
# I Latin American Powder Diffraction Conference - LAPDiC

## BOOK OF ABSTRACTS

**October 13 to 14, 2025**

**Fortaleza, Brazil**



# PREFACE

It is with great pleasure that we present this **Book of Abstracts**, compiling the scientific contributions shared during the **I Latin American Powder Diffraction Conference (LAPDiC 2025)**, held in Fortaleza, Brazil, from **October 13$^{th}$ to 14$^{th}$**, 2025, at the Universidade Federal do Ceará. This event brought together the vibrant **Powder Diffraction** community of Latin America and colleagues from around the globe, marking a significant meeting for the advancement of the field in Brazil's Northeast region.

This collection represents the diverse and cutting-edge research presented across the broad spectrum of **Powder Diffraction science and its applications**. Within these pages, you will find contributions spanning from structure refinement using X-ray and neutron powder diffraction to materials analysis, software development, and new methodologies for phase studies, residual stress, and many other fascinating areas.

The abstracts gathered here showcase the scientific vitality demonstrated at **LAPDiC 2025** and underscore the crucial role that **Powder Diffraction** plays in addressing contemporary scientific and industrial challenges. We are confident that the research shared during the event sparked stimulating discussions, fostered new collaborations, and inspired further advancements in the field.

We extend our sincere gratitude to all the **authors** for their excellent contributions, to our **invited speakers** for sharing their expertise, to the **scientific and organizing committee** for its tireless dedication, and to our **sponsors** for their invaluable support in making the meeting a success.

We hope this Book of Abstracts serves as a valuable record of the science presented at **LAPDiC 2025** and a useful resource for the scientific community.

Sincerely,

**The LAPDiC 2025 Organizing Committee**

**Organizing Committee – LAPDiC**

The successful realization of the **I Latin American Powder Diffraction Conference (LAPDiC 2025)** was made possible by the dedication and tireless hard work of the following committee members, to whom we extend our sincere gratitude:

