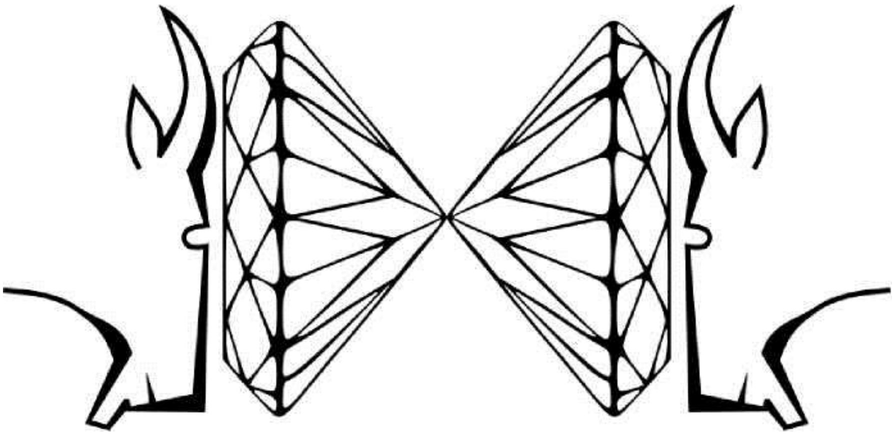
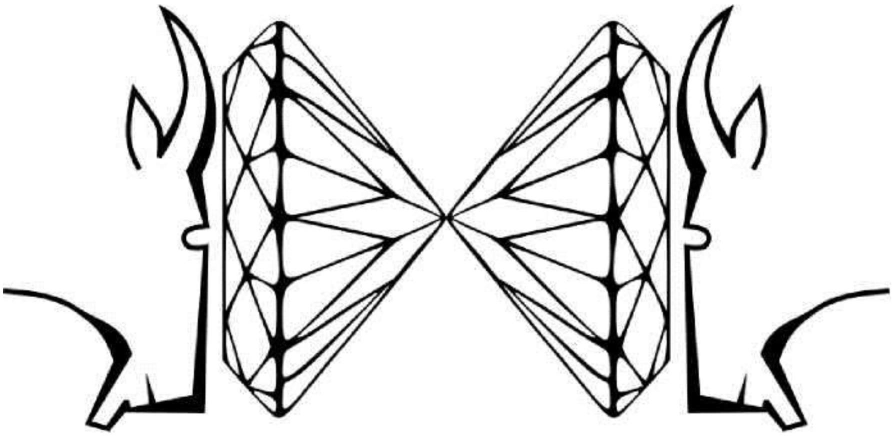


8TH FROLIC GOATS
WORKSHOP ON HIGH PRESSURE DIFFRACTION

Abstract Book

Poznań, 26-28 April 2015



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Welcoming address

The 8th Frolic Goats Workshop on High-Pressure Diffractometry continues the mission of disseminating the application of extreme conditions in crystal chemistry, physics and biology. Of various thermodynamical conditions for many years pressure remained most neglected to due complicated experimental and hence expensive, for equipment required diffraction measurements. However, after the advent of diamond-anvil cell (DAC), pressure calibration by ruby-fluorescence and 2-dimensional x-ray detectors, the high-pressure experimental become cheaper and easier. During the Workshop we will explain the operation of the most powerful tool for modifying crystal environment. For nearly three decades it has been our aim to facilitate high-pressure diffraction experiments. They consist of several stages, involving the handling and alignment of the DAC, its loading, pressure calibration and running the diffractometer measurement in an optimal way for high-pressure experiments. During our Workshop the high-pressure techniques will be explained and practiced. Apart from theoretical lectures, the specific tasks of handling the DAC can be performed together with experienced colleagues. But most importantly, it was our intention to create a forum of high- pressure scientists for direct exchange of experience, views and discussion.

Traditionally, the Workshop includes a social event, for establishing informal ties between the participants. This year, the young group of organizers decided to visit and better learn the historical beautiful city of Poznań, but not in a traditional way. Instead of plain sightseeing we would like to engage you in the Urban Game. Divided into groups, you will try to decrypt a message from Clues located in the city center.

This Frolic Goats Workshop to be fully held in the Morasko Campus in the new Collegium Chemicum building and for the first time we will also use the Wielkopolska Center for Advanced Technologies . Finally, it is my pleasure to thank all the sponsors of the Workshop, the Faculty of Chemistry, Olympus Polska for financial support.

I also thank all the members of the Organizing Committee and all teachers in the Workshop for their efforts.

Wishing you the fruitful sessions,

Andrzej Katrusiak

Scientific programme

Sunday, 26 April 2015

13^{30}		Urban Game

Monday, 27 April 2015 Wlkp. *Center for Advanced Technology, Umultowska 89c Morasko Campus*

8^{30}		*Registration, room 111-5*
9^{00}	A. Katrusiak	*Opening Ceremony*
9^{05}	M. Szafrański	*High-pressure studies of physical properties of solids*
9^{50}	E. Patyk	*α-D-glucose transformations at high pressure*
10^{10}	M. Kaźmierczak	Strong and weak Interactions
10^{30}	D. Pinkowicz	Photomagnetic behavior at extreme conditions: high pressure, low temperature, intense illumination
11^{00}	*Coffee break*	
11^{30}	P. Guńka	*High-pressure structural study of cubic As2O3: helium clathrate of arsenolite*
12^{00}	J. Marciniak	*High-pressure solvate of Z'>1 compound*
12^{20}	M. Anioła	*High-pressure reactions of 4,4'-bipyridine hydrobromide*
12^{40}	H. Tomkowiak	*Pressure-induced transformations in thiourea*
13^{00}	A. Gładysiak	*Negative area compressibility in a porous material*
13^{20}	M. Andrzejewski	*Synthesis and structural characterization of two novel cobalt-organic frameworks*
13^{40}	K. Rajewski	*High-pressure studies of 2,4,5-tribromo and 2,4,5-triiodoimidazoles*
14^{00}	*Poster session and lunch*	

Tuesday, 28 April 2015 Collegium Chemicum Novum, Umultowska 89b, room 2.42

9^{30}		*Laboratory exercises: DAC construction, alignment, loading, diffraction experiment - room 2.42, Collegium Chemicum, Morasko Campus*

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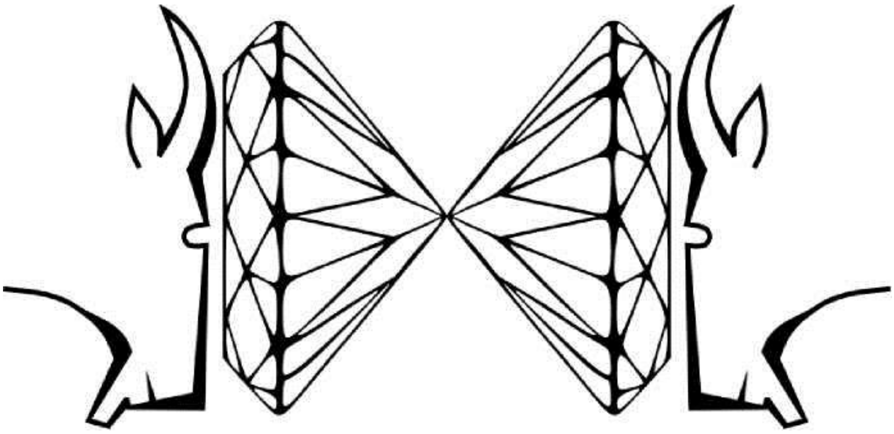

Lectures

High-pressure studies of physical properties of solids

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High-pressure studies of physical properties of solids require specific experimental techniques. In particular, an apparatus to generate pressure in large volume is often desirable for such applications. Here we present and shortly characterise an experimental set up designed for high-pressure measurements on macroscopic samples. The exemplary experimental results are shown and discussed with an emphasis on information which can be derived from the high-pressure dielectric spectroscopy and calorimetry.

