Colloque de l'AFC

Association Française de Cristallographie

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Livret de résumés du colloque de l'Association Française de Cristallographie
30 juin-3 juillet 2026, Lille

Rédaction : Lydia Karmazin (IMEC - Institut Michel Eugène Chevreul, Lille) et Pascal Roussel (UCCS – Unité de Catalyse et Chimie du Solide, Lille)

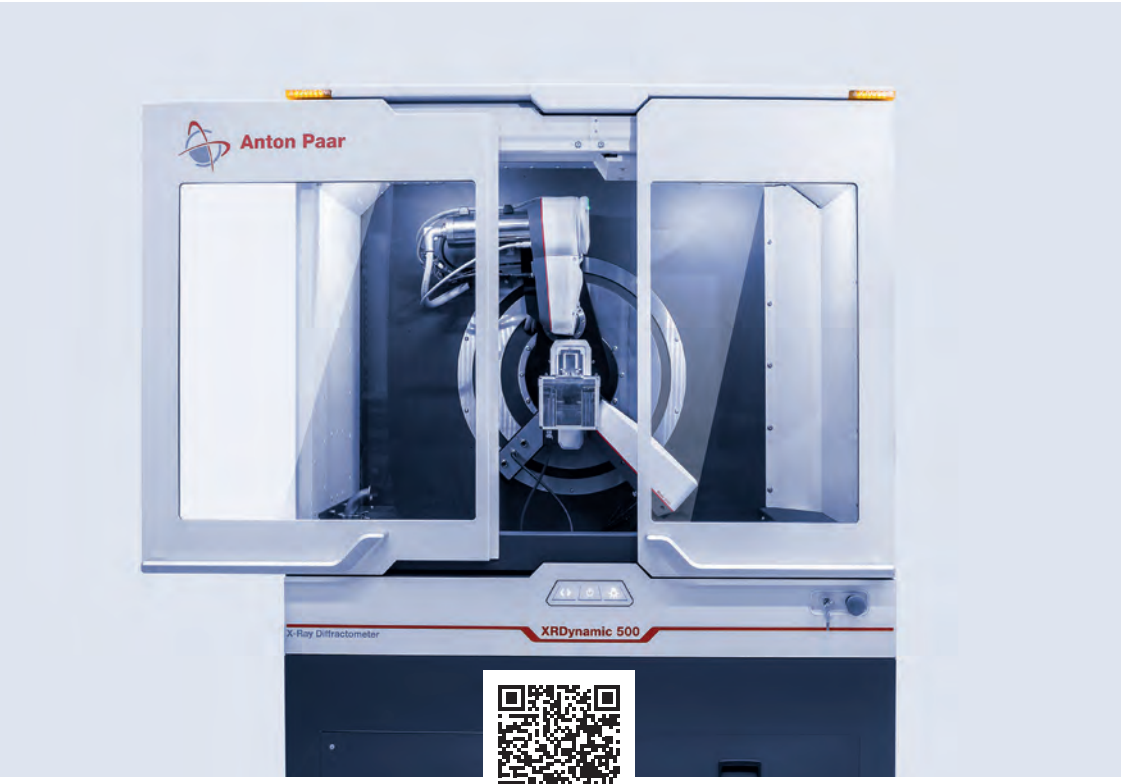
Assemblage : VertCom

Impression : Université de Lille

Couverture : Pascal Roussel, Lydia Karmazin et Frédéric Capet

Juin 2026

Anton Paar



La DRX à un niveau supérieur

XRDynamic 500

Diffractomètre à rayons X polyvalent pour poudres

Système optique et alignement entièrement automatisés

Trajet du faisceau sous vide, grand rayon

Idéal pour la DRX et le SAXS en conditions non ambiantes

Mot de bienvenue

Bienvenue à toutes et à tous au Lilliad Learning Center Innovation de Villeneuve d'Ascq pour l'édition 2026 du colloque de l'Association Française de Cristallographie (AFC2026). La communauté des cristallographes lillois est ravie de vous accueillir pour cet événement important pour la cristallographie locale, réunissant les différentes disciplines (physique, chimie, biologie) au cœur de notre association.

Nous tenons à exprimer notre profonde gratitude au Conseil d'Administration de l'Association Française de Cristallographie pour nous avoir accordé sa confiance pour organiser cet événement majeur pour la communauté cristallographique française. Nous adressons également nos vifs remerciements aux membres du comité scientifique et aux animateurs de session pour leur contribution à l'élaboration du programme, ainsi qu'aux étudiants bénévoles pour leur aide logistique indispensable. Enfin, ce colloque n'aurait pu voir le jour sans le soutien financier des institutions, des collectivités et de nos partenaires privés.

AFC 2026 vous propose 14 sessions couvrant divers thèmes issus des sciences structurales en biologie, chimie, physique ainsi que des sujets transversaux comme la croissance cristalline, l'enseignement, la médiation scientifique et l'utilisation de l'intelligence artificielle au service de notre science. Nous aurons le plaisir d'accueillir nos trois conférenciers « thématiques » en session plénière : Laurence Croguennec pour la chimie (ICMCB, Bordeaux), Sylvain Ravy pour la physique (LPS, Orsay), Herman van Tilbeurgh pour la biologie (I2BC, Paris-Saclay), et dans le cadre de la session « l'AFC invite », Simon Coles (NCS, Southampton) et Gideon Davies (YSBL, York), de l'association britannique de cristallographie (BCA). Nous aurons également le plaisir d'accueillir le Professeur Patrick Cordier qui donnera une conférence grand public au Palais des Beaux-Arts de Lille ayant pour titre « Quand les cristaux se désorganisent : de l'amorphisation des minéraux aux matériaux du futur ». Un autre moment fort de cette édition 2026 sera la remise du troisième prix André Guinier à Marianne Quiquandon et Denis Gratias, de l'Institut de Recherche de Chimie Paris (IRCP) / Chimie ParisTech. L'AFC décernera également ses prix de thèse le jeudi 2 juillet. N'oublions pas la journée satellite la veille du congrès organisée par le réseau ЯÉCIPROCS, sur le thème « Méthodes avancées d'analyse des données à travers la Cambridge Structural Database ».

Profitez pleinement de ce colloque pour échanger avec vos pairs, découvrir la fameuse « Grand-Place », le beffroi de l'hôtel de ville ou flâner sur les pavés des ruelles du Vieux Lille !

En attendant, nous vous souhaitons à toutes et à tous un excellent colloque.

Pour les organisateurs de l'AFC2026 :

Lydia Karmazin et Pascal Roussel

L'Association Française de Cristallographie (AFC) est une société savante qui rassemble les physiciens, chimistes et biologistes qui utilisent les cristaux et la cristallographie pour leurs recherches ou développent des méthodes en cristallographie. (www.afc.asso.fr)

Comités

Comité Scientifique

GT-Biologie:

- Xavier HANOULLE – BSI, Villeneuve d'Ascq
- Dominique HOUSSET – IBS, Grenoble
- Solange MORERA – I2BC, Gif-sur-Yvette
- Claude SAUTER – IBMC, Strasbourg

GT-Chimie:

- Michel GIORGI – FSCM, Marseille
- Olivier HERNANDEZ – IMN, Nantes
- Lydia KARMAZIN – IMEC, Villeneuve d'Ascq
- Carmelo PRESTIPINO – CRISMAT, Caen

GT-Physique:

- Delphine CABARET – IMPMC, Paris
- Natália T. CORREIA – UMET, Villeneuve d'Ascq
- Sébastien PILLET – CRM2, Nancy
- Sandrine DOURDAIN – ICSM, Marcoule

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- Marie COLMONT - Unité de Catalyse et Chimie du Solide (UCCS)
- Natália T. CORREIA - Unité Matériaux et Transformations (UMET)
- Xavier HANOULLE - Biologie Structurale Intégrative (BSI)
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- Alexandre MUSSI - Unité Matériaux et Transformations (UMET)
- Pascal ROUSSEL - Institut Michel-Eugène Chevreul (IMEC) et Unité de Catalyse et Chimie du Solide (UCCS)

Liste des sessions et modérateurs/modératrices

1. Cristallogenèse et Croissance cristalline

- Gabin Gbabode SMS – Sciences et Méthodes Séparatives (Rouen)
- Fátima Mota IM2NP – Institut Matériaux Microélectronique Nanosciences de Provence (Marseillle)

2. Science des matériaux, texture, microstructure et contraintes résiduelles

- Daniel Chateigner CRISMAT – Cristallographie et Sciences des Matériaux (Caen)
- Marie-Vanessa Coulet MADIREL – Matériaux Divisés, Interfaces, Réactivité, Électrochimie (Marseille)

3. Matériaux pour l'énergie : de la production au stockage

- Nathalie Audebrand ISCR – Institut des Sciences Chimiques (Rennes)
- Pascal Roussel UCCS – Unité de Catalyse et Chimie du Solide (Lille)

4. Chimie moléculaire et supramoléculaire, interactions et reconnaissance moléculaire

- Olivier Jeannin ISCR – Institut des Sciences Chimiques (Rennes)
- Laure Vendier LCC – Laboratoire de Chimie de Coordination (Toulouse)

5. Une nouvelle ère pour la cristallographie : intelligence artificielle et cristallographie quantique

- Enrique Espinosa CRM2 – Cristallographie, Résonance Magnétique et Modélisations (Nancy)
- Arie van der Lee ICGM – Institut Charles Gerhardt (Montpellier)

6. Multisources, multitechniques, conditions atypiques, *in situ*

- Brice Kauffmann IECB – Institut Européen de Chimie et Biologie (Bordeaux)
- Magali Putero IM2NP – Institut Matériaux Microélectronique Nanosciences de Provence (Marseille)

7. Diffraction et imagerie 3D à l'échelle nanométrique

- Philippe Boullay CRISMAT – Cristallographie et Sciences des Matériaux (Caen)
- Stéphanie Kodjikian – Institut Néel (Grenoble)

8. Biologie et chimie pour la Santé, Drug design

- Sonia Fieulaine I2BC – Institut de Biologie Intégrative de la Cellule (Gif sur Yvette)
- Jean-Denis Pedelacq IPBS – Institut de Pharmacologie et de Biologie Structurale (Toulouse)

9. Biologie Structurale Intégrative

- Coralie Bompard UGSF – Unité de Glycobiologie Structurale et Fonctionnelle (Lille)
- Pierre Legrand – Synchrotron SOLEIL (Saint-Aubin)

10. Enseignement et médiation scientifique

- Delphine Cabaret IMPMC – Institut de minéralogie, de physique des matériaux et de cosmochimie (Paris)
- Mathieu Marchivie ICMCB – Institut de Chimie de la Matière Condensée (Bordeaux)

11. Structures résolues en temps

- Elke De Zitter IBS – Institut de Biologie Structurale (Grenoble)

12. SAXS, WAXS et structure à l'échelle nano

- Elise Guerinoni ICSM – Institut de Chimie Séparative de Marcoule (Marcoule)
- Grégory Stoclet UMET – Unité Matériaux et Transformations (Lille)

13. Patrimoine culturel et naturel, Sciences de la terre

- Jean-Paul Itié – Synchrotron SOLEIL (Saint-Aubin)
- Pauline Martinetto – Institut Néel (Grenoble)

14. Désordre, apériodicité, structure locale

- El-Eulmi Bendeif CRM2 – Cristallographie, Résonance Magnétique et Modélisations (Nancy)
- Olivier Perez CRISMAT – Cristallographie et Sciences des Matériaux (Caen)

Le Prix Guinier

Le prix « André Guinier » de l'Association Française de Cristallographie récompense une personnalité francophone, ou ayant effectué une grande partie de sa carrière en France, qui a contribué de façon exceptionnelle à la cristallographie. Le prix 2026 sera remis à Marianne Quiquandon et Denis Gratias, de l'Institut de Recherche de Chimie Paris (IRCP) / Chimie ParisTech.

Marianne Quiquandon entame son parcours scientifique par une maîtrise en physique fondamentale à l'université Paris VI, avant de rejoindre en 1982 le CECM de Vitry/Seine pour y préparer sa thèse sur la théorie dynamique de la diffraction des électrons rapides par les cristaux et quasicristaux. Elle y développe le formalisme proposé par Denis Gratias et Richard Portier en 1981, inspiré des théories de perturbations dépendantes du temps en mécanique quantique. En 1984, alors que Denis Gratias séjournait à l'Institut de Physique Théorique de l'Université de Californie à Santa Barbara, il est confronté, dans le cadre d'une collaboration interdisciplinaire organisée par John Cahn, au problème des diffractions d'ordre 5 observées en 1982 par Danny Shechtman. Avec l'aide à distance d'Ilan Blech, les trois chercheurs publient en octobre 1984 un article annonçant la découverte des quasicristaux, une avancée qui vaudra à Shechtman le prix Nobel de chimie en 2011. À partir de 1985, une collaboration fructueuse s'installe entre Denis Gratias, Marianne Quiquandon, John Cahn et André Katz, marquant le début de contributions majeures en cristallographie.

Parmi leurs réalisations, on compte les premières simulations numériques d'images de microscopie électronique à haute résolution de quasicristaux icosaédriques, menées directement dans un cadre quasipériodique, sans recourir à des approximations par des cristaux à grandes mailles. Ce travail constitue le cœur de la thèse de Marianne Quiquandon, soutenue en 1988 sous la présidence d'André Guinier. Par ailleurs, le schéma d'indexation des diffractions des phases icosaédriques, développé à partir de 1986 et étendu par Quiquandon en 1999, permet de décrire ces structures comme des cristaux à six dimensions, dont le quasicristal réel est une coupe irrationnelle en 3D. Cette approche a ouvert la voie à des reconstructions de Fourier en 6D et à l'exploration de l'espace perpendiculaire.

En 1991, l'équipe réalise la première détermination expérimentale d'une phase icosaédrique ($Al_{62}Cu_{25.5}Fe_{12.5}$) grâce à un modèle de surfaces atomiques polyédrales. Cette avancée repose sur l'expertise métallurgique du CECM et sur deux éléments clés : le schéma d'indexation, qui révèle que les réflexions se divisent en quatre branches distinctes dans l'espace perpendiculaire, et les travaux d'André Katz sur les surfaces atomiques polyédrales. Le modèle proposé, sans paramètres ajustables, décrit avec précision plus de 95 % des atomes des structures actuelles. Par ailleurs, le lien géométrique entre les quasicristaux et leurs phases approximantes est établi en considérant ces dernières comme le résultat d'un cisaillement linéaire dans l'espace perpendiculaire, transformant une structure quasipériodique en une structure périodique.

En 2000, Denis Gratias et Marianne Quiquandon rejoignent le Laboratoire d'Étude des Microstructures (LEM) à Châtillon, où Gratias dirige l'unité mixte CNRS-ONERA jusqu'en 2009. Ils y

mènent une étude approfondie des environnements locaux atomiques dans les phases icosaédriques de type F, aboutissant à la proposition d'un modèle 6D générique pour ces phases. À partir de 2014, après le passage de Denis Gratias à l'éméritat, ils intègrent l'IRCP/Chimie ParisTech. Leurs recherches se concentrent alors sur les symétries N-dimensionnelles et les défauts de module dans certains alliages, généralisant les descriptions classiques des macles dans les cristaux. Plus récemment, dans le cadre d'un projet ANR, ils développent le cadre formel de la bi-cristallographie, étudiant les symétries globales générées par deux copies d'un même objet dans l'espace. Leurs applications aux bicouches 2D, comme celles du graphène, et aux figures de moiré, relient pavages géométriques et fonctions analytiques continues. Leur travail actuel explore une cristallographie « douce », où les notions strictes de périodicité et de symétrie cèdent la place à des concepts plus souples, comme la presque-périodicité et la presque-symétrie, au sens de Harald Bohr.

Marianne Quiquandon et Denis Gratias Crédit photo: @nolwenn.buvat

Les conférenciers pléniers

Laurence Croguennec est Directrice de Recherche CNRS à l'Institut de Chimie de la Matière Condensée (ICMCB- CNRS, France) de l'Université de Bordeaux. Elle a obtenu son doctorat en 1996 à l'Université de Nantes, à l'Institut des Matériaux Jean Rouxel, et a passé un an comme chercheuse postdoctorale à l'Université de Bonn (Allemagne). Elle est devenue chercheuse CNRS à l'ICMCB en 1997, a dirigé le groupe de recherche « Énergie : matériaux et batteries » entre 2004 et 2021, et est directrice adjointe de l'ICMCB depuis 2022. Elle participe également activement au Réseau français sur le stockage électrochimique de l'énergie (RS2E), ainsi qu'à l'Institut Européen de Recherche ALISTORE dédié à la recherche sur les batteries, et au programme d'accélération France 2030 avec les batteries PEPR. Laurence Croguennec travaille depuis plus de 25 ans sur la chimie cristalline des matériaux d'électrodes développés pour les batteries à ions métalliques, et plus récemment pour les batteries tout-solides, ainsi que sur la caractérisation des mécanismes impliqués dans leur cyclage, en particulier pour les oxydes stratifiés et spinelles et les matériaux d'électrodes positives de type polyanionique. Elle mène également des recherches en collaboration avec de grandes installations européennes de neutrons et de synchrotrons pour la caractérisation in situ et operando des matériaux pendant le fonctionnement des batteries.

Sylvain Ravy est directeur de recherche au CNRS. Il a soutenu sa thèse en 1988 au Laboratoire de Physique des Solides, CNRS/Université Paris-Saclay ; celle-ci portait sur des études de systèmes moléculaires désordonnés par diffusion diffuse des rayons X. Il est ensuite rentré dans ce même laboratoire Chargé de Recherche au CNRS. De 1995 à 1997 il a été visiteur à l'Université de Wisconsin-Milwaukee et utilisateur du synchrotron ALS de Berkeley, pour des études de physique des surfaces. De retour en France, il s'est impliqué dans le projet de synchrotron SOLEIL, où il a été détaché 11 ans, de 2004 à 2014, pour y être responsable de la ligne de diffraction CRISTAL. En 2015, il a été nommé directeur du Laboratoire de Physique des Solides, et depuis 2021 il occupe la fonction de Directeur Adjoint Scientifique à CNRS Physique, en charge des infrastructures de recherche.

Herman van-Tilbeurg a obtenu une maîtrise en chimie à l'université de Gand (1976-1980), puis un doctorat en biochimie dans la même institution (1981-1986), en se concentrant sur les mécanismes enzymatiques. En 1994, il obtient l'habilitation à diriger des recherches de l'université d'Aix-Marseille II, ce qui lui permet d'accéder au poste de professeur titulaire et de superviser des doctorats de manière indépendante en France. Sa carrière professionnelle s'étend à l'industrie, aux organismes de recherche nationaux et à des postes de direction dans l'enseignement supérieur. Il commence sa carrière en tant que chercheur scientifique

chez Plant Genetic Systems (Gand, Belgique, 1986-1991), où il utilise la cristallographie des protéines pour améliorer les enzymes industrielles. Il rejoint ensuite le CNRS en tant que post-doctorant à Marseille (1991-1993), puis il est nommé Chargé de Recherche (CR1) au Centre de Biochimie Structurale à Montpellier (1993-1997). En 1997, il est nommé professeur de biologie structurale à l'université d'Aix-Marseille I, puis en 2001, professeur de biochimie structurale à l'université Paris-Saclay, où il continue à diriger des activités de recherche et d'enseignement. Il dirige l'équipe de recherche FAAM (Fonction et Architecture des Assemblages Macromoléculaires) à l'Institut de Biologie Intégrative de la Cellule, un institut commun au CNRS, au CEA et à l'Université Paris-Saclay.

Simon Coles a obtenu sa licence et son doctorat en systématique structurale et modélisation moléculaire à l'Université du Pays de Galles, à Cardiff, avant d'être nommé chercheur postdoctoral à la Royal Institution pour construire la première ligne de faisceau au monde dédiée aux monocristaux de petites molécules, au synchrotron de Daresbury. En 1998, Simon s'installe à Southampton pour créer un nouveau laboratoire et gérer le Service National de Cristallographie. Simon devient Directeur du Service National de Cristallographie (www.ncs.ac.uk) en 2009, Directeur du Service britannique de Science des Données en Sciences Physiques en 2019, et est aujourd'hui responsable de l'Infrastructure des Données en Sciences Physiques. Simon est l'auteur de plus de 1 000 articles appuyant la synthèse chimique, dans de nombreux domaines de la chimie structurale et dans l'information numérique/chimique. Il est l'un des cristallographes chimistes les plus prolifiques au monde, se classant au 20e rang des contributeurs les plus importants de tous les temps à la Cambridge Structural Database.

Gideon Davies, membre de la Royal Society, dirige un groupe de recherche au Département de Chimie de l'Université de York, Royaume-Uni. Après avoir obtenu son doctorat à Bristol, au Royaume-Uni, il a travaillé à l'EMBL Hamburg Outstation et à Grenoble, avec des séjours à Uppsala et à Vancouver. Il est surtout connu pour ses travaux sur les enzymes actives sur les glucides, leurs structures 3D et la dissection structurelle de leurs mécanismes réactionnels. Il a été le pionnier de l'analyse des chemins conformationnels de réaction (en utilisant la cristallographie pour montrer comment les enzymes déforment leurs substrats de sucres) et applique désormais ses connaissances mécanistiques et structurelles au développement de nouveaux inhibiteurs et sondes enzymatiques.

La conférence grand public

Quand les cristaux se désorganisent :
de l'amorphisation des minéraux aux matériaux du futur

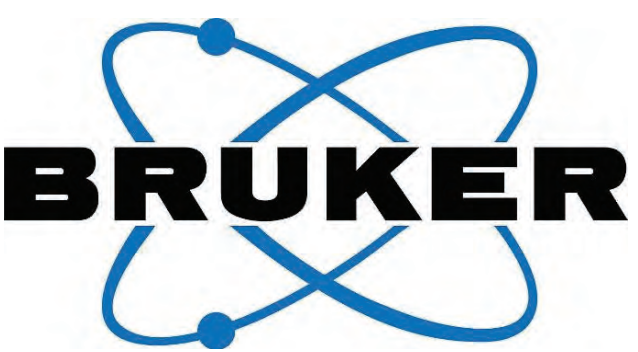
Par **Patrick Cordier**
Professeur à l'Université de Lille
et membre de l'Institut Universitaire de France

Les cristaux fascinent par leur ordre parfait et leurs symétries. Pourtant, certains minéraux peuvent perdre cet ordre et devenir amorphes sans fondre. Cette conférence propose une exploration des processus d'amorphisation à l'état solide, lorsque la structure d'un cristal se désorganise sous l'effet de pressions extrêmes, d'ondes de choc, du rayonnement ou de réactions chimiques. Nous verrons comment la nature produit des verres d'impact, des minéraux métamictes ou des phases amorphisées dans les zones de faille, comment ces transformations sont reproduites en laboratoire, et pourquoi la maîtrise de l'amorphisation ouvre aujourd'hui de nouvelles perspectives, depuis la compréhension des processus astrophysiques et géologiques jusqu'au développement de technologies fondées sur la commutation entre états cristallin et amorphe.

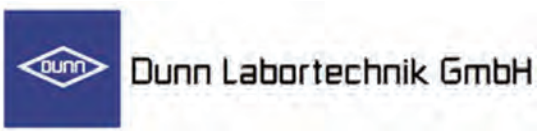
Patrick Cordier est professeur de physique à l'Université de Lille et membre de l'Institut Universitaire de France. Après une formation initiale d'ingénieur en science des matériaux, il a obtenu son doctorat à l'Université de Lille sur l'influence de l'eau sur la plasticité du quartz. Il s'intéresse particulièrement aux mécanismes de déformation des minéraux et des roches des intérieurs planétaires. Ses approches de prédilection sont la microscopie électronique à transmission, la modélisation numérique multi-échelle et diverses techniques expérimentales impliquant des hautes pressions ou des essais nanomécaniques. Il est Fellow de la Mineralogical Society of America et de l'American Geophysical Union. Il a reçu la médaille Dana de la Mineralogical Society of America.

Les sponsors

Partenariat en diamant



Partenariat en or



Partenariat en argent

Les partenaires institutionnels

Lieu du Colloque

L'édition 2026 de notre colloque aura lieu au pôle événementiel de LILLIAD Learning center Innovation, espace dédié à l'accueil de congrès, implanté sur le campus Cité scientifique de l'Université de Lille. L'exposition constructeur se tiendra dans le Hall évènementiel, les posters seront affichés dans les salles C et D, et les présentations auront lieu dans les amphithéâtres indiqués sur le programme. Enfin, le salon P.I accueillera les activités liées à la médiation scientifique.

Adresse Postale : LILLIAD Learning center Innovation
Université de Lille, campus Cité scientifique
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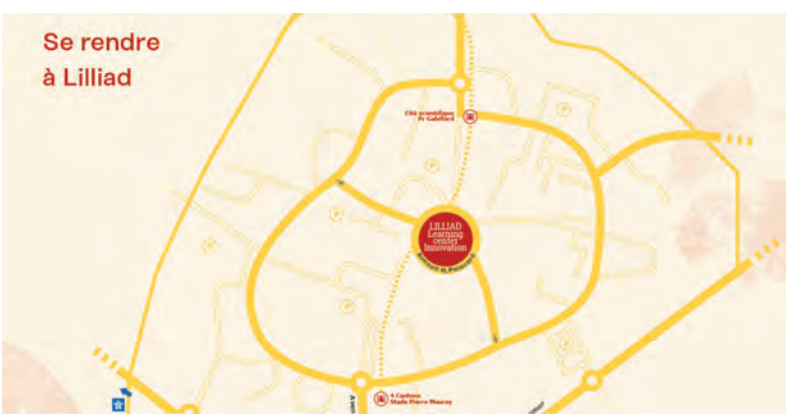


En métro, prendre la ligne 1, direction 4 Cantons-Stade P. Mauroy et descendre à la station Cité Scientifique-Pr Gabillard. Marcher environ 300m et vous êtes arrivés !

Lieu de la soirée de gala

Le diner de gala aura lieu jeudi 2 juillet à 20h, au restaurant de l'hôtel Alliance « Couvent des Minimes »,

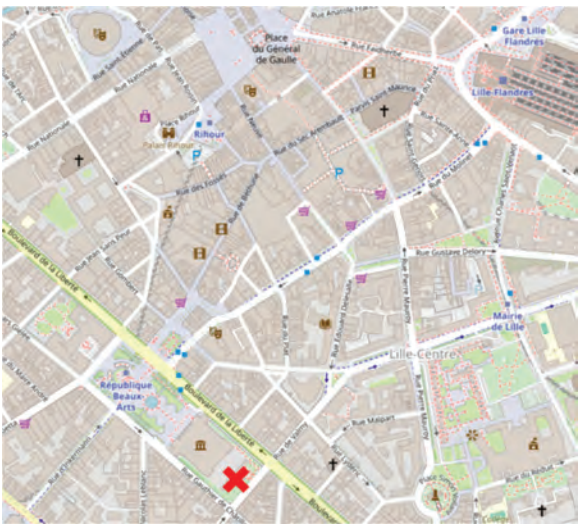
situé 17 Quai du Wault – 59800 Lille.

« L'Hôtel Couvent des Minimes et son restaurant Le Jardin du Cloître offrent un cadre d'exception : un couvent du XVIIe siècle classé monument historique, empreint de charme et d'élégance. Installé sous une majestueuse verrière, au cœur du patio du couvent, le restaurant Le Jardin du Cloître vous invite à vivre un moment unique dans une atmosphère intime et apaisante. »

En Métro, prendre la ligne 1, direction CHU Eurasanté, et descendre à la station Rihour. Marcher environ 10 minutes et vous êtes arrivés !

Lieu de la conférence grand public

La conférence grand public aura lieu mercredi 1er juillet à 19h, au Palais des Beaux-Arts de Lille. L'entrée, à cette heure-ci, se fera au 18 bis rue de Valmy, à l'arrière du bâtiment. En raison des consignes Vigipirate, il est conseillé d'arriver un quart d'heure plus tôt



En métro : ligne 1, station République Beaux-Arts

Programme – vue d'ensemble

	Mardi 30/06	
8h00	Accueil	
8h30	Mot de bienvenue	
9h00	Conférence plénière Biologie	
10h00	Pause-café	
10h30	MS13 (Amphi A) Patrimoine culturel et naturel, Sciences de la terre	MS11 (Amphi B) Structures résolues en temps
12h30	Pause déjeuner	
14h00	L'AFC invite la British Crystallographic Association	
15h30	Session Exposants	
16h30	Session Posters - Pause café	
17h30	Prix Guinier	
18h30	Soirée de bienvenue Ch'ti	

	Mercredi 1er/07	
8h30	Conférence plénière Physique	
9h30	Pause-café	
10h00	MS3 (Amphi A) Matériaux pour l'énergie : de la production au stockage	MS9 (Amphi B) Biologie Structurale Intégrative
12h00	Assemblée Générale AFC	
13h00	Pause déjeuner Photo de Groupe	
14h30	MS10 Enseignement et médiation scientifique	
16h00	Session Posters - Pause café MS10	
18h00	Enseignement et médiation scientifique (suite)	
19h00	Conférence grand public	

Programme – vue d'ensemble

	Jeudi 2/07	
8h30	Conférence plénière Chimie	
9h30	Pause-café	
10h00	MS8 (Amphi A) Biologie et chimie pour la santé, Drug design	MS14 (Amphi B) Désordre, apériodicité, structure locale
12h00	Pause déjeuner	
13h00	Prix de thèse	
14h00	MS7 Diffraction et imagerie 3D à l'échelle nanométrique	MS4 Chimie moléculaire et supramoléculaire, Intéractions et reconnaissance moléculaire
16h00	Session Posters - Pause café	
17h00	MS2 Science des matériaux, texture, microstructure et contraintes résiduelles	MS12 SAXS, WAXS et structure à l'échelle nano
19h00		
20h00	Dîner de gala *Lieu : Hôtel Couvent des Minimes, 17 Quai du Wault, Lille*	

	Vendredi 3/07	
8h30	MS5 Une nouvelle ère pour la cristallographie : intelligence artificielle et cristallographie quantique	
10h30	Pause-café	
11h00	MS6 (Amphi A) Multisources, multitechniques, conditions atypiques, *in situ*	MS1 (Amphi B) Cristallogenèse et Croissance cristalline
13h00	Clôture et remise des prix	

Programme détaillé (mardi 30-06-2026)

	Mardi 30/06	
8h00	**Accueil**	
8h30	**Mot de bienvenue**	
9h00	**Conférence plénière Biologie** *Modératrice : Solange Morera* **Herman van Tilbeurgh** (I2BC, Paris-Saclay) "Has the remarkable success story of protein crystallography come to an end?"	
10h00	**Pause-café**	
10h30	**MS13 (Amphi A)** **Patrimoine culturel et naturel, Sciences de la terre** *Modérateurs : Pauline Martinetto et Jean-Paul Itié* Invitée : **Emilie Bérard** (ICMMO, Paris-Saclay) "Révéler la nature et les techniques de mise en forme des alliages anciens par SR-DRX" Invité : **Victor Gonzalez** (PPSM, Paris-Saclay) "Multi-scale structural analysis of historical inorganic pigments" **Killian Fourie** (CINaM, Marseille) "La porosité de l'hématite comme archive thermique : vers un paléothermomètre de l'hématite de synthèse en archéométrie" **Coline Théron** (Edytem, Le Bourget-du-Lac) "Characterisation of pigments from the Otello shelter (Bouches-du-Rhône): a study that is not so simple, in which the combination of X-ray diffraction and fluorescence is essential" **Damien Jacob** (UMET, Villeneuve d'Ascq) "Non-Negative Matrix Factorization for 4D-STEM data analysis and phase mapping of astromaterials samples"	**MS11 (Amphi B)** **Structures résolues en temps** *Modératrice : Elke De Zitter* Invité : **Nicolas Caramello** (IBS/ESRF, Grenoble) "Mapping the slow propagation of disorder across a protein photoreceptor's tertiary structure with time-resolved crystallography" **Raphaël De Wijn** (XFEL, Schenefeld) "Capabilities and highlights of the SPB/SFX Scientific Instrument at the European XFEL" **Maria Davila** (IBS, Grenoble) "Visualizing the B12-dependent photoreceptor CarH in action with time-resolved electron cryo-microscopy" **Damien Léa** (Institut de Physique de Rennes) "Ultrafast electron diffraction study of insulator to metal photoinduced phase transition"
12h30	**Pause déjeuner**	
14h00	**L'AFC invite la British Crystallographic Association** *Modératrices : Coralie Bompard et Lydia Karmazin* Invité : **Simon Coles** (University of Southampton) "The synergy between X-ray and Electron single crystal diffraction in dealing with challenging samples" Invité : **Gideon Davies** (University of York) "Crystallographic studies of enzyme reaction coordinates and the design and exploitation of activity-based probes"	
15h30	**Session Exposants** *Modératrice : Natália T. Correia* Anton Paar Rigaku Quantum Design / Eldico Malvern Panalytical	
16h30	**Session Posters - Pause café**	
17h30	**Prix Guinier** *Modérateur : Matias Velazquez* **Marianne Quiquandon** et **Denis Gratias** (IRCP, Paris) "L'aventure des quasicristaux"	
18h30	**Soirée de bienvenue Ch'ti**	

