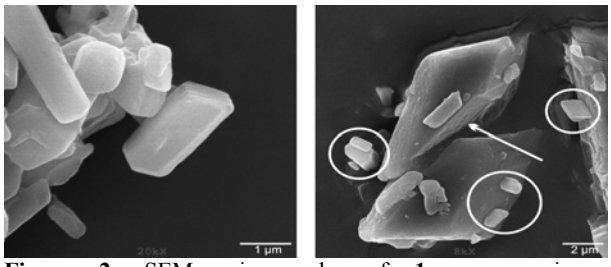


Figure 2. SEM micrographs of **1** grown via sonocrystallization: (a) before and (b) after photoreaction. Circles show intact crystals, while the arrow shows crack in a large crystal.

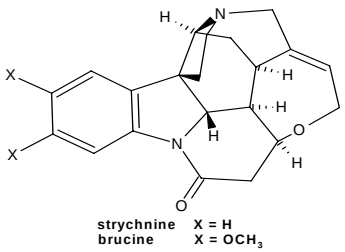
[1] Takahashi, S.; Miura, H.; Kasai, H.; Okada, S.; Oikawa, H.; Nakanishi, H. *J. Am. Chem. Soc.*, 2002, *124*, 10944.
[2] D.-K. Bučar, L. R. MacGillivray *J. Am. Chem. Soc.*, 2007, *129*, 32.

MS15 P04

Racemic resolution of *N*-protected DL-amino acids by crystallization of brucinium and strychninium diastereomeric salts. Agata Białońska, Zbigniew Ciunik *Faculty of Chemistry, University of Wrocław.Wrocław, Poland.* E-mail: bialonsk@eto.wchuwr.pl

Keywords: self-assembly supramolecular chemistry, recognition molecular, hydrogen bonding

Racemic resolution by fractional crystallization of diastereomeric salts remains one of the most useful methods in endeavor to obtain pure optically active compounds [1]. Therefore, it seems peculiar that only one pair of crystal structures of brucinium salts, as a result of racemic resolution, is known [2]. It seems to be a high selective for a given enantiomer, and reveals a low selectivity for various compounds (CSD, V. 5.27, 2006) [3]. For a given N-benzoyl-amino acid, there is known only one brucinium or strychninium diastereomeric salt. Pairs of crystals of brucinium or strychninium diastereomeric salts of *N*-4-nitrobenzoyl-D- and of *N*-4-nitrobenzoyl-L-amino acids, as well as, solid solutions have been obtained. Similarly, pairs of the diastereomeric salts of *N*-3,5-dinitrobenzoyl-D- and *N*-3,5-dinitrobenzoyl-L-amino acids have crystallized. In some other cases, strychnine and brucine appear an ineffective resolving agents in racemic resolution of *N*-3,5-dinitrobenzoyl-DL-amino acids, and the crystals of the double salts have been obtained. Thus, the selectivity of strychnine and brucine for a given enantiomer decrease in series of racemic resolution of *N*-benzoyl- > *N*-4-nitrobenzoyl- > *N*-3,5-dinitrobenzoyl-DL-amino acids. As far as we can see, there are the first cases of solid solutions of brucinium salts, as well as, of double brucinium salts, for which structures were determined.



In most reported crystals containing brucine or strychnine moieties, among many only few donors participate in weak C-H...O and C-H...π hydrogen bonds stabilizing common alkaloid self-assemblies. Depending on alkaloid self-assemblies, various active sites are formed at their surface. The active sites, by recognition of suitable part of anions or solvent molecules, provide appropriate surfaces for interactions in hydrophilic environment, from which the brucinium or strychninium salts were crystallized. Donor/acceptor capabilities of surfaces of alkaloid self-assemblies are related to donor/acceptor properties of resolved compound [4] or solvent [5]. Chiral discrimination depends on the nature of hydrogen bonds networks, involving resolving agent, solvent molecules and tertiary amine N atom.

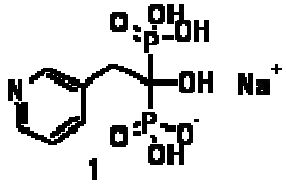
[1] Jacques J., Collet A., Wilen S.H., *Enantiomers, Racemates and Resolutions*, Krieger Publishing Company, Malabar; Florida, 1991.
[2] Kuwata S., Tanaka J., Onda N., Yamada T., Miyazawa T., Sugiura M., In Y., Doi M., Inoue M., Ishida T., *Bull. Chem. Soc. Jpn.*, 1993, 66, 1501.
[3] Allen F.H., *Acta Cryst.*, 2002, B58, 380.
[4] Białońska A., Ciunik Z., Cryst. Eng. Comm., 2004, 6, 276,
[5] Białońska A., Ciunik Z., Acta Cryst., 2004, C60, o853.

MS15 P05

Crystal structures of the osteoporosis drug risedronate
J. Brüning[a], E. Alig[a], B. Nachtsheim[b], M. Bolte[a], and M. U. Schmidt[a] [a]*Institute of Inorganic and Analytical Chemistry,* [b]*Institute of Organic Chemistry and Chemical Biology*, [a,b]*University of Frankfurt, Max-von-Laue-Str.7, 60438 Frankfurt am Main, Germany*.
E-mail: bruening@chemie.uni-frankfurt.de

Keywords: risedronate, crystal structure, polymorphism

Risedronate (**1**) is a pyridinyl bisphosphonate inhibiting the osteoclast-mediated bone resorption. It is the most effective drug against osteoporosis up to now [1,2].



During a polymorph screening with over 100 experiments using different temperatures and solvents (e.g. dimethylsulf-oxide, N-methyl-pyrrolidone, alcohols, ethers and esters), we found 11 polymorphic and pseudopolymorphic forms, which we identified and characterised by X-ray powder diffraction and differential thermal analysis. 3 crystal structures of **1** were determined by single crystal X-ray diffraction.

[1] Diez-Perez A., *Maturitas*, 2002, 43, 19.
[2] Redman-Furey, N. et al., *J. Pharm. Sci.*, 2005, 94, 893.