

50 ANS D'AVENTURE XAFS EN FRANCE

des pionniers du LURE
aux sources de 4^e génération

X - FS50.FR

50 YEARS OF XAS
IN FRANCE



PROGRAMME
DE RECHERCHE
MATÉRIAUX
ÉMERGENTS



PROGRAMME
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Ondes et de la
Matière

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Bienvenue

C'est avec un réel plaisir que nous vous accueillons au synchrotron SOLEIL à l'occasion de la conférence XAFS50.FR. Cet évènement se veut une célébration des 50 ans de la spectroscopie XAFS en France. Durant cinq jours, nous retracerons le parcours accompli depuis les pionniers du LURE, qui ont développé les expériences et donné aux communautés scientifiques françaises l'accès aux formalismes du XAFS. Dès son émergence, le XAFS a été adopté par de nombreuses communautés qui en ont fait très rapidement une technique pluridisciplinaire à fort impact sur les avancées scientifiques dans leurs domaines.

Grâce à l'investissement du comité scientifique et du comité local d'organisation, nous vous avons préparé un programme particulièrement riche.

La première journée sera consacrée à retracer ces débuts et à faire partager l'enthousiasme de cette époque fondatrice par les chercheuses et chercheurs qui ont permis à tant d'entre nous de pratiquer la spectroscopie d'absorption des rayons X. La visite de Sciences ACO, le musée de la Lumière et de la Matière, classé site de l'European Physical Society et inscrit à l'inventaire supplémentaire des monuments historiques de France, conclura ce moment fort en souvenirs où les anecdotes croiseront l'Histoire des Sciences.

Les conférences et posters présentés lors des trois jours qui suivront montreront les évolutions de la spectroscopie XAFS, intimement liées à celle du rayonnement synchrotron, ainsi que les apports de la technique pour différentes communautés scientifiques. La place de la théorie et de ses progrès au cours de ces 50 ans occupera également une part importante du programme.

Transmettre a été une force motrice de ces 50 années d'évolution de la technique, à travers des écoles et des développements de logiciels portés par les pionniers, et dont le développement comme l'enseignement se perpétuent toujours aujourd'hui. C'est pourquoi nous avons souhaité proposer, pour la dernière journée de la conférence, deux formations aux simulations de l'EXAFS et du XANES, dispensées par les auteurs des programmes.

L'organisation de cette conférence n'aurait pu se faire sans la générosité des sponsors et la présence des exposants. Nous remercions en particulier le synchrotron SOLEIL de l'accueillir en ses murs et de contribuer à son organisation financière et logistique. Nous remercions également la région Île de France qui, grâce au soutien financier du DIM PAMIR, a permis de proposer la visite de Sciences ACO et l'apéritif qui s'ensuivra, inscrivant ainsi l'évènement dans cette dimension de l'« Histoire des Sciences » que nous souhaitons. Le soutien financier du DIM MaTeRe, des PEPR DIADEM et LUMA, montre combien la spectroscopie d'absorption des rayons X est devenue une technique majeure dans l'arsenal des outils de caractérisation des matériaux, notamment ceux à fort impact sociétal.

Enfin, nous vous remercions tous pour avoir répondu présent à l'évènement et nous espérons que vous profiterez de ces journées avec autant de plaisir que nous avons eu à les organiser.

Valérie Briois et Delphine Vantelon



October 5th - 9th, 2026
Synchrotron SOLEIL

Welcome

It is with great pleasure to welcome you to SOLEIL Synchrotron for the XAFS50.FR conference. This event celebrates 50 years of modern XAFS spectroscopy in France. Over the course of five days, we will trace the journey taken since the days of the LURE pioneers, who developed the first experiments and provided the French scientific community with access to XAFS formalisms. From its inception, XAFS was adopted by numerous scientific communities, rapidly evolving into a multidisciplinary technique that has significantly impacted advances in their respective fields.

Thanks to the commitment of the scientific and local organizing committees, we have prepared a particularly rich program for you.

The first day will be devoted to retracing those early years and sharing the enthusiasm of this pioneering period with the researchers who enabled so many of us to practice X-ray absorption spectroscopy. This day, rich in memories and personal anecdotes, will conclude with a visit to *Sciences ACO*, the Museum of Light and Matter, a site recognized by the European Physical Society and listed in France's Supplementary Inventory of Historic Monuments.

The talks and posters presented over the following three days will highlight the evolution of X-ray absorption spectroscopy, closely intertwined with the development of synchrotron facilities, as well as the contributions of the technique to a broad range of scientific communities. These sessions will also illustrate the essential role of theory and the major advances in theoretical approaches over the past 50 years.

Finally, knowledge transfer has been a driving force behind the 50 years of development of XAFS spectroscopy, and it has also been a central motivation in the preparation of this conference. For this reason, we have chosen to devote the final day to two training sessions dedicated to EXAFS and XANES simulations.

This conference could not have taken place without the generosity of our sponsors. We would like to warmly thank SOLEIL for hosting the event and for its substantial contribution to its organization. We are also very grateful to the Île-de-France Region, whose financial support through the DIM PAMIR is contributing to the visit to "*Sciences ACO*" and the reception that will follow, thereby giving the event the dimension of the "History of Science" that we wished to emphasize. Financial support from DIM MaTeRre and the PEPR DIADEM and LUMA highlights just how vital X-ray absorption spectroscopy has become within the toolkit for characterizing materials—particularly those with significant societal impact.

Finally, thank you all for attending this event; we hope you enjoy these days as much as we have enjoyed organizing them.

Valérie Briois et Delphine Vantelon



October 5th - 9th, 2026
Synchrotron SOLEIL

Committees

Conference Chairs

- Valérie Briois – *UR1-CNRS, SOLEIL synchrotron*
- Delphine Vantelon – *SOLEIL synchrotron*

Scientific Committee

- Christophe Den Auwer – *Université Côte d'Azur*
- Valérie Briois – *UR1-CNRS, SOLEIL synchrotron*
- Delphine Cabaret – *Sorbonne Université*
- Sylvain Cristol – *Université de Lille*
- Emiliano Fonda – *SOLEIL synchrotron*
- Jean-Louis Hazemann – *Institut Néel*
- Amélie Juhin – *Sorbonne Université*
- Karine Provost – *Université Paris Est Créteil*
- Jérôme Rose – *Aix-Marseilles Université*
- Delphine Vantelon – *SOLEIL synchrotron*

Local Organizing Committee

- Anthony Beauvois – *SOLEIL synchrotron*
- Sylvie Bonnardel – *SOLEIL synchrotron*
- Amélie Bordage – *Université Paris-Saclay*
- Valérie Briois – *UR1-CNRS, SOLEIL synchrotron*
- Cheila Cleland – *SOLEIL synchrotron*
- Mary-Anna Destermes – *SOLEIL synchrotron*
- Camille Enjommet – *SOLEIL synchrotron*
- Frédérique Fraissard – *SOLEIL synchrotron*
- Angéla Lambert – *SOLEIL synchrotron*
- Gautier Landrot – *SOLEIL synchrotron*
- Solenn Reguer – *SOLEIL synchrotron*
- Delphine Vantelon – *SOLEIL synchrotron*
- Stéphanie Yao – *SOLEIL synchrotron*



Practical information

ACO museum visit (Orsay)

Monday, October 5th, 2026 at 18:30

Bus transfer in front of the SOLEIL reception building



The museum visit is funded by the Région Ile de France

Conference Dinner at La Coupole (Paris)

Tuesday, October 6th, 2026 at 20:00

Bus transfer at 18:30 in front of the SOLEIL reception building

Posters

Posters can be installed on Monday afternoon until the end of the conference

Photobooth

Photobooth will be available on Monday and Wednesday afternoons



**October 5th - 9th, 2026
Synchrotron SOLEIL**

Sponsors



IRELEC

ALCEN

Ecole de Spectroscopie d'Absorption X

14-18 Juin 2027 – Université de Montpellier



La spectroscopie d'absorption des rayons X (SAX) est une technique puissante de caractérisation de la structure locale de la matière. Elle est aujourd'hui largement utilisée dans de nombreux domaines : chimie, physique, catalyse, matériaux, géologie, pharmacologie ou sciences de l'environnement.

Cette école propose une formation complète aux **principes théoriques et instrumentaux de la SAX, ainsi qu'à l'analyse des données**. Une place importante est consacrée aux **travaux pratiques d'analyse et d'interprétation des données EXAFS**. L'objectif est de donner aux participants les connaissances et compétences nécessaires pour **analyser de manière autonome leurs propres spectres EXAFS**.

Public concerné : chercheurs, ingénieurs et doctorants en chimie, physique, biologie et sciences des matériaux, débutants ou souhaitant actualiser et approfondir leurs connaissances.

Renseignements :

Jean Louis Bantignies : Jean-louis.bantignies@umontpellier.fr

Christophe Den Auwer : christophe.denauwer@univ-cotedazur.fr



Exhibitors



MIRION
TECHNOLOGIES



Quantum Design

Program

Monday, October 5th, 2026

From LURE to the 4th Synchrotron Radiation facility History and Driving Concepts

SOLEIL Auditorium – Main Building

13:30 – 14:00 *Registration*

13:55 – 14:00 Welcome
Valérie Briois, Delphine Vantelon – UR1-CNRS & Synchrotron SOLEIL, France

14:00 – 14:35 Les dix premières années de l'EXAFS à LURE
Pierre Lagarde, Denis Raoux – Synchrotron SOLEIL, France

14:35 – 15:10 XAFS de surface et XAFS dispersif ; l'usage essentiel de la polarisation du photon dans le champ naissant des interfaces et multicouches
Dominique Chandesris, Alain Fontaine – CNRS, France

15:10 – 15:27 XAFS in France before synchrotron facilities
Christian Brouder – Sorbonne University, Paris, France

15:27 – 15:45 Apprendre à se connaître : Rayonnement synchrotron et sciences de la Terre à la fin du XX^e siècle
Georges Calas – IMPMC, Paris, France

15:45 – 16 :00 First in situ XAFS investigation of the electrochemical interface, DCI/LURE 1991
Abderrahmane Tadjeddine – Institut de Chimie Physique, CNRS

16:00 – 16:30 *Coffee Break setting up in front of the SOLEIL Auditorium - Main Building*

16:30 – 17:05 La spectroscopie d'absorption sous pression : une spécialité française, du LURE à SOLEIL en passant par l'ESRF et SLS
Alain Polian, Jean-Paul Itié – IMPMC, Sorbonne University & Synchrotron SOLEIL, France

17:05 – 17:40 Chemistry & XAFS@LURE at the edge of the 80s, Science, People, Driving Concepts
Christophe Cartier Dit Moulin, Michel Verdaguer, Françoise Villain – IPCM, CNRS Sorbonne Université, Paris, France

17:40 – 18:15 The Journey of XAFS beamlines from the 1st to the 4th generation of Synchrotron Radiation Facilities
Jean Daillant, Michèle Sauvage – ESRF & Synchrotron SOLEIL, France

18:30 – 20:30 Museum visit - ACO (Anneau de Collision d'Orsay)
18:30 - *Bus departure in front of the SOLEIL reception building*



The museum visit is funded by the Région Île de France

Tuesday, October 6th, 2026

XAFS Contribution to the Past and Today Science

SOLEIL Auditorium – Main Building

- | | |
|-----------------|---|
| 09:00 – 09:05 | Introduction |
| 09:05 – 10:00 | XAFS Theory: 50 Years of Breakthroughs
John Rehr – <i>Department of Physics. University of Washington, United States</i> |
| 10:00 – 10:35 | XAS utilization in glass studies: potentialities, difficulties and blind corners
Stéphanie Rossano – <i>LGE, Université Gustave Eiffel, France</i> |
| 10 :35 – 11 :00 | <i>Coffee Break setting up in front of the SOLEIL Auditorium - Main Building</i> |
| 11:00 – 11:35 | X-ray Spectroscopy at the European X-ray Free Electron Laser
Sakura Pascarelli – <i>European XFEL, Germany</i> |
| 11:35 – 11 :53 | First-principles XANES calculations within the pseudopotential framework: 17 years of XSpectra
Delphine Cabaret – <i>Sorbonne Université, IMPMC, France</i> |
| 11 :53 – 12 :29 | XAFS in liquids and disordered systems: early and recent advances from LURE times to today
Andrea Di Cicco – <i>Università di Camerino, Italy</i> |
| 12:30 – 14:00 | <i>Lunch</i> |
| 14:00 – 15:30 | Parallel sessions – 2 different rooms <ul style="list-style-type: none">• Disordered Materials & Cultural Heritage – <i>SOLEIL Auditorium – Main Building</i>• Catalysis – <i>SOLEIL Auditorium – Reception Building</i> |
| 15 :30 – 15 :55 | <i>Coffee Break setting up in front of the SOLEIL Auditorium - Main Building</i> |
| 15:55 – 16:30 | Evolution of Synchrotron XAS for Heterogeneous Catalysis: From early studies to operando strategies and data analysis, at IFPEN
Christèle Legens – <i>IFP Energies Nouvelles, France</i> |
| | Meet our sponsors and exhibitors <ul style="list-style-type: none">• Advanced Detection Solutions for Synchrotron Applications
Greateyes – Fenton Mann – 10'• Iliad: A Next-Generation EELS Platform for X-ray Absorption Spectroscopy–Like Information in the TEM by Thermofisher - Arno Meingast – 10'• Dispersive Lab-based X-ray Spectrometers from LynXes by LynXes Innovation - Zoltán Németh – 10'• PEPR DIADEM• PEPR LUMA• DIM MaTerRe• DIM PAMIR |
| 16:30 – 17:40 | |
| 17:40 – 18:30 | Poster session |
| 18:30 | <i>Bus departure in front of the SOLEIL reception building</i> |
| 20:00 – 23:30 | <i>Conference diner at La Coupole</i> |

Parallel Session

Disordered Materials & Cultural Heritage

SOLEIL Auditorium – Main Building

- 14:00 – 14:18 Local Structure in Silicate Glasses and Melts at Extreme Conditions
Max Wilke – *Universität Potsdam, Germany*
- 14:18 – 14:36 From liquid to solid: The contribution of XANES at the structure and properties of glasses and melts
Daniel Neuville – *IPGP CNRS, France*
- 14:36 – 14:54 Comparative Pt L₃-edge XAS study of Pt nanoparticles with different morphologies in solution and tumor cells
Raul Magro Hernandez – *UCM, Spain*
- 14:54 – 15:12 XAFS to study Cultural Heritage Metallic Materials
Solenn Reguer – *Synchrotron SOLEIL, France*
- 15:12 – 15:30 Harnessing X-rays to Reveal Historical Productions in Late Iron Age Britain
Susan Nehzati – *Diamond Light Source, United Kingdom*

Catalysis

SOLEIL Auditorium – Reception Building

- 14:00 – 14:18 Catalyst Design Guided by XAS Spectroscopy
Xavier Carrier – *Sorbonne Université, France*
- 14:18 – 14:36 XAS's contribution to IRC(IRCELYON) research in the field of catalysis
Christophe Geantet – *CNRS, France*
- 14:36 – 14:54 Operando XAFS Reveals Interfacial Redox Chemistry of Supported Metal Catalysts as the Switch Descriptor Between Base- and Tip-Growth Carbon Filaments During Catalytic Methane Splitting
Wilson Henao – *Instituto de Tecnología Química (ITQ), Spain*
- 14:54 – 15:12 Combined XAFS/DRIFTS/MS: a powerful technique for operando catalysts investigations
Davide Salusso – *ESRF, France*
- 15:12 – 15:30 Monitoring the Formation of a Single-Atom Catalyst with Operando Quick XAFS
Franck Morfin – *IRCELYON, France*

Wednesday, October 7th, 2026

Theory and Data Analysis – Technique Evolutions

SOLEIL Auditorium – Main Building

- 09:00 – 09:05 Introduction
- 09:05 – 10:00 Simulating X-ray Absorption Spectroscopies Can Be Useful
Yves Joly – Institut Néel. Université Grenoble Alpes, France
- 10:00 – 10:35 X-ray Spectromicroscopy or Microspectroscopy? A Methodological Perspective
Jean Susini – Synchrotron SOLEIL, France
- 10:35 – 11:00 Coffee Break - setting up in front of the SOLEIL amphitheater
- Parallel sessions** – 2 different rooms
- 11:00 – 12:30
- [Energy Related Materials](#) – SOLEIL Auditorium – Main Building
 - [Geosciences Part I](#) – SOLEIL Auditorium – Reception Building
- 12:30 – 14:00 Lunch
- 14:00 – 14:35 X-ray Absorption Spectroscopy: A Key to Deciphering and Optimizing Battery Materials
Laurence Croguennec – ICMCB, France
- 14:35 – 14:53 Understanding chemical bonds with valence RIXS
Oana Bunau – ESRF, France
- 14:53 – 15:11 Orbital Engineering in Graphene/SiC: An ELNES and FDMNES Study
Vinod Kumar Paidi – CNRS, INSA Lyon, UCBL, MATEIS, UMR 5510, France
- 15:11 – 15:29 Unraveling Local Environment in Multicomponent Materials using Reverse Monte Carlo Simulations
Alexei Kuzmin – Institute of Solid State Physics, Latvia
- 15:29 – 15:47 Multiple-Edge EXAFS/RMC Refinement for 3D Local Structure in SrTiO₃
Andris Anspoks – Institute of Solid State Physics, Latvia
- 15:50 – 16:10 Coffee Break setting up in front of the SOLEIL Auditorium - Main Building
- 16:10 – 16:45 Dichroisms at LURE and SOLEIL
Philippe Saintavit – IMPMC. Sorbonne Université, France
- 16:45 – 17:03 Introducing « FITNESS »: a new program, built-in to Fastosh, to efficiently perform shell-by-shell fitting of the EXAFS
Gautier Landrot – Synchrotron SOLEIL, France
- 17:03 – 17:21 From Monometallic to Bimetallic NiCo/SiO₂: Element-Resolved Active-Site Identification by Operando XAS and MCR-ALS
Shuchi Zhang – Eindhoven University of Technology, Netherlands
- 17:21 – 17:39 Resolving the "Coloring Problem" in Crystallography: The Synergistic Power of EXAFS and Resonant Diffraction
Carmelo Prestipino – CRISMAT, France
- 17:39 – 17:57 EXAFS Unbound: Unlocking Atomic Scale Insights Beyond The Next Edge
Rene Bes – University of Helsinki, Finland

	In Memoriam Prof. Alain Michalowicz
17:57 – 18:20	Karine Provost – ICMPE. Université Paris Est Créteil, France Emiliano Fonda – Synchrotron SOLEIL, France
18:30 – 20:00	Poster session and Aperitif

Parallel Session

Energy Related Materials SOLEIL Auditorium – Main Building

11:00 – 11:18	From Ex Situ to Operando Soft X-rays Microscopy of Photoactive Materials: Case of Hematite-based Photoanodes Stefan Stanescu – Synchrotron SOLEIL, France
11:18 – 11:36	Operando XAS Strategies for Rational Design of Electrochemical Energy Materials Francesco Paparoni – Synchrotron SOLEIL, France
11:36 – 11:54	Tuning Redox Chemistry in Gd-Doped Ceria: Gd-Driven Suppression of Ce ³⁺ Formation in Reducing Condition Alessandro Longo – ESRF, France
11:54 – 12:12	The Optical Dispersive EXAFS (ODE) Beamline at Soleil Synchrotron and Its Studies Qingyu Kong – Synchrotron SOLEIL, France
12:12 – 12:30	Combined Operando XRD And XAS Study Of Electrochemically Active Li ₂ MChO Anti-perovskites Mikhail Gorbunov – Karlsruhe Institute of Technology, Germany

Geosciences Part I SOLEIL Auditorium – Reception Building

11:00 – 11:18	Coupling High-Energy Resolution Fluorescence Detected X-ray Absorption Spectroscopy (HERFD-XAS) with microfluidic cells for in situ monitoring of tungsten nanoparticles redox transformation Melanie Auffan – CEREGE CNRS, France
11:18 – 11:36	Vanadium enrichment in lateritic soils: mineralogical and pedoclimatic controls Myriam Boudieb – Sorbonne Université, France
11:36 – 11:54	Redox speciation of cerium in the presence of colloids Terjini Kiththangodage – IPGP, France
11:54 – 12:12	XAS Studies Of Metal Transport In Hydrothermal Fluids: Autoclaves And Photons Denis Testemale – Institut Néel, CNRS, France
12:12 – 12:30	Probing the Speciation of Transition Metals (Mo, W) in Hydrothermal Fluids by In-Situ XAS Maria Kokh – Institut für Geowissenschaften Universität Potsdam, Germany

Thursday, October 8th, 2026

Technique Evolutions – 4th Generation and Beyond

SOLEIL Auditorium – Main Building

09:00 – 09:05	Introduction
09:05 – 10:00	XAS Data Analysis in the Era of Machine Learning Janis Timoshenko – Fritz Haber Institute. Max-Planck-Gesellschaft, Germany
10:00 – 10:35	Tracing cadmium from soil to foodstuff using X-ray techniques Geraldine Sarret – Institut des Sciences de la Terre. Université Grenoble Alpes, France
10:35 – 11:00	Coffee Break setting up in front of the SOLEIL Auditorium - Main Building
	Parallel sessions – 2 different rooms
11:00 – 12:30	• Chemistry – SOLEIL Auditorium – Main Building • Geosciences Part II – SOLEIL Auditorium – Reception Building
12:30 – 14:00	Lunch
14:00 – 14:35	XAFS and nuclear fuels Philippe Martin – CEA Marcoule, DES/ISEC/DMRC, France
14:35 – 14:53	Beyond XAS with RIXS Jean-Pascal Rueff – Synchrotron SOLEIL, France
14:53 – 15:11	Study of the genesis of HDS catalysts by in situ photon-in photon-out spectroscopy Sylvain Cristol – Université de Lille, France
15:11 – 15:29	Understand 4f covalency by ab initio calculated RIXS plane Sihan Zhang – ESRF, France
15:29 – 15:47	Tender X-ray photon-in/photon-out spectroscopy at ESRF ID26 Pieter Glatzel – ESRF, France
15:50 – 16:10	Coffee Break setting up in front of the SOLEIL Auditorium - Main Building
16:10 – 16:45	Multi-scale characterization of environmental colloidal materials Laurent Michot – Laboratoire de PHENIX, France
16:45 – 17:03	Developments of the setup for pump-probe X-ray absorption fine structure (XAFS) and a capability of EXAFS at FXE instrument, European XFEL Yohei Uemura – European XFEL, Germany
17:03 – 17:21	FAME-PIX: The Pioneering French High-Resolution Hyperspectral 3D X-Ray Microscope at the ESRF Julio C. Da Silva – Institut Néel, CNRS, France
17:21 – 17:39	Operando Spatio-Temporal Study of CO oxidation reaction in Pd Catalysts using Full-Field Hyperspectral XAS imaging Marlon Muniz Da Silva – CNPEM, Sirius, Brazil
17:39 – 17:57	X-ray Absorption Spectroscopy meets Imaging to Unveil Inclusion Puzzles in Deep Diamonds Helio Tolentino – LNLS/CNPEM, Brazil
18:00	Conclusion & Goodbyes

Parallel Session

Chemistry

SOLEIL Auditorium – Main Building

- 11:00 – 11:18 Unraveling the fate of anticancer gold complexes in cells through HERFD-XAFS
Tom Lacoma – Sorbonne Université, France
- 11:18 – 11:36 A short retrospective of actinide hexacyanoferrate exploration initiated at LURE
Christophe Den Auwer – Université Côte d'Azur, France
- 11:36 – 11:54 Magnetic hyperthermia at the nanoscale
Jerome Fresnais – CNRS, Laboratoire PHENIX, France
- 11:54 – 12:12 Understanding Nanoparticles Formation mechanisms at ITODYS Laboratory: Insights from investigations at ROCK Beamline
Lorette Sicard & Marion Giraud – ITODYS, Université Paris Cité, France
- 12:12 – 12:30 Time- and Space-Resolved Formation Stannosilicates by in situ XAS
Aram Bugaev – Paul Scherrer Institute, Switzerland

Geosciences Part II

SOLEIL Auditorium – Reception Building

- 11:00 – 11:18 Redox speciation of contaminants at magnetite surface
Rémi Marsac – Institut de physique du globe de Paris, CNRS, France
- 11:18 – 11:36 Mineralogical Controls on the Fate of Emerging Contaminants in Laterites Revealed by XAFS
Quentin Bollaert – Synchrotron SOLEIL, France
- 11:36 – 11:54 Thermal transformation of clay minerals probed by in situ soft-X-ray absorption spectroscopy
Erwan Paineau – Laboratoire de Physique des Solides, CNRS, France
- 11:54 – 12:12 Assessing the Sensitivity of X-ray Absorption Spectroscopy to Distinguish Various Forms of Nucleic Acids and Their Surface Structures on Imogolite Nanotubes
Jerome Rose – CNRS CEREGE, France
- 12:12 – 12:30 X-ray absorption spectroscopy in surface science on LUCIA
Nicolas Trcera – Synchrotron SOLEIL, France

Friday, October 9th, 2026

Fastosh & FDMNES Tutorials

SOLEIL Auditorium – Main Building

09:30 – 12:00	Fastosh Tutorial - Simulation of EXAFS: a live introduction to FITNESS, a new software built-in to Fastosh Gautier Landrot – <i>Synchrotron SOLEIL, France</i>
12:00 – 13:30	<i>Lunch</i>
13:30 – 16:30	FDMNES Tutorial - Simulation Of XANES Using The FDMNES Code Yves Joly – <i>Institut Néel, CNRS, UGA</i>



From LURE to the 4th Synchrotron Radiation facility

History and Driving Concepts

SOLEIL Auditorium – Main Building

XAFS in France before synchrotron facilities

Christian Brouder

Sorbonne Université, Muséum National d'Histoire Naturelle, UMR CNRS 7590, Institut de Minéralogie, de Physique des Matériaux et de Cosmochimie, IMPMC, 75005 Paris, France

ABSTRACT

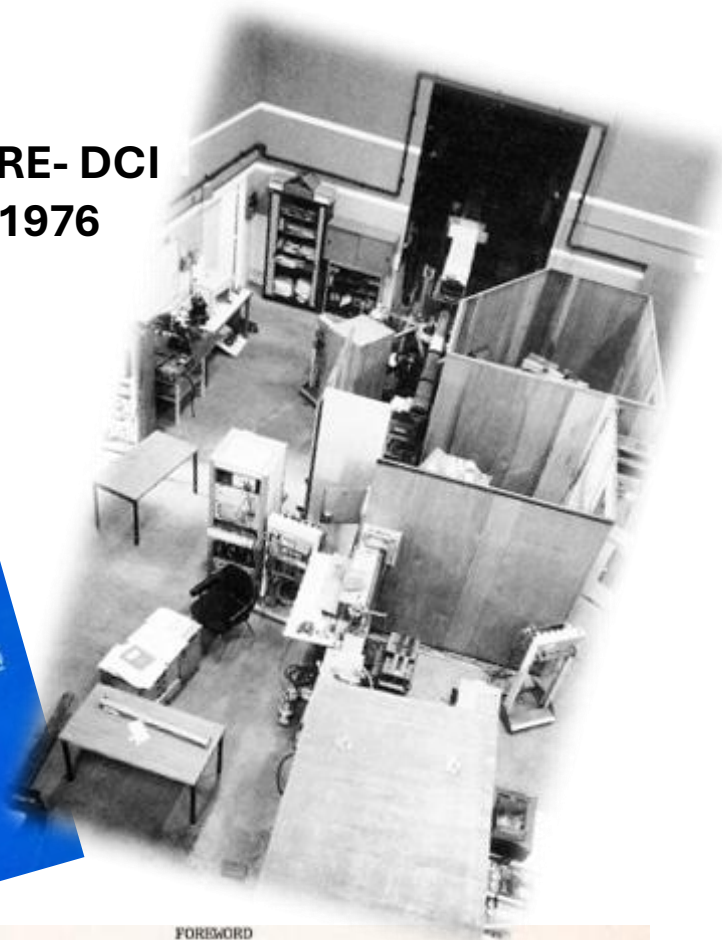
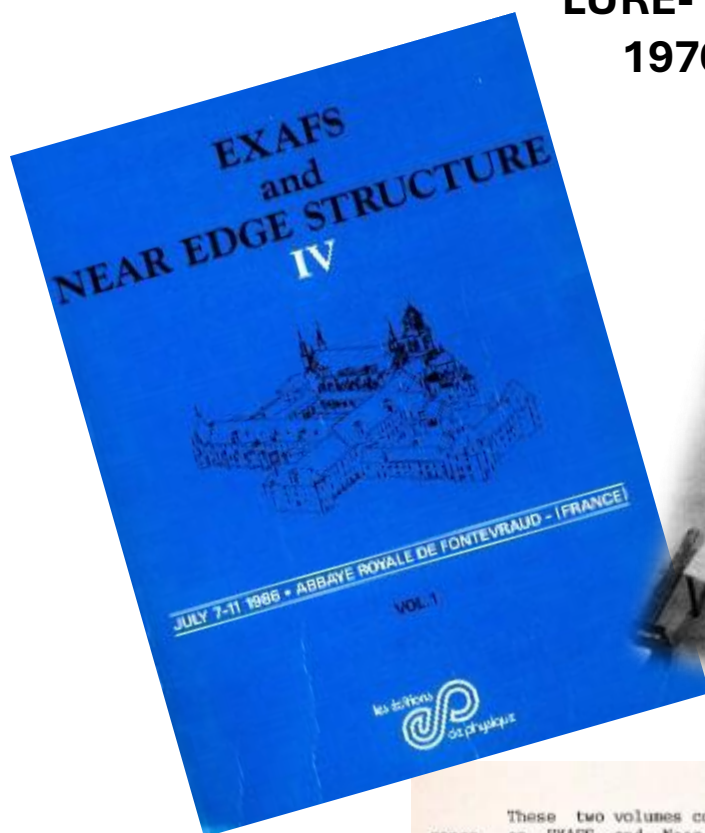
It is generally considered that the first x-ray absorption spectrum was obtained in 1913 by Maurice de Broglie [1]. Relying on the historical works by Roberte Stumm von Bordwehr [2] and others, we show that there is a continuous filiation, mainly due to Yvette Cauchois [3-5], from Maurice de Broglie to the first XAFS spectra measured at the LURE synchrotron facility.