Programme détaillé (mercredi 01-07-2026)

	Mercredi 1er/07	
8h30	**Conférence plénière Physique** *Modérateur : Sébastien Pillet* **Sylvain Ravy** (LPS, Paris-Saclay) "Infrastructures de recherche et cristallographie"	
9h30	**Pause-café**	
10h00	**MS3 (Amphi A)** **Matériaux pour l'énergie : de la production au stockage** *Modérateurs : Nathalie Audebrand et Pascal Roussel* Invitée : **Christelle Tamain** (CEA Marcoule) "Apport de la cristallographie et de la spéciation au cycle du combustible nucléaire" **Maxime Oliva** (Centrale Lille, Villeneuve d'Ascq) "Crystal growth and structural investigation of the new NbM1.5Cl5 (M=Mn, Co, Fe and Ni) family obtained by chemical vapor transport" **Maria Diaz Lopez** (Institut Néel, Grenoble) "Reimagining Interface Processing in Solid-State Batteries via Electrochemical Flash Sintering: Spatial Insights and Millisecond Structural Dynamics from Total Scattering" **Christy Sahyoun Conut Elias Mondo** (UCCS, Villeneuve d'Ascq) "Structural design and ionic conductivity in low-dimensional oxychlorides" **Killian Fourie** (CINaM,Marseille) "Comprendre le confinement du soufre dans des matrices poreuses par la caractérisation structurale et spectroscopique"	**MS9 (Amphi B)** **Biologie Structurale Intégrative** *Modérateurs : Coralie Bompard et Pierre Legrand* Invité : **Yvain Nicolet** (IBS, Grenoble) "Structural insights into nitrogenase femo-co assembly" **Armelle Vigouroux** (I2BC, Paris-Saclay) "New insights into the mechanism of iron transport through the bacterial Ftr system present in pathogens" **Sofia Kapetanaki** (MAX IV, Lund) "Study of the conformational dynamics of a bacterial photoactivated adenylate cyclase" **Kamel El Omari** (Diamond Light Source, Didcot) "Light atoms identification and location by anomalous scattering" **Juliette Devos** (ILL, Grenoble) "Deuteration of biomolecules for neutron structural biology"
12h00	**Assemblée Générale AFC**	
13h00	**Pause déjeuner** **Photo de groupe**	
14h30	**MS10** **Enseignement et médiation scientifique** *Modérateurs : Delphine Cabaret et Mathieu Marchivie* Invitée : **Paola Giura** (IMPMC Paris) "Enseignement et médiation scientifique, le rôle clé de l'experience" Invité : **Arie van der Lee** (IEM, Montpellier) "The Cambridge Structural Database is not only a database but also a knowledge base – workshops in France" **Michel Giorgi** (FSCM, Marseille) "A single crystal X-ray diffractometer scale model for scientific dissemination" **Sébastien Pillet & Elodie Tailleur** (CRM2, Nancy) "The Fourier-Maton : a visual demonstration of the Fourier transform methods in crystallography" **Claude Sauter & Agnès Gaudry** (IBMC, Strasbourg) "An immersive experience in biocrystallography" **Stéphanie Kodjikian** (Institut Néel, Grenoble) et al. '3D-Electron Diffraction": a training workshop focused on the practical aspects of 3D electron diffraction in materials and life sciences" **Peter Horton** & **Simon Coles** (Univ. Southampton) "Hands-on, challenge-based teaching in crystallography undergraduate practicals" **Thierry ROISNEL** (ISCR, Rennes) CRYSCALC, a crystallographic calculator **Carmelo Prestipino** (CRISMAT, Caen) & **Thierry Roisnel** (ISCR, Rennes) "A Python ecosystem for powder X-ray diffraction data visualization, editing, and analysis: XRDPlotter, PlotTxrd and PCReditor" **Nicolas Casaretto** (LCM, IPP, Palaiseau) et al. Atelier vidéo : "je suis cristallographe"	
16h00	**Session Posters - Pause café** **MS10** **Enseignement et médiation scientifique (suite)** Découverte des ateliers	
18h00		
19h00	**Conférence grand public** *Modérateur : Pascal Roussel* **Patrick Cordier** (Université de Lille, Institut Universitaire de France) "Quand les cristaux se désorganisent, de l'amorphisation des minéraux aux matériaux du futur" *Lieu : Auditorium du Palais de Beaux-Arts, 18bis rue de Valmy, Lille*	

Programme détaillé (jeudi 02-07-2026)

	Jeudi 2/07	
8h30	**Conférence plénière Chimie** *Modérateur : Carmelo Prestipino* **Laurence Croguennec (ICMCB, Bordeaux)** "Le défi matériaux pour inventer des nouvelles chimies et technologies de batteries"	
9h30	**Pause-café**	
10h00	**MS8 (Amphi A)** **Biologie et chimie pour la santé, Drug design** *Modérateurs : Sonia Fieulaine et Jean-Denis Pedelacq* Invité : **Alain Baulard** (CIIL, Lille) "Alpibectir/Ethionamide: The Long and Winding Road to a New Therapeutic Concept" **Mathieu Guérain** (UMET, Villeneuve d'Ascq) "Résolution de la structure de cocristaux d'intérêts pharmaceutiques et étude des relations structure-propriétés" **Théodore Arnaud** (ILL, Grenoble) "Neutron Crystallography Study of Host–Pathogen Recognition Enhanced by Hydrogen/Deuterium Exchange on Carbohydrates" **Hornela Yemene Tagoue** (UMET, Villeneuve d'Ascq) "Mechanism and Kinetics of Milling-Induced Polymorphic Transformation in Mefenamic Acid"	**MS14 (Amphi B)** **Désordre, apériodicité, structure locale** *Modérateurs : El-Eulmi Bendeif et Olivier Pérez* Invitée : **Claire Colin** (Institut Néel, Grenoble) "Modulation of the modulated magnetic structure of an Ho i-MAX phase" **Arie van der Lee** (IEM, Montpellier) "Incommensurate organic hydroxy channel structures: when the first-order satellite reflections are as strong as the main reflections" **Hassan Khoder** (ICSM, Marcoule) "Local structure and dynamics of nanoconfined water and electrolyte solutions" **Fantine Massicot** (CRM2, Nancy) "Complémentarité entre les simulations théoriques et résultats expérimentaux : applications aux matériaux photo-commutables" **Olivier Mentré** (UCCS, Villeneuve d'Ascq) "Electronic factors governing the modulated vs. periodic structural and spin lattices in the BaMP2O7 series"
12h00	**Pause déjeuner**	
13h00	**Prix de thèse** *Modérateur : Claude Sauter* Biologie : **Nicolas Caramello** (IBS, ESRF, Grenoble) Chimie : **Sabrina Grenda** (LMI, Lyon) Physique : **Antoine Gallo--Frantz** (LPS, Orsay) Prix spécial : **Ronald Rios-Santacruz** (IBS, Grenoble)	
14h00	**MS7 (Amphi A)** **Diffraction et imagerie 3D à l'échelle nanométrique** *Modérateurs : Philippe Boullay et Stéphanie Kodjikian* Invitée : **Tatiana Gorelik** (Ernst Ruska-Centre for Microscopy and Spectroscopy with Electrons, Jülich) "Serial Electron Diffraction – Targeting Nanocrystals" **Christophe Lepoittevin** (Institut Néel, Grenoble) "Structural study by Transmission Electron Microscopy of bulk calcium-doped neodymium Nickelates" **Antonin Raynal** (Institut Néel, Grenoble) "3D-Electron Diffraction unveils new melanotekite-based intergrowth series (Pb2FeO3)(Pb2Ge2Fe2O9)n" **Emre Yörük** (Holodiag, Val de Reuil) "Diffraction d'électrons pour l'industrie pharmaceutique" **Dominique Housset** (IBS, Grenoble) "3D ED platform at IBS: Examples of results in structural biology and material sciences and future developments"	**MS4 (Amphi B)** **Chimie moléculaire et supramoléculaire, Intéractions et reconnaissance moléculaire** *Modérateurs : Laure Vendier et Olivier Jeannin* Invitée : **Valérie Marvaud** (IPCM, Paris) "Magnetic dendrimers: tacking the challenge of synthesis and characterization" **Elen Duverger-Nédellec** (ICMCB, Bordeaux) "Keep Cool with Crystals: the Refrigeration of the Future" **Lise-Marie Chamoreau** (IPCM, Paris) "Structural characterization of molecular coordination networks based on oxamate ligands" **Hrudya Pariyacheri Padikkal** (Institut des Sciences Chimiques de Rennes) "Chalcogen bonding in organic conductors" **Nathalie Guillou** (Institut Lavoisier, Versailles) "Low-temperature transitions in the 2D [Cu2(1,3-BDT)]n coordination polymer"
16h00	**Session Posters - Pause café**	
17h00	**MS2 (Amphi A)** **Science des matériaux, texture, microstructure et contraintes résiduelles** *Modérateurs : Marie-Vanessa Coulet et Daniel Chateigner* Invité : **Yassine El Mendili** (ESTP Paris) "Microstructure et DRX pour comprendre et optimiser les matériaux de construction bas carbone" **Fatima Mota** (IM2NP, Marseille) "Etude de la croissance facettée et de la dynamique des défauts pendant la solidification dirigée du salol" **Arthur Souesme** (IRCER, Limoges) "Réseau de neurones convolutionnels pour l'analyse haut-débit de diagrammes de diffraction des rayons X" **Noussaïba Merdjane** (UCCS, Villeneuve d'Ascq) "Cation ordering and low-temperature symmetry lowering in $Li_2V_3GaO_8$" **Timmo Weidner** (UMET, Villeneuve d'Ascq) "Dislocation electron tomography in a SEM"	**MS12 (Amphi B)** **SAXS, WAXS et structure à l'échelle nano** *Modérateurs : Elise Guerinoni et Grégory Stoclet* Invitée : **Isabelle Morfin** (LiPhy/D2AM Grenoble) "SANS, SAXS, WAXS of soft matter and biomaterials" **Arie van der Lee** (IEM, Montpellier) "From WAXS to SAXS: experiences at the Montpellier X-ray beamline" **Gabin Gbabode** (LSMS, Rouen) "Polymorphic selection of biclotymol in thin films"
19h00		
20h00	**Dîner de gala** *Lieu : Hôtel Couvent des Minimes, 17 Quai du Wault, Lille*	

Programme détaillé (vendredi 03-07-2026)

	Vendredi 3/07	
8h30	**MS5**	
	Une nouvelle ère pour la cristallographie : intelligence artificielle et cristallographie quantique	
	Modérateurs : Enrique Espinosa et Arie van der Lee	
	Invité : **Alessandro Genoni** (Politechnico, Milan)	
	"Advancing Quantum Crystallography: New Directions for the X-ray Restrained Wavefunction Method"	
	Invité : **Antoine Taly** (IBPC, Paris)	
	"No, the folding problem is not solved"	
	Marc Lensink (UGSF, Villeneuve d'Ascq)	
	"CAPRI: catalyzing biomolecular interaction prediction for 25 years"	
	Léonard Imbert (IMN, Nantes)	
	"Crystalline phase projection and identification from high temperature diffraction patterns using graph neural networks"	
	Clément Poissonnet (CRISMAT, Caen)	
	"Analyse combinée DRX-IA pour des bétons"	
10h30	**Pause-café**	
11h00	**MS6 (Amphi A)**	**MS1 (Ampi B)**
	Multisources, multitechniques, conditions atypiques, *in situ*	**Cristallogenèse et Croissance cristalline**
	Modérateurs : Magali Putero et Brice Kauffmann	*Modérateurs : Fátima Mota et Gabin Gbabode*
	Invitée : **Marie-Ingrid Richard** (CEA Grenoble)	Invitée : **Joséphine de Meester** (NISM, Namur)
	"Probing Energy Materials at the Nanoscale: In Situ Bragg Coherent Diffraction Imaging"	"Single-crystal formation in double-emulsion (water/resine/water) microcapsules"
	Elodie Tailleur (CRM2, Nancy)	**Fabian Puga Montesdeoca** (UMET, Villeneuve d'Ascq)
	"Ferroelectricity in 3D and 2D Hybrid organic-inorganic perovskites"	"Coformer Dependent Thermal Collapse of Channel Type Carbamazepine Cocrystals"
	Nano Kingue Mouangue (IEMN, Villeneuve d'Ascq)	**Patrick Douglas Shaw Stewart** (Douglas Instruments, Hungerford)
	"Multimodal Operando Characterization of Li-Ion Thin Film Electrodes"	"Using phase diagrams with microseeding to prepare crystal samples for routine and advanced data collection techniques"
	Jérôme Rouquette (ICG, Montpellier)	**Anna Bonnardel** (IBS, Grenoble)
	"Transition de phase structurale induite par un champ magnétique à l'origine de la magnétostriction macroscopique dans le terfenol-D"	"Nanocrystallisation and chemical cross-linking: towards thermostable and recyclable crystal-based biocatalysts for industry"
	Olivier Pérez (CRISMAT, Caen)	**Eric Girard** (IBS, Grenoble)
	"Unveiling a Charge Density Wave in the m = 2 Monophosphate Tungsten Bronze: A Comprehensive Multitechnique Approach to Structure and Physical Properties"	"The nucleating agents, crystallophores, to boost your crystal production"
13h00	**Clôture et remise des prix**	

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L’aventure des quasicristaux

Denis GRATIAS (CNRS IRCP/Chimie ParisTech)
Marianne QUIQUANDON (IRCP, UMR 8247 (CNRS – Chimie ParisTech))

Nous discuterons des circonstances historiques de la rédaction de l’article fondateur de 1984 annonçant la découverte des quasicristaux de 1982 par Dan Shechtman et les multiples réactions scientifiques qui ont suivi sa publication.

Nous retracerons ensuite les étapes principales franchies pour aborder le difficile et nouveau problème de la détermination structurale des quasicristaux icosaédriques stables depuis les difficultés expérimentales d’élaboration métallurgique jusqu’à celles du traitement des données dans le formalisme de la cristallographie 6-dimensionnelle : indexation des spectres de diffraction, positions et formes des surfaces atomiques dans l’espace 6D et coupe finale par l’espace physique 3D engendrant la structure atomique quasipériodique réelle.

Nous conclurons cet exposé en discutant quelques conséquences les plus marquantes que ces études ont apportées à la cristallographie d’aujourd’hui dans ses concepts fondamentaux : périodicité et diffraction de Bragg ? symétrie : superposabilité ou indiscernabilité ? la presque-périodicité, une triviale généralisation de la périodicité ?

Quand les cristaux se désorganisent, de l’amorphisation des minéraux aux matériaux du futur

M Patrick CORDIER (Université de Lille)

Les cristaux fascinent par leur ordre parfait et leurs symétries. Pourtant, certains minéraux peuvent perdre cet ordre et devenir amorphes sans fondre. Cette conférence propose une exploration des processus d’amorphisation à l’état solide, lorsque la structure d’un cristal se désorganise sous l’effet de pressions extrêmes, d’ondes de choc, du rayonnement ou de réactions chimiques. Nous verrons comment la nature produit des verres d’impact, des minéraux métamictes ou des phases amorphisées dans les zones de faille, comment ces transformations sont reproduites en laboratoire, et pourquoi la maîtrise de l’amorphisation ouvre aujourd’hui de nouvelles perspectives, depuis la compréhension des processus astrophysiques et géologiques jusqu’au développement de technologies fondées sur la commutation entre états cristallin et amorphe.

Has the remarkable success story of protein crystallography come to an end?

M Herman VAN TILBEURGH (Institut de biologie intégrative de la cellule (I2BC))

Ever since the first diffraction pattern was observed on pepsin crystals about 90 years ago by D. Crowfoot and J. Bernal, protein crystallography has become a very efficient and successful method to study the three-dimensional structure of macromolecules. It developed slowly at first due to the huge technical problems that had to be solved, but its impact on understanding biological processes at the molecular level became predominant in the second half of the 20th century.

Structural data obtained by crystallography has been central to understanding many biological phenomena, as illustrated by the numerous Nobel Prizes awarded to researchers using this technique. It took decades and great perseverance by very talented scientists to make the isomorphous replacement phasing methodology work, as shown by the structures of hemoglobin and myoglobin.

Many technological and methodological advances since then have contributed to the spread of X-ray diffraction in the structural biology community. In this paper, the evolution of the technology at the instrumental, theoretical, and biochemical levels will be discussed.

At the end of the century, the community became aware of the widening gap between the massive amount of protein sequence data generated by genome sequencing and the available 3D structures. Structural genomics projects were launched worldwide to accelerate protein structure determination, and tens of thousands of new structures were solved. These data greatly contributed to the success of AlphaFold algorithms, which now predict 3D structures from sequences at spectacular speed and with high accuracy.

Did these algorithms dig the grave of protein crystallography, or are there still relevant biochemical questions beyond the reach of AlphaFold?

The synergy between X-ray and Electron single crystal diffraction in dealing with challenging samples

Prof Simon COLES (University of Southampton)

The UK National Crystallography Service (NCS, www.ncs.ac.uk) is amongst the most powerful and highest throughput single crystal X-ray diffraction facilities in the world, and examines samples that a typical crystallography unit cannot. Recently, Electron Diffraction (3D-ED) has the potential to revolutionise small molecule crystal structure determination for samples that stubbornly refuse to grow suitable sized crystals.

X-ray and 3D-ED are perfectly complementary – samples tend to diffract X-rays down to a few microns in size and when it is only possible to produce crystallites of 1 micron to 100 nanometres then electrons can be used. Firstly, there are studies where a particular sample tends to produce small crystals and secondly there are entire areas of science that only produce powders. Accordingly, across chemical and materials science, particularly inorganic solid-state, MOFs and catalysis, a new wealth and volume of crystal structure information will become available. However, 3D-ED has some limitations: dynamical diffraction can mean data processing and structure refinement don't produce the quality of results we are used to, while ultra-high vacuum and radiation damage can cause complications.

This talk will summarise experiences of the National Electron Diffraction Facility, a new arm of the NCS and how it is driving the development of new methods in 3D-ED. Results from developments in a) Energy Dispersive Spectroscopy to probe sample composition and b) in-situ controlled sample environments e.g. variable temperature and gas adsorption, will be discussed. Results will be presented, followed by a discussion of their trustworthiness and what the structures produced can be used for. Moreover, as the volume of 3D-ED structures is set to rapidly increase, some insights will be given into how we should approach responsible publication, such as definitions of structure quality, comparison with X-ray results and requirements for validation e.g. the availability of raw data.

Crystallographic studies of enzyme reaction coordinates and the design and exploitation of activity-based probes

Gideon DAVIES (University of York)

Carbohydrates play myriad roles in nature, from structural support and energy storage to molecular recognition in health and disease. Over the past several years, we have used X-ray crystallography to dissect the reaction coordinates of carbohydrate-active enzymes such as glycosidases. These studies have revealed a rich landscape of sugar-ring distortions that facilitate catalysis - now mapped as enzyme-specific “reaction coordinates”[1]. Individual enzymes follow distinct conformational pathways, and the challenge is now to exploit these conformations in the design of enzyme inhibitors[2], including activity-based probes (ABPs[3,4]) and therapeutic agents. Building on mechanistic insight and conformational mimicry, we have developed precision chemical tools for diverse classes of glycosidases, many with clinical potential, including anti-influenza[5] and anticancer[6] agents. In this BCA lecture, I will summarise our work using crystallography both to map enzyme reaction-coordinates and to harness this knowledge for enzyme-specific inhibition and imaging[*].

References

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[*]Funded by ERC "Carbocentre" with Overkleeft (Leiden) and Rovira (Barcelona).

Infrastructures de recherche et cristallographie

M Sylvain RAVY (Laboratoire de Physique des Solides, CNRS, Université Paris-Saclay)

La France est actionnaire ou partie prenante dans un certain nombre d'infrastructures de recherche (IR), situées sur son territoire ou ailleurs en Europe, où des études de cristallographie (au sens large) sont menées. Sources de photons ou de neutrons, ces IR jouent un rôle clé pour la communauté des cristallographes. Cette présentation vise à expliquer comment ces infrastructures sont choisies, gérées et financées par leurs tutelles, et à esquisser leur trajectoire possible dans un monde en pleine mutation.

Le défi matériaux pour inventer des nouvelles chimies et technologies de batteries

Laurence CROGUENNEC (ICMCB, CNRS UMR 5026, Université de Bordeaux, Bordeaux INP)

Quelle que ce soit la chimie ou la technologie de batterie envisagée, chaque avancée repose sur la découverte et l'optimisation de matériaux. Par exemple, les batteries sodium-ion, attractives du fait de l'abondance, la disponibilité et le moindre coût du sodium par rapport au lithium, exigent la découverte de matériaux d'électrodes innovants leur permettant d'atteindre des performances compétitives avec celles de la technologie Lithium-ion graphite-$LiFePO_4$. Mais, chaque solution passe par une recherche approfondie sur les propriétés des matériaux, et par une compréhension des mécanismes mis en jeu au cours du fonctionnement de la batterie pour les optimiser. Le contrôle de la relation composition-structure-défauts-propriétés est essentiel !

J'illustrerai ces défis à travers deux exemples. Nous avons récemment (i) découvert, en combinant calculs théoriques et expériences, une nouvelle famille de matériaux de type structural NASICON $Na_xV_2(PO_4)_3$, et (ii) démontré comment la chimie des anions mixtes et en particulier la compétition entre la liaison ionique V-F et la liaison vanadyle, covalente, de type V=O a un impact sur les propriétés de $Na_3V_2(PO_4)_2F_{3-y}O_y$. Les modifications de la composition et de la structure des matériaux doivent être étudiées dans leur environnement (*in situ*), et en temps réel (*operando*) lors de leur préparation ou du fonctionnement de la batterie car elles sont le plus souvent dans des conditions hors équilibre. Les expériences ainsi menées, le plus souvent aux grands instruments, permettent d'étudier la dynamique des réactions, essentielle pour la compréhension du matériau et l'optimisation de ses performances dans le système de stockage électrochimique.

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*) patent US 10847336

SINGLE-CRYSTAL FORMATION IN DOUBLE-EMULSION (WATER/RESINE/WATER) MICROCAPSULES

Joséphine DE MEESTER (UNamur - Namur Institute of Structured Matter)
Marie METTLER (Secoya Technologies)
Adrien DEWANDRE (Secoya Technologies)
Jean SEPTAVAUX (Secoya Technologies)
Benoit SCHEID (ULB - TIPs)
Johan WOUTERS (UNamur - Namur Institute of Structured Matter)

Crystallization plays a crucial role in the separation and purification of molecules and it allows to screen various solid forms, such as solvates and polymorphs. Moreover, single-crystal X-ray diffraction (SC-XRD) enables to determine molecular structures.[1,2] Microfluidic approaches have emerged as powerful tools for studying and optimizing crystallization by confining small volumes in droplets, offering rapid screening and precise control of crystal growth conditions, and low reagent consumption.[3,4]

Building on previous work that demonstrated the feasibility of crystallization in double-emulsion (water/resin/water) microcapsules cross-linked by UV irradiation,[5] our research aims to broaden the scope of this technology. We are focusing on:

(i) Crystallization of multi-component phases (salts and cocrystals) to validate the robustness of the microfluidic system,

(ii) In situ SC-XRD analysis of proteins, by incorporating glycerol as cryoprotectant into the capsules. Two glycerol incorporation strategies will be explored: direct encapsulation and diffusion through the shell,

(iii) Developing an organic/resin/organic system to broaden the scope of molecules that can be crystallized using the microfluidic system.

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Coformer Dependent Thermal Collapse of Channel Type Carbamazepine Cocrystals

Fabian PUGA MONTESDEOCA (Universite de Lille)
Natália CORREIA (Lille University)
Florence DANEDE (Lille University)
Frédéric AFFOUARD (Lille University)

Channel like carbamazepine (CBZ) cocrystals have been described as structurally disordered phases [Childs et al., 2008], yet their stoichiometry, stability, and thermal behavior remain insufficiently resolved. This uncertainty obscures the fundamental factors governing the formation and evolution of channel type molecular frameworks. In this study, we revisit four reported CBZ channel like cocrystals, formed with oxalic acid (OA), maleic acid (MA), malonic acid (MO), and 4 hydroxybenzoic acid (4HBA), to clarify their crystallogenesis and thermal transformation pathways.

Cocrystals were prepared by liquid assisted grinding and solvent evaporation. Powder X ray diffraction combined with Rietveld refinement confirmed that all phases adopt the previously reported channel like framework and can be reproducibly obtained with well-defined stoichiometries: 3:1 for CBZ:OA, CBZ:MA, and CBZ:MO, and 4:1 for CBZ:4HBA. Their diffraction patterns match the corresponding entries in the Cambridge Structural Database, demonstrating that the channel type architecture forms robustly across different coformers and crystallization routes.

Differential scanning calorimetry reveals melting behaviors that differ from earlier reports, pointing to previously unrecognized features in the thermal evolution of these phases. In situ variable temperature powder X ray diffraction shows that the channel like framework is intrinsically fragile: it collapses upon heating below the melting point. The collapse pathway depends strongly on the coformer, leading to the formation of either a 1:1 cocrystal, CBZ form I, or CBZ dihydrate.

These results demonstrate that channel like CBZ cocrystals sharing an identical crystallographic framework can nevertheless exhibit distinct thermal responses. They further establish that the chemical structure of the coformer residing within the channels governs the thermal behavior and collapse pathway of the channel type architecture, clarifying the transformation pathways and the structural factors that control the evolution of these channel-like molecular frameworks.

Using phase diagrams with microseeding to prepare crystal samples for routine and advanced data collection techniques

Patrick Douglas SHAW STEWART (Douglas Instruments Ltd)
Stefan KOLEK (Douglas Instruments Ltd)
Jack STUBBS (MAX IV Laboratory)
Peter BALDOCK (Douglas Instruments Ltd)

Serial data collection and microED techniques require slurries of tiny, well-ordered crystals, while neutron diffraction demands very large single crystals. Producing suitable samples is often complex, requiring multiple rounds of optimization. To guide this process, protein crystallizers work with a notional phase diagram comprising four zones: undersaturated (protein remains in solution), metastable (crystals grow from seeds), nucleation (spontaneous crystal appearance), and precipitation. Since real phase diagrams vary considerably between proteins, experimental determination of individual target protein phase diagrams is very valuable. Douglas Instruments, in collaboration with the University of Southampton, has developed a rapid, automated method for generating custom phase diagrams using just 15–60 µL of protein. The primary approach uses the microbatch-under-oil method, which prevents sample concentration (unlike vapor diffusion setups). Running the same procedure with and without a seedstock identifies the metastable zone. Microbatch also scales readily to 50 µL or larger batches (also using robotics),generating the higher sample volumes that advanced techniques often demand. A newer variant eliminates oil entirely by using a sitting-drop setup in which reservoir solutions are dispensed to exactly balance drop concentrations. We present case studies demonstrating how experimentally-determined phase diagrams improve control over crystallization conditions and crystal quality for both routine and advanced data collection.

Nanocrystallisation and chemical cross-linking: towards thermostable and recyclable crystal-based biocatalysts for industry

Anna BONNARDEL (CNRS - Institut de Biologie Structurale)
Charlotte RAYBAUD (CNRS)
Bruno FRANZETTI (CNRS - Institut de Biologie Structurale)
Eric GIRARD (CEA-Institut de Biologie structurale)

Enzymatic modifications are increasingly being used in the food industry to produce hydrolysates. Enzymes derived from extremophilic organisms are of great interest in this field, particularly because of their remarkable robustness and ability to function under extreme conditions (temperature, pH and pressure). Nevertheless, the use of extremozymes in this field is currently limited by their robustness wich prevent their inactivation as requested by the curent regulation.

In this context, we developed an immobilisation strategy by chemical cross-linking on crystallized enzymes to remove, recycle, and reuse them without loss of activity. I will present the setup of an immobilisation strategy in which diffusion limitations that reduce enzymatic activity are counterbalanced by the use of nanocrystals. The nano-crystallised enzymes are then immobilised via a two-step chemical cross-linking process, thereby maintaining the catalytic efficiency and significantly increasing their thermal and chemical stability.

In this study, we focused on TET aminopeptidases isolated from the hyperthermophilic archaeon Pyrococcus horikoshii. These enzymes have a distinctive structure, forming large oligomers (approximately 0.5 MDa) with a unique, self-compartmentalised organisation and tetrahedral shape (Appolaire et al., 2016). The contrasting substrate specificity found in this family of extremozymes (Chagny et al., 2025) can be harnessed for industrial processes such as producing functionalised hydrolysates (Patents No.: EP3723514, EP3724328 and EP3724329) with biological activities, such as antibiotic, antiviral, or antitumoral properties (Akbarian et al., 2022).

The protocol and results of the study of TET aminopeptidases led to a patent application, and our recent research shows that the two-step immobilisation protocol can be applied to other enzyme families. This study paves the way for the broader use of crystal-based catalysts.

The nucleating agents, crystallophores, to boost your crystal production.

Eric GIRARD (Institut de Biologie Structurale)
Amandine ROUX (Univ. Lyon, ENS de Lyon, CNRS UMR 5182, Université Claude Bernard Lyon 1, Laboratoire de Chimie,)
Natacha GILLET (Univ. Lyon, ENS de Lyon, CNRS UMR 5182, Université Claude Bernard Lyon 1, Laboratoire de Chimie)
Olivier MAURY (Univ. Lyon, ENS de Lyon, CNRS UMR 5182, Université Claude Bernard Lyon 1, Laboratoire de Chimie)

The crystallophore is a lanthanide complex combining phasing and nucleating properties [1], successfully exploited to produce crystalline forms free of crystal defects often encountered by crystallographers such as low-resolution diffracting samples or crystals with twinning [2], to generate crystals can from enriched fractions containing several proteins [2] leading to the structure determination of a protein complex [3] and to induce nucleation directly from the protein solution, as exemplified by the crystallization of hen egg white lysozyme in water [4].

The nucleating properties of the crystallophore have been challenged for the production of crystals with the appropriate size for either serial crystallography (SX) experiments or electron diffraction of 3D nanocrystals [5]. Moreover, these crystals were generated in the minute time-scale opening new opportunities in time-resolved SX. We will also show the input of crystallophore variants bearing chemical modifications to expand the possibility to control the number and size of crystals [6]. Finally, to facilitate crystal detection, a crystallophore with optimized imaging property completes the toolbox [7].

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Funding:

Authors acknowledge financial supports Agence Nationale de la Recherche (ANR Ln23-13-BS07-0007-01; ANR GlowCryst ANR-23-CE29-0002) and Region Auvergne Rhône Alpes (projects Xo4-2.0 and Crysfrag).

Curcumin Cocrystal Formation: Structural Consistency Across Three Crystallization Routes

Florence DANÈDE (UMET, Université de Lille)
Levine F. TEGUIMDJIO (UMET, Université de Lille)
Maulana ARIEFAI (UMET, Université de Lille)
Natalia T. CORREIA (UMET, Université de Lille)

The development of multicomponent crystalline forms is a relevant strategy for tuning the solid state properties of bioactive molecules. Curcumin, despite its broad interest, remains limited by low solubility and modest stability. This work investigates the formation of a curcumin cocrystal with a selected coformer using complementary crystallization routes, with the aim of comparing their efficiency and assessing the consistency of the resulting solid forms.
Three synthesis strategies were examined: liquid assisted grinding (LAG), solvent evaporation, and microwave assisted crystallization, the latter introduced for the first time in our group as a rapid and solvent saving approach. Several experimental conditions were screened for each method. From a crystallographic standpoint, all three routes yield similar solid forms, with no significant differences in the powder X ray diffraction patterns. DSC reveals a slight shoulder in the melting event of the LAG material, indicating a minor thermal deviation without affecting the overall structural consistency.
Overall, the results show that microwave assisted crystallization affords the same well defined cocrystal phase as the two conventional methods, while requiring minimal solvent and markedly shorter processing times. This combination of structural equivalence and operational efficiency highlights the specific advantage of the microwave assisted route.

Microstructure et DRX pour comprendre et optimiser les matériaux de construction bas carbone

Prof Yassine EL MENDILI (ESTP)

Le développement de matériaux de construction bas carbone (liants alternatifs, substitutions cimentaires, activation alcaline, valorisation de coproduits et ressources recyclées, matériaux terre stabilisés) repose sur une maîtrise fine de la microstructure, qui gouverne à la fois la réactivité, la prise, les performances mécaniques et la durabilité. Cette conférence présentera une approche “microstructure–performances” appliquée à plusieurs familles de matériaux, en s’appuyant sur la diffraction des rayons X (DRX) comme outil central de suivi des phases et de leurs transformations.

