



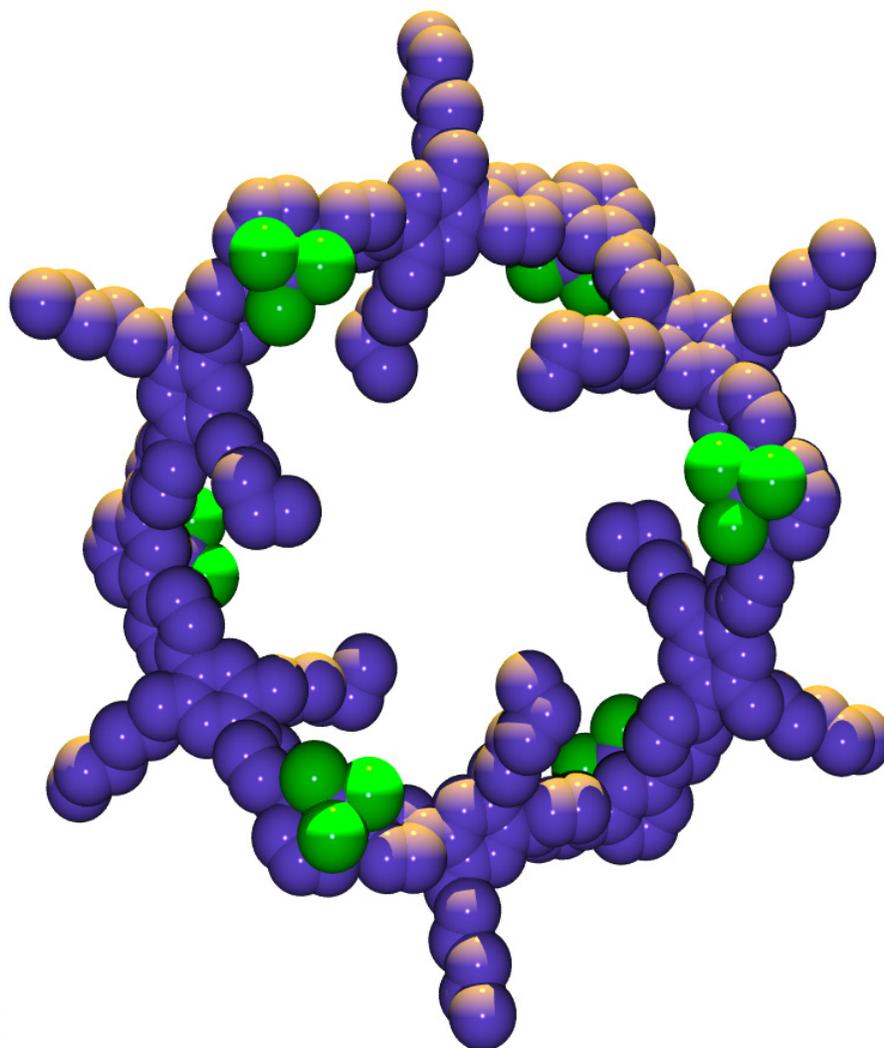
MIT Department of Chemistry
X-Ray Diffraction Facility



18TH ANNUAL BRUKER/MIT SYMPOSIUM

MODERN APPROACHES TO CRYSTAL STRUCTURE SOLUTION

Friday, February 21st to Sunday, February 23rd 2014



A Message from the Director of the Diffraction Facility

Dear participants of the Bruker/MIT Symposium,

As the director of the X-Ray Diffraction Facility of the MIT Department of Chemistry, it is my pleasure to welcome you to the 18th annual Bruker/MIT Symposium.



This year's motto is **Modern Approaches to Crystal Structure Solution**. *Solving a crystal structure*, as opposed to refining the structure, is the step of solving the phase problem. Solving a structure means assigning more or less accurate phase angles to the measured intensities (that is the squared structure factor amplitudes). Although for most X-ray single-crystal small-molecule structures, the phase problem can be solved reliably and quickly with the available software, there is still progress underway and new approaches to solve the phase problem are still actively developed. Especially protein structures or structures based on data from neutron or electron diffraction experiments can still be difficult to solve and even for structures that are solved easily with current software, this progress can be welcome.