We also point out some other French researchers, such as Constantin Kurylenko [6,7], who carried out interesting and forgotten contributions to x-ray spectroscopy.

REFERENCES

1. Lytle, F. W., *J. Synchrotron Rad.* **6**, 123-134, (1999).
2. Stumm von Bordwehr, R., *Ann. Phys. Fr* **14**, 377-466 (1989).
3. Cauchois, Y., *J. Phys. Rad.* **3**, 320-336 (1932).
4. Cauchois, Y., *J. Phys. Rad.* **3**, 320-336 (1932).
5. Cauchois, Y., Bonnelle, C., Missoni, G., *C. R. Acad. Sci. Paris*, **257**, 409-412 (1963).
6. Kurylenko, C., *Franges au voisinage de la discontinuité K des rayons X*, Ph. D. Thesis, Paris, 1939.
7. Kurylenko, C., *Ark. Fys.* **40**, 571-583 (1971)

LURE- DCI 1976



FOREWORD

These two volumes contain the proceedings of the fourth International Conference on EXAFS and Near Edge Structure held at Fontevraud, in the French Val-de-Loire, in July 1986. The interest among scientists in this technique, already remarkable in the previous meetings, has continued to rise steeply with the number of participants around 250 and the number of communications at nearly 300 : this represents more than a factor two over the last meeting (Stanford) which still was considered a great success. This large attendance forced us to manage parallel oral sessions and wide poster exhibits covering two sections simultaneously.

The classification we used during the meeting, and for the edition of these volumes now seems quite a classical one. Besides its use as a structural tool, the fundamental aspects of X-ray spectroscopy are still a subject of investigation through theoretical and experimental studies, mainly in the region close to the edge. Chapter I, "Fundamental Aspect of EXAFS and XANES", is quite important, revealing the efforts and the successes of scientists in explaining in detail the whole energy domain of the absorption spectrum.

Because of the recent development of dedicated synchrotron radiation facilities, and definite plans for new ones like the photon factory in Japan, ESRF or the 6 GeV American machine, the interest for new experimental tools is still strong and new experimental skills are appearing. We were surprised by the number of communications more or less devoted to experimental technique, and therefore we decided to dedicate one session of the meeting to this problem, gathering the results in Chapter II of the book.

Five chapters are devoted to material science : we have divided the studies of amorphous materials into covalent systems (chapter IV), metallic alloys (included in Chapter IX together with the metal studies) while those involving oxide and halide glasses have been grouped together in Chapter VII named "Inorganic Materials" which also covers the field entire of the chemistry of solids. The wide use of EXAFS as a standard structural technique is very apparent through the diversity and the number of subjects in all these domains.

EXAFS has always been powerful in two kinds of research : the study of catalysts and that of the metalloproteins in biophysics. As in the preceding Conferences, we have reserved a chapter for each of these subjects. The growth of surface science using synchrotron radiation facilities appears clearly in Chapter V which is devoted to this field. Finally edge studies, related of course to the new theoretical developments on the interpretation of the near edge structure, take up a separate chapter (VIII) in the important case where f-electrons are involved in the absorption process.

P. LAGARDE, D. RAOUX, J. PETIAU

First in situ XAFS investigation of the electrochemical interface: DCI/LURE 1991

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ABSTRACT

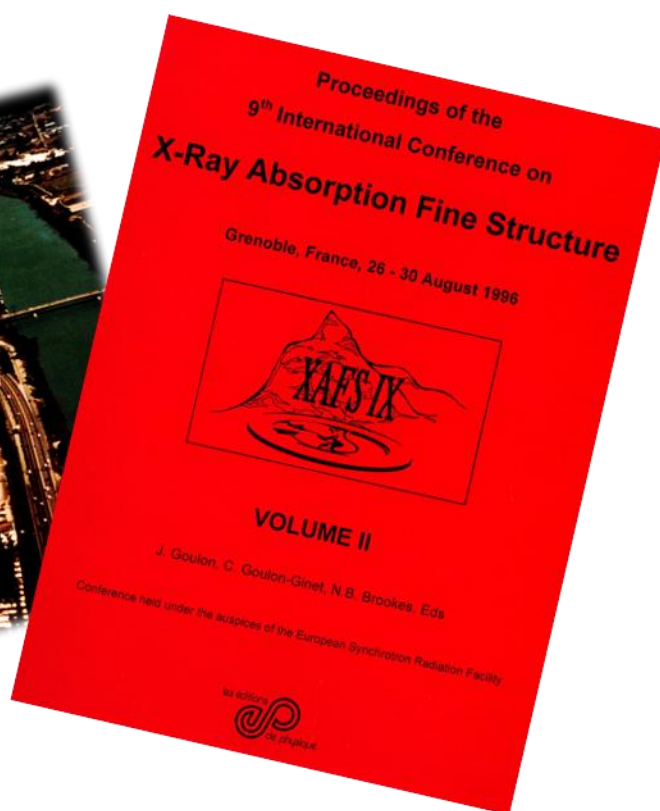
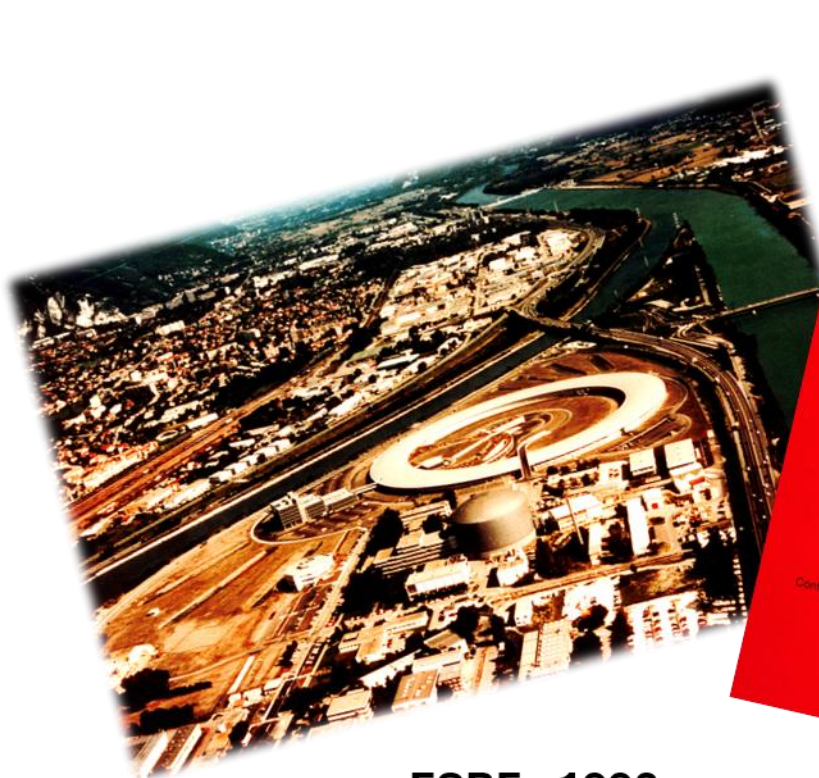
The development at LURE of a fluorescence detection system based on a plastic scintillator (1) has opened the possibility to investigate in situ, under potential control the electrochemical interface. We have designed a spectroelectrochemical cell to fit with the so-called "Detecteur Lemonnier" to study the underpotential deposition of Cu on gold in well-defined chemical conditions required by the long acquisition time of the spectra collection. The XANES data were collected in scans of 15 min with an energy step of 0.5 eV and added to obtain a good signal-to-noise ratio and ten scans (2.5h. For the EXAFS experiments recorded over a 600 eV energy domain, 15 scans of 20min each 55h° and an energy step of 2eV were averaged (2).

Preliminary test experiments were performed using (100) mono-oriented Au (100) sheet of 2 cm² area experiments. In situ X-ray absorption spectra of deposited monolayer and bilayer of copper immersed in an electrolyte solution were obtained using fluorescence detection under grazing incidence, the X-rays being polarized parallel to the gold surface. This study revealed two distinct structural configurations, depending on the Cu layer thickness.

We then moved to the investigation of Cu on Au(111) to investigate the electronic and structural properties of underpotentially deposited submonolayers under potential control (3). We found various ordered adlayer structures, dependent on coverage, time, and direction of the potential scan and a strong interaction of Cu and oxygen and charge transfer from Cu to Au. During the deposition, the initial (V3xV3) structure at 0.3 monolayer evolved to an equilibrium with the c(5x5). The latter and the (1x1) phase at 0.6 and 1 monolayer, respectively. The three structures are present at 0.6 monolayer during the stripping.

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ESRF - 1992

Preface

These two volumes regroup the Proceedings of the 9th International Conference on X-Ray Absorption Fine Structure (XAFS IX) held on August 26th-30th in Grenoble (France). The first International Conference focusing on XAFS was organised in 1981 in Daresbury (U.K.) as a "study week-end" with 80 participants. Fifteen years later, the 9th edition of this very successful series of conferences was organised by the European Synchrotron Radiation Facility (ESRF) and attracted over 550 registered delegates from all around the world. This rapid growth certainly reflects the vitality of the community of X-ray spectroscopists nearly one hundred years after the historical report by W.K. Röntgen of the discovery of X-rays and of their differential absorption by various elements in matter. The success of the XAFS IX Conference may perhaps be correlated to the fact that the ESRF was the first high energy synchrotron radiation source of 3rd generation which began user operation in 1994: it is not fortuitous, therefore, that the next conferences will be organized in 1998 by the user community of the American Advanced Photon Source and in 2001 by the Japanese user community of the new Spring-8 machine which is just starting operation.

José Goulon
Conference Chairman

Plenary Lectures

SOLEIL Auditorium – Main Building

Tuesday, October 6th, 2026

09:05 - 10:00 XAFS Theory: 50 Years of Breakthroughs
PL-01 **John Rehr** – *Department of Physics. University of Washington, United States*

Wednesday, October 7th, 2026

09:05 - 10:00 Simulating X-ray Absorption Spectroscopies Can Be Useful
PL-02 **Yves Joly** – *Institut Néel. Université Grenoble Alpes, France*

Thursday, October 8th, 2026

09:05 - 10:00 XAS Data Analysis in the Era of Machine Learning
PL-03 **Janis Timoshenko** – *Fritz Haber Institute. Max-Planck-Gesellschaft, Germany*



October 5th - 9th, 2026
Synchrotron SOLEIL

XAFS Theory: 50 Years of Breakthroughs

John J. Rehr

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ABSTRACT

There has been dramatic progress over the past five decades in the theory of x-ray absorption fine-structure (XAFS). Attaining an efficient and quantitative theory is challenging since the spectra cover a broad range of energy, length, and time-scales, where traditional methods like density functional theory (DFT) can become inadequate. As illustrated in the modern EXAFS equation, the theory needs to include many-electron effects such as inelastic losses, mean-free paths, and vibrational damping. Meeting these challenges required a number of theoretical and algorithmic breakthroughs. Here we present a chronological summary. Instead of wave-function based DFT approaches, a real-space Green's function (RSGF) approach is advantageous [1]. This real-space multiple scattering (RSMS) approach treats curved-wave scattering with effective scattering amplitudes f_{eff} and separable-propagators. The exchange-correlation functional of DFT is replaced with an energy-dependent self-energy to account for mean-free path damping. Further developments were needed to achieve quantitative *ab initio* treatments, including self-consistent scattering potentials, Dirac-Fock atom effects, core-hole screening, and correlated Debye-Waller factors. A major breakthrough over the past decade or so is the development of cumulant Green's function treatments of *intrinsic* inelastic losses such as the many-body amplitude factor S_0^2 in the EXAFS equation. Extensions to finite temperatures and related spectra are also briefly discussed.

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Simulating X-ray Absorption Spectroscopies Can Be Useful

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GOAL

The objective is to demonstrate how to obtain quantitative information on the chemical and physical properties of materials using *ab initio* simulations, by comparing them with experimental spectra.

SIMULATION OF X-RAY SPECTROSCOPIES

Depending on the absorption edge under consideration, various theoretical tools can be used to simulate X-ray spectroscopies. FDMNES is one such code based on density functional theory (DFT). We present its origins, key features, and capabilities, illustrated by a series of representative applications. These cover not only XANES and XMCD techniques but also related spectroscopies such as X-ray Raman scattering and resonant diffraction. We will see that each technique possesses specific characteristics that allow for the determination of distinct properties. All rely on the transition of a core electron to unoccupied states; consequently, their cross-sections depend on the electronic and magnetic structure of the material being probed. Thus, simulation enables us to obtain not only the expected spectra but also, potentially, the density of states and the degree of covalency between the absorbing atom and its ligands.

EXAMPLES

Three examples will be presented in greater detail. The first focuses on the study of hematite at room temperature under electric and magnetic fields. A very weak dichroic signal is observed upon reversal of the electric field. This signal changes sign when the magnetic field is reversed. We quantitatively reproduce all these features through simulation. We show that they are associated with very small atomic displacements and interpret the result in terms of electromagnetic multipoles. The second example concerns sulfur doping of TiO_2 (anatase phase). Various atomic models are used for the XANES simulations. We find that only a few of these models are plausible, implying the coexistence of two oxidation states: 6^+ and 2^- . The final example concerns an electrochemical interface between a Pt(111) electrode and an electrolyte, studied using surface-sensitive resonant X-ray diffraction. The so-called Helmholtz layer, located adjacent to the surface, can be analyzed in terms of composition (H_2O and H_3O^+), distance from the surface, and the resulting charge of the surface Pt layer.

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XAS Data Analysis in the Era of Machine Learning

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ABSTRACT

X-ray absorption spectroscopy (XAS) is a premier technique for tracking the structure of working functional materials due to its sensitivity to atomistic and electronic structure of the material, element specificity, and applicability to a broad range of different samples and experimental conditions [1]. Nonetheless, the interpretation of XAS data remains often challenging, and is particularly difficult, if the investigated material is a mixture of different species.

Recent breakthrough developments in data science methods provide a solution to this problem. In particular, the development of unsupervised machine learning methods and their combination with X-ray absorption near edge (XANES) spectroscopy is helpful for understanding the speciation of working functional materials [2]. At the same time, we have demonstrated that the supervised machine learning (SML) methods are well suited for quantitative analysis of XANES [3] but also EXAFS (Extended X-ray Absorption Fine Structure) spectra [4]. As we have recently demonstrated, the SML-EXAFS approach is particularly useful for understanding the evolving structures of complex heterogeneous materials, which feature absorbing sites in different crystallographic environments [5]. Alternatively, SML methods can also be used to guide the identification of the spectra for pure species from a series of spectra for heterogeneous mixtures, which at the next step can be analyzed using more simple approaches [6].

Here we demonstrate the potential of machine learning-based data analysis on an example of studies of working catalysts for electrocatalytic CO₂ conversion [4,6,7] and for oxygen evolution in alkaline electrolytes [5]. The compelling evidences about the nature of the catalyst active states, obtained from operando time-resolved XAS coupled with advanced data analysis approaches allow us to decouple the contributions of different species coexisting in working catalysts to the catalysts' functionality, and provide guidelines for the further optimization of catalytic systems and reaction protocols.

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Invited Talks

SOLEIL Auditorium – Main Building

Tuesday, October 6th, 2026

- 10:00 – 10:35 XAS utilization in glass studies: potentialities, difficulties and blind corners
IT-01 **Stéphanie Rossano** – LGE, Université Gustave Eiffel, France
- 11:00 – 11:35 X-ray Spectroscopy at the European X-ray Free Electron Laser
IT-02 **Sakura Pascarelli** – European XFEL, Germany
- 11:53 – 12:28 XAFS in liquids and disordered systems: early and recent advances from LURE times to today
IT-03 **Andrea Di Cicco** – Università di Camerino, Italy
- 15:55 – 16:30 Evolution of Synchrotron XAS for Heterogeneous Catalysis: From early studies to operando strategies and data analysis, at IFPEN
IT-04 **Christèle Legens** – IFP Energies Nouvelles, France

Wednesday, October 7th, 2026

- 10:00 – 10:35 X-ray Spectromicroscopy or Microspectroscopy? A Methodological Perspective
IT-05 **Jean Susini** – Synchrotron SOLEIL, France
- 14:00 – 14:35 X-ray Absorption Spectroscopy: A Key to Deciphering and Optimizing Battery Materials
IT-06 **Laurence Croguennec** – ICMCB, France
- 16:10 – 16:45 Dichroisms at LURE and SOLEIL
IT-07 **Philippe Saintavit** – IMPMC. Sorbonne Université, France
- 17:57 – 18:20 In Memoriam Prof. Alain Michalowicz
IT-08 **Karine Provost** – ICMPE. Université Paris Est Créteil, France
Emiliano Fonda – Synchrotron SOLEIL, France

Thursday, October 8th, 2026

- 10:00 – 10:35 Tracing cadmium from soil to foodstuff using X-ray techniques
IT-09 **Geraldine Sarret** – Institut des Sciences de la Terre. Université Grenoble Alpes, France
- 14:00 – 14:35 XAFS and nuclear fuels
IT-10 **Philippe Martin** – CEA Marcoule, DES/ISEC/DMRC, France
- 16:10 – 16:45 Multi-scale characterization of environmental colloidal materials
IT-11 **Laurent Michot** – Laboratoire PHENIX. Sorbonne Université, France



October 5th - 9th, 2026
Synchrotron SOLEIL

XAS utilization in glass studies: potentialities, difficulties and blind corners

Stéphanie Rossano

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ABSTRACT

Glass is a fascinating natural and synthetic material used since prehistorical times by Men and Women. Obsidians were shaped similarly to flint stones to produce tools. Since the Egyptian period, synthetic glass objects have been produced by Men and Women. At the beginning, small, precious, and rare objects were produced. With the evolution of raw materials, recipes and techniques, glass objects become transparent which is a key factor of science development in the Middle Ages with chemical glass ware and optical instruments for example. The Middle Ages is also an important period for glass windows in what is now northern Europe. With the industrial revolution, glass becomes more and more common with a wide range of uses that we know nowadays. However, the emergence of new glass compositions and uses are still empirical as the glass structure at the atomic scale remains largely unknown and the relationship between structure and properties unpredictable.

Exploring glass structure is thus still a necessity but is also very challenging. Disorder effects and the absence of long-range order hinder the possibility to extract structural information from powerful tools as X-ray diffraction. In this context, the chemical selectivity of XAS appeared to be very interesting to explore the structure from various points of view (at the edge of so called glass formers like Si or Al, but also minor elements, also called glass modifiers, used as structural probes like transition elements for example).

Based on different studies using EXAFS at the Fe K-edge, XANES at the Mg K-edge or at the Mn K-edge, pre-edge analysis at the Fe K-edge, we will explore the potentialities of XAFS – most of the time coupled to other techniques, to modelling or to calculation - to derive information on glass disorder, on element environment or oxidation state, and on glass alteration. For each example, experimental and analytical difficulties as well as blind corners will be discussed.

X-ray Spectroscopy at the European X-ray Free Electron Laser

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ABSTRACT

X-ray Free Electron Laser are opening novel avenues in the investigation of fundamental processes in many areas of science, from physics to chemistry, biology and materials science, because they allow investigation of matter at the time scales of electron and nuclear dynamics (down to fs), with chemical selectivity and bulk sensitivity.

In this talk I will briefly introduce the European XFEL and describe the XAFS and other X-ray spectroscopy platforms operational at both soft and hard X-ray instruments. I will report on recent scientific highlights in the fields of materials science, biochemistry, and high energy density science. I will also present our first attempts to investigate site-resolved electron dynamics in molecules by XAS. Finally, I will mention ongoing developments towards the generation and use of attosecond pulses and higher spectral densities.

XAFS in liquids and disordered systems: early and recent advances from LURE times to today

Andrea Di Cicco

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ABSTRACT

Some of the most interesting achievements in the application of the XAFS (X-ray Absorption Fine Structure) technique to liquids and disordered systems are reviewed from a personal perspective, including the pioneering works carried out by A. Filipponi and myself at LURE and in Frascati in the late 1980s and early 1990s. Those works were important in establishing the importance of applying the XAFS technique for measuring fine details of the short-range pair and higher-order distribution functions in disordered materials also under extreme high temperature and/or high pressure conditions. It is shown that the initial experiments (1989-1995) performed in collaboration with many LURE scientists¹ (see for example [2-6]) and interpreted with novel and advanced multiple-scattering methods [7,8] were really inspirational also for further applications carried out later on at the ESRF, Elettra, Soleil and at many other modern light sources including FELs.

In this communication, the continued efforts in studying liquids and disordered systems by XAFS are reviewed by examples of applications to selected substances, with a special emphasis on the first achievements obtained at LURE. Experimental and data-analysis methods, proven to be successful in dealing with highly disordered systems, are briefly summarized highlighting the potential of the XAFS technique. Recent experiments and results obtained under high-pressure and/or high temperature conditions are also mentioned [9] in connection with present experimental and computational capabilities.

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¹ A non-exhaustive list includes (co-authors) J. P. Itié, A. Polian, P. Lagarde, A. M. Flank, and later on F. Baudelet.

Evolution of Synchrotron XAS for Heterogeneous Catalysis: From early studies to operando strategies and data analysis, at IFPEN

Christèle Legens

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ABSTRACT

Over the past twenty years, X-ray absorption spectroscopy (XAS) has progressively evolved from a conventional characterization technique into a powerful tool for understanding heterogeneous catalysts under increasingly realistic conditions. This presentation illustrates this evolution through research carried out at IFPEN on molybdenum disulfide-based catalysts, with a particular focus on CoMo systems used for hydrodesulfurization (HDS). The development of more efficient HDS catalysts requires a detailed understanding of their activation, the formation of the active phase, their behaviour under operating conditions, and their deactivation mechanisms. XAS is particularly well suited to address some of these questions because providing local element-specific electronic and structural information while offering the possibility of in situ and operando measurements.

Early developments focused on monitoring the activation of supported CoMo catalysts during gaseous sulfidation¹. Using Quick-XAS and simultaneous measurements at the Mo and Co K-edges, the transformation of oxide precursors into sulfide species was followed in real time. These experiments revealed intermediate Mo oxysulfide species and highlighted the simultaneous sulfidation of cobalt and molybdenum as an important step in the formation of the active CoMoS phase. A high-pressure reactor was subsequently developed to reproduce industrial liquid-phase sulfidation conditions². Coupled with chemometric approaches such as principal component analysis and multivariate curve resolution (MCR-ALS), XAS enabled the identification of different activation pathways depending on the supply of the sulfiding agent.

The research then extended beyond the sulfided phase to investigate the origin of catalyst oxide species during the impregnation step. XAS combined with linear combination fitting allowed the quantitative identification of several molybdenum-containing species that form when the precursor solution interacts with the alumina support. More recently, hyperspectral full-field XAS imaging has provided spatially and temporally resolved information on the diffusion and speciation of molybdenum throughout catalyst extrudates³. This approach revealed significant changes in Mo speciation during impregnation, driven in particular by local pH variations and interactions between phosphorus-containing species and the alumina surface.

Finally, new XAS methodologies are being explored for photocatalysis, with the objective of identifying species directly involved in catalytic reactions⁴. Modulation excitation combined with phase-sensitive detection (MES-PSD) was applied to a Mo-based photocatalyst for CO₂ conversion to methane, revealing a photo-induced signal associated with the more active oxysulfide phase. Overall, these developments demonstrate the close evolution of XAS and catalyst research from conventional measurements to in situ/operando experiments, spatially resolved imaging, and advanced chemometric analysis.

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X-ray Spectromicroscopy or Microspectroscopy? A Methodological Perspective

Jean Susini

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ABSTRACT

Beyond the semantic difference between X-ray spectromicroscopy and microspectroscopy, the evolution of these two methods must be viewed within the broader context of advances in X-ray instrumentation and the goal of developing multidimensional techniques with chemical sensitivity. This presentation will trace the evolution of these techniques in the context of advances in synchrotron sources and instrumentation. It will conclude with a brief discussion of the advantages and disadvantages of fourth-generation synchrotron sources.

X-ray Absorption Spectroscopy: A Key to Deciphering and Optimizing Battery Materials

Laurence Croguennec

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ABSTRACT

X-ray Absorption Spectroscopy (XAS) has emerged as a powerful tool for probing the electronic and local structural environments in Metal-ion battery electrode materials. This talk will highlight how XAS provides unique insights into the redox mechanisms, phase transformations, and degradation processes in battery electrodes. By elucidating the oxidation state and coordination of key elements, transition metals but also anions, XAS enables the rational design of high-performance, durable, and safe battery systems. Case studies will also highlight how the development of dedicated electrochemical cells allowed to get in-depth insights into the mechanisms involved during the operation of the batteries. The possible effect of beam damages will also not be eluded.

Dichroisms at LURE and SOLEIL

Philippe Saintavit

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ABSTRACT

In this presentation we will outline the pioneer work by Jacquelin Petiau and Georges Calas in the field of X-ray absorption spectroscopy when playing with the polarization of light. A special attention will be dedicated to the instrumental resolution and to technical developments that were undertaken in order to optimize it.

In addition, we will also underline the role played by some laboratories in Paris, Saclay, Nancy and Strasbourg who contributed to the development of outstanding instruments at LURE and SOLEIL such as a hybrid asymmetric wiggler, high resolution monochromators, harmonic rejection devices, wire chambers for fluorescence detection, quarter-wave plates, high magnetic field magnets, and sub-kelvin cryostats.

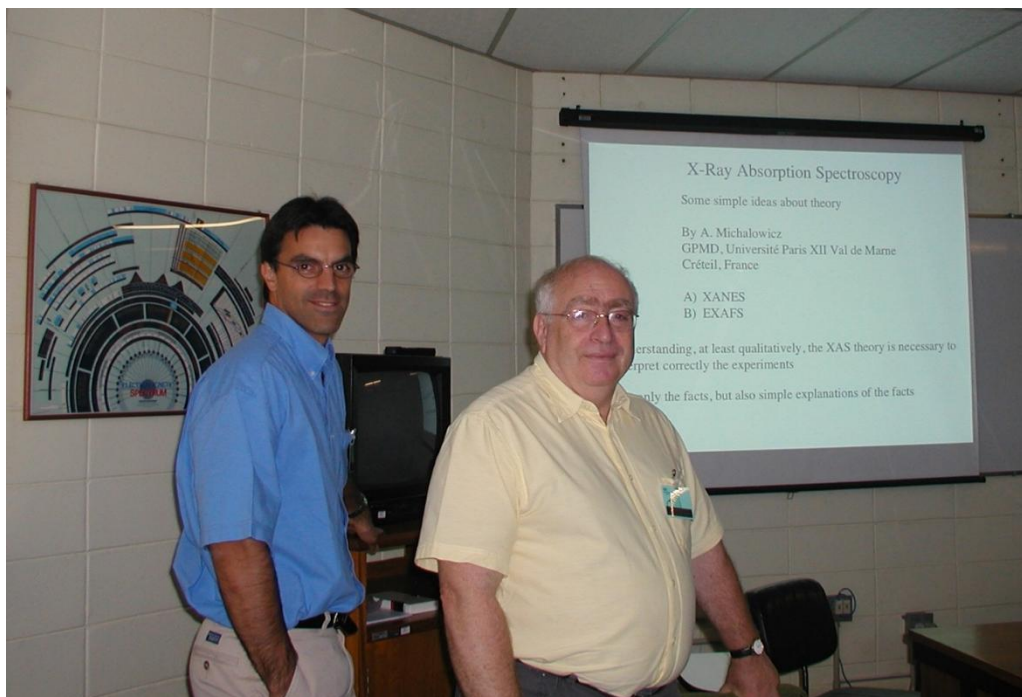
In Memoriam Prof. Alain Michalowicz

Karine Provost¹, Emiliano Fonda²

¹ICMPE, Université Paris Est Créteil, France ; ²Synchrotron SOLEIL, France

Alain Michalowicz began his academic career in September 1974 as an assistant professor at Paris 12 University's structural physico-chemistry laboratory in Créteil, under Michel Renaud and Roger Fourme. He helped build the LURE D1 beamline and, guided by Fourme, turned to studying metallo-proteins via X-ray absorption spectroscopy, installing the first fluorescence detector on the EXAFS 1 branch. A key collaboration with José Goulon shaped his development of accessible EXAFS analysis software—first for Mac, with the set *EXAFS for the Mac* and later *MAX: Multiplatform Applications for XAFS*. His 1990 state thesis focused on analyzing local order and disorder in inorganic materials.