À partir d’exemples issus de formulations cimentaires à forte substitution, de systèmes activés alcalinement (N-A-S-H / C-(A)-S-H) et de matériaux incorporant des carbonates réactifs (ex. coquillages), nous montrerons comment la DRX permet de suivre l’évolution des assemblages minéralogiques au cours de l’hydratation, de la polymérisation et de la carbonatation, et comment ces évolutions se traduisent dans l’organisation multi-échelle (produits de réaction, porosité, interfaces). L’exposé discutera également l’intérêt de combiner DRX (quantification lorsque pertinent, prise en compte de la fraction amorphe) avec des caractérisations complémentaires (Raman/FTIR, TG/DSC, MEB/EDS, sorption) afin d’obtenir une lecture robuste de la microstructure dans des systèmes complexes et souvent multiphasiques.

L’objectif est d’illustrer comment le suivi microstructural permet d’identifier des mécanismes dominants (réactivité des précurseurs, rôle des carbonates, stabilité des hydrates, évolution des phases secondaires), d’expliquer les différences de comportement entre formulations, et de dégager des leviers d’optimisation compatibles avec les contraintes de mise en œuvre et les exigences de durabilité des ouvrages.

Etude de la croissance facettée et de la dynamique des défauts pendant la solidification dirigée du salol

Fatima MOTA (IM2NP UMR 7334 CNRS)
Anassya RAAD (IM2NP UMR 7334 CNRS)
Nathalie BERGEON (IM2NP UMR 7334 CNRS)
Nathalie MANGELINCK-NOËL (IM2NP UMR 7334 CNRS)

Pendant la solidification à partir du bain fondu, la morphologie de l'interface solide-liquide dépend des paramètres physico-chimiques du matériau et du procédé. Il est essentiel de caractériser cette interface, car elle joue un rôle clé dans la définition des propriétés finales du matériau. Les interfaces facettées à l'échelle macroscopique sont une morphologie typique générée lors de la solidification de nombreux matériaux, tels que les semi-conducteurs et les quasi-cristaux. L'attachement des atomes depuis le liquide à l'interface est anisotrope et lent, ce qui influence la croissance cristalline et donne des surfaces lisses à l'échelle atomique. Comprendre la dynamique de cette morphologie facettée reste un défi, nécessitant des informations clés qui ne peuvent être obtenues que par une observation in situ et en temps réel du processus.

Dans ce travail, nous étudions les mécanismes impliqués dans la croissance des interfaces facettées, en utilisant des expériences de solidification dirigée d'un matériau organique transparent. Nous présenterons les résultats obtenus avec le salol pur, un cristal orthorhombique qui, en raison de son fort degré d'anisotropie, croît à partir du liquide avec une interface solide-liquide fortement facettée. Les expériences ont été réalisées dans un four de type Bridgman, équipé d'un microscope optique permettant d'observer en temps réel et in situ la dynamique de l'interface solide-liquide. La solidification dirigée permet un contrôle précis des paramètres du processus (gradient thermique et vitesse de tirage) et donc l'étude de l'évolution de la morphologie et de la dynamique de l'interface en fonction de ces paramètres.

Nous présenterons les observations les plus marquantes sur la dynamique de croissance des facettes. Les défauts tels que les bulles, les marches de croissance ou les macles apparaissent fréquemment, et leur origine ainsi que leur dynamique seront discutées. La relation entre la vitesse de la facette, sa nature cristallographique et le sous-refroidissement sera également explorée.

Réseau de neurones convolutionnels pour l'analyse haut-débit de diagrammes de diffraction des rayons X

Arthur SOUESME (IRCER CNRS UMR 7315, Université de Limoges, Limoges, France)
René GUINEBRETIÈRE (IRCER CNRS UMR 7315, Université de Limoges, Limoges, France)
Olivier CASTELNAU (PIMM CNRS UMR 8006, École Nationale Supérieur des Arts et Métiers, Paris, France)
Alexandre BOULLE (IRCER CNRS UMR 7315, Université de Limoges, Limoges, France)

La diffraction des rayons X est largement utilisée en science des matériaux pour analyser la microstructure de matériaux polycristallins. La détermination des paramètres microstructuraux, comme la taille moyenne des cristaux et les microdéformations, à partir de diagrammes 1D, est généralement réalisée à l'aide de méthodes de simulation globale du diagramme.

Les avancées récentes concernant les instruments de mesures, notamment dans les grandes installations (synchrotron, XFEL), permettent de réaliser des expériences résolues dans le temps, in situ ou operando, entraînant l'explosion de la quantité de données obtenues. Bien qu'efficaces, le caractère chronophage des méthodes d'analyses traditionnelles constitue un facteur limitant majeur et rend crucial le développement de nouvelles méthodes rapides et fiables pour le traitement des données.

Dans ce travail, nous présentons une méthode basée sur la mise en œuvre de réseaux de neurones convolutionnels (CNN) que nous avons développée pour permettre l'analyse de diagrammes de diffraction des rayons X 1D en temps réel [1]. Nous avons développé un générateur de diagrammes de diffraction étiquetés pour l'entraînement du CNN. Ces diagrammes sont générés avec des caractéristiques structurales et microstructurales contrôlées, aisément adaptables à chaque cas considéré. Nous démontrerons le potentiel de notre approche au travers d'une étude in situ à haute température (1200 °C) de la formation à l'état solide du spinelle MgAl2O4, réalisée sur la ligne BM01 de l'ESRF. Nous montrerons durant cette communication comment notre approche a permis l'analyse de plusieurs ensembles de diagrammes de taille différente en quelques secondes. Au terme de cette étude, nous avons pu déterminer simultanément la proportion des phases présentes, la taille moyenne des cristallites et le taux de microdéformations.

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Cation ordering and low-temperature symmetry lowering in Li?V?GaO?

Noussaiba MERDJANE (CNRS UMR8181 UCCS-CS)

$Li_2V_3GaO_8$ crystallizes in an orthorhombic *Pmma* structure with room temperature lattice parameters a = 5.85862 Å, b = 5.78778 Å, c = 8.38599 Å and V = 284.356 Å^3. This symmetry lowering from the ideal cubic spinel reflects cation ordering on both tetrahedral and octahedral sublattices, with Ga^{3+} and Li^+ occupying the tetrahedral (A) sites and V and Li distributed over the octahedral (B) sites. The vanadium sublattice exhibits a mixed valence state of +3.66, resulting in inequivalent V-O bond distances within the BO_6 octahedra. The octahedral framework forms a distorted network of corner-sharing octahedra derived from the pyrochlore topology, where the combination of mixed valence and geometric constraints can promote electronic instabilities. In vanadium-based spinels, such instabilities are often relieved at low temperature through charge ordering and associated structural distortions, leading to symmetry lowering and differentiation of crystallographic sites. Laboratory X-ray diffraction measurements performed down to 16 K reveal clear and reversible changes in the intensity and shape of selected Bragg reflections below approximately 70 K. These observations indicate the onset of a subtle structural distortion, consistent with a low-temperature symmetry lowering, potentially driven by charge ordering and/or lattice effects within the vanadium sublattice. In addition to temperature-driven effects, cation substitution via controlled delithiation (replacement of two Li^+ by one divalent cation M^{2+}, M = Cu, Co, Mn) preserves charge neutrality and therefore does not modify the V^{3+}/V^{4+} ratio. Instead, it alters site occupancy, local symmetry, and magnetic exchange pathways, providing a means to tune the structural and electronic properties of the system. $Li_2V_3GaO_8$ thus represents a cation-ordered mixed-valence spinel in which the interplay between lattice distortion and electronic degrees of freedom gives rise to a low-temperature structural transition.

Dislocation electron tomography in a SEM

Timmo WEIDNER (Université de Lille)
Alexandre MUSSI (Université de Lille)
Romain GAUTIER (Université de Lille)
Antoine GUITTON (Université de Lorraine)
Dr Alexandre MUSSI (Université de Lille)

The plastic deformation of crystalline solids is controlled by the motion of dislocations, nanoscale linear defects whose occurrence and dynamics determine mechanical response. These defects have long been difficult to observe directly, but diffraction contrast in transmission electron microscopy (TEM) makes them visible. Realizing three dimensional visualization of dislocations constitutes a major objective because 3D imaging permits unambiguous identification of slip, climb, and mixed climb systems; unraveling of complex dislocation interactions; and characterization of the collective processes that control plastic deformation. Electron tomography, developed roughly sixty years ago to reconstruct small biological specimens with nanometer resolution [1], provided the methodological basis for this study. Its application to dislocation studies, however, was particularly difficult, and dislocation electron tomography (DET) only matured about twenty years ago [2] owing to the particular difficulties of diffraction contrast tomography, notably extreme sensitivity to orientation conditions and the complex interpretation of contrast.
In this talk, we present the DET performed under ECCI conditions using a scanning electron microscope (SEM). This new technique, which we have named ECCT [3], allows for the analysis of larger areas than is possible with TEM and opens up the possibility of performing quasi-4D analyses with SEM.

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Localisation des cations dans les pores d'un MOF luminescent à base de terbium utilisé comme capteur chimique

Lowik DEWEZ-RASZKA (INSA Rennes)
Corinne LAGROST (Université de Rennes, CNRS Insittut des Sciences Chimiques de Rennes)
Erik ELKAIM (Synchrotron SOLEIL)
Catherine DEJOIE (ESRF)
Yan SUFFREN (INSA Rennes)
Nathalie AUDEBRAND (Université de Rennes, INSA Rennes)

Les Metal-Organic Frameworks (MOFs) sont des polymères de coordination largement étudiés pour leur porosité et leurs applications de stockage et de séparation de molécules. Le degré de coordination élevé des ions lanthanides (Ln) permet de former des MOFs stables. De plus les Ln présentent une signature en luminescence intéressante avec de nombreuses bandes d'émission très fines souvent dans le visible comme dans le vert pour l'ion Tb3+. Il est possible de piéger des analytes à l'intérieur de la structure hôte des MOFs à base de Ln et d'obtenir une réponse rapide par l'estimation de l'intensité de la luminescence, faisant ainsi de ces matériaux des capteurs chimiques. Un MOF à base de ligand 2-hydroxy-1,4-benzènedicarboxylate et de formule chimique Ln(OH-C8H4O4)(OH-O2H8)4O3H8Ca été rapporté pour certains lanthanides. Il possède une structure cubique (Ia¯3) avec de grands canaux à section carrée (11x11 Å). Le MOF isotype hétéronucléaire (Tb3+, La3+, le lanthane étant nécessaire à la stabilisation de cette structure) a été synthétisé par précipitation et est luminescent dans le vert. Il a été mis en suspension sous forme de poudre dans des solutions d'analytes (Cr3+ et Fe3+). Les matériaux résultants, dénommés analyte@MOF, présentent une atténuation importante de la luminescence faisant du MOF un détecteur de Cr3+ et Fe3+. Les interactions chimiques entre la structure hôte du MOF et les analytes ont été étudiées à partir de données de diffraction des rayons X par la poudre (lignes CRISTAL de SOLEIL et ID22 de l'ESRF) pour les matériaux Cr@MOF et Fe@MOF. Les premiers affinements de Rietveld semblent démontrer que les analytes sont insérés au sein des pores de la charpente hôte. Les distances Cr-Tb sont suffisamment courtes pour provoquer un effet d'extinction de la luminescence. Les résultats sont également discutés au regard d'expériences de spectroscopie XPS.

Magneto-electric phase diagram of ferrotoroidic LiCrSi?O? pyroxene.

Salma EL MAZOUNI (Institut Néel - CNRS)
Claire COLIN (Institut Néel - CNRS)
Fabio DENIS ROMERO (Institut Néel - CNRS)
Eric RESSOUCHE (CEA Grenoble)
Jérôme DEBRAY (Institut Néel - CNRS)
Frédéric GAY (Institut Néel - CNRS)

The potential of antiferromagnetic spintronics hinges on the ability to manipulate antiferromagnetic states electrically, which necessitates exploring innovative material platforms to overcome this challenge. Ferrotoroidic compounds are promising candidates for achieving this level of control.

In this context, we explore the magnetoelectric properties of the clinopyroxene mineral $LiCrSi_2O_6$, which crystallises in the $P2_1/c$ space group. At T = 11.5 K, the material exhibits long-range antiferromagnetic order (k = 0), featuring an antiferromagnetic arrangement of spins within each S = 3/2 chain and a magnetic space group of $P2_1'/c$. To date, all measurements of this material have been performed on powder samples [1]. We have recently succeeded in growing the high-quality millimeter-sized single crystals using the flux method in order to investigate its anisotropic properties under different crystallographic orientations and to determine the magneto-electric phase diagram combining specific heat measurements and magnetisation measurements at Neel Institute and neutron diffraction on D23 at ILL .

At zero-field, a coherent picture emerges from the combined observation of a linear magnetoelectric effect - revealed by pyroelectric measurements – consistent with $P2_1'/c$ symmetry refined from 2K neutron diffraction data, with a magnetic moment $M(Cr^{3+})$ = 2.47(3)μB. Consequently, a ferrotoroidal moment, T, is allowed in the ac-plane and a significant toroidic moment can be calculated T= (-0.0032, 0, 0.0087) $\mu_B Å^{-2}$.

Under a magnetic field, a metamagnetic transition along the a and a* directions around 4T was first identified through magnetisation measurements. Refinement of the high-field neutron diffraction data does not allow a clear distinction between $P2_1'/c$ and $P2_1/c'$ symmetries. However, pyroelectric measurements under magnetic field reveal a term that is only compatible with the $P2_1/c'$ symmetry, thereby complementing the neutron results and demonstrating the spin-flop nature of the metamagnetic transition.

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Corrélation des techniques de diffraction XRD et EBSD sur films minces métalliques pour l'étude des microstructures

Frederic FILLOT (Univ. Grenoble Alpes, CEA, Leti, F-38000 Grenoble, France.)
Mathieu LOYER (Univ. Grenoble Alpes, CEA, Leti, F-38000 Grenoble, France.)
Maria-Luisa CALVO-MUÑOZ (Univ. Grenoble Alpes, CEA, Leti, F-38000 Grenoble, France.)

Les caractéristiques microstructurales des matériaux influencent fortement leurs propriétés physiques. Pour une caractérisation complète et cohérente de la microstructure, l'utilisation de plusieurs techniques complémentaires est pertinente pour obtenir des informations à la fois locales et globales. Effectivement, les techniques de diffraction sont adaptées pour obtenir les propriétés structurales des matériaux. Alors que la diffraction des rayons X (XRD) donne des informations statistiques, moyennes et résolues dans l'espace réciproque, la diffraction par électrons rétrodiffusés (EBSD) donne des informations locales et spatialement résolues sur l'état structural.

Dans ce travail, des considérations générales sont abordées pour mettre en évidence la complémentarité de ces deux techniques. Des résultats obtenus avec les deux techniques sur des couches minces de cuivre sont présentés. Ces résultats sont croisés, comparés, interprétés séparément et distingués, puis une tentative de corrélation est proposée. Les caractéristiques instrumentales, la physique des interactions, les forces et les faiblesses, les limites et les spécificités de chaque technique seront abordées.

« Ce travail, réalisé (en partie) sur la Plate-Forme de Nano-Caractérisation (PFNC), a bénéficié d'aides de l'Etat gérées par l'Agence Nationale de la Recherche au titre des programmes « Recherche Technologique de Base » et France 2030 (ANR-22-PEEL-0014) »

X-ray characterization of chiral materials

Sophie NOWAK (Université Paris Cité)
Vanshika SINGHANIA (Université Paris Cité)
Vincent REPAIN (Université Paris Cité)

The influence of the structure of the chiral surfaces and interfaces (h≠k≠l for cubic systems) on the magnetic and the spintronic properties of ultrathin films are studied. In order to understand these properties, we need to characterize the structure of the surfaces and interfaces using X-rays.

Various techniques were used: X-ray fluorescence and reflectivity (to estimate the thickness of the deposited layers) and, mainly, X-ray diffraction was conducted to obtain pole figure measurements. For this study, various devices of an Empyrean diffractometer (@Malvern-Panalytical) were used:

- X-ray lenses to obtain a nearly parallel beam in both directions (axial and equatorial)

- or a hybrid monochromator combined with a triple-axis system to produce a purely monochromatic and parallel beam.

The purpose of this poster is to describe the various configurations tested, present the results obtained, and draw conclusions regarding their relevance and limitations. This topic will also provide an opportunity to show the wide range of equipment available at the X-ray Platform.

Composite Phases with Translational Disorder

Agnese PRIMIERI (CRISMAT, CNRS, Normandie University, ENSICAEN, UNICAEN)
Carmelo PRESTIPINO (CRISMAT, CNRS, Normandie University, ENSICAEN, UNICAEN)
Olivier PEREZ (CRISMAT, CNRS, Normandie University, ENSICAEN, UNICAEN)

The MCr_2X_4 family of compounds (M = Pb, Sr, Ba, Eu, Sn; X = S, Se) exhibits incommensurate channel-type structures, with subunits sharing periodicity along a and b but differing along c. Previous studies on $BaCr_2Se_4$ and $SnCr_2S_4$ using electron diffraction revealed translational disorder within the channels. This structural complexity can strongly influence transport, making these materials promising for thermoelectric applications. $PbCr_2S_4$ and vanadium-substituted derivatives, $PbCr_{2-x}V_xS_4$ (x = 0.4, 0.6, 0.8, 1), were synthesized via high-temperature solid-state reactions. Powder X-ray diffraction shows a systematic change in lattice parameters with increasing vanadium content, confirming incorporation into the structure and suggesting a route to tune structural and electronic properties.

Apport de la cristallographie et de la spéciation au cycle du combustible nucléaire.

Christelle TAMAIN (CEA Marcoule)
Thomas DUMAS (CEA, Marcoule)
Dominique GUILLAUMONT (CEA Marcoule)
Stéphane GRANDJEAN (CEA Marcoule)
Bénédicte ARAB-CHAPELET (CEA Marcoule)
Murielle RIVENET (UCCS Lille)

La France a fait le choix du retraitement du combustible nucléaire usé afin de valoriser le potentiel énergétique des éléments recyclables et de réduire la quantité de déchets ultimes. Le traitement du combustible repose sur des procédés de séparation par extraction liquide-liquide permettant de récupérer l'uranium(VI) et le plutonium(IV) sous forme de flux purifiés. Ces actinides sont ensuite convertis en phase solide, conduisant aux oxydes correspondants, lesquels peuvent être pastillés puis frittés pour former de nouveaux combustibles. Les produits de fission et les actinides mineurs sont, quant à eux, vitrifiés en vue de leur entreposage en couche géologique profonde.

Les actinides présentent une chimie particulièrement riche et versatile en raison de la présence d'électrons f et de la proximité énergétique des orbitales 7s et 6d, ce qui se traduit par l'existence de nombreux degrés d'oxydation. L'étude de la spéciation des différents éléments au cours des procédés mis en œuvre constitue un élément clé pour comprendre et prédire leur comportement. Ces données contribuent également à l'enrichissement de modèles permettant, à terme, la simulation et le pilotage des procédés. Dans cette présentation, nous proposons d'illustrer l'apport de la cristallographie, et plus particulièrement de la diffraction des rayons X sur monocristal (DRX monocristal), aux différentes étapes du retraitement du combustible. Au-delà de l'enrichissement des bases de données structurales autorisant l'identification des phases solides formées, la DRX monocristal permet d'obtenir des modèles moléculaires complets et fiables, pouvant être corrélés à des données spectroscopiques acquises en solution (notamment via des spectres de référence). Ce lien entre phase solide et solution constitue une approche méthodologique particulièrement pertinente pour la détermination de la spéciation des actinides — stœchiométrie, modes de coordination et constantes de complexation — dans les différentes solutions intervenant au cours du procédé.

Crystal growth and structural investigation of the new NbM1.5Cl5 (M=Mn, Co, Fe and Ni) family obtained by chemical vapor transport

Maxime OLIVA (Centrale Lille)
Marie COLMONT (Centrale Lille)
Murielle RIVENET (Centrale Lille)
Claire LAVALETTE (ORANO)

Researches into the development of chloride molten salts reactors (MSR) are currently in full swing in particular by the possibility they offer to recycle fissile materials such as plutonium and americium [1]. The growth of unexpected phases could impair the reactor's performances and limit its durability [2]. This work aims to shed light on the reaction mechanisms occurring between the reactor materials and the primary salt or the chloride gaseous phases [3]. To do so, various systems involving the corrosion products (Fe, Ni, Cr, Mo, Nb..) likely to diffuse from the alloys mixed with the elements from a primary simulant molten salt (Ce,Na..) are under investigation, the main purposes being to supplement existing crystallographic databases and to list the phases likely to grow inside the reactor.

Chemical Vapor Transport (CVT) is a well-known technique for the crystal growth of halides [4] In the chemical systems Nb/M/Cl (M= Mn, Fe, Co and Ni), a structure type was evidenced. Its crystal structure was refined by single crystal X-Ray diffraction analysis. It shows a versatile 2D-layered Kagome-type crystal structure, similar with the well-known Mott insulator phase Nb3Cl8. [5]

Combining several experimental techniques, including single-crystal X-Ray diffraction, powder X-Ray diffraction, electron diffraction study and Raman spectroscopy we elucidated the crystalline structures of the NbM1.5Cl5 (M= Mn, Co, Fe and Ni) family of compounds. This comprehensive structural analysis, complemented by the characterization of thermal properties provides a full understanding of the growth mechanisms and reactivity of these compounds inside the MSR. This new phase is opening up a wide perspective by 2D-layered crystal structure modification through the possible incorporation of elements of the salt or fission products by insertion or substitution of sodium or tellurium. [6][7]

All references are available if needed.

Reimagining Interface Processing in Solid-State Batteries via Electrochemical Flash Sintering: Spatial Insights and Millisecond Structural Dynamics from Total Scattering

MARIA DIAZ LOPEZ (Institut Neel)
Claire COLIN (Institut Neel)
Riku FUKADA (Institut Neel 38000 Grenoble)

Flash sintering has emerged as a powerful route for the rapid densification of ceramics, but its application to solid electrolytes remains challenging because purely ionic conductors are limited by blocking electrodes, strong thermal gradients, and interfacial degradation. In this work, we investigate electrochemical flash sintering (EFS) as a route to fabricate self-supported, monolithic all-solid-state battery-type architectures composed of $Li_3V_2(PO_4)_3$–$Li_{1.4}Al_{0.4}Ti_{1.6}(PO_4)_3$ composite electrodes and an LATP electrolyte layer. By operating under AC conditions, densification is achieved in both composite and electrolyte layers while maintaining coherent electrode–electrolyte interfaces without preferential cracking or secondary-phase enrichment.

To understand the structural mechanisms governing this ultrafast process, we combine spatially resolved total scattering with automated large-scale diffraction analysis. Bragg diffraction and pair distribution function (PDF) reveal pronounced heterogeneities generated by current localization and Joule heating, including hotspot formation, localized decomposition, amorphization, and phase redistribution. Analysis of large diffraction datasets using non-negative matrix factorization (NMF) enables efficient identification of minor and spatially localized impurity phases, providing a statistical view of phase evolution across the pellet.

To probe transient reaction pathways, in-situ total scattering experiments were performed in an adapted cell[2] at the XPDF/i15-1 beamline of the Diamond Light Source using the new ARC detector[3], achieving ~50 ms time resolution. This enables direct observation of structural dynamics during the 2s-long flash event, including the rapid formation of metastable and amorphous intermediates, their crystallization within seconds, and the coupled evolution of local and average structure. By correlating these transformations with processing variables, we identify the parameters controlling densification, hotspot formation, and decomposition pathways.[4]

These results demonstrate how advanced scattering methods can resolve both spatial and temporal complexity in non-equilibrium field-assisted processing. More broadly, they provide design rules for the rapid fabrication of dense solid electrolytes, robust interfaces, and next-generation solid-state battery architectures.

Structural design and ionic conductivity in low-dimensional oxychlorides

Christy SAHYOUN CONUT ELIAS MONDO (UCCS)
Victor DUFFORT (UCCS Lille)
Marielle HUVE (UCCS Lille)
Olivier MENTRE (UCCS Lille)

This work aims to develop innovative chloride conducting materials based on inorganic bismuth oxychlorides for electrochemical energy storage systems of the future generations. Because of their natural abundance and low cost, chloride ions (Cl^-) are being studied as alternative charge carriers in order to solve the environmental and economic limits of present lithium-ion technologies, which rely on rare and essential materials. In this context, solid-state oxychloride electrolytes are emerging as a balanced solution, combining stability and performance while benefiting from the safety and longevity of solid-state battery designs.

Bismuth oxychlorides, such as Bi12Cl6O15 and Bi24Cl10O31, look like promising materials due to their distinctive structural features: 1D triangular tunnels and 2D buckled chlorine layers, respectively. In order to introduce chloride ion vacancies to improve ionic conductivity, Pb2+ substitution was attempted, but this modification led to unexpected structural transformations.

The resulting structures were found to belong to the previously reported [PbnBi10-nO13] [Bi2O2]nCl4+n family described between n=2 and 4. However, subtle variations in peak positions observed in X-ray powder diffraction patterns for certain compositions suggest that the proposed unified models do not fully account for the structural complexity. This indicates the possible existence of a much larger and more intricate family of compounds.

This presentation will focus on the challenges encountered in identifying the correct unit cell, even when employing advanced techniques such as single crystal diffraction and electron diffraction. To overcome these difficulties, we resorted to manually constructing the different members of this oxychloride family to search for compatible cells.

Finally, preliminary measurements of chloride ion mobility within these structures will be presented, offering new insights into their potential applications in materials science.

Comprendre le confinement du soufre dans des matrices poreuses par la caractérisation structurale et spectroscopique

Killian FOURIE (Aix-Marseille Université, Madirel, CINaM)
Marie-Vanessa COULET (Aix-Marseille Université, CNRS, Madirel, CINaM)
Daniel FERRY (Aix-Marseille Université, CNRS ,CINaM)
Denis MORINEAU (Université de Rennes 1, CNRS, Institut de Physique de Rennes)
Basma DUPONT (Université de Rennes 1, CNRS, Institut de Physique de Rennes)
Olivier GRAUBY (Aix-Marseille Université, CNRS ,CINaM)
Amélie DEMOULIN (Aix-Marseille Université, Madirel)
Vasile HERESANU (Aix-Marseille Université, CNRS ,CINaM)

À l'heure de la transition énergétique, les matériaux poreux dopés au soufre présentent un intérêt majeur pour les batteries Li-S. Les méthodes couramment utilisées pour doper ces matériaux sont la fonctionnalisation et l'imprégnation liquide. Pour un meilleur contrôle du remplissage de la porosité, nous utilisons une troisième voie : l'imprégnation en phase vapeur. Afin de mieux comprendre les propriétés thermodynamiques, structurales et dynamiques du soufre confiné dans différents matériaux poreux, nous étudions l'évolution de la taille des pores, de leur géométrie, ou encore de la chimie (de surface) du composite en fonction de la quantité de soufre insérée.

Nous présentons en détail les caractéristiques physico-chimiques de nos matrices de carbone et de silice établies par couplage de diffraction de rayons X, de spectroscopie Raman, et de tomographie électronique en transmission pour la silice, afin d'établir les caractéristiques dimensionnelles et topologiques de son réseau poreux.

La manométrie d'adsorption d'azote et l'analyse thermogravimétrique mettent en évidence le rôle prépondérant des micropores de la matrice carbonée dans les effets de confinement du soufre, et montrent l'existence de différents régimes de remplissage suivant la taille des pores (micro-/méso-). Elles permettent également de déterminer la masse volumique du soufre confiné.

Nous montrons par des mesures de diffraction de rayons X que le soufre confiné existe sous forme amorphe ou cristallisée, selon qu'il est adsorbé dans une matrice de carbone ou de silice ou selon qu'il est dans des micropores ou des mésopores. La spectroscopie Raman nous permet d'identifier la nature des sites d'adsorption potentiels, d'estimer l'énergie d'interaction soufre-matrice par l'analyse fine des spectres, et de montrer que le soufre a une affinité plus grande avec la matrice carbonée qu'avec la silice.

Nos résultats apportent un nouvel éclairage sur les caractéristiques physico-chimiques du soufre confiné dans des matrices poreuses pouvant être d'intérêt pour les batteries Li-S.

Nous remercions l'Agence Nationale de la Recherche (ANR STaR-S) de soutenir ce projet.

Ba(PO4)2M5+2O3 mixed metal systems (M = Nb, Ta, W) at the edge between dielectrics and 2D-metals.

SANDY AL BACHA (CNRS-UCCS)
Angel M. ARÉVALO-LÓPEZ (CNRS-UCCS)
Olivier MENTRÉ (CNRS-UCCS)
Robert GLAUM (Institut für Anorganische Chemie)
Andrés CANO (Institut NEEL CNRS/UGA UPR2940)

The recently discovered series of layered monophosphate tungsten bronzes, $Ba(PO_4)_2W_mO_{3m-3}$ (m = 2-5, W^{5+}/W^{6+}), provides a versatile framework for designing original two-dimensional (2D) electronic materials. Their structure consists of $[Ba(PO_4)_2]^{2-}$ spacer layers separating bidimensional perovskite-like tungsten-oxide slabs, forming a unique platform for electron confinement. Unlike conventional MPTBs prone to charge-density-wave (CDW) instabilities, LMPTBs remain stable 2D metals down to low temperatures due to their inherent "trigonality," which suppresses the in-plane tungsten displacements driving CDW formation. As a result, these materials constitute ideal systems for tuning 2D electron confinement through targeted cationic substitution on different crystallographic sites.

In this work, the focus is on investigating the chemical and electronic tunability of LMPTBs by substituting W^{5+} (d^1) with electronically passive d^0 M^{5+} cations. We focus on the m = 2 phases $Ba(PO_4)_2M_2O_3$ (*M* = Nb, Ta), previously reported and their mixed-metal derivatives, which lie at the boundary between wide-gap dielectrics and two-dimensional (2D) metallic systems. Composed exclusively of pentavalent transition metals, this family is particularly well suited for such a study. To this end, we successfully synthesized the two end-members along with their mixed-metal intermediates $Ba(PO_4)_2Nb_{2-x}Ta_xO_3$ and characterized them using synchrotron, dielectric measurements, and DFT calculations.

Initial structural analysis reveals a revision of the crystal symmetry from *R*-3*m* to the chiral *R*32, highlighting the role of antiferroelectric-like metal displacements in governing octahedral tilts. These distortions are linked to the preferential occupation of external metal sites adjacent to phosphate groups. In this context, the interplay between electronically inactive d0 cations (Nb^{5+}/Ta^{5+}) and active d^1 W^{5+} species provides a versatile platform for tuning carrier density, electronic confinement, and emergent states. Thus, these findings open promising perspectives for enhancing two-dimensional electronic confinement through mixed-metal substitution across various LMPTB members, with ongoing studies probing their magnetic and electronic properties using advanced spectroscopy and DFT calculations.

In-operando battery study of NMC811 and beyond: What a benchtop XRD can achieve

Eric BERTHIER (Thermo fisher scientific)
Carlotta GIACOBBE (Thermo fisher scientific)
Simon WELZMILLER (Thermo fisher scientific)
Weicai JU (Thermo fisher scientific)

This study demonstrates the capabilities of benchtop X-ray diffraction for investigating structural dynamics in both conventional and next-generation battery materials. By utilizing PYTHON scripts on large datasets, it is possible to conveniently correlate analytical data from different sources. Initially, we present a detailed in-operando analysis of LiNi0.8Mn0.1Co0.1O2 (NMC811) cathode materials during electrochemical cycling (cf. Figure 1 left). Under controlled conditions (2.7-4.3V), two complete charge-discharge cycles revealed significant structural transformations. Derived from Rietveld refinements of NMC811 [2] using Profex [1], during charging, the c lattice parameter expanded from 1.42 nm to 1.45 nm, while the a parameter contracted from 0.284 nm to 0.281 nm, corresponding to lithium deintercalation (cf. Figure 1 right). The material demonstrated high specific capacity (180 mAh/g) [3-5] and excellent Coulombic efficiency (91.8% first cycle, 99.8% second cycle). Equally, it is possible to extend such an investigation to solid-state batteries utilizing e.g. sulfide-based electrolytes (Li2S-P2S5 system), where XRD analysis enables monitoring of interfacial reactions and structural stability during cycling. Therefore, a specialized test cell needs to be used to pressurize the solid-state electrolyte. The methodology successfully tracks phase evolution and potential degradation mechanisms in both the cathode material and the solid electrolyte, providing crucial insights for optimizing these next-generation battery systems. This comprehensive approach demonstrates how benchtop XRD can effectively support the development of both conventional and solid-state battery technologies.