α-D-Glucose transformations at high pressure

Ewa Patyk[1]

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Glucose is one of the most significant representative of carbohydrates. It is not only energy carrier in human organism but also takes part in many important biochemical processes. It is thanks to its unique structural pattern of hydroxyl groups and ability to create multiple intermolecular contacts that it can be recognized and processed by enzymes. It was proven for (+)-sucrose, that the solid-state preferences of bonding pattern can be significantly influenced by high pressure [1]. Similar study was carried out for α-D-glucose crystals [2] proving its high-pressure behavior to be similar to (+)-sucrose.

Crystals of α-D-glucose were mounted in the Merrill-Bassett diamond-anvil cell (DAC) [3] and compressed. 4-Circle diffractometer was used for sample measurement in the 0.2 – 6.2 GPa pressure range.

Isostructural phase transition of α-D-glucose was show in discontinuity of its unit-cell volume and parameters at 5.40 GPa. Orthorhombic symmetry, space group $P2_12_12_1$ (Z'=1), of α-D-glucose crystals is preserved on transformation from phase I to phase II, but other changes occur. Compression leads to rearrangement of molecular aggregation enforcing modification of intermolecular contacts pattern. Number of O-H···O hydrogen-bonds decrease above 5.4 GPa, showing how these strong interactions diminish their role. At the same time, weaker C-H···O contacts increase their number and therefore importance in phase II.

High-pressure behavior of α-D-glucose is to some point similar to one reported for (+)-sucrose. It makes us believe that structural similarities of carbohydrates can have an influence on the pressure-induced transformations of carbohydrate crystals.

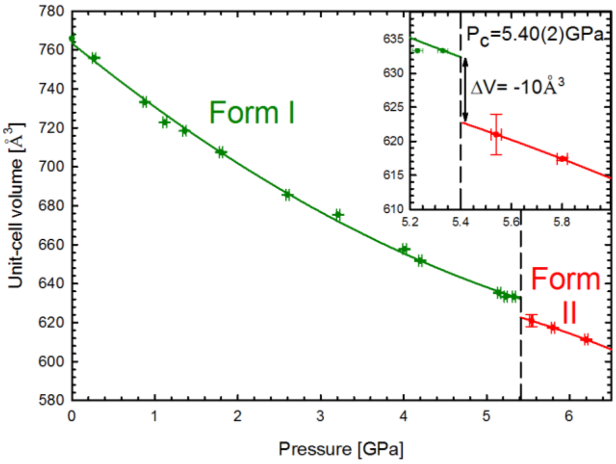

***Fig. 1** Pressure dependence of unit-cell volume of α-D-glucose Phase I and II, marked in green and red, respectively. Critical pressure for phase transition (Pc) is marked with vertical dashed line.*

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Strong and weak Interactions

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Intermolecular contacts are ones of the most important factor responsible for building crystal structure. Strong and weak contacts have different influence on cohesion forces, therefore so significant is to analyse its separately. Beside computation methods [1],[2] statistic method can be also used [3].

Distribution of the shortest intermolecular interactions according to the van der Waals radii obtained by survey of organic and metalorganic crystal structures deposited in Cambridge Structural Database (CSD) [4] have clearly bimodal character shown in Fig. 1.

Distribution maxima of specific types interactions falls into peak I (weak interactions) or peak II (strong interactions). Integration of two area separated by gap shown that weak interactions are twice more common as shortest intermolecular interactions.

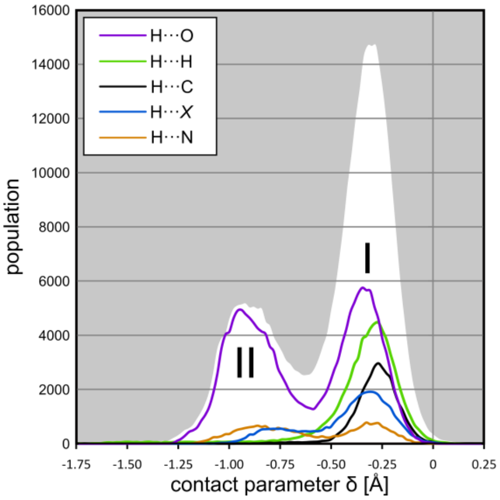

Fig. 1. *Most popular types of shortest contacts in crystal structures deposited in CSD.*

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Photomagnetic behavior at extreme conditions: high pressure, low temperature, intense illumination

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B. Sieklucka[1]

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Multifunctional solids are currently a hot topic within the chemistry, physics and materials science due to their application potential. One of the most exciting classes of such compounds are photomagnets, which exhibit photo-induced magnetization changes related to the ‘generation of magnetic moments’ using light [1]. Despite some progress in this field, investigation of the photomagnetic effect still remains underdeveloped. In the following contribution it will be demonstrated that photomagnetism can be enforced in a non-photomagnetic compound by adjusting the thermodynamic conditions [2]. The non-photomagnetic cyano-bridged $\{[Fe^{II}(pyrazole)_4]_2[Nb^{IV}(CN)_8]\cdot 4H_2O\}_n$ coordination polymer [3] has been subjected to high-pressure structural, magnetic and photomagnetic studies at low temperature, which revealed a wide spectrum of functionalities including the pressure-induced photo-magnetic behavior and piezochromism. As a result the first pressure-induced photomagnet has been discovered (Figure 1).

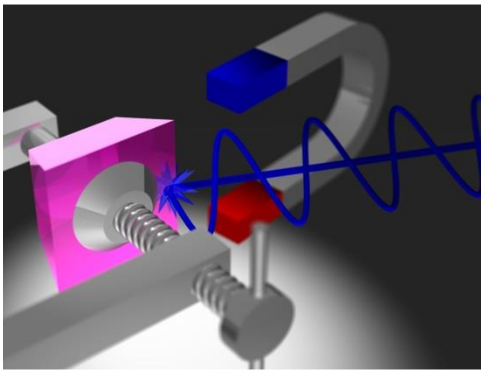

Fig. 1. *A visual representation of the pressure-induced photomagnet.*

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High-pressure structural study of cubic As2O3: helium clathrate of arsenolite

Piotr A. Guńka[1], *Kamil F. Dziubek*[2,3], *Andrzej Gładysiak*[3], *Maciej Dranka*[1], *Michael Hanfland*[4], *Andrzej Katrusiak*[3], *Janusz Zachara*[1]

