YCM1

1st Franco-German YOUNG CRYSTALLOGRAPHERS MEETING

26-29 September 2023

UNIVERSITÉ DE STRASBOURG

Institut de Science et d'Ingénierie Supramoléculaires

Université franco-allemande Deutsch-Französische Hochschule

B-ID Integrative Biophysics and Structural Biology For the Study of InFection and Disease

USIAS University of Strasbourg Institute for Advanced Study

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		09.45 - 10.30	Talks C01/B01/C02			10.00 - 10.45	Industry plenary session / K04
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		11.30 - 12.45	Talks P01/C03/ B02/C04/P02	11.15 - 12.00	Talks P07/P08/B08	11.15 - 12.15	Talk AFC Doctoral Prize 2022
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19.00 - 20.30	Welcome cocktail	20.30 - 23.00	Traditional Alsatian dinner	20.00 - 22.30	Dinner		

SCIENTIFIC PROGRAM

Tuesday 26th September

16.00 – 18.00 **Arrival & Check-in**

18.00 – 18.15 **Opening remarks**

18.15 – 19.00 **Plenary session / K01**
"Teasing out the secrets of subtle protein dynamics"
Helen Ginn
DESY, Hamburg, Germany

19.00 – 20.30 **Welcome cocktail**

Wednesday 27th September

09.00 – 09.45 **Plenary session / K02**
"The challenging crystallography of mixed-anion inorganic compounds"
Romain Wernert
University of Oxford, United Kingdom

09.45 – 10.00 **Talk / C01**
"Structural analyses and properties of complex sulphides in the Cr-Sn-S system"
Florentine Guiot
University of Rennes, France

10.00 – 10.15 **Talk /B01**
"Unusual peptide-binding proteins guide pyrroloindoline alkaloid formation in crocagin biosynthesis"
Dazhong Zheng
University of Glasgow, United Kingdom

10.15 – 10.30 **Talk / C02**
"A series of supramolecular metalacyclic assemblies of CuI8MII/III/IV: how structural conformers can influence the photophysical properties"
Constance Lecourt
Institut des Sciences Chimiques de Rennes, France

10.30 – 11.30 **Coffee break**
Poster session

11.30 – 11.45 **Talk / P01**
"Innovative synthesis at high pressure and high temperature of new compounds in the ternary B-C-Si system"
Martin Demoucron
Institut de Minéralogie, de Physique des Matériaux et de Cosmochimie, France

11.45 – 12.00 **Talk / C03**
"Linkage isomerism in a photoswitchable palladium nitro complex"
Artem Mikhailov
Université de Lorraine, France

12.00 – 12.15 **Talk / B02**
"Time-resolved crystallography for the study of protein dynamics using X-ray Free-Electron Lasers (XFELs)"
Ronald Rios-Santacruz
Université Grenoble Alpes, France

12.15 – 12.30 **Talk / C04**
"Structural and theoretical study of the unexpected flexibility of CALF-20 MOF"
Joanna Drwęska
Adam Mickiewicz University, Poland

12.30 – 12.45 **Talk / P02**
"Observing growth of silver thin films in situ with synchroton radiation"
Michal Kaminski
Karlsruhe Institute of Technology, Germany

12.45 – 14.30 **Lunch buffet**
Poster session

14.30 – 14.45 **Talk / B03**
"Emergence of order from proteins under nucleation"
Dimitris P. Triandafillidis
Universität Hamburg, Germany

14.45 – 15.00 **Talk / C05**
"Building New Metal-Organic Frameworks from Iron-Thiolate Layers"
Nusik Gedikoglu Lauritzen
Institut des Matériaux Jean Rouxel de Nantes, France

15.00 – 15.15 **Talk / P03**
"Characterization of [Au]core@[Ag]shell nanorods by ultra low frequency Raman scattering"
Charles Vernier
Sorbonne Université, France

15.15 – 15.30 **Talk / B04**
"Optimising Protein Production for Functional and Structural Studies: Example of the NYN proteins"
Léna Coudray
Institut de Biologie Moléculaire et Cellulaire, France

15.30 – 15.45 **Talk / C06**
"Study of Organolanthanide Complexes with Cyclononatetraenyl (cnt) and Cyclooctatetraenyl (cot) Ligands"
Nicolas Casaretto
Laboratoire de Chimie Moléculaire, Centre National de la Recherche Scientifique, École Polytechnique, France

15.45 – 16.15 **Coffee break**

16.15 – 16.30 **Talk / P04**
"Unraveling intermolecular protein interactions: insights from molecular rotors"
Yevgeniya Karibjanova
Laboratoire de Génie Chimique, France

16.30 – 16.45 **Talk / B05**
"Structural characterization of the BLUF photoreceptor OaPAC using time-resolved crystallography"
Anaïs Chretien
European XFEL, Germany

16.45 – 17.00 **Talk / C07**
"Structure Dynamics in a Spin-Crossover Crystal Probed with Ultrafast Electron Diffraction and Serial Femtosecond X-ray Diffraction"
Yifeng Jiang
European XFEL, Germany

17.00 – 17.15 **Talk / P05**
"Structural study of piezoelectric $LnCa_4O(BO_3)_3$ at high pressures and cryogenic temperatures"
Fatiha Azrour
Institut Charles Gerhardt Montpellier, France

18.30 – 20.00 **Visit to the historic wine cellar of Strasbourg Hospices**

20.30 – 23.00 **Traditional Alsatian dinner**

Thursday 28th September

09.00 – 09.15 **Talk / C08**

"Effect of nanostructuring on the structural and physical properties of functional molecular nanomaterials: Applications to bioactive glasses"

Amira Ghneim
Cristallographie, Résonance Magnétique et Modélisations, Université de Lorraine, France

09.15 – 09.30 **Talk / C09**

"Crystallography for molecular magnetism"

Nathalie Bridonneau
Institut de Chimie Moléculaire et des Matériaux d'Orsay, France

09.30 – 09.45 **Talk / P06**

"X-ray optical activity in achiral crystals of coordination complexes"

Elen Duverger-Nédellec
Institut de Chimie de la Matière Condensée de Bordeaux, France

09.45 – 10.00 **Talk / B06**

"Capabilities and developments at the SPB/SFX Scientific Instrument at the European XFEL"

Raphael de Wijn
European XFEL, Germany

10.00 – 10.15 **Talk / C10**

"Enhanced Circular Dichroism and Polarized Emission in an Achiral, Low Bandgap Bismuth Iodide Perovskite Derivative"

Jakob Möbs
Department of Chemistry and Material Sciences Center, Philipps-Universität Marburg, Germany

10.15 – 10.30 **Talk / B07**

"The in crystallo optical spectroscopy toolbox"

Nicolas Caramello
European Synchrotron Radiation Facility

10.30 – 11.15 **Coffee break**
Poster session

11.15 – 11.30 **Talk / P07**
"Milk fat polymorphism in the butter-making process"
Valentin Demaille
Université de Bordeaux, France

11.30 – 11.45 **Talk / P08**
"Hexagonal 2H-SiGe in Nanostructures by Phase Transformation"
Theo van den Berg
Centre de Nanoscience et Nanotechnology, France

11.45 – 12.00 **Talk / B08**
"The mechanism of the SorC protein family revealed"
Markéta Šoltysová
Institute of Organic Chemistry and Biochemistry of Czech Academy of Sciences, Czech Republic

12.00 – 14.00 **Lunch**

14.00 – 15.00 **Plenary session / K03**
"Molecular machines and motors: A historical perspective"
Jean-Pierre Sauvage
University of Strasbourg, France

15.00 – 16.30 **'Work with us' session**
15.00 Introductory presentations
15.30 Poster session

Participants
Loïc Le Dréau ~ Bruker
Loïc Mazé ~ Malvern Panalytical
Laurent Loos ~ Rigaku
Laura Folkers ~ STOE

17.00 – 19.30 **Boat trip & visit to Strasbourg**

20.00 – 22.30 **Dinner**

Friday 29th September

09.00 – 10.00 **French young crystallographers meeting**

10.00 – 10.45 **Industry plenary session / K04**
"Crystallographers in the pharma world of drug discovery"
Laure Delarbre
Sanofi R&D, France

10.45 – 11.15 **Coffee break**

11.15 – 11.35 **AFC Doctoral Prize 2022 Winner Talk / AFC01**
"Discovery and structure-function studies of key enzymes behind the non-canonical ZTGC-DNA found in phages"
Dariusz Czernecki
MRC Laboratory of Molecular Biology, United Kingdom

11.35 – 11.55 **AFC Doctoral Prize 2022 Winner Talk / AFC02**
"Resolution of the cationic distribution in $Cu_{22}Fe_8Ge_4S_{32}$ using synchrotron resonant powder diffraction: A case study and guidelines for analogous compounds"
Laura Paradis-Fortin
Lawrence Berkeley National Laboratory, California, USA

11.55 – 12.15 **AFC Doctoral Prize 2022 Winner Talk / AFC03**
"Unraveling Catalysis: Harnessing the Power of X-rays and Quantum Chemistry"
Corentin Chatelier
CEA-Grenoble, IRIG, MEM, NRX, France

12.15 – 12.30 **Poster and talk prizes**
Closing remarks

12.30 – 13.00 **Lunch box**
Departure

ABSTRACTS – PLENARY SESSION

K01

Teasing out the secrets of subtle protein dynamics

Arwen Pearson[1], Briony Yorke[2], Helen Ginn[3]

[1] University of Hamburg, [2] university of Leeds, [3] DESY, Hamburg, Germany

Structural biology experiments are focusing increasingly with the small, subtle motions underpinning protein function. Our collective understanding of how these subtle motions translate to functional impact is limited, and this is hampering efforts in protein engineering and drug development. The Protein Machinists' lab develops conceptual frameworks for defining, modelling and navigating the protein's conformational space, focusing on the representation of protein entities (RoPE). The first tool of RoPE is a simple and powerful visualisation of conformational space from experimental data. This talk will illustrate with examples how these tools help find the narrative in modern multi-dataset experiments and drive hypothesis development

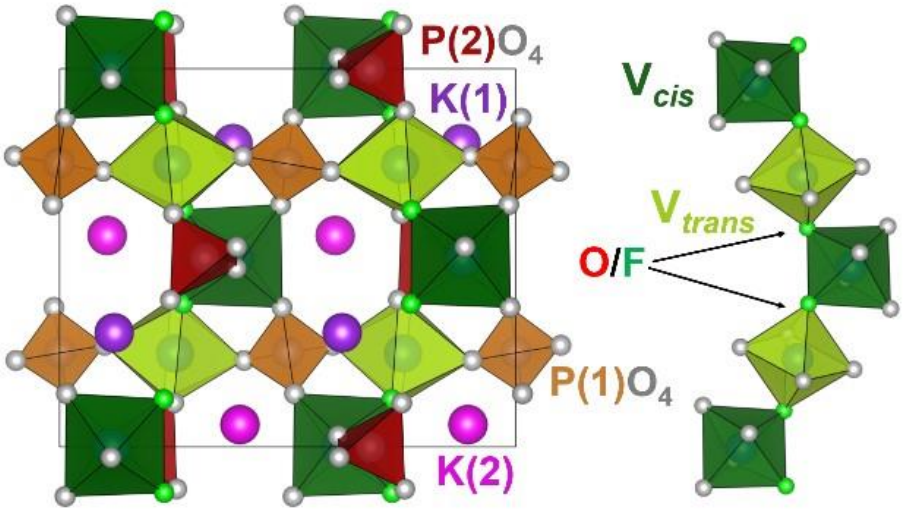
ABSTRACTS – PLENARY SESSION

K02

The challenging crystallography of mixed-anion inorganic compounds

Dr. Romain Wernert
University of Oxford, United Kingdom

Solid state compounds form the basis of modern technologies and are very often based on transition metal oxides. The combination of multiple anions in a single compound is challenging due to the strong thermodynamic preference of the cation(s) towards only one of them. However, progress have been made in the synthesis of such compounds using soft topochemical reactions for example. While some of these mixed anion compounds are relatively easy to characterize due to a large difference in their ionic radii and their X-ray scattering factor, it is not the case of oxyfluorides. To precisely characterize the anion arrangement or absence of thereof, a multi-technique approach combining spectroscopies with conventional diffraction techniques is necessary. This will be illustrated though the example of $K_xVPO_4F_{1-y}O_y$- battery materials whose crystal structure at various O/F substitution rate and K^+ deintercalation rate could only be understood with the help of EXAFS, ^{31}P NMR spectroscopy and DFT calculations along with powder X-ray diffraction.



Crystal structure of $KVPO_4F$, built from corrugated chains of VO_4F_2 octahedra with alternating cis *and* trans *fluorine arrangement. In $KVPO_4F_{0.5}O_{0.5}$, F and O are statistically distributed on the green site.*

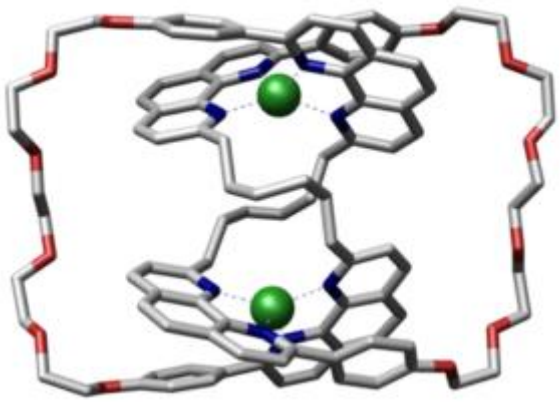
HONOUR GUEST – PLENARY SESSION

K03

Molecular Machines and Motors: A Historical Perspective

Prof. Dr. Jean-Pierre Sauvage
University of Strasbourg, France

In a conference entitled «Molecular Machines and Motors: A Historical Perspective» Dr. Jean-Pierre Sauvage will talk about various fields related to molecular sciences such as coordination photochemistry and solar energy conversion, CO_2 electrocatalytic reduction, chemical topology (interlocking or knotted rings), light-induced charge separation, multifunctional porphyrins as models of the photosynthetic reaction centre as well as molecular machine prototypes. His group got interested in this new field more or less by accident, which demonstrates that jumping in a new field can be very beneficial. The merit of his group goes to a large extent to the young and less young researchers he has been working with.



Knotane – Source Wikipedia

ABSTRACTS – INDUSTRY PLENARY SESSION

K04

Crystallographers in the pharma world of drug discovery

Dr. Laure Delarbre
Sanofi R&D France

Sanofi is a world-leading pharmaceutical company, committed to providing drugs to patients in a wide array of therapeutic areas, such as vaccines, oncology, immune inflammation, neurologic and metabolic diseases. Its research teams bring together scientists with a wide variety of backgrounds, from cell biologists and veterinaries to artificial intelligence specialists, including crystallographers. We will describe the main steps of drug research and development, the daily work of multidisciplinary project teams and how the role and missions of small-molecule crystallographers and macromolecule crystallographers concur to the objectives of a pharmaceutical company. Finally, we will provide examples of crystallography impact on drug research projects

ABSTRACTS – CHEMISTRY

C01

Structural Analyses and Properties of Complex Sulphides in the Cr-Sn-S System

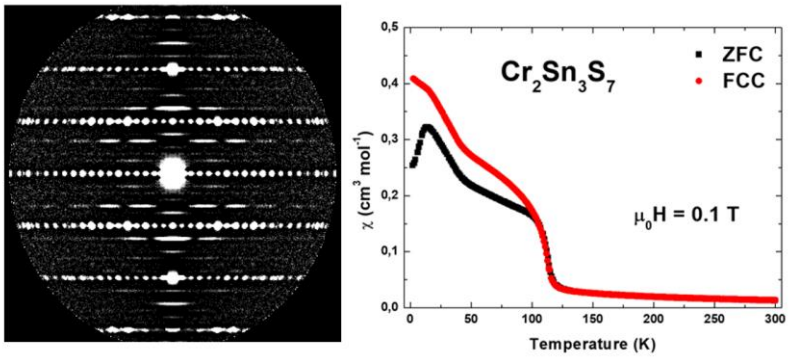
Florentine Guiot[1], Vincent Dorcet[1], Emmanuel Guilmeau[2], Bernard Malaman[3], Thierry Schweitzer[3], Pierric Lemoine[3], Carmelo Prestipino.[1]*

[1] Univ. Rennes, CNRS, ISCR-UMR 6226, F-35000 Rennes, France ; [2] Laboratoire CRISMAT, UMR 6508, CNRS, ENSICAEN, 14050 Caen, France; [3] Institut Jean Lamour, UMR-CNRS 7198, Université de Lorraine, 54011 Nancy, France

The design and optimization of thermoelectric (TE) materials rely on the intricate balance between thermopower (S), electrical resistivity (ρ) and thermal conductivity (κ). Perfecting such a balance is the key to reach high TE performances - determined by the dimensionless figure of merit $ZT = S^2T/\rho\kappa$ - necessary to improve energy recovery systems and thermoelectric cooling devices.(1) Among the most promising TE materials at medium temperature, complex copper-based sulphides are of double interests as they are usually made of eco-friendly and low cost elements(2) and exhibit intrinsically low thermal conductivity.(3) However, the use of copper-based sulphides in TE devices is limited by the lower TE performances of the *n*-type materials compared to those of the *p*-type.(4) Hence, it appears necessary to develop more performant *n*-type sulphide materials.