Theoretical Charge Density Study, NBO analysis and nonlinear optical (NLO) properties of benzo[4,5] imidazol [1,2-c] thiazole-1(3H)-thione

Dr TABTI CHAREF (Université de mostaganem, Algerie)
Megrouss YOUCEF (Université de chlef, Algerie)
Chouaih ABDELKADER (Université de Mostaganem, Algerie)

Determine the electron density distribution in a crystal enables the investigation of the specifics of the intraand inter-molecular interactions withbenzo[4,5]imidazo[1,2-c]thiazole-1(3H)-thione (BIThZThN), among other things. The electron density distribution was then modeled using the Hansen-Coppens multipolar model, and the interactions in crystals were carefully analyzed using the Atoms-In-Molecules refinement.Correlations between the geometrical characteristics of the contacts and the features of their critical points were analyzed in detail.The quantum chemical calculations of the molecule were performed using the density functional method (DFT)/B3LYP with the 6-311G(d,p) basis set in the ground state for the gas phase. The topological analysis has been also performed for both inter and intramolecular interactions. A number of intermolecular interactions were identified ranging from strong C-H….O hydrogen bonds through C-H….O to C-H…π and π….π interactions. The molecular dipole moment and the electrostatic potential were calculated and compared with those obtained using theoretical calculations. The molecular structures, HOMO-LUMO energies, NLO properties and Fukui functions were calculated andinterpreted. The RDG-NCI analysis of the molecule was performed to determine the non-covalent interactions present within the molecule. Finally, The investigation of the molecule's electron charge density is the main goal of this effort. By transferring the multipole parameters of the electron density, which are derived from accurate X-ray diffraction experiments, the study aims to compute the molecular dipole moment and model the aspherical features of the atomic electron density that result from the low-temperature chemical bonding of the molecule. The distribution of the molecule's electrostatic potential in the crystal was examined in order to comprehend the nature of intramolecular and intermolecular charge transfer.

Understanding the structural transitions of Inorganic Salt Hydrates for Thermal Energy Storage: A comprehensive In-situ X-ray Diffraction study

Dr Asma HASIL (Mines Saint-Etienne, CNRS, UMR 5307 LGF, Centre SPIN, F - 42023 Saint-Etienne France)

Inorganic salt hydrates are promising materials for thermochemical energy storage (TCES) due to their high energy density, cost-effectiveness, and availability [1]. However, their practical implementation is limited by performance degradation during repeated dehydration–rehydration cycles, including phase separation and loss of storage capacity [2]. Despite extensive studies on bulk thermophysical properties, the atomic-scale structural dynamics governing reversibility remain poorly understood.

A key limitation lies in the lack of detailed structural understanding of intermediate hydration states formed during cycling. Although these phases govern the reaction pathway, their crystallographic evolution and transformation kinetics remain insufficiently resolved [3–5].

This study investigates the structural evolution of $SrBr_2 \cdot xH_2O$ powder under thermal conditions relevant to TCES using in situ techniques. Variable-temperature powder X-ray diffraction (PXRD) is used to track phase transitions and identify intermediate phases, complemented by in situ infrared and Raman spectroscopy to probe hydration states.

By linking phase evolution to reaction progression, this work identifies key transformation steps and conditions leading to incomplete reversibility. These insights provide guidance for optimizing operating conditions and improving the stability of thermochemical energy storage materials.

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Organic functionalization to rationally improve the thermal properties of hybrid layered solid-solid phase change materials

Come ARCHINARD (Institut des Matériaux de Nantes)
Elias DJANFFAR (Institut des Matériaux de Nantes)
Ali SAAD (CEA-LITEN)
Bernard HUMBERT (Institut des Matériaux de Nantes)
Oivier HERNANDEZ (Institut des Matériaux de Nantes)
Gregory LARGILLER (CEA-LITEN)
Hubert CHEVREAU (Synchrotron SOLEIL)
Thomas DEVIC (Institut des Matériaux de Nantes)

Layered hybrid solids consisting of alternating sheets of inorganic anions and organic alkylammonium cations have been identified as early as in the 70-80s as solid-solid phase change materials (SS-PCMs); in line with the raising concerns regarding energy savings, they are nowadays investigated with a renewed interest for their potential applications in thermal storage and thermal management.[1] Compared to conventional SS-PCMs working in the same range of temperature (sugar alcohols, polymers), these crystalline solids offer a high level of tunability. For example, the latent heat and temperature of transition of 2-D perovskites formulated (CnH2n+1NH3)2MIICl4 (M = Cu, Mn, Co, Cd) evolve linearly with the length of the alkyl chain n and can thus be rationally selected.[2] Structural and spectroscopic studies have shown that the phase transitions are associated to the modification of the orientation, conformation and motion of the alkyl chains. We thus anticipated that the modification the supramolecular interactions between the alkylammonium cation (which are purely van der Waals in the above-mentioned materials) should strongly affect the thermal properties. For this purpose, hydrogen bond donor and acceptor groups are here introduced at the end of the alkyl chains. We show that once selected the proper functional group, strengthening the organic-organic supramolecular interactions through O-H··O hydrogen bonds leads a noticeable increase of the latent heat (+40 to 70%).[3] This increase directly correlates to the variation of the hydrogen bond strength during the phase transition, and can be easily quantified by infrared spectroscopy. We also show that such a functionalization led to a strong increase of the thermal conductivity thanks to the rigidification of the organic-organic interface.

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Exploring perovskite material properties for green environmental applications

Mme Salima LABIDI (University)

Perovskite materials have garnered significant attention for green environmental applications, possess interesting and unique properties, which can offer potential technological applications in Solar Cells, Energy Storage (Batteries and Supercapacitors), Carbon Capture and Sequestration, Photocatalytic Applications for Environmental Remediation,Water Splitting for Hydrogen Production.
The aim of this work is to envisage the effects of magnetoelectric and magnetocaloric in perovskite materials for green environmental applications by using the ab initio calculations and Monte Carlo simulations which are used as input for experimental study.

Structure determination of new ionic conductors from complex mixtures

Loic MAZE (Malvern Panalytical)
Gwilherm NÉNERT (MalvernPanalytical)
Maxim AVDEEV (The University of Sydney)

While combinatorial synthesis coupled to theoretical calculations is gaining more and more attention for the discovery of new materials, it remains a challenge to determine the crystal structure of new materials from mixtures. In the last decade, despite that many efforts have been dedicated to the combinatorial synthesis and the characterization of the obtained phases, it usually concentrates on single phase materials [1]. Single phase materials are obtained usually when the phase diagram is known, which is usually not the case when exploring new systems. At the exception of serial rotation electron diffraction [2], typically the structure determination of new phases remains unanswered when dealing with complex mixtures. This challenge was addressed recently using a full software solution from phase identification to pattern deconvolution and Rietveld refinement [3].

In this contribution, we are reporting on the exploration of the system Li/Na-Sn-P-O. We could solve the crystal structures of 2 new candidates for ionic conductors obtained from phase mixtures, namely A2Sn(PO4)2 (A = Li, Na) from 5 min laboratory X-ray scan data. Both materials exhibit a new structural type within the A2M(PO4)2 series (A= Alkali ion, M = Zr, Ti, Sn). The crystal structure accuracy was further confirmed by DFT calculations. Investigation of the ion conduction pathways from bond valence suggests that those materials are good candidates for ionic conduction. The ability to combine phase identification and to disentangle the various contributions from a phase mixture for structure solution opens the possibility to fully exploit the capabilities of combinatorial chemistry using laboratory powder diffraction.

Keywords: Combinatorial Chemistry; Structure Solution from Powder Data; Phase Mixture; electrode material

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Process-controlled defect engegnering and intrinsic low thermal conductivity in layered Cu2ZrS3

Dr Carmelo PRESTIPINO (Crismat)
Lucas LE GARS (CRISMAT UMR6508(Caen))
Olivier PEREZ (CRISMAT UMR6508(Caen))
Emmanue GUILMEAU (CRISMAT UMR6508(Caen))
Philippe BOULLAY (CRISMAT UMR6508(Caen))

Controlling lattice thermal conductivity (κL) is pivotal in designing materials for thermoelectric and thermal management applications. Sulfide-based compounds offer an eco-friendly, earth-abundant alternative to tellurides and selenides but generally suffer from high κL. Here, we investigate Cu_2ZrS_3, a layered metal sulfide recently reported to exhibit intrinsically low κL (~0.35 $W{\cdot}m^{-1}{\cdot}K^{-1}$ at 683 K), attributed to its unique Cu-based trigonal coordination and strong lattice anharmonicity.[1]
Through a combined approach of controlled synthesis, advanced diffraction techniques, and first-principles modeling, we uncover the existence of two novel structural polytypes of Cu_2ZrS_3, resulting from different thermal histories. These polytypes display distinct stacking sequences (ABCB and ABC), variations in atomic displacement, and levels of structural disorder, all of which critically impact phonon transport. Notably, diffuse scattering and mixed-site occupancy indicate the presence of stacking faults that has been simulated and quantitatively evaluated.
Heat capacity measurements, Raman spectroscopy, and phonon calculations confirm the presence of low-energy optical phonons associated with Cu, facilitating strong optical-acoustic phonon interactions that intrinsically suppress κL. These findings establish a clear correlation between structural polytypism and thermal transport in Cu-rich sulfides, offering new pathways toward sustainable, cost-effective materials with tailored thermal properties.
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Magnetic dendrimers: tacking the challenge of synthesis and characterization

Valérie MARVAUD (CNRS)
Cédric PÉCOU (Institut Parisien de Chimie Moléculaire - Sorbonne Université - CNRS)
Ingrid SUZANA (Ecole Centrale Méditerrannée)
Jérémy FORTÉ (Institut Parisien de Chimie Moléculaire - Sorbonne Université - CNRS)
Malischewski MORITZ (Freie Universität)

Despite the vast potential of dendrimer chemistry, magnetic dendrimers remain relatively scarce in the literature due to perplexing challenges in their synthesis and characterization. Consequently, designing such multimetallic and monodisperse starburst architectures endowed with magnetic properties poses a significant challenge. Initial efforts focused on the coupling of tetra- and penta-nuclear complexes, with promising results. The Gd5Cu16 complex showed magnetization compatible with S = 51/2, while the Tb5Cu16 equivalent showed single molecule magnetic behavior but no crystallographic structure.
Using a supramolecular "complex as ligand" approach, we successfully address the challenge through synthesis, characterization (including X-ray diffraction) and investigation of magnetic dendrimers. Our synthetic strategy relies predominantly on oxalate-based coordination chemistry as the core of the architecture, alongside trinuclear complexes, LnCo2, for the branches, which exhibit single-molecule magnetic properties. The integration of these building blocks allows the formation of supramolecular and dendritic assemblies. The versatility of this approach allows the anticipation of properties based on the choice of metal cations involved in the structure, such as single molecule magnets et giant spin values. Consequently, we have obtained hetero-tri-metallic dendrimers, MLn3Co6 and ZrLn4Co8 (where M=Co or Cr, and Ln=La, Tb, Dy, etc.), fully characterized by X-ray diffraction. The observed magnetic properties are in good agreement with theoretical models, suggesting potential applications in information storage, quantum computing or magnetic refrigeration.

Keep Cool with Crystals: the Refrigeration of the Future.

Elen DUVERGER-NÉDELLEC (ICMCB-UMR5026)
Liza EL KHOURY (ICMCB-UMR5026)
Nathalie DARO (ICMCB-UMR5026)
Guillaume CHASTANET (ICMCB-UMR5026)
Philippe GUIONNEAU (ICMCB-UMR5026)
Michaël JOSSE (ICMCB-UMR5026)
Mathieu MARCHIVIE (ICMCB-UMR5026)
Patrick ROSA (ICMCB-UMR5026)

Refrigeration devices we use in everyday life (air conditioners, refrigerators, etc.) are responsible for 8% of global greenhouse gas emissions. An alternative to refrigerant gases would be the use of a technology based on a solid, easily handled and recyclable, and its mechanical response to a pressure/depression cycle [1]. Among these materials, known as barocaloric materials, molecular spin-crossover (SCO) compounds have been identified as having promising barocaloric potential [2,3]. Although obtaining these materials in single-crystal form is complex, large quantities of polycrystalline powder can be produced from non-critical, inexpensive, abundant, and non-toxic elements. However, using powder in a technological application is challenging. Being able to process it into a more compact form, in order to facilitate the handling of the material of interest, is therefore a prerequisite for technological development.

While Cool-SPS (Spark Plasma Sintering) pelletizing has been demonstrated on various model systems to produce compact, centimeter-sized monoliths of brittle compounds [4], only one study has been reported for a molecular spin-crossover (SCO) material [5] leading to resulting densities comprising between 80 and 90% of the density of the corresponding crystal structure. The densification via Cool-SPS of new molecular SCO compounds has been investigated within the framework of FROSTBIT project (First Regenerative Solid-State Barocaloric Refrigerator) and will be presented. The FROSTBIT project relies on these molecular pellets to develop a regenerative cooling device with the aim of producing a fully functional refrigeration prototype.

Acknowledgements: The authors thank CNRS-Innovation (projet BAROQUE) and the European Innovation Council (projet FROSTBIT, Pathfinder Challenge GA n°101161137).

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Structural characterization of molecular coordination networks based on oxamate ligands

Ang LI (Wuhan Business University, College of Food Science and Technology)
Bhramar BISWAS (Institut Parisien de Chimie Moléculaire - Sorbonne Université - CNRS)
Lise-marie CHAMOREAU (Institut Parisien de Chimie Moléculaire - Sorbonne Université - CNRS)
Benoît BAPTISTE (Institut de Minéralogie, de Physique des Matériaux et de Cosmochimie)
Ludovic DELBES (Institut de Minéralogie, de Physique des Matériaux et de Cosmochimie)
Yanling LI (Institut Parisien de Chimie Moléculaire - Sorbonne Université - CNRS)
Yves JOURNAUX (Institut Parisien de Chimie Moléculaire - Sorbonne Université - CNRS)
Laurent LISNARD (Institut Parisien de Chimie Moléculaire - Sorbonne Université - CNRS)
Pierre FERTEY (Synchrotron SOLEIL)

Phenyl-oxamate ligands have been used in our group for a long time as synthetic building blocks, due to their ability to create molecular assemblies which show interesting magnetic properties. The use of these multi-polydentate ligands has led to the obtention of various hetero-metallic architectures among which 2D or 3D networks. To fully characterize these compounds, single-crystal x-ray diffraction was systematically used to elucidate the structural models. We pay particular attention to the environement of the metallic cations and the connectivity of the ligands. Powder diffraction experiments were employed to investigate the thermal stabilty of the species. Some of them showed phase a transition before degradation and we then tried, with more or less success, to carry out some single-crystal to single-crystal transitions with the aim to better understand the structural evolution observed with temperature. Finally, different synthetic pathways were also investigated and compared through XRD.

Chalcogen bonding in organic conductors

Hrudya PARIYACHERI PADIKKAL (UMR-6226)
Olivier JEANNIN (UMR-6226)
Ie-Rang JEON (UMR-5031)
Marc FOURMIGUE (UMR-6226)

Redox active electron rich molecules have been attaining significant attraction in the recent years because of their interesting physical and chemical properties that make them capable of rendering promising applications in organic electronics. Multifunctionality can be induced in these molecules by introducing a paramagnetic metal complex to favor interaction between π electrons of the cation radical salts and metallic d spin. Here, we take advantage of an intermolecular interaction called chalcogen bonding (ChB), to induce these possibly strong spin-charge couplings. We aim to synthesize organic electron donors different from the commonly used tetrathiafulvalene derivatives because of the weak spin density on the extremity in these molecules. In the newly designed donors, the spin density is present in positions readily available for spin coupling with magnetic counter ions, in their cation radical state. For example, the family of naphthalene peri-dichalcogenides and their radical cations, with their spin density mostly concentrated on the Se atoms seems to be very promising for magnetic interactions. Moreover, these molecules in their radical state, will develop electron deficient regions, σ-holes, around Se atoms. Consequently, Se atoms, on the oxidized donor bearing the spin density, have the possibility to be engaged in so-called chalcogen bond, with the magnetic anions, such as $FeCl_4^-$, $FeBr_4^-$ acting as ChB acceptors, achieved through electrocrystallization.

Similarly, organic donors can also give rise to charge transfer complexes (CTCs) with electron acceptors like 7,7,8,8-tetracyanoquinodimethane (TCNQ) and/or fluorinated TCNQ derivatives (TCNQF, $TCNQF_2$, $TCNQF_4$) mediated by ChB interactions by co-crystallization techniques. The degree of charge-transfer in these organic conductors is one of the key parameters characterizing their ground state properties. These properties on 2, 3, 7, 8-tetramethoxy selenanthrene and their CTC s are studied with the help of IR and Raman spectroscopy techniques.

Low-temperature transitions in the 2D [Cu2(1,3-BDT)]n coordination polymer

Chloé ANDRADE (CNRS UMR 5256, IRCELYON)
Alexis GIRAUDON (CNRS UMR 5256, IRCELYON)
Régis DEBORD (CNRS UMR 5306, ILM)
Nathalie GUILLOU (CNRS UMR 8180, Institut Lavoisier de Versailles)
Erik ELKAÏM (Synchrotron SOLEIL)
Florent PERRET (UMR CNRS 5182, ENS)
Aude DEMESSENCE (CNRS UMR 5256, IRCELYON)
Stéphane PAILHÈS (CNRS UMR 5306, ILM)

While thermoelectric devices are primarily based on inorganic materials, coordination polymers (CPs) - constructed from metal ions connected to organic ligands - are attracting growing interest. Indeed, their structural diversity (from 1D to 3D networks) and hybrid nature enable new strategies for tuning electronic properties. Combined with low lattice thermal conductivity and relatively high thermal stability, these features offer new opportunities in thermoelectricity (heat-to-electricity conversion). In this context, our focus has been on the study of $[Cu_2(1,3\text{-BDT})]_n$, a CP based on Cu(I) and 1,3-benzenedithiolate. Its structure, solved from powder X-ray diffraction, consists of chains in which copper atoms are coordinated to three sulfur atoms and vice versa, forming distorted Cu_3S_3-type hexagons (C. Andrade et al., *J. Mater. Chem. C*, **2023**, 11(42), 14540). These chains are then connected by bridging ligands to form a 2D network. This CP exhibits p-type semiconductor properties and a record Seebeck coefficient, which can be explained by the presence of the 1D tubular inorganic $\{Cu\text{-}S\}_n$ network. A semiconductor-to-metal transition in its electrical resistivity was observed below 150 K (C. Andrade et al., *Synthetic Metals*, **2025**, 314, 117912). This is accompanied by a change in the sign of the Seebeck coefficient with a drastic increase of its absolute value, and combined to the emergence of paramagnetism. To understand this behaviour, powder X-ray diffraction data were recorded on the CRISTAL beamline (SOLEIL synchrotron) between 290 and 6 K. The unit cell volume of $[Cu_2(1,3\text{-BDT})]_n$ decreases linearly up to 12 K, the temperature at which it increases drastically. Rietveld refinement of the various diffraction patterns recorded before and after this structural transition will be presented and the structural modifications carefully analysed, in order to better understand the electrical behaviour of $[Cu_2(1,3\text{-BDT})]n$.

Structural Studies of Four Polymorphs of Sodium Vanadium Phosphite NaV(HPO?)?

Thierry BATAILLE (ISCR - Ecole Nationale Supérieure de Chimie de Rennes)
Eric LE FUR (ISCR - Ecole Nationale Supérieure de Chimie de Rennes)

Polymorphism is a key phenomenon in solid-state chemistry, often leading to significant variations in the physicochemical properties of materials with identical chemical compositions. In this context, sodium vanadium phosphite, $NaV(HPO_3)_2$, represents a remarkable case, with four distinct polymorphs obtained through closely related synthetic routes.

Single-crystal and powder X-ray structure determination revealed the existence of a high-symmetry cubic phase ($Pn\bar{3}c$), two distinct monoclinic phases crystallizing in the space group C2/c, and a lower-symmetry monoclinic phase described in $P2_1/n$. Despite their different symmetries, all polymorphs are built from three-dimensional framework composed of corner-sharing VO_6 octahedra and HPO_3^{2-} groups, with structural variations mainly affecting the local arrangement of the framework and the coordination environment of sodium cations.

At present, the mechanisms governing the formation and stabilization of each polymorph remain unexplained. Although different synthesis conditions lead preferentially to the expected polymorph, frequently traces of some others are observed. No straightforward correlation has yet been established between the synthetic parameters and the resulting phase. These observations point to a complex energy landscape for $NaV(HPO_3)_2$, involving closely related structural minima that may be controlled by subtle thermodynamic and/or kinetic factors.

The $NaV(HPO_3)_2$ system therefore provides an attractive model for investigating the interplay between polymorphism, crystal symmetry, and synthesis conditions in inorganic phosphite materials. Further studies will be required to elucidate the factors responsible for polymorph selection and stability within this family of compounds.

Combining scXRD, VCD, ECD, and Quantum Chemistry for Absolute Configuration and Intermolecular Interaction Studies

Jean-Valère NAUBRON (Fédération Sciences Chimiques Marseille (UAR1739))
Michel GIORGI (Fédération Sciences Chimiques Marseille UAR 1739)
Sara CHENTOUF (CRCMM)

Determining the absolute configuration of chiral molecules is a critical challenge in catalysis, materials science, and pharmaceuticals. While **single-crystal X-ray diffraction (scXRD)** remains the gold standard for structural elucidation, **electronic circular dichroism (ECD)** and **vibrational circular dichroism (VCD)** provide complementary approaches to assign stereochemistry with high confidence.

Both VCD and ECD exploit the differential absorption of left- and right-handed circularly polarized light by chiral molecules—VCD in the mid-IR region and ECD in the UV-vis range. By comparing experimental spectra with quantum chemistry calculations (DFT, TD-DFT), the absolute configuration can be reliably assigned: a matching sign between measured and calculated spectra confirms the correct stereochemistry.

Beyond absolute configuration, the synergy of **scXRD, VCD, ECD**, and **quantum chemistry modeling** enables the investigation of subtle intermolecular interactions crucial for fully characterizing chiral properties. This poster highlights three case studies demonstrating their complementary insights:

• **VCD spectroscopy complementing scXRD** to reveal an isotopic chirality phenomenon.

• **scXRD/VCD analysis of a dynamically frozen chirality phenomenon.**

• Examining **chirality induction effects** when achiral and chiral molecules interact in close proximity, emphasizing the role of local environments on chiroptical responses.

By integrating these techniques, we establish a robust framework for resolving stereochemistry and probing intermolecular interactions, paving the way for deeper understanding of chiral systems in chemistry and materials science.

Étude cristallographique sous pression de la stabilisation inattendue de l'état haut spin du fer(II) dans des complexes à ligands bipyridinium

Laure VENDIER (CNRS, Laboratoire de Chimie de Coordination)
Livia GETZNER (LCC - Laboratoire de chimie de coordination)
Yasmine REMILI (CNRS, Laboratoire de Chimie de Coordination)
Damian PALIWODA (European Spallation Source ERIC)
Gábor MOLNÁR (LCC - Laboratoire de chimie de coordination)
Saioa COBO (LCC - Laboratoire de chimie de coordination)
Azzedine BOUSSEKSOU (LCC - Laboratoire de chimie de coordination)

L'étude cristallographique, réalisée par diffraction des rayons X sur monocristal sous pression, porte principalement sur le matériau $\{Fe(CH_3\text{-}pbpy)_2[\mu_2\text{-}Pd(CN)_4]_2 \cdot 2H_2O\}$ complétée par des données sur des composés analogues de Pt ou des ligands substitués. Elle est essentielle pour comprendre les transitions de spin observées. Elle permet de suivre précisément l'évolution des distances métal-ligand (Fe–N), des paramètres de maille et des déformations structurales en fonction de la pression.

À pression ambiante, le composé est dans l'état haut spin (HS1), caractérisé par des distances Fe–N longues (~2,16 Å). Lorsque la pression atteint 1,2 GPa, ces distances diminuent significativement (~1,93 Å), indiquant une transition complète vers l'état bas spin (LS1), plus compact.

L'observation la plus remarquable intervient entre 1,2 et 2,0 GPa : les distances Fe–N augmentent à nouveau (~2,12 Å), preuve d'un retour inattendu à un état haut spin (HS2). Enfin, à 2,5 GPa, une nouvelle contraction (~1,97 Å) confirme une seconde transition vers un état bas spin (LS2). Malgré ces changements locaux, le volume de la maille diminue continûment avec la pression, respectant ainsi la thermodynamique.

Au-delà des distances Fe–N, l'analyse révèle des transformations structurales importantes des ligands bipyridinium : torsions accrues, déformations hors du plan et modification des angles interplans. En particulier, dans l'état HS2, les ligands deviennent fortement non plans, favorisant des interactions π–π renforcées entre couches.

Ces changements structuraux sont associés à un transfert d'électrons induit par la pression entre les unités cyanure et les ligands organiques. Ce phénomène modifie le champ ligand autour du fer, expliquant la stabilisation inattendue de l'état haut spin sous pression.

Ainsi, l'étude cristallographique met en évidence un couplage étroit entre structure, transfert électronique et état de spin, à l'origine du comportement atypique du matériau.

Référence
Pressure-Induced Unexpected Stabilization of the High-Spin State of Iron(II) in a Metal–Organic Framework, Livia Getzner et al, J. Am. Chem. Soc. 2025, 147, 50, 46497–46504

Advancing Quantum Crystallography: New Directions for the X-ray Restrained Wavefunction Method

Prof Alessandro GENONI (Department of Chemistry, Materials and Chemical Engineering "Giulio Natta" - Politecnico di Milano)

The X-ray restrained wavefunction (XRW) method, also known as X-ray constrained wavefunction (XCW) approach, was introduced in 1998 by Dylan Jayatilaka [1, 2]. Since then, the technique has steadily improved and has become a cornerstone computational tool in modern quantum crystallography [3].

From a theoretical standpoint, the method has been continuously refined. Initially introduced within the restricted Hartree–Fock approximation, it has subsequently been extended to include more advanced quantum chemical approaches, such as relativistic methods and multi-determinant valence-bond techniques.

In terms of applications, the approach has proved valuable across several research areas. It has contributed to the reinterpretation of traditional chemical bonding concepts such as hypervalency, supported investigations of optoelectronic properties in systems with strong nonlinear optical responses, and enabled the study of biologically and pharmaceutically relevant compounds.

This presentation will begin with an introduction to the theoretical foundations of the XRW method, followed by a discussion of its current and future developments. In particular, the presentation will touch on some possible directions: the potential use of the Jayatilaka approach in developing new exchange–correlation functionals for density functional theory; the integration of Compton scattering data alongside, or in place of, traditional Bragg diffraction data; and the possible extension of the XRW method within quantum computing frameworks to efficiently generate large multi-determinant wavefunctions consistent with experimental observations.

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No, the folding problem is not solved

Antoine TALY (UMR8266, UP Cité/CNRS)

The advent of deep learning-driven tools such as AlphaFold has revolutionized the prediction of biomolecular structures, offering unprecedented accuracy and accessibility for proteins, RNA, and their complexes. While these tools have demonstrated remarkable success in benchmarking competitions and enabled experimentalists to generate models with ease, their widespread use has also highlighted persistent challenges. These include difficulties in assessing model confidence, limitations in predicting transmembrane domains, conformational diversity, and interactions with ions or ligands, as well as the tendency to misfold intrinsically disordered regions (IDRs).

This presentation will highlight the strengths and limitations of current AI-based structure prediction tools through illustrative examples with a particular emphasis on the impact of ion modelling. We discuss the importance of reporting confidence metrics in publications to avoid over-interpretation. We also explore the impact of ions on predictions, demonstrating how their inclusion can induce conformational changes, while also revealing biases in certain predictions. Furthermore, we address the unique challenges of RNA structure prediction, where data scarcity and structural complexity limit the performance of both classical and deep learning methods. Our analysis underscores the need for continued methodological advancements, integration of complementary computational tools, and expansion of high-quality experimental datasets

CAPRI: catalyzing biomolecular interaction prediction for 25 years

Marc LENSINK (University of Lille)
Alexandre BONVIN (University of Utrecht)

CAPRI is a community-wide blind prediction experiment, aimed at the improvement of computational protein interaction models. It relies on experimentalists who provide unreleased target structures. The first Round of the experiment took place over the summer of 2001, making 2026 CAPRI's 25th year of catalyzing method development.
The most recent Evaluation period has witnessed the transition to AI-driven prediction tools such as AlphaFold and related methods. While the AlphaFold baseline registers remarkable success on previous CAPRI targets, even when those targets fell outside its training set, recent Rounds have highlighted its limitations on targets that lack conservation in their binding regions, such as antibodies and nanobodies.
Most CAPRI targets, including these, fall in the difficult-to-predict category, underscoring the challenging nature of the CAPRI endeavor. Human predictors continue to outperform AI methods on such complex cases.
CAPRI gratefully acknowledges the continued commitment of participating groups, who persist despite their frustration of having to tackle such difficult targets. We also acknowledge the generosity of experimentalists who provide structures prior to publication. Continued contributions from experimentalists offering targets to blind experiments will remain essential to overcoming the remaining challenges in structural prediction for complex biomolecular systems.

Crystalline phase projection and identification from high temperature diffraction patterns using graph neural networks

Léonard IMBERT (L'Institut des Matériaux de Nantes Jean Rouxel)
Christopher L. HASSAM (L'Institut des Matériaux de Nantes Jean Rouxel)
Olivier HERNANDEZ (L'Institut des Matériaux de Nantes Jean Rouxel)
David BERTHEBAUD (L'Institut des Matériaux de Nantes Jean Rouxel)
David PORTEHAULT (Sorbonne Université, CNRS)
Romain GAUTIER (L'Institut des Matériaux de Nantes Jean Rouxel)

Powder X-ray diffraction (PXRD) is a common technique for crystallographic phase identification, as each crystalline phase produces a unique diffraction pattern unique to its atomic structure. However, most structural databases are populated with patterns measured at ambient temperature and pressure, which may not be representative of the experimental condition. This is the case for example in high-temperature molten salts. Additionally, identifying phases in unknown materials remains difficult due to the absence of reference patterns.

In this presentation, we introduce a Graph Neural Network (GNN) designed to extract distinct phases from multiphase thermodiffraction patterns and estimate their room-temperature diffraction patterns. Our method is based on the temperature-dependent shifts of diffraction peaks—linked to lattice expansion—to distinguish between phases and reconstruct their ambient-temperature signatures.

Analyse combinée DRX-IA pour des bétons

Clément POISSONNET (CRISMAT / Université de Caen)
Daniel CHATEIGNER (CRISMAT / Université de Caen)
Frédéric JURIE (GREYC)
Yassine EL MENDILI (ESTP)

L'identification-quantification de phases et l'estimation des paramètres de maille à partir des mesures de diffraction X (DRX) demeure un problème complexe pour caractériser des bétons. Composés de nombreuses phases, d'états cristallins parfaits à beaucoup plus défectueux, voire amorphes, les diagrammes DRX montrent alors des superpositions de pics ou de bosses. L'analyse de Rietveld devient elle aussi fastidieuse, pour le choix préalable des phases modèles, résultant en un temps d'analyse important, de plusieurs jours à semaines d'experts pour la détermination précise de la composition.

Dans ces travaux, nous proposons une méthode de deep learning pour régresser les paramètres cristallographiques de chaque phase ainsi que leur proportion, à partir des mesures DRX. Un dataset synthétique de 10 000 échantillons, combinant 10 phases, a été généré grâce au logiciel d'affinement Rietveld MAUD en faisant varier les paramètres de mailles des phases selon les distributions statistiques observées dans la Crystallography Open Database (COD), afin d'assurer un réalisme physique aux variations de données générées.

Nous avons alors entraîné un modèle contenant 4 couches de convolutions et 3 couches linéaires, qui prend en entrée le diagramme DRX et qui régresse les paramètres du matériau. Grâce aux contraintes géométriques des groupes d'espace des phases, un ensemble réduit de 40 paramètres (au lieu de 70) a été régressé pour les 10 phases. Sur nos données de test, nous obtenons une mesure de Rwp moyen d'environ 5% entre les spectres simulés et les spectres obtenus à partir des paramètres régressés.

L'utilisation des méthodes telles que la régression de quantiles et les prédictions conformelles nous permettent de produire des intervalles de prédiction à 90% de confiance pour les paramètres ciblés.

La méthode développée permet donc de régresser les paramètres cristallographiques d'un matériau complexe à partir d'un diagramme DRX avec un temps de calcul réduit par rapport à un affinement Rietveld.

Probing Energy Materials at the Nanoscale: In Situ Bragg Coherent Diffraction Imaging

Dr Marie-Ingrid RICHARD (CEA-Grenoble)

The emergence of 4th generation x-ray light sources offers an unparalleled opportunity for conducting *in situ* and *operando* studies of nanoparticle structures in complex environments, especially in the field of energy materials. Gaining insights into the dynamic strain behavior of catalysts is critical for developing cost-effective, efficient, and long-lasting catalytic systems. In this talk, I will show how Bragg coherent x-ray imaging (BCDI) [1] enables three-dimensional (3D) nanoscale imaging of strain, defect dynamics, and re-faceting processes within nanoparticles during catalytic reactions.