At this year's Bruker/MIT Symposium, we have among the speakers three of the world's leading heads of structure solution: Carmelo Giacovazzo whose approach to the negative quartets are used by practically all direct methods programs; George Sheldrick, author of the SHELX software package, whose unsurpassed programming tricks is making the most of many direct methods; and Lukáš Palatinus, programmer of Jana and one of the fathers of charge flipping. All three have made too many seminal contributions to the field of structure solution to list here. It is an honor to have these wonderful scientists here at MIT and I look forward to a successful and enjoyable meeting.

Sincerely,

Peter Mueller
Director, X-Ray Diffraction Facility
MIT Department of Chemistry

ABSTRACTS:

NEW AND EXCITING AT BRUKER

Michael Ruf, Bruker AXS Inc., 5465 East Cheryl Parkway, Madison, WI 53711-5373, e-mail: michael.ruf@bruker-axs.com

Bruker continues to push beyond the limits of X-ray source and detector technologies. The METALJET liquid Gallium source and the PHOTON 100 had a tremendous impact in the community over the last year. At the more economical end of the instrument spectrum the D8 QUEST ECO puts cutting edge technology in a low footprint package into the hands of smaller institutions.

This presentation will highlight some of the achievements that were made at Bruker since the last MIT Bruker meeting.

THE CRYSTALLOGRAPHIC PHASE PROBLEM

Peter Müller, Mass. Institute of Technology, 77 Massachusetts Ave., Cambridge, MA 02139, e-mail: pmueller@mit.edu

The 2014 Bruker/MIT Symposium is about modern approaches to crystal structure solution. The term “structure solution”, as opposed to structure refinement, is generally used to describe the step of assigning phases to the structure factors. In other words, to solve a structure means to solve the phase problem. This presentation will briefly and on a simple level introduce the crystallographic phase problem and highlight some of the classic methods of solving it.

SIR2014, PHASING METHODS AND RESULTS

Carmelo Giacovazzo, Istituto di Cristallografia, CNR, Bari, Italy, e-mail: carmelo.giacovazzo@ic.cnr.it

SIR2014, a still unreleased package, is designed for crystal structure solution via *ab initio* or *non ab initio* techniques, as here down summarized:

ab initio methods

- a) Modern direct Methods (*MDM*)
- b) *VLD* (Vive la Difference)
- c) Patterson techniques

Non ab initio

- d) Molecular Replacement
- e) Simulated annealing

Ab initio methods will be the main object of the talk. The theoretical bases of each technique and the various algorithms involved in the phasing process will be briefly elucidated.

Extended applications will be summarized, showing that:

1. Each of the three *ab initio* methods can be usefully applied to organic as well as to inorganic structures. That may be unexpected, particularly for Patterson techniques, which are considered to be suitable only when heavy atoms are in the unit cell.
2. The resolution limits, traditionally set to about 1 Å for *ab initio* techniques, may be by far overcome. In favorable conditions, it may be reset to 2 Å resolution.
3. The size limits may also be extended. Proteins up to 8000 atoms in the asymmetric unit are today solvable provided heavy atoms are present in the unit cell.

SIR2014 is fully automatic: from simple information on the unit cell, space group and unit cell content, it is able, in default conditions, to provide an isotropically refined molecular model. The user however, if he likes, may choose his own solution pathway by personally fixing directives.

SOLVING CRYSTAL STRUCTURES WITH CHARGE FLIPPING

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The charge-flipping algorithm is a member of the diverse family of dual-space iterative phasing algorithms. These algorithms use alternating modifications in direct and reciprocal space to find a solution to the phase problem. Unlike other algorithms from the diverse family of *ab initio* structure solution methods, in dual-space algorithms neither of the two spaces is dominant in the structure solution, but it is the iteration between the two that makes the structure solution possible. The dual-space algorithms and especially the charge-flipping algorithm have proven to be useful in a range of crystallographic applications, including – apart from the “standard” structure solution –

the solution of complex structures from powder diffraction data or the solution of incommensurately modulated crystals and quasicrystals. The algorithms require neither prior knowledge about the chemical composition of the crystal, nor prior information on the space group. Thus, in practice, they allow for the solution of the crystal structure even in cases when the chemical or symmetry information is ambiguous or wrong.