In this context, we have synthesized and studied the structural and physical properties of two phases in the Cr-Sn-S ternary system: $Cr_2Sn_3S_7$ and Cr_2SnS_4. The former is characterised by a semi-ordered crystal structure and a n-type semiconductor behaviour, and the latter is characterised by a complex crystal structure (Figure 1) and a p-type semiconductor behaviour. The intrinsic structural complexity of these materials leads to very low thermal conductivities, which are promising features to develop new performant thermoelectric materials.

In this presentation, I will (i) present results obtained from X-ray diffraction, scanning and transmission electron microscopies, magnetic measurements, spectroscopy techniques and transport measurements and (ii) discuss on the relationships between chemical compositions, crystal structures and properties (TE and magnetic) of $Cr_2Sn_3S_7$ and Cr_2SnS_4. (5)



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(1) Hébert, S.; Berthebaud, D.; Daou, R.; Bréard, Y.; Pelloquin, D.; Guilmeau, E.; Gascoin, F.; Lebedev, O.; Maignan, A. J. Phys.: Condens. Matter 2016, 28 (1), 013001.
(2) Caballero-Calero, O.; Ares, J. R.; Martín-González, M. Adv. Sustainable Syst. 2021, 2100095.
(3) Powell, A. V. Journal of Applied Physics 2019, 126 (10), 100901.
(4) Guélou, G.; Lemoine, P.; Raveau, B.; Guilmeau, E. J. Mater. Chem. C 2021, 9 (3), 773–795.
(5) Sleight, A. W.; Frederick, C. G. Materials Research Bulletin 1973, 8 (1), 105–107.

Keywords: Structure / Properties / Thermoelectric

ABSTRACTS - CHEMISTRY

C02

A series of supramolecular metalacyclic assemblies of CuI8MII/III/IV: how structural conformers can influence the photophysical properties

Constance Lecourt[1], Jana Schiller[1], Florent Moutier[1], Raquel Utrera Melero[1], Ingrid Suzanna[2], Valérie Marvaud[2], Guillaume Calvez[1], Karine Costuas[1], Christophe Lescop[1].

[1] ISCR (Institut des Sciences Chimiques de Rennes) - CNRS UMR 6226, INSA Rennes, Univ. Rennes ; [2] IPCM (Institut Parisien de Chimie Moléculaire) – CNRS UMR 8232, Sorbonne Université, Paris

An increasing interest is devoted to Cu(I) metal complexes as a competitive alternative to build attractive new luminescent materials for lighting and stimuli-sensitive sensor applications. The synthesis of these molecular derivatives takes advantage of both the various accessible photophysical properties and the large flexibility of the coordination sphere of this ion.[1] Inspired by a general synthetic approach previously developed,[2] a series of polymetallic metalacyclic assemblies of general formula $[Cu_8(dppm)_8(CN)_xM](PF_6)_y$ (dppm = bis(diphenyphosphino)methane) was synthetized, denoted hereafter as CuI8MII/III/IV, where MII = Pd, Pt, Ni (x = 8, y = 2), MIII = Fe, Co (x = 10, y = 1) and MIV = Mo, W (x = 11, y = 1) (Figure 1).

Polycyanometallate precursors, such as $M(CN)_4^{2-}$, $M(CN)_6^{3-}$ and $M(CN)_8^{4-}$, are acting as templates to connect the binuclear precursor '$\{Cu_2(\mu 2\text{-}dppm)_2\}$'[2] into octanuclear metallacycle assemblies of Cu(I) ions. The nature of the central metalloligand has a crucial influence on the structural arrangement of the conformer, the crystal packing and the photophysical properties. For example, on one hand, molecular derivatives with M = Pd(II) crystallize in two conformers that show luminescence properties sensitive to external stimuli. On the other hand, the open-shell electronic structure for M = Fe(III), Mo(IV), W(IV) leads to strong absorbing compounds overall the visible range. Moreover, by adding these paramagnetic centres in the middle of the edifice, we are targeting to elaborate multifunctional molecular materials.[3]

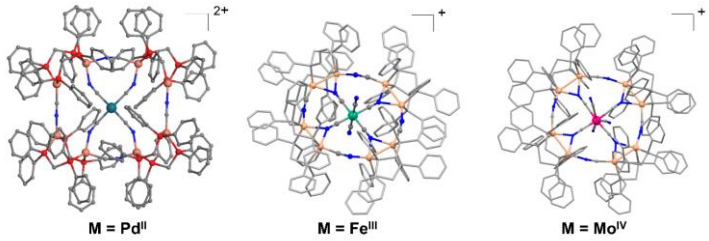

Figure 1. Structures of CuI8PdII, CuI8FeIII and CuI8MoIV derivatives.

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[1] a) Yam, V. W.-W.; Au, V. K.-M.; Leung, S. Y.-L. Chem. Rev. 2015, 115, 7589; b) Czerwieniec, R.; Leitl, M. J.; Homeier, H.H.H.; Yersin, H. Coord. Chem. Rev. 2016, 325, 2; c) Perruchas, S. Dalton Trans. 2021, 50, 12031; d) Lescop, C. Chem. Rec. 2021, 21, 544.

[2] a) Lescop, C. Acc. Chem. Res., 2017, 50, 885; b) Evariste, S.; Khalil, A.M.; El Sayed Moussa, M.; Chan, A. K.-W.; Hong, E. Y.-H.; Wong, H.-L.; Le Guennic, B.; Calvez, G.; Costuas, K.; Yam, V. W.-W.; Lescop, C. J. Am. Chem. Soc., 2018, 140, 12521. c) El Sayed Moussa, M.; Khalil, A. M.; Evariste, S.; Wong, H.-L.; Delmas, V.; Le Guennic, B.; Calvez, G.; Costuas, K.; Yam, V. W.-W.; Lescop, C. Inorg. Chem. Front. 2020, 7, 1334-1344; d) Khalil A. M.; Xu, C.; Delmas, V.; Calvez, G.; Costuas, K.; Haouas, M.; Lescop, C. Inorg. Chem. Front. 2021, 8, 4887 ; e) Moutier, F.; Schiller, J.; Lecourt, C.; Khalil, A. M.; Delmas, V.; Calvez, G.; Costuas, K.; Lescop, C. Chem. Eur. J. 2022, doi.org/10.1002/chem.202104497.

[3] Bridonneau, N. ; Quatremare, P. ; von Bardeleben, H. J. ; Cantin, J-L. ; Pillet, S. ; Bendeif, E-E. ; Marvaud, V. Eur. J. Inorg. Chem. 2018, 370-377

Keywords: Cu(I)/conformers/luminescence

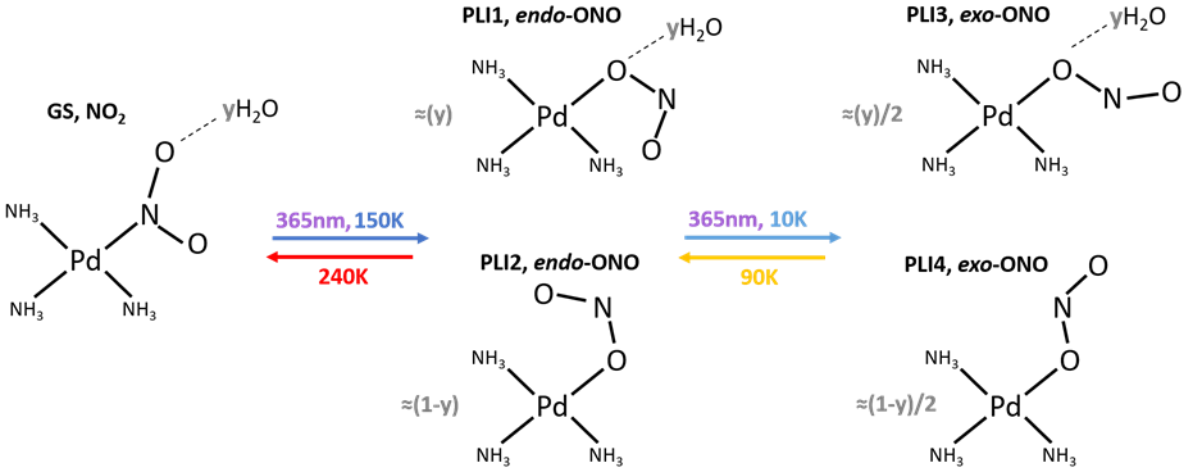
ABSTRACTS - CHEMISTRY

C03
Linkage isomerism in a photoswitchable palladium nitro complex

Artem Mikhailov[1], *Krzysztof A. Konieczny*[1, 2], *Sébastien Pillet*[1], *Dominik Schaniel*[1]

[1] Universite´ de Lorraine, CNRS, CRM2, UMR 7036, Nancy 54000, France; [2] Institute of Advanced Materials, Wrocław University of Science and Technology, Wybrzeże Wyspiańskiego 27, 50-370 Wrocław, Poland

The properties of photoinduced linkage isomers (PLIs) in a square planar complex $[Pd(NH_3)_3NO_2]^+$ being a part of the complex salts $[Pd(NH_3)_4][Pd(NH_3)_3NO_2][MOx_3]\cdot yH_2O$ (M = Cr (Cr), Rh (Rh), Co (Co), Ox = oxalate) were investigated. It was shown by XRD, that the irradiation at 365 nm at 150 K of the single crystals of Cr, Rh and Co leads to the formation of endo-ONO isomers PLI1 and PLI2. PLI1 and PLI2 differ in their intermolecular surrounding, where the partial occupation of a neighboring site by a solvate water molecule prevents the photoinduced formation of PLI2 (see Scheme 1). Further, on the example of Cr compound, PLIs were photogenerated at 10 K. Photocrystallographic analysis reveals the formation of exo-ONO isomers PLI3 and PLI4, which are not sufficiently stable at 80 K. Thus, four different PLIs can be photogenerated in $[Pd(NH_3)_3NO_2]^+$, moreover, the type and population of PLIs could be influenced by the amount of solvent H_2O molecules, by the type of $[MOx_3]_3$- and by the temperature of the photogeneration. A gradual heating of the PLIs showed that endo-ONO and exo-ONO isomers in the complexes have a different thermal stability and different decay paths. Namely, the exo-ONO isomer decays to the endo-ONO, further heating of the endo-ONO leads to its decay to the GS nitro isomer NO_2. Thus, the thermal decay of exo-ONO and endo-ONO isomers occurs via the scheme exo-ONO→endo-ONO→NO2 (see Scheme 1).



Scheme 1: The photo- and thermally induced transformations in $[Pd(NH_3)_4][Pd(NH_3)_3NO_2][MOx_3]_3$-y H_2O, M = Cr (Cr), Rh (Rh), Co (Co), Ox = oxalate). The coefficients roughly represent the populations of the linkage isomers.

Keywords: photocrystallography / isomerism / metastable states

ABSTRACTS - CHEMISTRY

C04

Structural and theoretical study of the unexpected flexibility of CALF - 20 MOF

Joanna Drwęska[1], Filip Formalik[2,3], Kornel Roztocki[1], Randall Q. Snur[3], Leonard J. Barbour[4], Agnieszka Janiak[1]

[1] Faculty of Chemistry, Adam Mickiewicz University, Poznań, Poland; [2] Department of Micro, Nano, and Bioprocess Engineering, Faculty of Chemistry, Wroclaw University of Science and Technology, Wroclaw, Poland; [3] Department of Chemical and Biological Engineering, Northwestern University, Evanston, Illinois, United States; [4] Department of Chemistry and Polymer Science, Stellenbosch University, Stellenbosch, South Africa

Abstract Calgary Framework 20 (CALF-20) is a readily obtained and reproducible zinc-based metal-organic framework that exhibits excellent durability in the CO_2 sorption process[1]. Previous reports indicate that the stability of materials may be attributed to their potential flexibility[1,2]. To verify this, we found the proper conditions for a single-crystal-to-single-crystal phase transition of CALF-20, hereafter referred to as CALF-20α_s (s – solvent molecule). A new phase dubbed CALF-20β_s is formed after heating CALF-20α_s at 80°C for seven days, during which a water molecule is statistically ligated to half of the zinc cations in the MOF. This phenomenon causes changes in the 3-D framework, resulting in a decrease in porosity.

To better understand the nature of the CO_2 sorption process at the atomic level, we carried out an in situ single-crystal X-ray diffraction experiment under controlled CO_2 gas pressure using an environmental gas cell, thus obtaining CALF-20αCO_2. The experiment revealed that no structural changes occur upon the gas sorption and allowed us to identify real carbon dioxide positions in the framework. We also determined that CO_2 interacts with CALF-20 through electrostatic interactions.

Theoretical analysis of CALF-20 flexibility proved that the water molecule is a key factor in stabilizing the structure, resulting in exceptional stability of the framework in the presence of bases, acids, and steam. Nevertheless, under anhydrous conditions, the energetically favourable phase is CALF-20α, which is also reflected in the experimental results. Furthermore, we predict the presence of an additional CALF-20β contracted phase, which can only exist at very low temperatures.

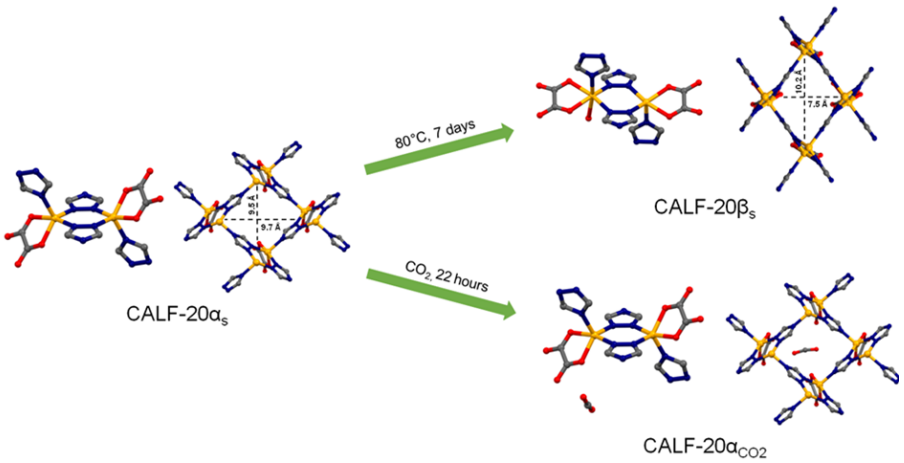


Figure 1: Structural changes of CALF-20 under different conditions

Keywords: CALF-20 / CO_2 capture / Flexible materials

ABSTRACTS - CHEMISTRY

C05
Building New Metal-Organic Frameworks from Iron-Thiolate Layers

Nusik Gedikoglu[1], Pablo Salcedo[1], Long Nguyen[2], Philippe Poizot[1], Lorenzo Stievano[2], Thomas Devic[1]

[1] Institut des Matériaux Jean Rouxel de Nantes, Nantes, France ; [2] Institute Charles Gerhardt Montpellier, Montpellier, France

Metal-Organic Frameworks (MOFs) are a class of materials constructed by the coordination of metal ions or clusters by organic ligands into three-dimensional structures [1]. MOFs exhibit a diverse range of structures due to the ability to control the arrangement of metal ions and ligands. Their porosity provides a large surface area, making them suitable for gas adsorption, catalysis, and storage of small molecules. Ongoing research focuses on developing MOFs with tailored properties for specific technological challenges. We are interested in synthesizing MOFs with high valance cations, such as Fe^{3+}, and sulfur-bearing ligands. Sulfur atoms are soft and polarizable and display a strong affinity towards heavy metal ions and coinage metals. Thus, MOFs with sulfur ligands might be suitable for various applications such as capture for heavy metal ions [2] and advanced drug delivery agents when coupled with plasmonic nanoparticles [3]. Meanwhile, high valance cation Fe^{3+} usually leads to MOFs with high hydrothermal and chemical stability. The redox activity of Fe^{3+} and strong charge transfer between the Fe3+ and sulfur groups might lead to compounds with improved electrical conductivity. Such compounds might be promising candidates for electrochemical energy storage applications [4].