We successfully mapped the 3D lattice displacements and strain distribution of a platinum nanoparticle under electrochemical conditions [2] and during CO oxidation [3,4]. We achieved sub-second time resolution during *operando* chemical reactions, detecting oscillatory strain changes with a 6.4-second periodicity, directly associated with site-specific CO adsorption during oxidation [5], with a benchmark resolution of 0.25 seconds. I will also present our latest findings on the core-shell transition in NiFe catalysts during annealing [6], discuss the potential of measuring particles as small as 20 nm [7] and demonstrate high-energy imaging of embedded materials [8] using BCDI.

We acknowledge funding from ERC under the European Union's Horizon 2020 research and innovation programme and under the European Union's Horizon Europe programme under grant agreement no. 818823 and no. 101201163.

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Ferroelectricity in 3D and 2D Hybrid organic-inorganic perovskites

Elodie TAILLEUR (Université de Lorraine - CRM2)
Aser Javier GARCIA BEAMUD (Université de Lorraine - CRM2)
Fantine MASSICOT (Université de Lorraine - CRM2)
Tanushree CHANGKAKOTI (Université de Lorraine - CRM2)
Emmanuel WENGER (Université de Lorraine - CRM2)
Sébastien PILLET (Université de Lorraine - CRM2)

Hybrid organic–inorganic perovskites (HOIPs) are a versatile class of semiconductors composed of organic cations embedded within an inorganic framework. By varying the size of the organic cations, the dimensionality of the structure can be tuned from three-dimensional (AMX_3) to lower-dimensional systems such as 2D (A_2MX_4), 1D, or 0D. This structural flexibility strongly influences their optical and electronic properties, giving rise to effects such as quantum and dielectric confinement and Rashba splitting. These features make HOIPs highly promising for applications in photovoltaics, optoelectronics, and spintronics.

Some HOIPs crystallize in non-centrosymmetric polar structures and exhibit ferroelectricity, characterized by a switchable spontaneous polarization. This property offers a powerful way to control structural and physical behaviors. While ferroelectricity in 3D HOIPs remains debated, it has been clearly demonstrated in several 2D systems, although its role in optoelectronic and photovoltaic processes is still not fully understood.

In this work, we investigate structure–property relationships in ferroelectric HOIPs using single-crystal optical and structural characterization under various external stimuli (T, hv, E). We first study $MAPbBr_3$, a model 3D HOIP. Combined photoluminescence and X-ray diffraction experiments under electric field and laser excitation reveal the origin of photostrictive and electrostrictive effects [1], highlighting an unprecedented photoelectrostrictive response.

We then turn to 2D ferroelectric HOIPs, focusing on $(BzA)_2PbCl_4$ [2] and its fluorinated derivative $(2\text{-F-BzA})_2PbCl_4$ [3]. Temperature-dependent single-crystal X-ray diffraction is used to identify the structural instabilities driving the paraelectric–ferroelectric phase transition. Symmetry-mode analysis allows us to determine the nature of ferroelectricity by tracking distortion modes as a function of temperature. Ongoing experiments under electric fields, including SHG and photoluminescence measurements, aim to further clarify the ferroelectric mechanisms in these materials.

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Multimodal Operando Characterization of Li-Ion Thin Film Electrodes

Nano KINGUE MOUANGUE (CNRS & IEMN)
Martine TRENTESAUX (UCCS Lille)
Laurent FUGERE (CNRS & IEMN)
Christophe LETHIEN (Université de Lille)
Wan Yu TSAI (Université de Lille)
Jérémie CHAILLOU (CNRS & IEMN)
Pascal ROUSSEL (UCCS Lille)
Lydia KARMAZIN (Institut Michel-Eugène Chevreul)
Pierre-Louis TABERNA (Université de Toulouse)
Patrice SIMON (Université de Toulouse)

Li-ion batteries (LIBs) are known as one of the most common energy storage solutions. Their high energy density and cycling stability make them a reliable power source for portable devices or electric vehicles for example. For the past 50 years, research has led to the discovery of many different electrode materials for LIBs. Among the most significant directions for improvement is rate capability. Next generation LIBs are expected to withstand faster and faster charge (up to 80% charge within 3 minutes).

LiFePO4 (LFP) is often used as a battery cathode material for its availability, low cost and remarkable cycling stability. Its charge (delithiation) reaction: LiFePO4 -> FePO4 + Li+ + e- occurs at 3.4V vs Li/Li+, making it suitable for most battery applications. However the unidimensional diffusion of Li+ ions and the low electronic condutivity make LFP unsuitable for high rate capability applications.

Enhancement of LFP conductivity was explored through N+ implantation. The introduction of defects is expected to increase conductivity in the LFP thin film, improving its rate capability.

In an attempt to better understand the charge storage mechanism and the effect of charge rate, 400nm thin film LFP electrodes were prepared by magnetron sputtering on a transparent substrate. These electrodes were discharged (lithiated) and charged (delithiated) in organic electrolyte (1M LiClO4 in EC/DMC 1/1) and analysed by multimodal operando characterization at different charge rates.

The combination of operando X-ray diffraction (XRD), UV/vis/NIR spectroscopy and Raman provides original insight into the charge storage mechanism in LFP thin films. XRD and Raman spectroscopy allow us to track thin film structure, whilst UV/vis/NIR spectroscopy shows the optical properties of the (de)lithiated thin films as well as the band gap energy.

In addition to understanding charge storage mechanisms, the development of thin film electrodes can be useful for miniaturization of energy storage...

Transition de phase structurale induite par un champ magnétique à l'origine de la magnétostriction macroscopique dans le terfenol-D

M Jerome ROUQUETTE (Institut Charles Gerhardt)

Le couplage entre champs externes et transitions de phase structurales dans les matériaux fonctionnels est à l'origine de nombreux phénomènes et applications, de la commutation ferroélectrique à l'actionnement magnétostrictif. Les alliages à magnétostriction géante comme le Terfenol-D ($Tb_{0.3}Dy_{0.7}Fe_2$) constituent des systèmes modèles pour étudier l'interaction entre ordre magnétique, symétrie cristalline et déformations macroscopiques. Grâce à sa magnétostriction exceptionnelle à température ambiante, ce matériau est largement utilisé dans les actionneurs, capteurs et transducteurs.

Ses propriétés remarquables sont liées à sa proximité avec une frontière morphotropique (MPB), séparant des phases ferromagnétiques de symétries et d'axes de facile aimantation distincts : une phase rhomboédrique (<111>) et une phase tétragonale (<001>). L'alliage de terbium et de dysprosium, aux anisotropies opposées, conduit à une compensation d'anisotropie et à un paysage énergétique aplati près de la MPB, favorisant la mobilité des parois de domaines et la rotation de l'aimantation.

Des effets similaires sont bien établis dans les ferroélectriques comme le PZT, où des transitions de phase induites par champ électrique amplifient la réponse piézoélectrique. Par analogie, un mécanisme comparable a été suggéré pour les matériaux magnétostrictifs, sans preuve structurale directe jusqu'à présent.

Nous montrons ici, grâce à des expériences de diffraction des rayons X synchrotron *in situ* réalisées en fonction du champ magnétique (jusqu'à 0,55 T) et de la température (200–320 K) sur la ligne ID22 de l'ESRF, l'existence d'une transition de phase structurale induite par champ dans le Terfenol-D. Cette transition réversible est directement corrélée à l'apparition de la magnétostriction macroscopique. Ces résultats apportent un éclairage nouveau sur les mécanismes en jeu et ouvrent la voie à une ingénierie des phases induites par champ dans les matériaux ferroïques.

Unveiling a Charge Density Wave in the m = 2 Monophosphate Tungsten Bronze: A Comprehensive Multitechnique Approach to Structure and Physical Properties

Olivier PEREZ (Cnrs)
Arianna MINELLI (Neutron Scattering Division, Oak Ridge National Laboratory)
Alain PAUTRAT (Cnrs / CRISMAT)
Marc DE BOISSIEU (SIMAP / CNRS)
Alexei BOSAK (ESRF)
Marek MIHALKOVIC (Institute of Physics, Slovak Academy of Sciences,)
Johnathan BULLED (ESRF)
Adrien GIRARD (Sorbonne Université, CNRS)
Elen DUVERGER-NEDELLEC (Univ. Bordeaux, CNRS)

Charge density waves (CDWs) are collective electronic states resulting from the interplay between lattice distortions and electronic instabilities in low-dimensional materials. Within the monophosphate tungsten bronze family, the m = 2 member had long been regarded as the only compound lacking a low-temperature electronic instability. In this work, we report the discovery and comprehensive characterization of a previously unidentified CDW phase in this material, with a transition temperature close to 290 K and a lock-in transition to a commensurate state near 130 K.

Our study emphasizes the strength of a multitechnique approach combining single-crystal X-ray diffraction, temperature-dependent, diffuse and inelastic X-ray scattering, electrical transport measurements, and ab initio molecular dynamics simulations. Diffraction experiments reveal the formation of satellite reflections associated with an incommensurate structural modulation, while diffuse and inelastic scattering measurements evidence phonon softening and a clear Kohn anomaly, highlighting the dynamical precursor of the transition. Complementary electrical transport measurements demonstrate non-linear conductivity and depinning behavior, confirming the collective nature of the electronic state.

Beyond the identification of the CDW phase, this combined structural and physical investigation provides key insight into the mechanism driving the transition. Structural refinements and simulations indicate that the modulation is dominated by cooperative rotations of WO_6 octahedra and tilting of phosphate units, consistent with an order–disorder scenario rather than a purely Peierls-type instability.

Overall, this work illustrates how integrating advanced crystallographic techniques with physical property measurements enables a unified understanding of charge density wave formation in low-dimensional oxides.

New MCuFe2O5 (M = Mn and Co) High-Pressure Oxides

Angel AREVALO-LOPEZ (CNRS UCCS Université de Lille)
Leelashree SOLAIAPPAN (CNRS UCCS Université de Lille)
Olivier MENTRÉ (CNRS UCCS Université de Lille)
Clemens RITTER (ILL, Grenoble)

$CaFe^{2+}Fe_2^{3+}O_5$ stabilizes a long-range collinear magnetic order below ~280 K. However, high-pressure and high-temperature (HPHT) synthesis allows Fe^{2+} to be substituted by Jahn–Teller active Cu^{2+} likewise Ca^{2+} by Mn or Co, giving rise to $CaCuFe_2O_5$, $MnFe_3O_5$ and $CoFe_3O_5$ with diverse magnetic ground states. Building on these concepts, we combined (i) $Fe^{2+} \rightarrow Cu^{2+}$ substitution and (ii) Ca → Mn/Co substitution and successfully synthesized the novel $MCuFe_2O_5$ (M = Mn, Co) oxides at 10–20 GPa and 1273 K using a two stage press with a Multi-Anvil Walker-type module.

Intriguingly, $MnCuFe_2O_5$ adopts a distinct orthorhombic *Pnma* structure, in contrast to $CoCuFe_2O_5$, which retains the *Cmcm* aristotype structure. This structural divergence originates from differences in cation coordination, particularly involving Cu and Fe polyhedra, reflecting the strong influence of Jahn–Teller distortions and cation size effects. Magnetic susceptibility measurements reveal two successive transitions in both compounds, at T_{N1} = 195 (105) K and T_{N2} = 77 (39) K for Co (Mn), respectively. Above the ordering temperatures, Curie–Weiss analysis yields large negative Weiss constants θ = -700 (-218) K and enhanced effective moments μ_{eff} = 5.1 (6.6) μ_B/f.u., exceeding spin-only values and indicating strong AFM exchange interactions along with significant spin–orbit coupling contributions, particularly from Co^{2+}.

Field-dependent magnetization at 2 K confirms ferrimagnetic behavior in both systems, with evidence of spin canting and competing magnetic interactions. In $CoCuFe_2O_5$, the interplay between Fe–Fe, Fe–Co, and Co–Cu exchange pathways, combined with antisite disorder and strong spin–orbit coupling, stabilizes a canted ferrimagnetic ground state with notable magnetic anisotropy and high coercivity. These results will be discussed in the context of structure–property relationships in HPHT oxides, highlighting how targeted cation substitution and local distortions can be exploited to tune complex magnetic ground states.

High pressure macromolecular crystallography revealed equilibria between conformational states of the Ras oncogene protein

Nathalie COLLOC'H (CNRS UMR6030-ISTCT)

Ras protein is a small GTPase involved in central regulatory processes and acts as a molecular switch between active GTP-bound and inactive GDP-bound states. These regulatory processes occur in multiple conformational states. An allosteric network between the effector binding regions and the membrane interacting regions is involved in Ras cycling. The conformational states which coexist simultaneously in solution with low occupancies possess higher Gibbs free energy than the ground state. Equilibria between these states can be shifted by applying high hydrostatic pressure favoring conformations with lower molar partial volumes.

The allosteric transitions between conformational states of Ras have been investigated using high pressure crystallography, which is a powerful tool to characterize at the molecular level the different high-energy states.

We have determined at high resolution the crystallographic structures of Ras(wt).Mg2+.GppNHp at pressures up to 900 MPa. We have observed a transition above 300 MPa in the crystal leading to more stable conformers. The comparison of these different structures gives insight to per-residue descriptions of the structural plasticity involved in allosteric equilibria between conformers. The different segments of Ras protein which remains in the ground-state conformation or undergo structural changes, adopting excited-energy conformations corresponding to transient states, have been mapped out at atomic resolution.

Such in-crystallo phase transitions induced by pressure opens the possibility to finely explore the structural determinants related to switching between Ras allosteric sub-states without any mutations nor exogenous partners.

Charge Density Waves Tuned by Biaxial Tensile Stress

Vincent JACQUES (Laboratoire de Physique des Solides - CNRS/Université Paris Saclay)
Antoine GALLO--FRANTZ (Institut Néel - CNRS)
Luc ORTEGA (Laboratoire de Physique des Solides - CNRS/Université Paris Saclay)
David LE BOLLOC'H (Laboratoire de Physique des Solides - CNRS/Université Paris Saclay)
Abdellali HADJ-AZZEM (Institut Néel - CNRS)
Pierre MONCEAU (Institut Néel - CNRS)
Aleksandr SINCHENKO (Laboratoire de Physique des Solides - CNRS/Université Paris Saclay)

Two-dimensional correlated systems display a broad range of competing or coexisting electronic and magnetic phases, such as superconductivity, charge density wave (CDW), Mott insulating states and many others, as a function of temperature, pressure, or doping. In the last years, strain has been more and more used as an external parameter, applied either along one or two axis of crystals (uniaxial or biaxial strain), in tensile and/or compressive way, to induce new electronic states or tune the existing ones in 2D systems [1]. We have recently built a new cryogenic biaxial tensile stress device compatible with structural and electronic probes (XRD, electronic transport, optical spectroscopy) that works in a wide range of temperatures (15-400K), to explore new electronic states of 2D systems under biaxial tensile strain, up to few percents [2]. Here, we will show how such a multi-technique approach allowed us to unambiguously determine that the in-plane crystal assymetry drives a CDW reorientational transition in the nearly-tetragonal $TbTe_3$, with huge transition temperatures variations (more that 30K) under strain [3]. Besides, a coexistence phase is found with two orthogonal CDW forming micrometer-size domains. In a second part, we will show how the two CDWs of the quasi-1D $NbSe_3$ are affected by uniaxial strains applied either along or perpendicular to the chains directions, and affect the transition temperatures in a different way, suggesting very different characteristics for the high- and low-temperature CDWs [4]. Finally, we will show how strain affects the non-linear dynamics of the CDWs of $TbTe_3$ and $NbSe_3$.

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Tuning the structural and optoelectronic properties of metal halide perovskites by hydrostatic pressure

Sébastien PILLET (Laboratoire CRM2 - Université de Lorraine)
Maxime DEUTSCH (Laboratoire CRM2 - Université de Lorraine)
Ashanti BERGONZONI (Institut PHOTON, INSA Rennes)
Jacky EVEN (Institut PHOTON, INSA Rennes)
Samuel MATHIEU (Laboratoire CRM2 - Université de Lorraine)
Mathieu STOFFEL (Institut Jean Lamour)
Wouter VAN GOMPEL (Hybrid Materials Design - MATCHEM)
Arthur MAUFORT (Hybrid Materials Design - MATCHEM)

Pressure is a fundamental thermodynamic variable that governs the state and properties of materials. Changes in pressure can alter bond lengths and angles and may even induce transitions between different crystallographic phases. Hybrid metal halide perovskites are relatively soft, making their structures and properties especially sensitive to pressure variations. Structural distortions induced by pressure, such as octahedral tilting, bond compression, or symmetry changes, can significantly modify their electronic structure. In particular, pressure can tune the band structure and band alignment by altering orbital overlap and the relative positions of the conduction and valence bands. Moreover, pressure-induced structural asymmetry can influence spin–orbit coupling related phenomena, such as the Rashba effect, by modifying local inversion symmetry. Several relevant examples of pressure induced control of optoelectronic properties will be presented in the poster.

A ‘Swiss-Knife’ laboratory diffractometer for Simultaneous Multi-Techniques and Multi-Stimuli Studies of Functional Materials

Emmanuel WENGER (Université de Lorraine, CNRS, CRM2, 54000 Nancy, France)
Elodie TAILLEUR (Université de Lorraine, CNRS, CRM2, 54000 Nancy, France)
Sébastien PILLET (Université de Lorraine, CNRS, CRM2, 54000 Nancy, France)
Dominik SCHANIEL (Université de Lorraine, CNRS, CRM2, 54000 Nancy, France)

Crystallography of functional materials aims to explain the observed functional properties based on the underlying structural features, ideally under 'in situ' or 'operando' conditions. We present a laboratory single-crystal X-ray diffraction (XRD) setup designed for the acquisition of diffraction data under static stimuli (temperature, pressure) as well as time-resolved conditions (light excitation, electric field).

In addition, the setup enables 'in situ' measurements of optical properties (luminescence and absorption) under continuous-wave (cw) or pulsed laser excitation of the sample. While the temporal resolution of the XRD experiments lies in the millisecond range, the optical measurements can reach nanosecond time resolution, enabling simultaneous multi-techniques characterization of time-dependent phenomena. This capability allows the investigation of a wide range of stimulus-induced processes and facilitates the establishment of structure–property relationships.

The poster will present the measurement principles, the technological implementation, and key metrological considerations of the instrument. Two distinct research areas are illustrated using this original experimental platform: time-resolved photocrystallography of photoswitchable compounds, and single-crystal XRD experiments under electric field applied to ferroelectric materials. Representative examples from each field will be discussed.

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Serial Electron Diffraction – Targeting Nanocrystals

Tatiana GORELIK (Ernst Ruska-Centre for Microscopy and Spectroscopy with Electrons, Forschungszentrum Jülich)
Dieter WEBER (Ernst Ruska-Centre for Microscopy and Spectroscopy with Electrons, Forschungszentrum Jülich)
Karina RUZAEVA (Institute for Advanced Simulations – Materials Data Science and Informatics (IAS-9), Forschungszentrum Jülich)
Brendan CLARKE (TESCAN GROUP)
Stefan SANDFELD (Institute for Advanced Simulations – Materials Data Science and Informatics (IAS-9), Forschungszentrum Jülich)
Rafal DUNIN-BORKOWSKI (Ernst Ruska-Centre for Microscopy and Spectroscopy with Electrons, Forschungszentrum Jülich)

Electron diffraction, with its ability to probe minute sample volumes, enables crystal-structure determination from micro- and nanoscale crystals. The current lower size limit for 3D electron diffraction is governed by goniometer stability and the beam sensitivity of the crystals. We investigate whether this limit can be extended using serial electron diffraction—analogous to XFEL experiments.

In serial crystallography, a single diffraction pattern is recorded from each individual crystal. Although information on the relative orientations of patterns is lost, this approach greatly simplifies automation and allows acquisition of very large numbers of patterns. Crystal decay under the probe is also mitigated, since each pattern originates from a fresh, previously unexposed crystal.

The serial data-processing workflow comprises unit-cell determination, indexing of individual patterns, and merging of intensities into a consolidated dataset. Because of the short electron wavelength, the Ewald sphere in electron-diffraction experiments is extremely flat, posing a geometric challenge for unit-cell determination from randomly oriented patterns.

This presentation outlines possible solutions for unit-cell determination from serial ED data and demonstrates ab initio crystal-structure determination across several material classes.

Structural study by Transmission Electron Microscopy of bulk calcium-doped neodymium Nickelates

Christophe LEPOITTEVIN (Université Grenoble-Alpes)
Nathan BASSOT (Institut Néel)
Pierre TOULEMONDE (Université Grenoble-Alpes)
Céline DARIE (Polytech Grenoble)

In 2019, the group of H. Y. Hwang reported the first observation of superconductivity in a thin film of the two-dimensional nickelate $Nd_{0.8}Sr_{0.2}NiO_2$, with a critical temperature $T_c \approx 15$ K. This compound adopts the so-called “infinite-layer” structure, characterized by NiO_2 planes analogous to the CuO_2 planes found in high Tc cuprates, which has sparked considerable interest within the scientific community [1]. Subsequently, the chemical doping range was expanded to $Ln_{1-x}A_xNiO_2$ phases (Ln = La, Pr, Nd, Eu; A = Sr, Ca [2-4]), with superconductivity emerging over the doping range $0.1 \leq x \leq 0.3$.

By tuning the nickel valency through partial substitution of Nd^{3+} with Ca^{2+}, the polycrystalline solid solution $Nd_{1-x}Ca_xNiO_2$ ($0 \leq x \leq 1$) was fully synthesized and characterized. In this context, Transmission Electron Microscopy (TEM) was employed as a primary tool to investigate the structure at the atomic scale.

For $0 \leq x < 0.6$, the targeted “infinite layer” phase was successfully stabilized. However, low temperature physical measurements revealed no evidence of superconductivity, likely due to the high density of structural defects within the crystallites, including twinning, nanoscale domains, and oxygen vacancies. High Angle Annular Dark Field - Scanning Transmission Electron Microscopy (HAADF-STEM) enabled direct visualization of these defects in the samples.

For $x = 0$ and $x > 0.6$, two new phases, the monoclinic $NdNiO_{2.33}$ and rhombohedral $Ca_{3-d}Nd_dNi_2O_6$, were isolated, and their crystal structures were determined by 3D electron diffraction. Stepwise precession electron diffraction datasets were collected for both compounds. Data reduction was carried out using PETS2 [5], and structure models were calculated via charge flipping using Superflip [6] implemented in JANA2020 [7]. The resulting models were subsequently refined using dynamical diffraction theory and systematically validated against HAADF-STEM imaging.

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3D-Electron Diffraction unveils new melanotekite-based intergrowth series (Pb2FeO3)(Pb2Ge2Fe2O9)n

Antonin RAYNAL (Univ. Grenoble Alpes, CNRS, Grenoble INP, Institut Néel)
Stephanie KODJIKIAN (Univ. Grenoble Alpes, CNRS, Grenoble INP, Institut Néel)
Christophe LEPOITTEVIN (Univ. Grenoble Alpes, CNRS, Grenoble INP, Institut Néel)
Fabio DENIS-ROMERO (Univ. Grenoble Alpes, CNRS, Grenoble INP, Institut Néel)

Transmission electron microscopy is a highly efficient characterization tool, as it enables local structural information to be combined with chemical composition information.

The exploration of the PbO-GeO2-Fe2O3 ternary diagram evidenced a new phase Pb10Ge8Fe9O39, and its crystal structure was solved using Single-Crystal XRD (SCXRD), leading to cell parameters a=27,1018 Å, b=10,1980 Å, c=13,2843 Å, β=92,320° in the P21/c space group. However, zone axis selected area electron diffraction patterns showed diffuse scattering along the a* direction, whose origin was highlighted by High Angle Annular Dark Field STEM imaging: The atomic scale Z-contrast images revealed a disorder consisting of three related unit cells, differing in a and β parameters, alternating randomly in the form of intergrowth defects.

A 1° stepwise precession electron diffraction dataset was recorded on a particular zone of the sample that presented a majority of the smallest unit cell. Then 3D-Electron Diffraction (3D-ED) software and methods were used to solve and refine its structure. Its cell parameters are a=21,4479 Å, β=82.232° and its chemical formula is Pb8Ge6Fe8O30.

It has been possible to extrapolate these three observed structures to a larger family of intergrowth consisting of the alternation of one Pb2FeO3 layer with n Pb2Fe2Ge2O9 melanotekite-type layers. A general structural model valid for all members (Pb2FeO3)(Pb2Ge2Fe2O9)n (n>0) was devised, and it turns out that the members n=3 and n=4 were the structures solved by SCXRD and 3D ED respectively. The 3rd structure, corresponding to the member n=5, of parameters a=33,1139 Å and β=98,531°, was computed using VESTA. It was then refined against a 3D-ED dataset recorded on particles containing reflections of all three structures.

Acknowledgements: The TEM facility JEOL NEOARM at CNRS Institut Néel was co-financed by the European Union under the European Regional Development Fund (ERDF, contract no. RA0023813).

Diffraction d’électrons pour l’industrie pharmaceutique

M EMRE YÖRÜK (Holodiag / ICROM)
Pierric MARCHAND (Holodiag / ICROM)

La diffraction d'électrons 3D (3D ED) (Gemmi et al., 2019) devient aujourd'hui particulièrement pertinente pour la caractérisation structurale de composés pharmaceutiques inaccessibles aux méthodes conventionnelles en rayons X. La résolution de structures cristallines, identification des polymorphes et la détermination de structures absolues est possible à partir de particules sous forme de micro- ou nanocristaux dans une poudre, supprimant la nécessité de croitre de cristaux. Le développement des diffractomètres à électrons dédiées (Simoncic et al., 2023) permet un débit expérimental élevé, en particulier grâce à l'automatisation de l'acquisition de données.
On présente ici les récents cas d'application de la 3D ED sur des composés d'intérêt pharmaceutique et des petites molécules où la caractérisation par la diffraction des rayons X n'était pas possible. L'impact important de l'optimisation des conditions d'irradiation est démontré (Yörük and Naumov, 2025). La prise en compte de la diffusion multiple des électrons, l'impact de la mosaïcité et de la morphologie des cristaux sur les intensités de diffraction permet des améliorations significatives de la précision des affinement structuraux.

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3D ED platform at IBS: Examples of results in structural biology and material sciences and future developments

Christine MORISCOT (ISBG & IBS - UMR 5075 UGA, CNRS, CEA)
Eric GIRARD (IBS - UMR 5075 UGA, CNRS, CEA)
Dominique HOUSSET (IBS - UMR 5075 UGA, CNRS, CEA)

Electron diffraction of 3D nanometer sized crystals, most commonly named microED or 3D ED, has recently emerged as a new technique to solve the structure of materials, small organic molecules and proteins [1,2]. Electrons are actually a very interesting probe for small samples as they strongly interact with matter, and more importantly, they deposit much less energy than X-rays per diffracted particle. 3D ED is clearly a promising technique as it can provide complete 3D diffraction data out of a few nanometer sized crystals. Sub-atomic resolution data can routinely be collected for small organic compounds and data up to 0.9 Å resolution have been obtained from lysozyme crystals [3]. The IBS 3D ED platform includes a side entry 200 kV Tecnai F20 electron microscope, a hybrid pixel detector from ASI and room temperature and cryo-holders, enabling data collection on hydrated materials and protein crystals. A few examples of electron diffraction data recently collected and published on materials and protein crystals will be shown. Specificities of 3D ED on data processing, phasing and refinement will be presented. A special focus will made on the preparation of protein nanocrystal, with two possible approaches. One uses cryo-sectioning after high pressure freezing of dextran embedded lysozyme crystals [4]. The second approach used minute made lysozyme nano-crystals in the presence of Tb-Xo4 [5]. Future developments such as the implementation of serial ED, the use of FIB milled lamella, improved nano-crystal detection or the use of a Gata K2 detector on a 200 kV Glacios will be discussed.

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3D ED for Nanoscale Structural Analysis

Philippe BOULLAY (Laboratoire de Cristallographie et Sciences des Materiaux (CRISMAT))
Erica CORDERO OYONARTE (Now at Department of Fundamental Microbiology (DMF), Faculty of Biology and Medicine)
Sara PASSUTI (Now at Laboratory of Electron Microscopy (LAME), AREA Science Park)

Accurate determination of crystalline structures is essential for understanding material properties and guiding the rational design of new compounds. However, X-ray diffraction, while remaining the reference technique, has limitations for studying nanomaterials due to their small size. In this context, 3D electron diffraction (3D ED) [1] has become an indispensable method for nanoscale structural analysis.

Once considered a major obstacle for reliable quantitative analysis, multiple scattering is now accounted for in dynamical refinements, a breakthrough that has revolutionized this field over the past decade. These refinements significantly improve the consistency of results [2] and enable the resolution of fine structural details, such as the localization of light atoms (H, Li), anisotropic atomic displacement parameters, and mixed site occupancies. Our recent work has demonstrated that such precise analysis is achievable for crystals as small as 10 nm [3], provided that experimental conditions such as beam size, cryogenic temperatures, and the use of high-sensitivity detectors are optimized.

The analysis of nanodomains in thin films, ceramics, or minerals is complicated by their microstructure, particularly the presence of adjacent crystalline domains. Innovative strategies, such as Scanning Precession Electron Diffraction Tomography (SPET) [4] or 4D-STEM Tomography [5], enable the simultaneous acquisition of high-quality 3D ED data from multiple regions of interest. By using parallel beams of a few tens of nanometers or less, these approaches reveal subtle structural variations at the nanoscale in complex functional materials.

Acknowledgments

The authors acknowledge funding from the European Union's Horizon 2020 research and innovation program under the Marie Skłodowska-Curie grant agreement No. 956099 (NanED – Electron Nanocrystallography – H2020-MSCA-ITN).

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Developing Devices and Approaches for Comparing Gas Absorption across Laboratory X-ray and Electron Diffraction Techniques.

Eleanor KEIL (The University of Southampton)
Graham TIZZARD (National Crystallography Service, UK)
Daniel RAINER (National Electron Diffraction Facility, UK)
Simon COLES (The University of Southampton)
Mark LIGHT (The University of Southampton)

Producing a carbon capture material has been the aim for many crystallographers in the battle to slow global warming. The *in situ* study of Carbon Dioxide absorption in Metal-Organic Frameworks largely occurs via neutron and X-ray powder diffraction, or via single crystal X-ray diffraction at specialised synchrotron facilities. As access to Rigaku Synergy-ED devices is becoming increasingly frequent, developing replicable procedures and understanding the limitations of *in situ* gas experiments, using electron diffraction, is vital.

An adaptable gas cell was successfully created and tested for use on 'in-house' commercial single crystal X-ray diffractometers. The project improves upon previous gas cell designs from The University of Southampton and Diamond Light Source, UK, [1] using established CALF-20 findings as a foothold into the study of Metal-Organic Framework absorption [2]. This X-ray diffraction hardware has become a benchmark for comparative testing towards the Hummingbird gas cell holder used for electron diffraction gas experiments at the National Electron Diffraction Facility, UK.

Consequent work will study the absorption capabilities of CALF-20 and some analogous crystals before venturing into the study of unknown absorption of Metal-Organic Frameworks, accelerating the study of Metal-Organic Frameworks as a tool against global warming. Eventually, definitive comparisons will describe the pitfalls of this static, single crystal X-ray diffraction gas cell against those of the dynamic flow electron diffraction gas cell and nanocrystals of the same species.

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A challenging Pb-Sr-Fe-oxide structure solved by 3D Electron Diffraction

Christophe LEPOITTEVIN (Université Grenoble-Alpes)

Strontium ferrites and cobaltites, $(Sr_{4-x}Ca_x)(Fe_{6-y}Co_y)O_{13\pm\delta}$, exhibit mixed ionic–electronic conductivity and thermoelectric properties, both strongly affected by oxygen non-stoichiometry [1-2]. The incommensurately modulated structure of $Sr_4Fe_6O_{13\pm\delta}$ has been accurately determined using single-crystal X-ray diffraction within a 4D formalism [3].
To induce structural distortions, Sr^{2+} was partially substituted with isovalent Pb^{2+}. Although similar in ionic radi-us, Pb^{2+} introduces significant distortions due to its stereochemically active $6s^2$ lone pair. This substitution led to the discovery of a new phase in 2006, for which only a preliminary structural model could be proposed because of its complexity [4].
Recent advances in 3D electron diffraction, combined with improved microscope performance, now enable the solution of such complex structures. This motivated a reinvestigation of the unresolved phase.
A dataset from −60° to +60° was collected and processed, and the structure was solved using charge flipping and refined through both kinematical and dynamical approaches. The compound crystallizes in a monoclinic C2/c unit cell (a = 34.782(8) Å, b = 5.522(2) Å, c = 24.962(7) Å, β = 99.11(2)°). The final model shows excellent agreement with HAADF STEM imaging.