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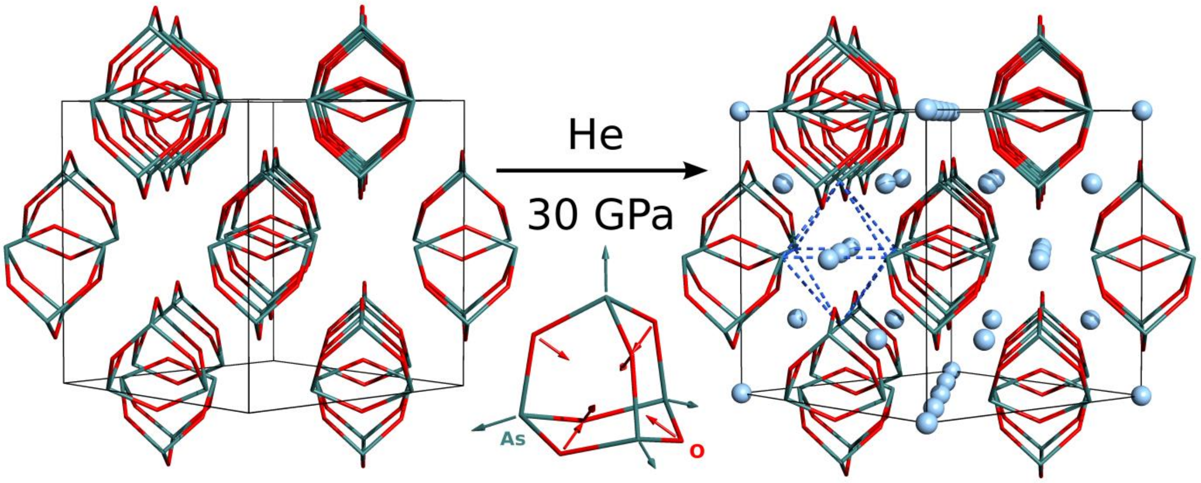
Crystal structure of arsenolite, the cubic polymorph of molecular arsenic(III) oxide, has been determined up to 30 GPa. The bulk of the crystal is monotonically compressed with no detectable anomalies to 60% of the initial volume at 30 GPa. The experimental As_4O_6 crystal compression exceeds that obtained by various theoretical models within the DFT framework. The As_4O_6 molecules are deformed toward more tetrahedral shape. Pressure above 3 GPa favors the formation of $As_4O_6 \cdot 2He$ clathrate in the surface layer increasingly deeper with pressure. Interestingly, this is the first example of helium clathrate formed *in situ* with a solid molecular oxide and a proof that helium may permeate even non-porous single crystals in high-pressure (HP) diffraction studies. This indicates it is an important and general phenomenon that needs to be taken into account when doing HP diffraction studies in helium.



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High-pressure solvate of Z’>1 compound

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Compounds with more than one symmetry-independent molecule (Z’>1) are likely to form solvates, according to the survey of solvate-unsolvate pairs structures determined so far.1 The Z’>1 implies inequivalent crystal environments of molecules; high-pressure increases the energy difference of their interactions which can destabilize the monotonic compression. The Z’ reduction to 1 or less eliminates differences in the environments of molecules. High pressure inevitably improves the close packing in the crystal through monotonic compression and phase transitions, but also by change in the mode of single- and multi-component crystallizations and can favor solvates and cocrystals. At normal conditions 2,4,5-triiodoimidazole, $C_3H_1N_2I_3$, forms crystals with Z’=3 in the structure. 2 It has been recrystallized in isobaric and isochoric conditions from methanol solution and its hemisolvate $2C_3H_1N_2I_3{\cdot}CH_3OH$ has been obtained.

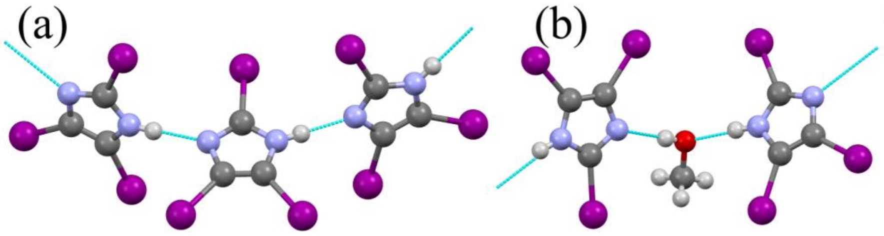

Fig. 1. *Asymmetric units of (a) unsolvated tIIm and (b) 2tIIm·*CH_3OH *solvate.*

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High-pressure reactions of 4,4’-bipyridine hydrobromide

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High pressure and temperature trigger chemical reactions of 4,4’-bipyridine hydrobromide monohydrate (44’biPyHBr·H_2O) in methanol solution. Above 0.2 GPa and 423 K 4,4’-bipyridininium dibromide salt (44’biPy2HBr) precipitates, while 4,4’-bipyridine free base remains dissolved in the methanol:water mixture, whereas at 0.35 GPa and 473 K the reaction of N-methylation of both pyridine moieties occurs and N,N-dimethylbipyridinium dibromide (44’biPy2CH_3Br) is formed. None of the high-pressure 44’biPy2HBr and 44’bPy2CH_3Br is solvated, which contrasts with the solvation of analogues 1,4-diazabicyclo[2.2.2]octane (dabco) salts: dabco2HI [1], dabcoCH_3I [1] and dabcoCH_3Br salts [2].

High-pressure experiments were performed in a Merrill-Bassett diamond-anvil cell (DAC) [3] modified by mounting the anvils directly on steel supports with conical windows. The gasket was made of tungsten foil 0.3 mm thick with a hole 0.4 mm in diameter. The DAC chamber was filled with the saturated methanol solution of 44’-bipyHBr·H_2O. For pressure calibration the ruby-fluoroscence method was used [4] and a Photon Control Spectrometer affording the precision of 0.02 GPa.

It is characteristic that in both reactions of 44’biPyHBr·H_2O the unsolvated disalt products were formed and that free base 44’biPy and H_2O were released into the solution. At ambient conditions 44’biPyHBr preferentially forms hydrate 44’biPyHBr·H_2O, whereas anhydrous dabcoHBr and dabcoHI are obtained from aqueous solution at 0.1 MPa. The dabco2HI·3CH_3OH solvate was obtained at 1.2 GPa.

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Pressure-induced transformations in thiourea

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Thiourea, $(NH_2)_2CS$, has been extensively studied at normal conditions and at various temperatures and pressures [1-5]. At normal conditions it crystallizes in orthorhombic space group Pnma, Z=4 (phase V). Below 202 K it undergoes a sequence of phase transitions between five phases – one ferroelectric and four modulated. High-pressure studies revealed that at 0.34 GPa thiourea transforms from ambient-pressure phase to phase VI (space group Pnma, Z=12). Gesi [6] postulated that there is another phase (phase VII) above 0.54 GPa and the transition was associated mainly with a dielectric anomaly. Noteworthy, we have found that at this pressure thiourea can easily form hydrates [7].

Single crystals of thiourea have been in situ grown in a Merrill-Bassett diamond-anvil cell (DAC) [8] and the structures were determined by X-ray diffraction. High-pressure crystallizations at isochoric and isothermal conditions lead to the unsolvated crystals (methanol solution) or two different hydrates (aqueous solution): $(NH_2)_2CS\cdot H_2O$ at 0.55 GPa and $(NH_2)_2CS\cdot 2/3H_2O$ at 0.70 GPa.

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Negative area compressibility in a porous material

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Most materials shrink in all directions under hydrostatic pressure; only few exceptions expand in one direction [1] and even fewer are capable of expanding in two directions when hydrostatically compressed [2–4]. Such expansion in one direction is termed negative linear compressibility (NLC). This effect is rare and usually very small in magnitude. The expansion in two directions of a compressed crystal is much less common and even weaker in magnitude, and it is known as negative area compressibility (NAC).

