

formula $C_{19}H_{22}F N_3 O_3 \cdot 6(H_2O)$ which crystallizes in a triclinic crystal system with space group $P-1$. Water molecules of the network array of hydrogen bonding interactions of the type O-H...H with distances that are within the range of 1.93 a 2.23 Å and as a few examples, in this case shows that some water molecules have three hydrogen bonds at the same time, which favors a two-dimensional crystal growth with the *motif* $R^6_6(12)$, giving rise to the supramolecular arrangement in a lamina.

In the dimeric complex of copper acetate (II) with molecular formula $C_8 H_{16} Cu_2 O_{10}$, have a monoclinic crystal system with space group $C 2/c$, in this compound the water molecules establish three hydrogen bonds in the range of 2.03 to 2.58 Å, giving rise to *motif* $R^2_2(8)$ mainly. While for crystals of $[Fe(H_2O)_6]SO_4 \cdot 2(H_2O)$ (II) with molecular formula $Fe H_{12} O_6 \cdot 2(SO_4) \cdot 2(H_2O)$ crystallizing in a monoclinic crystal system with space group $P 21/n$; the large number of water molecules present in this complex supports a large number of such interactions O-H...H with hydrogen bond lengths are in the range of 1.85 to 2.19 Å.

In a global analysis of data from X-ray diffraction, is that the magnitude of the hydrogen bonds are present in the energy range of 4 to 15 Kcal, being strong bonds with weak covalence and dominant electrostatic contribution, these properties are required for the design and application engineering of crystals and / or molecular devices

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Structural analysis of aziridine-2-methanol derivatives

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Aziridines are widely used as versatile building blocks for the synthesis of a variety of biologically and pharmaceutically important molecules [1]. Among the available methodologies for the preparation of functionalised aziridines, the lithiation/trapping sequence of simple parent aziridines is growing in importance [2]. Present work reports the results concerning the structural study of aziridine-2-methanol derivatives by dynamic nuclear magnetic resonance (DMNR) [3] and single crystal X-ray diffraction. A careful examination of the aziridine 2 (see figure 1) by NMR data, shows that at room temperature the meta protons and the protons of the two ortho methyl groups of the mesityl ring gives featureless lumps, likely as consequence of a restricted rotation around the Csp^3-Csp^2 bond between the carbinolic carbon and the ipso carbon of the mesityl ring. Also in the solid state, an almost identical arrangement of the mesityl ring, with respect to the other two phenyl rings, has been observed. In this case, the aziridine nitrogen substituent was found in a syn relationship with respect to the carbinolic group so preventing the possibility of forming a hydrogen bond with the hydroxyl group. This evidence underlines a slow rotation of the mesityl group instead of a nitrogen inversion as usually is expected. By X-ray analysis of 3 and *diast-3* (figure 1), it is estimated that the nitrogen substituents set on the opposite side with respect the carbinolic carbon. *Via* study of crystal structures, the presence of hydrogen bonds between the hydroxyl group and the aziridine nitrogen lone pair was ascertained for aziridine 3 but not for *diast-3* (figure 1). Such

a hydrogen bond, which is persistent also in solution, could prevent the formation of conformational diastereoisomers by rotation around the bond between the carbinolic carbon and the aziridine quaternary carbon. This hypothesis has been demonstrated by

NMR experiments. This investigation allowed us to calculate some Csp^2-Csp^3 and Csp^3-Csp^3 rotational barriers and highlight a sort of “geared” rotation between aryl and alkyl substituents; this is possible if the structure is not in a locked conformation by an hydrogen bond as demonstrated by NMR and X-ray diffraction.

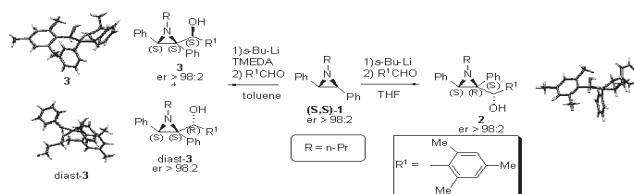


Figure 1

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Proton migration in molecular complexes of urea and its derivatives

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Systems showing proton disorder, transfer and migration are an area of much interest in recent times with widespread implications for areas such as ferroelectrics and enzyme action. These often subtle processes can affect the properties of systems and are often observed in materials containing short strong hydrogen bonds. They can be probed using variable temperature diffraction experiments. We aim to identify and characterise systems that show such effects, with a view to predicting and controlling this potentially “tunable” behaviour. These processes have previously been observed in complexes of urea [1].

Single crystal X-ray and neutron diffraction have been used to study molecular complexes of urea and its derivatives with a particular focus on characterising the hydrogen atom behaviour. The proton behaviour is monitored over variable temperatures, with the aim of observing movement of the proton with changing temperature. A limited number of such mobile proton effects have been observed using variable temperature diffraction measurements, and this project aims to build on these observations by extending these investigations to further, related, systems. The instrument used for the neutron studies is the single crystal Laue diffractometer VIVALDI at the ILL in Grenoble which is ideal for use here due to its potential for high throughput single crystal diffraction of small samples.

Temperature dependent proton migration in the 2:1 complex of N,N-dimethylurea and oxalic acid has been observed by single crystal X-ray and neutron diffraction data confirming the existence of this phenomenon. Accurate determination of the hydrogen atom parameters in this system is vital in understanding this phenomenon and also in explaining a single-crystal to single-crystal phase transition