the home system allows collection of data as soon as the crystals are produced to get the initial solution of novel structures and is invaluable in the quick turnover often required in ligand-binding studies.

We will describe how the combination of the updated Agilent Technologies SuperNova, a highly efficient compact diffractometer, with the new version of fully automated CrysAlisPro data collection and processing software, optimized for macromolecular crystallography, makes an ideal home lab solution complementing synchrotron data collection.

New unique features of CrysAlisPro and several examples of high quality results obtained with the system will be presented.

Keywords: macromolecular, experiment, software

MS73.P01

Acta Cryst. (2011) A**67,** C661

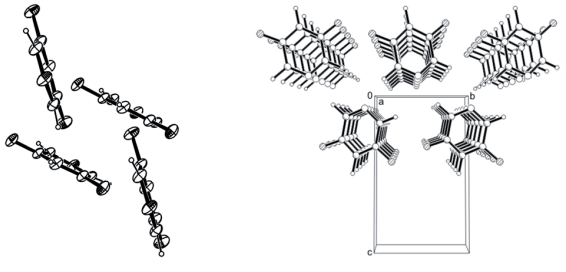
Fluorine determines the aggregation of pyridines. Experiment vs. theory

Vera Vasylyeva,[a] Klaus Merz,[a] Oleg V. Shishkin,[b] [a]*Inorganic Chemistry I Department, Ruhr-University Bochum, Bochum (Germany).* [b]*Institute for Single Crystals, National Academy of Science of Ukraine, Kharkiv (Ukraine).* E-mail: vira.vasylyeva@rub.de

Fluorine is a unique element. The question about the role of Fluorine in intermolecular interactions is discussed controversially. Well known is the influence of fluorine on the electronic structure of aromatic backbones and therefore on the entire molecules. On the other hand, fluorine forms only weak intermolecular interactions and seems to have no influence on the crystal packing. Pauling's definition of the hydrogen bond would imply that fluorine, as the most electronegative atom, should be a stronger hydrogen-bond acceptor then oxygen and nitrogen. But the C-F group, the so-called "organic fluorine", does not form hydrogen bonds commensurate with electronegativity considerations in contrast to the C-O and C-N groups.

We investigated a range of partially fluorinated pyridines and analysed their crystal packings experimentally and theoretically. Low temperature *in situ* crystallisation on the diffraktometer was used to investigate crystal structures of low melting fluorinated pyridines followed by analysis of the crystallisation behavior. Interesting tendencies were observed in crystal packings depending on the fluorination degree.

But still the general question we are interested in, is: what determines the crystal packing in the absence of strong intermolecular interactions? Theoretical study of the energies of weak intermolecular interactions is an innovative method for research of the basic motives in the solid state. The comparison of our experimental and theoretical findings shows how fluorine atoms influence the aggregation of substituted pyridines. The picture below shows the difference between basic structural motives in the experimental (a) and theoretical (b) crystal packing of 3,5-difluoropyridine.



[1] V. Vasylyeva, K. Merz, *J. Fluorine Chem.* **2010**, *131*, 446-449. [2] V. Vasylyeva, K. Merz, *Cryst. Growth Des.* **2010**, *10*, 4250-4255. [3] K. Merz and V. Vasylyeva, *CrystEngComm* **2010**, *12*, 3989-4002. [4] V. Vasylyeva, O.V. Shishkin, K. Merz *Cryst. Growth Des*. **2011,** submitted.

Keywords: crystal engineering, fluorine, halogen bonding

MS73.P02

Acta Cryst. (2011) A**67,** C661

TMA alcohol solvates: filling the gaps and increasing the dimensionality

Mark R.J. Elsegood and Sophie H. Dale, *Chemistry Department, Loughborough University, Loughborough, LE11 3NL, (UK).* E-mail: m.r.j.elsegood@lboro.ac.uk

Benzene-1,3,5-ticarboxylic acid, or trimesic acid (TMA), has been the focus of research interest for many years due to its symmetry, hydrogen bonding ability, ability to form salts and solvates, and use as an organic linker in metal organic frameworks. We have previously reported 1:1 and 1:2 TMA:MeOH solvates that demonstrated the stepwise dissolution of TMA via disruption by methanol of the $R^2_2(8)$ head-to-tail carboxylic acid dimer H-bonding pattern. This common pattern is seen in many pure carboxylic acids including the three-fold interpenetrated honeycomb lattice of pure TMA [1]. The disruption occurs via the insertion of an alcohol OH group into the $R^2_2(8)$ ring to generate an expanded $R^3_3(10)$ motif. More recently, the related structures of the higher alcohol homologues 1-butanol, 1-pentanol, and 1-hexanol were reported by Perepichka and Rosei [2]. This work revealed a structural dependence on the length of the alcohol's alkyl chain.