Here we describe a soft porous material $[Zn(L)_2(OH)_2]_n \cdot$Guest (where L is 4-(1H-naphtho[2,3-d]imidazol-1-yl)benzoate, and Guest is water or methanol), exhibiting the strongest ever observed NAC. This counterintuitive strong NAC arises from the interplay of flexible $[–Zn–O(H)–]_n$ helices with puckered layers of $[–Zn–L–]_4$ rings comprising large channel voids. The compression of helices and flattening of puckered rings combine to produce a giant piezo-mechanical response. This is the first NAC reported in MOFs and its magnitude, clearly visible and by far the highest of all known materials, can be reversibly tuned by exchanging guests adsorbed from hydrostatic fluids.

Our finding and the analogous forthcoming materials with an extremely large NAC effect can find versatile high-technology applications, for example as strain-compensating composites, smart adjusters as well as ultrasensitive sensors and actuators [5].

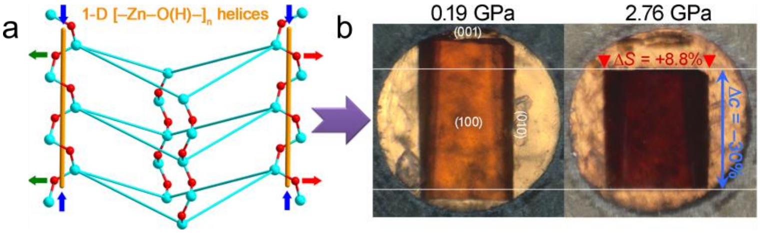

Fig. 1. *(a) Schematic view of $[Zn(L)_2(OH)_2]_n \cdot$Guest framework structure and its geometric response to hydrostatic pressure. (b) A single crystal of $[Zn(L)_2(OH)_2]_n \cdot H_2O$ hydrostatically compressed in the diamond-anvil cell.*

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Synthesis and structural characterization of two novel cobalt-organic frameworks

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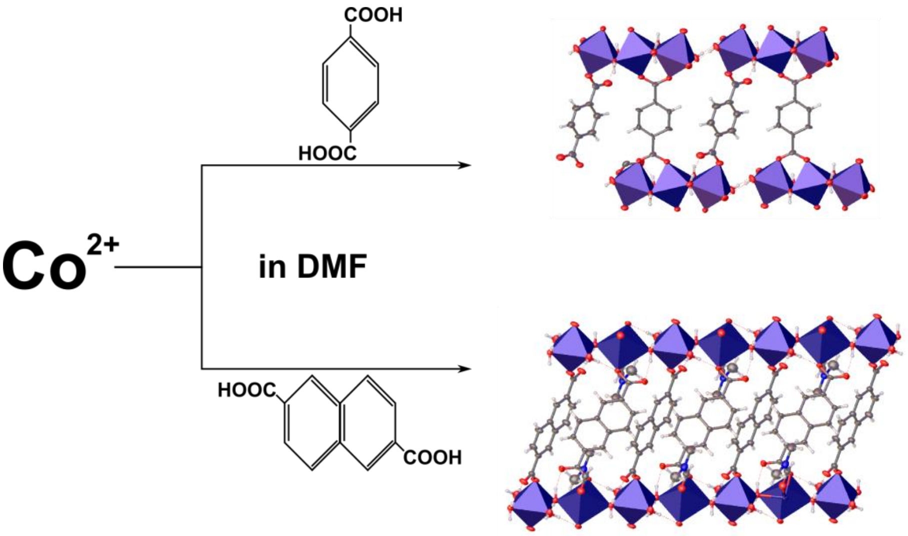
Metal-organic frameworks are under intensive investigation due to their potential applications in fields of gas adsorption, selective catalyst, magnetism, luminescence [1, 2]. There are many factors affecting synthesis of coordinating polymers like stoichiometry, thermodynamic conditions, reaction time. A small change in reaction conditions may lead to a new product with different properties. Reaction of Co2+ with terephthalic acid or 2,6-naphthalenedicarboxylic acid in DMF may give a few products depending on experimental conditions. Single crystals of these compounds may exhibit magnetic properties. As recently proved, they are ferromagnetic, antiferromagnetic or metamagnetic [3, 4]. Hence, such crystals may be applied in molecular based electronics.



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High-pressure studies of 2,4,5-tribromo and 2,4,5-triiodoimidazoles

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Switchable polarization of NH···N hydrogen bonds remains in a great interest of crystallographers due to their ferroelectric properties. This effect has been recently reported for dabco salts and halobenzimidazoles [1]. At ambient conditions imidazole crystallize in centrosymetric space group, however high-pressure noncentrosimetric β phase has been reported [2]. This suggested that imidazole derivatives can exhibit ferroelectric properties associated with the bistable NH···N bonds in polar crystals. Recently we published structures of di- and trihaloimidazoles at ambient conditions [3]. NH···N bonds lengths shortened significantly in comparison to unsubstituted imidazole (from 2.756 Å in β-imidazole at 3.5 GPa to 2.734 Å in 2,4,5-triiodoimidazole at ambient conditions). High pressure experiments led to a new high-pressure 2,4,5-triiodoimidazole ring arrangement. Phase transition is isostructural and nondestructive. Yet 2,4,5-tribiromoimidazole has no high-pressure phase transition.

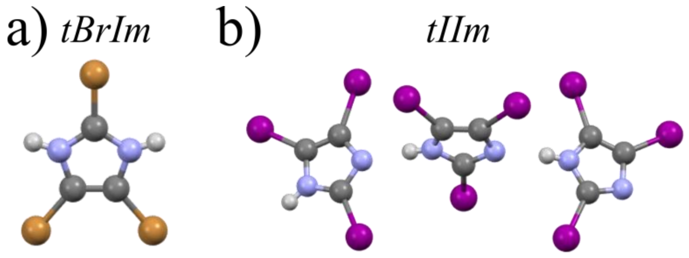

Fig. 1. *a) asymmetric unit of 2,4,5-tribromoimidazole, b) asymmetric unit of 2,4,5-triiodoimidazole.*

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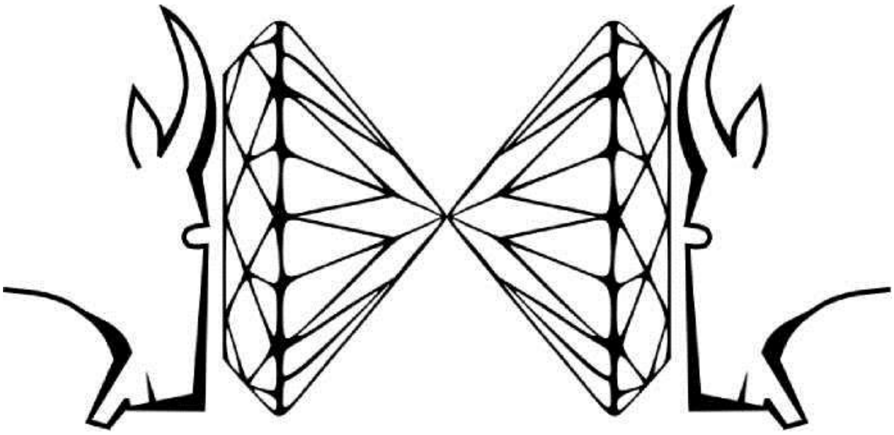

Posters

Structural studies of iodinated imidazole cocrystals

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Cocrystals formation is one of the most important challenge for crystal engineering, design of pharmaceuticals in particular [1]. Halogen-halogen interactions which can be as strong as hydrogen bonds may force a creation of new architectures and arrangements in organic molecular crystals. Iodination of imidazole can result in formation of cocrystals as a by-product after a partial substitution of an imidazole ring [2]. This effect was not observed during neither chlorination nor bromination. A new cocrystal of 2,4-diiodoimidazole:4-iodoimidazole was separated and its crystal structure was determined by X-ray diffraction techniques at 293 K/0.1 MPa. The structural analysis including the interplay between halogen-halogen interactions and NH···N hydrogen bonds was done including comparison with the known cocrystal.

