Structural chemistry of piperidine hydrates

Pawel Socha1, Łukasz Dobrzycki1, Michał K. Cyrański1, Roland Boese1

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

e-mail: psocha@chem.uw.edu.pl

The aim of this report was to investigate the possibility of creating hydrates or clathrates for piperidine-water system. Piperidine is a heterocyclic aliphatic amine with six-membered ring. Crystal structure of the amine is known, molecules build columns in crystal lattice [1]. Hydrates were crystalized on the diffractometer, using in situ crystallisation technique with IR laser [2]. Five hydrates have been received during the project, consisting of 0.50, 2.00, 8.10, 9.75 and 11.00 water per one amine molecule. Structures of hydrates were determined with single crystal X-ray diffraction. Interestingly, hydrates with high concentration of water were very similar to gas clatrathates [3], however there were hydrogen bonds between amine and water in crystal lattice and the water framework including positions of H2O molecules was disordered. What is more, piperidine hydrates consisting 9.75 and 11.00 water were isostructural with an analogue structure of tert-butylamine hydrates [4, 5]. Packing diagrams of these two structures are presented in the Figure below. Moreover, piperidine hydrate 8.10 were isostructural with iso-propylamine hydrate 8.00 [6, 7]. All synthesised crystals were also characterised with Raman spectroscopy and X-ray powder diffraction.

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

References

Keywords: Hydrates, Clathrates, Piperidine, in situ crystallization, Crystal Engineering, Single Crystal Diffraction, Raman Spectroscopy

Figure 1. Unit cell content of 9.75 and 11.00 hydrates of piperidine. Alternative positions of H2O molecules indicating disorder of water framework are presented as yellow spheres.