QUALITY DATA: ECONOMICAL AND CUTTING-EDGE

Bruce Noll, Scott D. Phillips, and Michael Ruf,
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Bruker's D8 VENTURE METALJET and D8 QUEST ECO diffractometers represent opposite ends of the equipment line-up. The air-cooled D8 VENTURE METALJET, employing a liquid gallium target, is our brightest laboratory source, and has proven itself in its ability to produce measureable diffraction from specimens that showed no evidence of diffraction on other sources. Samples that would otherwise be sent to a synchrotron can be handled in the home lab, without delays of shipping and scheduled beamtime. Small, sensitive samples can be examined quickly, and problems of air- and moisture-sensitivity or solvent loss can be addressed within the lab, without the worry of the shipping and handling at the synchrotron. As will be shown, gallium has proven to be effective for determination of absolute configuration of light-atom structures. Coupled with our APEX2 software suite, the structure determination is straightforward and direct, from data collection, through structure solution, and into generation of reports and publication materials.

The D8 QUEST ECO provides an economical and ecological solution for X-ray structure determination. With an entirely air-cooled design, the system has a minimal carbon footprint, appealing to, and required at, many institutions. Available with either copper or molybdenum X-ray sources, it is attractive to the service laboratory as a workhorse diffractometer using Mo radiation and accessible to users of all levels of experience. With Cu radiation, the system is well-suited to determination of absolute configuration for light-atom structures, making it very useful for pharmaceutical research. Also integrated with APEX2, the system is easy for new users to operate, and powerful for more experienced researchers.

SHELXT: INTEGRATING SPACE GROUP DETERMINATION AND STRUCTURE SOLUTION

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SHELXT is intended for robust routine solution of small molecule crystal structures. It makes the simple but powerful assumption that the structure consists of resolved atoms, but unlike classical direct methods it is not required that the atoms are 'equal'. This enables it to succeed with poor or incomplete data but makes it unsuitable for structures that are twinned, modulated or contain severe (e.g. 'whole molecule') disorder. SHELXT is a dual-space program that starts with a Patterson minimum superposition and iteratively employs the random omit procedure (also used in SHELXD), but does not use phase probability relations or solvent flipping. In the SHELX system it will probably obsolete SHELXS but not SHELXD, which is better for large equal-atom and twinned structures. SHELXT reads any legal SHELX format .ins and (HKLF3 or 4) .hkl files. It extracts the Laue group and tries to find the best space group(s) in this Laue group and makes an approximate assignment of element types using the elements specified on the SFAC instruction (and maybe a couple more). This is followed by an isotropic refinement and an attempt to assign the absolute structure if the space group is non-centrosymmetric.

It is hoped to release SHELXT as a normal part of the SHELX system in time for the 2014 Montreal IUCr Meeting.

Poster abstracts

Trapping an Elusive Bürgi-Dunitz intermediate: A Crystallographic-Computational Joint Study

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Hydroxamic acids are important ligands in bioinorganic chemistry and have recently emerged as highly valuable anchors for dye sensitized solar cells. Despite the great attention shown in bioinorganic literature due to their connection with siderophores, there are only few examples of pure coordination chemistry. We have found $[\text{Cp}^*\text{Ir}(\text{OH})_3\text{IrCp}^*]\text{OH}$ reacts with PhCONHOH to give $[\text{Cp}^*\text{Ir}(\eta_2\text{-ONCOPh})]$ in which the doubly deprotonated NHOH unit binds side-on via N and O, an otherwise unrecorded binding mode. The X-ray structure shows unprecedented pyramidalization at Ir together with secondary bonding between the carbonyl oxygen and iridium ($\text{Ir}-\text{O} = 2.87 \text{ \AA}$). The related o-hydroxyphenyl hydroxamic acid gives a conventional chelate structure in which both sp^3 O atoms are bound in deprotonated form. The distorted conformation in $[\text{Cp}^*\text{Ir}(\eta_2\text{-ONCOPh})]$ is stabilized by weak π - π interactions in the solid state. Computational studies suggest that being a transient conformation in solution that could be classified as a Bürgi-Dunitz intermediate. These results demonstrate the importance of weak interactions in the solid state to gain access to otherwise inaccessible structures.