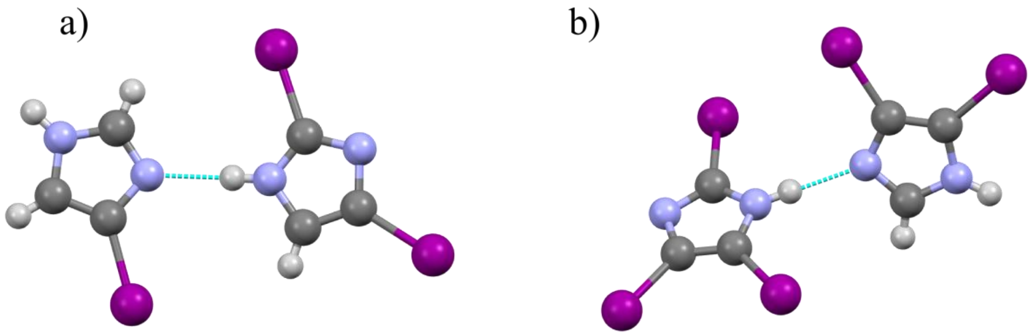

Fig. 1 *Crystal structures of iodinated imidazole cocrystals: a) 2,4-diiodoimidazole:4-iodoimidazole and b) 2,4,5-triiodoimidazole:4,5-diiodoimidazole.*

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Pressure enhanced durability of unstable solvate

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According to the CSD [1] there are 24 different benzene-1,4-diol (hydroquinone) solvates. In most of the cases a solvent molecule fills an accessible space in a clathrate like β form (~ 5% of the unit cell volume) or α form (~ 1%) leaving a host structure unaffected. There are nine examples where the solvent forces a different molecular arrangements of hydroquinone molecules. We report the hydroquinone acetone solvate (C2/c), unstable under ambient conditions, previously obtained in a glass capillary [2], presently investigated in high pressure. Experiments at high pressure were performed in a modified Merrill-Bassett diamond-anvil cell (DAC). Hydroquinone interacts with an acetone molecule via OH···O bonds forming zigzag chains along the [101] direction (Fig. 1). Moreover, there is a significant influence of pressure on the c-parameter, which shortens approximately 0.4 Å up to 0.6 GPa and at approx. 0.7 GPa becomes shorter than the b-parameter. It is possible due to an alternating layer system with acetone and hydroquinone molecules. Assuming that the surface is not affected by defects, we can suppose that the top or bottom surface consists of either A or B layer in equilibrium with the solvent – acetone. When the crystal is exposed to air, the acetone layer evaporates quickly (acetone boiling point is 329-330 K, under standard conditions). Acetone evaporates also from the bulk of the crystal, which collapses.

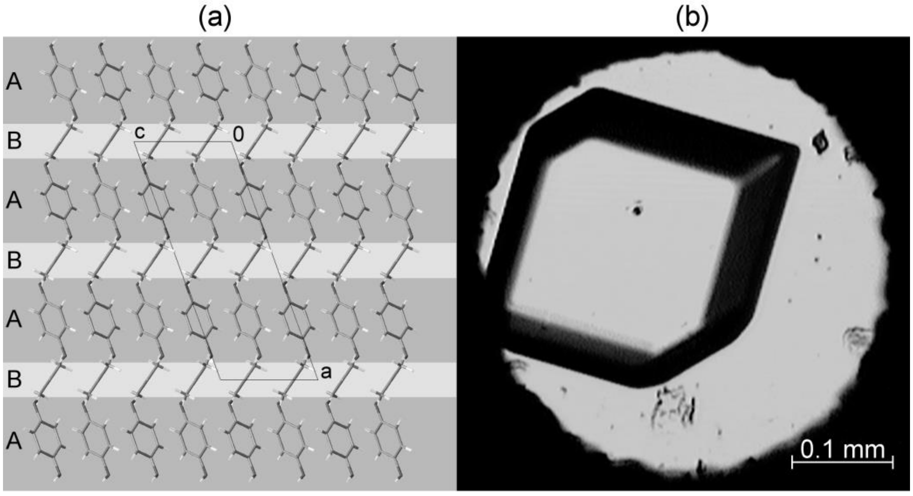

***Fig. 1** (a) Hydroquinone (A) and acetone (B) alternating layers and in the cocrystal structure, projected onto the (101) surface; (b) the cocrystal obtained at 0.63GPa, 296 K.*

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Understanding octahedral distortion factors in bis(1,4-H2-1,2,4-triazolium) pentabromidoantimonate(III)–1,4-H2-1,2,4-triazolium bromide

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The hybrid materials belonging to the family of halogenidoantimonates(III) with organic cations represent new, relatively easy to synthetize and design, generation of materials. They may be obtained *via* self-assembly of metal oxide/halide with an organic linker, in an appropriate solvent, to form the synthesis-conditions dependent three-dimensional arrangements usually showing different topologies. These materials have been extensively investigated during the past decades because of their interesting physicochemical properties and potential applications *e.g.* as ferroic materials [1,2].

The inorganic substructures of halogenidoantimoantes(III) are almost always distorted from the ideal octahedral/square pyramidal geometry. The observed differences from regularity in the bond lengths and angles are correlated with two main factors: (i) primary deformation connected with the tendency of inorganic polyhedra to share halogen atoms resulting in the formation of more complicated structural arrangements and (ii) secondary deformation that arises from the interactions of the inorganic framework with the oppositely charged organic cations. In most structures of those compounds these two factors are inseparable and it is very difficult to understand and unambiguously explain the polyhedral distortion. Bis(1,4-H_2-1,2,4-triazolium) pentabromidoantimonate(III)–1,4-H_2-1,2,4-triazolium bromide, $(C_2H_4N_3)_2[SbBr_5]\cdot(C_2H_4N_3)Br$ (BTPTB) is quite unique with its relatively strong hydrogen bonds formed to selected Br atoms. This is also one of the very few examples where hydrogen bonds are formed between organic cations giving the plausible reason for their ordering and a lack of any order-disorder phase transition characteristic for this group of crystals [3].

The poster will show that the room- and low-temperature studies of BTPTB together with the structure determination of its 'parent' simple organic 1,4-H_2-1,2,4-triazolium bromide $(C_2H_4N_3)Br$ (TB) supported by the Hirshfeld surface analysis (Fig. 1) and analysis of the data retrieved from the Cambridge Structural Database allow to characterize and explain the above mentioned distortion factors.

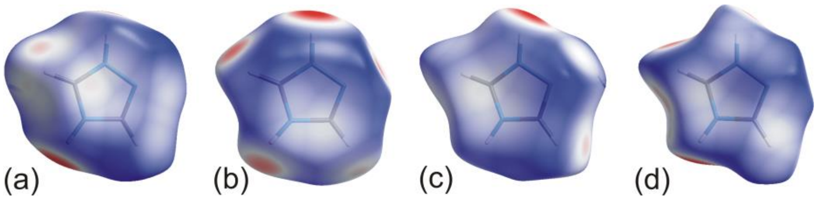

***Fig. 1** The Hirshfeld surfaces for the 1,4-H_2-1,2,4-triazolium cations in the low-temperature structures of BTPTB (a - c) and TB (d).*

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High-pressure polymorphism of β-D-mannose

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Mannose is a monosaccharide widely used in pharmaceutical industry. This epimer of glucose plays an important role in keeping urinary track in good health. Because of different, in respect to glucose, orientation of hydroxyl groups it is barely processed in human organism and almost entirely ends up in bladder. There, it is competitively bound by *Escherichia coli* leading to its removal [1]. In this respect mannose is like any other carbohydrate, owing its properties to the unique arrangement of hydroxyl groups. As in (+)-sucrose [2] and *α*-D-glucose [3] preferences of H-bonging pattern of *β*-D-mannose can be influenced by external stimuli such as high pressure. Herein we not only present crystal structure of high-pressure polymorph of *β*-D-mannose, but also unknown till now ambient-condition structure [4].