We investigated the reactivity between sulfur-bearing ligand, 2,5-disulfanylbenzene-1,4-dicarboxylic acid (H4DSBDC), and Fe^{3+} sources and isolated a series of compounds. Under solvothermal conditions, H4DSBDC ligand and Fe^{3+} cations lead to iron-thiolate layers, formulated [Fe(DSBDC)-]n, which exhibit superior thermal and chemical stability compared to their class of materials. The addition of cations of various charges in the synthesis medium leads to 3D networks where iron-thiolate layers are bridged through cations (see Figure). Flexible structures and redox-active behavior is observed for some materials.

We will here describe the synthesis and the crystal structure of these compounds together with their electrochemical properties. The crystal structures were solved using powder and single crystal XRD techniques. The materials were characterized thoroughly: thermal and chemical stability of the materials were identified by thermogravimetric and diffraction techniques, oxidation states of Fe cations in the materials were investigated through Mossbauer spectroscopy, and the flexibility of a structure and its effects were studied by ss-NMR spectroscopy.

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Keywords: metal-organic frameworks / new materials / electrochemistry

ABSTRACTS - CHEMISTRY

C06

Study of Organolanthanide Complexes with Cyclononatetraenyl (cnt) and Cyclooctatetraenyl (cot) Ligands

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Sandwich complexes of lanthanides have recently attracted a considerable amount of interest due to their applications as Single Molecule Magnet (SMM). In this context, a series of heteroleptic lanthanide sandwich complexes ligated by the cyclononatetraenyl (Cnt) and the cyclooctatetraenyl (Cot) ligand [Ln(Cot)(Cnt)] (Ln=Tb, Dy, Er, Ho, Yb, and Lu) has been reported in 2021 by Tricoire et al.. The investigation of the coordination behavior of the Cnt ligand along the series showed different coordination patterns in the solid-state depending on the size of the corresponding lanthanide ion without altering its overall anisotropy.
This presentation will discuss the coordination properties of those large aromatic rings through the study of various organo-lanthanide coordination complexes, such as the sandwich complexes or bi-metallic complexes.

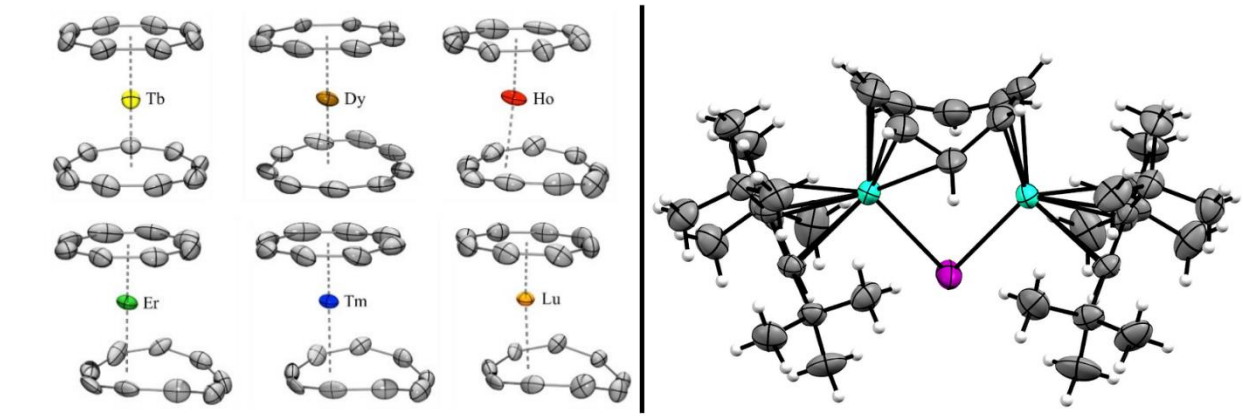

Figure 1: (left) ORTEP of lanthanide sandwich complexes [Ln(cot)(cnt)]; (right) ORTEP of a bi-metallic lanthanide complex Dy2I(cnt)(cpttt)2

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Keywords: organometallic / complexes / lanthanide

ABSTRACTS - CHEMISTRY

C07

Structure Dynamics in a Spin-Crossover Crystal Probed with Ultrafast Electron Diffraction and Serial Femtosecond X-ray Diffraction

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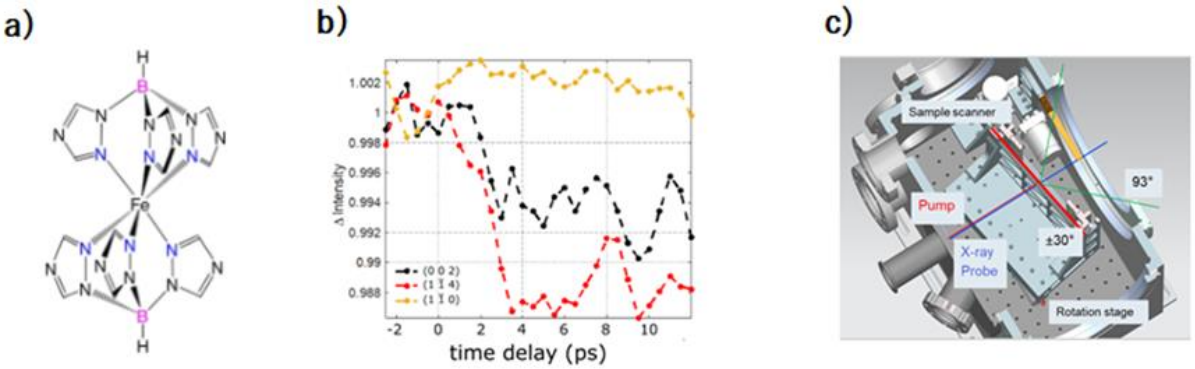
Spin-crossover (SCO) compounds are photoswitchable materials that exhibit a reversible phase transition between a low-spin (LS) and a high-spin (HS) state that can be triggered under various external perturbations (e.g. light). Photoinduced SCO compounds are of interest for applications as solar energy converters and photo-redox catalysts arising from their spin transition and electronic dynamics1. Among the SCO systems, $Fe(HB(tz)_3)_2$ (HBTZ) (tz= 1,2,4-triazol-1-yl) (Fig.1) is very attractive: it has an extremely abrupt first-order, isostructural spin transition above the room temperature with an high resilience upon repeated photoswitching[2]. HBTZ can be deposited on different substrates to prepare smooth, dense, highly oriented nanocrystalline films with the orthorhombic c-axis normal to the surface[3]. These unique properties of HBTZ open a promising perspective for fast response photoswitches at room temperature.

Our team has performed MeV-UED experiments on HBTZ nanofilms to investigate the photoinduced SCO dynamics with ~100 fs temporal resolution, but due to the epitaxial orientation of the thin film with the c-axis perpendicular to the surface and the high electron energy (MeV), only Bragg peaks of the (h k 0) family have been detected. This renders it impossible to refine the atomic motions during the HBTZ phase transition and study the lattice volume expansion along the c-axis, which requires Bragg peaks (h k l) ($l \neq 0$). However, from our electron diffraction results, one noteworthy feature is a plateau behavior observed for time delay 2 ps to 12 ps (Fig. 2b), which is after the initial photoinduced SCO and before the latter lattice response. This is indicative of the formation of the photoexcited high-spin state in unrelaxed lattice.

In this contribution, we will discuss the MeV-UED results and the serial femtosecond XRD as complementary tool to the MeV-UED for having a better understanding of the molecular dynamics in the HBTZ thin film. We carried out the fs time-resolved XRD experiments at the FXE end-station at the European XFEL. Pump laser pulses centered at 267 nm have been used to photoexcite the sample. Thanks to the high resolution of X-rays, we have been able to study Bragg peaks from more crystal lattice planes than in electron diffraction and, since the X-rays beam is also more focused than in MeV-UED, we could even study the Bragg peaks shift, elucidating the development of nanometric strain on the thin film in ultrafast timescale. Finally, we propose a new method for the full 3D structure determination of photoinduced high spin state of the HBTZ with rotational X-ray diffraction (Fig. 1c).



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Keywords: XFEL/MeV/UED/UXRD/spin crossover/structure determination

ABSTRACTS - CHEMISTRY

C08

Effect of nanostructuring on the structural and physical properties of functional molecular nanomaterials: Applications to bioactive glasses

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Over the past two decades, there has been significant progress in the development and use of a new generation of mesoporous bioactive glasses (MBGs) in the medical field. Based on their biocompatibility and unique bioactive properties, these nanobiomaterials are mainly used for bone reparation and regeneration processes. They have the ability to form bioactive bonds with the body's cells and tissues through the presence of carbonate hydroxyapatite (HCA) phase. When exposed to a physiological medium or simulated body fluid (SBF), the innovative glass undergoes a conversion process leading to the formation of an amorphous calcium phosphate phase (ACP). This amorphous coating gradually transforms into a crystalline apatite layer (HCA) and becomes firmly attached to the surface of bio-implants. It is important to monitor the formation and growth of HCA on mesoporous bioglass (MBGs) surfaces when they are submerged in SBF, as well as to study their structure. In this work, we present a comparative study of the bioactive properties of two MBGs, 58S and 70S30C, determined in two physiological media: c-SBF (classic Simulated Body Fluid) and m-SBF (modified Simulated Body Fluid). We used X-ray diffraction technique to detect the presence of a crystalline structure: carbonate hydroxyapatite (HCA) in order to validate the bioactivity of two synthetic bioglasses. However, since this technique is only sensitive to crystalline structure, we also utilized X-ray total scattering coupled with pair distribution function (PDF) analysis to analyze the development and transformation of an ACP layer into HCA layer. Other complementary experimental techniques such as spectroscopy Raman and scanning electron microscopy SEM were also used to track the presence of different species on the bioglasses.

ABSTRACTS - CHEMISTRY

C09
Crystallography for molecular magnetism

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We are coordination chemists working in the field of molecular magnetism. We synthesize compounds of different sizes containing one or more metal centers (mostly single molecules, but also coordination polymers, 2D networks, nanoparticles) and study their magnetic properties (single molecule magnets, molecular quantum bits). The magnetic behavior of our compounds strongly depends on the surrounding of the metal centers in these molecules: coordination sphere of the metal ion, nature of the bridging ligands, presence or absence of intermolecular interactions. The interaction between metal centers depends on the nature and amplitude of the orbital overlap of the magnetic orbitals. Thus, an important part of our work consists in synthesizing our compounds in crystal form in order to obtain the crystallographic structure of the molecules. This structural information is crucial to our understanding of the magnetic behavior of our compounds. It enables us to define magnetic interaction models, establish magneto-structural correlations and rationalize them through theoretical studies. This presentation will attempt to demonstrate this approach through examples drawn from our current research projects.

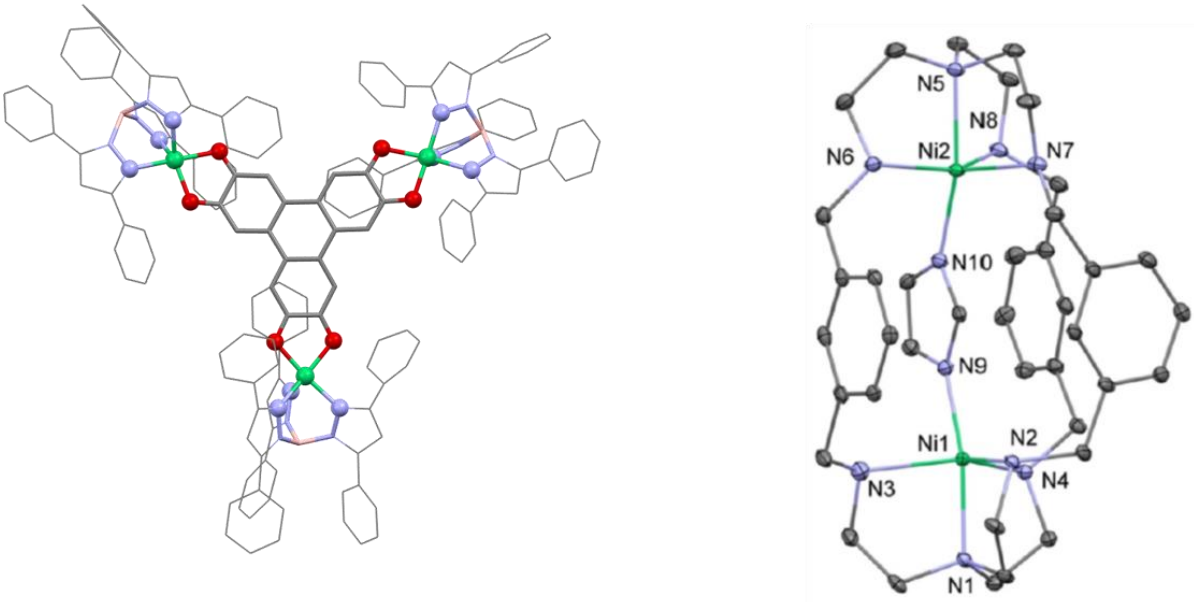

Figure 1: Examples of Nickel-based magnetic complexes obtained in our group

Keywords: Molecular magnetism / coordination chemistry

ABSTRACTS - CHEMISTRY

C10

Enhanced Circular Dichroism and Polarized Emission in an Achiral, Low Bandgap Bismuth Iodide Perovskite Derivative

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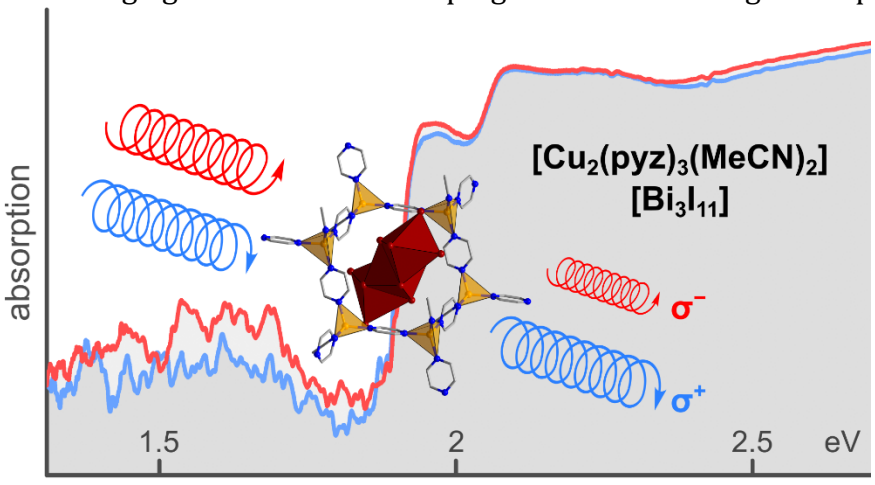
Lead halide perovskites and related main group halogenido metalates offer unique semiconductor properties and diverse applications in photovoltaics, solid-state lighting, and photocatalysis. Recent advances in incorporating chiral organic cations have led to the emergence of chiral metal-halide semiconductors with intriguing properties such as chiroptical activity and chirality-induced spin selectivity, enabling the generation and detection of circularly polarized light and spin-polarized electrons for applications in spintronics and quantum information. However, understanding the structural origin of chiroptical activity remains challenging due to macroscopic factors and experimental limitations. In this work, we present an achiral perovskite derivative $[Cu_2(pyz)_3(MeCN)_2][Bi_3I_{11}]$ (CuBiI; pyz = pyrazine; MeCN = acetonitrile), which exhibits remarkable circular dichroism attributed to the material's non-centrosymmetric nature. CuBiI features a unique structure as a poly-threaded iodido bismuthate, with $[Bi_3I_{11}]^{2-}$ chains threaded through a cationic two-dimensional coordination polymer. The material possesses a low, direct optical band gap of 1.70 eV. Notably, single crystals display both linear and circular optical activity, with a large anisotropy factor of up to 0.16. Surprisingly, despite the absence of chiral building blocks, CuBiI exhibits a significant degree of circularly-polarized photoluminescence, reaching 4.9%. This value is comparable to the results achieved by incorporating chiral organic molecules into perovskites, typically ranging from 3–10% at zero magnetic field. Our findings provide insights into the macroscopic origin of circular dichroism and offer design guidelines for developing materials with high chiroptical activity.