We are now able to report the intervening 'missing' TMA–alcohol solvate crystal structures with EtOH, 1-propanol, and 2-propanol, two of which are twinned. Their structures are placed in the context of the preceding work and our new findings plug the gap in current knowledge. We also report the structures of two diols which extend the dimensionality from 1D ladders (Fig. 1) to 2D sheets. The diol solvates/co-crystals exhibit further disruption of the $R^2_2(8)$ rings.

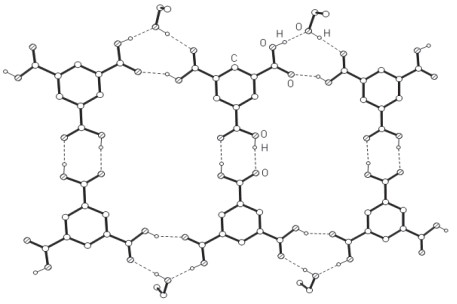


Fig. 1. 1D ladder structure adopted by TMA·EtOH with $R^2_2(8)$ and $R^3_3(10)$ H-binding motifs.

[1] S.H. Dale, M.R.J. Elsegood, S.J. Richards, *Chem. Commun.,* **2004**, 1278-1279. [2] K.G. Nath, O.Ivasenko, J.M. MacLeod, J.A. Miwa, J.D. Wueat, A. Nanci, D.F. Perepichka, F. Rosei, *J. Phys. Chem. C.,* **2007,** *111*, 16996-17007.

Keywords: hydrogen bonding, crystal engineering, trimesic acid

MS73.P03

Acta Cryst. (2011) A**67,** C661-C662

Hydrogen bonding, Z' and stability of diclofenac amine salts

Carl H. Schwalbe,[a,b] Miren Ramirez,[a] Barbara R. Conway,[c] Peter

Timmins,[d] [a]*School of Life & Health Sciences, Aston University, Birmingham B4 7ET, (UK).* [b]*Cambridge Crystallographic Data Centre, 12 Union Rd, Cambridge CB2 1EZ, (UK).* [c]*School of Applied Sciences, University of Huddersfield, Huddersfield HD1 3DH, UK.* [d]*Bristol-Myers Squibb, Reeds Lane, Moreton, Cheshire CH46 1QW, (UK).* E-mail: c.h.schwalbe@aston.ac.uk

We report four structures of primary ammonium salts of the anti-inflammatory agent diclofenac (D), and we complete the salt sequences from t-butylammonium to Tris and mono- to triethanolammonium, seeking to optimize formulation properties.

Primary ammonium salts form columns made from hydrogen bonded (HB) rings. The highest-melting primary ammonium salts have $R_4^3(10)$ columns (melting points by DSC in triplicate; estimated errors in parentheses). The six independent ion pairs of the cyclohexylammonium salt are related by pseudosymmetry.

R group	Z'	Graph set for HB ring(s)	M. p. /°C
t-butyl	1	$R_4^2(8)$ & $R_4^4(12)$	166(2)
cyclohexyl	6	2 independent $R_4^3(10)$	191(2)
benzyl	1	$R_4^4(12)$	108(2)
adamantyl	1	$R_4^3(10)$	242(2)

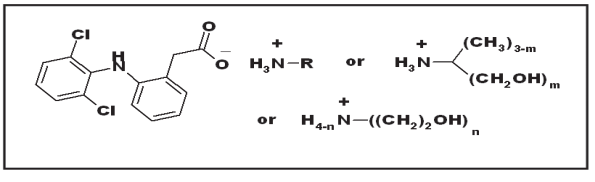
Hydroxymethyl derivatives have HB to OCO^- from OH as well as NH^+. Although $R_4^3(10)$ columns persist when m=1, they are distorted and presumably bring less stability.

m	Z'	H bond donors to OCO^-	M. p. /°C
1	1	$R_4^3(10)$, $R_2^2(9)$ NH^+&OH...O^-	143.7(6)
2	1	$R_3^3(8)$ NH^+&OH...O^-; $R_4^2(14)$	179(2)
3 (TUDPIR)	1	$R_3^3(8)$ NH^+&OH...O^-; $R_4^2(14)$	207.2(3)

Hydroxyethyl derivatives with n=1 or 2 also use these groups, but when n=3 the NH^+ is blocked [1]. Two ion pairs of the mono-hydroxyethyl compound differ in their HB: one D lacks the ubiquitous $S_1^1(7)$ intramolecular N-H…O^-, and only one pair has cation-cation NH^+…OH. Pseudosymmetry relates the other two.

n	Z'	H bond donors to OCO^-	M. p. /°C
1	4	$R_4^2(8)$ NH^+...O^-; $R_2^2(9)$	140.3(2)
2 (ZIKPOY)	1	$R_4^2(8)$ NH^+...O^-; $C_2^2(9)$	131.2(3)
3 (TEKVAG)	1	$C_2^2(12)$ OH...O^-	135(2)

HB motifs but not Z' obviously affect stability. We thank the NCS, Southampton, for data on these often difficult specimens.