Use of a Virtual Multifunctional X-Ray Diffractometer for Teaching Science and Engineering Courses

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The poster will present a simulation-based Virtual X-ray Powder Diffractometer designed to enable students to become familiar with X-ray diffraction methods and equipment. It allows students to gain practical skills required for conducting actual experiments and acquire the knowledge needed for collecting, analyzing and interpreting experimental data. Embedded auxiliary simulations help students learn fundamental physical principles underlying the analytical methods and equipment design in very visual and interactive ways.

The virtual diffractometer enables students to watch and explore what is going on “under the hood” of the equipment and thus overcome the educational disadvantage of contemporary fully computerized X-ray equipment that feel like a “black box”, letting students access only a computer monitor and a sample holder. An

interactive online lesson, as well as prerecorded video lectures, animations, quizzes and other learning resources, can be called up within virtual experiments to provide “just-in-time” learning opportunities that address the educational needs of the student.



The virtual diffractometer has also been used for preparing undergraduate and graduate students to perform the research studies. The v-Labs can be easily integrated with traditional online courses including MOOC courses available from *edX*, *Coursera*, and *Udacity* and other courseware providers

Complexation of Small Gases by a Heterocapped Cryptophane-111 Derivative

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Cryptophanes are cage-like container molecules with an enforced internal lipophilic cavity that can house and interact with smaller molecules and gases. Cryptophane-111, (\pm)-**111**, is the second smallest molecular cage with a cavity volume of 74 \AA^3 . (\pm)-**111** is known to bind small gases such as CH_4 , C_2H_4 , C_2H_6 and xenon.^[1] Particular interest is for the isotope ^{129}Xe ($I = 1/2$) which has potential applications in magnetic resonance imaging. In fact, (\pm)-**111** exhibits the highest affinity for Xe ($K_a = 10^4 \text{ M}^{-1}$ at 293 K) in organic solvents.^[2] The first water-soluble derivative of (\pm)-**111** was synthesized within our group by the addition of pentamethylcyclopentadienyl ruthenium $[\text{Cp}^*\text{Ru}(\text{II})]^+$ moieties to the arene rings, forming the chloride salt $(\pm)\text{-}[(\text{Cp}^*\text{Ru})_6\text{-111}]\text{Cl}_6$. This derivative exhibits among the highest binding constants for Xe in any solvent, $2.9 \times 10^4 \text{ M}^{-1}$ at 293 K in D_2O .^[3] To date, all reported derivatives of (\pm)-**111** were obtained by post-synthetically modifying (\pm)-**111** which is prepared by the covalent capping of two cyclotriphenylene (\pm)-CTP

cups. In effort to make more available functionalized derivatives, trisbromocryptophane-111, (\pm)-**Br₃-111** was successfully synthesized by the heterocapping of (\pm)-**CTP** with trisbromocyclotriphenylene (\pm)-**Br₃-CTP**. Single crystal X-ray diffraction data were obtained for the guest free (\pm)-**Br₃-111** host as well as for complexes of Xe and N₂. Also, CH₄ complexation by (\pm)-**Br₃-111** in CD₂Cl₂ was studied using ¹H NMR spectroscopy to better understand the substituent effect of the bromide on the (\pm)-**111** core in terms of the binding of gases.

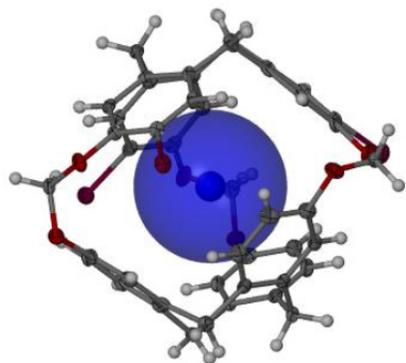


Figure 1: Thermal ellipsoid plot of 0.96Xe@(±)-**Br₃-111** at 60% probability. Only major occupancy positions of disordered species are shown. The van der Waals volume of Xe is shown as a transparent blue sphere.