Keywords: bismuth / iodide / optical activity

ABSTRACTS - PHYSICS

P01

Innovative synthesis at high pressure and high temperature of new compounds in the ternary B-C-Si system.

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Boron carbide is a ceramic of utmost importance for both its mechanical and nuclear properties. It has been selected as the (only) neutron absorber material of generation IV nuclear reactors: this is due to both the presence of ^{10}B isotopes and to the high structural stability under neutron irradiation. Boron carbide, B_4C, is also the third hardest materiel known after diamond and cubic Boron nitride. In addition, it is a very light ceramic which makes it a perfect shielding material. Despite its very good mechanical properties, when Boron carbide is impacted beyond is Hugoniot Elastic Limit, the ceramic shows a gradual loss of strength. This could be the consequence of a weakness inside the atomic structure, at the C-B-C chain level. The Boron atom doesn't remain at high constraint and some vacancies appear in the structure.
In this study, we try to reinforce B_4C by the formation of a ternary compound Boron-Carbon-Silicon. The Silicon doping in B_4C has already shown a positive effect on Boron carbide amorphization, however, no synthesis of a pure Si doped Boron carbide has been referenced. One of the main obstacles is the low solubility of Silicon inside B_4C. To push back this solubility, which is about a few atomic percent at High Temperature, we added High Pressure to High Temperature during the synthesis. Some promising and reproducible results have been observed both by *ex situ and in situ XRD* characterizations on B-C-Si samples. Some Rietveld refinements have been used to process the synchrotron data. The important weight of the synchrotron data has forced us to proceed batch refinement, including quantitative phase analysis.

This work is supported by Agence Nationale de Recherche (project ANR-21-CE08-0018)

ABSTRACTS - PHYSICS

P02

Observing growth of silver thin films in situ with synchrotron radiation

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Silver thin films are used in a number of applications (e.g., transparent and conductive electrodes and architectural glazing), which require a continuous layer with thickness below a few nanometers. However, Ag films grown by magnetron sputtering have the tendency to form 3D-structures on weakly interacting substrates. It is reported that the use of gas additives (particularly N_2 [1]) and the growth of Ag on amorphous Ge seed layers [2] allow for obtaining a continuous layer at an earlier deposition stage. We want to gain a thorough understanding of the mechanisms during the deposition process. Synchrotron radiation makes it possible to investigate the growth process in real-time [3]. In particular, we use real-time in situ grazing incidence small angle x-ray scattering (GISAXS), grazing incidence diffraction (GID) and substrate curvature measurements. With our approach we can establish a correlation between atomic-scale mechanisms and resulting film morphology and understand the role of additives in modifying Ag growth. Our results we will provide guidelines for further optimisation of the growth. I will present the general experimental approach and first results showing the impact of the additives on the film morphology. The main focus will be set on the topic of the analysis of the GISAXS data obtained for the curved sample.

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Keywords: silver thin films / synchrotron / GISAXS

Acknowledgements: The work is performed within the frame of the ANR-DFG project IRMA. The synchrotron experiments were performed at the SIXS beamline at SOLEIL.

ABSTRACTS - PHYSICS

P03

Characterization of [Au]core@[Ag]shell nanorods by ultra low frequency Raman scattering

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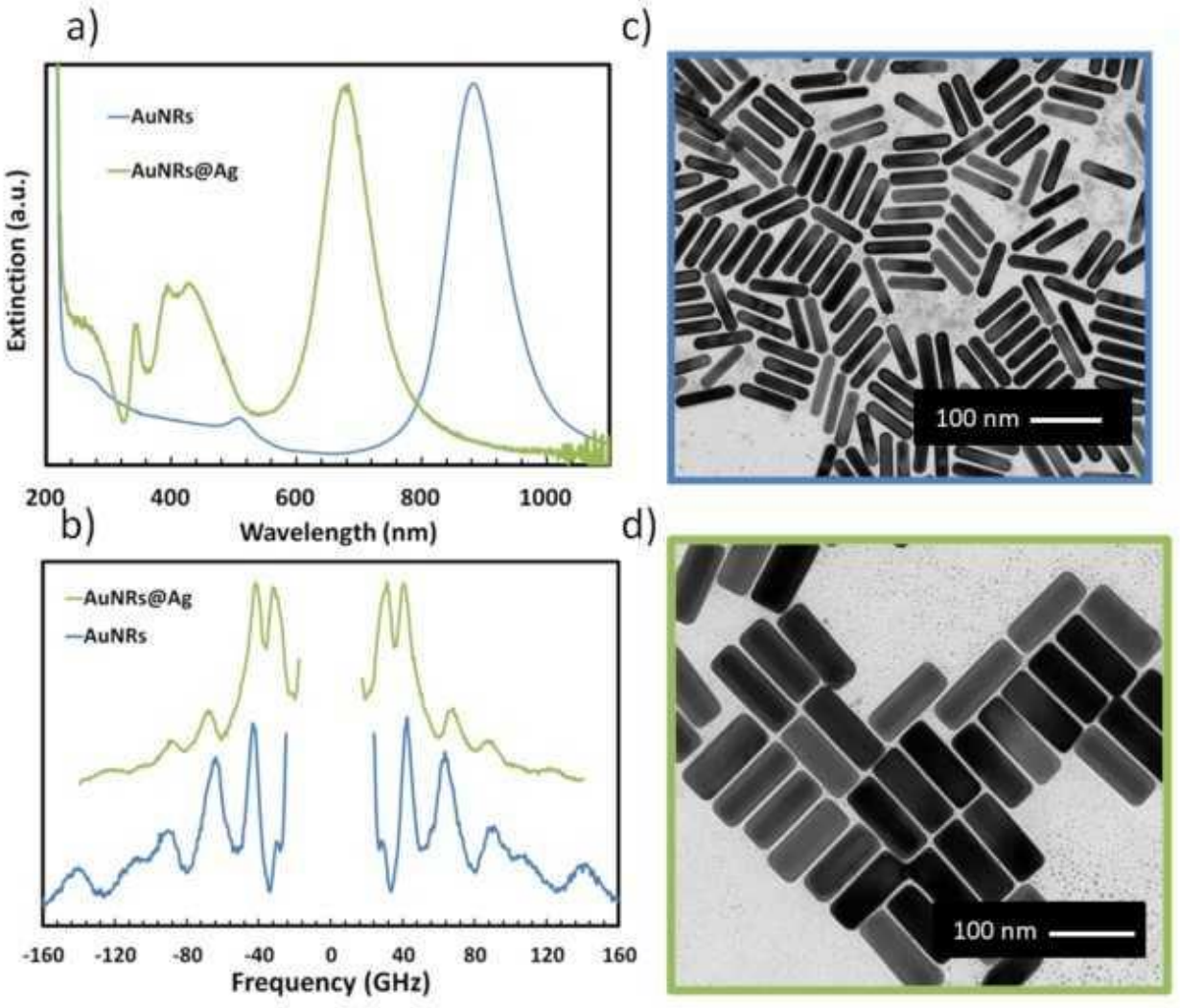
Low-frequency Raman scattering (LFRS) is a high-performance tool for detecting acoustic phonons confined in solids of small size relative to the light wavelength, such as metallic nanoparticles. It is now clearly established that spherical polycrystalline nanoparticles of gold or silver (isotropic elasticity) mainly scatter light by the quadrupolar vibrational mode (l=2). Their single-crystal counterparts also have a very specific LFRS signature [1]: in the case of nano-objects with cubic elasticity, the l=2 mode splits into two modes, with irreducible representations E_g and T_{2g}. However, the question remains open for inhomogeous nanoparticles composed of two metals with cubic elasticity. Do they retain the signature of elastic anisotropy? In theory, core-shell systems consisting of a single-crystal Au core surrounded by an Ag shell are expected to exhibit specific features in their LFRS spectra, provided that the shell growth is epitaxial [2].

We propose here an adapted protocol for the synthesis of monodisperse gold nanorods (AuNRs) and their subsequent coating with silver, leading to [Au]core-[Ag]shell nanorods (AuNRs@Ag). Changing the amount of silver used in the protocol, core-shell AuNRs@Ag with Ag shell of various thicknesses were synthesized (e = 3 nm, 9 nm, and 15 nm). Using transmission electron microscopy, we observed epitaxial and homogeneous growth of silver on the AuNRs. Logically, looking at the UV-vis extinction spectra measured from AuNRs@Ag in which the Ag shell thickness was progressively increased, localized surface plasmon resonances both related to gold and silver are observed, with an increasing contribution from the band corresponding to the Ag shell. We also show that the LFRS is sensitive to the crystallinity of AuNRs and the thickness of the silver shell, as well. This work aims to illustrate the contribution of LFRS as a technique for characterizing metal nanoparticles that leads to useful information, complementary to that provided by microscopy and spectrophotometry.

(a) Extinction spectra of pure AuNRs (blue curve) and Au@Ag core-shell NRs with Ag shell thicknesses e = 9 nm (green curve). (b) Anti-Stokes and Stokes LFRS of AuNRs and AuNRs@Ag. TEM images of (c) AuNRs and (d) AuNRs@Ag are also shown.



References:
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Keywords: Raman scattering / core-shell / nanorods

ABSTRACTS - PHYSICS

P04

Unraveling intermolecular protein interactions: insights from molecular rotors

Yevgeniya Karibjanova[1], *Isaac Rodriguez-Ruiz*[1], *Sébastien Teychené*[1]

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In protein solutions, the interplay between short-range attraction and long-range molecular repulsion leads to a complex phase behavior giving rise to transient dense liquid phases, equilibrium clusters, and kinetically arrested states. The role of liquid-liquid phase separation (LLPS) in crystal nucleation is still debated, and the high viscosity of dense phases is known to be a parameter limiting nucleation within the dense phase. Therefore, achieving suitable conditions for protein crystallization remains a significant challenge and is in great demand in the field of structural biology.

In light of these challenges, we propose a methodology based on fluorescence lifetime imaging microscopy (FLIM) to comprehensively characterize protein interactions, investigate the impact of intermediate liquid phases on crystal nucleation, and potentially facilitate the prediction of the conditions suitable for protein crystallization. Since phase transition implies local changes in the solution structure, we propose to use environment-sensitive fluorophores, molecular rotors. The fluorescence lifetime of the rotors correlates with the local viscosity or free volume present in their immediate surroundings. Moreover, molecular rotors have the ability to interact with proteins within the solution, and the nature of these interactions can be influenced by various environmental parameters.

We have studied the relation between protein-protein interactions and fluorescence lifetime in lysozyme solutions using Sulforhodamine-B (SRh-B) as a molecular rotor. Interestingly, the evolution of fluorescence lifetimes exhibited a nonmonotonic trend (Fig. 1A) upon the gradual introduction of salt (precipitant). Subsequent small-angle X-ray scattering (SAXS) analyses confirmed that changes in the solution's structure factors occurred near the minima of the lifetime curves, indicating a transition from a repulsive to an attractive interaction regime. Similar trends were confirmed for different salts in Hoffmeister series.

We also characterized the dense liquid phase formed upon lysozyme LLPS. Preliminary results have shown higher fluorescence lifetime and intensity inside the protein-rich droplets (Fig.1B), indicating a crowded environment for the rotor. Droplets formed in different conditions (concentration of salt or protein) exhibit different fluorescence lifetime. Rotor-protein interactions have additionally been studied using X-ray Crystallography and found to be non-specific. SAXS data has shown no changes in the structure factors of the protein solutions in presence or absence of SRh-B. Therefore, we propose fluorescence lifetime of molecular rotors as an indicative of protein interactions, and FLIM as a promising tool for characterizing transient phases in nucleation studies.

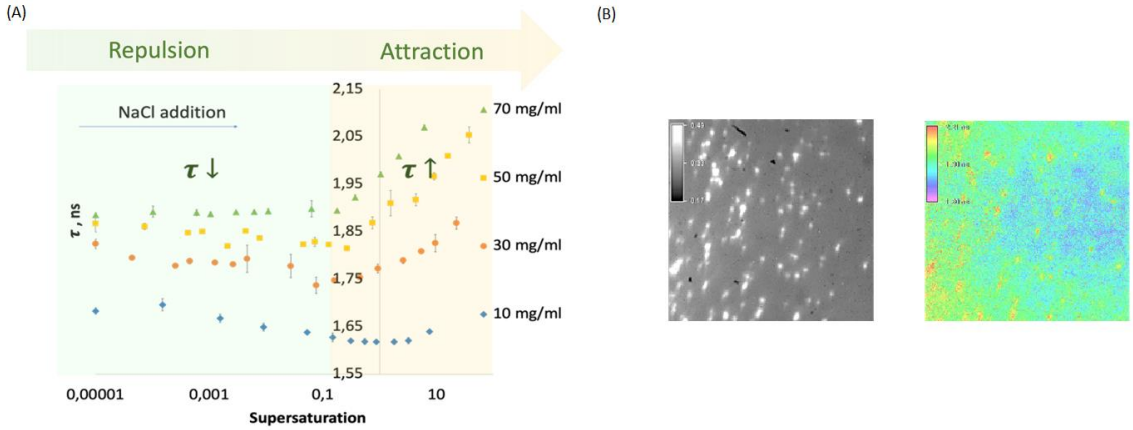

Figure 1: (A) Evolution of fluorescence lifetime with supersaturation in the lysozyme solutions, (B) Fluorescence intensity and lifetime maps of LLPS formed in lysozyme solution of 50 mg/ml at 7 wt% NaCl

Keywords: protein crystallization, molecular interactions, molecular rotors

ABSTRACTS - PHYSICS

P05

Structural study of piezoelectric $LnCa_4O(BO_3)_3$ at high pressures and cryogenic temperatures

F. Azrour[1], F.P. Yu[3, 4], S.J. Zhang[3,4], J. Long[1,2], R. Viennois[1], P. Hermet[1], M.Beaudhuin[1], J. Rouquette[1]

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In spite of their small piezoelectric effect, quartz crystals continue to dominate as components for frequency control since the early days of radio engineering, due to their extremely sharp resonance curves, which are stable with respect to temperature and aging. Lanthanide oxyborate crystals, LnCOB ($LnCa_4O(BO_3)_3$ with Ln = La to Yb are found to be quite attractive as they exhibit excellent piezoelectric properties more than three times that of quartz in a large temperature range (300≤T ≤1000°C). However up to now, they were not investigated either at cryogenic temperatures and/or under high-pressure in spite of important concern in piezoelectric application in orbit and LT/HP-sensors. This is therefore the aim of the present study.

Ln-oxyborates crystallise in the monoclinic Cm space group which was found to be stable from our DFT-calculations. Ln- and Ca-ions exhibit distorted edge-shared octahedra connected by BO3 groups along *c**, Fig a. Optimal piezoelectric properties were found to be linked to the Ln-ion radius (best for rLn^{3+} similar to rCa^{2+}). High quality single crystals [1] have been characterized in house using 4-circle Bruker D8 diffractometer using Mo K_α-radiation in the 100 ≤ T ≤ 350K temperature range. Additionally, Nd and Er-based oxyborate crystals were studied at high pressure (1 ≤ P ≤ 6 GPa) and low temperature (10-300 K) using single crystal X-ray diffraction at the XPRESS beamline at ELETTRA synchrotron using 0.5 Å wavelength photons and PILATUS 3S 6M detector. High-pressure experiments were conducted using a diamond anvil cell using helium as pressure transmitting medium.

Whereas NdCOB was found to keep the Cm monoclinic structure in the 100-350K temperature range, LaCOB transformed below 150K into an incommensurate structure, Fig b. Additionally, ErCOB and TmCOB were found to exhibit (Ca/Ln) site disorder, Fig c, which could explain the observed lower piezoelectric properties [1].

Cryogenic structural characterization under high pressure led to a similar incommensurate transition of NdCOB to that previously determined for LaCOB below 30K in the entire pressure range. However, such a transformation was absent for ErCOB, probably due to the site-disorder previously described. These structural characterizations have now to be compared with on-going cryogenic temperatures and/or under high-pressure piezoelectric measurements.

