

MS34-P14 3-pyrroline hydrates: *in situ* crystallization and structural investigations

Patryk Rzepinski¹, Lukasz Dobrzycki¹, Michał K. Cyrański¹,
Roland Boese¹

1. Czocharlski Laboratory of Advanced Crystal Engineering,
Faculty of Chemistry, University of Warsaw, Żwirki i Wigury 101,
02-093 Warsaw, Poland

email: przepinski@chem.uw.edu.pl

3-pyrroline (2,5-dihydropyrrole, C₄H₇N) is a cyclic amine, liquid at ambient conditions. Using the *in situ* crystallization technique [1] assisted by IR laser focused radiation we obtained two hydrates of 3-pyrroline (three- and hexahydrate) presented in the figure below. The trihydrate crystallizes in *P2₁/c* space group (*V*=690 Å³) with H₂O molecules forming layers. The amine molecules are attached to these layers via N...O hydrogen bonds. The hexahydrate belonging to *P2₁/m* space group (*V*=538 Å³) contains 3D network of interacting water molecules. In the structure the amine molecules are incorporated to this network thus the hexahydrate is example of semiclatrate [2]. This structure is isostructural with hexahydrate of pyrrolidine [3] - saturated analogue of 3pyrroline. Contrary to the pyrrolidine hexahydrate however, corresponding structure with 3-pyrroline does not undergo order-disorder phase transition. In all presented structures containing 3-pyrroline water molecules are disordered what manifests in alternative positions of hydrogen atoms similarly like in the hexagonal ice I_h crystal structure.

Acknowledgements The work has been supported by the National Science Center grant (NCN 2011/03/B/ST4/02591).

References

- [1] Boese, R. (2014). *Z. Kristallogr.*, **229**, 595.
- [2] Fowler, D. L., Loebenstein, W. V., Pall, D. B., Kraus, C. A. (1940). *J. Am. Chem. Soc.*, **62**, 1.
- [3] Dobrzycki, Ł., Taraszewska, P., Boese, R., Cyrański, M. K. (2015). *Crystal Growth & Design*, **15**, 4804.

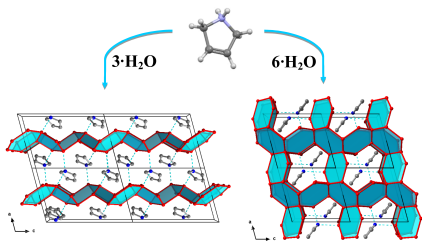


Figure 1. Packing diagrams of pyrroline tri- and hexahydrate

Keywords: Hydrates, Clathrates, 3-pyrroline, *in situ* crystallization, Crystal Engineering, Single Crystal Diffraction, Raman Spectroscopy

MS34-P15 Separation of alcohol isomers by Host-Guest Chemistry

Nicole Sykes¹, Luigi R. Nassimbeni¹, Hong Su¹, Susan A.
Bourne¹, Edwin Weber²

1. University of Cape Town
2. Technische Universität Bergakademie Freiberg

email: nixsykes@gmail.com

Host-Guest chemistry is a useful separation technique when the components of a targeted mixture have similar boiling points, rendering distillation inefficient.

We have chosen pairs of alcohols from a total of 14 isomers of propanol (2 isomers), butanol (4 isomers) and pentanol (8 isomers), which have close normal boiling points. Starting with equimolar mixtures of the selected pairs, we have exposed the guest mixtures to diol host compounds (3,3'-bis(9-hydroxy-9-fluorenyl)-2,2'-binaphthyl and 9,9'-(Biphenyl-2,2'-diyl)difluoren-9-ol).

The crystal structures of the inclusion compounds with the single alcohol isomer and that of the targeted mixture have been elucidated. The non-bonded interactions in the structures have been analysed with the programme Crystal Explorer and the thermal characteristics of all the crystalline compounds have been measured by thermal gravimetry (TG) and differential scanning calorimetry (DSC).

Selectivity curves and the effect of multiple hosts have been analysed in order to understand the effect of changes to the mother liquors from which the crystals were grown.

The analysis of the percentage of each guest which was entrapped, was obtained from the refined crystal structures and confirmed independently by NMR spectroscopy and gas-liquid chromatography.

Keywords: host-guest, isomer, selectivity, inclusion, alcohols