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Crystallographic Insight in the Chirality of the Potent Monofunctional Anticancer Agent Phenanthriplatin and the Nature of Its Interaction with DNA

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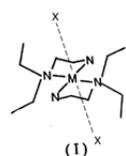
Despite widespread use of platinum drugs in the clinical treatment of cancer, a range of side effects and the occurrence of resistance prompt the development of new metal-based therapeutics. Platinum drug development has typically focused on the preparation of complexes that obey strict structure-activity relationships requiring square planar geometry, charge neutrality, and exactly two labile ligands. We have recently reported, however, that the cationic monofunctional compound phenanthriplatin, or *cis*-[Pt(NH₃)₂Cl(Am)]⁺ where Am is phenanthridine, displays significant potency in vitro. Unlike cisplatin, phenanthriplatin can only form one bond to DNA,

precluding the generation of DNA crosslinks as the origin of anticancer activity. Solution of the crystal structure of *cis*-[Pt(NH₃)₂Cl(phenanthridine)]OTf revealed that this compound is chiral. The chirality of the complex arises from the asymmetry of the phenanthridine ligand about the platinum coordination plane. The subsequently solved crystal structures of the chloride, nitrate, and tetraphenylborate salts of phenanthriplatin provide insight into the internal conformational dynamics of the chiral monofunctional complex. This fluxionality was further characterized by dynamic NMR spectroscopy. Further NMR spectroscopic studies revealed that phenanthriplatin adducts of 9-alkylguanine, small molecule models of the phenanthriplatin-DNA adduct, form with diastereoselectivity. The crystal structure of *cis*-[Pt(NH₃)₂(phenanthridine)(9-methylguanine)]OTf₂, along with NOE NMR experiments, revealed which diastereomeric form is present in both the solid state and in solution. Computational studies provide insight into the origin of the conformational preference and discussion is given of the role that this preference may have on the biological activity of phenanthriplatin.

Tracing Pathways of Thermochromic Transition in Copper Complexes

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Copper(II) complexes of the type shown in (I) are known since long^[1] to exhibit first order reversible thermochromic red to blue transitions in solid state. The temperatures (T_c) at which the colour change takes place vary with anions (X) present in the complex (I).



X	T _c (° C)
BF ₄	24
ClO ₄	44
NO ₃	150



Although, structural investigations have been carried out to understand the mechanism of colour change^[2,3], the factors that trigger the structural phase transition upon heating have remained somewhat obscure. To observe these events in crystals, attempts are made to determine structure of the ClO₄ complex at temperatures starting below the transition and reaching those beyond the colour change. The variable temperature diffraction measurements on a single crystal over short time intervals were possible only because of the availability of Australian Synchrotron source. The copper ion 'appears' in the square planar coordination environment in both the forms, shortest approach of the anion Cu...O (ClO₄) being > 3.5 Å in low and high temperature forms (figure right above of the BF₄

complex). The onset of the transition is seen to be triggered by the orientational disorder of the anions that weakens the interactions with the ligand (N-H \cdots O and C-H \cdots O contacts). The Diethyl groups are thus free to change the conformation allowing anions to interact with the Copper ion in the high temperature form. Analysis of the structures for the first time reveals a pathway of phase transition that goes with a remarkable co-operativity in the entire crystal.

References:

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Automated Patterson methods in ab initio structure determination of 32 base pair RNA

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Direct methods have been rarely applied to RNA crystal structures in spite of the growing interest in RNA structure. We determined the crystal structure of a 32 base pair RNA using only the native 1.05 Ångstrom diffraction data by ab initio direct methods using the program SIR2011 starting with phases derived from peaks automatically found by Patterson superposition. One strand was in the asymmetric unit (32 nucleotides or 675 non-H RNA atoms). The opposing strand was generated by crystallographic symmetry. This is one of the largest nucleic acid structures determined to date ab initio by direct methods. All 31 phosphates in the asymmetric unit were found and almost all of the base and sugar atoms were automatically placed by peak picking in the E-maps. The weighted mean phase error of the SIR2011 map was 23.2 degrees.