Author of over 100 papers, he worked on both materials science (spin-transition complexes, electrochromic compounds, ferroelectric perovskites) and biomedical applications (cobalamin compounds, vitamin B12 derivatives, platinum anticancer complexes). Known for combining X-ray diffraction with absorption spectroscopy and for his rigorous statistical approach, he served on the IUCr XAFS commission (1999–2008) and co-authored an influential 2000 report on error handling in EXAFS analysis. From 2007 to 2023, he taught error calculation at the Montpellier XAS summer school. Later in his career, he explored multivariate analysis of X-ray absorption spectra to track chemical reaction mixtures, updating his software accordingly.



Jacques Moscovici (at the left) and Alain Michalowicz (at the right) during a XAFS School at the Brazilian Synchrotron Light Laboratory (LNLS, Campinas) in 2004.

Tracing cadmium from soil to foodstuff using X-ray techniques

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ABSTRACT

Cadmium (Cd) is a toxic metal classified as a Group 1 carcinogen by the World Health Organization (WHO). For the general population, food is the main route of cadmium exposure. Various mitigation strategies are under study to decrease cadmium in crops. Understanding how cadmium is accumulated in the plant and how it is transferred to edible parts is key to help plant breeding and cultivar selection, and developing other mitigation strategies. X-ray absorption spectroscopy and micro X-ray fluorescence are particularly adapted to answer to these questions. A major challenge is the low Cd concentrations (generally $< 1 \text{ mg kg}^{-1}$) in realistic agricultural systems. We will show how the techniques have progressed over time on sample preservation, sensitivity and resolution. Results obtained on various crops, including cereals [1] and cacao [2-4] will be presented.

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XAFS and nuclear fuels

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ABSTRACT

Closing the nuclear fuel cycle is essential for developing more sustainable nuclear energy systems. In France, plutonium recovered from spent uranium oxide (UOx) fuel is recycled into mixed uranium–plutonium oxide (U,Pu)O₂ (MOx) fuels and used in Pressurized Water Reactors (PWR). MOx fuels are also leading candidates for several Generation IV reactor designs, including sodium-cooled fast reactors (SFRs). Because of the complexity and long timescale involved in working with spent fuel samples, the experimental strategy involves first working with fresh MOX fuel and/or surrogate materials. The latter, known as SIMMOX, consists of (U,Pu)O₂ doped with up to 12 stable fission products (FPs).

Over the past 30 years, XAFS in combination with other characterization techniques ((SP)-XRD, Raman microscopy, SEM-EDS, SEM-EBSD, EPMA, ...) has been used in nuclear fuel studies. In particular, XAFS has recently been applied to investigate the fabrication MOX fuels and their associated microstructural properties^{1,2}, the behaviour of FPs^{3,4,5} and the influence of Pu content on the MOX oxidation processes⁶. Several illustrations will be presented during the talk. It should be emphasized that these studies were made possible only by the development of synchrotron beamlines capable of accommodating highly radioactive samples, such as ROBL at the ESRF⁷ and MARS at SOLEIL⁸.

Nevertheless, the available beamtime remains limited, and additional constraints, such as the preparation of radioactive samples and their transport to the beamline, have motivated the development of a complementary laboratory-scale X-ray absorption spectroscopy (XAS) apparatus. As part of a collaboration between CEA Marcoule and the University of Helsinki, a new, compact, laboratory-scale XAS apparatus optimised for actinide materials, has been installed in the ATALANTE hotlab facility (CEA Marcoule, France). This setup uses a low-power X-ray tube and a spherical bent crystal analyser. It can operate in both symmetric and asymmetric Johann configurations⁹. The first results obtained with this experimental setup will be presented.

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Multi-scale characterization of environmental colloidal materials

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ABSTRACT

Colloids are microscopic particles with sizes in the range 1 to 0.01 μm . Because of their small size, they are sensitive to Brownian motion and can then be transported over short and long distances. Furthermore, they have a very high specific surface area that makes them highly reactive towards adsorption. Understanding their structure and adsorption properties and their evolution under evolving physico-chemical conditions is then of prime importance for environmental monitoring and is crucial for understanding and modelling the various processes at play in environmental systems. We will show in this talk how X-ray absorption spectroscopy has strongly deepened our knowledge of the structure and adsorption properties of environmentally relevant colloidal materials. We will also present the interest of analyzing in parallel colloidal materials systems at higher length scales, through the use of scattering techniques (light, neutron, x-rays) and spectromicroscopic methods such as STXM or TXM among others.

Contributed Talks

Tuesday, October 6th, 2026

XAFS Contribution to the Past and Today Science

SOLEIL Auditorium – Main Building

- 11:35 – 11:53
CT-01 First-principles XANES calculations within the pseudopotential framework: 17 years of XSpectra
Delphine Cabaret – Sorbonne Université, IMPMC, France

Parallel Session: Disordered Materials, Chemistry & Cultural Heritage

SOLEIL Auditorium – Main Building

- 14:00 – 14:18
CT-02 Local Structure in Silicate Glasses and Melts at Extreme Conditions
Max Wilke – Universität Potsdam, Germany
- 14:18 – 14:36
CT-03 From liquid to solid: The contribution of XANES at the structure and properties of glasses and melts
Daniel Neuville – IPGP CNRS, France
- 14:36 – 14:54
CT-04 Comparative Pt L₃-edge XAS study of Pt nanoparticles with different morphologies in solution and tumor cells
Raul Magro Hernandez – UCM, Spain
- 14:54 – 15:12
CT-05 XAFS to study Cultural Heritage Metallic Materials
Solenn Reguer – Synchrotron SOLEIL, France
- 15:12 – 15:30
CT-06 Harnessing X-rays to Reveal Historical Productions in Late Iron Age Britain
Susan Nehzati – Diamond Light Source, United Kingdom

Parallel Session: Catalysis

SOLEIL Auditorium – Reception Building

- 14:00 – 14:18
CT-07 Catalyst Design Guided by XAS Spectroscopy
Xavier Carrier – Sorbonne Université, France
- 14:18 – 14:36
CT-08 XAS's contribution to IRC(IRCELYON) research in the field of catalysis
Christophe Geantet – CNRS, France
- 14:36 – 14:54
CT-09 Operando XAFS Reveals Interfacial Redox Chemistry of Supported Metal Catalysts as the Switch Descriptor Between Base- and Tip-Growth Carbon Filaments During Catalytic Methane Splitting
Wilson Henao – Instituto de Tecnología Química (ITQ), Spain
- 14:54 – 15:12
CT-10 Combined XAFS/DRIFTS/MS: a powerful technique for operando catalysts investigations
Davide Salusso – ESRF, France
- 15:12 – 15:30
CT-11 Monitoring the Formation of a Single-Atom Catalyst with Operando Quick XAFS
Franck Morfin – IRCELYON, France

Wednesday, October 7th, 2026

Theory and Data Analysis – Technique Evolutions

SOLEIL Auditorium – Main Building

- 14:35 – 14:53
CT-12 Understanding chemical bonds with valence RIXS
Oana Bunau – ESRF, France
- 14:53 – 15:11
CT-13 Orbital Engineering in Graphene/SiC: An ELNES and FDMNES Study
Vinod Kumar Paidi – CNRS, INSA Lyon, UCBL, MATEIS, UMR 5510, France
- 15:11 – 15:29
CT-14 Unraveling Local Environment in Multicomponent Materials using Reverse Monte Carlo Simulations
Alexei Kuzmin – Institute of Solid State Physics, Latvia
- 15:29 – 15:47
CT-15 Multiple-Edge EXAFS/RMC Refinement for 3D Local Structure in SrTiO₃
Andris Anspoks – Institute of Solid State Physics, Latvia
- 16:45 – 17:03
CT-16 Introducing « FITNESS »: a new program, built-in to Fastosh, to efficiently perform shell-by-shell fitting of the EXAFS
Gautier Landrot – Synchrotron SOLEIL, France
- 17:03 – 17:21
CT-17 From Monometallic to Bimetallic NiCo/SiO₂: Element-Resolved Active-Site Identification by Operando XAS and MCR-ALS
Shuchi Zhang – Eindhoven University of Technology, Netherlands
- 17:21 – 17:39
CT-18 Resolving the "Coloring Problem" in Crystallography: The Synergistic Power of EXAFS and Resonant Diffraction
Carmelo Prestipino – CRISMAT, France
- 17:39 – 17:57
CT-19 EXAFS Unbound: Unlocking Atomic Scale Insights Beyond The Next Edge
Rene Bes – University of Helsinki, Finland

Parallel Session: Energy Related Materials

SOLEIL Auditorium – Main Building

- 11:00 – 11:18
CT-20 From Ex Situ to Operando Soft X-rays Microscopy of Photoactive Materials: Case of Hematite-based Photoanodes
Stefan Stanescu – Synchrotron SOLEIL, France
- 11:18 – 11:36
CT-21 Operando XAS Strategies for Rational Design of Electrochemical Energy Materials
Francesco Paparoni – Synchrotron SOLEIL, France
- 11:36 – 11:54
CT-22 Tuning Redox Chemistry in Gd-Doped Ceria: Gd-Driven Suppression of Ce³⁺ Formation in Reducing Condition
Alessandro Longo – ESRF, France
- 11:54 – 12:12
CT-23 The Optical Dispersive EXAFS (ODE) Beamline at Soleil Synchrotron and Its Studies
Qingyu Kong – Synchrotron SOLEIL, France
- 12:12 – 12:30
CT-24 Combined Operando XRD And XAS Study Of Electrochemically Active Li₂MChO Antiperovskites
Mikhail Gorbunov – Karlsruhe Institute of Technology, Germany

Parallel Session: Geosciences Part I

SOLEIL Auditorium – Reception Building

- 11:00 – 11:18
CT-25 Coupling High-Energy Resolution Fluorescence Detected X-ray Absorption Spectroscopy (HERFD-XAS) with microfluidic cells for in situ monitoring of tungsten nanoparticles redox transformation
Melanie Auffan – CERECE CNRS, France
- 11:18 – 11:36
CT-26 Vanadium enrichment in lateritic soils: mineralogical and pedoclimatic controls
Myriam Boudieb – Sorbonne Université, France
- 11:36 – 11:54
CT-27 Redox speciation of cerium in the presence of colloids
Terjini Kiththangodage – IPGP, France
- 11:54 – 12:12
CT-28 XAS Studies Of Metal Transport In Hydrothermal Fluids: Autoclaves And Photons
Denis Testemale – Institut Néel, CNRS, France
- 12:12 – 12:30
CT-29 Probing the Speciation of Transition Metals (Mo, W) in Hydrothermal Fluids by In-Situ XAS
Maria Kokh – Institut für Geowissenschaften Universität Potsdam, Germany

Thursday, October 8th, 2026

Technique Evolutions – 4th Generation and Beyond

SOLEIL Auditorium – Main Building

- 14:35 – 14:53
CT-30 Beyond XAS with RIXS
Jean-Pascal Rueff – Synchrotron SOLEIL, France
- 14:53 – 15:11
CT-31 Study of the genesis of HDS catalysts by in situ photon in photon out spectroscopy
Sylvain Cristol – Université de Lille, France
- 15:11 – 15:29
CT-32 Understand 4f covalency by ab initio calculated RIXS plane
Sihan Zhang – ESRF, France
- 15:29 – 15:47
CT-33 Tender X-ray photon-in/photon-out spectroscopy at ESRF ID26
Pieter Glatzel – ESRF, France
- 16:45 – 17:03
CT-34 Developments of the setup for pump-probe X-ray absorption fine structure (XAFS) and a capability of EXAFS at FXE instrument, European XFEL
Yohei Uemura – European XFEL, Germany
- 17:03 – 17:21
CT-35 FAME-PIX: The Pioneering French High-Resolution Hyperspectral 3D X-Ray Microscope at the ESRF
Julio C. Da Silva – Institut Néel, CNRS, France
- 17:21 – 17:39
CT-36 Operando Spatio-Temporal Study of CO oxidation reaction in Pd Catalysts using Full-Field Hyperspectral XAS imaging
Marlon Muniz Da Silva – CNPEM, Sirius, Brazil
- 17:39 – 17:57
CT-37 X-ray Absorption Spectroscopy meets Imaging to Unveil Inclusion Puzzles in Deep Diamonds
Helio Tolentino – LNLS/CNPEM, Brazil

Parallel Session: Chemistry
SOLEIL Auditorium – Main Building

- 11:00 – 11:18 Unraveling the fate of anticancer gold complexes in cells through HERFD-XAFS
CT-38 **Tom Lacoma** – Sorbonne Université, France
- 11:18 – 11:36 A short retrospective of actinide hexacyanoferrate exploration initiated at LURE
CT-39 **Christophe Den Auwer** – Université Côte d'Azur, France
- 11:36 – 11:54 Magnetic hyperthermia at the nanoscale
CT-40 **Jerome Fresnais** – CNRS, Laboratoire PHENIX, France
- 11:54 – 12:12 Understanding Nanoparticles Formation mechanisms at ITODYS Laboratory: Insights from investigations at ROCK Beamline
CT-41 **Lorette Sicard & Marion Giraud** – ITODYS, Université Paris Cité, France
- 12 :12 – 12 :30 Time- and Space-Resolved Formation Stannosilicates by in situ XAS
CT-42 **Aram Bugaev** – Paul Scherrer Institute, Switzerland

Parallel Session: Geosciences Part II
SOLEIL Auditorium – Reception Building

- 11:00 – 11:18 Redox speciation of contaminants at magnetite surface
CT-43 **Rémi Marsac** – Institut de physique du globe de Paris, CNRS, France
- 11:18 – 11:36 Mineralogical Controls on the Fate of Emerging Contaminants in Laterites Revealed by XAFS
CT-44 **Quentin Bollaert** – Synchrotron SOLEIL, France
- 11:36 – 11:54 Thermal transformation of clay minerals probed by in situ soft-X-ray absorption spectroscopy
CT-45 **Erwan Paineau** – Laboratoire de Physique des Solides, CNRS, France
- 11:54 – 12:12 Assessing the Sensitivity of X-ray Absorption Spectroscopy to Distinguish Various Forms of Nucleic Acids and Their Surface Structures on Imogolite Nanotubes
CT-46 **Jerome Rose** – CNRS CEREGE, France
- 12 :12 – 12 :30 X-ray absorption spectroscopy in surface science on LUCIA
CT-47 **Nicolas Trcera** – Synchrotron SOLEIL, France



First-principles XANES calculations within the pseudopotential framework: 17 years of XSpectra

Delphine Cabaret

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ABSTRACT

XSpectra is the X-ray absorption spectroscopy module of the Quantum ESPRESSO suite [1], enabling first-principles calculations of X-ray absorption near-edge structure (XANES) spectra within a density-functional-theory-based framework that uses pseudopotentials [2]. Originally introduced in 2002 by Taillefumier et al. [2], the method reached a broader community with the publication of the XSpectra implementation in 2009 [3] and has since been employed in several hundred scientific studies across physics, chemistry, materials science, and geosciences.

In this presentation, I will review the major developments of XSpectra over the past 17 years, highlighting the methodological advances [5-8] that have progressively extended its range of applicability. I will also illustrate the impact of the code through a selection of representative applications, demonstrating its role as a widely adopted tool for the interpretation and prediction of XANES spectra from first principles.

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Local Structure in Silicate Glasses and Melts at Extreme Conditions

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ABSTRACT

The local structure in non-crystalline matter at high pressure is largely ruled by closed packing of atoms without the demand for long-range order as in crystalline systems. The lack of long-range order also represents one of the biggest obstacles of constraining the structure of these systems as standard XRD methods cannot be applied. Silicate melts are ubiquitous materials in the deep Earth that have been ruling the dynamics and chemical evolution of the Earth from the core to the atmosphere over geological times. Here, we will present latest advances by X-ray spectroscopy, XANES and EXAFS, at extreme pressure and high temperature using diamond anvil cells (DAC) to characterize the local structure around Ge, Sr and Y in silicate glasses and melts. Measurements at high temperature were performed either by laser-heating in the melt above the liquidus or in the supercooled melt by resistive heating. While Ge as structural analogue of Si, transforms from tetrahedral to octahedral coordination up to ca. 25-30 GPa with considerable increase of Ge-O distance, the larger cations increase their coordination from octahedral to 7 or even 8-fold at almost constant distance to O [1,2,3,4]. At higher pressure, all cations show only polyhedral compression. High temperature shifts the transformation pressure only slightly [5]. This indicates only weak influence of kinetics and superimposition of thermal expansion on the compression. I will discuss the application of bond-valence theory to evaluate resulting data on the structural properties. Finally, I will suggest implications for the structure of silicate melts at extreme pressures as well as for magmas at extreme depth of the Earth or planetary bodies.

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From liquid to solid: The contribution of XANES at the structure and properties of glasses and melts.

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ABSTRACT

X-ray absorption is a powerful and element-specific probe for investigating the structure of materials: crystals, liquids, and glasses. Initially, such studies were limited to heavy elements in crystalline or amorphous solids, but thanks to the upgrades implemented on the LUCIA beamline, successful measurements on light elements were first achieved at room temperature and subsequently extended to high temperatures. We will present a series of experiments performed from room temperature up to 2400 K at the K-edges of Si, Al, Ca, and Fe in amorphous, crystalline, and liquid systems. These experiments have provided new insights into several fundamental processes:

- i) they revealed the unique behavior of aluminum in molten silicates, leading to the proposal of a new model for the dynamics of molten silicates;

- ii) they demonstrated that calcium rapidly occupies a pre-nucleation site when glasses are heated just above the glass transition temperature;

- iii) they confirmed the local structure of SiO₂ polymorphs;

- iv) they elucidated the structure of molten alumina;

- v) they showed how aluminates influence the redox state of iron.

Together, these results demonstrate the unique capability of high-temperature X-ray absorption spectroscopy to probe element-specific local environments under extreme conditions, providing fundamental insights into the atomic-scale mechanisms governing the structure, dynamics, and crystallization of oxide materials.

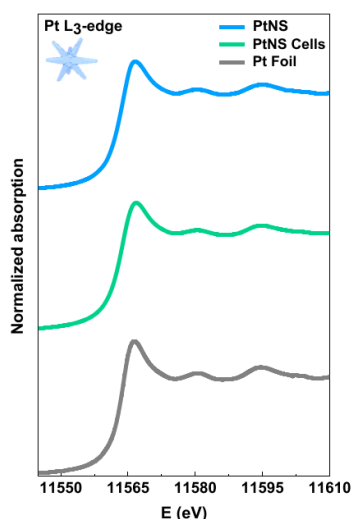
Comparative Pt L₃-edge XAS study of Pt nanoparticles with different morphologies in solution and tumor cells

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ABSTRACT

Platinum (Pt) nanoparticles are promising therapeutic nanomaterials whose physicochemical properties, catalytic performance, photothermal efficiency, and structural stability are strongly influenced by their morphology [1,2]. Moreover, once internalized by cells, Pt nanostructures may undergo transformations that affect their therapeutic action. In this work, Pt L₃-edge X-ray absorption spectroscopy (XAS) was used to compare Pt-based nanoparticles with different morphologies, including nanostars, polyhedral nanoparticles, and nanodendrites [3]. XANES and EXAFS analyses were performed on Pt nanoparticles in solution and after incubation with tumor cells, enabling direct comparison of their oxidation state and local structure before and after cellular uptake [4]. This approach provides insight into morphology-dependent structural changes and the intracellular stability of Pt nanostructures. Experiments were carried out at the BM23 beamline (ESRF, Grenoble, France). Additional studies on nanothermometric effects will be conducted at the SAMBA beamline (SOLEIL, France).



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XAFS to study Cultural Heritage Metallic Materials

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ABSTRACT

The presentation will look back over more than 20 years of investigations carried out using synchrotron beamlines on metallic cultural heritage materials. Advances in XANES and EXAFS techniques have made possible to address various and crucial scientific questions: assessing the extent of deterioration and the processes of metal corrosion both over the very long term and in a variety of environments. These techniques supported the set-up of reliable diagnostic methods while developing and improving conservation strategies.

The analyses were based on the need to distinguish and locate the constitutive phases of the corrosion layers because of their different reactivity. In that way, metallic samples from different objects and archaeological and historical sites have been thoroughly studied, including wrought-iron bars from Gallo-Roman shipwrecks [1], tie rods from Amiens Cathedral [2], elements of architectural heritage, as well as medieval and ancient objects [3] or even aluminium airplanes [4]. The combination of micro-XRF and macro/micro XANES and EXAFS measurements at the Fe K-edge and Al K-edge made it possible to identify alteration products and to map the oxidation states according to the exposure environment (atmosphere, soil, or marine environment). The role of specific elements, especially S, P and Cl, on phase's stability and the chemical form and distribution of these elements in the alteration layers, was also investigated at S, P [5] and Cl K-edges [6,7].

Advances in 2D/3D imaging methods marked the transition from point-by-point spectral measurements to a comprehensive visualization of the distribution of chemical states throughout the entire object. This approach offered a twofold opportunity: on the one hand, it deepened our understanding of long-term alteration processes under buried or submerged conditions; on the other hand, it enabled the practical optimization of the conservation of ancient metals through a fine understanding of their behavior at a micrometric scale, thereby guiding the development and evaluation of more effective stabilization and protection processes.

In addition, the development of specific samples environments played a key role in such studies. Thanks to the design of dedicated (electro)chemical cells, in situ analyses were conducted to study in real time the corrosion [8] and/or restoration mechanisms, such as those involved in dechlorination treatments [9, 10] and protection using metal soaps [11].

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Harnessing X-rays to Reveal Historical Productions in Late Iron Age Britain

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ABSTRACT

Heritage materials provide valuable records of past human activity and researching them helps illuminate key transitions in historical cultural development. A decorated Iron Age terret currently displayed in the European Prehistory Gallery of the Ashmolean Museum, Oxford, provides a unique opportunity to investigate glass production and exchange in late Iron Age Britain. Found in Suffolk, England, and dated 50 BCE–75 CE, this copper-alloy terret is ornamented in the characteristic La Tène style, featuring incised embellishments and coloured glass inlays. The red glass is identified as the distinctive “sealing wax” red [1] commonly found in prestigious metal artefacts of this period, yet the origin and significance of the dark, translucent blue glass is less well understood.



Late pre-Roman Iron Age terret used in analysis.

Using X-ray fluorescence imaging (XRF) and X-ray absorption near-edge structure (XANES) at Diamond Light Source, we examined the chemical composition and production technology of the glass inlays. We characterised the chemical composition of the terret glass, compared with Corning glass reference standards [2], particularly focusing on iron, copper, and lead.

The results provide new evidence for the production, use, and circulation of coloured glass in pre-Roman Britain, enabling a more detailed reconstruction of the object's biography. More broadly, the study contributes to our understanding of craft practices, material exchange, and social networks in late Iron Age Britain.

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Catalyst Design Guided by XAS Spectroscopy

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ABSTRACT

Rational synthesis of catalytic materials plays a pivotal role in the industry due both to the enormous economic impact of these materials and their requirement for establishing more sustainable chemical processes, reducing pollution and producing energy. Despite this considerable economic and environmental importance, catalyst preparation has long relied on trial-and-error approaches. Innovative synthesis strategies are now being developed to achieve a truly rational design of catalytic materials, for example, to enable nanoscale control over the size and growth of supported active-phase particles. One of the most suitable molecular-level characterization techniques for guiding this rational design is X-ray absorption spectroscopy, which combines unique advantages (namely element specificity and insight into the electronic and local environment of the probed element). The purpose of this presentation is to present several examples of heterogeneous catalysts preparation (metals, sulfides, carbides) for which XAS experiments performed at French synchrotron beamlines (notably under operando conditions thanks to the Equipex funding for the ROCK project) have played a key role in the design of improved catalytic formulations.

The atomic distribution of metals is of particular importance in catalysis, as surface atoms play a key role in catalytic performance. Furthermore, when bimetallic nanoparticles are exposed to various gas atmospheres (including reaction conditions) and heated to high temperatures, atomic redistribution may occur. Two examples obtained using ROCK [1] will be presented (Au-Ag nanorods and Au-Cu nanoparticles): they allow us to track the redistribution of metals (Au-Ag) and the onset of reduction (Au-Cu) to demonstrate that the kinetics of these processes depend strongly on the metal composition and particle volume.

A 2nd example will focus on sulfide catalysts (MoS₂) supported on alumina, which are key components in hydrosulfurization (removal of sulfur from petroleum feedstocks) and in the conversion of biomass for biofuel production. In this case, a surface-science approach was developed to enable better control of the speciation of surface sorption sites, using α -Al₂O₃ single crystals with different orientations as model supports. Polarization-dependent surface EXAFS experiments [2], conducted on SAMBA and DIFFABS, revealed a preferential orientation of MoS₂ slabs on surfaces exhibiting reduced metal-support interactions. The metal-support binding strength appears to be a decisive descriptor for surface speciation and, consequently, for describing the surface-dependent volcano plot for hydrotreating activity.

Finally, HERFD XANES experiments conducted on FAME-UHD [3] using carbon-supported Mo and W carbide catalysts will be presented to investigate the role of the carburization sequence in the formation of these Mo-W carbides. A clear carburization order is observed, governed not only by the intrinsic reducibility of the metals but also by the extent of overlap between the predominant domains of reduced MoO₂ and WO_{3-x}. These results show that the material can be tailored to produce either homogeneous bimetallic nanoparticles or phaseseparated carbides, thereby laying the foundation for a rational design of catalysts.

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XAS's contribution to IRC(IRCELYON) research in the field of catalysis

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ABSTRACT

From the early beginnings of the use XAS at LURE until now at Soleil and the ESRF (as well as other SR), the “Institut de recherche sur la Catalyse” (IRC, now IRCELYON) has been involved in the development of *in situ* or *operando* studies related to several domains of catalysis and types of catalysts. We will summarize some of the main findings and developments performed in collaboration with beamlines scientists for the benefit of one of the major fields of application of XAS. Three years after the creation of LURE in 1973, A Renouprez and B. Moraweck from IRC submitted a proposal to solve the structure of supported Pt nanoparticles¹ and a few years later built up a cryofurnace for combining heat treatment and further analysis at liquid nitrogen temperature. The application of EXAFS was at that time mainly addressing Pt supported catalysts rapidly extended supported carbonyl clusters², nickel-based catalysts (either Raney or supported), Pd based catalysts and their alloys³. In the 1990, via a collaboration with M. Danot of IMN Nantes, started the investigation of many types of transition metal sulfides and the development of cells of *in situ* sulfidation of studying industrial catalysts in collaboration with IFPEN or EURECAT⁴. In parallel, J. M. Millet was investigating partial oxidation catalysts following the variation of oxidation states by XANES.⁴ P. Afanasiev was the last user of a XAS beamline at LURE!

The French community of XAS took benefit of the development of CRG beamlines (IF then FAME and FAME UHD) at the ESRF which allowed the investigation by fluorescence of low loading catalysts, photocatalyst or poisons, first QEXAFS studies of catalysts, use of high resolution XANES (for instance Pt/CeO₂). Then, the construction of Soleil SR brought beamlines (Samba and then Rock) in perfect adequation for studying the dynamic of catalysts during pre-treatment or catalysis. Activation or *operando* studies performed by QXAS, brought to catalysis community outstanding results for instance with ultradispered catalysts⁵. This presentation will give an overview, throughout history, of the contributions of our Institute in the application of XAS to the field of heterogeneous catalysis.

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Operando XAFS Reveals Interfacial Redox Chemistry of Supported Metal Catalysts as the Switch Descriptor Between Base- and Tip-Growth Carbon Filaments During Catalytic Methane Splitting

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ABSTRACT

Catalytic methane splitting offers a direct route to CO_x-free hydrogen and valuable solid carbon products, yet catalyst performance is dictated by a nanoscale question that last as difficult to resolve: why do some metal nanoparticles remain anchored and drive carbon base-growth while others detach and ride at the filament tip? Here, operando quick-XAFS at the Ni and Co K-edges coupled with online mass spectrometry identifies the metal-support perimeter as a redox-active switch controlling base- versus tip-growth in metal nanoparticles supported on *non-porous* Al₂O₃. The study tracks size-controlled NiO/Al₂O₃ and Co₃O₄/Al₂O₃ catalysts during H₂ reduction, He flushing to remove residual H₂ and subsequent CH₄ splitting at 873 K.