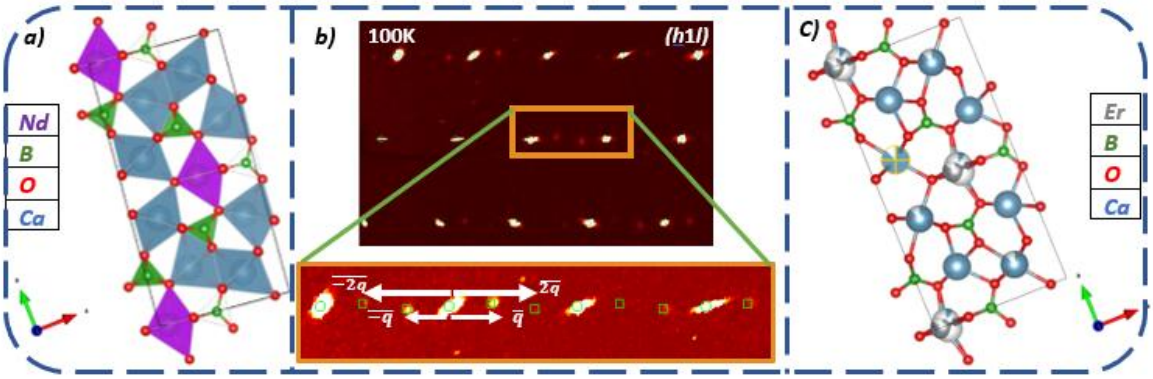

Figure 1: a) Cm monoclinic structure of NdCOB, b) h1l reconstruction of the reciprocal space showing the appearance of satellites for q⃗=0.625a+0.01c*, c) site-disorder characterized for ErCOB (R1=2.9%)*

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Keywords: Piezoelectric, transition, monoclinic, $ReCa_4O(BO_3)_3$

ABSTRACTS - PHYSICS

P06

X-ray optical activity in achiral crystals of coordination complexes

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Natural Circular Dichroism (NCD), the differential absorption of right- and left-circularly polarized light, is an efficient technique, widely used in pharmacology, for absolute configuration determination of chiral molecules and macromolecules. While this technique is most often applied in the UV-visible energy range, core-level optical activity using X-rays demonstrates some unique features such as element specificity and orbital selectivity, but is limited to single crystal measurements.[1] The majority of X-ray Natural Circular Dichroism (XNCD) studies reported in the literature have been performed using synchrotron sources, especially the ESRF ID12 beamline at the K-edge of light transition metals[2] or at the $L_{2,3}$-edge of heavier atoms, such as lanthanides.[3]

XIMTEX project (Experimental investigation of microscopic theory of Natural Circular Dichroism using X-rays) aims to explore XNCD signal produced by coordination complexes, understand the influence of the metal, the ligands and the symmetry on the shape and intensity of the signal and experimentally verify the XNCD theoretical angular dependencies predicted by Natoli et al.[4] using model compounds.

Investigations were performed at the ID12 beamline on the model compounds $[FeCl_4][PPh_4]$ and $[CuCl_4][TEA][TMA]$ crystallizing in the achiral tetragonal space groups *I*-4 and *P*-42_1m, respectively. For the first time, is was proved that achiral systems can generate XNCD. The polar and azimuthal dependences of these crystal systems were fully characterized, supporting theoretical predictions.[4]

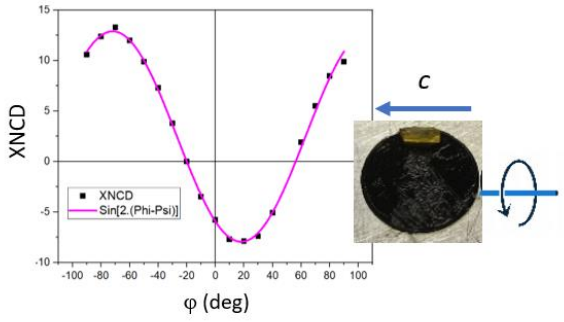

Figure 1: Angular (azimuthal) dependence of the XNCD intensity of an achiral iron coordination complex.

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ABSTRACTS - PHYSICS

P07

Milk fat polymorphism in the butter-making process

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Raw milk and cream contain fat confined in microscopic globules. These globules are surrounded by membranes made of phospholipids and proteins, which prevent the fat in milk from merging into a single mass. After a step of physical maturation, butter is produced by intense agitating of the cream, the so-called churning process, which damages these membranes and allows fat globules to undergo massive coalescence and clumping. Churning produces small butter grains floating in the water-based portion of the cream. In the final stage, butter is kneaded under fast cooling conditions.

The manufacture of puff pastry butters requires features that guarantee industrial or artisanal manufacturers control over the puffing of their pastries. Fat crystallization needs to be mastered in order butters to have the appropriate texture. Fat crystallization is dependent on the fatty acid composition [1] but it can be modulated by the physical and mechanical treatments applied to it, whether in cream or after churning. To meet users' requirements in terms of functionality and to comply with regulations (quality and origin labels) and the butter expected characteristics, fat crystallization can be directed only during the phases of physical maturation of the cream and final kneading + cooling of the butter. The difficulties associated with this technological choice lie in the seasonal and geographical variability of the fatty acid composition [2], and in the multiple stages involved in the transformation process that imposes physical constraints to the material. Ideal puff pastry butter must be tough enough to ensure water isolation between the laminated dough and plastic enough to follow the folds without breaking (Figure). The optimization of butter's properties after shear and temperature treatments needs to be fully understood to fit production's parameters for every season.

To ensure a better comprehension of the mechanisms involved during butter manufacturing, we have adopted a multiscale approach. Nanoscale characterization has been realized by X-ray diffraction to identify the three main crystal polymorphs of milk fat (α, β, β') and different lamellar packings (2L, 3L, …) [3]. Differential scanning calorimetry and rheological measurements allow to identify polymorphic changes and changes in the microstructure, respectively [4]. Such measurements have been conducted at different stages of the process.

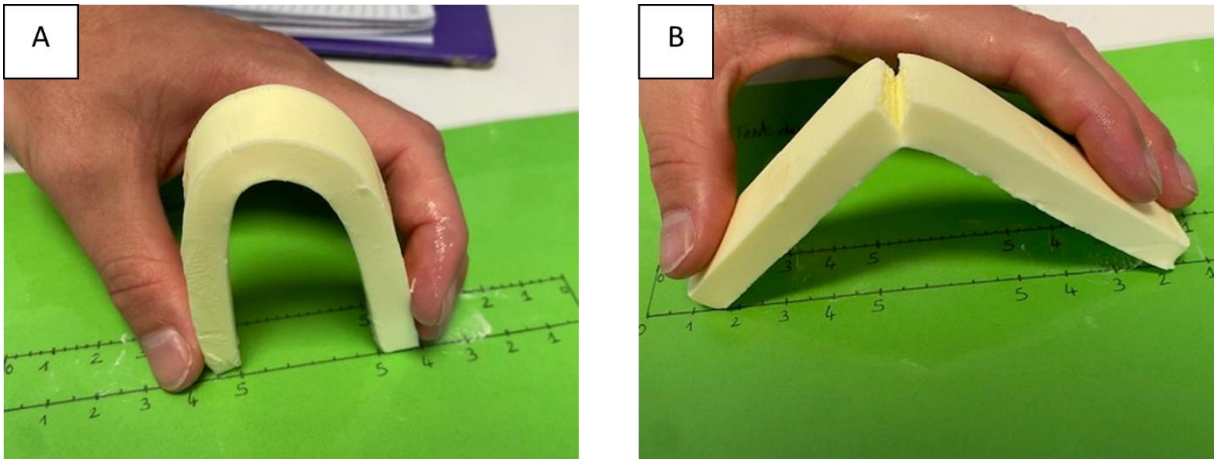

Figure: A- Puff pastry butter with optimal plasticity at 4 °C. B- Puff pastry butter breakable at 4 °C

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Keywords: Polymorphism / Milk Fat / Texture

ABSTRACTS - PHYSICS

P08

Hexagonal 2H-SiGe in Nanostructures by Phase Transformation

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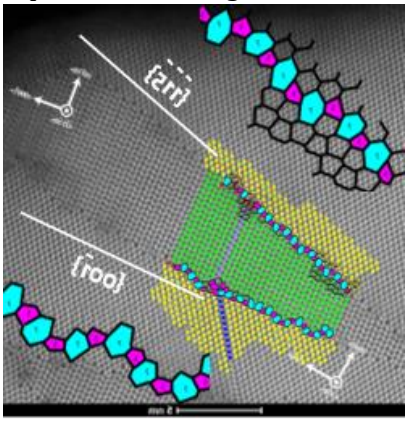
We developed a method to partially transform Si and Ge nanostructures from the cubic 3C phase to the hexagonal 2H phase. For the transformation process to occur, a HSQ resist is spun around nanostructures to embed them. The HSQ is then heated, densifying it to silica. The densification of the HSQ induces shear stress on the nanowires, combined with heat, this is the driving force of transformation. Our research investigates the mechanisms behind this phase transformation.
To distinguish between the 3C and 2H phase (S)TEM is used. The phase transformation for both Si and Ge in <111>-axial nanowires is characterized by the formation of multiple 2H bands that are formed through the whole radius of the nanowire. These domains have a length of up to 35 nm along the nanowire at 600°C. Only nanowires that have a diameter smaller than 240 nm show transformation. The martensitic transition between the 3C and 2H domains exhibit a {115} habit plane. Along this habit plane, the atoms arrange themselves in rings of 5 and 7 atoms that repeat in a 5-7-5-7-chain. This arrangement leaves no dangling bonds, stabilizing the interface. A less common {113} habit plane has also been identified.
The situation for <100>-axial nanowires is different. Transformation rarely occurs and if any the 2H domains are small and triangular; mostly twinning is observed. A shear stress along the <110> crystal direction may account for this. For <111>-axial nanowires, this <110>-direction is almost parallel to the nanowire; for <100>-axial nanowires it is parallel to the substrate. Thus, asymmetry is likely to have an important impact. Therefore, <111> vertical nanofins in an array have been studied. Fins feel an asymmetric shear stress in the outer fins. This has also been confirmed by Comsol simulations. For the outer nanofins, more than 400 nm long 2H-bands are observed, and none for the center fins. For <100> vertical nanofins, asymmetry is expected to also result in bands through the fin.
Also, Ge shows phase transformation but with a different threshold temperature of respectively 400°C instead of 550°C. Therefore, taking advantage of the lower transformation temperature for Ge, a transformed domain can be localized in a Ge layer within a Si/Ge/Si heterostructure. There is no sign of diffusion between Si and Ge, indicating a martensitic nature. However, Si in contact with Ge-2H is transformed below the threshold temperature, hinting at a cascade effect of bond breaking



Keywords: Transformation 2H-phase STEM

ABSTRACTS - BIOLOGY

B01

Unusual peptide-binding proteins guide pyrroloindoline alkaloid formation in crocagin biosynthesis

Sebastian Adam, Dazhong Zheng, Andreas Klein, Carsten Volz, William Mullen, Sally L. Shirran, Brian O. Smith, Olga V. Kalinina, Rolf Müller & Jesko Koehnke

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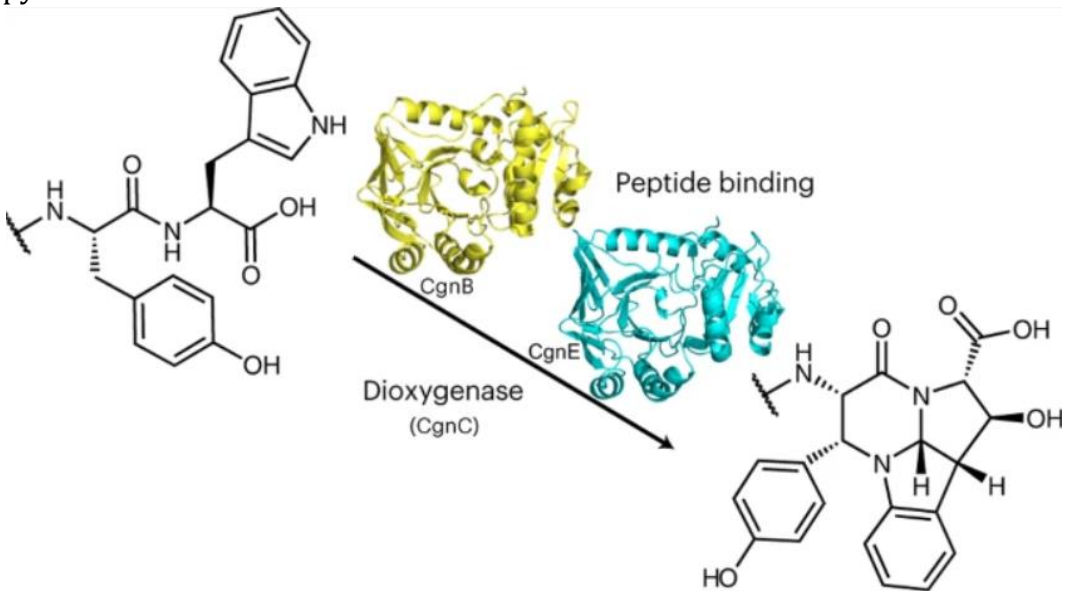
Ribosomally synthesized and post-translationally modified peptide natural products have provided many highly unusual scaffolds. This includes the intriguing alkaloids crocagins, which possess a tetracyclic core structure and whose biosynthesis has remained enigmatic. Here we use in vitro experiments to demonstrate that three proteins, CgnB, CgnC and CgnE, are sufficient for the production of the hallmark tetracyclic crocagin core from the precursor peptide CgnA. The crystal structures of the homologues CgnB and CgnE reveal them to be the founding members of a peptide-binding protein family and allow us to rationalize their distinct functions. We further show that the hydrolase CgnD liberates the crocagin core scaffold, which is subsequently N-methylated by CgnL. These insights allow us to propose a biosynthetic scheme for crocagins. Bioinformatic analyses based on these data led to the discovery of related biosynthetic pathways that may provide access to a structurally diverse family of peptide-derived pyrroloindoline alkaloids.



Production of the crocagin main motif, a pyrroloindoline fused to a tetrahydropyrimidinone moiety, by its main enzymes, CgnB, CgnC, and CgnE.

ABSTRACTS - BIOLOGY

B02

Time-resolved crystallography for the study of protein dynamics using X-ray Free-Electron Lasers (XFELs)

Ronald Rios-Santacruz[1], Harshwardhan Poddar[2], Jacques-Philippe Colletier[1], Nicolas Coquelle[1], Elke De Zitter[1], Samantha Hardman[2], Michiyo Sakuma[2], Shigeki Owada[3], Kensuke Tono[3], Takehiko Tosha[3], David Leys[2], Derren J. Heyes[2], Nigel S. Scrutton[2], Giorgio Schirò[1], Martin Weik[1]

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XFELs (X-ray Free-Electron Lasers) are a revolutionary technology that has transformed the field of crystallography in the last ten years. These lasers produce extremely bright and ultra-short X-ray pulses that allow the observation of molecular dynamics on unprecedented temporal and spatial scales. In structural biology, XFELs enable time-resolved serial femtosecond crystallography (TR-SFX) that allowed studying molecular processes such as those involved in photosynthesis, vision, and enzyme catalysis. Recently performed TR-SFX studies by our research team have elucidated photoactivated intermediated states in a newly discovered photoreceptor. The ability to observe these processes in the native state of proteins and in real-time allows for a better understanding of the molecular dynamics, which has important implications from fundamental research to drug design.

Keywords: XFEL, structural biology, photoreceptors, time-resolved crystallography

ABSTRACTS - BIOLOGY

B03
Emergence of order from proteins under nucleation

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Protein crystallization has a crucial role in the determination of protein structures, but also in the efficient delivery of therapeutic pharmaceuticals. Despite its tremendous importance, the underlying mechanisms that lead from a soluble macromolecule to a well-ordered solid phase, *i.e.* crystal, are still not completely understood. Although nucleation is commonly considered as a single-step process, an increasing number of publications report multi-stepped protein nucleation with intermediate steps such as oligomerization, cluster formation or phase separation[1]. The complex and transient nature of these processes makes them especially hard to monitor, yet mechanistic understanding of the nucleation process would allow effective guidance of crystallization towards desired outcomes, *e.g.* polymorphs of interest, desired crystal size and number, *etc*.

Human insulin is an especially interesting model system for studying protein nucleation and the competitive effects of the underlying steps that lead to nuclei formation, because of its well-studied polymorphism, in terms of molecular conformations and crystal forms which can be induced by pH, anions or small ligands. Using X-Ray Cross-Correlation Analysis (XCCA) we can monitor the local symmetry of the particles that assemble from protein molecules as order emerges from the amorphous state on the way to formation of a crystal.