Single crystals of *β*-D-mannose were mounted in the Merrill-Bassett diamond-anvil cell (DAC)[5] and gradually compressed. X-Ray diffraction experiments were performed in the 0.48-2.85 GPa pressure range.

At 1.95 GPa *β*-D-mannose undergoes an isostructural phase transition evidenced not only by the discontinuity of its unit-cell volume and parameters but also by the significant change in crystal shape (Figure 1). Compression of *β*-D-mannose crystals forces molecules to rearrange their positions at the same time preserving its orthorhombic symmetry, space group $P2_12_12_1$ (Z'=1). Those changes are inseparably connected to transformations of H-bonding pattern and weaker intermolecular interactions.

β-D-Mannose, just like previously reported sugars, [2,3] undergoes pressure-induced phase transition, showing behavior in some respects similar to theirs and it is the most easily pressure-transformed carbohydrate studied till now.

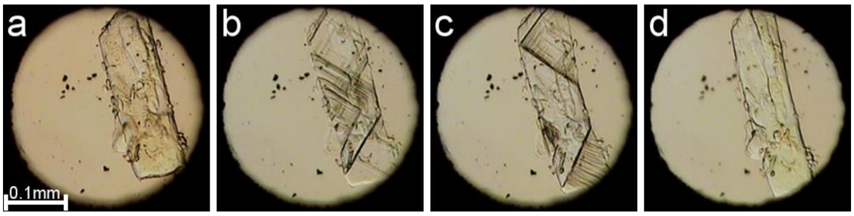

Fig. 1. *Single crystal of β-D-mannose: Phase I (a); undergoing the transition (b-c), Phase II (d).*

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Structural studies of bis(4-(trimethylammonium) benzoate) hydrogen iodide monohydrate

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4-Aminobenzoic acid (PABA) and its derivatives have variety biological activity. PABA has been called vitamin B10 or Bx, however PABA is part of vitamin B11 skeleton, benzocaine and procaine are its esters. Therefore so important investigation of other derivatives, especially in assailable for system form as salts and hydrates.

Crystal of bis(4-trimethylammonum) benzoate) hydrogen iodide monohydrate was obtained from methanol solution [1]. The diffractometric experiment was performed using Xcalibur. The main structural features were solved by direct methods of SHELXS [2].

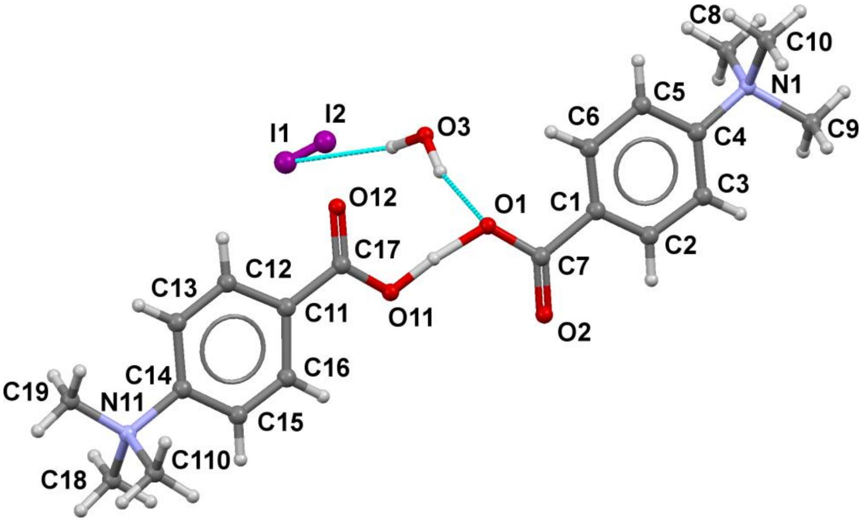

Fig. 1. *Structure of bis(4-(trimethylammonium)benzoate) hydrogen iodide monohydrate.[3] Iodide anion is located in two half-occupied positions*

References

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The course of the photochemical reaction in crystals at different pressures

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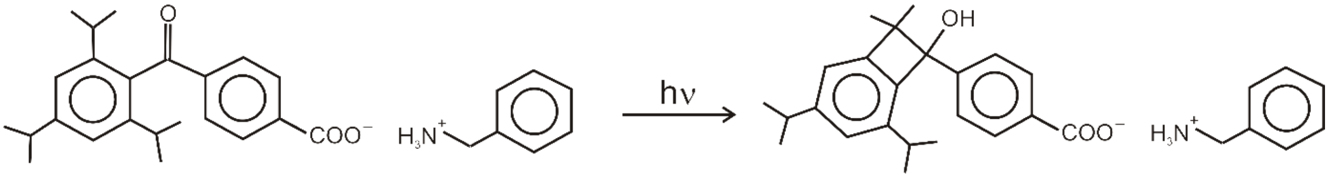
We are interested in the monitoring of structural changes brought about by photochemical reactions proceeding in crystals in ambient and extreme conditions.

Here, we will present the results of crystallographic studies of benzylammonium 4-(2,4,6-triisopropylbenzoyl)benzoate undergoing the Norrish-Yang reaction:



The influence of pressure on the molecular and crystal geometry, the influence of the photochemical reaction conducted at high pressure on the molecular and crystal geometry and the influence of pressure on the reaction course will be discussed in terms of:

- variations of the cell parameters and cell volume *versus* pressure,
- changes in the molecular shape and geometry *versus* pressure,
- analysis of hydrogen bonds geometry *versus* pressure,
- variations of the cell parameters along with the photochemical reaction progress at different pressures,
- variations of the molecular geometry along with the photochemical reaction progress at different pressures,
- changes in hydrogen bonds geometry along with the photochemical reaction progress at different pressures,
- difference between the kinetics of the photochemical reaction for different pressures.

Giant strain in urea crystals

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Urea -the first molecular crystal of an organic compound studied by x-ray diffraction [1].
High-pressure phases of urea were discovered by P. Bridgman in 1916 [2], however the structure of high-pressure phases III and IV were determined only recently [3]. Above 2.8 GPa in phase IV, the voids collapse within the retained H-bond network of phase I, however the occurrence of intermediate phase III between 0.48 and 2.80 GPa, where every fourth of NH···O bond is broken, remained unclear. Hence, the continuation of our study on urea, aimed at gathering more precise information about the structural transitions between phases I, III and IV.
At high pressure the urea crystal collapses in two stages. At 0.48 GPa the crystal exhibits a giant negative linear compressibility: in phase III the crystal expanding to b_{III}/b_I=148% along [010], while shrinking to a_{III}/a_I=65% along [100] and to 95% along [001]. Above 2.8 GPa when transforming to phase IV the crystal the still shows negative linear compression to c_{IV}/c_{III}=106% along [001].