To resolve evolving chemical states within the operando XAFS datasets, we applied a chemometric workflow combining principal component analysis (PCA), multivariate curve resolution–alternating least squares (MCR-ALS), and linear-combination fitting (LCF). The XAFS-derived speciation reveals two sharply different interfacial redox pathways: under reductive activation at 873 K, NiO nanoparticles reduce directly to a predominantly metallic Ni phase, followed by a minor surface reoxidation during He flushing. This limited interfacial oxygen reservoir is rapidly carboreduced under CH₄ exposure, leaving mobile Ni particles that detach to yield tip-grown carbon nanofibers. In sharp contrast, small Co₃O₄ nanoparticles follow a sequential reduction pathway toward CoO and Co, retaining substantial interfacial CoO- and CoAl₂O₄-like environments that become enriched during He flushing. Upon CH₄ introduction, these oxygen-containing species are only partially carboreduced, leading to persistent Co-O-Al anchoring motifs that stabilize Co at the alumina perimeter and promote base-grown, open-ended carbon nanotubes. The mechanism is size dependent: larger Co nanocrystals lose the CoAl₂O₄-like signature and switch toward tip-growth like Ni.

Environmental TEM observations directly capture this divergence as particle rounding and lift-off for Ni, versus carbon-induced flattening and sustained anchoring for Co. DFT further rationalizes this behavior through stronger Co–Al₂O₃ adhesion and a higher probability of Co substitution into the interfacial oxide. These results establish operando XAFS as a decisive atomic probe of reactive metal-support interfaces and provide actionable descriptors like nanoparticle size, oxophilicity and interfacial oxygen persistence, for designing methane-splitting catalysts that steer carbon morphology while sustaining hydrogen production.

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Combined XAFS/DRIFTS/MS: a powerful technique for operando catalysts investigations

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ABSTRACT

Diffuse Reflectance Infrared Fourier Transformed (DRIFTS) and X-ray Absorption (XAS) spectroscopies are two of the most employed techniques for catalyst characterization under reaction conditions. However, comparison of kinetics from the two experiments is not trivial due to the differences of the setups. In this talk the key advantages of measuring simultaneous XAS and IR spectra within the same reactor will be presented.

The set-up developed at BM23 and ID24-DCM ESRF beamlines combines a commercial infrared (IR) spectrometer where custom Au-mirrors guide the light on an in-house developed reactor cell allowing to simultaneously collect DRIFTS and XAS either in transmission or in fluorescence modes. The latter is of paramount importance since current catalytic research favors lower metal loadings that maximize atom efficiency. Moreover, the reactive gases pass through the sample reproducing a tubular reactor while products are collected and identified by an online Mass Spectrometer (MS).

In the panorama of catalytic reactions, simultaneous measurement of DRIFTS/XAS/MS allows to identify the catalysts surface and bulk species associated to the catalytic activity. In this presentation the set-up capacities will be presented through the study of NiCeO₂ catalysts under Sabatier reaction ($\text{CO}_2 + 4\text{H}_2 \rightarrow \text{CH}_4 + 2\text{H}_2\text{O}$, 300°C) where the results provided a clear correlation between NiO/Ni - Ce⁴⁺/Ce³⁺ redox properties, surface carbonates coordination and methane production. [1,2]

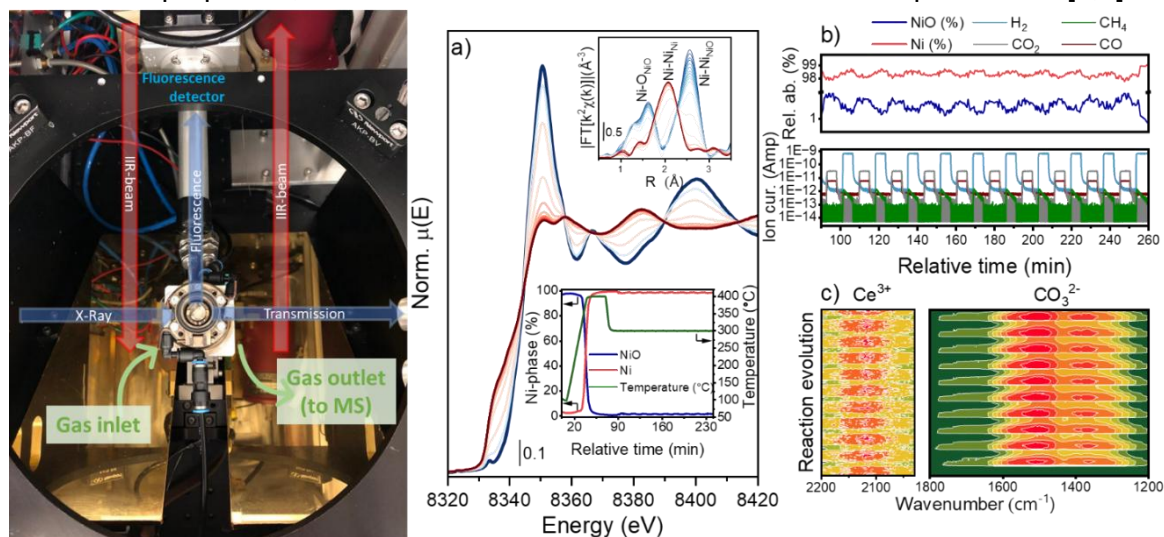


Figure. (left) DRIFTS/XAS reactor. Operando a) Ni K-edge XANES and (top inset) FT-EXAFS spectra with (low inset) MS results. Temperature increases from blue to red line. b) Detail of (top) NiO/Ni modulation with (low) MS data. c) Ce³⁺ and CO₃²⁻ DRIFTS maps measured during CO₂/H₂ modulation.

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Monitoring the Formation of a Single-Atom Catalyst with *Operando* Quick XAFS

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ABSTRACT

Single-atom catalysts (SACs) have demonstrated their ability to enhance selectivity,¹ reduce the need for expensive metals,^{1,2} and even replace molecular catalysts.^{2,3} Among SACs, M-N-C materials, which consist of isolated metal (M) atoms stabilized by nitrogen dopants in a carbon matrix, are the most widely studied, with applications spanning electro-, photo- and thermo-catalysis. These catalysts are typically synthesized via impregnation/pyrolysis methods, where processing parameters dictate the final metal coordination and thus the catalytic performance.

In this study, we investigated the thermal evolution of Pt and Co precursors initially adsorbed on a carbon powder, using established synthesis protocols^{4,5} and X-ray absorption spectroscopy (XAS) at the SOLEIL ROCK beamline. Multivariate curve resolution – alternating least squares (MCR-ALS) analysis of the spectra reveals complex interconversion processes, with pyrolysis temperature and flowing gas (N₂ or H₂) as critical parameters.

As an illustration, Figure 1 presents the XAS evolution and MCR-ALS decomposition results for the formation of a Co-N-C catalyst, which exhibited high activity and selectivity in CO₂ electroreduction. The process begins with decomposition of adsorbed cobalt(II) acetate, followed by gradual cobalt reduction and single-atom formation (6.3 ± 2.5 light neighbors – optimal fraction around 350 °C) in parallel with the separate development of slightly oxidic cobalt dimers and few-atom clusters (1.0 ± 0.4 and 2.6 ± 0.6 Co neighbors, respectively), and concludes with the appearance of large nanoparticles (8.3 ± 1.6 Co neighbors). These findings offer valuable guidelines for the rational synthesis of SACs.

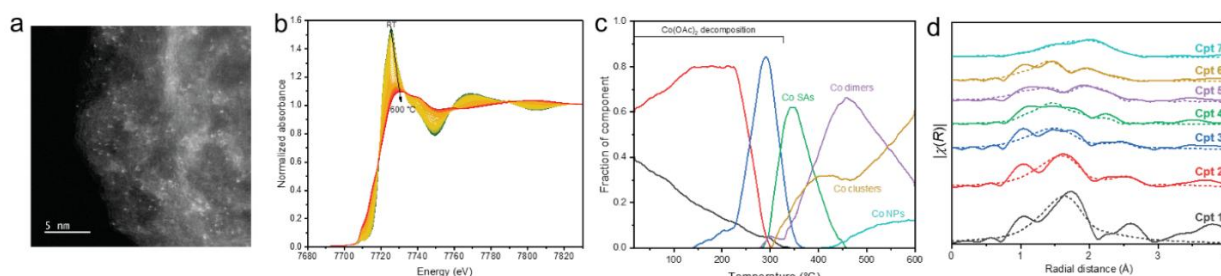


Figure 1. a) STEM-HAADF image of a Co-N-C SAC. b) Evolution of Co-K edge XAS spectra during pyrolysis of Co(OAc)₂ and 1,10-phenanthroline on carbon black, from RT to 600 °C under N₂ flow. c,d) Corresponding MCRALS component fractions and XAFS FT moduli of components (extracted from full RT-600-RT-900°C sequence).

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Understanding Chemical Bond with Valence RIXS

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ABSTRACT

Valence resonant inelastic X-ray scattering (RIXS) provides simultaneous sensitivity to occupied and unoccupied electronic states, making it a promising tool for probing chemical bonding in transition-metal compounds. In this contribution, we present a methodology for the analysis of valence RIXS data acquired at the ID26 beamline at ESRF^{1,2}, at the K and L₃ edge in representative 3d and 4d polycrystalline compounds, respectively. The approach aims to define quantitative metrics that can be extracted from RIXS maps, including descriptors related to the electronic gap and chemical bond. We discuss the conditions under which resonant emission provides additional information on the electronic structure beyond the combination of high energy resolved fluorescence detected (HERFD) absorption and non-resonant X-ray emission spectroscopy. *Ab initio* calculations performed with the FDMNES package³ are used to support the interpretation of the experimental spectra in terms of local electronic structure and metal–ligand hybridization.

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Orbital Engineering in Graphene/SiC: An ELNES and FDMNES Study

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ABSTRACT

With silicon-based logic approaching its fundamental scaling plateau, epitaxial graphene on SiC represents a leading platform for next-generation electronics due to its unique interface-driven properties [1]. While pristine graphene's high carrier mobility is well-reported, its lack of an electronic bandgap limits its use in logic transistors. Recent attention has focused on the "buffer layer" (BL)—the first carbon layer grown on the SiC(0001) facet. Unlike subsequent layers, the buffer layer is semiconducting, offering a ~ 0.6 eV bandgap and hole mobilities superior to Germanium [1].

To probe the substrate-driven orbital engineering and the resulting loss of graphene's metallicity, we used core-loss electron energy-loss spectroscopy (EELS) in a probe-corrected scanning transmission electron microscopy (STEM) environment. Operating on the identical physical principles of dipole-dominated core-excitations shared with X-ray absorption spectroscopy (XAS), electron energy-loss near-edge structure (ELNES) provides complementary, sub-nanometer spatial resolution. We investigated a complex heterostructure comprising a graphene layer and a semiconducting buffer layer grown on 6H-SiC(0001). To decode the localized bonding and electronic structure, theoretical modeling of the C K-edge was performed using the finite difference method approach within the FDMNES code [3], applied to atomic coordinates relaxed via density functional theory (DFT) [4]. Our results reveal two critical phenomena: i) Orbital quenching, where $\sim 25\%$ of carbon atoms undergo sp^3 -like covalent bonding with the Si-substrate, disrupting long-range conjugation; and ii) Symmetry breaking, where a lifting of p_x/p_y orbital degeneracy in non-bonded atoms suggests that van der Waals and electrostatic "pinning" fundamentally alter the electronic topology. This study highlights the power of pairing advanced XAS formalisms (FDMNES) with site-specific electron spectroscopy to uncover hidden electronic states in 2D heterostructures [5].

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Unraveling Local Environment in Multicomponent Materials using Reverse Monte Carlo Simulations

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ABSTRACT

X-ray absorption spectroscopy provides insight into the local atomic structure surrounding a specific element and is ideally suited for studying multicomponent compounds such as solid solutions and high-entropy materials. Simultaneous analysis of the extended X-ray absorption fine structure (EXAFS) spectra of all constituent elements allows the determination of a robust structural model but requires the use of specialized methods. Multi-edge EXAFS analysis using reverse Monte Carlo (RMC) simulations [1] has proven to be a reliable approach for addressing this task [2]. The RMC method makes it possible to probe subtle structural effects beyond the first coordination shell, in the medium-range-order region where coordination shells overlap and contributions from many-atom distribution functions, known as multiple-scattering effects, become important.

Here, the application of the RMC method to the analysis of local structural distortions in tungstate compounds is demonstrated through three specific case studies: the observation of exchange-coupled Ni^{2+} ions in wolframite-type NiWO_4 and $\text{Zn}_c\text{Ni}_{1-c}\text{WO}_4$ solid solutions with high nickel content ($c \leq 0.6$) [3]; the suppression of the Jahn-Teller distortion of $[\text{CuO}_6]$ octahedra in the high-entropy tungstate $(\text{MnCoNiCuZn})\text{WO}_4$ [4]; and the relaxation of the local atomic structure around W^{5+} polaronic centers in proton-intercalated scheelite-type tungstates [5].

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Multiple-Edge EXAFS/RMC Refinement for 3D Local Structure in SrTiO₃

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ABSTRACT

Conventional EXAFS analysis is still often performed edge by edge, although many functional materials contain several absorber atoms whose local environments are strongly coupled. A single-edge fit can reproduce an EXAFS spectrum while leaving correlated distances, disorder factors, multiple-scattering contributions and light-element positions underdetermined. We present a practical multiple-edge strategy in which Sr-K and Ti-K edge EXAFS spectra of SrTiO₃ are fitted simultaneously to one reverse Monte Carlo (RMC) structural model [1-4]. The aim of this presentation is methodological: to show how two absorption edges provide complementary constraints that make the local oxygen geometry and angular distortions observable within standard hard X-ray EXAFS. It also aims to facilitate use of RMC for EXAFS analysis as RMC remains underused in routine EXAFS analysis, especially as a complement to conventional shell fitting.

Transmission spectra of SrTiO₃ collected from 26 to 300 K were analyzed with EvAX using a 6×6×6 tetragonal supercell (4320 atoms), multiple scattering paths, and Morlet wavelet-transform comparison [2].

The SrTiO₃ benchmark illustrates why multiple-edge modelling matters. Earlier single-edge RMC analyses could not resolve the antiphase TiO₆ octahedral rotation, whereas simultaneous Sr-K/Ti-K refinement recovers radial distribution functions, MSRDs, Voronoi volumes, and oxygen-position distributions from one consistent 3D model [1]. The local model shows rigid Ti-O bonding, softer Sr dynamics and an in-plane TiO₆ rotation reaching 2.7° at 26 K. This approach also builds on one of the early two-edge EXAFS/RMC-EA demonstrations in MnWO₄ and Mn_{0.7}Co_{0.3}WO₄ [4,6]. Multiple-edge EXAFS/RMC therefore offers the XAS community a robust and practical route from spectra to three-dimensional local structure.

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Introducing « FITNESS »: a new program, built-in to Fastosh, to efficiently perform shell-by-shell fitting of the EXAFS

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ABSTRACT

To purposely coincide with the 50th anniversary of the use of XAFS in France, it is here presented for the very first time « FITNESS » (« FITting Numerically the Exafs using Sound Strategies »), a new program dedicated to shell-by-shell fitting of the EXAFS. This type of data treatment approach, although widely employed, can be very tedious to perform from a practical standpoint, especially when multiple theoretical paths are involved in the model. Indeed, using the data treatment software that are commonly utilized until now to perform EXAFS shell-by-shell fitting, the expressions of all path parameters (i.e. SO_2 , ΔE_0 , ΔR , N , and σ^2), and the definition of all fit parameters involved in them, must be entirely defined by hand when initially building, or subsequently modifying, the theoretical model. Additionally, this parameterization step is not straightforward as it heavily relies on some fundamental aspects of the modelling approach, which can be overlooked by non-expert users, who consequently may struggle to successfully employ this data-treatment approach. Therefore, this parameterization step arguably represents to everyone the main bottleneck to rapidly and accurately perform EXAFS shell-by-shell fitting. The new program FITNESS overcomes this difficulty, thanks to its automatic parameterization functions that are applied to:

- all single scattering paths. Their path parameter expressions and associated fit parameters are automatically defined using some « *sound strategies* », as the latter take into account 1) the intrasect modelling resolution in R space; 2) the relative amplitude of each path; and 3) the number of degrees of freedom available.
- all multiple scattering paths. This is achieved strictly using the fit parameters intervening in the path parameter expressions of the single scattering paths. Therefore, the number of degrees of freedom is not affected by this step.

Additional unique functions are proposed in FITNESS, including some that allow to perform useful tasks prior to/during calculations by FEFF8L (included in FITNESS), such as:

- importing, and subsequently treating in FEFF, structures featuring partial occupancies
- performing all at once more than one FEFF calculation if the absorber is present in more than one crystallographic position
- viewing in 3D (and other basic 3D characterization functionalities): 1) the unit cell; 2) the atomic environment around the absorber; 3) each FEFF path, individually or inside the atomic environment around the absorber
- modifying the atomic environment around the core (e.g. shell distances, doping level).

Some real EXAFS fitting examples will be provided in this presentation to showcase the capabilities of FITNESS. Its initial trial version is built-in to the latest version of Fastosh, which can be freely downloaded from SAMBA website, Synchrotron SOLEIL.

From Monometallic to Bimetallic NiCo/SiO₂: Element-Resolved Active-Site Identification by Operando XAS and MCR-ALS

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ABSTRACT

Operando X-ray absorption spectroscopy (XAS) is a powerful technique for probing the electronic and local structural evolution of catalysts under working conditions. However, the obtained spectra often contain mixed structural information under reaction conditions. Multivariate Curve Resolution–Alternating Least Squares (MCR-ALS) is an effective method for analyzing linearly mixed datasets and can decompose them into independent components, which can help a lot in XAS data analysis¹.

Our previous work on Ni/SiO₂-catalyzed CO₂ methanation revealed CO* as the dominant surface species, while operando XAS gas-switching experiments showed a corresponding change in the Ni spectral features². Recently, at ROCK beamline (SOLEIL), we performed meticulous transient gas switching experiments (H₂↔CO₂+H₂) followed by XAS over Me/SiO₂ (Me= Ni or Co) and NiCo/SiO₂. We applied MCR-ALS analysis to operando XAS datasets to further identify the intermediates dynamic changes on the surface. We confirmed the intermediates mainly composed by CO* by FTIR (Figure.1a). CO* coverage was calculated using chemisorption and Steady-state isotopic transient analysis (SSITKA) (Figure.1b), also performs as the reference value of MCR-ALS analysis, which gives us the XAS spectra of both metal (S1) and CO-adsorbed metal (S2) (Figure.1c). Substituting these two standard spectra as reference components for the NiCo/SiO₂ sample in MCR-ALS, we obtained the CO coverage on the Ni and Co, respectively, as 6.7% (Ni bases) and 2.0% (Co bases), which highly indicated the activity of both metals. This provides an effective element-resolved method for identifying active sites and their activities on alloy catalysts.

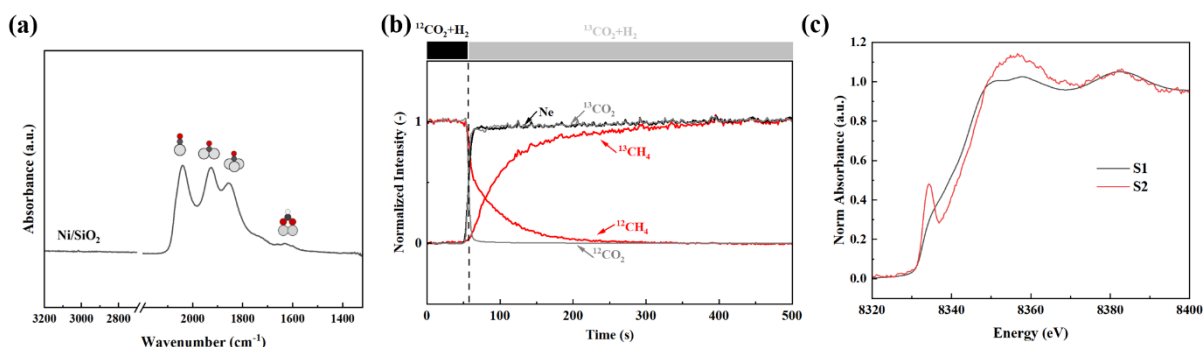


Figure1. (a) FTIR spectra of Ni/SiO₂ during CO₂ hydrogenation at 220 °C. (b) SSITKA results during ¹²CO₂+H₂ switch to ¹³CO₂+H₂ during steady state CO₂ hydrogenation at 220 °C. (c) Pure spectra obtained by MCR-ALS using 4.2% CO* adsorbed Ni as S2.

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Resolving the "Coloring Problem" in Crystallography: The Synergistic Power of EXAFS and Resonant Diffraction

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ABSTRACT

The accurate determination of cation distributions in complex inorganic materials—often termed the "coloring problem"⁽¹⁾—remains a persistent challenge in crystallography, particularly when elements with similar atomic numbers (e.g., Fe/Ni, Cu/Fe, or Ge/Cu) or oxidation states coexist. Conventional X-ray or neutron diffraction struggles to distinguish such species due to their nearly identical scattering factors, leading to ambiguities in site occupancy and local structure. This talk demonstrates how **combining Extended X-ray Absorption Fine Structure (EXAFS) and resonant diffraction (anomalous or multi-edge anomalous diffraction, MEAD)** provides a robust, element-specific solution to this problem, enabling the resolution of cation ordering in technologically relevant materials.

Using three case studies as exemplars:

1. **Stannoidite (Cu₈Fe₃Sn₂S₁₂)**: MEAD at Fe and Cu K-edges, coupled with EXAFS, revealed that Fe preferentially occupies the interstitial 2a, 2b, and 2c sites, resolving a long-standing ambiguity in its cation ordering. EXAFS confirmed Fe–Fe/Cu interactions, while DFT calculations validated the electronic structure and metal-metal bonding trends.
2. **Germanite (Cu₂₂Fe₈Ge₄S₃₂)**: A combinatorial approach integrating resonant powder X-ray diffraction (at Cu/Ge edges) with EXAFS and single-crystal data uncovered a Ge-rich deviation from nominal stoichiometry. The interstitial 2a site was shown to be fully occupied by Fe, with Ge localized on 6d (or 6c) sites, and residual Fe on 12f sites^[2].
3. **Lacunar CsCl-related phase (Fe_{1.5}Ni_{1.5}Ga₄)**: Resonant scattering near the Fe K-edge disproved Fe/Ni ordering, confirming statistical distribution in the 4a Wyckoff site (*1̄a3d* symmetry). DFT further elucidated the local environment and spin-glass behavior, with competing ferromagnetic/antiferromagnetic interactions arising from near-degenerate electronic states.

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EXAFS Unbound: Unlocking Atomic Scale Insights Beyond The Next Edge

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ABSTRACT

Extended X-ray Absorption Fine Structure (EXAFS) is a cornerstone technique in modern spectroscopy, offering non-destructive, element-specific insights into atomic-scale environments across solids, liquids, and gases. Its unparalleled sensitivity to local coordination geometry has made it indispensable in fields ranging from analytical chemistry to material physics and environmental science.

The precision of EXAFS critically depends on the accessible energy range above the absorption edge, which is often constrained by the presence of nearby edges from other elements. This limitation has long hindered the full exploitation of EXAFS in chemically complex systems, effectively capping its resolution and applicability.

The pioneering work of Gu et al. [1] demonstrated that simultaneous detection of total and partial fluorescence yield allows for the subtraction of overlapping signals, thereby extending the usable energy range, unlocking unprecedented structural insights in systems previously deemed inaccessible.

Despite its promise, this method remains underutilized with only a few studies reported in almost 20 years, mainly on the Mn local environment in the plant photosystem II [2], and on inorganic materials such as LaOCl [7], Pt doped SnO₂ [8], CoFe₂O₄ [9], PtAu catalysts [10-12] and Nd_{0.5}Sr_{0.5}MnO₃ [13-14].

We will discuss how this approach offers unprecedented insight into short-range order in multi-element systems such as CrMnFeCoNi and VNbMoTaW alloys, unlocking previously inaccessible structural information through EXAFS.

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From Ex Situ to Operando Soft X-rays Microscopy of Photoactive Materials: Case of Hematite-based Photoanodes

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ABSTRACT

We will present here a journey in the soft X-rays microscopy world, using mainly XANES as contrast mechanism, following several recent developments allowing today to track chemistry and chemical coordination evolution in operando conditions, at nanoscale.

Over the last few years, we successfully applied soft X-rays microscopies, STXM [1-3], spectro-ptychography and XPEEM [4], to characterize hematite-based photoanodes dedicated to solar assisted water splitting. Prototypical photoanode material with perfectly matched band gap for efficient solar light absorption, cheap, abundant, and ecofriendly, hematite offers a perfect sandbox for band engineering approaches, as Ti doping or Oxygen vacancy control. We will first present an overview of the results obtained in ex situ conditions, both from STXM and XPEEM perspective, emphasizing the need for operando approaches as unique way to tackle the mechanisms at the origin of charge transfer at the photoanode/electrolyte interface. In the second part we will address the in situ and operando methodology, under development, along with some preliminary results demonstrating the feasibility of our approach.

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Operando XAS Strategies for Rational Design of Electrochemical Energy Materials

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ABSTRACT

Electrolysis is a cornerstone technology for green hydrogen production, yet large-scale adoption remains limited by high energy requirements and expensive electrolyzers reliant on scarce elements. Recent efforts have focused on narrowing the performance gap between transition-metal oxides and noble-metal-based systems, to develop cost-effective devices through precise structural and electronic tuning¹. *Operando* X-ray Absorption Spectroscopy (XAS) has become a key technique for probing the dynamics of catalyst active sites during water reduction (HER) and oxidation (OER). We will explore the capability of *operando* XAS with the setup developed at the SAMBA beamline through two specific case studies: MoO₂/Ni cathodes and Ni-doped maghemite photoanodes.

After introducing the sample environments developed to perform *operando* XAS², we investigate MoO₂ electronic reconfiguration upon Ni interface formation. Compared with previous works³, the combination of *operando* XAS and Raman spectroscopy allows us to follow the dynamics of the active sites, revealing that Ni enhances conductivity and the binding energy of reactants by forming O vacancies that activate MoO₂. The second case study focuses on the structural and electronic effects of Ni doping in ultra-thin γ -Fe₂O₃ films for solar-driven OER. By varying Ni concentrations and employing *operando* XAS, the study reveals an optimal low-Ni doping (Ni/Fe $\approx$ 0.04) that significantly improves photoelectrochemical activity and lowers the OER overpotential compared to undoped samples and even hematite analogs. The improvement arises from balancing improved surface activity via oxygen-vacancy formation, band-gap narrowing, and OH⁻ binding-energy optimization. Using a novel *operando* pump-and-probe X-ray absorption method, we reveal the rapid formation of metastable surface superoxo species at Fe octahedral sites during illumination.

Our findings underline the critical role of *operando* XAS for rational defect engineering, offering pathways to design cost-effective catalysts that can be extended to a wide range of functional materials.

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Tuning Redox Chemistry in Gd-Doped Ceria: Gd-Driven Suppression of Ce³⁺ Formation in Reducing Condition

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ABSTRACT

Ceria (CeO₂) and rare-earth-doped ceria are among the most important oxide materials for catalytic applications and solid oxide fuel cells (SOFCs), owing to their exceptional oxygen storage capacity and high oxygen-ion conductivity. In particular, gadolinium-doped ceria (GDC) exhibits enhanced ionic conductivity through the formation of oxygen vacancies introduced for charge compensation. Although conductivity measurements have long suggested that Gd doping suppresses the reduction of Ce⁴⁺ to Ce³⁺ under reducing conditions, direct spectroscopic evidence of this effect has remained elusive.

Here, we combine **in situ** high-energy-resolution fluorescence-detected X-ray absorption spectroscopy (HERFD-XAS) at the Ce L_{2,3} edges and X-ray Raman scattering (XRS) at the Ce N_{4,5} edges with X-ray pair distribution function (PDF) analysis and density functional theory (DFT) calculations to directly probe the evolution of the cerium electronic structure during reduction. HERFD-XAS provides direct sensitivity to Ce³⁺ formation through the quadrupole-induced pre-edge feature at the Ce L₃ edge, while high-momentum-transfer XRS quantitatively follows changes in the Ce 4f multiplet structure, enabling direct determination of the Ce⁴⁺/Ce³⁺ ratio under operando conditions.

Our results provide the first direct spectroscopic demonstration that Gd doping inhibits Ce³⁺ formation during reduction by stabilizing oxygen vacancies generated through charge compensation, thereby preserving predominantly ionic conduction over a broader range of reducing conditions. Complementary PDF analysis and DFT calculations reveal that increasing Gd concentration induces significant local structural relaxations around oxygen vacancies, which progressively limit their participation in oxygen-ion diffusion. These findings identify an optimal Gd concentration in the range of **x = 0.10–0.25**, where suppression of electronic reduction is achieved while maintaining high oxygen mobility. This work establishes a direct relationship between dopant concentration, local structure, and electronic response, providing important guidelines for the rational design of ceria-based electrolytes for intermediate- and low-temperature SOFCs and other redox-active oxide materials.