In XCCA experiments, the intensity of a 2D speckle pattern is correlated between two different q vectors separated by an angle Δ using the correlation function $C_{q1,q2,\Delta} = \langle I_{q1},I_{q2},\varphi+\Delta\rangle / \langle I_{q1},\varphi\rangle\langle I_{q2},\varphi\rangle$ [2]. Certain symmetries in the speckle pattern will result in peaks in C, e.g. hexagonal symmetries lead to a peak for Δ=60°. XCCA is increasingly used in experiments on nanoparticle assemblies[3], while it can also reveal otherwise hidden symmetries, as demonstrated for colloidal glasses[4,5].

Using XCCA we aim to characterize the low-range symmetry of protein nuclei formed under crystallization native conditions. Such experiments could pave the road for identifying off-pathways that act as roadblocks preventing crystallization, and developing generic experimental tools to track these processes and aid crystallization.

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Keywords: human insulin, protein nucleation, xcca, polymorphism

ABSTRACTS - BIOLOGY

B04

Optimising Protein Production for Functional and Structural Studies: Example of the NYN proteins

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RNA maturations and modifications play a crucial role in the living by enabling a variety of RNAs to fulfil their functions. These include the cleavages carried out by ribonucleases, which are characterised by binding domains enabling them to interact with RNAs (RBDs) or other partners and catalytic domains holding/responsible for their cleavage function. In 2006, a new globular domain associated with RBDs was identified in several eukaryotic and prokaryotic proteins including YacP [1] and N4BP1. Structural predictions suggested an α/β domain with 5 strands (S) and 4 helices (H), perfectly congruent with secondary structures observed in the PIN (PilT N-terminal) and FLAP catalytic domains. The presence of 2 conserved Asp (D) at the end of S1 and S4 as well as 2 acidic residues in the N-terminal part of H2 and H4 suggests a potential nuclease function for this new domain, which was then named NYN for N4BP1, YacP-like Nuclease domain [2]. Subsequently, this PIN-like domain superfamily was split into 5 major structural subclasses including “NYN” (true NYN) and “PRORP” (Protein only RNase P [3]) [4]. Although several proteins with NYN domains have already been identified, the huge diversity of domains associated with NYN suggests a variety of functions and targets, from which very little is known at present.

The structural (X-ray crystallography, cryoEM, SAXS), biophysical (DLS, NanoDSF) and functional (in vitro cleavages, RNA-seq) study of these proteins can lead to a better understanding of their role in vivo and, in the longer term, to the possibility of diverting their function to solve agronomic or health problems. To perform these analyses, particular attention was given to the optimisation of their cloning, production and purification. Such optimisation approaches can be readily extended and applied to other samples.

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Keywords: Ribonucleases / RNAs / Sample optimisation

ABSTRACTS - BIOLOGY

B05

Structural characterization of the BLUF photoreceptor OaPAC using time-resolved crystallography

Anaïs Chretien[1], Sabine Botha[2], David von Stetten[3], Joachim Schulz[1], Arwen Pearson[4], Kristina Lorenzen[1], Robin Schubert[1]

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Reactions of biological macromolecules can be studied by time-resolved crystallography (TRX), as it provides high special and temporal resolution. TRX requires the ideally instantaneous initiation of the reaction of interest, to ensure that fine structural changes are not "blurred out" by the different molecules in the crystal reacting in a non-synchronized manner. Luckily, naturally light-sensitive signaling proteins such as photoreceptors allow the investigation of biochemical processes from the picosecond time range, as these can be efficiently and uniformly activated by a short laser pulse in pump-probe experiments. Photoreceptors containing a Blue-Light sensor Using Flavin (BLUF sensor domain) have one of the fastest photoreaction cycles of all known photoreceptors and are therefore highly attractive for a broad range of applications. Understanding the signal transmission in the complete BLUF sensor–effector system is however required to pave the way for future bioengineering applications.

In this project, a photoactivated adenylate cyclase (PAC) protein is studied. PAC contains a BLUF sensor domain coupled to an adenylyl cyclase effector domain, involved in the conversion of ATP into cAMP. For this, PAC protein was produced, purified and characterized to verify stability and functionality. Complete X-ray crystallography datasets of PAC were collected in its dark state with ATP bound in the active site. Pump-probe time-resolved crystallography using XFELs (Fig. 1) in combination with ultrafast spectroscopy was performed. Structural changes around the FMN chromophore for several time points could already be elucidated and additional experiments are ongoing.

The data shows the rotation of the Gln48, which initiates a change in FMN hydrogen bond network. Notably, the observed kinetics from the TR-SFX experiments differ from spectroscopy data in solution. Onset of the displacement of Met92 initiates signal transmission to the adenylate cyclase domain.

Cryo-trapping experiment also enabled to capture reaction time-points in a light-activated state by flash-freezing protein crystals after few seconds of illumination. By this, we could show that PAC also adopts the so called "Tryptophan-in" conformation (Trp90) in the light activated state, which is known from some BLUF proteins, but was not seen for BLUF-PAC so far. The performed experiments help to better understand the signalling process of PAC, which can serve as a basis to design novel optogenetic tools.

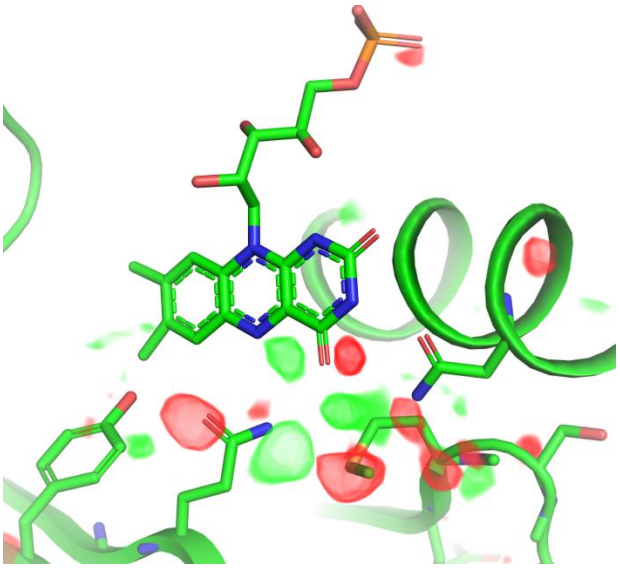

Figure 1: Time-resolved serial femtosecond crystallography study of BLUF activation via blue-light excitation of FMN chromophore in OaPAC. Fo(light)-Fo(dark) difference map for pump–probe time delay Δt = 2.3 μs contoured at +3 σ (green) and -3 σ (red).

Keywords: BLUF / Photoreceptors / XFEL

ABSTRACTS - BIOLOGY

B06

Capabilities and developments at the SPB/SFX Scientific Instrument at the European XFEL

Raphael de Wijn[1], Valerio Bellucci[1], Johan Bielecki[1], Sarlota Birnsteinova[1], Juncheng E[1], Sravya Kantamneni[1], Konstantin Kharitonov[1], Chan Kim[1], Jayanath Koliyadu[1], Faisal Koua[1], Romain Letrun[1], Diogo Melo[1], Adam Round[1], Abhisakh Sarma[1], Tokushi Sato[1], Marcin Sikorski[1], Egor Sobolev[1], Patrik Vagovic[1], Dmitrii Zabelski[1], Richard Bean[1]

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The unique capabilities of X-ray Free Electron Lasers (XFELs), and especially high repetition-rate XFELs, offer an unprecedented ability to generate large quantities of serial crystallography data. At the European XFEL (EuXFEL), the megahertz repetition rate and the very short and ultra-bright X-ray pulses provide a fertile ground for the development and usage of innovative experiment designs and methods. The Single Particles, Clusters, and Biomolecules & Serial Femtosecond Crystallography (SPB/SFX) scientific instrument is dedicated to conducting SFX and single particle time resolved experiments, by providing optimised configurations exploiting the unique EuXFEL capabilities. At the same time, the instrument provides the flexibility and setups for new developments. This presentation will give an overview of the capabilities of the SPB/SFX scientific instrument and associated user facilities at EuXFEL. In addition, some of the science performed to date will be presented, focusing on SFX and including megahertz repetition rate serial crystallography, time-resolved studies, as well as new methods. An outlook to future capabilities and upgrades will also be shown.

Keywords: XFEL, Serial Fentosecond Crystallography, Scientific Instrument

ABSTRACTS - BIOLOGY

B07
The *in crystallo* Optical Spectroscopic toolbox

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The rise of time-resolved macromolecular crystallography (TRMX) prompted an interest in optical spectroscopy as a companion biophysical characterization methods applicable to molecules both *in crystallo* (*ic*OS) and in solution [1]. Its use was since extended to the study of radiation damage [2,3] and the characterisation of metalloproteins [4] and flavoenzymes redox states [5]. The use of the extremely time coherent XFEL pulses for the collection of single diffraction images from slurries of micro-crystals renewed the interest in TRMX, and with it the use of *ic*OS. In several XFEL diffraction studies [5,6], the position of various UV-vis absorbance peaks are compared in different conditions and timescales to determine the electronic states. However, these shifts in a spectrum are often narrow, and may change for a different crystal or orientation. This is owed to, among others, Rayleigh scattering within the crystalline material [7], reflexion on the crystal surface, and polarization of light through the crystalline lattice. Fortunately, these phenomena can be modelled, and corrected if necessary, and the quality of a spectrum can be assessed via a series of diagnostic plots. The present communication summarises these procedures and showcases a suit of utilities encased in a graphical interface for the an easy analysis of spectroscopic data gathered on macromolecular crystals.

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ABSTRACTS - BIOLOGY

B08
The mechanism of the SorC protein family revealed

Markéta Šoltysová[1], Jana Škerlová[1], Petr Pachl[1], Jiří Brynda[1], Jiří Nováček[2], Karel Škubník[2], Michal Babiak[2], Irena Sieglová[1], Milan Fábry[1], Pavlína Řezáčová[1]

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Proteins belonging to the SorC family are transcription regulators involved in carbohydrate metabolism and quorum-sensing control [1, 2]. They consist of a DNA-binding domain (DBD) and an effector-binding/oligomerization domain (EBD). Based on the sequence similarity of DBDs, the family is further divided into two subfamilies, SorC/DeoR and SorC/CggR. Several SorC structures of their EBDs and two full-length proteins have been determined so far [3-6]. However, there was no structural information on their complex with their cognate DNA (operator).

We used an integrative approach of structural biology combining X-ray crystallography and cryogenic electron microscopy (cryo-EM) to structurally characterize two SorC prototypes in the complex with their operators, *bs*DeoR and *bs*CggR. X-ray and cryo-EM studies of the full-length repressor-DNA complexes gave us low-resolution information revealing the general mechanism of binding. To gain insight into the repressor-DNA atomic interactions, we determined 2.3 and 2.1 Å resolution crystal structures of *bs*DeoR and *bs*CggR DBDs in complex with half-operator duplexes. Putting all the information together, we propose the SorC protein family mechanism of the function, which might be used for further basic and applied research.

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ABSTRACTS – AFC DOCTORAL PRIZE 2022

AFC01

Discovery and structure-function studies of key enzymes behind the non-canonical ZTGC-DNA found in phages

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The non-canonical nucleobase 2-aminoadenine (or diaminopurine, Z) replaces adenine (A) in the DNA of cyanophage S-2L (1) and several related bacteriophages. In their genomes, 2-aminoadenine and thymine (T) form the Z:T pair bound by a fully saturated triple hydrogen bond. Together with the standard G:C pair they form double-stranded ZTGC-DNA which is resistant to bacterial hosts' restriction enzymes (2). However, the enzymatic pathway allowing for A-to-Z substitution was previously unknown. Using the structure-function approach, I dissected this pathway and generated high to ultrahigh (1.72 - 0.86 Å) resolution crystallographic structures of four enzymes with their respective ligands. These structures explain the specificities observed in catalytic tests - or lack thereof.

In my studies, I focused on cyanophage S-2L, the originally-described bearer of 2-aminoadenine. I identified a DNA primase-polymerase PrimPol, responsible for S-2L replication, with surprisingly similar activity towards dATP and dZTP substrates. This prompted the investigation of other enzymes conserved among the phages of interest. First, I showed that the activity of DatZ, a dATP-specific triphosphatase, explains how dATP is removed at the pre-replication stage. I obtained three structures of DatZ with its substrate, cofactors and product. This allowed me to propose the two-metal-ion mechanism as a general mechanism of dephosphorylation in HD phosphohydrolases. Secondly, I found that PurZ of phage S-2L's, a key enzyme in diaminopurine production (3), is not only an ATPase but also a dATPase, contributing to dATP removal. I also identified a nucleotide pyrophosphatase MazZ as the third essential component of the conserved Z biosynthetic pathway. I showed that MazZ converts dGTP into dGMP, thus generating one of the substrates of PurZ – the precursor of 2-aminoadenine.

Finally, I characterized the structure of a Z-specific family A DNA polymerase DpoZ found in a related vibriophage φVC8, yet absent in S-2L's genome. Its crystallographic structure in polymerase-exonuclease "thumb-exo open" and "thumb-exo closed" states offers an explanation for the observed specificity. The complete description of 2-aminoadenine pathways in phages S-2L and ϕVC8 could be subjected for bioengineering purposes.

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ABSTRACTS – AFC DOCTORAL PRIZE 2022

AFC02

Resolution of the cationic distribution in $Cu_{22}Fe_8Ge_4S_{32}$ using synchrotron resonant powder diffraction: A case study and guidelines for analogous compounds

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The present work investigates the cationic distribution of a complex CuS compound belonging to a promising class of materials with significant prospects for thermoelectric generator and photovoltaic applications. We propose the use of powder resonant X-ray scattering in a combinatorial experimental approach with X-ray powder diffraction and single crystal, as a valid, general, and practical method to unravel the complex cation ordering encountered in the synthetic germanite $Cu_{22}Fe_8Ge_4S_{32}$. The generation and testing of all the possible structural models is rapid and rigorous as it is based on two modules written in python language: a module that takes care of model creation and ordering (permutation algorithm associated with some filtering and ordering algorithms) and a python parser for the refinement software (FullProf) input and output. Each possible cationic model is tested through combined Rietveld refinement of resonant X-ray data at selected edges and analyzed considering its global $\chi 2$ (Bragg contribution) agreement factor. This approach can easily integrate information coming from other techniques, as in our case EXAFS spectroscopy and single crystal X-ray diffraction. In spite of the complexity of the germanite, we were able to define the main characteristics of the cationic ordering (space group $P\bar{4}3n$): (i) the "interstitial" site 2a is fully occupied by Fe, (ii) Ge is located only on the 6d site (or the symmetry equivalent one 6c), and (iii) the remaining Fe atoms are located preferentially on the 12f site and possibly on the 6d (or 6c) site along with Ge. Moreover, we single out a probable enrichment of Ge at the expense of Fe. The approach developed in this case study can be used as a guideline for the crystal structure resolution of analogous compounds.

Keywords: thermoelectricity, complex structure, germanite, sphalerite, quaternary Cu-S, $Cu_{22}Fe_8Ge_4S_{32}$

ABSTRACTS – AFC DOCTORAL PRIZE 2022

AFC03

Unraveling Catalysis: Harnessing the Power of X-rays and Quantum Chemistry

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Replacing noble metal (Pd, Pt, Au) catalysts with inexpensive, environmentally harmless, active, selective, and stable substitutes is a big challenge for the chemical industry. Several aluminium-based complex intermetallic compounds have shown promises for alkynes and alkenes hydrogenation reactions, which are of interest in the chemical industry. It is the case for Al_5Co_2, $Al_{13}Co_4$ and $Al_{13}Fe_4$ quasicrystalline approximants. The study of their catalytic properties demands different approaches, both theoretical and experimental, in order to determine first their surface structures under ultra-high vacuum or reaction conditions, then their catalytic properties. The combination of surface science experiments (scanning tunneling microscopy, surface X-ray diffraction) and theoretical chemistry calculations (surface energies, adsorption energies and reaction pathways) allows for a better understanding of the key parameters behind the promising catalytic properties of these materials.