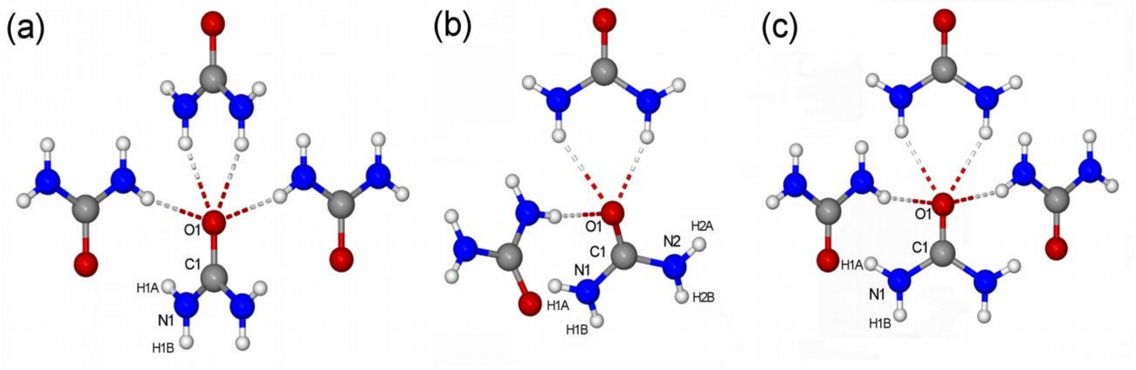

Fig. 1. *The coordination of the carbonyl oxygen in urea: (a) phase I at 0.47 GPa; (b) phase III at 0.80 GPa; and (c) phase IV at 3.10 GPa [3].*

References

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Crystallization of ribose: enantiomer vs. racemate

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Ribose, carbohydrate widespread in Nature, building block of RNA, plays very important role in all leaving organisms. Though it has been studied for years, its recalcitrant crystallization and tendency to form syrup, features common for all natural sugars made its crystal structure a mystery for a long period of time. It was not until 2010 when two forms of D-ribose were reported [1] and 2012 when two polymorphs (one very unstable) of DL-ribose were discovered [2]. We have investigated the reason for facile crystallization of DL-ribose II (Figure 1), yielding large crystals built only from β-pyranose molecules and difficult crystallization of D-ribose, resulting in low-quality crystals of mixed α and β-pyranose anomers. We have rationalized preferential racemate crystallization in the terms of the interplay between their directional interactions in crystal structures and conformational energy of ribose molecules [3].

DL-Ribose II was crystallized from saturated ethanol:isopropanol mixture (vol. 2:1). Obtained crystals were measured at 100 and 295K with a single-crystal Excalibur KM4-CCD diffractometer with graphite monochromated Mo X-ray radiation. H-bonding patterns and potential energy (Ep) of all conformers, existing in the solid state, for D-ribose I and II and DL-ribose II were compared.

The preferential crystallization of racemic ribose is plausibly caused by favorable geometry of H-bonding pattern (short distances and almost linear geometry coherent for all existing H-bonds), molecules adjusting their conformation to the one of the lowest energy and easier fitting of molecules of opposite chirality. Moreover, crystals of D-ribose I and II must try to adapt their rings and H-bonds to match each other. It entails existence of more than 1 molecule in asymmetric part of the unit cell and partial disorder involving α-pyranose anomers.

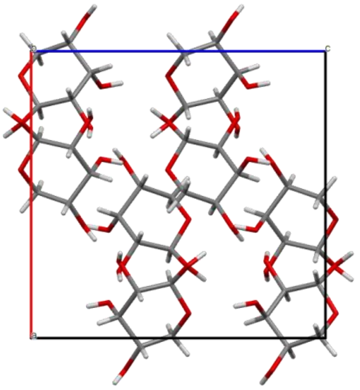

Fig. 1. *Crystal structure of dl-ribose II shown along direction [010].*

References

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High-pressure crystallization of 2,4,5-tribromo and 2,4,5-trichloroimidazole

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Structural stability of 2,4,5-tribromoimidazole was investigated with X-ray diffraction techniques in a diamond-anvil cell (DAC) [1]. In situ crystallization above 0.5 GPa yields a double methylated imidazole bromide hydrate. Moreover subsequent recrystallization at 1.08 GPa led to dehydration and symmetry change. Pressure induced amine methylation was recently reported for dabco hydrobomine and hydroiodine salts [2]. Alkyl derivatives of imidaziole are precursors for ionic liquid [3] what consider pressure techniques as their synthesis methods. Crystal structures of both hydrated and pure 1,3-dimethyl-4,5-dibromoimidazolium bromide have been determined. High-pressure studies of 2,4,5-trichloroimidazole did not give us enough structural information but we can assume that this compound undergo the same reaction as its isostructural equivalent.

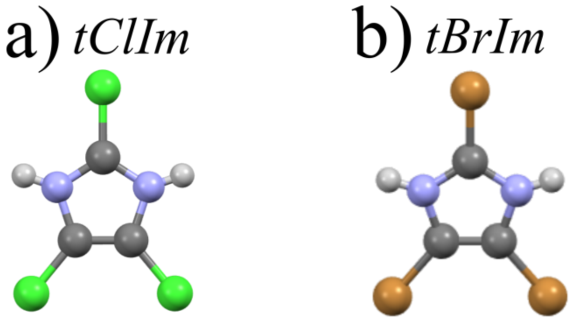

Fig. 1. *a) 2,4,5-trichloroimidazole compound, b) 2,4,5-tribromoimidazole compound.*

References

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Structural studies of 3,3-dimethyl-2-butanone

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Pinacolone (3,3-Dimethyl-2-butanone), the product of pinacol rearrangement [1], is liquid at normal conditions. This important ketone is a precursor to many herbicides and fungicides synthesis, like metribuzin and triadimefon [2]. Furthermore, pinacolone can be used as a photosensitizer, in an inclusion compound with deoxycholic acid, allowing the chemical storage of solar energy. [3] The molecular conformation and intermolecular interactions in pinacolone have not been investigated experimentally and it was the aim of our study to determine these information for the crystalline pinacolone. Freezing point of pinacolone was determined in piston-cylinder press. Based on compressibility chart, two high-pressure crystallizations were carried out in a diamond-anvil cell (DAC) [4,5]

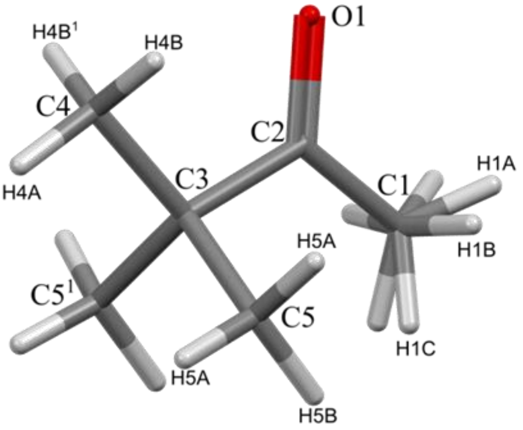

Fig. 1 *Pinacolone (3,3-dimethyl-2-butanone).*

References

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Thiourea from aqueous solution under high pressure

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Thiourea, $(NH_2)_2CS$, one of common chemical compounds, is widely used in chemical practice and industry, mainly for the production of fertilizers, but also insecticides, dyes, plant protection agents, pesticides, corrosion inhibitors, fungicides and explosives.