The Optical Dispersive EXAFS (ODE) Beamline at Soleil Synchrotron and Its Studies

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ABSTRACT

The Optical Dispersive EXAFS (ODE) beamline at Soleil Synchrotron is a state-of-the-art facility dedicated to advancing in-situ X-ray Absorption Spectroscopy (XAS) studies across a wide range of scientific disciplines. Our research focuses on investigating dynamic processes in electrochemistry [1,2], catalysis [3], and photochemistry [4], utilizing the beamline's unique capabilities to probe electronic and structural changes in real time. Additionally, we conduct XAS measurements under extreme conditions, such as high pressure and low temperatures, to explore material behavior and properties under challenging physical environments [5-7]. By integrating cutting-edge instrumentation with innovative experimental techniques, we aim to uncover fundamental insights into the structural and electronic dynamics of materials, driving progress in energy, environmental science, and materials research.

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Combined Operando XRD And XAS Study Of Electrochemically Active Li_2MChO Anti-perovskites

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ABSTRACT

Recently, there has been a plenty of discussion dedicated to cubic anti-perovskites and their applicability in metal-ion batteries. Specifically, they have been proposed as solid-state electrolytes or cathodes for lithium-ion batteries¹⁻⁴. Although the latter is a matter of doubts, the exceptional chemical flexibility of the Li_2MChO ($\text{M} = 3d$ metal; $\text{Ch} = \text{S}, \text{Se}$) crystal lattice (Figure 1) results in a huge variety of properties of anti-perovskites apart from (or combined with) electrochemical activity⁴. Therefore, the materials of this group represent also a valuable model for fundamental physical and physicochemical studies. One of the cases are changes in crystal and electronic structure, induced by electrochemically varying the $\text{Li}_{2-x}\text{MChO}$ composition. The results of such studies might later be used for designing more practice-relevant battery materials.

The presented work is dedicated to Li_2MSeO ($\text{M} = \text{Fe}, \text{Co}, \text{Mn}$) series. Their structural stability against reversible de-lithiation was studied by a combination of operando XRD and XAS, involving synchrotron-radiation facilities. While changes in the average structure were monitored by the diffraction experiment, charge-compensation mechanism was evaluated from the spectroscopy. Subsequent EXAFS calculations performed on $3d$ elements as well as Se, revealed certain peculiarities in the initial local structure as well as provided information about its irreversible degradation for some members of the series⁵. Besides fundamental interest, this study is showcasing the way EXAFS results complement XRD.

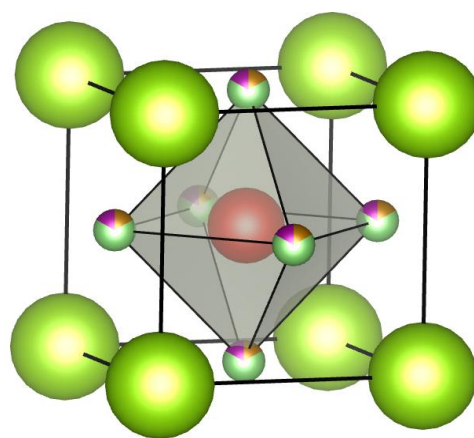


Figure 1. Crystallographic model for $Pm\text{-}3m$ anti-perovskites. Oxygen occupies the centre of the unit cell, lithium and transition metal(s) share the apices of the octahedron, while sulphur is in the apices of the cubic unit cell.

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Coupling High-Energy Resolution Fluorescence Detected X-ray Absorption Spectroscopy (HERFD-XAS) with microfluidic cells for *in situ* monitoring of tungsten nanoparticles redox transformation

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ABSTRACT

Tungsten is a redox-sensitive transition element of strategic importance for many industrial applications. The functionalities of tungsten-based materials are highly dependent on W atomic structure and oxidation state (0, IV and VI), and the same applies to the toxicity of tungsten compounds. However, assessing tungsten speciation and structural changes in complex materials and environmental matrices (e.g. sediments, biota), poses significant challenges necessitating highly sensitive analysis as well as *in situ* monitoring at short timescale.

Conventional XANES spectra at the W L₃-edge (10 207 eV) were obtained on the FAME BM30b beamline (French Absorption spectroscopy beamline in Material and Environmental science) at the ESRF, Grenoble, France using a Si(220) two-crystal monochromator. HERFDXANES spectra at the W L₃-edge with L_{α1} (~8 397 eV) and L_{β2} (~ 9 961 eV) emission lines were obtained on the FAME-UHD BM16 beamline (French Absorption spectroscopy beamline in Material and Environmental sciences at Ultra-High Dilution) at the ESRF with a similar monochromator than on BM30b. For the experiment with the L_{α1} emission line, the Crystal Analyzer Spectrometer (CAS) used 14 crystals Si(444) while for the experiment performed with the L_{β2} emission line the CAS used 14 crystals Si(555).

We demonstrated that the better energy resolution in HERFD-XAS (energy bandwidth between 1.2 to 1.5 eV) at the L₃-edge with L_{α1} and L_{β2} emission lines would allow the separation, characterization and quantification of subtle changes in the XAS spectrum associated with specific electronic transitions in W(0), W(IV) and W(VI). Moreover, HERFDXAS combined with a microfluidic chip proved suitable for *in situ* monitoring of changes in W redox states with second-scale temporal resolution.

We pointed out that such an experimental setup would be particularly useful for providing direct insight into the rapid changes in W speciation that would occur under oxidizing environmental conditions.

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Vanadium enrichment in lateritic soils: mineralogical and pedoclimatic controls

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ABSTRACT

Vanadium (V) is considered a critical raw material due to its increasing importance in metallurgy and energy storage technologies. Although its average abundance in the continental crust is relatively high, economically exploitable deposits remain scarce, and the mechanisms responsible for its enrichment are still poorly understood. Lateritic covers, which occupy nearly one-third of the continental land surface, provide valuable natural archives for understanding vanadium concentration processes, as the weathering gradients record its mobilization, oxidation, and sequestration throughout the alteration process.

In this perspective, this study aims to identify the mineralogical and geochemical processes controlling vanadium enrichment in two lateritic profiles developed under contrasting geological and climatic settings: the Syerston profile (Australia), developed on ultramafic rocks, and the Leo Falls profile (Suriname), developed on metamorphic schists. Despite moderate vanadium concentrations in the parent rocks (70–80 mg kg⁻¹), both profiles display significant enrichment, reaching approximately 1,000 mg kg⁻¹ at Syerston¹ and up to 3,700 mg kg⁻¹ at Leo Falls² questioning the origin of such differences. On the basis of preliminary geochemical and mineralogical analyses, we used an X-ray absorption spectroscopy (HERFD-XANES) approach to identify vanadium host phases and track its redox evolution along the weathering profiles.

The results demonstrate that HERFD-XANES V-K edge sensitivity to oxidation state and local electronic environment makes it an appropriate tool to follow V speciation along the studied alteration profiles. The coupling between *in situ* spectra obtained through microbeam analyses and acquisition on bulk samples reinforces our ability to identify the proportion of the distinct V-bearing phases and the mechanisms by which V is trapped. The evolution of V speciation along the two studied profiles, developed over distinct parent rocks and under contrasted pedoclimatic conditions, helps discuss the mechanisms responsible for V accumulation during supergene weathering.

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Redox speciation of cerium in the presence of colloids

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ABSTRACT

Understanding the interactions between trace metals and colloids is crucial due to their significant influence on metal speciation, mobility, toxicity, and bioavailability. Cerium (Ce), a lanthanide element, is of particular interest because of its abundance and unique redox properties, since it exists in both Ce(III) and Ce(IV) oxidation states. This redox flexibility makes Ce a powerful proxy for paleo-environmental reconstructions.¹ However, distinguishing between Ce(III) and Ce(IV) in colloids like organic matter (OM) and Manganese dioxide (MnO₂) remains challenging, especially at environmentally relevant concentrations. X-ray absorption spectroscopy (XAS) at either Ce K-, L₃- and M_{4,5}-edges offer powerful tools to directly resolve Ce oxidation states under these conditions.

In this study, Ce M_{4,5}-edge XAS was performed at the SOLEIL DEIMOS beamline for the CeOM system (pH 5-10) under both aerobic and anaerobic conditions. DEIMOS beamline offers a perfect sample environment for redox-sensitive elements with its Ar-glovebox connected to the end station. Potential formation of CeO₂ nanoparticle and their structure was determined at Ce K-edge EXAFS on SNBL BM31 beamline. Ce L₃-edge XANES for the Ce-MnO₂ and CeOM-MnO₂ systems (pH 6.5) were conducted at the SOLEIL LUCIA beamline.

In the Ce-OM system, the results showed a strong pH dependent Ce oxidation in the presence of OM under ambient atmospheric conditions, with negligible Ce(IV) at pH 5 and nearly complete oxidation (~94%) at pH 10. Dissolved oxygen (O₂) was identified as the primary oxidant, as anoxic conditions lead to negligible Ce oxidation. Results show a preferential complexation of Ce(III) by OM, which stabilizes it over Ce(IV). Ce K-edge EXAFS and transmission electron microscopy showed that oxidation of Ce(III) to Ce(IV) is driven by hydrolysis and precipitation of ~2 nm CeO₂ nanoparticles. In both Ce-MnO₂ and Ce-OM-MnO₂ systems, Ce L₃-edge XANES demonstrated the formation of Ce(IV), indicating that MnO₂ effectively promotes Ce(III) oxidation and facilitates the accumulation of surface-bound Ce(IV) on MnO₂ even in the presence of OM.

Overall, this study clarifies the mechanisms controlling Ce redox behavior in the presence of OM and MnO₂ across different pH conditions. These findings can be directly used in developing risk assessment and management strategies for Ce in aquatic environments. Longer term, they can also be incorporated into predictive models of Ce redox speciation, improving our ability to evaluate and manage Ce contamination in natural systems.

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XAS Studies Of Metal Transport In Hydrothermal Fluids: Autoclaves And Photons

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ABSTRACT

Hydrothermal fluids, though minor in the Earth's lithosphere (~1%), play a pivotal role in forming economic metal deposits by extracting, transporting, and precipitating metals. Central to this process is the solubility and speciation of metals and volatiles in the fluid phase [1]. In situ synchrotron X-ray Absorption Spectroscopy is the method of choice to probe metal species, offering direct insights into their molecular structure and concentration. When combined with *ex situ* hydrothermal methods (e.g., batch and flow-through systems) and thermodynamic modeling, XAS has revolutionized our understanding of metal transport by geological fluids.

A key challenge lies in performing *in situ* XAS measurements under extreme conditions. This requires high-pressure, high-temperature experimental cells capable of withstanding the chemical reactivity of hydrothermal fluids while allowing X-ray access. Among the available technologies, autoclaves stand out for their versatility and robustness [2]. In this contribution, we present XAS methods developed using high-pressure, high-temperature autoclaves at the FAME and FAME-UHD french CRG beamlines at ESRF. These methods enable *in situ* acquisition of X-ray transmission and fluorescence spectra for hydrothermal fluids and silicate melts, providing both solubility and speciation data for metal complexes. Our autoclaves operate across a broad range of conditions, from liquid- to vapor-like hydrothermal fluids (metal concentrations: <1–10,000 ppm) to magmatic fluid-melt systems at 1000°C [3]. Scientific examples highlight the advantages of our spectroscopic "microbatch" reactors, while integration with the FAME-UHD crystal analyzer spectrometer further enhances their capabilities.

During the last 25 years the FAME and FAME-UHD beamlines have pioneered the development of specialized XAS methods based on autoclaves, enabling unprecedented *in situ* studies of metal speciation and transport in hydrothermal systems [4]. These advancements allow for the calibration of solubility constants and quantitative predictions of metal transport, bridging the gap between laboratory experiments and natural processes.

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Probing the Speciation of Transition Metals (Mo, W) in Hydrothermal Fluids by In-Situ XAS

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ABSTRACT

Hydrothermal fluids are among the most challenging samples for X-ray absorption spectroscopy (XAS). They are characterized by high temperatures and pressures, as well as ongoing dissolution–precipitation processes. Additionally, the concentrations of dissolved metals may be very low, further complicating XAS measurements. The hydrothermal autoclave developed at the FAME beamline (BM30) combines a vitreous-carbon reaction cell with movable pistons positioned inside a furnace, all enclosed within a helium-pressure vessel equipped with water-cooled beryllium windows, enabling in-situ XANES and EXAFS measurements of hydrothermal fluids in both transmission and fluorescence modes [1]. Successive improvements in beamline optics, reactor design, fluorescence detection, and data acquisition have progressively expanded the accessible experimental conditions and lowered practical detection limits from the ppm range to a few tens of ppb for selected elements such as Mo [2]. The expertise developed at BM30 also contributed to the design of similar hydrothermal XAS instrumentation at the P65 beamline of PETRA III (DESY), demonstrating the broad impact of the ESRF hydrothermal research on the international synchrotron community [3].

In this contribution, the evolution of this methodology will be illustrated by Mo and W case studies. In molybdenum-bearing sulfur-rich fluids (300 °C, 50 MPa), in situ XAS resolved the first coordination shell of dissolved molybdenum species and provided direct structural evidence for tetrahedral thiomolybdate (MoS_4^{2-}), together with a previously unknown mixed-ligand complex, $\text{MoS}_3(\text{S}_3)^-$, with three sulfide ions and one trisulfur radical ion [2]. In tungsten-bearing sulfur-free fluids, in-situ XAS distinguished tetrahedral tungstate species in alkaline fluids (WO_4^{2-}) from octahedral complexes in acidic fluids ($[\text{H}_3\text{WO}_4^+]\cdot 2\text{H}_2\text{O}$) and provided insights into the stability of polymeric tungsten species at temperatures to 400 °C and pressures to 60 MPa [4]. These studies demonstrate how advances in synchrotron instrumentation and autoclave design have enabled the direct structural characterization of increasingly dilute aqueous complexes under realistic hydrothermal conditions.

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Beyond XAS with RIXS

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ABSTRACT

Inelastic x-ray scattering (IXS) and its resonant counterpart (RIXS) have become a well-established, powerful spectroscopic technique to investigate the physico-chemical properties of materials with a broad range of application from quantum materials, catalysis or inorganic chemistry, to magnetic systems or high-pressure research [1-4].

Here, we will review the interests of IXS / RIXS to overcome the limitations of x-ray absorption spectroscopy (XAS) through selected results obtained especially at the GALAXIES beamline at SOLEIL Synchrotron [5]. More precisely, we will see how IXS / RIXS can:

- Improve the spectral resolution of the absorption spectra below the core hole lifetime broadening effect using high energy resolution fluorescence detected (HERFD) XAS.
- Access low-energy absorption edges with bulk sensitivity using hard x-ray via X-ray Raman scattering
- Provide higher sensitivity to local, element sensitive electronic properties through 2D spectral imaging by combining XAS and emission spectroscopy in a coherent process.
- Review exotic, yet remarkable effects such as the access to non-dipolar resonances or the use of polarization of the light.

Finally, we will discuss the possible evolution of RIXS in the context of SOLEIL II.

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Study of the genesis of HDS catalysts by in situ photon-in photon-out spectroscopy

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ABSTRACT

The advent of in situ/operando spectroscopies in the field of heterogeneous catalysis was a key turning-point making possible a fundamental understanding of the relationship linking the active phase structure, reactivity, and selectivity. However conventional and widely applied X-ray spectroscopic techniques (e.g., X-ray Absorption (XAS), X-ray Photoelectron Spectroscopy (XPS)...) gives a weighted averaged signal over all the transition metal ion speciation and it is not possible to obtain a set of pure spectra corresponding to the numerous speciation (spin, covalency, oxidation state...) that the absorber undergoes during the synthesis/activation of the catalyst and the catalytic reaction. This last decade have seen the implementation of two complementary photon in photon out spectroscopies: High Energy Resolved Partial Fluorescence Detection XAS (HERPFD-XAS) and Resonant Inelastic X-ray Scattering (RIXS). By aligning the detector at an appropriate energy, HERPFD-XAS allow access to a pure XAS spectrum of only one speciation of the absorber metal. 1s-2p RIXS gives access to different electronic intermediate states of the XAS pre-edge structure whereas in classical XAS K edge, the pre-edge corresponds to the sum of these states.

We used these two spectroscopies to study the genesis of hydrodesulphurization (HDS) catalysts. The active phase of this class of catalyst is consisting of Co promoted nanometer scale MoS_2 particles dispersed on γ -alumina support. Combination of Mo with Co gives a synergetic effect in HDS activity due to the formation of the active CoMoS phase. However, the exact structure and localization of cobalt remains unclear due to the segregation of the Co element in different phases during the catalyst activation step. HERPFD XAS and 1s 2p RIXS spectra were recorded at SOLEIL synchrotron facility on GALAXIES beamline. To follow the genesis of the active phase, the catalyst was heated up to 400 °C in a ramp of 1 °C/min under a flux of $\text{H}_2/\text{H}_2\text{S}$. Using Co K edge HERPFD-XAS we were able to separate the sulfide and the remaining oxide phase. Thanks to the partial fluorescence detection and to the removal of cobalt oxide from the global signal, XAS spectrum of CoMoS shows sharper features compared to those published in the literature making comparison to theoretical XAS spectra possible. CoMoS structures showing S/M-edges were generated by DFT calculation and their corresponding Co K edge XANES spectra were calculated. The best agreement between experiment and theory is observed for tetrahedral Co localized at S-edges of the MoS_2 layers. *In situ* Co K edge 1s2p RIXS of the sulfided catalyst shows excitations to band-like unoccupied states revealing the metallic nature of the CoMoS active phase which questioning earlier XPS data processing A new XPS fitting procedure, considering the exact electronic structure of the active phase is proposed and gives rise to 20 % of Co in oxide phase and 80% in CoMoS phase. This fit is in agreement with HERPFD-XAS results unlike previous fit available in literature where a disagreement between XAS and XPS is reported (XAS show an almost total sulfidation while XPS adjustment shows a sulfidation of 40-60% of Co oxide phase).

In conclusion HERPFD-XAS and 1s2p RIXS are very powerful to characterize selectively both electronic and geometric structure making possible the localization of Co in HDS catalysts (S-edge) and showed that the promotion rate is underestimated. These findings pave the way to future investigations on the impact of Co S/M-edges substitution on the final properties of the catalyst.

Understand 4f covalency by *ab initio* calculated RIXS plane

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ABSTRACT

We present a combined experimental and theoretical investigation of covalency in cerium dioxide (CeO_2) using resonant inelastic X-ray scattering (RIXS). Within the Restricted Active Space (RAS) framework, together with our recently developed convolution approach, the full RIXS planes with polarization can be reconstructed fully *ab initio*. This enables a direct and detailed interpretation of the spectral features.

Our calculations show that the characteristic two-manifold peak structure comes from the synergy between metal-oxygen covalency and the core-hole effect. These same mechanisms also naturally generate an additional feature in the valence-to-core (vtc) RIXS region. Importantly, this feature exhibits a strong angular dependence, providing a sensitive probe of the symmetry and orbital character of the involved states. In conclusion, the topic will provide a deeper connection to the theoretical concepts of covalency, local symmetry, and scattering tensors.

As an outlook, we will also present a many-body quantum model for X-ray spectroscopy to address why cerium dioxide represents such a particular case. This model further allows us to explore whether similar signatures may be expected in other lanthanide compounds, and under what conditions such behavior can be observed experimentally.

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Tender X-ray photon-in/photon-out spectroscopy at ESRF ID26

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ABSTRACT

The energy range between 1.5 and 4.5 keV (tender X-rays) covers the K-edges of Al, Si, P, S, Cl, K and Ca, the L-edges of 4d transition metals and the M-edges of actinides. 4d transition metals and actinides can also be measured in the hard x-ray range but the smaller lifetime broadening and favorable selection rules to probe the valence orbitals directly make the tender X-ray range interesting also for those elements. X-ray emission spectroscopy (XES) in combination with X-ray absorption spectroscopy (XAS), photon-in/photon-out spectroscopy, can provide sharper spectral features and allows studying the occupied and unoccupied density of electronic states thus providing a wealth of information.

Tender X-ray emission spectrometers have been realized in dispersive and scanning geometries at various synchrotron radiation end-stations and at laboratory X-ray sources. Beamline ID26 at the ESRF features an instrument in non-dispersive, scanning geometry that employs eleven Johansson or Johann crystals in combination with a gas proportional counter or a pixel detector [1]. The 80 mm long Si crystal wafers are cylindrically bent to 1m radius in the meridional plane. The sagittal dimension is 25mm requiring a large detector surface of 50 x 25 mm². The angular range of the instrument is 35 to 85 degrees.

The short absorption length in the tender X-ray range poses several challenges with respect to spectral quality, normalization and sample environment. The need for in situ and operando studies of materials has dramatically grown with the transition to renewable sources of energy. Carrying out such studies in the vacuum chamber of the ID26 tender X-ray instrument has thus become a top priority. To this end, we developed operando cells for (electro)catalysis experiments. The presentation will present some fundamental considerations for tender X-ray photon-in/photon-out spectroscopy as well as the ID26 spectrometer with the operando cells.

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Developments of the setup for pump-probe X-ray absorption fine structure (XAFS) and a capability of EXAFS at FXE instrument, European XFEL

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ABSTRACT

X-ray Absorption Fine Structure (XAFS) is one of the most essential techniques for materials characterisation. XAFS is applied to various systems, such as solid-state materials or molecules in solution in synchrotrons. Besides, XAFS has been employed to study photoexcited states of materials on ultrafast timescales since the X-ray Free Electron Lasers (XFELs) began operating. The Femtosecond X-ray Experiments (FXE) instrument at the European X-ray Free Electron Laser (EuXFEL) is dedicated to the study of photoexcited states of materials using a pump-probe methodology. At FXE, non-resonant X-ray Emission Spectroscopy (nr-XES) has been regularly employed to track photoexcited states of molecules in solution, taking advantage of the full XFEL flux since nr-XES requires neither monochromatic x-rays nor energy scanning. Resonant X-ray spectroscopies such as XAFS, which require monochromatic X-rays at XFELs, have been more challenging because of the intrinsically stochastic X-ray spectral features of the self-amplified spontaneous emission (SASE) process.

At FXE, we have recently been focusing on developing XAFS measurements. The unique operation mode at EuXFEL, i.e. burst operation, allows us to use more X-ray photons compared to other XFEL facilities, which should translate into XAFS spectra with a good signal-to-noise (S/N) ratio and shorter acquisition times. In addition, the XAFS capability is important for our user community since XAFS is a standard measurement for materials science, and advanced X-ray spectroscopic measurements, such as high-energy-resolution fluorescence-detected x-ray absorption spectroscopy (HERFD-XAS), also become possible if XAFS measurements at FXE is established to a sufficiently high level.

In Fig. 1, pump-probe XAFS of $\text{Cu}(\text{dmp})_2$ molecules measured at FXE is displayed. The X-rays are monochromatized using a set of Si (111) crystals, and the incident and fluorescence X-ray intensities are measured using PIN photodiodes. 1000 pulses/s were used to measure XAFS and each data point was accumulated for 10 s (~10 min/spectrum). The signal-to-noise ratio (S/N) of pump-probe XAS (i.e. $\Delta\text{XAS}_{\text{positive_delay}}/\Delta\text{XAS}_{\text{negative_delay}}$) is estimated to be ~100. More X-ray pulses at EuXFEL enabled a good S/N for the 10-s data acquisition. 10000 pulses are available at EuXFEL for 10 s while 300 – 1000 pulses are available for 10 s in other facilities. The S/N of XAFS at EuXFEL can be 3 times better than in other facilities. In addition to many X-ray pulses, a shorter burst period can reduce noise from jet fluctuations, which are more pronounced at longer pulse periods. Owing to the establishment of the XAFS setup, resonant XES is also available in combination with the setup of nr-XES. Furthermore, EXAFS can be measured by scanning the monochromator and the undulator together. In this presentation, we will discuss our recent achievements in XAFS.

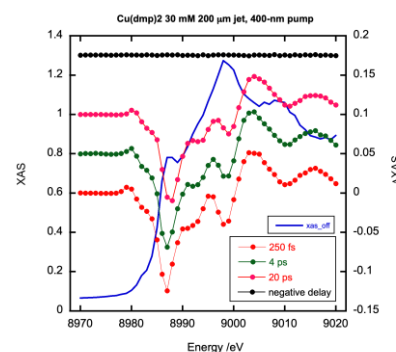


Fig. 1 pump-probe XAFS of $\text{Cu}(\text{dmp})_2$ molecules using a liquid jet (diameter: 200 μm)

FAME-PIX: The Pioneering French High-Resolution Hyperspectral 3D X-Ray Microscope at the ESRF

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ABSTRACT

While data acquisition in Ptychographic X-ray Computed Tomography (PXCT) has advanced, achieving hyperspectral nanoimaging still necessitates further speed enhancements. Identifying element locations and chemical speciation within a sample's microstructure demands numerous PXCT experiments at various photon energies. To enhance this capability, we can optimize instrumentation for expedited data collection and adjust analysis methods to minimize data requirements without compromising image quality. This can be achieved through instrumentation upgrades and refined data processing techniques that optimize data volume while preserving high resolution. We have already made progress in data analysis methods [1,2,3]. Presently, the MAGNIFIX project funding enables the construction of a new endstation on the FAME-PIX beamline at the ESRF. FAME-PIX integrates all aspects of spectral ptychography and spectral PXCT, making it the pioneer X-ray microscope in France with these capabilities. Advancements in instrumentation broaden the applications, while the need to characterize diverse samples also dictates the requirements for instrumentation. Reconstruction of the beamline has been achieved, except the experimental nanoprobe which is being prepared in the manufacturer's workshop. FAME-PIX is being superseding the preceding FAME beamline at the ESRF port BM30. FAMEPIX will benefit from the small stable source of this port of the upgraded ESRFEBS, which is approximately 40µm × 40µm. In this oral presentation, we will introduce the FAME-PIX ptychography endstation, the novel F-CRG instrument that will confer PXCT with spectral capabilities, thereby unlocking novel opportunities for characterizing heterogeneous materials.

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Operando Spatio-Temporal Study of CO oxidation reaction in Pd Catalysts using Full-Field Hyperspectral XAS imaging

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ABSTRACT

The ROCK beamline at the SOLEIL synchrotron has recently implemented Full-Field (FF) hyperspectral Quick-XAS imaging, combining element-specific XAS with micrometer-scale spatial resolution and second-scale temporal resolution for investigating the reaction pathways of functional materials under *operando* conditions [1,2]. Hyperspectral cubes containing 170 to 580 images of the sample, recorded at energy values scanning an X-ray absorption spectrum, were acquired using a pixelated CMOS camera (1.625 μm pixel size over a 3.3 mm field of view) with acquisition times as short as 2.5 s for unraveling the spatial dynamics of hydride formation during H_2 pretreatment and their subsequent impact on the CO oxidation reaction over supported Pd nanoparticle catalysts.

Hyperspectral datasets were processed using in-house developed Jupyter notebooks and analyzed by Multivariate Curve Resolution with Alternating Least Squares, enabling extraction of transient species and their spatial concentration profiles. During H_2 pretreatment, the initial reduction of the oxidized catalyst led to the formation of PdH_x species under 5% H_2 or metallic Pd under 0.08% H_2 . Depending on the support (Al_2O_3 or TiO_2), these transformations propagated through the catalyst bed as reduction wavefronts. During CO oxidation reaction, the catalysts exhibited distinct spatial and temporal phase evolution, correlated with the reaction stage and the support–metal interactions that govern the spatial variations. Below 200 °C, the catalyst remained predominantly metallic, a state associated with high activity through a Langmuir–Hinshelwood reaction pathway [3]. As the temperature increased to 300 °C, the catalyst progressively evolved towards a highly oxidized state following the heater direction, suggesting a transition towards a Mars–van Krevelen-type reaction pathway [4].

Beyond these mechanistic insights, FF hyperspectral XAS imaging also uncovered pronounced beam-induced effects for the RT H_2 uptake that remain undetectable in conventional XAS measurements. These effects were significantly more pronounced for the TiO_2 -supported catalyst, highlighting the strong interplay between the support properties, the X-ray beam, and catalyst reactivity. As the higher photon flux delivered by 4th-generation synchrotron sources becomes increasingly available, spatially resolved *operando* XAS will be essential not only for distinguishing intrinsic catalytic behavior from probe-induced artefacts, but also for improving the reliability of *operando* catalyst characterization and guiding the design of more efficient and robust heterogeneous catalysts.

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X-ray Absorption Spectroscopy meets Imaging to Unveil Inclusion Puzzles in Deep Diamonds

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ABSTRACT

X-ray absorption spectroscopy and X-ray microscopy have converged at 4th-generation synchrotron sources, enabling correlated nanoscale chemical and structural imaging. The CARNAÚBA beamline at Brazil's SIRIUS source, operating in the range 2.05–15 keV, combines absorption spectroscopy, diffraction, fluorescence, luminescence, and 2D/3D imaging in a single achromatic nano-focused platform. Here, we showcase this multimodal approach by investigating mineral inclusions trapped in deep-Earth diamonds—natural time capsules that record mantle conditions. Spectromicroscopy reveals highly inhomogeneous, defect-rich mineral phases. As a case study, we examine an iron oxyhydroxide inclusion: XANES mapping at the Fe K-edge shows point-to-point spectral variations deconvolved into goethite-like (FeOOH) and hematite-like (Fe₂O₃) components. This heterogeneity suggests that the mineral experienced extreme stress and multiple high-pressure/high-temperature phase transitions, followed by back-transformations during diamond ascent. These findings point to an unexpected water-related source in deep-mantle processes and illustrate how next-generation X-ray nanoprobe can unravel complex geological histories with unprecedented detail.