Keywords: Catalysis, surfaces, aluminium, intermetallics, butadiene, DFT, SXRD

POSTERS - CHEMISTRY

P-C01

In situ diffraction study of the phase transformations occurring in the thermoelectric colusite Cu26V2Sn6S32

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Interest in thermoelectric (TE) technology has been continuously growing in the last decade driven to the necessity to limiting waste heat during energy transformation. Among the most promising TE materials at medium temperature, complex copper-based sulphides are of double interests as they are usually made of eco-friendly and low cost elements[1] and exhibit intrinsically low thermal conductivity.[2,3] Derivatives of the natural mineral colusite, with general formula $Cu_{26}A_2E_6S_{32}$, (A = V, Nb, Ta, Cr, Mo, W; E = Ge, Sn, As, Sb), are an emerging class of excellent thermoelectric materials.[4] As example, the ZT value of the colusite $Cu_{26}V_2Sn_6S_{32}$ rises to near unity at 675K, making this material one of the best p-type TE in this temperature region.[5] Its performances are mainly related to the coexistence of an ordered (*P*-43*n*) and a disordered (*F*-43*m*) forms obtained after sintering at 1023 K (i.e. sample H), leading to a very low thermal conductivity.[5,6] In addition, colusite $Cu_{26}V_2Sn_6S_{32}$ is known to exhibit an intrinsic exsolution phenomenon supposed to be related to the coexistence of Sn-rich and Sn-poor colusites.[7,8]

In this study, we investigated by in-situ synchrotron powder diffraction the solid-state phase equilibrium as function of the temperature between the ordered and disordered forms of colusite $Cu_{26}V_2Sn_6S_{32}$. The use of high-resolution setup revealed a complex behavior with several phase transformations, probably related to a mutual interaction and kinetic effects.

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Keywords: Thermoelectric, phase transformations

POSTERS - CHEMISTRY

P-C02

Influence of the crystal structure and nature of the ligands on the valence of uranium in binary chalcogenides: a HERFD-XANES and RIXS study.

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Thanks to its 5*f* orbitals, uranium benefits from several valence states, from U^{3+} to U_6^+, in inorganic compounds and possesses a wide crystal-chemistry. The radial expansion of these orbitals leads to energetically close crystal field (dominating in the case of 3*d* elements) and spin-orbit (dominating in the case of 4*f* rare earths) interactions, and subsequent rich and exotic physical properties [1] (e.g. coexistence of superconductivity and ferromagnetism). Associated with a chalcogen element (Q = S, Se, Te), uranium forms inorganic compounds characterized by various crystallographic structures leading to unique uranium polyhedral structures [2] with e.g. the presence of $(S_2)_2$- dimers.

However, due to the limited number of known uranium chalcogenides, practically no experimental information on the nature of the U-Q bond is available in the literature. As a consequence, understanding the localization of 5*f* electrons of actinides in solid-state, a great challenge for theoretical physicists, remain limited to the study of oxides [3] and intermetallic materials.

To increase the field of investigation, some binary uranium chalcogenides (S, Se, Te) have been characterized and studied by HERFD-XANES and RIXS spectroscopies at the U M_4 edge. These preliminary measurements enabled to determine the oxidation states of uranium for these binary compounds. The results will be presented and discussed towards the influence of crystal structure and ligands on this oxidation state. For example, HERFD-XANES suggests different U valence states for isostructural compounds UQ_3 ($P2_1/m$).

In addition, some ternary compounds from U-M-S systems (M = 3*d* metal) have been synthetized for further investigation of their physical properties.

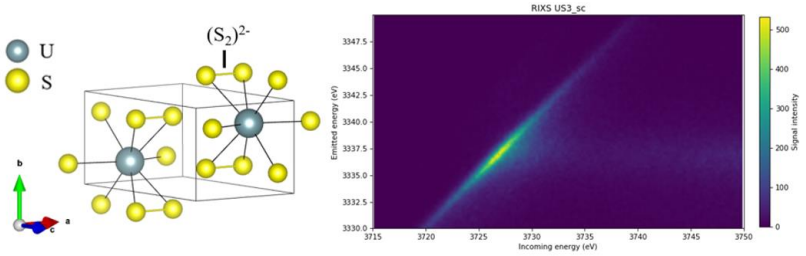

Figure 1: Crystal structure and U 3d4f RIXS map of US3 ($P2_1/m$)

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Keywords: Uranium / HERFD-XANES

POSTERS - CHEMISTRY

P-C03
DTE chemistry: contribution and limitation of single-crystal X-Ray diffraction

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Single crystal X-Ray diffraction (SCXRD) is nowadays a routine technique in most academic laboratories that occupies an essential and central position in the molecular chemist analytical toolbox. Indeed, SCXRD is not only complementarity to other technics but, very often, it also provides the ultimate proof of existence of a chemical species to researchers due to the unequivocal nature of the three-dimensional structural model resulting from this technique. Still, depending on the chemistry of interest, crystals which are obtained can be challenging for the crystallographer.

The study of organic compounds of the dithienylethenes family (DTE) is a central thematic in Rennes at the ISCR, which is driven by their very interesting photochromic behavior. The wide variety of DTE under study makes it interesting from a structural point of view with very different types of crystals and as many associated problematics in terms of measurement and structural resolution (small crystals, with low diffraction at high angle, sometimes disordered and/or twinned).

In this context, we will here illustrate the contribution of SCXRD to different topics of molecular chemistry that share the DTE organic function as a common motif: POM-DTE chemistry [1], transition-metal catalysis [2] and Single Molecule Magnets [3]. The difficulties encountered at different stages of the analysis as well as the limitations of the method will also be discussed.

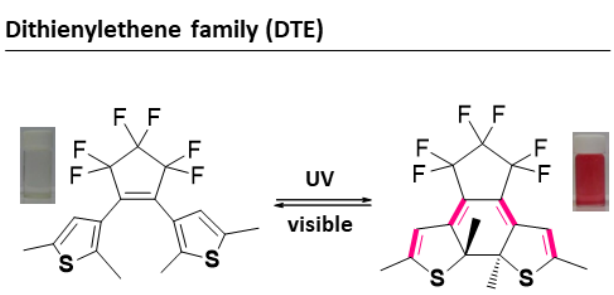

Fig 1: photochromism effect of DTE

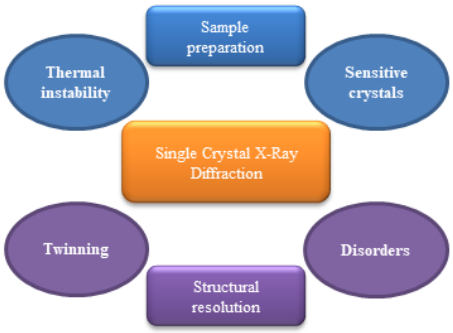

Fig 2: problems encountered with SCXRD

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Keywords: SCXRD / DTE

POSTERS - CHEMISTRY

P-C04

X-Ray structures of Magnetic Dendrimers

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Developed in the 1980s, dendrimer chemistry has opened up new perspectives in fields as diverse as catalysis, molecular electronics and artificial photosynthesis (1). Surprisingly, however, magnetic dendrimers are not common in the literature due to puzzling difficulties in their synthesis and
characterization (2). In this context, the design of such multimetallic and monodisperse starburst architectures, endowed with magnetic properties, is therefore a real challenge (3).
Following a supramolecular "complex as ligand" approach, we succeeded in this challenge by carrying out the synthesis, characterization (including X-Ray diffraction) and study of magnetic dendrimers. Our synthetic strategy essentially relies on (i) oxalate based coordination chemistry as the core of the architecture and (ii) trinuclear complexes, $LnCo_2$ for the branches (with Ln a lanthanide and Co, the Klaui ligand) that feature single-molecule magnet properties. The combination of these two building blocks allows us to obtain supramolecular and dendritic assemblies.
The versatility of this approach makes it possible to anticipate the properties according to the choice of metal cations involved in the structure (single molecule magnets or giant spin values, etc.). Thus we obtained hetero-tri-metallic magnetic dendrimers, MLn_3Co_6 and MLn_4Co_8 (with M=Co or Cr, and Ln: La, Tb, Dy, Er, …) fully characterized by X-ray diffraction (4)(5). The magnetic properties are in good agreement with the expected theoretical models. This type of compound could be of great interest for applications in information storage

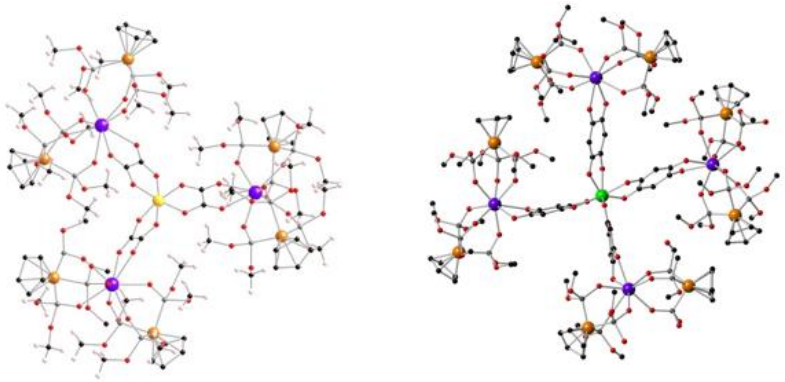

Figure 1: X-ray structures of magnetic dendrimers: $CrEu_3Co_6$ and $ZrGd_4Co_8$

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Keywords: Dendrimers, molecular magnetism, X-ray structure

POSTERS - CHEMISTRY

P-C05

Mechanochemical synthesis of theobromine cocrystals with chosen coformers. X-ray structural analysis, solubility and thermal properties

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Theobromine as a purine alkaloid can be classified as an active pharmaceutical ingredient (API) because it is a vasodilator, heart stimulant and affects the human nervous system. This compound is less soluble in polar solvents than its analogs (e.g. theophylline and caffeine). [1] This is a limitation due to its limited use in the pharmaceutical industry. One of the techniques used to improve the physicochemical properties of medicines is the synthesis of pharmaceutical cocrystals with appropriately selected coformers. This group of complexes is defined as multicomponent solids which consist of molecular and/or ionic compounds connected by non-covalent interactions. [2]
Cocrystallization experiments of theobromine using selected carboxylic acids were performed. Single crystals were obtained by cocrystallization from the solution. The X-ray structural analysis made it possible to determine the role of non-covalent interactions in the arrangements of molecules in the crystal lattice of the obtained systems. The powder patterns confirmed the possibility of synthesis of these complexes by neat or liquid-assisted grinding using a ball mill. Steady-state UV-Vis spectroscopy showed the impact of theobromine cocrystallization on its solubility in water. The high thermal stability of obtained multicomponent complexes was confirmed using TGA and DSC techniques. [3, 4, 5]

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Keywords: cocrystals, grinding, theobromine

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POSTERS - PHYSICS

P-P01

High-pressure and high-temperature transformations of chevkinite group minerals

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The subject of this work is part of a larger project on understanding the processes of mobilization and concentration of Rare Earth Elements (REE) in the Earth's crust. The chevkinite-group minerals (CGM) are dominantly monoclinic REE-Ti-Fe sorosilicates, with REE_2O_3 contents up to ~50 wt%. The crystals of chevkinite-(Ce) studied originated from the pegmatite of Harramosh, Pakistan, and had an average formula: $(Ce_{1.8}La_{0.81}Nd_{0.65}Ca_{0.44})_4Fe^{2+}(Fe^{2+}_{1.02}Ti_{0.77}Mn_{0.14}Mg_{0.10})_{2.0}Ti_{2.0}(Si_2O_7)_2$. CGM have complex chemical compositions and complicated atomic arrangements. The crystal structure of chevkinite-(Ce) is described in the literature in space groups $P2_1/a$ or $C2/m$. These variants have different but close symmetry. This varying symmetry appears to be related to temperature, pressure of the formation of the crystals, as well as later alteration processes. Both space groups belong to the $2/m$ point group, but $P2_1/a$ is of lower symmetry than $C2/m$. [1] The factors responsible for a given atomic arrangement have not been discussed in the literature. The aim of this project was to examine these factors and determine transformation pathways. We carried out a series of single-crystal X-ray diffraction measurements on natural and crystals hydrothermally altered in the laboratory. The crystal symmetry of natural and altered samples was determined as $P2_1/a$ but the natural crystals were closer to $C2/m$ symmetry than the hydrothermally altered crystals. After single-crystal XRD experiments selected crystals were annealed in an oven or loaded into Diamond Anvil Cell in order to test the influence of high pressure, high temperature on the structure of chevkinite. The experiments in the oven were carried out at temperatures ranging from 550 to 1000°C with durations ranging from 6h to 18 days. The changes due to temperature were evaluated by further comparative single-crystal XRD measurements on each of the crystals. Transformations were observed in both directions (from $P2_1/a$ to $C2/m$, from $C2/m$ to $P2_1/a$) depending on the conditions of annealing. In the other part of the project, I examined the influence of high pressure on the crystal structure of chevkinite-(Ce). We carried out a series of single-crystal XRD experiments in DAC up to 5 GPa for selected crystals. High pressure was in all cases promoting the gradual transformation of the crystals from $P2_1/a$ to $C2/m$ symmetry. We were able to quantify the changes induced by high pressure and high temperature statistically by analyzing the changes of intensities of reflections forbidden in the $C2/m$ symmetry.

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Keywords: high-pressure crystallography / experimental mineralogy

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POSTERS - PHYSICS

P-P02

Electric Field Stimulated Hadamard Time-resolved X-ray Crystallography

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Understanding the relationship between the function, dynamics, and structure of biomacromolecules requires techniques with high spatiotemporal resolution, such as time-resolved X-ray crystallographic methods. Most of these techniques rely on photoactivation or rapid mixing to initiate the change of interest, but these are challenging to achieve uniformly across all molecules in a sample. In contrast, electric field-stimulated X-ray crystallography (EF-X) applies an electric field pulse across a protein crystal, creating a precise pattern of forces localized to the charges natively present on each macromolecule. Laue X-ray diffraction data are recorded at a series of time-delays after the electric field initiation. There are two major limitations of the current implementation of EF-X. The first is the need for a Laue source as the X-ray probe, of which there are relatively few worldwide. The second is that the method is not high throughput, requiring time-consuming sample exchange. Thus, we will implement EF-X at a monochromatic macromolecular crystallography beamline and develop novel hardware to improve sample throughput, by multiplexing the experiment. To tackle this challenge, we will join forces between electric field-stimulated X-ray crystallography and Hadamard time-resolved X-ray crystallography (EF-HATRX). Hadamard time-resolved X-ray crystallography (HATRX) uses the Hadamard transform to encode time points within a sequence, decoupling time resolution from exposure time. The Hadamard transform increases the achievable time resolution, enabling the use of EF-X at monochromatic beamline to observe short chemical and biological reactions.

Keywords: time-resolved crystallography, electric field, Hadamard Transform

POSTERS - BIOLOGY

P-B01

Structural characterization of resistance profile of SARS-CoV-2 Main Protease against Nirmatrelvir and Ensitrelvir

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SARS-CoV-2 is the causative agent of COVID-19. The viral Main Protease (M^{pro}) is a key enzyme for viral cycle and one of the most promising targets for drug development. The recent emergency approved drug Paxlovid contains Nirmatrelvir, which is a M^{pro} inhibitor that had showed near 90% reduction in hospitalization during phase III clinical trials, in addition, the clinical candidate Ensitrelvir from Shionogi also demonstrate potential as antiviral drug. Despite its high efficiency against wild-type M^{pro}, very little is still known about how M^{pro} active site mutations could generate resistance to Nirmatrelvir and Ensitrelvir. Therefore, we here identified from genomic databases sixteen mutation that exist in the radius of action of Nirmatrelvir and Ensitrelvir and could affect its activity. M^{pro} mutants demonstrated to be active and able to recognize and cleave the fluorogenic substrate with similar catalytic activity, except for Q189K and G143S, which shown catalytic efficiency, respectively reduced by 33, 3-fold. Nirmatrelvir retained its inhibition in low nanomolar range against most polymorphism tested, while mutants G143S and Q189K exhibited higher resistence. For ensitrelvir, higher resistance was observed for polymorphisms M49I, G143S and R188S, but not for Q189K, meaning a possible distinct resistance profile between inhibitors. The crystal structures of M^{pro} polymorphism revealed the structural basis for resistance generation. In addition, these results can assist the monitoring of potential resistant strains, as well as guide the development of a next generation of M^{pro} inhibitors, including the development of combined therapy.