[1] C. Castellari, S. Ottani, *Acta Cryst.* **1996**, *C52*, 2619-2622.

Keywords: diclofenac, H-bond, stability

MS73.P04

Acta Cryst. (2011) A**67,** C662

Halogen Interactions Using Variable Temperature Single Crystal X-ray Diffraction

Angshuman Roy Choudhury,[a] Piyush Panini,[b] Gurpreet Kaur,[a] and Deepak Chopra,[b] [a]*Indian Institute of Science Education and Research Mohali, Sector 81, S.A.S. Nagar, Manauli PO, Punjab, (India), 140306.* [b]*Indian Institute of Science Education and research Bhopal, Govindpura, Bhopal, (India), 462023*. E-mail: angshurc@iisermohali.ac.in.

The studies of intermolecular interactions involving halogen atoms in small organic molecules have been a controversial topic in contemporary research [1]. While a number of research groups have indicated that F behaves differently than Cl and Br in building crystal lattices [2]. mostly using Cambridge Structural Database; many other research groups have shown significant amount of experimental evidence of the active role of weak interactions such as C-H···F, C-F···F and C-F···π like C-H···X, C-X···X, C-X···π (X = Cl and Br) in crystal packing [3]. The natures of these interactions are yet to be well understood. The importance of fluorine in pharmaceutical industry, especially in medicinal application of small fluorinated organic molecules have gained significant interest in scientific literature [4]. We have been interested in this field as fluorine is one of the major substitutions found in drugs and drug intermediates and the C-F group in these play a major role in their biological activity [4b,c].In order to understand the nature and modes of weak interactions offered by a C-F group, we have chosen few model molecules such that these interactions can be studied both in the presence and absence of stronger hydrogen bonding interactions. We have also studied the crystal structures of the same set of compounds where F is replaced by Br or Cl. The altered packing modes of these molecules show that aromatic C-F groups provide significant interactions to stabilize crystal packing. These crystal structures have been studied at different temperatures to get an indication about the nature (attractive or repulsive) of these interactions. The salient features of our recent studies will be presented and the significance of weak interactions offered by the so called "organic fluorine" will be highlighted.

[1] (a) J.D. Dunitz, R. Taylor. *Chem. Eur. J.* **1997**, *3*, 89-98, (b) J A.K. Howard, V.J. Hoy, D. O'Hagan, G.T. Smith, *Tetrahedron* **1996**, *52*, 12613-12622, (c) V.R. Thalladi, H.C. Weiss, D. Bläser, R. Boese, A. Nangia, G.R. Desiraju, *J. Am. Chem. Soc.* **1998**, *120*, 8702-8710. [2] (a) G.R. Desiraju, R. Partasarathy, *J. Am. Chem. Soc.* **1989**, *111*, 8725-8726, (b) L. Shimoni, J.P. Glusker, *Struct. Chem*. **1994**, *5*, 383397. [3] (a) A.R. Choudhury, U.K. Urs, P.S. Smith, R. Goddard, J.A.K. Howard, T.N. Guru Row, *J. Mol. Struct*. **2002**, *641*, 225-232. (b) A.R. Choudhury, T.N. Guru Row, *Cryst. Growth Des.* **2004**, *4*, 47-52. (c) D. Chopra, T.N. Guru Row, *CrystEngComm* **2008**, *10*, 54-67. (d) D. Chopra, T.N. Guru Row, *CrystEngComm*, **2011**, *13*, 2175-2186, [4] (a) S. Purser, P.R. Moore, S. Swallow, V. Gouverneur, *Chem. Soc. Rev.* **2008**, *37*, 320-330, (b) Y. Benitex, A.M. Baranger, *J. Am. Chem. Soc.* **2011**, *133*, 3687-3689, (c) N.H. Campbell, D.L. Smith, A.P. Reszka, S. Neidle, D. O'Hagan, *Org. Biomol. Chem.* **2011**, *9*, 1328-1331, (d) C. Dalvit, A. Vulpetti, *ChemMedChem*, **2011**, *6*, 104-14.

Keywords: halogen interactions, disorder, crystal engineering

MS73.P05

Acta Cryst. (2011) A**67,** C662-C663

Subtle Interplay of Hydrogen Bonds and Weak Interactions in Drugs

Maheswararao Karanam and Angshuman Roy Choudhury, *Indian Institute of Science Education and Research Mohali, Sector 81, S. A. S. Nagar, Manauli PO, Punjab, (India), 140306.* E-mail: maheswararao@iisermohali.ac.in.

Classical hydrogen bonds [1] are known to take the leading role in generating the supramolecular assemblies, both in the solution and in the solid state and influence the formation of various crystal lattices of all organic, organometallic and biological macromolecules [2]. Weak