The Crystal Structure of a Six-Fold Non-Merohedrally Twinned Inclusion Compound

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Urea forms inclusion compounds with many long chain guest molecules, but simple *n*-alkanes are of particular interest because weak host-guest interactions typically do not induce commensurability, giving rise to various aperiodic phases. However, eicosane/urea is uniquely commensurate with 2 guests for 5 hosts repeats. Solving a commensurate structure is usually straightforward, but

here guests are dynamically disordered in the high temperature paraelastic phase, and exhibit six-fold non-merohedral twinning in the low-temperature, ferroelastic phase. We attempted to circumvent the twinning problem by using a thermomechanical clamping technique to bias the twin populations in the ferroelastic phase of this material. Here, a uniaxial stress is applied across opposite prismatic faces of the crystal above the phase transition temperature. While under stress, the crystal is cooled through the ferroelastic phase transition (190K) to selectively populate the favored twin. Although the "detwinned" data were sufficient to solve the host structure without invoking a twin model, incomplete detwinning, coupled with the large size of the crystal required for this experiment, gave rise to scaling problems that prevented a complete structure solution. We later developed a twin model to solve the structure using data collected at APS Beamline 14-BM-C using a Quantum 315 detector. The frames were converted to Bruker format using a program developed by Weiguang Guang of McMaster University. Reflections from all six domains were separated manually in RLATT to refine six unit cells used for multi-component integration in SAINT. TWINABS was used to further partition and scale reflections. With XPREP, we identified space group $P2_1$ with $a = 8.261$ Å, $b = 55.002$ Å, $c = 13.8291$ Å, and $\beta = 90.001^\circ$. In stark contrast with the direct methods structure solution, SHELXT (intrinsic phasing) was able to identify all host atoms and most guest atoms. The structure was refined in SHELXL using single and composite reflections for all domains. Our structure confirms the counterintuitive prediction by Fukao^[1] that guests molecules pack head-to-head along the channel axis rather than herringbone.

Reference:

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Design and Applications of Functional Molecular Frameworks

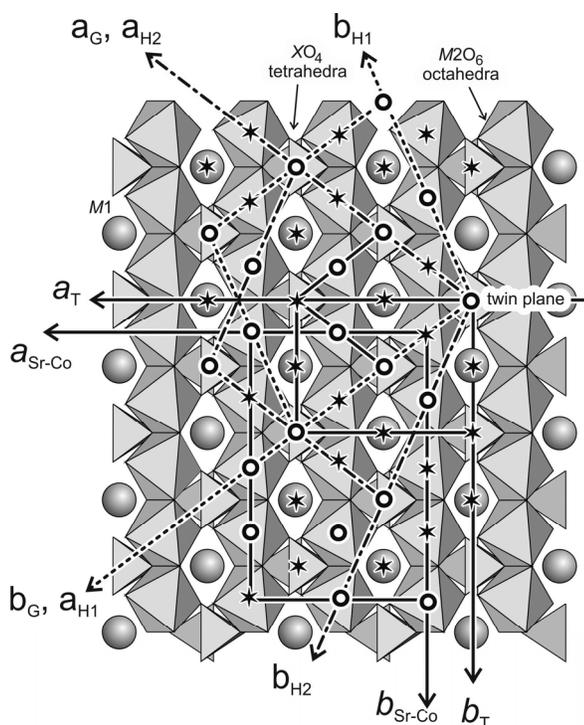
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Inclusion compounds comprise host frameworks, assembled through non-covalent interactions, and guest molecules. A particularly attractive feature of inclusion compounds is the ability to incorporate functional guests within a host framework with controllable architecture. Whereas most crystal engineering strategies rely on structural control and function combined in the same molecular building block, inclusion compounds allow separation of structure and function, permitting improved control of material properties. Herein we reported a reliable strategy to engineer hydrogen bonded frameworks with variable cavities' sizes and morphologies through molecular design.

Structural variants among tsumcorite-type arsenate

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In order to understand the role of arsenic in the environment, one has to investigate structural features and stabilities of naturally occurring arsenic compounds. In addition, a study of mineral-related synthetic phases should be very helpful because they can appear as a result of human activities. $\text{SrCo}_2(\text{AsO}_4)(\text{AsO}_3\text{OH})(\text{OH})(\text{H}_2\text{O})$ and $\text{BaMg}_2(\text{AsO}_4)_2(\text{H}_2\text{O})_2$ were hydrothermally synthesized (Teflon-lined steel autoclaves, 493 K, 3-4 days) during an on-going research on natural and synthetic arsenates, with a focus on their structural and spectroscopic classification.