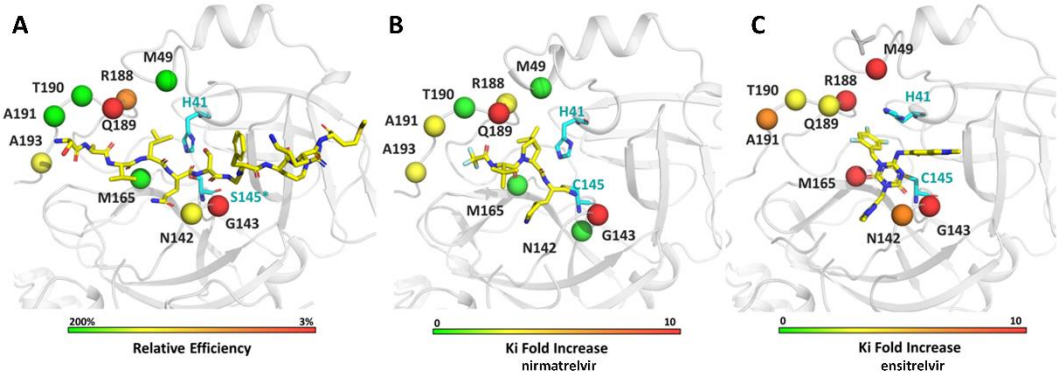

Figure 1: A) Structural representation of M^{pro} mutations using the substrate bound cryo-EM structure (PDBid 7S82) . B) Structural representation of M^{pro} mutations using the nirmatrelvir bound WT crystal structure (PDBid 8DZ2). C) Structural representation of M^{pro} mutations using the ensitrelvir bound WT crystal structure (PDBid 8DZ0) Residues where polymorphisms were identified are displayed as spheres and coloured according to M^{pro} (A) relative activity, (B) Nirmatrelvir affinity and (C) ensitrelvir affinity Active site residues are displayed as sticks, H41 and C145 are colored in cyan. Substrate peptide, Nirmatrelvir and ensitrelvir are represented as ball and stick and colored in yellow.

Keywords: Main Protease / Nirmatrelvir / Ensitrelvir

POSTERS - BIOLOGY

P-B02

Integrating X-ray Crystallography, CryoEM, and Biochemical Approaches to Unravel the role of CAK Complex in Transcription and DNA Repair.

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X-ray crystallography has revolutionised our understanding of the natural world and has been applied to various scientific disciplines. In biology, it has played a pivotal role in deciphering the structures of biomolecules, thus establishing the field of structural biology. By providing detailed structural information on proteins, nucleic acids, and other biological macromolecules, X-ray crystallography has offered invaluable insights into cellular processes. Recent advancements in synchrotron radiation technology and data processing software have significantly expanded the applications of X-ray crystallography in structural biology, such as understanding enzyme dynamics and facilitating structure-based drug design. Given the complexity of the crystallisation processes of some flexible proteins, cryo-electron microscopy (cryoEM) has become an essential tool that complements X-ray crystallography. The integration of both methods has been instrumental in understanding crucial cellular processes, including the central dogma of biology. A notable example of such proteins is RNA polymerase II, which plays a key role in transcription. At the preinitiation complex, it assembles with multiple transcription factors at the promoter DNA, one of which is TFIIH, which is also involved in DNA repair. TFIIH includes a kinase module known as Cyclin-Dependent Kinase Activating Kinase (CAK). CAK is responsible for crucial phosphorylation modifications of the C-Terminal Domain (CTD) of RNA pol II. The CTD has a tail composed of heptapeptide repeats with the consensus sequence Y1S2P3T4S5P6S7. These CTD repeats serve as a platform for multiple post-transcriptional modifications (PTMs) that regulate various steps of transcription. Our research aims to employ both X-ray crystallography and cryoEM to unravel the phosphorylation and recognition mechanisms of CAK towards CTD. In addition to structural methods, we will employ other biochemical and biophysical approaches to support the structural data. CAK exhibits high specificity towards serine 5 in CTD, and the mechanism by which CAK differentiates it from other residues in the heptapeptide repeats is not yet fully understood. A deeper understanding of this mechanism would shed light on the mechanisms of other CTD PTMs, particularly phosphorylation by transcriptional Cyclin-Dependent Kinases (CDKs). Furthermore, it will provide insights into the interaction of CAK with other substrates in the context of cell cycle regulation and support the design of CDKs inhibitors, which can be developed as anti-tumour agents

Keywords: integrative structural biology / preinitiation complex / RNA POL ctd

POSTERS - BIOLOGY

P-B03

High-resolution crystal structure of the tyrosine-coordinated Mn-protoporphyrin (IX) protein

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Artificial metalloenzymes (ArMs) hold great promise for enhancing enzymatic activities by incorporating non-native cofactors into protein scaffolds. Here, we present a novel enzyme from Oligotropha carboxydovorans, which is a homologue of the hexameric tyrosine-coordinated heme protein (HTHP) found in Silicibacter pomeroyi. While the HTHP-homologue shares striking structural similarity with HTHP, it exhibits a unique dodecameric assembly (dimer of hexamer) in both solution and crystal structures, as confirmed by analytical size exclusion chromatography and high-resolution crystallography respectively. Thus, it is named dodecameric tyrosine-coordinated heme protein (DTHP). We present the crystal structures of DTHP complexed with manganese-substituted non-native heme (Mn-protoporphyrin IX) at an unprecedented high resolution of 0.94 Å.

The DTHP enzyme displays a barrel-shaped architecture with exceptional ring-like C6 symmetry, much like its hexameric counterpart. Each monomer within the dodecamer contains three α helices forming a binding pocket that accommodates a solvent-exposed Mn-PPIX cofactor. The central Mn ion of the porphyrin ring is coordinated with the phenolic oxygen atom of Tyrosine 46 from the proximal side. The dimer of hexamer is formed by a combination of hydrophobic and hydrogen bonding interactions.

Our study highlights the potential of metal substitution in cofactors as a strategy to engineer ArMs. The elucidation of the Mn-PPIX-bound DTHP structure provides valuable insights into the precise binding interactions and structural adaptations within the protein scaffold. This work not only expands our understanding of heme-binding enzymes but also offers new opportunities for rational design and optimization of ArMs with enhanced catalytic activities.

Keywords: Artificial Metalloenzyme (ArM) / Protein engineering / Heme cofactor

POSTERS - BIOLOGY

P-B04

Microfluidic chips for biomolecular crystal growth and serial crystallography

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Twenty years ago, microfluidics opened up new possibilities and brought many benefits for the crystallization of biomolecules. Indeed, microfluidic systems facilitate the manipulation of nano-volumes of sample solutions, as well as extreme miniaturization and parallelization of crystallization assays. In addition, they provide a convection-free environment that favors the growth of high-quality crystals [1].

As an illustration, a new multifunctional microchip will be presented that combines 1) the search and optimization of crystallization conditions of biomolecules by the counter diffusion method, 2) crystal identification by fluorescence microscopy, 3) microcrystalline seeding, 4) derivatization of crystals by substrate soaking, and 5) routine in situ crystal analysis at room temperature. The concept was already tested on a large panel of biomolecules including RNA and various soluble or membrane proteins [2-3]. A new chip design and our latest results in microcrystallization and in situ serial synchrotron crystallography at room temperature (RT-SSX) will be presented.

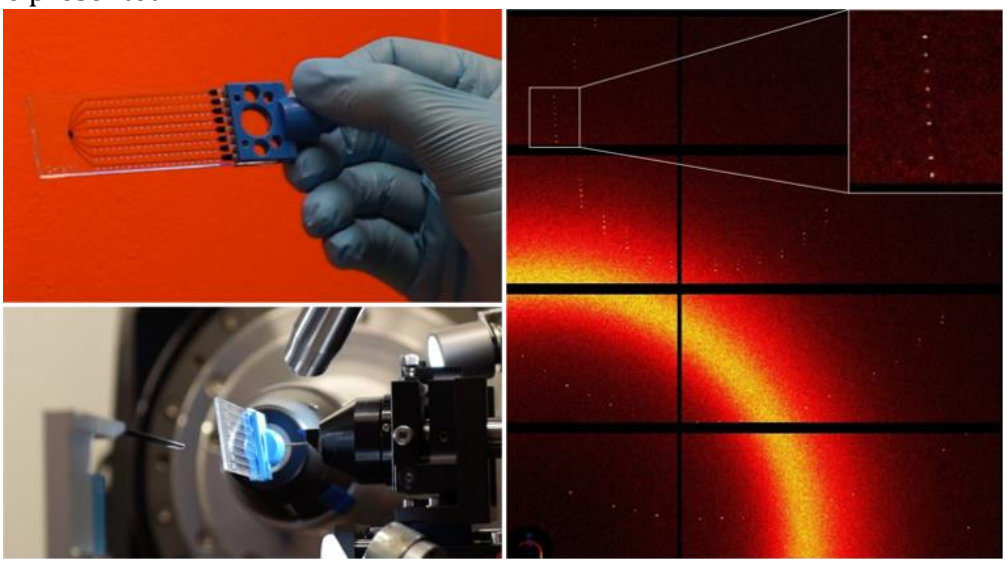

Figure 1: On-chip biomolecular crystal analysis by RT serial synchrotron crystallography

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Keywords: serial crystallography / microfluidics / microcrystals / seeding / ligand soaking

POSTERS - BIOLOGY

P-B05

Biocrystallography as a means of communication with the general public on the coronavirus and the RNA world

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For more than 60 years, biocrystallography has been revealing the secrets of biomolecules. These structural data are a valuable source of information for scientists, but also a great medium for wonder and exchange with the general public. After two years of pandemic, the ARN laboratory – ARN for architecture and reactivity of RNA – set itself a challenge: to use the data accumulated on the SARS-CoV2 coronavirus and its RNA genome to communicate on the burning news of RNA viruses on the occasion of the French science festival, a national event organized every year in October [1]. Various communication tools were deployed to answer questions of the greatest number of people. First of all, four posters were displayed at the back of the booth to illustrate 1) the central role of mRNAs in gene expression and protein synthesis, 2) the diversity of roles played by RNAs in the cell, 3) the variety of RNA viruses studied in the laboratory, and 4) the way in which 3D representations of these macromolecules are obtained. An experiment of fast protein crystallization allowed visitors to get their hands on it, while marveling at the appearance of multicolored crystals and discovering the first step of every crystallographic study. The exploration of protein and RNA architecture continued on a 3D screen, thanks to a series of scripted scenes in the PyMOL molecular visualization software [2]. Visitors could also reconstruct the Watson-Crick basepairs present in RNA thanks to the BasePairPuzzle designed by Prof. Jiro Kondo from Sofia University in Tokyo [3]. Finally, under the threatening gaze of a 3D printed model of the coronavirus [4], visitors were invited to enter the level 3 laboratory where public enemy number 1 is studied, and to immerse themselves in the biology of the beast by putting on a virtual reality helmet. We constituted a highly motivated team of PhD students, postdocs, technicians, engineers and researchers who animated in turn the booth during 3 days and exercised their communication skills. We will share the challenging and exciting experience of exchanges with the general public on sometimes complex subjects and educational materials implemented during the science festival weekend.

Figure 1: Exploring the world of RNA and coronavirus at the French Science Festival (Fête de la Science)

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[3] https://jkondo.wixsite.com/basepairpuzzle
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Keywords: biocrystallography / coronavirus / RNA / science outreach

POSTERS - BIOLOGY

P-B06
Protein dynamic events probed by time-resolved crystallography on the second to hour time scale

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Over the last decade, the rise of serial crystallography (SX) at X-ray Free Electron sources (XFEL) has rejuvenated time-resolved macromolecular crystallography (TRMX) and led to technical and methodological breakthroughs[1]. TRMX at XFELs usually addresses phenomena occurring on the picosecond to millisecond time scale, but many protein dynamic events take place on slower time scales[2]. We have applied RTMX to study the relaxation of a photostationary equilibrium built within a crystal of the LOV2 domain of phototropin 2 from *Arabidopsis thaliana*, whose photoadduct decays on the 10-min time scale[3]. We have monitored its relaxation in the dark by recording 1 s full oscillation data sets, as a follow-up to our study on the population build-up of the same photoadduct[4]. We could observe bond breakage in the photoadduct using both *in crystallo* UV-vis absorption spectroscopy and X-ray crystallography with similar decay time constants. Surprisingly, the return to the ground state of the chromophore is followed by additional large-scale protein rearrangements, which end up inducing a phase transition in the crystal. Our work demonstrates the possibility of performing time-resolved protein crystallography experiments from a small number of crystals using standard beamline equipment at synchrotrons for phenomena occurring on the second to the tens of minutes time scale.

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POSTERS - BIOLOGY

P-B07

Structural and functional characterization of lentiviral Gag polyproteins

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Human Immunodeficiency Virus and Feline Immunodeficiency Virus are etiological agents of immunodeficiency syndromes in human beings and in feline species. The virions are composed by the assembly of the three main retroviral proteins Gag, Pol and Env. The Gag proteins assemble around the dimer of genomic RNAs and form a spherical complex. After budding, the viral protease releases the MA, CA and NC subunits which reassemble respectively to form the matrix beneath the membrane, the capsid and the nucleocapsid binding the genome. For HIV-1, the structures of all subunits have been resolved separately, by crystallography for MA and CA, NMR for MA and NC and for FIV the structure of the MA subunit was, and a structure of the monomeric CA was determined as well but not assembled as a hexamer. Here, we focus on the understanding of the HIV and FIV assembly. The Gag assembly is mostly driven by the CA:CA subunit interactions to form hexamers in the immature form before the proteolytic cleavage. Gag proteins accumulate around the dimer of RNA through their interactions with the NC subunit and accumulate to the membrane through the MA subunit having an affinity for the membrane, with its basic region and its myristoyl anchor. Here, we studied the assembly of FIV CA subunit and the identification of an inhibitor of it using experimental data and modelling from a partial crystalline structure. We also study the assembly of the full-length Gag. The HIV-1 Gag proteins assemble around RNA and we show the obtention of Virus-Like-Particles by assembling proteins around fragments of the genomic RNAs. We are currently expressing and purifying the FIV Gag expressed from mammal cells so that they contain the myristoyl anchor which contributes to the folding of the Gag protein. Modelling of the Gag protein structure can be performed by combining all the partial structures of the protein obtained to help understand the assembly but experimental data remain required to have a clear understanding of the process. All together, formations of VLP particles, modelling and identification of ligands of the capsid could lead to the development of new drugs available to target lentiviral infections, inhibiting assembly, maturation or the binding of the viral genome to the NC subunit.

Keywords: retrovirus / modeling

POSTERS - BIOLOGY

P-B08

Structural Studies of Cancer-related Drug Target Carbonic Anhydrase IX with Sulfonamide-based Inhibitor

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During the tumor development, cancer cells face various forms of stress, such as low pH environment resulting from hypoxia-induced glycolysis metabolism. To manage this difficulty and maintain their intracellular pH, various types of carcinoma cells overexpress Carbonic Anhydrase IX (CA IX) on their cytoplasmic membrane. This enzyme belongs to a family of human α-CA metalloenzymes family which facilitate the reversible hydration CO_2 to HCO_3- and H+. All twelve enzymatically active isoenzymes share high sequence identity and a typical β-sheet structural fold of catalytic domain with Zn^{2+} ion in the active-site cavity.
In our research we focus on a unique characteristic of CA IX, which is the presence of N-terminal proteoglycan-like domain (PG). There is currently no structural information about the whole extracellular part of CA IX consisting of both PG and catalytic domain and our aim is to fill this knowledge gap.
We have successfully expressed recombinant CA IX comprising of both the catalytic and PG domains through heterologous expression in Escherichia coli. Various recombinant protein constructs include specific mutations that enhance yield and solubility, therefore enabling the production of sufficient quantities of high-quality protein for X-ray crystallography experiments. In parallel isotopically labeled CA IX was produced for NMR experiments. The integration of these two complementary approaches of structural biology will allow comprehensive investigations of CA IX, particularly concerning the structure and function of the PG domain. Furthermore, the insights gained from these studies will also aid in the discovery of specific inhibitors targeting this isoform.

Keywords: structural biology, drug design, cancer

POSTERS - OTHERS

P-O01

My life as an analytical academic scientist in crystallography

Jérémy Forté

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My name is Jérémy and I'm a crystallographer. I work for a french research lab and I love my job! I enjoy working with academic researchers and would like to present you the different aspects of my daily life.
Here goes!

Keywords: French / crystallographer / job / work

"WORK WITH US" SESSION

Participants:

Laura Folkers from **STOE**

Loïc Mazé from **Malvern Panalytical**

Laurent Loos from **Rigaku**

Loïc Le Dréau from **Bruker**

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