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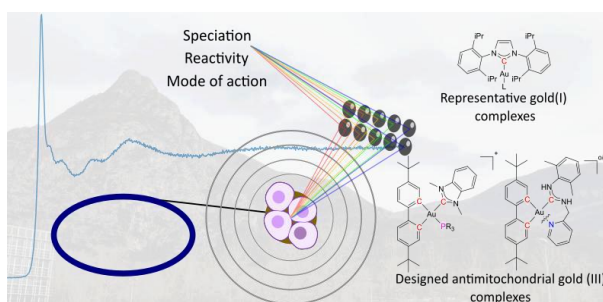
Unraveling the fate of anticancer gold complexes in cells through HERFD-XAFS

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ABSTRACT

Organogold complexes are increasingly studied for their anticancer properties both *in cellulo* and *in vivo*.^{1–3} Like any metal complex however, they are susceptible to a wide range of transformations in biological media (redox, ligand exchange) dictated by their structure and reactivity. Determining the speciation of such complexes is therefore key to understanding their mode of action, for which XAFS methodologies have proven very useful. These techniques have been employed to study the fate of Pt(IV),⁴ and Os(II)⁵ complexes in cell samples. XAFS has also been used to study the reactivity of some gold complexes with proteins^{6–8} but has never been employed to monitor their speciation *in cellulo* to date. Taking advantage of the high sensitivity and resolution of cryo-HERFD-XAS offered by the crystal analyzer spectrometer of FAME-UHD beamline (BM16, ESRF), we systematically studied the speciation of a library of representative gold(I) complexes in biological settings. A relationship between their speciation in bulk A549 lung cancer cells and their structure and biological activity was successfully established. Additionally, we could probe the speciation of two new families of biphenyl gold(III) N-heterocyclic carbene (BGC) complexes, and link their speciation to their physico-chemical properties and biological activities. This altogether allowed for a better understanding of the fate of these metallodrugs in cells and provided pivotal information regarding their mode of action.



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A short retrospective of actinide hexacyanoferrate exploration initiated at LURE

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ABSTRACT

The occurrence of actinide elements is related to the production of nuclear energy. But actinide elements are also interesting for their specific chemistry, largely due to their position at the bottom of the periodic Table and the occurrence of the *5f* orbitals in their electronic configuration. It is in the late 90s that synchrotron tools were used to investigate the speciation of actinide molecular compounds. The first article dealing with the speciation of plutonium with EXAFS goes back to 1993 with data recorded at SSRL.¹

The formation of a metal-ligand coordination bond is most often described as resulting from the use of a free electron pair around a ligand atom. The coordination chemistry of the actinides, although less well-developed than that of the *3d*, *4d*, and *5d* metals, is quite distinctive. In contrast, the lanthanide series, often presented as analogous since it also contains *f*-valence orbitals, exhibits only one stable oxidation state (with a few exceptions) and primarily ionic cation-ligand interactions.

In 1996-97 at CEA Marcoule was launch a program to study actinide molecular chemistry with X-ray Absorption techniques and this was initiated experimentally at D44 of LURE. One system of interest was the well-known Prussian Blue family of molecular edifices because they were studied for a long time with transition metals. Although the geometry of the hexacyano-metalate brick is very rigid and the octahedral geometry is favoured for both metal sites, different types of structures can be observed depending on the synthesis conditions. As a study case of fundamental actinide chemistry, we chose to combine the hexacyanoferrates entities $[\text{Fe}^{\text{III}}(\text{CN})_6]^{3-}$ and $[\text{Fe}^{\text{II}}(\text{CN})_6]^{4-}$ (respectively ferricyanide and ferrocyanide ions) as ligands with actinide and lanthanide cations at oxidation states III and IV.² These $\{\text{Fe}(\text{CN})_6\}$ entities, of rigid octahedral geometry have indeed the ability of forming bonds with a lot of cations by the way of the six terminal nitrogen atoms from the cyano ligands, the later becoming thus bridging. It appears that actinide(IV) compounds exhibit absorption spectra similar to those of hafnium ferrocyanide and significantly different from those of lanthanide ferrocyanides. In particular, the data recorded at the K-edge values for carbon and nitrogen indicate significant overlap between the valence orbitals of the actinides (or hafnium) and the π^* orbitals of the cyano ligand.³ For actinide(III) like americium or californium, analogy can be drawn with neodymium and gadolinium respectively.

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Magnetic hyperthermia at the nanoscale

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ABSTRACT

Magnetic hyperthermia is one of the key application of magnetic nanoparticles (MNPs) for catalysis or for biomedical applications ¹. Evidence for local effects of thermal activation, even at very low nanoparticle concentrations, and at their only vicinity was recently presented ². Still, the local heat exchange around those nanoprobe remains controversial with too few evidences on its origin and even on its existence. We recently used quick EXAFS to investigate heat generation inside aqueous suspensions of iron oxide nanoparticles. In line with what was published recently in the case of gold colloids³, we analysed the evolution of the Debye-Waller factor when the particles are subjected to an oscillating magnetic field, which allowed determining the local temperature. In contrast with some recent publications^{4,5}, we found the temperature inside particles to be higher than the macroscopic one. In parallel, we analysed by fluorescence decay spectroscopy the heat exchange on photosensitive ligands permanently and covalently bound to the surface of maghemite nanoparticles. The measurement of rhodamine B lifetime allowed evaluating the thermal increase, in very dilute nanoparticle dispersions, at 4 given distances ranging from 5 to 7 nanometres from the surface. We globally obtained coherent results compared to already published works achieved with thermo-labile ligands ^{5,6,7}, but without chemically cleavable groups at the surface of nanoparticles in solution.

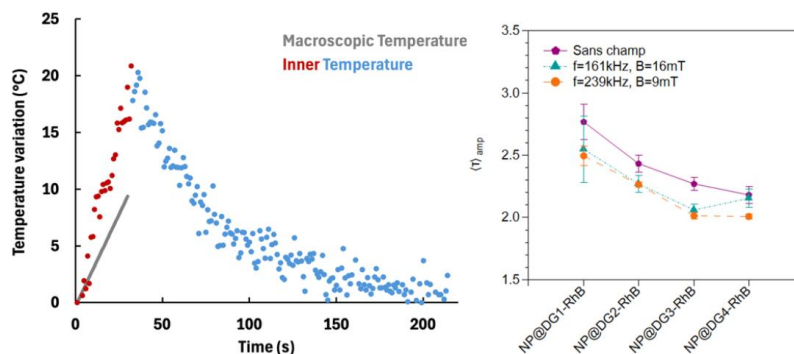


Figure 1: Temperature inside MNPs ($T_{on}=30s$ (red); $T_{off}=180s$ (blue)) and macroscopic temperature (grey) during hyperthermia determined by EXFAS analysis. The lifetime of Rhodamine B grafted on MNPs under magnetic hyperthermia.

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Understanding Nanoparticles Formation mechanisms at ITODYS Laboratory: Insights from investigations at ROCK Beamline

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ABSTRACT

Colloidal synthesis of nanoparticles (NPs) is very attractive because it gives access to a fine tuning of their size, shape and composition. However, it is still challenging to establish clear correlations between synthetic parameters and final NPs features. The Nanocat team at ITODYS laboratory develops new (electro)catalysts based on metallic alloys or oxides NPs for different applications like alcohol dehydrogenation or electrocatalytic hydrogen production and is using solutions routes (especially the polyol process) to synthesize them. In order to achieve a better control on the NPs characteristics, we are particularly interested in getting deeper insights into their mechanism of formation and for that purpose, X-ray Absorption Spectroscopy (XAS) is a powerful *in situ* characterization tool.

In particular, ROCK beamline at synchrotron SOLEIL is well-adapted for such investigations, combining Quick-XAS recording, jump-edge facility as well as several *in situ* and *operando* sample environments

The potentialities of ROCK beamline for investigating NPs formation, will be illustrated through two representative examples: the formation of Co-Ru nanoalloys by the polyol process¹ and the elaboration of hollow IrO_x cubes via Coordinating Etching Precipitating process starting from inorganic Cu₂O templates.²

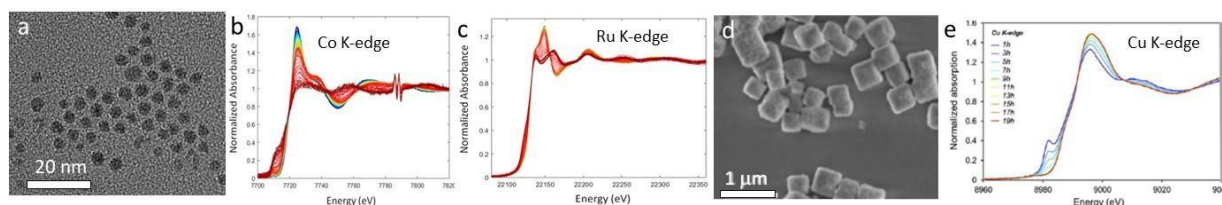


Figure 1. a. TEM image of CoRu nanoparticles and b. and c. evolution of the XAS spectra at the Co and Ru K-edges, respectively, during their formation from Co(II) and Ru(III) acetylacetonates in octan-1-ol; d. SEM image of IrO₂ nanocages and evolution of the XAS spectra at the Cu-K-edge during the transformation of Cu₂O cubes to IrO_x nanocages.

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Time- and Space-Resolved Formation Stannosilicates by *in situ* XAS

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ABSTRACT

Hydrothermal zeolite crystallization and especially the assembly of heteroatom-containing stannosilicates remain poorly understood at the atomic scale, despite their central role in designing Lewis-acid catalysts for biomass-derived transformations. In this work, we have designed a custom *in situ* cell that allows XAS measurements during Electro-Assisted Synthesis (EAS) of stannosilicates at elevated pressures and temperatures. In a series of examples, the cell was used to follow the crystallization of MFI-type materials with controlled potentials applied to Sn electrodes while measuring XAS with time and spatial resolution.

The cell was tested during two beamtimes at the SAMBA beamline of Soleil. With an internal diameter of 30 mm, it provides a sufficient X-ray path length for Sn *K*-edge XAS measurements in transmission. In a typical synthesis, the gel containing the silica source was loaded inside the cell, while two tin electrodes submerged in the gel were used as the Sn source. The applied potential followed either a square or sinusoidal shape with an amplitude of 2 or 4 V. XAS data collection started simultaneously with the synthesis, although a detectable edge jump was only obtained after Sn was released from the electrode into the gel. The cell was equipped with two 4-mm windows, allowing us to follow differences in Sn species along the horizontal and vertical directions.

Using PCA and MCR-ALS approaches, we show that at least two types of Sn species were formed during the EAS procedure. Both species correspond to Sn(II) with the same symmetry and a similar local environment. However, the second species possesses a second shell in FT-EXAFS attributed to a Sn-O-Si contribution that is absent in the first one. The absolute concentration of each species shows a clear correlation with the applied potential and the distance from the electrodes. Notably, the synthesis without a silica source showed only one type of Sn species, without the second-shell contribution. In contrast, standard batch crystallization without EAS results in Sn(IV) species and overall lower concentrations of Sn incorporated into the crystals. The *in situ* XAS results were also complemented by a wide range of other laboratory measurements and synchrotron SAXS data.

In summary, we have developed a custom *in situ* cell that was successfully used to follow Sn speciation during EAS crystallization of MFI-type stannosilicates. This provides an illustrative example of how complex the systems studied by XAS can be: mixtures of gas, liquid, and solid phases; crystallization under high pressures and temperatures; with the possibility to apply an external electric potential.

Redox speciation of contaminants at magnetite surface

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ABSTRACT

Magnetite (Fe₃O₄) is a mixed-valence iron oxide, with a Fe(II)/Fe(III) ratio ($0 \leq R \leq 0.5$) that critically controls its redox reactivity toward trace metals. While large magnetite particles are thought highly stable, their stoichiometry (R) are largely affected by pH and redox potential (Eh) at the nanoscale. For instance, XMCD at the L_{2,3}-edge and wet chemistry showed that 10 nm magnetite nanoparticles are not only sensitive to oxidant like O₂ or H₂O₂ but they release Fe(II) in solution at pH < 7.¹ A thermodynamic model was developed to predict effective R and redox potential (Eh) as a function of initial R and pH. Hence, nanomagnetites are particularly interesting model colloids to investigate contaminants (e.g. Cr(III/VI), Cu(II/I/0) or U(IV/V/VI)) redox behavior by XAS under controlled pH and Eh conditions, in order to develop and calibrate predictive models of contaminants speciation in the environment.

The Cr case is simple, as Cr(III) prevails at magnetite surface for $0.1 \leq R \leq 0.5$, as shown by Cr L₃-edge XAS at LUCIA beamline (SOLEIL), and as expected by modeling.² Conversely, Cu and U redox state distribution did not match thermodynamic calculations in the aqueous solution. Indeed, L_{2,3}-edge XAS results on DEIMOS beamline (SOLEIL), under moderately reducing conditions, Cu(II) prevailed at the magnetite surface while Cu(I) prevailed in solution. At lower Eh, Cu(II) was not reduced to Cu(0) but Cu(I) was stabilized at magnetite surface.³ Similarly, U L₃-edge HERFD-XAS at BL39XU beamline (SPring-8) revealed that U(VI) was not reduced to U(IV), but U(V) was found stable on a large range of pH and R conditions.⁴ If Cu(II) enhanced stability field can be explained by the high affinity of Cu²⁺ for surface -OH groups, Cu⁺ and UO₂⁺ very likely incorporate into magnetite, as they generally weakly bind to metal oxide surfaces under circumneutral pH conditions.

These results demonstrate that contaminants interaction with magnetite surface play a key role on their oxidation state distribution. This opens new paths for the improvement of geochemical models that help predicting contaminants mobility, toxicity, and environmental fate.

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Mineralogical Controls on the Fate of Emerging Contaminants in Laterites Revealed by XAFS

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ABSTRACT

The supply of critical metals (e.g., REE, PGM, Nb, Ta) is increasingly unstable due to the multiplication of conflicts and the growing demand of materials essential for a wide pan of industries including weapons, artificial intelligence and low-carbon technologies. To meet this demand and secure the supply, primary extraction, processing, and recycling have increased, resulting in substantially increased environmental fluxes of these elements¹. However, as their environmental release is relatively recent, these elements are considered emerging contaminants, for which their mobility and fate at the Earth's surface remain poorly understood.

The critical zone constitutes a major reservoir for critical metals. In particular, weathered regoliths such as laterites represent open-field laboratories for investigating their long-term geochemical cycling under intense weathering conditions. Nonetheless, the mineralogical and geochemical processes controlling the dynamics of critical metals remain largely unknown because these elements are commonly hosted by scarce, poorly crystalline, and submicrometric mineral phases that are difficult to characterize using conventional analytical techniques.

To tackle this issue, our team developed a molecular-scale approach funded on X-ray Absorption Fine Structure (XAFS) spectroscopy at synchrotron facilities to determine the speciation of critical metals in lateritic systems, i.e. the distribution of metals in mineral phases as well as their local atomic environment. Our work on a selection of critical metals showed their extensive redistribution during weathering, from primary magmatic minerals to secondary phosphates, oxides, and oxyhydroxides^{2,3}. The sensitivity of XAFS revealed the binding mechanisms of these metals, which are either structurally incorporated or adsorbed, depending on the intrinsic geochemical properties of the metal^{4,5}. Although they are poorly concentrated in metals or occurring as nanometric phases, these secondary minerals control the fate of critical metals in the environment.

As the use of these elements continues to expand rapidly, understanding their mineralogical controls is fundamental for predicting their environmental fate and help assess their ecotoxicity. In particular, this work contributes to assess the potential of mineral phases to scavenge these metals and effectively limit their concentrations and bioavailability in ecosystems.

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Thermal transformation of clay minerals probed by in situ soft-X-ray absorption spectroscopy

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ABSTRACT

For the past 20 years, the LUCIA beamline has provided the scientific community with soft-X-ray absorption spectroscopy (XAS) experiments to measure the K-edge of light elements such as Al, Si and Mg.¹ These elements are constituents of a large number of mineral phases, particularly clay minerals. A recurring question is determining how the atomic coordination in these nanostructures changes in response to external activation.

Thermal activation is one of the most commonly applied process to modify the structure, porosity and surface reactivity properties of aluminosilicate clay minerals. During thermal annealing, clay materials may proceed through intermediate and metastable ('meta') states, which are particularly suitable for use in the ceramics industry² or in the development of lowcarbon cements.³ Despite the strong interest in these phases, an overall description of the thermally induced atomic reorganization in clay minerals is missing.

The objective of this presentation is to provide an overview of the performance of the LUCIA beamline for in situ temperature measurements using a compact furnace capable of thermal annealing up to 1000°C while acquiring XAS fluorescence spectra in flyscan mode. This will be illustrated using natural halloysite nanotubes ($\text{Al}_2\text{Si}_2\text{O}_5(\text{OH})_4$)⁴ and synthetic imogolite-type nanotubes ($\text{Al}_2(\text{Si,Ge})\text{O}_3(\text{OH})_4$).^{5,6} We will demonstrate that it is possible to obtain new insights on the mechanisms of structural reordering during thermal transformation through the quantitative analysis of the acquired spectra using a multivariate curve resolution alternating least squares method. The findings will highlight the influence of initial morphology (tube vs. sphere vs. platelet) or chemical composition (e.g. Si vs. Ge) on thermal reactivity of the clay minerals. From a practical point of view, the comparison of different natural and synthetic samples chemically similar shows important differences on the temperature onset at which the octahedral Al site conversion begins, which could be economically attractive in terms of energy costs for industrial applications. These results also open up perspectives for (re)examining the temperature-dependent transformation of other 'oxide' phases using in situ low-energy XAS.

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Assessing the Sensitivity of X-ray Absorption Spectroscopy to Distinguish Various Forms of Nucleic Acids and Their Surface Structures on Imogolite Nanotubes

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ABSTRACT

The origin of life theory relies on the formation of not only simple organic building blocks, such as nucleobases, nucleosides, and nucleotides, but also their further polymerization into nucleic acids like DNA and RNA. While the prebiotic synthesis of monomers is widely accepted, the mechanisms driving their polymerization into oligomers remain elusive, however, a potential role of mineral surfaces in this reaction is suspected. This study explores the potential role of aluminosilicate nanostructures, specifically imogolite nanotubes found in volcanic soils, in facilitating the formation of oligonucleotides from nucleotides. A key objective was to evaluate whether X-ray Absorption Spectroscopy (XAS) could distinguish between the signals of monomers and oligomers adsorbed on imogolite surfaces.

XAS measurements were conducted using a Si(111) monochromator and a low-temperature cryostat, with signal detection via fluorescence. Nucleotide adsorption experiments on imogolite were performed at pH 6.5–7, using initial concentrations ranging from 5 to 300 μM /l. The study included a comprehensive set of reference compounds, such as DNA and RNA nucleotides (dCMP, dGMP, dTMP, dAMP, UMP, GMP, AMP), oligomers (Poly T, Poly C, Poly A, Poly G, TBA), and minerals (Al-PO_4 , apatite, Fe-PO_4 gels, phosphite salts). To ensure reliable data extraction and structural analysis, we employed a multi-faceted approach, integrating principal component analysis (PCA) of XANES and EXAFS spectra, linear combination fitting (LCF) for EXAFS data, and shell-by-shell EXAFS modeling for refined atomic-layer structural determination.

XAS successfully discriminated inorganic phosphate from nucleotides and ascertained surface bonding of nucleotides on imogolite, but it showed limited sensitivity in distinguishing monomers from oligomers. While using XANES oligomer identification was not conclusive, EXAFS provided partial differentiation through phosphodiester bond detection. The study confirmed XAS sensitivity for most species but highlighted challenges in resolving subtle structural differences, particularly for adsorbed oligomers.

In terms of nano-mineral surface reactivity, this study demonstrates the high affinity of nucleotides for imogolite nanotube surfaces, surpassing traditional clays, with XAS confirming P–O–Al bonds that validate direct phosphate–aluminum interactions. Near-complete adsorption at low concentrations, along with structural modeling, suggests imogolite may facilitate nucleotide stabilization and possible oligomerization, a step towards addressing the missing link in the origin of life theory.

X-RAY ABSORPTION SPECTROSCOPY IN SURFACE SCIENCE ON LUCIA

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ABSTRACT

Surface science studies quite often deal with the physics of deposits with thicknesses that can be as low as a tenth of a monolayer, including aggregates made up of a few atoms. Among information of interest, the atomic structure is particularly relevant, the challenge being to achieve the required sensitivity to small quantities. Thanks to a specific ultra-high vacuum facility, X-ray absorption spectroscopy offered by the LUCIA beamline [1] provides access to: (i) the electronic structure of the deposited element (chemical speciation), (ii) the local atomic structure around it, and (iii) the interactions between the deposited layer and the substrate. With this abstract, we propose to give an overview of the contribution of X-ray absorption spectroscopy to surface science through various experiments carried out on LUCIA beamline.

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Operando XAFS of Zr-based multicomponent Alloys for Ni–MH battery applications

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ABSTRACT

The C14 Laves phase is frequently observed in Zr-based multicomponent alloys and provides a versatile structural platform for reversible hydrogen storage under moderate temperature and pressure conditions. In this work, two C14-dominant alloys, $\text{Zr}_{33}(\text{CrMnFeNi})_{67}$ (Zr_1) and $\text{Zr}_{33}\text{Cr}_{22}\text{Mn}_{15}\text{Fe}_{25}\text{Ni}_5$ (Zr_5), were investigated as negative-electrode materials for Ni–MH batteries. Despite exhibiting similar gas–solid hydrogen-storage behavior, with hydrogen capacities of approximately 1.0–1.1 wt.% H_2 ^[1], the two alloys showed markedly different electrochemical responses. After optimization of the alkaline surface treatment, Zr_1 achieved an electrochemical capacity close to its theoretical value at low current density, whereas Zr_5 exhibited considerably lower capacity. This pronounced contrast indicates that favorable bulk hydrogen-storage properties alone are not sufficient to ensure efficient electrochemical hydrogen uptake. X-ray photoelectron spectroscopy (XPS), electrochemical impedance spectroscopy (EIS), and distribution of relaxation times (DRT) analysis were employed to investigate differences in surface chemistry, interfacial charge-transfer processes, and hydrogen-transport kinetics. In addition, *Operando* electrochemical Zr K-edge XAFS measurements were performed on Zr_1 at ROCK beamline to follow the electronic and local structural evolution during hydrogen cycling. Preliminary data treatment will be presented.

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Deciphering Li^+ and Mg^{2+} Intercalation Pathways in Chevrel-Phase Mo_6S_8 by Multi-Edge X-ray Absorption Spectroscopy

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ABSTRACT

Chevrel-phase Mo_6S_8 is a benchmark cathode for rechargeable Mg batteries because its open cluster framework can accommodate both monovalent and multivalent ions. In Mg-Li hybrid batteries, however, Li^+ and Mg^{2+} compete for interstitial sites, and their individual contributions are difficult to separate using electrochemistry or diffraction alone. Li^+ generally supports rapid insertion, whereas Mg^{2+} can experience desolvation barriers, site-specific trapping and sluggish transport. Here, electrochemical cycling is combined with Mo K-edge XANES and EXAFS and S K-edge XANES to compare charge-carrier-dependent insertion across selected states of charge. Mo K-edge XANES follows the Mo electronic state, while EXAFS resolves changes in local Mo-Mo and Mo-S coordination. The S K-edge probes unoccupied S 3p-Mo 4d hybridised states, providing a complementary fingerprint of sulfur-centred charge redistribution and Mo-S covalency. In situ X-ray diffraction tracks long-range phase and lattice evolution, while galvanostatic measurements, cyclic voltammetry and electrochemical impedance spectroscopy relate the spectroscopic response to kinetics, polarisation and reversibility. Initial electrochemical analysis indicates a faster and more reversible response for Li^+ insertion, whereas Mg^{2+} insertion is more kinetically constrained. The multi-edge approach tests whether these differences arise from distinct local structural rearrangements or from charge redistribution within the Mo_6S_8 cluster. By correlating Mo- and S-specific electronic and coordination changes with in situ diffraction and impedance, this work resolves ion-storage pathways and transient local structures that are obscured in diffraction alone. It also informs reliable operando XAS strategies for battery materials, including cell geometry, time resolution and multi-edge measurement sequencing.

Unlocking Reliable Operando XAS Characterization of Electrode Materials with Full-Field Hyperspectral Imaging.

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ABSTRACT

Since the 1990s, X-ray absorption spectroscopy (XAS) has been used as a chemical probe to characterize electrode materials, initially in ex situ experiments to determine the local structure and oxidation state of the involved cation¹. These early studies, however, faced limitations due to electrode removal from electrochemical cells (such as artefacts from washing, short-circuiting during disassembly, undesired air exposure, or potential state-of-charge variations)²⁻⁴.

To address these challenges, in situ approaches were developed, allowing measurements under more controlled conditions²⁻⁵. Yet another two decades passed before operando measurements became feasible, driven by advances in synchrotron technology⁶. The advent of 3rd- and 4th-generation synchrotrons, with their higher photon fluxes, enabled faster detection modes and improved signal-to-noise ratios. While these upgrades facilitated operando studies, they also introduced new issues: increased photon flux and brilliance could induce transient or permanent beam effects on the electrodes⁷⁻⁹. Despite significant improvements in electrochemical cell design leading to more reliable and reproducible performance, certain limitations persist¹⁰. To overcome these obstacles and enable robust operando characterization of electrode materials, full-field hyperspectral imaging in the Quick-XAS mode emerges as a promising approach¹¹. By combining a temporal resolution of a few hundred milliseconds with a spatial resolution of a few micrometres on the ROCK beamline, this technique provides unprecedented insights into the dynamic evolution of electrode materials. By integrating chemometric analysis and dose-density mask analysis, we further demonstrate that beam-induced and cell-related artefacts can be mitigated, ensuring more reliable interpretations of operando XAS measurements¹².

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Linking Dose to Damage: The Need for Dosimetry in Synchrotron Science

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ABSTRACT

The control of absorbed dose is a central issue in many fields where ionizing radiation is used. It is now well regulated and modeled in critical sectors such as medicine (radiotherapy, imaging) and nuclear applications. However, in the field of synchrotrons, with the exception of protein crystallography[1] and cultural heritage science [2,3], dosimetry remains relatively underdeveloped and rarely standardized.

This gap is all the more problematic given that modern synchrotrons deliver extremely intense X-ray beams, whose performance continues to improve with recent facility upgrades (source upgrades, increased brightness, finer focusing). Moreover, experiments involve a very wide variety of samples (biological, polymers, complex materials, in situ systems), each exhibiting very different absorption properties.

In this context, the lack of robust and accessible tools to estimate the absorbed dose constitutes a significant limitation: it can affect the reproducibility of experiments, complicate the interpretation of results, and lead to uncontrolled degradation of samples. The development of dosimetry tools adapted to the specific characteristics of synchrotrons therefore represents a major scientific and methodological challenge.

We present ADose, a software that aims to rapidly estimate the order of magnitude of the absorbed dose and dose rate for the very specific case of a sample irradiated by X-rays, covering the widest possible range of samples (solids, liquids, solutions, or multilayer stacks) and an energy range of 100eV -100keV. In ADose, the estimated dose accounts for photoelectric effect, Rayleigh effects, and Compton scattering. The implemented parameters allow ADose to provide an optimal description of a wide variety of experiment types (such as spectroscopy, diffraction, etc.) and sample types. We have performed a detailed validation based on comparison with experimental dose measurements with dosimeters and the RADDOS-3D code. Finally, ADose provides as a unique feature, the implementation of the heat equation to estimate the thermal heating of the sample. The limitations of this first version of ADose will be discussed. ADose is provided as an open-source jupyter notebook available under GNU AGPLv3 license [4,5].

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Spectroscopic Analyses Of “African” Glass Beads Highlighting Reciprocal Influences Between African And Europe Production (19th-20th Century)

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ABSTRACT

From the end of the 19th century European colored glass beads, also called “trade beads”, have been massively exported to Africa with the expansion of colonial trade networks. Mass production centres such as Murano near Venice or Bohemia adapted their design to the observed African tastes. Reciprocally a local craftwork emerged in several countries such as Ghana where the Krobo people developed their own design with Akoso or Bodom beads and even imitate now the *millefiori* beads initially invented in Italy. These mutual influences are a source of anthropological questions concerning the evolution of the materials choices, the technology and the manufacturing know-how of colored glass beads between these two continents.