$\text{SrCo}_2(\text{AsO}_4)(\text{AsO}_3\text{OH})(\text{OH})(\text{H}_2\text{O})$ and $\text{BaMg}_2(\text{AsO}_4)_2(\text{H}_2\text{O})_2$ belong to the tsumcorite group compounds. Up to now more than 30 natural and synthetic representatives of the group are known [1,2 and references therein]. The general formula of the group is: $M1M2_2(\text{XO}_4)_2(\text{H}_2\text{O},\text{OH})_2$: $M1^{1+,2+,3+} = \text{Na, K, Rb, Ag, NH}_4, \text{Ca, Pb, Bi, Tl}$; $M2^{2+,3+} = \text{Al, Mn}^{3+}, \text{Fe}^{3+}, \text{Co, Ni, Cu, Zn}$; $X^{5+,6+} = \text{P, As, V, S, Se, Mo}$. The $\text{M}2\text{O}_6$ polyhedra are edge-connected to chains which are linked by XO_4 tetrahedra to layers. They are connected by $\text{M}1$ atoms and by hydrogen bonds. For each of the cation sites $\text{M}1$, $\text{M}2$ and X at least two different valences are possible; the coupled exchange at these cation positions ensures electro neutrality. Furthermore, charge balance is also realized by adjusting the ratio $\text{OH}:\text{H}_2\text{O}$.

Majority of the group members crystallize in the parental structure type ($C2/m$, $Z = 2$), where H_3O_2 groups are formed, which involve a symmetry restricted hydrogen bond (tsumcorite type [3]). Three different unit cells with lower symmetry are known so far (see Figure) [1,2]. They are caused by distinct crystal-chemical requirements. ‘Helmutwinklerite type’ ($P\bar{1}$, $Z = 4$): two H_2O molecules p.f.u. require an avoidance of symmetry-restricted hydrogen-bonds; ‘gartrellite type’ ($P\bar{1}$, $Z = 1$): $\text{M}2$ atoms show different crystal-chemical behavior e.g., a Jahn-Teller effect. [2]. In $\text{SrCo}_2(\text{AsO}_4)(\text{AsO}_3\text{OH})(\text{OH})(\text{H}_2\text{O})$, monoclinic symmetry is maintained but the cell volume is doubled [1]. In addition, the C -centering is avoided and the space-group symmetry is lowered to the subgroup $P2_1/a$. The As position from the parental structure is split into two crystallographically independent sites and that the O1 atoms are no longer linked by an inversion center. The hydrogen bonding scheme is reorganized as compared to the ‘tsumcorite type’. The structural analysis of the possible fifth structure variant adopted by $\text{BaMg}_2(\text{AsO}_4)_2(\text{H}_2\text{O})_2$ is currently under analysis.

The room temperature single-crystal X-ray intensity data of $\text{SrCo}_2(\text{AsO}_4)(\text{AsO}_3\text{OH})(\text{OH})(\text{H}_2\text{O})$ was collected on a Nonius Kappa CCD single-crystal four-circle diffractometer (MoK α -radiation, graphite monochromator, $2\theta_{\text{max}} = 65^\circ$), equipped with a 300 mm diameter capillary-optics collimator. Single-crystal X-ray intensity data of $\text{BaMg}_2(\text{AsO}_4)_2(\text{H}_2\text{O})_2$ was collected at 293 K on a Bruker-Nonius APEX-II diffractometer with a CCD-area detector, equipped with a graphite monochromator and using MoK α -radiation). The intensity data were processed with the Bruker-Nonius program suite SAINT-Plus and corrected for Lorentz, polarization, background and absorption effects. A complete sphere of reciprocal space was measured for both crystals. Both structures were solved by direct methods using SIR97 [5] and refined on F^2 by full-matrix least-squares using SHELXL97 [6] and WinGX [7].