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| | Monday, Oct. 13th | | Tuesday, Oct. 14th |
|---|---|---|---|
| | LAPDiC | | LAPDiC |
| 09:00 | Registration | | |
| 09:30 | Plenary I<br>**Stefan Kycia**<br>*Advancing X-ray pair distribution function and diffraction capabilities at the Canadian Light Source* | | Plenary III<br>**Yang Ren**<br>*Synchrotron and neutron diffraction study of order/disorder and heterostructures* |
| 10:30 | **Coffee break** | | **Coffee break** |
| | MS 1 - Synchrotron X-ray Powder Diffraction | | MS 4 - General Interest Powder Diffraction |
| 11:00 | Keynote I<br>**Cristiane Barbieri Rodella**<br>*The Brazilian synchrotron facility for powder X-ray diffraction experiments: PAINEIRA beamline* | | Keynote III<br>**Verônica de Carvalho Teixeira**<br>*Carnaúba beamline: Advancing X-ray nanoscopy at Sirius* |
| 11:45 | Invited<br>**Leopoldo Suescun**<br>*$SrMnO_x$ ($2.25 \leq x \leq 3$) revisited: Oxygen-vacancy, charge and spin ordering in a not-so-simple perovskite* | | Invited<br>**Diego Germán Lamas**<br>*Micro-XPD analysis of archaeological materials using a laboratory SAXS/WAXS instrument* |
| 12:15 | **Otavio Luis Canton**<br>*Pressure-induced structural transitions in tantalum- and antimony-based trirutile oxides* | | **Flávia Regina Estrada**<br>*XRDplayground: an interactive educational tool for teaching powder X-ray diffraction (PXRD)* |
| 12:30 | **Sofia Baldoni Baúti**<br>*In situ synchrotron XRD evaluation of high-entropy perovskites: exploring redox cyclability for catalytic applications* | | **Fabiano Rafael Praxedes**<br>*Copper Oxidation and the Formation Pathways of $Cs_2CuSbCl_6$ and $Cs_2CuSbBr_6$ Double Perovskites* |
| 12:45 | | | **Patrícia Osório Ferreira**<br>*Diffractometric, spectroscopic, and thermal analysis characterization of valsartan - 4-aminobenzoic acid salt-solvate* |
| 13:00 | **Lunch** | | **Lunch** |
| | MS 2 - Total Scattering Methods for Materials Characterization | MS 3 - Multi-phase Identification and Quantification | MS 2 - Structure Determination and Refinement Using Powder Diffraction |
| 14:00 | Keynote IV<br>**Fanny Nascimento Costa**<br>*Polyamorphism in Pharmaceuticals? An overview and open questions* | Keynote V<br>**Thomas Blanton**<br>*Advanced materials characterization using powder diffraction techniques and the Powder Diffraction File™* | Keynote II<br>**Viviane Peçanha Antonio**<br>*Unravelling crystal structures with neutron powder diffraction* |
| 14:45 | Invited<br>**Ariane Schmidt dos Santos**<br>*The JATOBÁ (Joint Analysis by TOtal and wide-angle X-ray scattering BeAmline) beamline for PDF analysis at SIRIUS/LNLS* | Invited<br>**Carlos Eduardo Maduro de Campos**<br>*Real-time and in-situ Powder diffraction of mechanochemical synthesis of thermoelectric nanocrystalline materials* | Invited<br>**Fabio Furlan Ferreira**<br>*Solving crystal structures of pharmaceutical compounds with X-ray powder diffraction* |
| 15:15 | **Guilherme Strapasson**<br>*Probing Oxygen Vacancy-Induced Transformations in Fe-doped $TiO_2$ Using X-ray Total Scattering* | **Rodolpho Mouta**<br>*Monitoring the formation pathway and cation ordering of a rocksalt battery material via in-situ X-ray diffraction* | **Valentina Ordoñez Ulloa**<br>*Structural determination by X-ray powder diffraction and supramolecular analysis of the opioid dihydrocodeine bitartrate* |
| 15:30 | **Vinícius Danilo Nonato Bezzon**<br>*PDFxDDB: a database for pair distribution function patterns of amorphous drugs* | **Bryan Moncada Villegas**<br>*Mineralogical study of ash from the Poás Volcano: Geological and risk implications* | **Graciela Diáz de Delgado**<br>*Structure determination of racemic lomefloxacin Iodide using laboratory X-ray powder diffraction data* |

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# Plenary Lectures

# *Advancing X-Ray Pair Distribution Function and Diffraction Capabilities at the Canadian Light Source*

***S. Kycia***

University of Guelph, Canada skycia@uoguelph.ca

A series of significant advances in our acquisition and analysis of total scattering measurements at the Brockhouse X-Ray Diffraction and Scattering Sector (BXDS) of the Canadian Light Source has enabled atomic pair distribution functions (PDF) of unprecedented quality and sensitivity, along with simultaneous high-resolution powder diffraction for Rietveld analysis. An overview of the new methods and capabilities will be presented. The PDF and Rietveld methods have been applied for the structure study of polycrystalline nanomaterials, amorphous systems, and molecules in solution. This serves as an excellent analytical tool for structure determination and phase identification for ex-situ, in-situ, and in-operando studies.

**Keywords: Canadian Light Source; pair distribution function; Rietveld method; nanomaterials**

# *Frontiers of local structure determination and prospects for AI to extend them*

***S. J. L. Billinge***

[1]Columbia University, New York, NY, USA sb2896@columbia.edu

Local structure is at the heart of materials science studies for materials for next-generation devices. In practice, this presents a number of key data analysis and interpretation challenges because it implies we are studying ever more complicated samples, often in complex heterogeneous environments and in time-resolved operando setups, and we are interrogating our data for more and more subtle effects such as microstructures and evolving defects and local structures. Of particular interest is the study of nanomaterials and materials structure on different length-scales. In this talk, I will describe the frontier for studying such systems using x-ray, neutron, and electron diffraction. Various developments that leverage the latest data acquisition and analysis techniques are powered by artificial intelligence (AI) and machine learning (ML). I will describe our attempts to push these boundaries forward by developing these emerging tools in the application to local structure studies.