We selected a corpus of a few tens of beads from the Musée de l'Homme and from private collections corresponding to beads either produced in Europe or in Africa at the end of 19th century or during 20th century and always intended to the African market. The aim of this study was to characterize the composition of the glass matrix and the chromophore elements in order to gain insights into raw material sourcing, potential glass recycling practices and coloring techniques.

We used several spectroscopy and imaging techniques: synchrotron radiation based XRF and XAS at the PUMA beamline in the synchrotron SOLEIL, PIXE at the accelerator AGLAE (C2RMF), Raman and optical (UV-visible reflectance) spectroscopies. These techniques allowed to obtain elemental mappings of beads from sodium to heavy elements such as uranium complemented with punctual chemical analysis (XAS, Raman and UV-visible reflectance) in elementary homogenous regions of interest. The results obtained enabled us to discuss and precise the provenance of the beads as well as the production date thanks to the trace elements detected or to the coloring agent identified. In particular we highlight how XAS measurements provide complementary information to Raman and optical spectroscopies to characterize the coloring agents.

XANES Insights Into The Composition And Alteration Of Historical Painted Metal

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ABSTRACT

During the 19th century, the rise of industrialization led to a significant expansion in the production of metal objects. Many of these objects were subsequently painted, both for decorative purposes and to provide protection against corrosion. Today, many museums care for numerous collections composed entirely or partially of painted metal objects. Often associated with industrial and technical heritage, these collections are frequently accompanied by limited historical documentation concerning the formulation of their paints. Such gaps in knowledge present significant challenges for their conservation and restoration, while also hindering our understanding of the materials, techniques, and practices employed in their manufacture.

Martin Toys are one of the flagship collections of the Musée des Arts et Métiers (Paris, France). Small painted metal automatons depicting a variety of characters, these toys provide a true glimpse into French society in the late 19th and early 20th centuries. The present research addresses two complementary aspects. First, it focuses on the characterisation of the paint formulations used on these toys, including their pigments, fillers and binders. Second, it aims to investigate the alteration mechanisms occurring within the paint layers and at their interfaces, as well as at the metal substrate. This collection provides a unique opportunity to investigate the distinctive features and behaviour over time of such composite objects, through a complementary multi-technique and multi-scale approach.

Following preliminary analyses of the microsamples collected from a selection of objects using conventional analytical techniques (XRF, Raman, FTIR and SEM-EDS), synchrotron radiation-based μ -XRF mapping was performed at the DiffAbs beamline of synchrotron SOLEIL to further refine the elemental composition of the paint layers and metal support. In addition, μ -XANES analyses were conducted at the Cr and Zn K-edges and at the Pb L₃-edge to investigate the local environment and oxidation state of these elements. This complementary multi-technique approach confirmed the presence of lead white ($2\text{PbCO}_3 \cdot \text{Pb}(\text{OH})_2$), zinc white (ZnO) and lead chromate (PbCrO_4) in the paint layers, while revealing several alteration products, including lead oxide (PbO), lead acetate ($\text{Pb}(\text{CH}_3\text{COO})_2$), lead soaps and Cr(III) degradation products.

Phase-Selective Spectroscopy Of Mixed 2D Oxides Using Diffraction Anomalous Near-Edge Structure

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ABSTRACT

Understanding catalysts is crucial if we wish to move our society towards green technology. Having 2D model catalysts on single crystals let us mimic the condition of industrial catalyst. Even with such simple systems as mono- or bi-metal oxides give valuable information when measured in operando. Nevertheless, we must not expect to have only one single 2D model catalyst in the real applications. Usually, a more complex combination of crystal phases will be found intertwined on real sample surfaces.[1] It is on these multiphase structures we wish to detangle the signals and get spectra of the individual phases

Obtaining X-ray absorption near edge structure (XANES) from a specific phase in a multiphase system is difficult under normal conditions, as the data is averaged over all different XANES signals.

However, by using surface diffraction we can separate the individual phases on the crystal and by performing anomalous surface diffraction we can get chemical spectra for the individual elements in their respective phases. Measuring the energy across the near-edge regions gives us the diffraction anomalous near-edge structure(DANES)[2]. Thus, DANES gives a complete picture of the individual crystal structures and their chemical composition.

We scanned the energy across the Co and Fe k-edge while recording the diffraction intensities on a 2D detector. This allows for the detection of the two elements in the individual phases on the sample. In our study we have looked at two different samples of ultrathin films of Co and Fe oxides.

The first sample was made purely with Fe deposit using molecular beam epitaxy (MBE) in an oxygen atmosphere on Ag(100) single crystals. On the sample surface we found Fe as Fe²⁺ in monolayers of FeO and Fe³⁺ in a trilayer of Fe₃O₄ simultaneously. Knowing that we could detangle the signal from two almost identically phases we proceeded to a more complex sample with a mixture of different phases. Those we made by co-deposition of Co and Fe in an oxygen atmosphere with MBE onto an Ag(100) crystal. We identified the oxidation states of Co and Fe together with their presence in a 2D Fe₃O₄-like film as well as Fe₃O₄ in bulk structure. Co was additionally found as FCC Co-nanoclusters. All coexisting on the same sample surface.

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Local Structure of High-Entropy Tungstates

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ABSTRACT

High-entropy oxides (HEOx) are compositionally complex materials in which several cations share the same crystallographic sublattice, producing strong chemical disorder and unusual functional properties [1,2]. Understanding how this chemical disorder is structurally accommodated at the atomic scale is essential for relating the composition to the physical properties. Previous PDF/RMC [3] and EXAFS studies [4,5] have shown the importance of local distortions in HEOx, but systematic composition-dependent investigations remain scarce. So our team initiated a detailed study of the composition – local structure – property relationship thanks to a CSC PhD thesis and CNRS Chimie Emergence project. We are investigating the local structure of high-entropy tungstates with the wolframite-type structure $(M_5)WO_4$, where five cations share the M site. Laboratory X-ray diffraction and neutron diffraction @ILL show that the average wolframite structure is retained across the investigated compositions, but individual information on each type of M cation cannot be resolved from this average structure. Therefore, to probe the response of local structure to the chemical disorder, we take advantage of the element selectivity of XAS. HERFD-XAS on FAME-UHD@ESRF was thus employed at low temperatures to probe the W, Ni and Co environments in a series of model $(M_5)WO_4$ HEOx.

We will present our first results and conclusions at the Co and W edges. XANES spectra reveal only minor changes in oxidation state and electronic structure among the investigated compositions. In contrast, EXAFS data highlight distinct first-shell structural responses at each cation site. Within the Co-containing HEOx subset, the fitted Co–O bond-length splitting is systematically reduced relative to $CoWO_4$, whereas the W–O one increases. This opposite response indicates that the chemical disorder is accommodated differently around the Co and W sites, despite the preservation of the average crystal structure.

Further EXAFS measurements at additional absorption edges, together with neutron total scattering PDF/RMC analysis, will be used to determine how these local structural responses depend on the cation chemistry and how they propagate beyond the first coordination shell. Such a combined element-selective and total-scattering approach should provide a route toward establishing the link between the chemical composition, the local structural disorder and the thermal behaviour of high-entropy tungstates.

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High-Entropy Nanoalloys: Insight Into Formation And Structural Transformation By Electron Microscopy and X-ray Absorption Spectroscopy

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ABSTRACT

High-Entropy Alloys (HEAs) represent an emerging class of multimetallic materials with strong potential for catalysis and energy-related applications. Composed of at least five elements in near-equiatomic proportions, they enable fine tuning of their electronic and structural properties [1, 2]. However, their chemical complexity and the richness of their transformations under operating conditions make their characterization particularly challenging, limiting the understanding of structure-property relationships.

In this context, we propose a correlative approach combining transmission electron microscopy (TEM) and X-ray absorption spectroscopy (XAS), including *in situ* experiments, to investigate the formation of porous HEA-based nanocrystalline particles. These materials are synthesized through polymer templated spray-drying, a protocol that allows to easily tune the chemical composition [3]. We studied three systems: RuRhPdIrPt (S6), NiRuRhIrPt (S7) and NiRuRhPdIr (S16), with the objective of understanding the effect of replacing noble metals with non-noble metals on morphology and chemical segregation.

Ex situ XAS and TEM analyses revealed that the first two systems exhibit structurally and chemically homogeneous nanocrystals with narrow size distribution. However, replacing Pt by Ni leads to pronounced phase segregation into Pd-rich domains and NiRhIr ternary alloy identified by TEM and confirmed by XAS. In order to understand the origin of this segregation, we carried out *in situ* XAS and TEM characterization.

For *in situ* XAS, we took advantage of the edge jumping capability of the ROCK-SOLEIL Quick-EXAFS beamline, which permits the monitoring of XAS at different chemical element edges during the same reaction, to record sequentially and cyclically time-resolved XAS datasets over three energy ranges - 3d-XAS for Ni (8.3 keV), 5d-XAS covering L₃-edges of Ir (11.2 keV), Pt (11.6 keV) and 4d-XAS for Ru (22.1 keV), Rh (23.2 keV), Pd (24.4 keV) - along the heating ramp (1°C/min from 25 to 500°C) of the various polymer-metal precursors leading to the HEAs. Multivariate analysis of the *in situ* Quick-XAS data for NiRuRhPdIr (S16) demonstrated that the first nucleation event coincides with the reduction of approximately half of the PdCl₂ to metallic Pd, which occurs approximately 100°C earlier than the reduction of the other metallic precursors. The second occurs when the remaining elements reach approximately 50% reduction.

In situ TEM monitoring of calcination of NiRuRhPdIr (S16) composition revealed distinct two nanoparticle nucleation events well separated in temperature, whereas NiRuRhIrPt (S07) forms in a single event occurring in a narrower temperature range. These results indicate that the early reduction of Pd triggers the phase segregation observed in the final material, a phenomenon already observed in similar system [4].

This correlative study combining *in situ* TEM and XAS provides a multiscale understanding of the formation and structural evolution of high-entropy nanoalloys. Our results demonstrate that differences in reduction kinetics of the precursors can strongly influence the final chemical distribution, giving either homogeneous alloys or phase segregation.

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Chemical and structural insights from Al K-edge XANES – alumina and mixed-metal aluminates

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ABSTRACT

Aluminates make an essential group of materials for industrial applications in the energy and environment sectors. Extremely abundant, light and affordable, aluminates are used in the manufacturing of a great variety of adsorbents, catalysts and ceramics. The properties of such materials (thermal, chemical, electrical) may be tuned thanks to the diversity of compositions and structures that aluminates may take. From simple hydroxides to mixed metal oxides, the tuning of the functional properties of aluminates relies on the mastering of their synthesis, shaping and thermal transformation processes. The chemistry of aluminates must therefore be controlled from ambient aqueous conditions to high-temperature solid-state reactions. The description of the chemical and structural state of aluminum has benefited from various techniques such as FTIR, XRD and ²⁷Al-NMR which have provided a strong framework for rationalizing material manufacturing and properties tuning.

The XAS of aluminum has been investigated in several instances for studying natural and synthetic aluminates [1-3]. With an onset energy of 1.56 keV, the Al K-edge may be probed at synchrotrons in few dedicated soft X-ray beamlines with transmission or fluorescence spectroscopy setups. Highly sensitive to the coordination environment of Al³⁺, the XANES of the Al K-edge reveals interesting electronic properties of the solid compounds distinctive of tetrahedral, pentahedral and octahedral environments affected by various distortion and long-range order effects. Used with chemistry, time or space resolved spectroscopic methodologies, Al K-edge XANES was shown to reveal interesting chemical and structural behaviors of aluminate systems not accessible by other means [4].

This communication will present Al K-edge XANES data obtained from the HERMES and LUCIA beamlines of synchrotron SOLEIL on synthetic samples relevant to catalysis and thermochemistry. The experimental and computed spectral features of the different compounds will be discussed in reference to their structural parameters. Two cases will be described. First is the aqueous phase chemistry of aluminates leading to (oxy)hydroxides and the thermal transformation of the latter to transition alumina. Second is the high temperature (>800°C) transformation of mixed-metal aluminates in cycling redox atmospheres [4].

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XAFS reveals Copper Accumulation As Disordered Multinuclear Cu(I)–Thiolate Assemblies In Human Cancer Cells

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ABSTRACT

Copper (Cu) is an essential trace element involved in redox regulation, mitochondrial respiration, and cell proliferation. Many cancer cells display altered Cu uptake and intracellular handling, making Cu an emerging therapeutic target.¹ For instance, Cu stored within vesicular cellular structures can be efficiently remobilized by Cu(I) ionophores, promoting redistribution toward the cytosol and ultimately leading to cancer cell death.² Nonetheless, the molecular form in which excess Cu is stored inside cancer cells remains largely unknown.

We took advantage of the selectivity of XAFS to interrogate Cu in human lung adenocarcinoma (A549, H322, and H358) and hepatocarcinoma (HepG2/C3A) cell lines exposed to Cu. The cells were exposed to CuCl₂ in their culture medium for 24h, harvested, washed, and frozen in liquid nitrogen, in order to analyze the internalized Cu and preserve its speciation. XAFS spectra in total fluorescence yield mode were acquired on the beamline BM30-FAME at the ESRF.

The XANES spectra indicate that Cu(I) is the dominant oxidation state in all cell lines, and EXAFS shell fitting reveals a coordination sphere made of sulfur atoms from thiolate moieties. On average, Cu is coordinated by 2.2 to 2.5 S atoms at 2.25 to 2.26 Å, consistent with mixed digonal and trigonal geometries.³ In all cell lines, second-shell Cu–Cu interactions indicate heterogeneous, structurally disordered Cu clusters. Pulmonary cells (A549, H322, and H358) show multinuclear clusters averaging ~3–4 Cu second-shell neighbors per Cu absorber, with Cu–Cu distances of 2.66 to 2.87 Å. In HepG2/C3A cells, increased Cu nuclearity and structural disorder point to more aggregated and heterogeneous Cu assemblies. The fine analysis of the real part of the FT EXAFS signal was crucial to determine the nuclearity of the Cu clusters.

These findings, combined with single-cell XRF elemental nano-imaging performed on the beamline ID21 of the ESRF, suggest that pulmonary and hepatic cancer cells accommodate excess Cu predominantly as multinuclear Cu(I)–thiolate complexes, supporting a detoxification mechanism involving cysteine-rich intracellular components. Cu₄S₆- and Cu₅S₇-type motifs likely contribute to the intracellular Cu pool, particularly in pulmonary cancer cells. By performing Cu K-edge XAFS in cells, this work highlights the biological relevance of Cu(I)–S clusters, previously characterized only in vitro.³

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Deciphering the origin of Zn within the Orne watershed – insights from XAS data

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ABSTRACT

The Orne watershed in northeastern France was the place for intensive industrial activities that left behind steel making wastes, overlaying the river banks and enriched in different metals. Zn content ranges from 1200 to 80,000 mg/kg and is mainly associated to polymetallic sulfides [1]. Within the Orne River basin, supplementary Zn sources were highlighted, related to the geology fingerprint of the basin (geogenic) or linked to current human activities (car production industry, road dust, domestic wastes) [2]. During twelve years, river suspended particulate matter (SPM) was regularly collected in the Orne River in variable flow regime conditions and at different specific stations. The Beth station, located in the downstream part of the basin, was selected for its integrative character and was regularly monitored and in particular during winter flood events. SPM samples collected at Beth station display variable Zn content suggesting the contributions of different sources. However, Zn content and Zn isotopic fingerprint were not sufficient to decipher the sources of Zn transported by the Orne River [2].

XAS data at Zn K-edge were collected on SAMBA beamline, using the continuous mode, at low temperature (25K), with fluorescence detection and synchronous acquisition of reference spectrum for energy calibration. SPM samples were freeze-dried and protected from oxidation.

XAS data at Zn-edge (XANES and derivative) allowed to decipher and estimate Zn carriers in the different river SPM samples, and demonstrated the variable origin of Zn as a function of flow regime or season. Geogenic Zn, found in soils and river suspended matter (SPM) from the upstream part of the basin, is 100 % associated to clay minerals, inserted in the crystal network of lithogenic phyllosilicates. XANES signal display specific features with relatively intense absorption edge [3]. Zn in SPM was also shown to be present as Zn-sulfides, easily highlighted and quantified from XANES spectra and its first derivative). Quantification was reached using linear combination of reference spectra on XANES and first derivative data.

The combination of XAS data, Zn content and Zn isotopic fingerprint allowed the estimation of contributions of the different sources of Zn during several flood events. The presence of Zn sulfides could be easily demonstrated in Beth SPM samples, explaining the relatively higher Zn content, even when SPM are dominated by geogenic particles coming from the run-off of cultivated soils. This work highlights the input of XAS data for the study of Zn behavior in human-altered hydro-systems.

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Agricultural soil contamination by antimony: source, speciation, mobility

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ABSTRACT

The Allier department, located on the eastern edge of the Massif Central (France), is one of many regions with a rich mining history. Among the ore deposits, one was exploited until 1914 for antimony (Sb), and another until 1962 for tungsten (W), tin (Sn), and associated metals and metalloids (As, Zn, Sb). This mining activity generated over 335,000 m³ of residual materials, tailings, or waste from ore processing phases, whose chemical and mineralogical stability evolves depending on changes in the physicochemical conditions of the environment.

Part of this waste was used to backfill extraction galleries. At the same time, the surplus was stored on the surface without any specific protection, making it vulnerable to biotic and abiotic weathering processes. Under these conditions, potentially toxic elements (PTE) present in these residues can change speciation during the weathering of their bearing phases. Therefore, they may be dispersed with varying efficiency and at various distances from the source areas, either in dissolved or particulate forms.

In the case of the more recent residues from the sites of interest, exposure to supergene conditions for at least sixty years has led to the weathering of the tailings and the release of PTE, either through dissolution or particulate transfer. Therefore, the new distribution of Sb should be determined to understand its stability and toxicity for the local environment as in the surrounding agricultural soils.

To this end, samples from several soil profiles developed on or near mining residues were characterized using various physico-chemical methods (elemental composition, particle size analysis, mineralogical analysis by XRD and SEM-EDS of solids, Sb speciation by XANES) in order to quantify and determine the primary and secondary bearing phases of the PTE. Surface waters draining the impacted areas were also collected and analyzed for their PTE content. Leaching experiments and selective extractions provide information on the PTE mobility under environmentally relevant pH conditions and solid:solution ratios. Better understanding PTE dynamics helps anticipate and manage the environmental risks associated with PTEs in post-mining management.

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Mineralogical diversity of Bi and W evolution in human-impacted environments over time

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ABSTRACT

Currently, achieving mineral resource self-sufficiency is a critical strategic priority for nations seeking to secure supply chains and reduce vulnerability to dominant economic powers. While the historical mining industry left a legacy of millions of tons of tailings and extensive environmental contamination, these sites now present an opportunity. France is currently considering the resumption of mining operations, which necessitates the transformation of mining waste from an environmental liability into a valuable secondary resource. In this context, the use of X-ray spectroscopy is essential to unravel the geochemical cycling of Bismuth (Bi) and Tungsten (W) in anthropogenically impacted environments.

Moving beyond total concentration analysis, REXEL project¹ focuses on solid-phase speciation and temporal evolution within mining materials. The research is structured around two core objectives: first, mapping the alteration pathways from primary sulfides to secondary Fe-oxides; and second, identifying the crystallographic properties of neo-formed mineral phases. These phases - including iron oxides¹, phosphates^{2,3}, sulfates⁴- act as efficient sinks for Bi and W. Understanding these retention mechanisms is key to developing natural attenuation strategies and optimizing the recovery of these metals for industrial valorization.

The project employs a high-resolution analytical framework, combining laboratory and synchrotron-based XRF and XAS to achieve sub-micrometer spatial resolution for chemical mapping and phase speciation. By determining the local coordination environments and oxidation states of Bi and W, we can assess their stability and recombination potential within alteration products. Furthermore, the synthesis of representative mineralogical phases allows for a comparative analysis between synthetic and natural samples. This dual approach is designed to isolate the most stable phases responsible for metal sequestration, thereby preventing environmental leaching while identifying pathways for the economic recovery of these critical elements.

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HERFD-XAS Reveals Trace Metal Incorporation Mechanisms In Environmental Iron Sulfides

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ABSTRACT

High-Energy Resolution Fluorescence Detected X-ray absorption spectroscopy (HERFD-XAS) has recently emerged as a significant tool for examining local atomic structure and redox state of trace elements in complex environmental matrices. It filters emitted fluorescence through a narrow energy bandwidth, producing high-resolution spectra free of parasitic emissions from other elements in the material [1,2]. A notable example is the challenge of acquiring conventional XAS data on cobalt traces in iron-rich samples due to overlapping Co K α lines and Fe K β emission lines [3], which a HERFD setup effectively addresses. High-resolution XANES spectra provide direct information on redox and coordination states with sensitivity unattainable by conventional fluorescence detection. Compared with model compounds and ab initio calculations, pre-edge HERFD spectra distinguish tetrahedral from octahedral coordination in complex materials. Modern XAS beamlines such as FAME-UHD now enable simultaneous EXAFS data collection using the same HERFD setup. Complemented by ab initio EXAFS calculations on DFT models, EXAFS spectroscopy provides valuable supplementary information on local structures that cannot always be resolved from XANES data. Here, we will present recent examples of HERFD spectroscopy applied to environmentally relevant samples, with important implications for our understanding of the fate of trace metals in modern and ancient sulfide-rich media. In particular, authigenic pyrite (FeS₂) has been intensively studied because it can record the chemical signature of its precipitating medium, making it a valuable geochemical archive for interpreting past environments [4]. It also acts as an efficient sink for toxic and/or strategic metals in subsurface environments [5,6]. HERFD-XAS data at the Co and Cu K-edge reveal new pyrite precursor and formation pathways, thereby impacting our understanding of pyrite formation, trace elements at low temperatures, biogeochemical cycling, metal-sulfide evolution in sediments, and geochemical archives [7].

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Original Autoclave Design For Spectroscopic Studies Of Hydrothermal Fluids

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ABSTRACT

In support of D. Testemale's talk about *in situ* XAS studies of metal transport in hydrothermal fluids [1,2], this poster aims at presenting in more details the various technical elements of the design of our autoclave [3], and the way they are implemented to overcome the technical challenges of *in situ* spectroscopic measurements of hydrothermal fluids. Indeed, in such experiments: fluids need to be maintained at high pressure and temperature over long periods of time; the fluids can be chemically reactive (solvents, pH, etc.); their densities cover a wide range [0.1–1] g.cm⁻³ spanning from liquid-like to vapor-like fluids; the concentration of the metals investigated might be very low.

The different design and usage specificities are:

- internal cells made of glass-like carbon which makes low x-rays energy measurements possible.
- Original high-pressure windows made of beryllium or glass-like carbon [4].
- Sample volume: the importance of this volume and its ratio to the beam size will be explained.
- Pressure and temperature control and stability obtained through the development of original high-pressure valves [5].
- A version of the autoclave with HP windows and sample holder made of monocrystalline sapphire is designed for optical measurements such as Raman.

The integration of these autoclaves on FAME beamlines at ESRF will be demonstrated with a few examples. Also, even if this poster aims at being mainly technical, the methodology of XAS measurements and analysis to derive metal solubility and speciation will be recalled to picture a global view of our methods and put those technical developments into context.

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The Renaissance of Laboratory X-ray Absorption Spectroscopy: New Opportunities for Routine Characterization of Nuclear Materials

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ABSTRACT

Over the last several decades, the elemental selectivity and applicability to crystalline, amorphous, liquid, and heterogeneous materials have established X-ray absorption spectroscopy (XAS) as one of the most powerful methods for probing the local atomic and electronic structure of matter. In the nuclear field, XAS has provided unique insights into fuel fabrication, irradiation effects, actinide speciation, and waste-form evolution [1-5]. Historically, however, most XAS experiments have relied on synchrotron radiation facilities, whose exceptional performance has enabled major scientific advances.

The growing demand for synchrotron beamtime and the increasing oversubscription of many beamlines have recently renewed interest in laboratory-based XAS instrumentation. Significant progress in X-ray sources, optics, crystal analyzers, and detector technologies has led to a new generation of laboratory spectrometers capable of delivering high-quality XANES and, increasingly, EXAFS measurements for a broad range of applications [6-9]. These developments are progressively transforming XAS into a more accessible technique that can support routine characterization, method development, long-duration experiments, and rapid experimental feedback directly within research laboratories.

This renewed availability of laboratory XAS opens particularly attractive perspectives for the investigation of challenging materials. Radioactive and actinide-bearing samples remain difficult to study at synchrotron facilities because of transportation constraints, radiological regulations, and the limited number of beamlines authorized to handle significant quantities of transuranic elements. By enabling measurements directly within dedicated nuclear laboratories, modern laboratory XAS instruments can simplify experimental workflows while reducing the risk of sample alteration associated with preparation and transport [8, 10-11].

In this contribution, we present a historical perspective on the evolution of laboratory XAS, from its early developments to the technological advances driving its current resurgence. Emphasis will be placed on the opportunities offered by modern laboratory spectrometers as complementary tools to synchrotron facilities, with examples drawn from the characterization of actinides and nuclear materials. The emergence of readily accessible laboratory XAS is reshaping the practice of absorption spectroscopy and creating new opportunities for both routine and specialized investigations.

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XAS Studies On Nuclear Fuels: From Simulated to Spent Fuels Characterizations

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ABSTRACT

Fission of U and Pu during fuel irradiation in nuclear reactors leads to formation of more than 60 different chemical elements, in small or even very small quantities (< or << at.%). Determining the chemical and microstructural roles of the fission products (FPs) in the evolution of nuclear fuel physical properties is of crucial importance to develop reliable longterm predictions of the fuel behavior during reactor operation under normal and accidental conditions as well as for storage and recycling of spent fuel.

X-ray absorption spectroscopy (XAS) is the element-specific technique of choice to determine the speciation of an element, even very diluted, in potentially complex samples. However, the accurate determination of nuclear fuel chemistry is a very challenging task. First, fission products in UO₂ create a complex multi-component system, in which a given element can form various phases which may be different depending on the location within the pellet. Secondly, spent nuclear fuel is highly radioactive and requires specific radiological safety devices and protocols to be handled and studied. In some cases, where specific fission products are to be characterized under particular conditions, irradiated fuels can be replaced by model, low activity materials, such as SIMFUEL (UO₂ doped with stable isotopes of fission products) or ion-implanted UO₂ pellets.

Numerous XAS studies have been carried out at SOLEIL and ESRF on such model samples to characterize the chemical states and/or behaviors of various FPs (Mo, Xe, Kr, Cs, I, Ba, Zr, etc) in UO₂ as a function of concentrations, temperature and oxygen potential. These “separate effects” experiments aimed at, e.g., feeding thermodynamical databases with experimental data to improve modelling [1], or improving the understanding of the behavior of FPs during a severe accident [2]. They also help developing innovative advanced fuels.

Recently a massive sample of UO₂ spent nuclear fuel could be characterized for the first time by HERFD-XANES on the MARS beamline to determine the chemical states of Mo and Cs [3]. This first experiment opens up many prospects for future XAS characterizations of spent fuels.

An overview of all these XAS studies on model or spent fuels will be given. The technical and scientific challenges, as well as the required technical and methodological developments will be exposed.

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Probing Nb Environment in Non-Crystalline Structure Up to 5 Å Resolution using XAFS

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ABSTRACT

Non-crystalline materials are challenging to characterize because their broad distribution of local atomic environments produces overlapping spectroscopic contributions and significant signal broadening, complicating interpretation. Bioactive glasses have attracted considerable attention since their development by Professor Larry Hench in the early 1970s. Their metastable, non-crystalline structure enables bonding with hard and soft tissues, while their composition can be tailored with therapeutic ions such as niobium, which has been associated with enhanced alkaline phosphatase expression and osteoblast activity. This study investigated changes in the local chemical environment and network structure of a bioactive glass following Nb₂O₅ incorporation [1]. The composition was 46.1 mol% SiO₂, 24.4 mol% Na₂O, 26.9 mol% CaO, (2.6 – X) mol% P₂O₅, and X mol% Nb₂O₅, where X = 1.3 or 2.6 mol%, producing two niobium-containing glasses. Nb K-edge XAFS was used to investigate the local electronic and atomic environments of niobium, with structural information of 5 Å resolution. XANES indicated that niobium predominantly occupies a sixfold-coordinated environment. EXAFS revealed a highly disordered first coordination shell, evidenced by a large Debye-Waller factor and consistent with a distorted NbO₆ octahedral environment. Increasing Nb₂O₅ content increased the average Nb-O distance. No significant Nb-Nb backscattering contribution was resolved, suggesting that niobium is mainly dispersed as isolated or weakly interconnected NbO₆ units rather than forming an extensive Nb-O-Nb network [2]. This structural isolation may favor bioactivity because stable Nb-O-Nb linkages could reduce glass dissolution and restrict niobium release. Higher coordination shells, associated with neighboring Si, P, Ca, and Na atoms, also exhibited increased fitted distances and Debye-Waller factors after niobium incorporation, indicating expansion and greater disorder in the glass structure. The combined use of XANES and EXAFS provides element-selective information on oxidation state, coordination geometry, interatomic distances, and disorder. This is particularly valuable due to the ⁹³Nb NMR analysis interpretation is complicated by the strong quadrupolar interaction of the ⁹³Nb nucleus. Nb K-edge XAS therefore helps resolve local structural features that may be difficult to determine by NMR alone, which is one of powerful technics to probe chemical environment. Overall, elucidating the local and medium-range environments of niobium is essential for understanding the relationship between glass structure, dissolution behavior, and biological performance. These structural findings supported the in-vitro enhanced the osteogenic and osteoestimulative properties, while the in-vivo results demonstrate a significant effect on bone formation bone formation [3].