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Plateforme Française 3D - DE

Lydia KARMAZIN (CNRS, FR 2638-IMEC-Institut Michel-Eugene Chevreul, Université de Lille)
Jean-François LOHIER (CRISMAT, CNRS, Normandie University, ENSICAEN, UNICAEN)
Olivier PEREZ (CRISMAT, CNRS, Normandie University, ENSICAEN, UNICAEN)
Pascal ROUSSEL (Unité de Catalyse et Chimie du Solide - CNRS UMR 8181)

La diffraction électronique en mode 3D (3D-DE) est une technique de diffraction à partir d'électrons (méthode du cristal tournant) permettant l'acquisition de données de diffraction exploitables pour la détermination structurale à partir de cristaux de taille sub-micronique. Les données permettent de déterminer la structure cristalline ; leur affinement, en fonction de la précision souhaitée et de la nature des cristaux, se fera en mode cinématique ou dynamique. Le développement récent de diffractomètres à électrons permet de démocratiser son utilisation et de l'ouvrir au plus grand nombre

U³ Unveiling the Unseen and Undetectable: Automated Electron Diffraction and its growing role in solid state characterization

Dr GUSTAVO SANTISO QUINONES (Eldico Scientific AG)
Gunther STEINFELD (Eldico Scientific AG)
Giorgia FIORINI (Eldico Scientific AG)
Danny STAM (Eldico Scientific AG)
Doris BRAUN (University of Innsbruck)

Following its Science Breakthrough of the Year nomination in 2018,[1] electron diffraction (3D ED / MicroED, here referred to as ED) has emerged as a transformative method for solid-state characterization in both research and industry.[2] ED addresses many of the same application areas as X-ray powder diffraction (XRPD) and single-crystal X-ray diffraction (SC-XRD), while offering a key advantage: the ability to determine structures from nanometer-sized crystals within minutes. The rapid growth[3] in structures solved by ED is driven in part by the availability of dedicated commercial instrumentation, such as the ELDICO ED-1.[4] Its instrument design and expanded sample environment enable automation, making ED increasingly accessible to non-specialist users.

Automation in ED is not merely a convenience; it is a discovery engine. By systematically screening large numbers of nanosized crystals without user intervention, automated ED workflows can reveal solid-state forms that would remain undetected, or simply be missed, in a manual measurement campaign. This will be exemplified through three cases involving pharmaceutically relevant molecules. First, a new co-crystal form discovered in a well-characterized sample, where its presence had gone unnoticed by prior analyses. Second, the detection of unexpected crystallinity in a sample considered amorphous, in which complementary techniques had failed to reveal any crystalline content. Third, the discovery of a previously unknown polymorphic form of dapsone: despite over a century of study and five known polymorphs, automated ED identified a sixth form in a commercial sample — one not anticipated by crystal structure prediction (CSP) studies limited to $Z' = 1$ and 2, yet subsequently located within the computed energy landscape and independently confirmed by complementary experiments following isolation via slurry experiments.[5] Together, these examples highlight the power of automated ED to unveil the unseen and undetectable in solid-state chemistry.

Alpibectir/Ethionamide: The Long and Winding Road to a New Therapeutic Concept

Alain BAULARD (Inserm)

Mycobacterium tuberculosis possesses a remarkable capacity to modulate its own antibiotic susceptibility through intrinsic transcriptional regulatory networks. Ethionamide, a thioamide prodrug central to multidrug-resistant tuberculosis regimens, is a striking illustration: its bactericidal activity is contingent upon intramycobacterial bioactivation by the monooxygenase EthA, itself tightly controlled by the TetR-family repressor EthR. Deciphering this regulatory bottleneck launched a research program that has now culminated in the successful completion of a Phase IIa clinical trial in South Africa with alpibectir.

Alpibectir embodies a first-in-class mechanism of action, functioning as a molecular Trojan horse that hijacks the bacterium's own transcriptional circuitry. By derepressing alternative bioactivation pathways, it renders M. tuberculosis hypersusceptible to ethionamide at dramatically reduced doses, including in clinical isolates harbouring defined resistance determinants. This presentation will retrace the path from that clinical milestone back to the original conceptual discovery. It will describe how the comparative analysis of two independent EthR crystal structures, both serendipitously trapped in ligand-bound conformations, led to a pharmacophoric model that launched the structure-guided search for synthetic EthR inhibitors. Major scientific turning points will be highlighted, from the genetic dissection of ethionamide bioactivation to the iterative medicinal chemistry campaigns that ultimately produced alpibectir. The central role of structural biology throughout this journey will be emphasised, alongside several pivotal moments of serendipity that redirected the programme. More broadly, this talk will illustrate how sustained multidisciplinary collaboration spanning molecular genetics, medicinal chemistry, biophysics and pharmacology, combined with public/private partnerships, helped transform a mechanistic insight once dismissed as utopian into a validated therapeutic strategy now brought to the clinic.

Résolution de la structure de cocristaux d'intérêts pharmaceutiques et étude des relations structure-propriétés

Mathieu GUERAIN (UMET)
Frédéric AFFOUARD (UMET)
Hubert CHEVREAU (SOLEIL)
Natalia CORREIA (UMET)
Florence DANEDE (UMET)
Catherine DEJOIE (ESRF)
Yannick GUINET (UMET)
Alain HÉDOUX (UMET)
Laurent PACCOU (UMET)

La formation d'un assemblage moléculaire rassemblant dans la même maille cristallographique le principe actif lui-même et une molécule n'ayant pas d'effets délétères est une méthode permettant d'améliorer la biodisponibilité de certaines molécules pharmaceutiques. L'ensemble cristallographique formé est appelé cocristal, et présente les caractéristiques suivantes :

-Structure cristalline composée de deux molécules ou plus, avec un ratio stœchiométrique

-Composants purs sous forme solide à pression et température ambiante

-Pas de transferts de charge entre les composants au sein de la maille cristallographique

La cocristallisation de principes actifs permet donc d'accéder à de nouvelles formes cristallographiques, et donc non seulement d'améliorer leurs propriétés thérapeutiques, mais aussi de mieux comprendre les interactions qui régissent les molécules.

Nous présenterons ici les travaux réalisés sur des cocristaux impliquant des molécules d'intérêts pharmaceutiques (Ibuprofène, Caféine, Naproxène, Carbamazépine). En particulier, nous discuterons de l'influence de la méthode de synthèse sur la structure cristallographique formée menant à la possibilité d'obtenir plusieurs formes polymorphiques d'un même cocristal, de la détermination du domaine de stabilité de ces différentes formes, et enfin de l'influence de la structure cristallographique obtenue sur certaines propriétés du composé.

Pour cela, une connaissance fine des structures cristallographiques obtenues et des liaisons hydrogènes formées est nécessaire. Ces structures comportant souvent un grand nombre de molécules par mailles, elles sont en conséquence particulièrement délicates à résoudre. C'est pourquoi l'apport des lignes synchrotron de haute résolution (SOLEIL, ESRF) a été indispensable pour ces travaux, de même que les calculs de dynamique moléculaire.

Neutron Crystallography Study of Host–Pathogen Recognition Enhanced by Hydrogen/Deuterium Exchange on Carbohydrates

Theodore ARNAUD (Institut Laue-Langevin & CERMAV)
Chloe TATOL (Service de Chimie Bio-organique et Marquage, DMTS, CEA, Université Paris-Saclay)
Juliette DEVOS (Institut Laue-Langevin)
Lukas GAJDOS (Institut Laue-Langevin)
Sébastien VIDAL (Institut de chimie des substance naturelles)
Marek KORSAK (National centre for biomolecular research, faculty of science, Marsaryk university)
Emeline RICHARD-MILLOT (CERMAV)
Annabelle VARROT (CERMAV)
Michaela WIMMEROVA (National centre for biomolecular research, faculty of science, Marsaryk university)
Sophie FEUILLASTRE (Service de Chimie Bio-organique et Marquage, DMTS, CEA, Université Paris-Saclay)
Matthew BLAKELEY (Institut Laue-Langevin)
Anne IMBERTY (CERMAV)

Neutron macromolecular crystallography (NMX) is a powerful structural biology technique that allows the direct visualization of hydrogen atoms and protons, thereby revealing details of hydrogen-bonding, protonation and hydration in biomolecular systems. Visualization is enhanced when hydrogen atoms are replaced by deuterium through isotopic labeling strategies.

Many human glycans serve as targets for virulence factors produced by pathogens. Among these factors, lectins are carbohydrate-binding proteins involved in host–pathogen recognition that play a central role in bacterial adhesion, tissue colonization, and biofilm formation. The opportunistic pathogen Pseudomonas aeruginosa, a major cause of hospital-acquired infections and a critical antibiotic-resistant bacterium, produces two soluble lectins, LecA and LecB, which specifically recognize galactose and fucose, respectively. LecA acts as a membrane disruptor by specific binding to the oligosaccharide head group of globoside, a sphingolipid consisting of ceramide with a Gal(α1-3)Gal(β1-4)Glc glycan part. LecA emerged as a promising target for the development of anti-virulence therapeutics and thus precise structural data are needed for the design of glycomimetics that act as “pathoblockers”.

In this work, we present several deuteration strategies and crystallization approaches developed to investigate the interaction between the lectin LecA and carbohydrate ligands using NMX. These approaches include the production of perdeuterated protein, the preparation of deuterated carbohydrates (galactose, and galactose containing oligosaccharides and analogs) and the optimization of crystallization conditions to obtain large-volume crystals suitable for neutron diffraction experiments.

The neutron structures of perdeuterated LecA complexed with several deuterated ligands shed light on the importance of histidine protonation states in the active site, the water-mediated interactions, and hydrogen-bond networks within the LecA binding site. These structural insights significantly improve our understanding of lectin–carbohydrate recognition and provide valuable guidance for the rational design of novel anti-adhesive compounds targeting P. aeruginosa infections.

This presentation will be based on our recent publication (DOI: 10.1002/ceur.202500424).

Mechanism and Kinetics of Milling-Induced Polymorphic Transformation in Mefenamic Acid

Mme Hornela YEMENE TAGOUE (Université de Lille)
Florence DANÈDE (Université de Lille)
Mathieu GUERAIN (Université de Lille)
Juergen SIEPMANN (Université de Lille)
Jean-Francois WILLART (Université de Lille)

Mechanical milling is frequently employed in industrial processes to reduce the particle size of powders. However, it can also induce changes in the physical state that may alter the functional properties of the material. This is particularly critical in the pharmaceutical field, where a change in the state of an active ingredient can modify its stability, solubility, and bioavailability—and consequently, its efficacy. Despite their crucial importance, these milling-induced transformations remain poorly understood and, therefore, difficult to control.

We present herein a detailed kinetic study of the milling induced I to II polymorphic transformation of mefenamic acid, a non-steroidal anti-inflammatory drug. The objective is to identify the fundamental mechanisms governing this type of transformation. Specifically, this work aims to understand the origin of the induction time characteristic of such transformations and to determine whether the change of state is direct or involves, on the contrary, a transient amorphization step.

High-energy milling operations were performed at room temperature and at low temperature (-196°C) using a Retsch CryoMill®. Experiments were conducted in a 50 mL zirconium oxide jar containing a single zirconium oxide ball (Ø 25 mm) at a frequency of 20 Hz. Structural evolution was monitored ex situ by Powder X-ray Diffraction (PXRD) and Differential Scanning Calorimetry (DSC). Quantitative phase analysis by Rietveld refinement (MAUD) enabled the determination of phase fractions and the construction of the kinetic profile of the transformation.

The results indicate that milling leads to:

- a I to II polymorphic transformation at 20°C

- a near-absence of transformation at -196 °C.

- a co-amorphization when mefenamic acid is combined with a high Tg polymer.

These findings, combined with the analysis of transformation kinetics, have allowed us to progress in the identification of the fundamental mechanisms governing polymorphic transformations during mechanical milling.

Neutron Crystallography Study of Host–Pathogen Recognition Enhanced by Hydrogen/Deuterium Exchange on Carbohydrates

Theodore ARNAUD (Institut Laue-Langevin & CERMAV)
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Michaela WIMMEROVA (National centre for biomolecular research, faculty of science, Marsaryk university)
Sophie FEUILLASTRE (Service de Chimie Bio-organique et Marquage, DMTS, CEA, Université Paris-Saclay)
Matthew P. BLAKELEY (Institut Laue-Langevin)
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Many human glycans serve as targets for virulence factors produced by pathogens. Among these factors, lectins are carbohydrate-binding proteins involved in host–pathogen recognition that play a central role in bacterial adhesion, tissue colonization, and biofilm formation. The opportunistic pathogen Pseudomonas aeruginosa, a major cause of hospital-acquired infections and a critical antibiotic-resistant bacterium, produces two soluble lectins, LecA and LecB, which specifically recognize galactose and fucose, respectively. LecA acts as a membrane disruptor by specific binding to the oligosaccharide head group of globoside, a sphingolipid consisting of ceramide with a Gal(α1-3)Gal(β1-4)Glc glycan part. LecA emerged as a promising target for the development of anti-virulence therapeutics and thus precise structural data are needed for the design of glycomimetics that act as “pathoblockers”.

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The neutron structures of perdeuterated LecA complexed with several deuterated ligands shed light on the importance of histidine protonation states in the active site, the water-mediated interactions, and hydrogen-bond networks within the LecA binding site. These structural insights significantly improve our understanding of lectin–carbohydrate recognition and provide valuable guidance for the rational design of novel anti-adhesive compounds targeting P. aeruginosa infections.

This presentation will be based on our recent publication (DOI: 10.1002/ceur.202500424).

In Situ Discovery of Potent InhA Inhibitors via Kinetic Target-Guided Synthesis

Julien RIZET (Univ Toulouse, CNRS, SPCMIB et IPBS)
Mélina CHEBAIKI (Univ Toulouse, CNRS, SPCMIB et IPBS)
Deborah RECCHIA (Dipartimento di Biologia e Biotecnologie “Lazzaro Spallanzani”, University of Pavia)
Saïda DANOUN (Univ Toulouse, CNRS, SPCMIB)
Evelyne DELFOURNE (Univ Toulouse, CNRS, SPCMIB)
Pascal HOFFMANN (Univ Toulouse, CNRS, SPCMIB)
Joëlle AZÉMA-DESPEYROUX (Univ Toulouse, CNRS, SPCMIB)
Gabriel PUBLICOLA (Univ Toulouse, CNRS, IPBS)
Bastien DUMAIS (Univ Toulouse, CNRS, IPBS)
Lionel MOUREY (Univ Toulouse, CNRS, IPBS)
Giulia DEGIACOMI (Dipartimento di Biologia e Biotecnologie “Lazzaro Spallanzani”, University of Pavia)
Maria Rosalia PASCA (Dipartimento di Biologia e Biotecnologie “Lazzaro Spallanzani”, University of Pavia)
Laurent MAVEYRAUD (Univ Toulouse, CNRS, IPBS)
Giulia DEGIACOMI (University of Pavia)
Christian LHERBET (Univ Toulouse, CNRS, SPCMIB)

Tuberculosis, caused by Mycobacterium tuberculosis, remains the deadliest infectious disease worldwide, with 10.7 million new cases and 1.23 million deaths reported in 2024. The emergence of multidrug-resistant and extensively drug-resistant strains highlights the critical need for innovative therapeutics [1]. Mycolic acid biosynthesis, essential for bacterial cell wall integrity, is a validated target pathway. InhA, the enoyl-ACP reductase within the FAS-II system, is a key enzyme traditionally inhibited by isoniazid, a prodrug requiring activation by the catalase-peroxidase KatG, though resistance due to KatG mutations has compromised its effectiveness [2,3].

This study leverages kinetic target-guided synthesis (KTGS) to identify novel direct inhibitors of InhA. By deconstructing established inhibitors such as GEQ and triclosan [4] into fragments, we reconstructed them in situ, enabling the discovery of potent compounds that target a previously under-exploited hydrophobic region of the InhA active site. These findings demonstrate the potential of KTGS as a fragment-based screening method for developing new anti-tuberculosis agents (manuscript in preparation).

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C-Branched Iminosugars as Selective Pharmacological Chaperones of Lysosomal Alpha-Glucosidase for the Treatment of Pompe Disease

Mme Gerlind SULZENBACHER (AFMB, CNRS-AMU)

C-Branched Iminosugars as Selective Pharmacological Chaperones of Lysosomal Alpha-Glucosidase for the Treatment of Pompe Disease

Anaïs Vieira Da Cruz,a,‡ Valentine Perraudin,a,‡ Nadia Minopoli,b,‡ Roberta Iacono,c,d,‡ Véronique Roig-Zamboni,e Andrea Bossio,a Salia Tangara,a Martine Fayolle,a Alice Kanazawa,a Christian Philouze,a Antonietta Tarallo,b,f Jurriaan J. A. Heming,g Marta Artola,g Jean-Bernard Behr,h Herman S. Overkleeft,g Marco Moracci,*c,d Gerlind Sulzenbacher,e Giancarlo Parentib,f and Sandrine Pya

a Univ. Grenoble Alpes, CNRS, DCM, Grenoble, France

b Telethon Institute of Genetics & Medicine, Pozzuoli, Italy

c Department of Biology, Federico II University, Naples, Italy

d National Biodiversity Future Center (NBFC), Italy

e Architecture et Fonction des Macromolécules Biologiques, CNRS, Aix-Marseille University, Marseille, France

f Department of Translational Medical Sciences, Federico II University, Naples, Italy

g Department of Medical Biochemistry and Department of Bioorganic synthesis, Leiden Institute of Chemistry (LIC), Leiden, The Netherlands

h Université de Reims Champagne-Ardenne, CNRS, ICMR, Reims, France

We report herein the design and synthesis of a series of 5-C-alkyl-deoxynojirimycins from L-sorbose, through an efficient and scalable method amenable to prepare a large variety of analogues. The interaction of this class of compounds with human acid esadisoculg- (GAA), the genetically defective enzyme in patients suffering from Pompe disease, was investigated to identify pharmacological chaperones exhibiting high selectivity for this enzyme. Crystallographic analyses provided a rationale for their binding mode to GAA and chaperone activity. The effects of 5-C-phenethyl-DNJ (4c) were evaluated on GAA activity enhancement in cells from Pompe disease patients and in GAA-KO mice. The significant increase of GAA activity in the presence of 4c in various tissues, and more particularly in the diaphragm, encourage further studies on this class of small molecules towards the development of clinical drugs. Their chaperone activity and excellent selectivity may offer potential benefits over the current treatments of Pompe disease.

STRUCTURAL INSIGHTS INTO NITROGENASE FEMO-CO ASSEMBLY

Dr Yvain NICOLET (Institut de Biologie Structurale)

The use of transition metals in association with proteins enables living organisms to carry out chemical reactions that would otherwise be impossible with the only used of the 22 standard amino acids. Some of these reactions lie at the core of processes that emerged at the origin of life on Earth. Today, metalloproteins catalyze a wide range of essential biochemical transformations. In particular, nitrogenase is responsible for the fixation of atmospheric nitrogen, making it accessible to all living organisms. Its activity relies on complex metallocofactors, the biosynthesis of which requires the coordinated action of numerous accessory proteins.

In this presentation, we will discuss recent work aimed at elucidating the structure–function relationships of metalloproteins involved in the molecular machinery responsible for the biosynthesis and insertion of the metallocofactor essential for nitrogenase activity. We will show how the structural organization of these proteins is coupled to dynamic processes that drive the stepwise assembly of the cofactor and its subsequent insertion into the enzyme.

New insights into the mechanism of iron transport through the bacterial Ftr system present in pathogens

Armelle VIGOUROUX (CNRS I2BC)
Anne-Soisig STEUNOU (CNRS I2BC)
Magali AUMONT-NICAISE (CNRS I2BC)
Stéphane PLANCQUEEL (CNRS I2BC)
Alain BOUSSAC (CNRS I2BC)
Soufian OUCHANE (CNRS I2BC)
Solange MORÉRA (CNRS I2BC)

Iron is an essential nutrient in bacteria. Its ferrous form, mostly present in low oxygen and acidic pH environments, can be imported using the specific Ftr-type transport system, which encompasses the conserved FtrABCD system found in pathogenic bacteria such as Bordetella, Brucella and Burkholderia. The nonpathogenicity and versatile metabolism of Rubrivivax gelatinosus make it an ideal model to study the FtrABCD system. Here, we report a new aspect of its regulation and the role of the periplasmic proteins FtrA and FtrB using in vivo and in vitro approaches. We investigated the metal binding mode and redox state of copper and iron to FtrA by crystallography and biophysical methods. An 'as isolated' FtrA protein from the bacterial periplasm contained a copper ion (Cu+) identified by electron paramagnetic resonance (EPR). Copper is coordinated by four conserved side chains (His and Met) in the primary metal site. Structural analysis of R. gelatinosus FtrA and FtrA homologues revealed that copper binding induces a rearrangement of the His95 imidazole ring, releasing thereafter space, as well as both Asp45 and Asp92 side chains, for iron binding in the secondary metal site. EPR highlighted that FtrA can oxidize the bound ferrous ion into the ferric form by reducing the bound Cu2+ into Cu+ , both metal sites being separated by 7 Å. Finally, we showed that FtrB binds iron and not copper. These results provide new insights into the mechanism of ferrous iron utilization by the conserved FtrABCD iron transporter for which we propose a new functional model.

Study of the conformational dynamics of a bacterial photoactivated adenylate cyclase

Sofia KAPETANAKI (MAX IV Laboratory)
Norbert TROMBITAS (University of Pécs)
Zsofia ZALAI (University of Pécs)
Kevin POUNOT (European Synchrotron Radiation Facility)
Aenor BARRET (LOB, CNRS, INSERM, École Polytechnique, Institut Polytechnique de Paris)
Emoke BODIS (University of Pécs)
Zsuzsanna FEKETE (University of Pécs)
Jovana VITAS (Institut de Biologie Structurale, Université Grenoble Alpes, CEA, CNRS, Grenoble, France)
Eva HOFFMANNÉ SIMON (University of Pécs)
Mikhail KOZHAEV (European Synchrotron Radiation Facility)
Jonatan PASITKA (University of Pécs)
Ildiko PECSI (University of Pécs)
Nikolett VARNYUNÉ KIS-BICSKEI (University of Pécs)
Marten VOS (LOB, CNRS, INSERM, École Polytechnique, Institut Polytechnique de Paris)
Pascale CHANGENET-BARRET (LOB, CNRS, INSERM, École Polytechnique, Institut Polytechnique de Paris)
Martin WEIK (Institut de Biologie Structurale, Université Grenoble Alpes, CEA, CNRS, Grenoble, France)
Matteo LEVANTINO (European Synchrotron Radiation Facility)
Andras LUKÁCS (University of Pécs)
Giorgio SCHIRÒ (Institut de Biologie Structurale, Université Grenoble Alpes, CEA, CNRS, Grenoble, France)

Photoactivated adenylate cyclases (PACs) are blue-light photoreceptors coupled with adenylate cyclases and able to modulate the cyclic adenosine monophosphate (cAMP) levels in the cell upon blue-light illumination. OaPAC, a flavoprotein discovered ten years ago (1) is a member of the PAC family and is considered a highly promising candidate for optogenetic applications. Despite the various approaches that have been employed to date (1-8) the signal transduction pathway in OaPAC still remains elusive. In this work, we have applied time-resolved X-ray solution scattering (TR-XSS) (9), a technique that enables real-time structural characterisation under near-native conditions and combines high temporal resolution and structural sensitivity to probe the blue-light induced conformational dynamics in OaPAC. The implications of our findings are discussed.

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Light atoms identification and location by anomalous scattering

Kamel EL OMARI (Diamond Light Source)
Ramona DUMAN (Diamond Light Source)
Christian ORR (Diamond Light Source)
Vitaliy MYKHAYLYK (Diamond Light Source)
Armin WAGNER (Diamond Light Source)

More than a third of all known proteins bind metal ions. Metal ions play key roles in a broad range of cellular processes, they are involved in protein structure stability and catalysis; with traditional examples of zinc fingers in transcription factors and iron in haemoglobin. Therefore, identifying metal ion-binding sites is important for understanding the biological functions of proteins and further helps in designing potent therapeutics.

Experimental and computational methods have been developed to identify or predict metal ion ligand-binding residues. However, experimentally identifying and locating metal ions, such as calcium and potassium in protein structures can be challenging. The unique wavelength range of the macromolecular crystallography beamline I23 at Diamond Light Source allows identification and location of metal ions and lighter atoms of biological relevance (Ca, K, S, P and Cl) using X-ray anomalous scattering in crystal structure analysis.

In a typical experiment, anomalous datasets are collected at two wavelengths, above and below the ion or element absorption edge, and then processed to calculate phased anomalous Fourier difference maps. The difference in anomalous peak heights between these two datasets allows the direct identification and visualisation of the ion in the protein structure. We successfully used this method in different projects to experimentally map ions in crystal structures and some examples will be discussed.

Deuteration of biomolecules for neutron structural biology

Juliette DEVOS (Institut Laue-Langevin)
Valérie LAUX (Institut Laue-Langevin)
Martine MOULIN (Institut Laue-Langevin)
Frank GABEL (Institut Laue-Langevin)

In neutron experiments in biology, the replacement of the common hydrogen isotope protium (^{1}H) by its stable isotope deuterium (^{2}H) in biomolecules can be of crucial importance. Depending on the technique, different levels of deuteration are required to enhance contrast allowing selective visualization of molecular components in complex biological systems, to reduce incoherent scattering or to increase signal. This is particularly important in neutron macromolecular crystallography (NMX) where hydrogen (deuterium) atom positions and protonation states can be directly resolved.
The Institut Laue-Langevin Deuteration Laboratory (ILL D-Lab) is a platform specialised in the *in vivo* deuteration of biological molecules for neutron applications, including small-angle neutron scattering (SANS), protein crystallography (NMX), dynamics studies and neutron reflectometry (NR). Microorganisms such as bacteria and yeasts have been successfully adapted to growth in deuterated minimal media, enabling efficient isotope incorporation.

Large-scale protein deuteration by recombinant expression in high-cell density cultures was initially developed at the ILL D-Lab. The production of various labelled biomolecules required for the study of proteins, protein-nucleic acid complexes, protein-lipid complexes, glycoproteins, membrane proteins and stealth lipid nanodiscs will be presented.
The *in vivo* deuteration of small biomolecules of major functional importance will also be highlighted, together with recent methodological developments.
Access to the D-Lab platform is available to all neutron users through a peer-reviewed user program, enabling optimized integration of deuteration strategies alongside neutron beamtime proposals. Deuteration laboratories have become essential infrastructure, driving innovation for the study of biomolecular structure and dynamics using neutrons.

Subsite binding in the yin-and-yang Bacteroides thetaiotaomicron sialidase at 1.5 Å resolution

Aminata BATHILY (Université de Lille)
Shubham SEMWAL (Unité de Glycobiologie Structurale et Fonctionnelle (UGSF), UMR 8576 CNRS & University of Lille)
Clarisse BRIDOT (Unité de Glycobiologie Structurale et Fonctionnelle (UGSF), UMR 8576 CNRS & University of Lille)
Thomas BESSONET (Unité en Sciences Biologiques et Biotechnologies (US2B), UMR 6286 CNRS, Nantes University)
Amélie SAUMONNEAU (Unité en Sciences Biologiques et Biotechnologies (US2B), UMR 6286 CNRS, Nantes University)
Cyrille GRANDJEAN (Unité en Sciences Biologiques et Biotechnologies (US2B), UMR 6286 CNRS, Nantes University)
Franck DALIGAULT (Unité en Sciences Biologiques et Biotechnologies (US2B), UMR 6286 CNRS, Nantes University)
Sébastien GOUIN (Chimie Et Interdisciplinarité, Synthèse, Analyse, Modélisation (CEISAM), UMR 6230 CNRS)
Julie BOUCKAERT (Unité de Glycobiologie Structurale et Fonctionnelle (UGSF), UMR 8576 CNRS & University of Lille)

Inflammatory bowel disease (IBD) is increasing worldwide, highlighting the need to better understand gut microbiome functions and their therapeutic potential. The beneficial gut bacterium Bacteroides thetaiotaomicron contributes to intestinal health through the degradation of complex dietary glycans and the modulation of inflammatory responses. Its secreted sialidase, BtSA, is thought to play a key role in these processes, although the molecular basis of its high catalytic efficiency remains unclear.

To investigate the structure–function relationship of BtSA, we applied an integrative structural biology approach combining X-ray crystallography, small-angle X-ray scattering (SAXS), size exclusion chromatography-multi angle light scattering (SEC-MALS), mass photometry, surface plasmon resonance (SPR), and isothermal titration calorimetry (ITC). Structural and biophysical analyses revealed that BtSA forms a stable yin–yang-shaped dimer in solution, with the catalytic domain of one monomer positioned toward the carbohydrate-binding module (CBM) of the opposing monomer. Functional studies showed enhanced sensitivity to a divalent inhibitor compared with a related sialidase (1), suggesting cooperative interactions between the two monomers. In addition, SPR and ITC experiments demonstrated that substrate binding and catalytic activation are induced by the presence of a sialylated substrate rather than by the reaction product alone (free sialic acid).

Together, these findings support a mechanism in which dimerisation promotes catalytic turnover through cooperation between the catalytic site and the CBM during substrate recognition and processing. This work provides a molecular explanation for the high efficiency of BtSA and establishes a foundation for future studies investigating the function of the CBM and its potential ligands.

Reference: (1) Assailly, Coralie, et al. "Polyvalent Transition-State Analogues of Sialyl Substrates Strongly Inhibit Bacterial Sialidases." Chemistry–A European Journal 27.9 (2021): 3142-3150.

Association of Resources for Biophysical Research in Europe

Julie BOUCKAERT (UGSF - CNRS)

ARBRE is a community of technology and method-oriented scientists who constitute the human resources that enable researchers to answer questions about biological systems.

ARBRE focuses on molecular-scale biophysics. This is a dynamic interdisciplinary field that aims to study biological macromolecules and assemblies at an intermediate level between atomic-resolution structural descriptions and cellular-level observations (“Between atom and cell”), with significant applications in biomedicine, drug discovery, biotechnology, structural biology and interdisciplinary life sciences.

Its members are principally:

→ Core facility, research infrastructure and shared resource laboratory staff in academia and industry

→ Experimental scientists with a technological/methodological expertise

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→ Developers of novel softwares, databases and artificial intelligence algorithms

→ Instrumentation company R&D and application scientists

→ Contract Research Organization members

→ Students and early-stage post-docs using biophysical instrumentation and wishing to network with experts

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Charge transfer in a redox protein – insights from neutron dynamics studies and cryo-neutron crystallography

Juliette DEVOS (Institut Laue-Langevin)
Maxime CUYPERS (University of Rochester Medical Center)
Estelle MOSSOU (European Synchrotron Radiation Facility)
Marie-Hélène LEMÉE (Institut Laue-Langevin)
Tilo SEYDEL (Institut Laue-Langevin)
V. Trevor FORSYTH (Faculty of Medicine and LINXS Institute for Advanced Neutron and X-ray Science)

Rubredoxins are small monomeric iron-sulphur cluster proteins found in prokaryotes and in some eukaryotes. They act as electron carriers in a variety of biological activities such as fatty acid metabolism, processing of reactive oxygen species, and carbon fixation. Rubredoxin from *Pyrococcus furiosus* (*Pf*) is one of the most thermostable proteins known. As a redox protein, rubredoxin is also an important model system for an understanding of electron transfer processes associated with catalysis.
In previous work, a near atomic resolution study of perdeuterated *Pf* rubredoxin revealed several hydronium ions that are clearly implicated in the stability of both redox forms of the protein. We now present recent and unpublished studies on this exciting system. Using neutron spectroscopy, we have measured both inelastic and elastic fixed window data in order to investigate the possibility of tunnelling phenomena within the protein. While the data needs further careful analysis, the dynamical transition of the protein has been clearly identified. We also have recorded two high resolution (0.88 Å and higher) single crystal neutron diffraction experiments of perdeuterated rubredoxin at a temperature of 100 K. The presented work was carried out using the high resolution thermal diffractometer D19 at the Institut Laue-Langevin. The protein was produced in the ILL Deuteration Laboratory. The crystal growth, the sample mount and environment to obtain the best possible cryo data set will be highlighted. The structural analysis shows a striking network of Zundel and hydronium ions as well as ordered water molecules that link to the iron-sulphur cluster of the protein. These observations are highly suggestive of the way in which protonation shifts involving exotic ionic species may be involved in the charge transfer processes of redox proteins. The results are the first of their type in the study of redox biology.

