loop despite three mutations, salt bridged N- and C-termini, a disulfide bond in two chiralities, five internal water molecules and four sulfate anions, an intermolecular four-stranded  $\beta$ -sheet, highlighting their role in the formation of very stable, highly diffracting crystal that can be grown on the time scale of hours.

Recently, low temperature (100 K), sub-atomic resolution (0.87 Å) data have been obtained for the same mutant crystallized under similar conditions. The structure has been refined with anisotropic displacement parameters using both conjugate gradient and least-squares algorithms in a way similar to small-molecule refinement. The final R factor is 10.74 % for all 46,219 reflections and 6,615 parameters. The overall structure is similar to that observed at room temperature with localized differences. Some of the high B-value residues have been resolved into double conformations, there are changes in the solvent region including appearance of cryoprotectant molecules and of additional ions. This low-temperature and high-resolution data allow a detailed analysis of intra- and intermolecular interactions involving not only the classical ones but also weak interactions, like N/C-H...p, and C-H...O hydrogen bonds.

In this presentation, a comparison of the room-temperature and low-temperature structures will be given and aspects of various intermolecular interactions and of their effect on local geometry will be discussed.