The strong reflections of $\text{SrCo}_2(\text{AsO}_4)(\text{AsO}_3\text{OH})(\text{OH})(\text{H}_2\text{O})$ correspond with the unit cell and space-group symmetry of the tsumcorite-type. However, a number of weak reflections could clearly be seen; they are responsible for a doubled b parameter as well as for violating the C centering of the unit cell. The true space-group symmetry of $\text{SrCo}_2(\text{AsO}_4)(\text{AsO}_3\text{OH})(\text{OH})(\text{H}_2\text{O})$ is $P2_1/a$, $a = 9.136(2)$ Å, $b = 12.820(3)$ Å, $c = 7.520(2)$ Å, $\beta = 114.34(3)^\circ$, $V = 802.48$ Å³, $Z = 4$, $R(F) = 3.3\%$ and $wR(F^2) = 9.8\%$. The extremely strong anisotropic displacement parameters observed in the average structure for the oxygen atoms are avoided in the larger $P2_1/a$ unit cell.

The average structure of $\text{BaMg}_2(\text{AsO}_4)_2(\text{H}_2\text{O})_2$ correspond to the parental structure type of the tsumcorite-type (space group $C2/m$, $Z = 2$). Its crystal data are: $a = 9.3540(19)$, $b = 6.2190(12)$, $c = 7.2560(15)$ Å, $\beta = 103.20(3)$, $V =$

410.9(1) Å³, $R(F) = 0.0238$, $wR(F^2) = 0.0598$. However, the higher anisotropic displacement parameters for Ba atom are observed and solving its crystal structures in lower space-group, didn't lead to the real structure solution. Further structural data for barium compound were collected on the STOE StadiVari four-circle diffractometer with Dectris PILATUS 300K pixeldetector using MoK α -radiation. These measurement showed that within (hkl) layers, where $k = 2n+1$ and (hkl) layers, where $h = 2n+1$ comes to the appearance of the diffuse scattering lines, which appear in the form of the broken lines. The structural solution of BaMg₂(AsO₄)₂(H₂O)₂ is under construction

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Thanks be to

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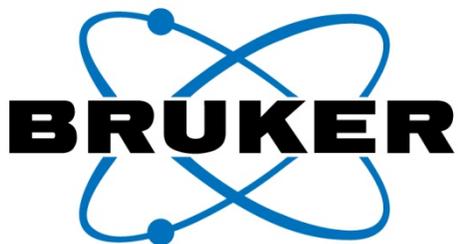
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SCHEDULE

Friday, February 21st: workshops (8:30 a.m. to 12:00 noon and 1:30 p.m. to 5:00 p.m.)
SHELXT Workshop: room **6-321** (both sessions);
SIR Workshop: room **18-378** (both sessions),
Superflip Workshop: room **6-233** (morning session) and room **12-122** (afternoon session).

Saturday, January 22nd: 8:30 a.m. to 5:00 p.m. in **room 66-110**

08:30 - 09:00 Light Breakfast

09:00 - 09:15 Opening Remarks

09:15 - 09:45 Michael Ruf (Bruker AXS)
New and Exciting at Bruker

09:45 - 10:45 Peter Müller (MIT)
The Crystallographic Phase Problem

10:45 - 11:00 Coffee Break

11:00 - 12:00 Carmelo Giacovazzo (Inst. of Crystallography, Bari)
SIR2014, Phasing Methods and Results

12:00 - 01:45 Lunch with Poster Session in **room 6-321** (Moore room)

02:00 - 03:00 Lukáš Palatinus (Institute of Physics, Prague)
Solving Crystal Structures with Charge Flipping

03:00 - 03:15 Coffee Break

03:15 - 03:45 Bruce Noll (Bruker-AXS)
Quality Data: Economical and Cutting-Edge

03:45 - 04:45 George Sheldrick (University of Göttingen)
SHELXT: integrating space group determination and structure solution

04:45 - 05:00 Poster Prize and Closing Remarks

Sunday, February 23rd: workshops (9:00 a.m. to 12:30 p.m.)
SHELXT Workshop: room **6-321**;
SIR Workshop: room **18-378**,
Superflip Workshop: room **6-233**.