Inducing Binding Hot Spots Enables Selection of Protein Binders to Defined Regions of a Target Protein

Ing Ines Li de la LI DE LA SIERRA-GALLAY (Institute for Integrative Biology of the Cell)

Inducing Binding Hot Spots Enables Selection of Protein Binders to Defined Regions of a Target Protein

Margarida Gomes, Agnès Mesneau[a], Augustin Degaugue, Florian Gourmelon, Raoudha Dammak[a], Guillaume Villain, Magali Noiray[a], Magali Aumont-Nicaise[a], Ines de Li la Sierra-Gallay[a], Philippe Minard[a], Marie Valerio-Lepiniec[a], Agathe Urvoas[a]

[a] Institute for Integrative Biology of the Cell (I2BC), CEA, CNRS, Université Paris-Saclay, 91198 Gif-sur-Yvette, France

It is now possible to generate proteins that bind specifically to a wide range of targets; however, conventional selection methods, such as combinatorial library screening methods (e.g., phage display), offer limited control over the binding site. Here, we propose an approach to direct selection toward a defined region by engineering the target protein surface. This strategy enables the generation of binders with site specificity, opening new opportunities for biotechnology and therapeutic applications. For example, DARPin-based proteins can be engineered as multifunctional therapeutics by combining binding domains against different targets within a single polypeptide (Caputi, 2020).
We developed a strategy to generate binders targeting a specific region of a protein surface by engineering artificial binding “hot spots”. Binder selection was performed using an αRep library. This approach was validated on three targets, including two engineered αReps (NB1 and NB2) and CheYtin, yielding specific binders in all cases. Crystal structures confirmed binding at the engineered site. These results highlight the potential of this strategy for generating targeted binders and designing affinity clamps.

Bibliographie

Achille Patrizio Caputi, Pierluigi Navarra. 2020.” Beyond antibodies: ankyrins and DARPins. From basic research to drug approval”. Current Opinion in Pharmacology. Volume 51: 93-101

Consequences of structural dynamics on the fluorescence properties of a family of infrared fluorescent proteins

Antoine ROYANT (Institut de Biologie Structurale, Grenoble)

This work constitutes a thorough structural study of a family of infrared fluorescent proteins (IFPs) derived from the bacteriophytochrome of a Gram-negative bacterium of the genus Bradyrhizobium, which uses biliverdin, a product of haem catabolism, as a chromophore. The studied IFPs include the protein mIFP and its blue-shifted variants iBlueberry and iBlueberry2. IFPs are of significant interest in whole-body imaging due to the existence of an optical tissue window between 650 and 900 nm, namely above the region where haemoglobin and deoxy-haemoglobin absorb, and below the region where water and lipids absorb and scatter light. The proteins studied here exhibit fluorescence emission maxima between 667 and 704 nm. The structural study reveals that a loop located near the biliverdin can adopt a wide range of conformations, unlike other phytochromes and IFPs studied to date, where this loop appears to have only one conformation. The consequence of this conformational flexibility is the presence of a chromophore configuration that has never been observed previously, leading to a compact conformation of the macrocycle formed by the four pyrrole rings of biliverdin. This is distinct from the linear, or extended, conformation typically observed. In crystallo UV-Visible absorption spectroscopy and fluorescence emission spectroscopy, applied directly to the crystals, demonstrated that the compact conformation of the chromophore results in a significantly red-shifted fluorescence emission peak for all three proteins. This conformation appears to be favoured by the crystalline packing and must only be weakly populated in solution. This work provides insights into how to modify the chromophore configuration within the protein through rational design, for instance using artificial intelligence-based methods. The goal is to develop new IFPs with emission maxima in the middle of the tissue optical window, thereby exploiting better the latter in multi-colour labelling experiments of tissue or whole-body imaging.

MS09-P36

A cis-peptide-defined mannose-binding pocket reveals how Mannitou IgM recognises paucimannose

Shubham SEMWAL (UGSF - CNRS)

Aminata BATHILY (Universite de Lille)

Alexis DESCAMPS (Dioscuri Center for Modelling of Posttranslational modifications, MCB, Jagiellonian University, Krakow, Poland)

Yehudi BLOCH (European Molecular Biology Laboratory, Hamburg Unit c/o DESY, Hamburg, Germany)

Simone DIESTEL (Institute of Nutrition and Food Sciences, University of Bonn, Bonn, Germany)

Kenneth VERSTRAETE (VIB-UGent Center for Inflammation Research, Ghent, Belgium. Unit for Structural Biology, Department of Biochemistry and Microbiology, Ghent University, Ghent, Belgium)

Savvas N. SAVVIDES (VIB-UGent Center for Inflammation Research, Ghent, Belgium. Unit for Structural Biology, Department of Biochemistry and Microbiology, Ghent University, Ghent, Belgium)

Julie BOUCKAERT (CNRS UMR 8576)

Paucimannosidic N-glycans are compact carbohydrate epitopes associated with altered glycosylation in biological and pathological contexts, yet the structural basis for their antibody recognition remains poorly understood. Mannitou, a murine IgM selective for paucimannose-containing glycans, offers a unique model to define how weak monovalent glycan interactions are converted into high-avidity immune recognition.

Here, we present an integrated structural and biophysical characterisation of Mannitou Fab. Investigation of the binding thermodynamic profile of Mannitou Fab to oligomannose-3 and heptyl α-d-mannopyranoside via ITC revealed micromolar binding, whereas characterisation of the binding kinetics by SPR and whole IgM binding data support a marked avidity enhancement driven by the decavalent architecture of IgM. MALLS, SEC-SAXS, SRCD, and mass-spectrometry further showed that the recombinant Fab contains monomeric and partially misfolded dimeric species and carries an N-linked glycan at Asn164 of the heavy chain.

The central advance of this work is the crystal structure of Mannitou Fab in complex with heptyl α-d-mannopyranoside to 1.7Å resolution. The carbohydrate ligand is reproducibly captured in two independent Fab molecules, revealing a conserved mannose-recognition pocket. The mannose hydroxyls are anchored by Glu50, Gly96, Gln99, Trp101, and Gly103, while Trp33, Tyr37, and Trp102 create an aromatic cage that accommodates C1-C2 of the mannose and its hydrophobic heptyl extension. Notably, a non-proline cis-peptide geometry involving the Gln99/Trp102 region organises the binding site and appears essential for shaping the pocket. A second crystal form revealed that the Asn164-linked M6F glycan engages a symmetry-related Fab through the conserved mannose-binding site, highlighting the relevance of this pocket across distinct glycan presentations.

Together, these structures explain how Mannitou Fab recognises paucimannose through an organised binding site/pocket, while IgM multivalency amplifies weak monovalent contacts into strong functional binding with nanomolar affinities. This work provides a molecular framework for understanding antibody recognition of a truncated N-glycan epitope.

Enseignement et médiation scientifique, le rôle clé de l'experience

Mme Giura PAOLA (Sorbonne Université-IMPMC)

Une personne célèbre a dit un jour : « La main est l'instrument de l'intelligence. » soulignant ainsi l'importance de manipuler des objets concrets : manipuler pour réfléchir et toucher pour mieux comprendre. Je suis fortement partisane de cette devise et depuis plus de sept ans, je teste l'impact de l'enseignement par l'expérience auprès d'un large éventail de publics aux parcours éducatifs variés. Un volet très important de la méthode d'apprentissage par l'expérience repose sur la proposition d'ateliers dont le but est d'appréhender les concepts scientifiques avec des mises en situation pas-à-pas. Ma présentation montre une sélection des actions que j'ai menées dans ce sens pour la médiation scientifique, la formation continue et l'enseignement supérieur, ainsi que les objets conçus et créés pour les mettre en œuvre. Le tout, bien entendu, axé sur les cristaux et leurs propriétés. Des exemples des outils proposés sont à découvrir dans l'espace « ateliers » de cette section.

Ref: 1. M. Montessori, L'esprit absorbant de l'enfant, 1949.

The Cambridge Structural Database is not only a database but also a knowledge base – workshops in France

Arie VAN DER LEE (IEM - Université de Montpellier - CNRS)

The Cambridge Structural Database (CSD) is world's largest database of curated organic and metallo-organic crystal structures that have been determined by diffraction techniques. The principal reason to query the database is to know whether a structure has been published before. All other reasons to use the data could lead to new knowledge. This was already foreseen in the early days of the CSD when its founder, Olga Kennard, wrote that "the collective use of data would lead to the discovery of knew knowledge, which transcends the results of individual experiments" long before the word 'machine learning' was used. The Cambridge Crystallographic Data Centre (CCDC) has developed a large set of tools to mine and analyse the data in the CSD and it also provides many on-line tutorials and courses to help scientists using these tools. However, nothing is more stimulating than an in-person training. For this reason, the CCDC stimulates CSD-ambassadors in many countries worldwide to organize in-person training sessions in their country. Several CSD training sessions have been organized throughout France in the last years: the pedagogical principles and the course of a typical workshop day will be presented. If time permits, one demonstration will be presented.

A single crystal X-ray diffractometer scale model for scientific dissemination

Michel GIORGI (Fédération des Sciences Chimiques de Marseille (UAR1739))
Ludovic DELBES (Institut de Minéralogie, de Physique des Matériaux et de Cosmochimie)

During the workshop time of the 'Teaching and Scientific Mediation' session, we will present an automated scale model of a four-circle single crystal X-ray diffractometer that has been developed by the professional network RÉCIPROCS (https://reciprocs.cnrs.fr) for scientific dissemination. This scale model is based on the one previously designed and created on behalf of the ACAM (Association de Cristallographie d'Aix-Marseille: http://acam.cristal-provence.fr)[1]. The purpose of this device is to reach out to the wider public and students to introduce them in a playful way to one of the laboratory apparatuses to which they do not usually have access, - or even know that it exists - and use it as a basis for explaining various phenomena and concepts in a playful way. Crystallography is a multi-disciplinary field that links many areas of mathematics, science and engineering. Moreover, the various technologies used in the design and operation of a four-circle single crystal X-ray diffractometer make it a very visual apparatus, likely to attract the eye and allowing to explain many physical phenomena or mathematical concepts - electromagnetic radiation, diffraction, interferences, Fourier transform - and to talk about crystallography in the broadest sense - state of matter, crystals, chemistry, biology etc... -. Such an approach can be useful to anyone involved in scientific dissemination and can also be developed for other laboratory equipment and other disciplines. This kind of device can also be the subject of scientific and technological projects in close collaboration with educational institutions. It was the case for the final year project of an apprentice in physics at the Laboratoire de Cristallogenèse de l'Ecole Polytechnique Fédérale de Lausanne, based on the former scale model.

[1] M. Giorgi & Y. Berchadsky
, J. Appl. Cryst. (2022) 55, 149-153

The Fourier-Maton : a visual demonstration of the Fourier transform methods in crystallography

Sébastien PILLET (Laboratoire CRM2 - Université de Lorraine)
Elodie TAILLEUR (Laboratoire CRM2 - Université de Lorraine)
Emmanuel WENGER (Laboratoire CRM2 - Université de Lorraine)
Cyril PALIN (Laboratoire CRM2 - Université de Lorraine)
Rémi CLAUSSE (Laboratoire CRM2 - Université de Lorraine)

Fourier transform methods are central to modern crystallography, providing the mathematical framework that links experimentally measured reciprocal space to real-space atomic structures. However, the theory of Fourier transform is often perceived as highly abstract by beginners in crystallography. This animation illustrates the foundational role of Fourier transforms in crystallography by introducing an intuitive, visual demonstration inspired by a “Photomaton” approach. It allows participants to explore how periodicity, defects, and motif shape influence the resulting reciprocal space. Finally, participants can visually examine the reconstructed object, gaining a clearer and more concrete understanding of the underlying principles.

An immersive experience in biocrystallography

Claude SAUTER (ARN - IBMC - CNRS)
Agnès GAUDRY (ARN - IBMC - CNRS)

There is nothing like a film to engage with the general public and introduce them to the scientific process, the lives of scientists, and their work environment. We embarked on this adventure in 2006 with our first short film illustrating the stages of a crystallographic study leading to the determination of the three-dimensional structure of an enzyme and its application in drug design. Two decades have passed, and thanks to new imaging technologies, we present a virtual reality remake that takes the viewer from the biology laboratory to the SOLEIL synchrotron to explore the secrets of life. Come and join the adventure!

Atelier vidéo “Je suis cristallographe”

Nicolas CASARETTO (LCM, Institut Polytechnique de Paris, CNRS)
Laureen BUSSON (LCM, Institut Polytechnique de Paris, CNRS)
Jeremy FORTÉ (Sorbonne Université, IPCM, UMR 8232)
Delphine CABARET (Sorbonne Université, IMPMC, UMR CNRS 7590)
Elen DUVERGER-NÉDELLEC (Univ. Bordeaux, CNRS, Bordeaux INP, ICMCB, UMR 5026)

La cristallographie est une science riche, profonde, et multiculturelle. Nous sommes souvent à l’interface de plusieurs disciplines et cela se reflète dans nos parcours et nos expériences. Dans ce contexte, les membres de notre communauté ne s’identifient que rarement en tant que cristallographes, ne se sentant pas représentés dans l’image d’Épinal du vieux sage avec les tables internationales sous le bras. Car il s’agit bien de représentation ici. Comment faire exister l’image du/de la cristallographe dans toute sa pluralité ?

Cet atelier propose de déconstruire l’identité scientifique des participants afin de dresser des portraits de cristallographes modernes sous formes de vidéos courtes qui seront ensuite diffusées sur les réseaux de l’AFC. Nous proposerons aux volontaires de répondre à des questions sur leur parcours devant notre caméra, et s’ils le souhaitent, d’illustrer leur quotidien dans des mises en scènes ludiques.

La question existentielle du sentiment d'appartenance va plus loin que la simple auto-réfection, elle est centrale dans la construction d’une communauté. Que ce soit la transmission du savoir ou le renouvellement des experts, tout prend racine dans la représentation.

Hands-on, challenge-based teaching in crystallography undergraduate practicals

Peter HORTON (University of Southampton)
Simon COLES (University of Southampton)

Following a need for increased hands-on experience with technical analytical instrumentation for undergraduate students, the University of Southampton has offered just such a practical course to third years for the past 15 years. This involves the preparation of crystalline materials, with subsequent analysis including the collection of single crystal X-ray diffraction data and work-up to generate fully refined crystal structures [1].
Over those years the practical has steadily evolved. Thus, the first practical which was predominantly 'cookbook' has now been superseded by a problem led exercise that takes place for one full day a week over three weeks. Students are presented with a marketplace of co-formers and tasks them to modify the physical properties of an API to be within a particular set of requirements. However, many of the fundamental principles of that original practical persist - ensuring students independently conduct all analyses (with no 'black box' results) to gain applied hands-on experience of a range of solid-state analytical methods, using cheap and readily available chemicals that can be considered safe if suitable precautions are taken. The practical allows for the students to gather results, reflect and then possibly take the work in a different direction in a subsequent session.
Here we will present how the practical has changed and continues to evolve, what has worked well (and has not), but also show that it is definitely possible for undergraduate students to grow a range of good, suitably sized crystals in a week, for which a publication quality dataset can be collected on a sealed tube diffractometer with CCD detector in an hour (or less).

[1] Coles, S.J. and Mapp, L.K., Conducting Reflective, Hands-On Research with Advanced Characterization Instruments: A High-Level Undergraduate Practical Exploring Solid-State Polymorphism, J. Chem. Educ., 2016, 93, 131–140.

“3D-Electron Diffraction”: a training workshop focused on the practical aspects of 3D electron diffraction in materials and life sciences

Stéphanie KODJIKIAN (Univ. Grenoble Alpes, CNRS, Grenoble INP, Institut Néel, 38000 Grenoble, France)
Christophe LEPOITTEVIN (Univ. Grenoble Alpes, CNRS, Grenoble INP, Institut Néel, 38000 Grenoble, France)
Dominique HOUSSET (Univ. Grenoble Alpes, CEA, CNRS, IBS, 38000 Grenoble, France)
Christian PHILOUZE (Univ. Grenoble Alpes, CNRS, DCM, 38000 Grenoble, France)
Olivier PEREZ (CRISMAT, CNRS, Normandie University, ENSICAEN, UNICAEN, 14000 Caen, France)

Electron diffraction for structural resolution is now becoming widespread, and all the relevant methods and techniques are now grouped under the umbrella term “three-dimensional electron diffraction (3D-ED)”.

Aware of this dramatic technological evolution and its impact on the profession of crystallography, the Reciprocs network has been running training initiatives for its members since 2018: Lecture on 3D-ED at the ANF workshop “Diffraction under extreme or atypical conditions: advanced techniques and tools”, followed by the 2022 ANF workshop “Electron diffraction for structural crystallography. Since 2023, Reciprocs has expanded its training offering to include 3D-ED technology workshops, open to all network members wishing to familiarise themselves with 3D-ED and its use in structural determination.

The three-day training course consists of a combination of demonstrations and practical sessions using transmission electron microscopes, alongside data analysis during computer-based tutorials. Trainees themselves implement data acquisition and processing methods in order to develop practical skills that can be transferred to their home laboratories. The first two days, devoted to materials science, take place at the Institut Néel (Grenoble), whilst the third day, dedicated to biomaterials, takes place at the IBS (Grenoble).

We will outline the key features of this course.

Acknowledgements:

1) Platforms of the Grenoble Instruct-ERIC center (ISBG; UAR 3518 CNRS-CEA-UGA-EMBL) within the Grenoble Partnership for Structural Biology (PSB), supported by FRISBI (ANR-10-INBS-0005-02) and GRAL, financed within the University Grenoble Alpes graduate school (Ecoles Universitaires de Recherche) CBH-EUR-GS (ANR-17-EURE-0003). The IBS-ISBG electron microscope facility is supported by the Région Auvergne-Rhône-Alpes, Fondation pour la Recherche Médicale (FRM), Fonds FEDER and the GIS-Infrastructures en Biologie Santé et Agronomie (IBiSA).

2) The TEM facility JEOL NEOARM at CNRS Institut Néel was co-financed by the European Union under the European Regional Development Fund (ERDF, contract no. RA0023813).

An Python ecosystem for powder X-ray diffraction data visualization, editing, and analysis: XRDPlotter, PlotTxrd and PCReditor

Carmelo PRESTIPINO (Crismat)

The analysis of powder X-ray diffraction (PXRD) data remains a cornerstone of crystallography and materials science, requiring efficient tools for visualization, preprocessing, and refinement workflow preparation. While established software solutions such as WinPLOTR provide robust and widely adopted graphical environments for powder diffraction, the growing use of Python in scientific computing calls for more flexible, scriptable, and extensible approaches.

In this contribution, we present an integrated ecosystem of Python tools dedicated to powder diffraction data: XRDPlotter, PlotTxrd, and PCReditor (available at https://prestipino.github.io/PlotTxrd.html). These complementary packages are designed to streamline the workflow from raw PXRD data inspection to refinement input preparation.

PlotTxrd provides an interactive environment for the visualization and manipulation of powder diffraction patterns, supporting flexible data handling, multi-pattern comparison, and advanced graphical control. It is particularly suited for rapid exploration of experimental datasets, including in situ and high-throughput measurements.

XRDPlotter extends these capabilities toward high-quality and publication-ready representations, enabling advanced customization and comparison between experimental and simulated PXRD data.

PCReditor complements this workflow by offering a dedicated interface for the preparation and editing of refinement input files, for Rietveld software FullProf. It simplifies interaction with complex file formats while preserving full user control over refinement strategies.

Together, these tools form a lightweight and extensible ecosystem specifically tailored for powder diffraction, promoting reproducibility, transparency, and user-driven customization. Their development is driven by practical needs in crystallography, including the treatment of non-standard datasets, variable instrumental conditions, and complex experimental workflows.

CRYSCALC, a crystallographic calculator

M Thierry ROISNEL (ISCR CNRS Univ Rennes)

CRYSCALC, a crystallographic calculator (makes crystallographer life easier)
T. Roisnel
In memory of Loïc Le Dréau [1984-2026]

CRYSCALC is a software that has been designed to help the crystallographer in its daily work ; it performs some basic crystallographic calculations and provide some crystallographic information on space groups. CRYSCALC is based in part on modular routines of the CRYSFML library [1] and uses keywords and arguments that user has to input through the command line or from an input file.
Among the main facilities implemented in CRYSCALC, we can itemize :

- Space groups : list of symmetry operators, Wyckoff positions, extinctions …
- Crystallographic calculations : unit cell volume, matrix transformations, interatomic distances and angles, connectivity, BVS…
- Atomic database (weight, radii, oxidation states…)
- Scattering database: scattering and absorption cross-sections for X-rays and neutrons
- Structure factor calculation for X-rays and neutrons
- Simulation of powder diffraction pattern
- Analysis of {hkl} list: search for space group, calculation of Rint and completeness, statistics…
- Input/output of crystal structure through common input files (.CIF, .INS, .PCR, .XYZ, Z-matrix …)
- Creation of final CIF archive file for CCDC deposit
- Creation of experiment reports (.HTML, .pdf) for single crystal experiment
- Miscellaneous: matrix calculation, references, update …

CRYSCALC is working in a CMD window; software features can be customized to the user preferences through a setting file, in which some external applications (editor, browser, molecular viewer…) or diffractometer features (radiation, profile function, instrumental resolution function, common software…) can be defined.
CRYSCALC is available free of charges for academic institutions and can be downloaded from the following URL: https://cdifx.univ-rennes.fr/progs/cryscalc

[1] Crystallographic Fortran 90 Modules Library (CrysFML): a simple toolbox for crystallographic computing programs, Juan Rodríguez-Carvajal and Javier González-Platas
https://www.iucr.org/resources/commissions/crystallographic-computing/mandate-and-history/newsletters/1/crysfml

Mapping the slow propagation of disorder across a protein photoreceptor's tertiary structure with time-resolved crystallography

Nicolas CARAMELLO (Department of Chemistry, National Taiwan University)
Yuhei HOSOKAWA (Department of Chemistry, National Taiwan University)
Kai-Chun YANG (Department of Chemistry, National Taiwan University)
Po-Hsun WANG (Department of Chemistry, Philipps University Marburg)
Sylvain ENGILBERGE (Institut de Biologie Structurale, Université Grenoble Alpes, CEA, CNRS, Grenoble, France)
Pei-Jyun LIU (Department of Chemistry, National Taiwan University)
Thierry GIRAUD (European Synchrotron Radiation Facility)
Ming-Daw TSAI (Institute of Biological Chemistry, Academia Sinica, Taiwan)
Lars-Oliver ESSEN (Department of Chemistry, Philipps University Marburg)
Antoine ROYANT (European Synchrotron Radiation Facility)
Manuel MAESTRE-REYNA (Department of Chemistry, National Taiwan University)

Order-disorder transitions allow many proteins to shift between compact and flexible/unfolded states, thereby acting as structural switches that regulate catalysis, signaling, recognition, and allostery. We applied time-resolved serial oscillation crystallography to map the progression of a light-triggered order-disorder transition in an animal-like crypto-chrome as a six-second-long molecular movie with all-residue near-atomic and millisecond resolutions. Our 29 structural snapshots established the structural kinetics of disorder propagation across the tertiary structure motifs of a cryptochrome's C-terminal region, from a single broken D321-R492 salt bridge. The order-disorder transition was slow, requiring up to five seconds, due to regions distal from the D321-R492 trigger requiring slow zero-order disordering before unfolding in a kinetic regime switch, a prerequisite for ultrasensitivity. Our per-residue density analysis provides a detailed structural-kinetic basis for the emergence of order-disorder transitions via directional propagation from across protein tertiary structure, as occurring in signaling, and many other biological systems.

Capabilities and highlights of the SPB/SFX Scientific Instrument at the European XFEL

Raphael DE WIJN (European X-ray Free Electron Laser Facility GmbH)
Johan BIELECKI (European X-ray Free Electron Laser Facility GmbH)
Juncheng E (European X-ray Free Electron Laser Facility GmbH)
Konstantin KHARITONOV (European X-ray Free Electron Laser Facility GmbH)
Chan KIM (European X-ray Free Electron Laser Facility GmbH)
Yoonhee KIM (European X-ray Free Electron Laser Facility GmbH)
Jayanath KOLIYADU (European X-ray Free Electron Laser Facility GmbH)
Faisal KOUA (European X-ray Free Electron Laser Facility GmbH)
Romain LETRUN (European X-ray Free Electron Laser Facility GmbH)
Diogo MELO (European X-ray Free Electron Laser Facility GmbH)
Tomas POPELAR (European X-ray Free Electron Laser Facility GmbH)
Tokushi SATO (European X-ray Free Electron Laser Facility GmbH)
Marcin SIKORSKI (European X-ray Free Electron Laser Facility GmbH)
Egor SOBOLEV (European X-ray Free Electron Laser Facility GmbH)
Patrik VAGOVIC (European X-ray Free Electron Laser Facility GmbH)
Mohammad VAKILI (European X-ray Free Electron Laser Facility GmbH)
Lena WORBS (European X-ray Free Electron Laser Facility GmbH)
Richard BEAN (European X-ray Free Electron Laser Facility GmbH)

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, including the recent launch of the Quick Access Screening Time (QUAST), a broad-brush approach to sample screening and rapid access data collection. 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. To conclude, an outlook to future capabilities and upgrades will be shown.

Visualizing the B12-dependent photoreceptor CarH in action with time-resolved electron cryo-microscopy

María DÁVILA (Institut de Biologie Structurale)
Ronald RÍOS SANTACRUZ (Institut de Biologie Structurale)
Alok BHARADWAJ (Department of Bionanoscience, Kavli Institute of Nanoscience, Delft University of Technology, Delft, Netherlands)
Wiel EVERS (Department of Bionanoscience, Kavli Institute of Nanoscience, Delft University of Technology, Delft, Netherlands)
Arjen JAKOBI (Department of Bionanoscience, Kavli Institute of Nanoscience, Delft University of Technology, Delft, Netherlands)
Guy SCHOEHN (Institut de Biologie Structurale)
Giorgio SCHIRÒ (Institut de Biologie Structurale)
Félix WEIS (Institut de Biologie Structurale)
Martin WEIK (Institut de Biologie Structurale)

Time-resolved cryo-electron microscopy (cryo-EM) is a technique that allows to take structural "snapshots" of biomolecules in action, by starting the reaction with a trigger, such as light, and then "freeze-trapping" reaction intermediates by vitrification after a well-defined, yet variable time delay. Unlike time-resolved X-ray crystallography, time-resolved cryo-EM does not require crystals and can therefore capture large conformational changes. Here we present an example of the complementarity of the two techniques, in the study of the later stages of the photoreaction of the bacterial transcription factor CarH, a vitamin B12–dependent photoreceptor. While time-resolved X-ray crystallography is limited to early time points (<10 ms) due to large domain movements disrupting the crystal packing, time-resolved cryo-EM offered us a way to freeze-trap intermediate states at later stages of the photoreaction (up to 600 ms) and determine their high-resolution structures . In particular, we found a previously unobserved reaction intermediate, adding new details to our understanding of the CarH photoactivation mechanism.

Ultrafast electron diffraction study of insulator to metal photoinduced phase transition

Damien LÉA (Institut de Physique de Rennes - Université de Rennes/CNRS (UMR 6251))
Hilaire MBA (Institut de Physique et Chimie des Matériaux - Université de Strasbourg/CNRS (UMR 7504))
Gael PRIVAULT (Institute of Pure and Applied Sciences, University of Tsukuba)
Riku SEIKI (Department of Materials Science, Institute of Pure and Applied Sciences, University of Tsukuba)
Matthieu PICHER (Institut de Physique et Chimie des Matériaux - Université de Strasbourg/CNRS (UMR 7504))
Arnaud ARBOUET (CNRS, Univ Rennes, DYNACOM - IRL2015, The University of Tokyo)
Florian BANHART (Institut de Physique et Chimie des Matériaux - Université de Strasbourg/CNRS (UMR 7504))
Hiroko TOKORO (Department of Materials Science, Institute of Pure and Applied Sciences, University of Tsukuba)
Masaki HADA (Institute of Pure and Applied Sciences, University of Tsukuba)
Maciej LORENC (Institut de Physique de Rennes - Université de Rennes/CNRS (UMR 6251))
Philippe RABILLER (Institut de Physique de Rennes - Université de Rennes/CNRS (UMR 6251))

In recent years, quantum materials attracted attention for their unusual properties, one of which is that ultrafast light can drive them into new, out-of-equilibrium states, enabling potential applications through controllable property changes such as phase transitions[1]. Ti_3O_5 is one of such materials, which can undergo a reversible photoinduced insulator-to-metal transition (IMT) from a semiconducting phase to a metastable metallic phase[2]. This strongly first order transition is isostructural but involves a substantial change of the c-lattice parameter ($\Delta c \sim 6\%$), while the a and b-lattice parameters change by less than 1%[3,4]. The importance of laser-induced elastic deformations for the propagation of the phase transition at ultrafast timescale in polycrystalline samples has been demonstrated recently[3]. However, direct proof through investigation of isolated nanoscale objects to unveil the effects of sample morphology along with said elastic effects, and their coupling to thermal effects, is still lacking.

To investigate the IMT dynamics in single crystal samples, we have conducted ultrafast electron microscopy and diffraction measurements on single-crystal nanoscale samples (~100nm thickness) prepared by focused ion beam milling and by ball-milling (~500nm diameter)[5]. We developed a series of tools to provide accurate phase identification and determine the sample misorientation. We also provide evidence of ultrafast phase transition at the ns-timescale. These results confirm the possibility of using tailor-cut samples to investigate the dependence of the phase transition on physical parameters, such as sample morphology or crystallographic orientation.

References:

[1] D. N. Basov, R. D. Averitt, and D. Hsieh, Nat. Mater., Nov. 2017, doi: 10.1038/nmat5017.
[2] S. Ohkoshi et al., Nat. Chem., Jul. 2010, doi: 10.1038/nchem.670.
[3] C. Mariette et al., Nat. Commun., Feb. 2021, doi: 10.1038/s41467-021-21316-y.
[4] H. Tokoro et al., Nat. Commun., May 2015, doi: 10.1038/ncomms8037.
[5] Y. Hu et al., J. Phys. Chem. C, Aug. 2024, doi: 10.1021/acs.jpcc.4c02685.

Time-resolved Crystallography as a Probe for Nonenzymatic RNA Copying

Jahmyl ESSEX (University of Chicago)

Modern life is thought to have emerged from an earlier RNA world. RNA is uniquely capable of acting as both an informational molecule and a catalyst, so it is reasonable that an RNA oligonucleotide, such as an autocatalytic RNA polymerase (replicase), could have driven replication before modern-day protein-catalyzed replication. Before the advent of a replicase—itself, the product of extensive evolution—replication must have been carried out by simpler, nonenzymatic processes.1 Nonenzymatic copying on a primer-template complex driven by chemically activated nucleotides is a potential candidate for early ribonucleotide replication.2

Several fundamental questions about this process remain unanswered. Prebiotic syntheses of nucleotides produce racemic mixtures. How does the presence of one enantiomer affect copying by the other? High concentrations of Mg^{2+} are required for nonenzymatic copying, yet this is both prebiotically unrealistic and damaging to RNA and potential primordial membranes. Can this requirement be circumvented? And how do nucleotide mismatches at the primer-template junction affect the reaction center?

Time-resolved X-ray crystallography offers a way to address these questions at the molecular level. By soaking activated nucleotides into the lattice of a self-complementary RNA duplex that serves as a primer-template mimic, and freezing crystals at defined points along the reaction trajectory, we obtain atomic-resolution snapshots of nonenzymatic RNA copying in action. This approach allows us to directly observe how chiral interference, metal-ion identity, and mismatched junctions influence nucleotide binding, alignment, and chemistry at the active site. Together, these snapshots provide a molecular framework for understanding the constraints on early replication and for guiding the design of more prebiotically plausible reaction conditions.

Ultrafast electron diffraction study of insulator to metal photoinduced phase transition

Damien LÉA (Institut de Physique de Rennes - Université de Rennes/CNRS (UMR 6251))
Hilaire MBA (Institut de Physique et Chimie des Matériaux - Université de Strasbourg/CNRS (UMR 7504))
Gaël PRIVAULT (Institute of Pure and Applied Sciences, University of Tsukuba)
Riku SEIKI (Department of Materials Science, Institute of Pure and Applied Sciences, University of Tsukuba)
Matthieu PICHER (Institut de Physique et Chimie des Matériaux - Université de Strasbourg/CNRS (UMR 7504))
Arnaud ARBOUET (CNRS, Univ Rennes, DYNACOM - IRL2015, The University of Tokyo)
Florian BANHART (Institut de Physique et Chimie des Matériaux - Université de Strasbourg/CNRS (UMR 7504))
Hiroko TOKORO (Department of Materials Science, Institute of Pure and Applied Sciences, University of Tsukuba)
Masaki HADA (Institute of Pure and Applied Sciences, University of Tsukuba)
Maciej LORENC (Institut de Physique de Rennes - Université de Rennes/CNRS (UMR 6251))
Philippe RABILLER (Institut de Physique de Rennes - Université de Rennes/CNRS (UMR 6251))

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References:

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[4] H. Tokoro et al., Nat. Commun., May 2015, doi: 10.1038/ncomms8037.
[5] Y. Hu et al., J. Phys. Chem. C, Aug. 2024, doi: 10.1021/acs.jpcc.4c02685.