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In Situ Spatial And Temporal Distribution Of Rare Earth Ions During Liquid-Liquid Extraction With Ionic Liquids

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ABSTRACT

Liquid-liquid extraction (LLE) plays a crucial role in the hydrometallurgical recycling of Rare Earth elements (REEs). Current industrial processes use large amounts of toxic volatile organic solvents and extractants. Ionic liquids (ILs) offer a promising alternative to organic solvents due to their lower toxicity, flammability and volatility than conventional solvents. Interestingly, it was shown that the use of ILs can remarkably enhance the extraction efficiency of REE ions from leaching solutions.^{1,2} However, ILs present high viscosities which slow down the mass transfer during extraction. To overcome this problem, Homogeneous liquid-liquid extraction (HLL), was developed using of thermomorphic ILs i.e. ionic liquids that exhibit temperature-dependent miscibility with water.^{3,4} By changing the temperature of the IL/water system, a reversible transition from a biphasic mixture to a homogeneous phase occurs, eliminating thus the interface and the diffusion barrier for extraction.⁴

This study is focused on a thorough understanding of the extraction mechanisms of critical metals, specifically lanthanides, La(III) and Ce(III), via HLL using a thermomorphic IL.: 2-hydroxyethyl-trimethylammonium bis-(trifluoromethanesulfonyl)imide [Choline][TFSI] coupled with a zwitterionic extractant (Betainium). The ternary phase diagram of the [Chol][TFSI]/betainium/water system, established via NMR spectroscopy, provided novel information on the reduced extraction efficiency observed for high betaine concentrations. To understand the mechanism of extraction, modelling was performed based on the system's ternary phase diagram and the metal extraction results. In addition, X-ray Absorption Spectroscopy (XAS) experiments were carried out to follow the speciation and concentration of La and Ce during HLL in the system [Chol][TFSI]/betaine/water. Furthermore, this work demonstrated, for the first time, the transient accumulation of REEs at the liquid-liquid interface prior to the high-temperature step of the process.⁵

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Probing Excited-State Auophilic Interactions in a Dinuclear Au(I) Complex by Time-Resolved X-ray Absorption Spectroscopy and Solution Scattering

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ABSTRACT

Bimetallic gold complexes have drawn attention in recent years by distinguishing themselves through their specific photophysical and photochemical properties. Their applications vary from areas such as homogeneous catalysis¹, materials science and energy-efficient optoelectronics to fluorescent markers, bioimaging and chemical sensing².

Despite the extensive studies of the ground state of gold dimers, the structures of the singlet (S1) and triplet (T1) excited states have not yet been sufficiently understood³.

Here, we investigate the photoinduced dynamics of the dinuclear gold(I) complex [Au₂(dcpm)₂](PF₆)₂ in acetonitrile using time-resolved X-ray absorption spectroscopy and X-ray solution scattering. Femtosecond measurements at the Au L₃-edge, including Total Fluorescence Yield X-ray Absorption Spectroscopy and High-Energy Resolution Fluorescence Detection at the European XFEL FXE instrument, were deployed to observe transient changes in the electronic and local coordination environment of the Au centers.

The local structural information obtained by X-ray absorption spectroscopy is complemented by time-resolved X-ray solution scattering (TR-XSS) measurements performed at the ESRF ID09 beamline. The results have been analyzed using structural models based on ORCA geometry optimizations and Debye scattering calculations. Candidate excited-state geometries with varied Au-Au distances are compared with the experimental scattering response to evaluate the sensitivity of the transient signal to changes in aurophilic interactions. Moreover, the photoinduced solvent contributions are independently characterized using a dye reference in acetonitrile.

These techniques provide valuable insight into the evolution of aurophilic interaction after photoexcitation and insights into the transient electronic and structural changes.

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Investigating Fe(III) Photoreduction in Potassium Ferrioxalate Using X-ray Absorption and X-ray Emission Spectroscopy

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ABSTRACT

Potassium tris(oxalato)ferrate(III), $K_3[Fe(C_2O_4)_3] \cdot 3H_2O$, undergoes $Fe(III) \rightarrow Fe(II)$ reduction upon light irradiation. Here, we investigate this photoreduction in the solid state using complementary Fe K-edge X-ray absorption spectroscopy (XAS) and Fe K β X-ray emission spectroscopy (XES).

Measurements were performed using a laboratory-based high-energy-resolution von Hámos spectrometer with a Si(531) analyzer. The sample was continuously irradiated with a 405 nm CW laser, while XAS and XES spectra were collected at different irradiation times.

The Fe K-edge XAS spectra show a progressive transformation consistent with $Fe(III) \rightarrow Fe(II)$ reduction. First-order analysis gives characteristic time constants of $\tau = 16.9$ min for the edge-energy shift and $\tau = 6.8$ min for the normalized white-line area, revealing different timescales for the observed electronic and structural responses.

The Fe K β XES spectra independently confirm the photoreduction. The spectral centroid shifts by approximately -1.25 eV between 0 and 100 min, with most of the change occurring within the first 10 min. The Integrated Absolute Difference (IAD) increases from 0.277 at 10 min to 0.303 at 100 min, indicating that the Fe electronic structure rapidly approaches a stable photoreduced state. Changes in the valence-to-core region further reveal modifications of the Fe–O electronic environment.

The combined XAS and XES measurements demonstrate a persistent $Fe(III) \rightarrow Fe(II)$ transformation and reveal a rapid initial electronic reorganization followed by stabilization. These results provide complementary insight into the evolution of the Fe electronic and local structural environment during ferrioxalate photoreduction.

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1s2p Resonant Inelastic X-ray Scattering Natural Circular Dichroism

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ABSTRACT

We present measurements using a recently developed, bulk-sensitive, element-specific, photon-in photon-out, hard x-ray spectroscopic technique: Resonant Inelastic X-ray Scattering Natural Circular Dichroism (RIXS-NCD). This technique offers a new tool for in-depth investigations of chiral systems via electric dipole-quadrupole transitions originating from the asymmetric local environment. Unlike conventional RIXS, which is largely mediated by purely electric dipole transitions into hybridised orbitals, selection rules dictate that in RIXS-NCD the quadrupole term will, for core-hole spectroscopy of transition metals, also directly probe the valence d-states providing more information on local symmetries, charge distributions and electron structure. 1s2p RIXS-NCD spectra of cobalt- and manganese-based chiral compounds exhibit clear circular dichroism arising from the metal centre chirality and resulting valence orbital mixing. We further demonstrate a dependence on the incident photon wavevector orientation relative to the crystal structure, and the scattering angle of the emitted photons. Finally, constant emission energy cuts of the RIXS-NCD spectra are compared with density functional theory calculations demonstrating strong agreement.

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Role of the electron Inelastic Mean Free Path in x-ray photoemission and x-ray absorption spectroscopy of solid copper

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ABSTRACT

The Inelastic Mean Free Path (IMFP) of electrons at low and intermediate kinetic energies plays a key role in determining the probing depth of various spectroscopic techniques based on the collection of photoemitted electrons (XPS, AEY, TEY ...) [1-2] and enters also directly in the interpretation of the x-ray absorption oscillations commonly referred to as X-ray Absorption Spectroscopy (XAS) [3]. While modern IMFP determinations and calculations are usually very accurate, there are discrepancies between the IMFP curves reproducing XAS data and those obtained by other techniques [4]. In this work we present a novel determination of the IMFP curve in solid copper obtained using the overlayer method and core photoemission spectroscopy using synchrotron radiation. In this context, new experimental procedures have been developed to improve the reliability of IMFP determinations from the overlayer method, discussing the role of elastic scattering [2]. Moreover, the IMFP curve is also determined by comparing ab-initio multiple-scattering simulations and experimental Cu K-edge data showing a very satisfactory agreement with the curve probed by photoemission, extended to a larger energy range. Detailed prescriptions on how the inclusion of the IMFP determination is realized in multiple-scattering simulations are reported. An experimental XANES analysis of the IMFP using the finite-difference-method proposed by Bourke and Chantler [4] has been also done, and found in reasonable agreement with the two above mentioned investigations. The resulting IMFP curve appears to be a solid result obtained combining three independent techniques which provides an accurate determination of IMFP in solid copper overcoming some previously observed discrepancies. The developed approach, validated in the case of crystalline copper, can be extended to the determination of the electron IMFP in unknown and technologically relevant materials [2].

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Simultaneous XAFS and XRD using a two-color beam

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ABSTRACT

X-ray diffraction (XRD) and X-ray absorption fine structure (XAFS) spectroscopy are widely used in materials science to determine crystal structure and chemical state, respectively, and both techniques are compatible with *in situ* sample environments. Numerous authors have highlighted the advantages of recording XAFS and XRD within a single experimental session at a synchrotron radiation beamline, as the combined data sets provide substantially deeper insight into the probed sample volume than either technique alone. Such combined experiments are now performed at many beamlines around the world.

However, XAFS measurements must be carried out at the absorption edge of the analyte atom, an energy that is often lower than desired for XRD, where shorter wavelengths are often used to avoid strong absorption and to access a larger reciprocal space range. Combined XRD/XAFS studies therefore typically require changing the incident energy over a wide range, which is problematic when the sample evolves on time scales faster than tens of seconds. Moreover, changing the monochromator angle can induce beam motion, causing the two techniques to probe different sample volumes.

Here we propose exploiting the first and third harmonics of a Si(111) double-crystal monochromator, combined with the 35 mm-period undulators at ESRF ID26, to enable truly simultaneous XRD/XAFS measurements. The main challenges of this approach are harmonic contamination in the spectroscopic data and the low photon flux available in the third harmonic for recording the XRD pattern. We will present and discuss our first feasibility studies on ID26 where the powder diffraction patterns were recorded using 4 Mythen detectors.

LUCIA: the tender X-ray beamline of SOLEIL

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ABSTRACT

The LUCIA beamline, dedicated to X-ray fluorescence (XRF) and X-ray absorption spectroscopy (XAS) experiments has been designed to cover the energy range from 0.8 to 8.5 keV. Within this energy range, the beamline is optimized to characterize the speciation of elements from Na to Co at the K-edge, from Ni to Gd at the L-edges, and of rare earths and actinides at the M-edges.

Using a pair of Kirkpatrick-Baez (KB) mirrors, the photon beam can be focused down to $2.5 \times 2.5 \mu\text{m}$, enabling the elemental mapping in a heterogeneous sample. LUCIA's strength lies then in its ability to couple XRF imaging with X-ray absorption near edge spectroscopy (XANES) and extended X-ray absorption fine structure (EXAFS) analysis at the micrometric scale. This allows for the determination of the speciation variability of a given atom as a function of its elemental environment. As XRF maps can be collected in fly-scan mode, hyperspectral data can be easily obtained, stacking the images using Jupyter notebooks developed at SOLEIL. This allows to collect a XAS spectrum in each pixel of the elemental map. Thanks to liquid He and N₂ cryostats, such analyses can be performed in cryo-conditions, on hydrated samples if needed. In addition, LUCIA can accommodate dedicated microfluidic chips to enable *in situ* observations of reactions, the quality of these measurements being enhanced by XAS data collection in fly-scan mode.

An upgrade to LUCIA in 2027 will enable XRF-XAS coupled maps with an 800 x 800 nm beam in a dedicated chamber.

Current and future capabilities will be illustrated by examples in the poster.

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A platform for XAFS studies in electrocatalysis: the SAMBA beamline at SOLEIL synchrotron

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ABSTRACT

Pathways to a cleaner and carbon neutral energy cycles may employ electrochemical methods to produce and consume hydrogen or reduce oxidized carbon from CO₂ to more valuable C1 or C2 alcohols or acids ready to be reemployed. All these processes are complex; they are often energy hungry like the oxygen evolution reaction (OER) and require catalysts to reduce the activation energies and increase the selectivity towards the products like in the CO₂ reduction reaction (CO₂RR). XAFS has an important role in catalysts characterization, most of them are present in the systems as single atoms, finely dispersed nanoparticles or dopants in nanocrystalline oxides. Thus, the capability of XAFS of providing the local structure of a selected element and information on its oxidation state is precious; this is particularly true for operando studies, where the highly penetrating hard x-ray XAFS spectroscopy enables a relatively faster adaptation of sample environments from lab test benches to the synchrotron station.

There is a multitude of processes and conditions that are proposed for study, often requiring dedicated cells that must be adapted to the beamline for XAFS studies. The degree of adaptation of the cells may often affect the success of experiments from the electrochemical or spectroscopy point of view. However, during the last ten years, the demand for operando x-ray absorption studies in electrocatalysis has moved from confidential to almost three quarters of the SAMBA [1] activity. This has stimulated our group to offer an increasing set of tools for all users.

We will showcase some results illustrative of the beamline capabilities through the usage of liquid half cells adapted to the beamline from market suppliers (Zahner GmbH), but also SAMBA prototypes like the cells Optymal [2] and CAPACE as well as a complete fuel cell setup [3].

In particular, the Optymal cell is optimized for very thin liquid layers to study at high and low pH catalysts on non-x-ray-transparent substrates. It is suitable for studies on epitaxial grown thin films. The CAPACE system is a new development for studies on catalysts dispersed on gas diffusion layers, it enables a very large angle of view, and it is intended to approach more realistic and high current conditions than cells where CO₂ is provided via electrolyte saturation.

At the horizon of 2030 post upgrade restart of SOLEIL-II, the objective will be to offer a coherent and benchmarked set of electrochemical cells and to transparently integrate them into the data acquisition process.

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FAME-PIX: The French CRG Beamline Offering A Spectral Ptychography End-station at the ESRF

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ABSTRACT

After 20 years of operation, the FAME beamline is becoming FAME-PIX. This new beamline will still offer a XAFS end-station (which has already reopen after a 36-month closure for upgrade) and a fully new end-station dedicated to ptychography. This poster will focus on this new end-station, and in particular on the conception and construction aspects.

Ptychography is an imaging technique based on the use of x-ray coherence, which has been increased a lot during the ESRF upgrade (EBS). The spectral ptychography principles will be explained in a simplified scheme.

Then, we will introduce the technical challenges raised during the design of the different elements. For example, a positioning system offering a precision better than $\pm 2\text{nm}$ is needed, implying the use of interferometers as encoders.

Since such measurements will be time consuming, we have already investigated several possibilities to reduce the acquisition time while keeping the desired data quality. A deep-learning-assisted optimized reconstruction strategy [1] will be presented.

Spectral ptychography is particularly well suited for the study of heterogeneous materials containing small aggregates of absorbing atoms finely mixed in a complex matrix. The accessible energy range being 4 to 12 keV, the transition metals can be targeted easily. Applications in the fields of catalysis and Earth sciences will be shown.

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FAME-UHD & FAME-PIX: A French Hub Dedicated To X-Ray Absorption Spectroscopies At The ESRF

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ABSTRACT

The French hub FAME (French Absorption spectroscopy beamline in Material and Environmental science) at the ESRF (European Synchrotron Radiation Facility) consists of two instruments: FAME-PIX (Ptychography Imaging with X-rays) and FAME-UHD (Ultra-High Dilution). Those instruments are dedicated to x-ray absorption spectroscopy studies. They cover a wide range of fields: condensed matter physics, material science, biophysics, chemistry, and mainly Earth sciences. For the latter, FAME instruments are particularly well suited since the majority of the geosciences probed elements are highly diluted. Measurements can be carried out at low temperature (e.g. to limit radiation damages), at high temperature, at high pressure or under controlled atmosphere for in situ or operando studies [1].

Thanks to two governmental fundings obtained jointly with the other ESRF French CRG beamlines [2], the original FAME beamline has been entirely rebuilt to become FAME-PIX and the FAME-UHD detection equipment has been upgraded. In details:

- (a) On FAME-PIX, new state-of-the-art mirrors have been installed to take the best benefit of the quality of the new ESRF source.
- (b) A new solid-state 16-element germanium detector as well as a new digital electronics have been bought for FAME-PIX. The photons emitted by the sample can now be detected with a better resolution and a higher counting rate compared to what was achieved with the equipment.
- (c) A new imaging end-station is being built on FAME-PIX, aiming at taking advantage of the increased coherence of the EBS source to carry out x-ray ptychographic measurements. Once achieved, samples will be imaged with a spatial resolution of about 50nm.
- (d) New crystal analysers have been acquired to increase the detection capacities of FAME-UHD: detection angles can be better optimised for each probed element, thus optimising the collected signal (energy resolution about 1eV).
- (e) On both beamlines, departing from previous step-by-step monochromator movements, continuous measurements have been implemented in order to allow faster analysis.

The construction of FAME-PIX imposed the closure of the previous FAME beamline for 30 months. Now, both FAME-PIX and FAME-UHD beamlines are in operation, except the ptychography end-station, still under construction.

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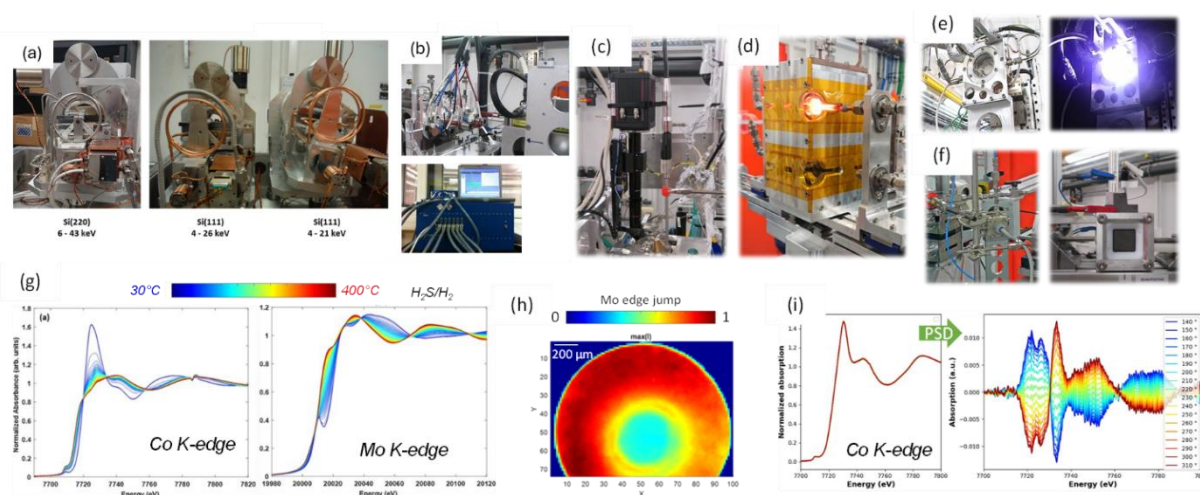
ROCK: a versatile beamline for *operando* quick-XAS monitoring at the second and micrometer resolution

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ABSTRACT

ROCK (Rocking optics for Chemical Kinetics) is the quick X-ray Absorption Spectroscopy beamline at SOLEIL¹ whose main applications are the in situ/operando XAS characterization of energy-related materials². ROCK is equipped with 3 cam-driven channel-cut monochromators working between 4 and 40 keV³ (Fig. 1a) with oscillation velocity ranged from 0.1 to 20 Hz for sub-second time resolution acquisition. A large portfolio of setups for monitoring the dynamic evolution of batteries (Fig. 1b) and catalysts (Fig. 1c to f) in working conditions is proposed to the users. The beamline is fully optimized to probe several elements on a single experiment by alternate XAS acquisition between several edges using the so-called edge jumping operation mode (Fig. 1g). Full Field (FF) XAS hyperspectral imaging (Fig. 1h) using a pixelated CMOS camera allows to reach a micrometer spatial resolution in addition to the second time resolution⁴. ROCK has gained a sound background in using chemometrics tools such as Multivariate Curve Regression with Alternating Least Square (MCR-ALS)⁵ fitting allowing to isolate from huge Quick-EXAFS dataset the species involved in the reaction and their concentration profiles. To overcome bulk sensitivity of XAS making difficult to differentiate the spectator species from the active species, modulation-excitation XAS (MEXAS) with phase sensitive detection (PSD) has been recently implemented to highlight the contribution of active species during a chemical process⁶ (Fig. 1i).



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DEIMOS: 15 years at the service of magnetism, from conception to SOLEIL II

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ABSTRACT

X-ray Magnetic Circular Dichroism (XMCD) has been a cornerstone of element-specific, atomic-scale magnetism for five decades^{1,2}. The DEIMOS beamline (Dichroism Experimental Installation for Magneto-Optical Spectroscopy), dedicated to soft-X-ray magnetic absorption measurements in the 350–2500 eV energy range, opened to users in March 2011 at SOLEIL synchrotron.³ On the occasion of the XAFS jubilee in France, we propose a journey through fifteen years of instrumental evolution that have shaped DEIMOS, in permanent dialogue with the scientific challenges of its time. From 1000 K to 220 mK, and from *in situ* transport to liquid environments, the experimental phase space accessible to soft-X-rays MCD has progressively expanded. Around its two experimental stations, CroMag equipped with a superconducting magnet (7 T axial, 2 T transverse, 2 K to 370 K) and MagneTwo fitted with a fast-field electromagnet (2 T, up to 1 Hz), DEIMOS has developed a unique set of specialized sample environments in response to emerging scientific topics. The MilleK insert extends the temperature range up to 1000 K, enabling XMCD studies of high-Curie-temperature magnetic materials, such as ferromagnetic van der Waals layers of low dimensionality above room temperature. The V²TI (Versatile Variable Temperature Insert) combines variable temperature control with multiple electrical connections, enabling *in situ* transport experiments coupled to XMCD, opening the way for functional device research.⁴ The microfluidic cell extends soft-X-ray MCD to liquid environments, including ferrofluids. Finally, the Dichro50 project integrated a ³He-⁴He dilution refrigerator into the CroMag cryomagnet, pushing the temperature limit to 220 mK under simultaneous X-ray irradiation and magnetic field, reaching a record-low temperature for XMCD measurements in the soft X-ray domain.⁵ Looking ahead to SOLEIL II opens a new chapter for DEIMOS. While scattering or imaging techniques will naturally benefit from the improved source emittance and the higher flux density of a fourth-generation ring, the impact on X-ray absorption spectroscopy is less transformative. For DEIMOS, the source upgrade therefore represents not simply an increase in performance, but an opportunity to rethink the beamline from within. The goal is to push the principles that have defined DEIMOS from the outset: high stability, the detection of extremely low signal levels and increasingly demanding sample environments.

Acknowledgements: *The authors warmly thank all the scientists, and experts collaborators whose contributions over the years to the design and development of the DEIMOS beamline were decisive.*

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MARS: the French Beamline Dedicated to Radioactive Samples

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ABSTRACT

The MARS beamline at SOLEIL provides a unique environment for the investigation of radioactive samples [1]. Born from a strategic partnership with the CEA, the beamline was designed in the mid-2000s to combine X-ray absorption spectroscopy (XAS) and X-ray diffraction (XRD) for the study of highly radioactive materials. Since its opening in 2010, MARS has undergone continuous technological and radiological expansion.

A significant milestone was reached in 2019 with successful experiments on spent nuclear fuel, leading to an increased radioactivity limit of 18.5 GBq per sample (and 185 GBq for storage). This allows for sophisticated experiments under diverse conditions, including extreme temperatures, high pressures, and in situ chemical environments.

The beamline currently offers a vast analytical toolkit across the hard X-ray domain (3–35 keV). This range enables synergistic investigations, such as combining actinide 5f-state analysis via RIXS at the M-edge with structural determination via EXAFS at the L-edge [2-4]. Additionally, the high-energy regime facilitates advanced X-ray diffraction and scattering (XRD, SAXS/WAXS) [5-6]. Recent advancements have further extended these capabilities into the realm of high-resolution imaging, utilizing microbeam [7] and full-field modes (e.g., FF-CT and multi-modal mapping combining μ XRF, μ XRD, and absorption contrast) to achieve 2D and 3D characterization. This presentation will outline the beamline's multifaceted capabilities and demonstrate them through recent high-impact X-ray spectroscopy experiments.

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Getting started with a laboratory spectrometer.

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ABSTRACT

Through this poster, we aim to present our objectives in acquiring a laboratory spectrometer, including preparation for synchrotron sessions, training new users in X-ray absorption, studying the aging of samples over time using the XANES approach, etc.

We will then provide some details about the EasyXAFS300+ instrument: its configuration (transmission or fluorescence measurements, the energy range, the set of currently available crystals and related accessible absorption edges etc.), before presenting preliminary results for a few samples: various reference materials for the K-edge of copper (Cu) or iron (Fe), as well as for element at the extremes of the energy range, such as those for titanium (Ti) and ruthenium (Ru).

Finally, it will be interesting to discuss the various sample environments: a liquid cell, a cryostat, or possibly electrochemical cells.

Tutorials

SOLEIL Auditorium - Main Building

Fastosh & FDMNES Tutorials

- | | |
|------------------------------|---|
| 09:30 – 12:00
T-01 | Fastosh Tutorial - Simulation of EXAFS: a live introduction to FITNESS, a new software built-in to Fastosh
Gautier Landrot – Synchrotron SOLEIL, France |
| 12:00 – 13:30 | Lunch |
| 13:30 – 16:30
T-02 | FDMNES Tutorial - Simulation Of XANES Using The FDMNES Code
Yves Joly – Institut Néel, CNRS, UGA |



Simulation of EXAFS: a live introduction to FITNESS, a new software built-in to Fastosh

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ABSTRACT

This tutorial will be mostly dedicated to showcase FITNESS, a new software built-in to Fastosh and dedicated to shell-by-shell fitting of the EXAFS. Since the focus of the tutorial will be on this program rather than on the overall EXAFS fitting methodology, the attendees ideally should have some prior knowledge and experience of the EXAFS shell-by-shell fitting procedure using other FEFF-based EXAFS fitting software. The session will be divided in two parts:

- In the first part, the instructor will provide a “walk-through” live demonstration of the main features of FITNESS. An experimental EXAFS spectrum will be simulated in front of the audience, from start to finish, to showcase:
 - 1) all the specific unique functionalities offered by the program at every step of the fitting procedure (i.e. data importation; preparation of the FEFF input file; management of FEFF results; creation of the theoretical chi ; refinement of the theoretical via least-square fitting minimization.
 - 2) the ability of the program to rapidly fit an EXAFS spectrum on a large R range in Fourier Transform space
- In the second part, an experimental spectrum will be simulated by the attendees. This “hands-on” experience should be help get personally familiar with the program functionalities.

MATERIAL

For the first part, since it will be fully provided by the instructor, no personal computer is required.

For the second part, the attendees should have the latest version of Fastosh installed on their personal computers. **Note:** An oral presentation introducing the program will be given at the conference XAFS50.fr on October 7th, which will represent the official birth of the software. Therefore, the installer (Windows, Mac, Linux) of the latest version of Fastosh, which will feature FITNESS, and data employed in this tutorial, **will be available for free download on SAMBA beamline's website** (<https://www.synchrotron-soleil.fr/en/beamlines/samba>) **starting from October 7th.**

Simulation Of XANES Using The FDMNES Code

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ABSTRACT

The goal of this tutorial is to understand the basic principles of X-ray absorption; this will be achieved through *ab initio* simulations using the FDMNES code. Participants will simultaneously learn to use this code, which is designed to be user-friendly.

After a short introduction, we will begin with a very simple case: a metal atom in an octahedral environment. We will examine the dipole and quadrupole contributions and test for anisotropy. We will then introduce distortions to observe their quantitative impact on the signal and on dichroism. Next, we will move on to crystalline materials, such as magnetite. We will look at the influence of the calculation radius on the results and the effect of the two iron sites. Subsequently, we will examine magnetic cases by performing XMCD calculations.

Given the limited duration of the tutorial, it will be difficult to carry out full calculations achieving optimal agreement between experiment and theory, but the methodology for doing so will be explained.

Depending on participant requests, we will look at how to calculate the absorption signal for dopants or cases where multiple chemical elements occupy the same site. We may also explore calculations for X-ray Raman scattering, X-ray emission, or resonant diffraction. Finally, if participants have specific examples, these can be addressed—at least to provide the right direction for analysis.

MATERIAL

The participants must have their own laptop (Windows, Mac or Linux) with a software to plot spectra (Origin, Kaleidagraph, or any other simple to use... (not Excel!). It is also possible to plot using Larix.

A special **package containing the FDMNES executable for the different operating system will have to be first downloaded using the following link:** <https://hub.neel.cnrs.fr/index.php/s/rGqmx28AnfeqNdt>. It is not the standard package but a more complete one containing special documentation and data files necessary for the tutorial.

Note that the Mac users could test first if the executable is working. If not, instructions are given in the “doc” directory of the package.