**Keywords: Local structure; AI; Pair distribution function analysis.**

# *Synchrotron and neutron diffraction study of order/disorder and heterostructures*

***Y. Ren***

Department of Physics, City University of Hong Kong Hong
Kong JC STEM Lab of Energy and Materials Physics
yangren@cityu.edu.hk

Synchrotron X-rays and neutron scattering provide indispensable scientific resources for investigating the structure and dynamics of materials across multiple length and time scale in multidisciplinary research fields. While synchrotron X-rays offer high brilliance, fine spatial resolution, and element-specific sensitivity, neutrons provide unique capabilities in light-element detection, magnetic structure determination, and deep penetration into bulk samples. In addition, neutrons and X-rays interact with matter in different ways, thus are often used as complementary tools for studying materials at the electronic, atomic and molecular levels. In this talk, we will discuss the difference and similarity of neutrons and synchrotron x-rays, and their complementary use in materials science research with special focus on synchrotron and neutron diffraction studies of order/disorder and heterostructures in various materials.

# *Peptide and protein powders: Harnessing the power of laboratory and synchrotron-XRPD and advanced technologies*

***I. Margiolaki***

[1]Department of Biology, Section of Genetics, Cell Biology and Development, University of Patras, GR-26500 Patras, Greece imargiola@upatras.gr

Knowledge of 3D structures of biological molecules plays a major role in both understanding important processes of life and developing pharmaceuticals. Intense X-rays available at powerful synchrotron beamlines provide macromolecular crystallographers with an incomparable tool for investigating biological phenomena on an atomic scale. The resulting insights into the mechanism's underlying biological processes have played an essential role and shaped biomedical sciences during the last 30 years, considered the "golden age" of structural biology. In addition to several methods available for structure determination, macromolecular X-ray Powder diffraction (XRPD) [1-2] has transformed over the past decades from an impossible dream to a respectable method. XRPD can be employed in biosciences for various purposes such as observing phase transitions [3-4], characterizing bulk pharmaceuticals [5], accurate structure determination [6] and detecting ligands in protein–ligand complexes (7). This presentation aims to provide selected case studies demonstrating the power of the state-of-the-art infrastructures available at the ESRF which include:

- The ongoing investigation of the molecular and crystal polymorphism of microcrystalline protein drugs against diabetes aiming in the improvement of diabetes drugs arising from both the development of microcrystalline pharmaceuticals, as well as the combination of human insulin (HI) with organic ligands with well-known pharmaceutical action.
- Studies of the structure and dynamics of proteins of newly emerging, high-risk
RNA viruses for antiviral drug design, and the exploration of the crystallographic fragment-screening as a method for drug discovery against viral proteins. The aim is to provide important information on specific virus proteins involved in several structural RNA viruses-related projects and establish pandemic preparedness (linking to the EVA & VIZIER EU Projects).
- The development and combination of methodologies for improving currently existing peptide-based drugs. The aim is to provide significant information for the enhancement of a drug's absorption, distribution, metabolism and excretion (ADME) characteristics; a route which requires extensive peptide/ protein polymorph screening.

[1] Margiolaki I., "Macromolecular Powder Diffraction", Book Chapter for the International Tables of Crystallography- Volume H: Powder Diffraction, chapter 7.1, 718-736, (2019).
[2] Karavassili F. & Margiolaki I., Protein & Peptide Letters, 23, 3, 232 (2016); ESRF Industrial case studies.
[3] Spiliopoulou M. et al., J. Appl. Cryst. 54, 963-975 (2021).
[4] Fili S. et al., IUCrJ. 4, 2, 534 (2015); Triandafillidis D. P. et al., Acta Cryst. D76, 1065-1079 (2020).
[5] Karavassili F. et al., Biomolecules 7, 3, 63 (2017).
[6] Margiolaki I. et al., J. Am. Chem. Soc. 129, 11865 (2007); Triandafillidis D. P. et al., Acta Cryst. D. 79, 374-386 (2023) & Cover article; Spiliopoulou M. et al., Acta Cryst. A77, 186-195 (2021). [7] ESRF News March 2015; Karavassili F. et al., Acta Cryst. D76, 4, 375-384 (2020).