Capturing enzymes in action using functional crystallography

Julien MARTEL (Institut de Biologie Structurale, Université Grenoble Alpes, CEA, CNRS, Grenoble, France)
Nicolas CARAMELLO (Institut de Biologie Structurale, Université Grenoble Alpes, CEA, CNRS, Grenoble, France)
Antoine ROYANT (Institut de Biologie Structurale, Université Grenoble Alpes, CEA, CNRS, Grenoble, France)
Eric MATHIEU (Institut de Biologie Structurale, Université Grenoble Alpes, CEA, CNRS, Grenoble, France)
Lucas PETIT (Institut de Biologie Structurale, Université Grenoble Alpes, CEA, CNRS, Grenoble, France)
Philippe JACQUET (Institut de Biologie Structurale, Université Grenoble Alpes, CEA, CNRS, Grenoble, France)
Alexandre APPOLAIRE (Institut de Biologie Structurale, Université Grenoble Alpes, CEA, CNRS, Grenoble, France)
Dominique MADERN (Institut de Biologie Structurale, Université Grenoble Alpes, CEA, CNRS, Grenoble, France)
Sylvain ENGILBERGE (Institut de Biologie Structurale, Université Grenoble Alpes, CEA, CNRS, Grenoble, France)
Antoine ROYANT (Institut de Biologie Structurale, Université Grenoble Alpes, CEA, CNRS, Grenoble, France)
Tristan WAGNER (Institut de Biologie Structurale, Université Grenoble Alpes, CEA, CNRS, Grenoble, France)

Time-resolved crystallographic methods provide a means to capture the structural dynamics that govern enzyme function. However serial approaches often remain experimentally challenging in terms of sample preparation and data processing, which can limit their accessibility for many laboratories. We have developed Time-Resolved Functional Rotation Crystallography (TR-FRX), a room-temperature single-crystal approach that combines conventional rotation data collection with rapid on-crystal ligand delivery. Initial applications demonstrate that TR-FRX can be used to resolve ligand binding and large-scale structural rearrangements on the hundreds of milliseconds to minutes timescales while using mostly standard synchrotron beamline instrumentation and minimal protein sample.

In this poster, we present the development of the TR-FRX workflow and its application to model systems that validate the method for capturing structural snapshots of catalytic reactions. These studies show that TR-FRX can detect sequential substrate binding and conformational rearrangements directly within crystals. Complementary UV-visible spectroscopy measurements in crystal were used to monitor reaction progression, guide crystallographic data-collection parameters, and compare reaction kinetics between spectroscopic and structural measurements.

We also outline the next phase of the project, which focuses on extending TR-FRX to enzymes involved in biological pathways responsible for atmospheric carbon fixation, where key mechanistic intermediates remain poorly characterized. Ongoing work aims to improve temporal resolution through multi-crystal data-merging strategies and simplify data interpretation through improved processing scripts and analysis workflows. Together, these developments position TR-FRX as a broadly accessible method for studying enzyme mechanisms.

SANS, SAXS, WAXS of soft matter and biomaterials

Mme Isabelle MORFIN (Laboratoire Interdisciplinaire de physique)

From soft matter to biomaterials, neutron and X-ray scattering techniques are powerful tools for observing complex multiscale architectures, as well as the effects of various stresses on these structures. Using different examples, we will explore SAXS, SANS, ASAXS, and WAXS techniques, highlighting their complementarity and/or specific features.
The complementarity between SANS and SAXS will be illustrated through the self-assembly of pluronic micelles, hyaluronic acid and essential oil, while ASAXS applied to soft matter will be discussed for a system of charged DOTAB micelles in presence of hyaluronic acid. Focusing on wood and its derivatives, the complementarity between WAXS and SAXS will be highlighted through the study of materials subjected to mechanical constraints or chemical reactions that induce structural changes.
Each of these examples will provide an opportunity to highlight the capabilities of several large scales facilities in France, with particular emphasis on the French CRG beamline BM02/D2AM at the ESRF, where both high-throughput setups and dedicated sample environments enable a wide range of in-situ simultaneous SAXS-WAXS measurements.

From WAXS to SAXS: experiences at the Montpellier X-ray beamline

Arie VAN DER LEE (PAC Chimie Balard, Univ Montpellier, CNRS, ENSCM, Montpellier, France)
Karim AISSOU (IEM - Université de Montpellier - CNRS)
Laura MARTY (Institut Charles Gerhardt, UMR 5253 CNRS-UM-ENSCM)
Gaulthier RYDZEK (Institut Charles Gerhardt, UMR 5253 CNRS-UM-ENSCM)
Romain VIENNOIS (Institut Charles Gerhardt, UMR 5253 CNRS-UM-ENSCM)
Elise VAIRON (Institut Charles Gerhardt, UMR 5253 CNRS-UM-ENSCM)
Mickael BEAUDHUIN (Institut Charles Gerhardt, UMR 5253 CNRS-UM-ENSCM)
Corine GERARDIN (Institut Charles Gerhardt, UMR 5253 CNRS-UM-ENSCM)

This presentation aims to give an overview of some of the recent research activities with data collected at the diffusion and diffraction setup of the Analysis and Characterisation Platform (PAC Chimie Balard Montpellier, UAR2041) of the Chemistry Department of the University of Montpellier. The detector to sample distance can be varied between approximately 50 and 1600 mm, giving the possibility to access a q-range between 0.03 nm^{-1} and 39 nm^{-1}. The minimal accessible q-value depends also on the beamsize and the experimental set-up. The specificity of the set-up and data analysis software lies in the fact that standardless absolute intensity calibration can be performed as validated with measurements on NIST's SRM3600 standard. We present examples of typical SAXS measurement of colloids, in particular of polymer-stabilized gold nanoparticles, of different sizes and shape and the formation of supracolloidal assemblies of gold nanoparticles. The formation of block copolymer membranes with double gyroid nanochannels was followed by (GI)SAXS measurements. Typical examples of (GI)WAXS measurements on semiconducting TBT polymers and chitosan/collagen-based hydrogels are shown. We also show how scaleless specular reflectivity measurements can be carried out with this set-up on all kinds of thin films.

Polymorphic selection of biclotymol in thin films

Gabin GBABODE (Université de Rouen Normandie)
Clementmanohar ARAVA (Université de Rouen Normandie)

Biclotymol is an active pharnaceutic ingredient (API) used as an antiseptic for Ear, Nose and Throat (ENT) affections. It is known to exhibit 2 polymorphic forms, named forms I and II, form I being the more stable at all temperatures up to its melting temperature (monotropic relationship). Form II can be obtained by crystallization from the amorphous solid upon an appropriate thermal treatment (melting and quench below biclotymol's glass tranition -around 20°C-). The goal of the present study was to explore the possibilities of crystallization in a geometrically confined media such as thin films to, not only favor the selection of the metastable form II in a simple step but also to be able to keep this polymorphic form for longer times than in conventional powder (reconversion to the stable form I occurs in a week). Considering metastable forms in the final drug formulation is of high interest as they inherently possess higher solubility than the stable crystal forms. Furthermore, crystallization in gemotrically confined media allows to reduce the crystal size which directly impacts the dissolution rate of the drug. Overall, this crystallization method might lead to drug products with enhanced efficacy.

Thin films of biclotymol have been fabricated via solvent-based depositon methods such as drop-casting and spin-coating, involving the use of small amount of solution (100 µL per film). The films have been deposited on pre-cleaned glass or silicon wafer substrates from biclotymol solutions of concentrations varying from 0.001 M to 0.5 M in ethanol, methanol and acetone. It appeared that performing the thermal treatment on drop casted films allowed to obtain form II reproducibly whatever the concentration. Furthermore, grazing incidence X-ray diffraction performed at ESRF showed that form II was always observed for the films obtained from solution concentration below a certain threshold, that is, the thinnest films.

Nanoscale Structuring and Crystallization Mechanisms of Uranium–Monoamide Systems: Insights into Solid Third Phase Formation

Mme Elise GUERINONI (ICSM, Univ Montpellier, CEA, CNRS, ENSCM, Marcoule, France)

The recycling of spent nuclear fuel requires efficient solvent extraction systems, in which monoamides have emerged as promising alternatives for uranium recovery. However, under high metal loading, these systems may exhibit the formation of a solid third phase, associated with the crystallization of uranium–monoamide complexes, potentially affecting process stability.

This work focuses on understanding the mechanisms leading to precipitation in uranium-loaded monoamide organic phases. Particular attention is given to the nanoscale organization of the solution prior crystallization and pathways governing the formation of solid phases. The influence of monoamide structure, especially alkyl chain variations, is investigated to assess its role in supramolecular interactions and aggregation behavior.

A combination of SAXS/WAXS/ XRD and spectroscopic techniques is used to probe structural changes across different length scales, from molecular organization in solution to crystalline order in the precipitated phase. These approaches provide insight into the relationship between nanoscale structuring and macroscopic phase behavior.

This study aims to contribute to a better understanding of precipitation phenomena in monoamide-based liquid-liquid extraction systems and to support the development of more robust processes for nuclear fuel recycling.

Etude de la dynamique macromoléculaire du Polylactide par Spectroscopie de correlation de photons X

M Grégory STOCLET (Université de Lille)
Frédéric LIVET (SIMAP)
Nils BLANC (Institut Néel)
Gilbert CHAHINE (SIMAP)
Maxime DUPRAZ (ANAXAM)

La spectroscopie de corrélation de photons X (XPCS) s'est imposée comme une méthode de choix pour l'étude des phénomènes dynamiques à l'échelle nanométrique dans une grande variété de matériaux. Cette avancée est rendue possible grâce aux progrès des sources de rayonnement synchrotron et à la modernisation des lignes de faisceaux. La récente mise à niveau de la source du synchrotron (ESRF - EBS) à Grenoble, en France, a considérablement accru la brillance et la longueur de cohérence du faisceau, facilitant ainsi les mesures par XPCS. Ce travail présente une configuration expérimentale dédiée, développée sur la ligne de lumière D2AM de l'ESRF, permettant d'effectuer simultanément des mesures par XPCS et par diffraction des rayons X à grand angle (WAXS). De plus, les potentialités de l'XPCS sont illustrées à travers l'étude du polylactide, en mettant l'accent sur l'influence de la température, du degré de cristallinité et de l'orientation macromoléculaire sur la dynamique des chaînes. Ce travail constitue une contribution originale à la compréhension de l'influence de la cristallinité sur la dynamique macromoléculaire, démontrant le potentiel de la XPCS pour l'étude approfondie des processus dynamiques dans les polymères.

Révéler la nature et les techniques de mise en forme des alliages anciens par SR-DRX

Mme Emilie BÉRARD (Université Paris-Saclay, CNRS,ICMMO)

Dans le domaine de la métallurgie, l'étude de la sélection des matériaux et des savoirs techniques de mise en forme est d'un grand intérêt pour enrichir notre compréhension des sociétés du passé. Si les approches fondées sur l'analyse des productions matérielles ont déjà montré leur efficacité, les analyses métallographiques couramment utilisées sont basées sur la réalisation de prélèvements, rarement autorisées sur des objets muséaux. Dans les cas favorables, elles demeurent limitées à l'étude d'échantillon de faible taille (millimétrique). Afin d'augmenter la représentativité des mesures sur les métaux anciens par nature hétérogènes, le développement d'analyses non invasives est primordial. La diffraction des rayons X est un outil de choix dans ce domaine. L'identification des phases cristallines et l'étude de la forme et largueur des pics renseignent à la fois sur la nature du métal mais également sur les traitements thermomécaniques de mise en forme.

Cette contribution présentera au travers d'un cas d'étude, la méthodologie développée sur les lignes DiffAbs-SOLEIL et ID22-ESRF, pour l'étude de pièces d'armures nurembergeoises du début du XVIe siècle. Grâce à la spécificité du rayonnement synchrotron et à une acquisition rapide, la multiplication des analyses sur DiffAbs a permis d'évaluer la variabilité des alliages utilisés (composition chimique, traitements thermiques...) à l'échelle de chaque objet. Afin d'augmenter la représentativité des analyses en épaisseur, un dispositif de balayage de déformation en mode haute résolution angulaire, a également été testé sur ID22. Ces mesures ont permis de mettre en évidence la présence d'assemblage de matériaux ferreux aux propriétés distinctes. Les résultats couplés et discutés avec des examens métallographiques, soulèvent de nouvelles questions quant aux procédés de fabrication des objets et aux choix réalisés par les artisans.

Multi-scale structural analysis of historical inorganic pigments

Dr Victor GONZALEZ (CNRS)

Omnipresent in paintings since the Antiquity, inorganic pigments are key materials of art history. Collecting accurate chemical information on them is essential to achieve a better understanding of ancient pictorial practices, as well as to develop new conservation strategies. However, this objective faces several scientific challenges. First, pigments were obtained in the past following complex chemical syntheses, whose parameters are not always known to us. Secondly, painters were combining these materials in variable formulations, and applied these formulations with their own unique artistic techniques, resulting in a strong heterogeneity of the hybrid [pigment(s) + binder] paint systems. Finally, paintings are dynamic objects: chemical interactions within paint layers can result in the *in-situ* formation of non-original organo-metallic and/or inorganic compounds. The presence of these neo-formed materials can threaten the optical and/or physical integrity of artworks.

This communication will present recent research aimed at deciphering the past synthesis, formulation by artists, and potential alteration mechanisms of historical pigments. A special focus will be put on the advantages of multi-scale chemical analysis to tackle the chemical complexity of the composite paint systems. At the micro-scale, the analytical power of synchrotron radiation, notably using structural analysis via X-ray Powder Diffraction (XRPD), enables to discriminate between the multiple inorganic compounds present in paint layers, but also to provide detailed information on their composition and microstructure. At the macro-scale, the development of X-ray based structural imaging prototypes enables the charting of crystalline species on the entire surface of historical paintings. The communication will illustrate the complementarity of structural and molecular data collected at the multi-scale on carefully design model samples, historical paint fragments and entire artworks. The case of lead and cobalt-based pigments and their associated alteration products will be specifically discussed.

La porosité de l'hématite comme archive thermique : vers un paléothermomètre de l'hématite de synthèse en archéométrie

Killian FOURIE (Aix-Marseille Université,Madirel,CINaM)
Olivier GRAUBY (Aix-Marseille Université, CNRS ,CINaM)
Daniel FERRY (Aix-Marseille Université, CNRS ,CINaM)
Marie-Vanessa COULET (Aix-Marseille Université, CNRS, Madirel, CINaM)
Vasile HERESANU (Aix-Marseille Université, CNRS,CINaM)

L'hématite est un pigment naturel rouge. Cependant, un traitement thermique de la goethite (????–FeOOH) au-delà de 250°C la transforme en hématite [1], qui pourrait alors être l'un des premiers pigments synthétisés par l'Homme, comme en témoignent des pigments de goethite pseudomorphosée en hématite dans la grotte du Moulin (Troubat ; France), occupée dès 45000 ans av. J-C. [2] Contrairement à l'hématite naturelle, l'hématite synthétisée par chauffage de goethite présente une porosité qui évolue avec la température. [3]

Nous étudions l'évolution des caractéristiques dimensionnelles et topologiques de la porosité de la goethite avec la température afin de définir un paléothermomètre de l'hématite de synthèse. Des cristaux de goethite sont pyrolysés à des températures comprises entre 250°C et 900°C puis étudiés par tomographie électronique en transmission. Les reconstructions tridimensionnelles obtenues à partir des séries tomographiques sont analysées pour déterminer les caractéristiques dimensionnelles et topologiques de la porosité et leur évolution avec la température.

Nous mesurons par thermogravimétrie la perte d'eau induite lors du changement de structure de la goethite en hématite et caractérisons les phases cristallines par diffraction de rayons X.

L'analyse des reconstructions tomographiques révèle une augmentation progressive i) de la taille des pores, ii) de la porosité et iii) de la surface spécifique jusqu'à un maximum à 700°C, au-delà duquel la porosité et la surface spécifique diminuent. Nos résultats permettent de déterminer si un pigment d'hématite est d'origine naturelle ou synthétique, et la température maximale atteinte par la goethite transformée.

Nous montrons que la tomographie électronique en transmission permet à la fois de quantifier les caractéristiques physiques 3D de la porosité de l'hématite et leur évolution avec la température et d'établir un paléothermomètre pour l'étude de vestiges archéologiques.

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Characterisation of pigments from the Otello shelter (Bouches-du-Rhône): a study that is not so simple, in which the combination of X-ray diffraction and fluorescence is essential

Coline THÉRON (Environnements, Dynamiques et Territoires de la Montagne (Edytem), Centre National de la Recherche Scientifique : UMR5204 / FRE 2641, Université Savoie Mont Blanc, Centre National de la Recherche Scientifique : UMR5204)

Pierre BORDET (Univ. Grenoble Alpes, Centre National de la Recherche Scientifique, Grenoble Institute of Engineering Univ. Grenoble Alpes, Institut Néel, 38000 Grenoble, France)

Emilie CHALMIN (Environnements, Dynamiques et Territoires de la Montagne (Edytem), Centre National de la Recherche Scientifique : UMR5204 / FRE 2641, Université Savoie Mont Blanc, Centre National de la Recherche Scientifique : UMR5204)

Claudia DEFRASNE (Environnements, Dynamiques et Territoires de la Montagne (Edytem), Centre National de la Recherche Scientifique : UMR5204 / FRE 2641, Université Savoie Mont Blanc, Centre National de la Recherche Scientifique : UMR5204)

Pauline MARTINETTO (Univ. Grenoble Alpes, Centre National de la Recherche Scientifique, Grenoble Institute of Engineering Univ. Grenoble Alpes, Institut Néel, 38000 Grenoble, France)

The Otello shelter is visible from a distance; its reddish-orange floor stands out against the limestone massif of the Alpilles. It is upon entering the cavity that forms the upper part of the site that one notices the presence of over 300 schematic painted figures dating from the Neolithic period. Interpreting the wall paintings is not straightforward, due to layers of overpainting and weathering, and no artefacts have been found at the site.

These enigmatic graphic remains raise many questions: what is the origin of these paintings? When and how often did people come here to paint? How did they go about it? Did they use local materials, particularly those found on the ground?

The characterisation of matter is one of the key avenues for providing answers to these questions.

We investigated the material by combining in situ X-ray fluorescence spectroscopy from synchrotron analysis (ESRF) of existing micro-samples (Hameau 2010) with X-ray diffraction (XRD) at high angular (ID22) and spatial (ID13) resolution.

This multi-analytical approach enables us, despite the superimposition and interweaving of natural and anthropogenic processes, to (i) characterise the phases comprising the paint material (kaolinite, iron and titanium oxides) and (ii) disentangle the stratigraphy (limestone substrate, sulphate and carbonate-type weathering).

This understanding of the phases and their positions within the stratigraphy enables us to envisage the construction of a multi-layer model to compare not only the stratigraphy but also the composition of the paint at different points on the wall. The paint material is also compared with soil samples from the rock shelter to determine the origin of the materials used, in particular by utilising the precision of high-angular-resolution X-ray diffraction (XRD) diagrams, which allow the microstructure to be investigated.

Non-Negative Matrix Factorization for 4D-STEM data analysis and phase mapping of astromaterials samples

Bahae-Eddine MOULOUD (CNRS, UMR 8207-UMET-Unite Matériaux et Transformations, Université de Lille)
Maya MARINOVA (CNRS, FR 2638-IMEC-Institut Michel-Eugene Chevreul, Université de Lille)
Sylvain LAFORET (CNRS, FR 2638-IMEC-Institut Michel-Eugene Chevreul, Université de Lille)
Corentin LE GUILLOU (CNRS, UMR 8207-UMET-Unite Matériaux et Transformations, Université de Lille)
Hugues LEROUX (CNRS, UMR 8207-UMET-Unite Matériaux et Transformations, Université de Lille)
Francisco DE LA PENA (CNRS, UMR 8207-UMET-Unite Matériaux et Transformations, Université de Lille)
Damien JACOB (CNRS, UMR 8207-UMET-Unite Matériaux et Transformations, Université de Lille)

4D-STEM has become a robust method for exploring materials at the nanometric scale. It enables the mapping of various physical properties, such as phase, orientation, strain, or magnetization, fully exploiting the information contained in diffraction patterns. However, due to the vast amount of data generated in a typical 4D-STEM experiment, automatic data processing methods are generally required for analysis. In particular, several combinations of dimensionality reduction and blind source separation algorithms, such as Singular Value Decomposition (SVD) and Non-Negative Matrix Factorization (NMF) have been demonstrated.

We introduce an innovative approach for analyzing meteorite (Orgueil) and asteroid (Ryugu, from the recent JAXA Hayabusa2 mission [5]) samples. These samples, beam sensitive, are characterized by a phyllosilicate-rich matrix, where fine-grained constituents form a nanometer-scale mixture, leading to overlapping diffraction patterns from different phases and orientations. 4DSTEM data sets have been acquired with a hybrid-pixel direct electron detector located behind a Gatan energy filter on a Thermo Fisher Titan 300kV instrument with a 10 nm probe size.

A first data treatment, producing a Bragg Vector Map (BVM) of the area under investigation together with its azimuthal integration, proved efficient for separating the various diffraction peaks contributing to the entire data set. Nevertheless, their indexation and classification as components of all the existing phases appeared quite tedious. As an improvement, we applied the NMF procedure on the azimuthal profile of each diffraction pattern pre-processed as a BVM. The resulting first components, whose number was given by an initial SVD procedure applied on the profiles set, where then more easily reassociated and combined to constitutive distinct phases. A robust phase map could then be reconstructed based on a limited number of components.

Modulation of the modulated magnetic structure of an Ho i-MAX phase

Claire COLIN (Université Grenoble Alpes)
Rafik BALLOU (Univ. Grenoble Alpes, CNRS, Grenoble INP, Institut Néel)
Tao QUANZHENG (Department of Physics, Chemistry and Biology, Linkopings Universitet)
Johanna ROSEN (Department of Physics, Chemistry and Biology (IFM), Linkopings Universitet)
Christine OPAGISTE (Univ. Grenoble Alpes, CNRS, Grenoble INP, Institut Néel)
Thierry OUISSE (Laboratoire des Matériaux et du Génie Physique)
Vaclav PETRICEK (Institute of Physics of the Czech Academy of Science)

Magnetic structures constitute a vast and particularly rich field, where significant complexity arises from commensurate or incommensurate modulations of the magnetic moment in direction or amplitude. It is therefore not uncommon to encounter structures in which several modulations coexist, the most typical examples being so-called multi-k structures. These correspond to magnetic configurations in which more than one arm of the star of the propagation vector k contributes to the spin arrangement, and are often associated with topologically non-trivial textures such as hedgehog and skyrmion states. In contrast, the coexistence of propagation vectors not related by symmetry remains rare, except in specific cases such as conical structures.
In this presentation, we focus on the Ho-based i-MAX phase, $(Mo_{2/3}Ho_{1/3})_2GaC$, which exhibits highly unusual magnetic behavior. To our knowledge, it represents a rare example where two incommensurate propagation vectors, not symmetry-related, coexist without phase separation. An additional peculiarity is that one modulation appears to be modulated by the other, as the satellites of the second wavevector are only observed around those of the first.
Neutron powder diffraction reveals that the magnetic ordering develops in two stages: first, a transition to an amplitude-modulated structure, followed by a second transition introducing modulation in a perpendicular direction. The resulting structure can be described as a longitudinal amplitude modulation that is itself modulated in a perpendicular direction. Remarkably, this complex arrangement is accurately captured using the superspace-group formalism with only a limited number of parameters, highlighting the strength of this approach. The microscopic origin of these stabilized magnetic phases will also be discussed.

C.V. Colin, Q. Tao, C. Opagiste, R. Ballou, J. Rosen, T. Ouisse and V. Petrıcek (2025). Acta Cryst. B81, https://doi.org/10.1107/S2052520624011053.

Incommensurate organic hydroxy channel structures: when the first-order satellite reflections are as strong as the main reflections

Arie VAN DER LEE (IEM - Université de Montpellier - CNRS)
Ioan STROIA (IEM - Université de Montpellier - CNRS)
Li-Bu HUANG (IEM - Université de Montpellier - CNRS)
Erol LICSANDRU (IEM - Université de Montpellier - CNRS)
István KOCSIS (IEM - Université de Montpellier - CNRS)
Mihail DELEANU (IEM - Université de Montpellier - CNRS)
Christian HÜBSCHLE (Bruker AXS)
Mihail BARBOIU (IEM - Université de Montpellier - CNRS)
Olivier PÉREZ (CRISMAT, CNRS, Normandie University, ENSICAEN, UNICAEN)

(In)commensurately modulated crystal structures are usually characterized by a set of strong main reflections representing a 3D average structure and a set of much weaker satellite reflections that give information about the structural modulations with respect to the basic structure. We present here two cases of related modulated organic hydroxy-channel compounds for which the first-order are (nearly) as strong as the main reflections. This gives severe problems in the initial indexing of the diffraction pattern: which are the main reflections and which are the satellite reflections? We propose that the ratio between the mean intensity of the satellite intensities and that of the main reflections is a measure of the average strength of the modulation in an (in)commensurate crystal structure. This idea is applied to a database of 117 other organic and inorganic modulated structures. In the particular case of the two hydroxyl channel structures the origin of the modulation is sought in the competition between strong orthogonal urea-urea and hydroxy-hydroxy interactions. We also present an alternative method to graphically represent a structural modulation in its basic unit cell; it combines a usual ball-and-stick representation of the basic structure with Lissajous curves for the spatial modulation mapped to the basic unit cell. The curves are colour-mapped with the modulation *t*-parameter which allows to see immediately which individual modulation displacements are in phase and which are not.

Local structure and dynamics of nanoconfined water and electrolyte solutions

Hassan KHODER (ICSM, Univ Montpellier, CEA, CNRS, ENSCM, Marcoule, France)
El-Eulmi BENDEIF (Synchrotron SOLEIL)
Bertrand SIBOULET (ICSM, Univ Montpellier, CEA, CNRS, ENSCM, Marcoule, France)
Diane REBISCOUL (ICSM, Univ Montpellier, CEA, CNRS, ENSCM, Marcoule, France)

Nanoporous materials are ubiquitous in many fields and consist of nanoscale-confined media typically filled with water and ions. Under such restricted environments, the properties of water deviate significantly from their bulk counterpart, strongly impacting reactivity, transport, and stability. These deviations arise from the combined effects of geometrical confinement, interfacial interactions, and the presence of solutes, leading to complex structural and dynamical behavior. Understanding these effects is essential for predicting the macroscopic behavior of nanoporous systems during operation and aging.

We investigate the structural organization of water confined in mesoporous silica using total X-ray scattering coupled with pair distribution function (PDF) analysis. Distinct structural signatures are identified, revealing distortions of the hydrogen-bond network, particularly within the interfacial region [1]. We then investigate the water dynamics by performing quasi-elastic neutron scattering experiments on water confined in anisotropic silica mesopores. These measurements provide access to directional-dependent diffusion, showing significant differences between motions parallel and perpendicular to the confining interfaces [2].

Finally, we extend this framework to electrolyte solutions under confinement. We show, using quasi elastic neutron scattering, that water dynamics depend on the nature (kosmotropic vs. chaotropic) and valency (monovalent vs. divalent) of the ions, revealing ion-specific effects particularly in the interfacial region [3]. X-ray scattering experiments were performed on these samples, and PDF analyses are currently being processed to probe the structural distortions induced by the ions.

This combined structural and dynamical approach provides new insights into the interplay between local structure and dynamics in nanoconfinement, offering a deeper understanding of the processes occurring at solid-aqueous interfaces.

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Complémentarité entre les simulations théoriques et résultats expérimentaux : applications aux matériaux photo-commutables

Fantine MASSICOT (CRM2 - Université de Lorraine)
Dominik SCHANIEL (CRM2 - Université de Lorraine)
El-Eulmi BENDEIF (CRM2 - Université de Lorraine)

Les matériaux hybrides de type photoswitch@MOF (Metal–Organic Frameworks) constituent une classe prometteuse de matériaux fonctionnels combinant une matrice poreuse cristalline et des molécules photosensibles capables de modifier réversiblement leurs propriétés sous irradiation lumineuse. Toutefois, la compréhension des relations entre structure, dynamique et propriétés dans ces systèmes hôte–invité reste un défi majeur en raison du désordre et de la mobilité des molécules invitées au sein de la matrice.
Ce travail de thèse propose de développer une approche intégrée visant à caractériser la structure et les interactions dans ces matériaux en s'appuyant sur une comparaison directe entre simulations théoriques et résultats expérimentaux. Des données de diffusion totale des rayons X analysées par fonction de distribution de paires (PDF) permettent de caractériser expérimentalement l'organisation structurale de ces matériaux à différentes échelles. En parallèle, des simulations de dynamique moléculaire obtenues par la méthode SCC-DFTB sont mises en œuvre afin de modéliser théoriquement le comportement des molécules photosensibles confinées dans les matrices MOFs. L'objectif est de confronter ces modélisations aux observations expérimentales afin d'identifier les motifs d'interaction hôte–invité et de mieux décrire la dynamique locale des systèmes de ce type. Cette démarche sera explicitée à travers les résultats obtenus sur le système spiropyrane@MOF5.
Cette comparaison étroite entre théorie et expérience vise à établir des corrélations entre structure atomique et propriétés optiques des matériaux, apportant ainsi des éléments de compréhension essentiels pour le contrôle de leurs performances.

Reference:

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Electronic factors governing the modulated vs. periodic structural and spin lattices in the BaMP2O7 series

Olivier MENTRÉ (UCCS-CNRS)
Hicham NIMOH (UCCS-CNRS)
Angel-Moise AREVALO-LOPEZ (UCCS-CNRS)
Claire COLIN (Institut NEEL CNRS/UGA UPR2940)
Isabelle BRAEMS (Institut des Matériaux de Nantes)
Robert GLAUM (Université Bonn)

The $BaM^{2+}P_2O_7$ (M= Cr to Zn) series constitutes a particularly rich playground, displaying unexpected structural complexity together with unconventional magnetic and magnetoelectric responses [1-6]. Structurally, these phases are characterized by spatially well-isolated one-dimensional chains built from metal dimers. It offers a versatile platform for diverse magnetic ground states [2,5] and, in some cases, multiferroic behavior [4]. Although this series has not been addressed in its globality from a comprehensive perspective, a striking trend emerges from individual case studies: depending on the nature of the M^{2+} cation, the structures exhibit either pronounced aperiodic modulations (M = Mn, Fe, Co, Ni) or regular, non-modulated lattices (M = Cr, Cu, Zn), without any obvious critical tolerance factor based solely on ionic radii arguments. In the aperiodic members, the incommensurate modulation is associated with unusually large atomic displacement waves, which locally alter the coordination of the transition metal between MO5+1 and distorted MO6 polyhedra. Such strong lattice distortions induce substantial spin–lattice coupling effects. This interplay is reflected in magnetic structures that are directly influenced by the structural modulation, either already at zero field [4] or along metamagnetic transitions [1,4]. On the basis of the novel compound BaCrP2O7, we will establish the raises the fundamental question of whether unified microscopic parameters govern the entire $BaMP_2O_7$ series. A central objective of the present work is therefore to elucidate how the nature of the 3d M cation controls the stabilization of either a modulated or a regular crystal structure, relying on conventional electronic-structure considerations and lattice-dynamical arguments.

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Dynamic disorder in crystalline solids revealed by broadband dielectric spectroscopy

Natalia T. CORREIA (UMET, Université de Lille)
Fabian PUGA MONTESDEOCA (UMET, Université de Lille)
Florence DANÈDE (UMET, Université de Lille)
Frederic AFFOUARD (UMET, Université de Lille)

Dynamic disorder in crystalline materials often arises from local molecular motions that remain hidden to diffraction based analyses focused on average long range order. Broadband dielectric spectroscopy (BDS), through its sensitivity to dipolar relaxations over wide frequency and temperature domains, provides direct access to these fluctuations and to the associated local structure. Two representative systems are examined to illustrate this complementarity. In plastic crystalline caffeine, BDS reveals the characteristic reorientational dynamics of the high symmetry orientationally disordered phase. In contrast, a channel like carbamazepine cocrystal exhibits a distinct dielectric response dominated by localized motions constrained by the anisotropic host framework. By correlating these dielectric signatures with structural motifs, BDS emerges as a powerful tool for characterizing dynamic disorder, probing local mobility regimes, and refining our understanding of structure-dynamics relationships in complex crystalline solids.

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