Single crystals of thiourea have been *in situ* grown in a Merrill-Bassett diamond-anvil cell (DAC) [1] and their structures were determined by X-ray diffraction. High-pressure crystallizations at isochoric conditions lead to the two different hydrates. Despite the fact that only neat $(NH_2)_2CS$ is obtained from aqueous solution at normal conditions (space group *Pnma*, orthorhombic system), pressure of 0.60 GPa induces the crystallization of $(NH_2)_2CS{\cdot}H_2O$, which could be isothermally compressed to 1.20 GPa. However, any crystallization above 1.20 GPa, involving heating the sample, resulted in the formation of phase VI of neat thiourea. At 0.70 GPa and 0.95 GPa new hydrate, $(NH_2)_2CS{\cdot}2/3H_2O$, is formed. The systematic extinctions for both hydrates clearly indicated the monoclinic system, however the unit-cell parameters differ significantly. Monohydrate, $(NH_2)_2CS{\cdot}H_2O$, crystallizes in space group $P2_1/c$, while hydrate $(NH_2)_2CS{\cdot}2/3H_2O$ – in space group $C2/c$.

This study was supported by the TEAM grant No. 2009-4/6 from the Foundation for Polish Science.

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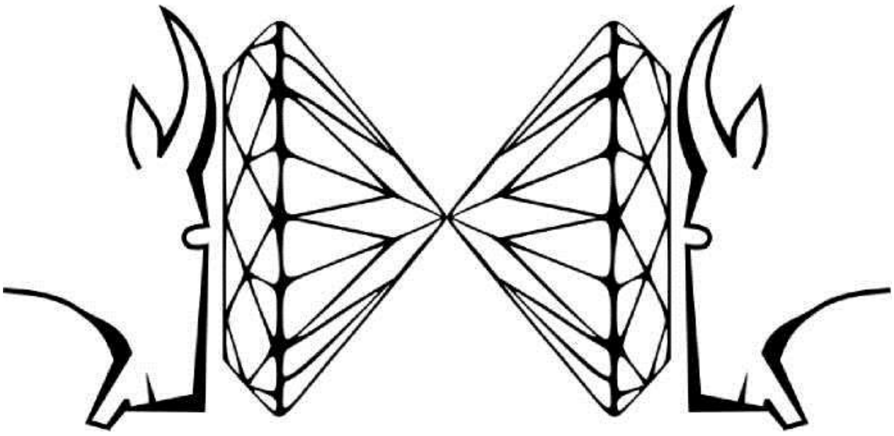

Laboratory work

High-pressure laboratories

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High-pressure technique is a convenient tool for crystallization and it is becoming more popular. First devices used for high-pressure experiments were big and the access to the studied substance was limited [1]. The next step was applying diamonds for generating high pressure [2, 3]. A breakthrough was inventing miniature diamond anvil cell in 1974 [4] and applying a ruby fluorescence method for pressure calibration [5]. Since then the diamond anvil cell (DAC) has become a convenient device for high-pressure experiments. It is a small, relatively cheap and save apparatus, suitable for diffraction structural studies, spectroscopy and many other types of physical measurements. Application of DAC enables people to learn and to understand the behavior of different substances in extreme conditions and the nature of intermolecular interactions. It also used for synthesizing new materials, polymorphs and solvates, that are difficult to obtain at normal conditions. Very often high-pressure experiments are more convenient than the low-temperature measurements and give the opportunity to choose the crystallization method (isothermal, isochoric) [6].

High-pressure laboratories involve practical exercises with the diamond-anvil cell (DAC), its assembling and loading with samples, centering the DAC on a CCD diffractometer, diffraction data collection and processing. Isothermal and isochoric crystallizations will be applied for growing single crystals of new phases of substances brought in by participants. The laboratories will include theoretical and practical parts for both beginner and expert high-pressure crystallographers. The aim of this workshop is to encourage the participants to start high-pressure experiments in their laboratories.

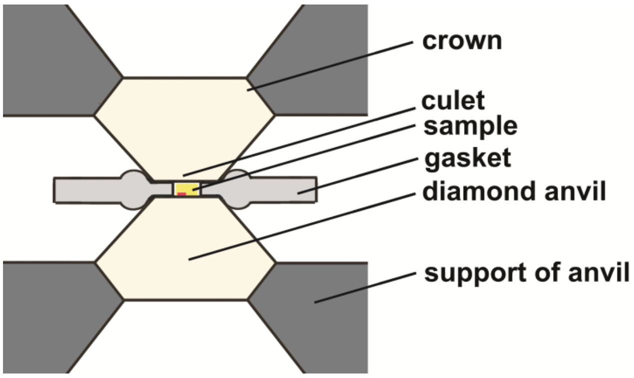

Fig. 1 *Schematic cross-section of the diamond-anvil cell: the diamond anvils, mounted on the steel supporting plates, plastically deforming the metal gasket, and compressing the hydrostatic fluid filling inside the centrally located chamber (a hole in the gasket). The fluid transmits pressure to the sample and to a piece of ruby for spectroscopic pressure calibration.*

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Membrane diamond-anvil cell for high-pressure experiments

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High pressure may be generated in a diamond-anvil cell manually by tightening screws. More convenient is to use a cell with membrane (Fig. 1). Due to an easy handling and possibility to increase pressure without moving cell, this type of cell is perfect for initial studies. Thus it is possible to observe optically phenomena like phase transitions, piezochromism, changes in crystal shape.

During the training participants will observe all steps during an experiment in a membrane cell including: preparing cell, a procedure of loading, controlling pressure inside and making an experiment.

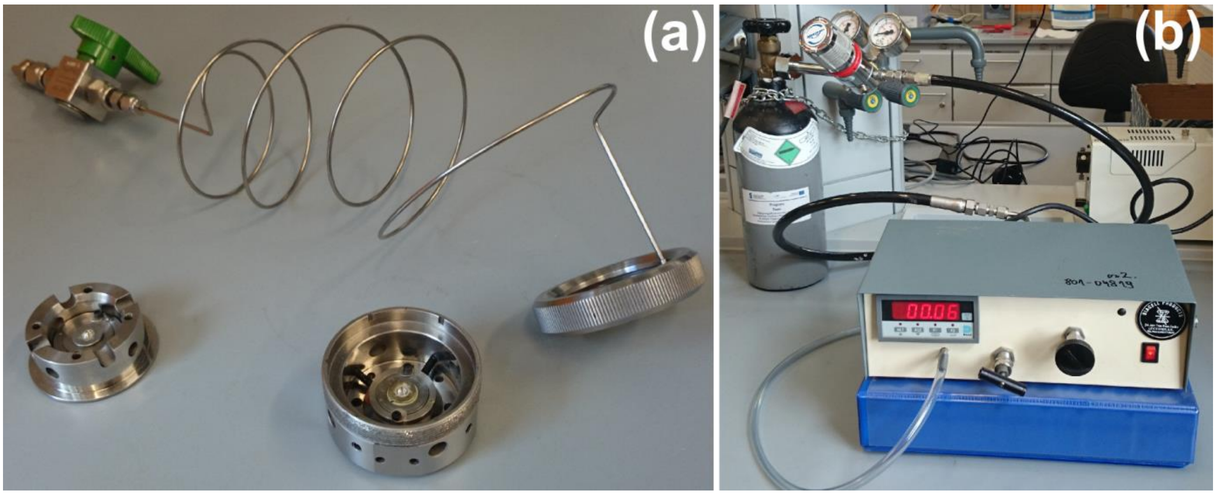

Fig. 1 (a) High-pressure membrane cell and (b) the system for pressure controlling.