# Keynote Lecture

# *The Brazilian synchrotron facility for powder X-ray diffraction experiments: PAINEIRA beamline*

***C. B. Rodella[1], F. R. Estrada[1], D. S. Gonçalves[1], A. I. Ferreira[1], J. C. Mauricio[1], J. L. B. Neto[1] and A. C. Lana[1]***

[1]Brazilian Synchrotron Light Laboratory (LNLS), Brazilian Center for Research in Energy and Materials (CNPEM), Campinas, São Paulo 13083-970, Brazil
E-mail: cristiane.rodella@lnls.br and paineira@lnls.br

The PAINEIRA beamline is a facility dedicated to the X-ray diffraction characterization of polycrystalline materials in operation at the Brazilian synchrotron radiation (SR) facility – SIRIUS. Its optical layout optimizes the high photon brightness of the fourth-generation synchrotron light source generated by an undulator [1]. The benefits of using SR-X-ray Diffraction (SR-XRD) for characterizing polycrystalline materials at PAINEIRA extend beyond high resolution and signal-to-noise ratio. SR-XRD experiments can be combined with appropriate sample environments, fluid handling systems, and large angular resolution and fast detectors to facilitate *in situ* and *operando* experiments, which can reveal transient crystalline phases or structures responsible for the performance of functional materials [2]. The beamline was designed to be efficient and flexible regarding data acquisition, providing high angular resolution using a multi-analyzer crystal detector (MAC) and/or a fast acquisition area detector (arc detector, PIMEGAE 450D). Furthermore, PAINEIRA will operate in high-throughput mode, supported by an automated system that includes a sample magazine, robotic arms, sensors, and experimental management software for fully automated remote data acquisition [1]. Automated experiments under variable temperature conditions will also be available [2]. In this keynote presentation, I will provide an overview of the PAINEIRA beamline, primarily focusing on the mechanical, automated, and computational tools [3] that enhance efficiency and create a user-friendly environment for powder X-ray diffraction experiments. Subsequently, apparatus for *in situ* and *operando* experiments, especially developed for catalysis, solar cells, and batteries, will also be presented. Ultimately, it will be demonstrated how this specialized synchrotron tool can investigate fundamental material properties at the atomic level, correlating them with their corresponding functions.

[1] Estrada, F. R., et al. (2022) J. Phys.: Conf. Series 2380, 012033.
[2] Ferreira, A. I., et al. (2025) J. Phys.: Conf. Series 3010, 012147.
[3] Biondo Neto, J. L., et al. (2025) J. Appl. Cryst. 58, 1061-1067.

**Keywords: PAINEIRA beamline, SR-XRD, mail-in, high-resolution, *in-situ* experiments**

*Acknowledgements: FAPESP and CNPEM*

# Polyamorphism in Pharmaceuticals? An overview and open questions

**F. N. Costa[1,2], S. Roy[2], A. Brown[2]**

[1]Bragg Centre for Materials Research, University of Leeds, LS2 9JT, Leeds - UK, [2]School of Chemical and Process Engineering, University of Leeds, LS2 9JT, Leeds - UK
f.nascimentocosta@leeds.ac.uk

The existence of more than one crystalline form for the same pharmaceutical compound is known as polymorphism, and considerable scientific effort has been applied to understand it comprehensively. However, the analogous concept of polyAmorphism— where more than one distinct amorphous form can exist—remains underexplored and controversial, particularly in the pharmaceutical field. In contrast to crystalline polymorphs, amorphous materials lack long-range order, yet they can exhibit local order. This presentation explores the challenges in distinguishing true polyamorphous forms from kinetically trapped or pseudo-polyamorphous states, as well as the analytical difficulties in detecting such subtle differences. Case studies including amorphous paracetamol, hydrochlorothiazide and ball-milled valsartan suggest the possibility of polyamorphic transitions driven by processing conditions [1-3]. Furthermore, the study of a co-amorphous valsartan–nicotinamide system, produced by multiple preparation routes, suggests pathwaydependent recrystallisation behaviour. Techniques such as PXRD, PDF, FT-IR, DSC, and microscopy are employed to interrogate structural, bonding, and thermal responses. More than just presenting polyAmorphism, such studies lead us to raise critical questions about how amorphous states in pharmaceutical systems are defined, stabilised, and identified. Considering historical delays in recognising polymorphism's relevance, should we now invest in understanding the intriguing possibility of the existence of polyamorphism?

