Session VI, Wednesday, September 14

L19

HOW TO GROW CRYSTALS OF BIOLOGICAL MACROMOLECULES?

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Crystallization of proteins is a multiparameter process that is very difficult predictable. Crystallization process is still the limiting step to obtain accurate 3D model of proteins by the means of X-ray (neutron) diffraction technique. The number of parameters affecting nucleation and growing of well-ordered crystals is too high and its prediction entails a degree of complexity comparable to the simulation of the protein-folding process.

There are several techniques that can be used to drive system into the supersaturation region, but mainly three of them are used the most frequently: batch, vapor diffusion and liquid-liquid diffusion [1]. Using of different crystallization techniques should be considered when trying to improve crystal quality.

Crystallization procedures and parameters affecting crystal growth will be discussed during the lecture.

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SL6

BIO-SAXS - BIOLOGICAL SMALL ANGLE X-RAY SCATTERING SERVICES AT CEITEC-MU

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The Core facility X-ray diffraction and Bio-SAXS of the CEITEC-MU located in Brno was inaugurated in September 2013. Since then, the core facility (CF) offers access to the monocrystal X-ray diffractometers and Bio-SAXS Kratky camera system. The list of CF services includes collection of diffraction and scattering data, X-ray structure determination, Bio-SAXS data analysis, long-term cryogenic storage of crystals or hardware support for synchrotron trips.

Here, selected examples from users community illustrates the possibilities of SAXS data analysis on biological samples as ab initio shape reconstruction, quaternary structure model building of macromolecular complexes and studies of intrinsically disordered proteins. The complex formation of an intrinsically disordered protein and telomeric DNA was chosen as a case study not straightforward to analyse by other methods of structural analysis. Two-phase ab initio SAXS-based modeling was used to reconstruct the shape of telomeric repeat-binding factor with telomeric DNA duplex.

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SL7

THE CRYSTALLOGRAPHY OF SUPRAMOLECULAR COMPLEXES BASED ON BAMBUSURIL, CUCURBITURIL AND SIMILAR MACROCYCLES

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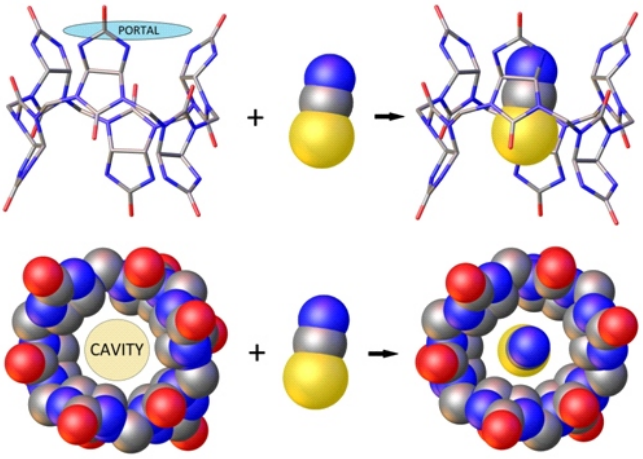
Host-guest complexes are supramolecular entities in which molecules are held together by non-covalent interactions; their formation, properties, and applications arouse considerable scientific interest. In case of inclusion compounds, the formation of the host-guest complex depends mainly on the interior of the host cavity and properties of the portals, whereas the exterior of the host influences properties such as solubility, interaction with the environment etc. Glycoluril-based macrocycles, such as bambus[n]urils and cucurbit[n]urils and their derivatives are popular hosts for

such inclusion compounds and are often studied as potential signaling molecules, catalysts, pharmaceutical transporters and "preservatives", or models for biological processes occurring at the surface of the membranes.

For this type of complexes, X-ray crystallography (among various NMR experiments) is usually the preferred method of structural analysis. However, the crystals usually have large unit cells with considerable quantities of solvents, which makes them somewhat similar to protein crystals regarding the data collection and refinement. Defects in crystal structure, such as a disorder in the solvent "area" around the hosts do occur quite often. A chemist is usually interested in remaining "non-solvent" parts of the structure, which means that some simplifications can be introduced. Practical aspects of X-ray diffraction studies on selected inclusion compounds of glycoluril-based macrocycles will be discussed in greater detail.



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Session VII, Wednesday, September 14

SL8

SOME NEW FINDINGS ABOUT THE POLYTYPISM OF THE MINERAL CRONSTEDTITE

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Polytypism of layer silicate cronstedtite ($Fe^{2+}_{3-x}Fe^{3+}_{x}$)($Si_{2-x}Fe^{3+}_{x}$)$O_5(OH)_4$, ($0.5 < x < 0.8$) was studied in numerous specimens from localities Eisleben (Germany), Pohled (Czech rep.) [1], Nižná Slaná (Slovakia) [2] by the four circle X-ray diffractometer with area detector. In addition, EMPA analyses of selected samples were done. Synthetic micrometer-size crystals were studied by electron diffraction tomography (EDT) [3]. These methods provide precession-like images of reciprocal lattice planes, relevant for the determination of OD subfamilies (A, B, C, D), and polytypes.

The rare 1*M* polytype (subfamily A, $a = 5.5033(3)$, $b = 9.5289(6)$, $c = 7.3328(5)$ Å, $= 104.493(7)$, space group *Cm*) was found as dominant in the synthetic run product [3]. A rare crystal from Eisleben allowed data collection and structure refinement [4]. More frequently it occurs in mixed crystals with 3*T*, another, more abundant polytype of the subfamily A ($a = 5.499(2)$, $c = 21.260(8)$ Å, space group $P3_1$). Such crystals were found in Pohled and Nižná Slaná samples. The newly discovered non-MDO polytype $6T_2$ (subfamily A, $a = 5.4976(3)$, $c = 42.601(1)$ Å, space group $P3_1$) was found in Pohled [1]. Its structure was refined and stacking sequence determined [5]. Another rare $2M_1$ polytype (subfamily A, $a = 5.497(2)$, $b = 9.507(2)$, $c = 14.267(6)$ Å, $= 97.25(3)$, space group *Cc*) occurs in Pohled in mixed crystals with $6T_2$, and in synthetic samples either with 3*T*, or isolated.

Polytypes of the subfamily A can be affected by twinning by reticular merohedry. The twinning operation - ($(2n\text{-}1) \times 60$ rotation parallel to the threefold axis) exchanges obverse/reverse settings of the *R* lattice of the rhombohedral subfamily A structure. The twinned crystals are common in Nižná Slaná (3*T* polytype), and rare in Pohled ($6T_2$, $3T + 1M$).

Mixed crystals of polytypes $2H_1 + 2H_2$, subfamily D, (lattice parameters of both polytypes $a = 5.5002(4)$, $c = 14.195(1)$ °, space groups: $P6_3cm$ ($2H_1$), $P6_3$ ($2H_2$)) were identified in Pohled, with various $2H_1/2H_2$ proportions. Almost pure $2H_1$ crystal was found in Nižná Slaná, and a totally disordered subfamily D crystal in the synthetic product.