[1] Turek, M., (2021). Mol. Pharmaceutics, 18, 1970−1984.
[2] Martins, I.C.B., et. al. (2023). Chem. Sci., 14, 11447.
[3] Thi, Y.N, et. al., (2015). CrystEngComm, 17, 9029–9036.

**Keywords: pharmaceuticals; amorphous pharmaceuticals; co-amorphous systems; polyamorphism.**

*This work is supported by EPSRC grant EP/S022473/1. I acknowledge contributions from Dr Daniel Irving and Dr Dean Keeble from Diamond Light Source, and Prof Thomas Rades' group from University of Copenhagen.*

# *Advanced Materials Characterization Using Powder Diffraction Techniques and the Powder Diffraction File$^{TM}$*

***T. N. Blanton***

*International Centre for Diffraction Data, Newtown Square, PA, USA*
tblanton@icdd.com

Powder X-ray diffraction (XRD) has historically been the analytical technique of choice for phase identification of crystalline materials. Today advances in radiation sources, optics and detectors, allow scientists to use XRD to probe beyond phase identification and extend studies to investigate material microstructure as well as nanostructure properties. Whether the material of interest being studied is crystalline or amorphous, randomly or preferentially oriented, inorganic or organic, powder or solid, there are many diffraction methods available that can be used to analyze a sample and provide help in understanding how material processing affects material properties. In addition to improvements in diffraction instrumentation, new developments in the ICDD Powder Diffraction File (PDF®) databases have produced an array of solid-state analysis tools resulting from a combination of single crystal and powder diffraction data. Advanced features include: atomic coordinates for Rietveld refinement techniques; amorphous and nano material references; digital simulation tools for evaluating X-ray, synchrotron, electron and neutron diffraction data as well as crystallite size and analysis of two-dimensional diffraction data.

Diffraction methods and the Powder Diffraction File together create a synergy between data collection and data analysis that has been proven to assist scientists in finding a more complete and correct answer to their materials characterization questions. The origins of the PDF will lead to a review of current capabilities of databases and software, resulting in solutions for materials analyses.

# *Carnaúba Beamline: Advancing X-ray Nanoscopy at Sirius*

***V. C. Teixeira[1], L. M. Kofukuda[1], E. Todeschini[1], A. C. Piccino-Neto[1], A. P. S. Sotero[1], F. M. C. Silva[1], C. A. Pérez[1], I. T. Neckel[1], R. Szostak[1], H. C. N. Tolentino[1]***

[1]Brazilian Synchrotron Light Laboratory (LNLS), Brazilian Center for Research in Energy and Materials (CNPEM), Campinas, Brazil veronica.teixeira@lnls.br

Carnaúba, an acronym for Coherent X-ray Nanoprobe beamline, is an X-ray nanoscopy facility that operates from tender to hard X-rays, enabling 2D, 3D, hyperspectral, and multispectral studies. With a multi-analytical approach based on techniques like X-ray excited optical luminescence (XEOL), X-ray fluorescence (XRF), X-ray absorption (XAS), X-ray diffraction (XRD), and coherent diffractive imaging (CDI), Carnaúba offers versatility for applications in a wide range of scientific fields, including nanoscience, cultural and natural heritage, energy materials, catalysis, photonics, agriculture, microelectronics, among others.

Carnaúba has an all-achromatic mirror-based optical design with focalization achieved through Kirkpatrick–Baez (KB) mirrors and a four-bounce Si (111) monochromator, operating with a resolving power of $\Delta E/E = 10^{-4}$. This beamline covers an energy range from 2.05 to 15 keV and comprises two experimental stations: Tarumã, *Tender-to-h***ar***d X-ray for s***u***b-***m***icro* **a***nalysis,* currently operational, and working with submicrometric beam and variable sample environment; and Sapoti, ***S****canning* **A***nalysis by* **P***tych***o** *for* **T***omographic* **I***maging*, which has been assembled and commissioned. It will operate with a diffraction-limited beam size (down to 30 nm x 30 nm), and cryogenics and ultra-high vacuum sample environment. Ptychography-based results at the Tarumã station have achieved an imaging resolution of 10 nm, up to this moment, the highest spatial for X-ray-based imaging at Sirius—the Brazilian 4th-generation synchrotron light source, operated by the Brazilian Synchrotron Light Laboratory (LNLS), which is part of the Brazilian Center for Research in Energy and Materials (CNPEM).

This talk will present highlights of the beamline, experimental setups, results, and ongoing developments that will open new opportunities for nanoscience at Sirius.

**Keywords: Synchrotron radiation; Carnaúba beamline; nanoscopy; coherent diffractive imaging; ptychography**

[1] Tolentino, Hélio CN, et al. "The CARNAÚBA X-ray nanospectroscopy beamline at the Sirius-LNLS synchrotron light source: developments, commissioning, and first science at the TARUMÃ station." Journal of Electron Spectroscopy and Related Phenomena (2023): 147340

*V.C. Teixeira acknowledges the São Paulo State Research Foundation (FAPESP) and the Brazilian Council for Scientific and Technological Development (CNPq)*

# *Unravelling crystal structures with neutron powder diffraction*

***V. Peçanha-Antonio,*** [1,2]

[1] ISIS Neutron and Muon Source, STFC Rutherford Appleton Laboratory, Didcot OX11 0QX, United Kingdom, [2] Department of Physics, University of Oxford, Clarendon Laboratory, Oxford OX1 3PU, United Kingdom viviane.antonio@stfc.ac.uk

In this talk, I will explore the capabilities of neutron diffraction to answer questions about structural and magnetic ordering of a very diverse host of compounds. Particularly for magnetic systems, neutron diffraction is crucial to elucidate magnetic moment directions, apart from being the only technique available allowing the quantification of magnetic moments for any long-range ordered compound. I will show results on magnetically frustrated compounds, where sometimes other techniques fail to display long-range order at the lowest possible temperatures. I will also explore more complicated magnetic structures displayed, for example, in intermetallic systems, where spin-density-wave states are common. Finally, I will show how combined results of neutron diffraction on powder and single-crystal samples can be used to ascertain crystallographic and magnetic structures.

**Keywords: Magnetism; frustration; neutron diffraction**

# Invited Talks

I Latin American Powder Diffraction Conference – LAPDiC

Fortaleza, October 13 and 14, 2025

# *$SrMnO_x$ (2.25<x≤3) revisited: Oxygen-vacancy, charge and spin ordering in a not-so-simple perovskite.*

***L. Suescun***

Cryssmat-Lab/DETEMA, Facultad de Química, Universidad de la República. Av. Gral. Flores 2124, Montevideo, 11800, Uruguay leopoldo@fq.edu.uy

Perovskite oxides were the stars of the solid-state chemistry and physics communities in the 90’s and first decade of the century due to their promising transport and magnetic properties and thanks to improved access to synchrotron and neutron powder diffraction facilities. This interest, however, shifted towards halides, chalcogenides and pncitides with the “boom” of perovskite solar panels and the discovery of superconductivity in the latter. In recent years, the main interest in perovskite oxides resides within the catalysis and solid-oxide cell communities where the old oxygen-vacant $ABO_{3-\square}$ oxides provide convenient ionic and electronic transport and catalytic activity for oxygenexchange reactions at high temperatures. The study of oxygen vacancy ordering and related phenomena in perovskites has fallen off the spotlights leaving some open questions and unfinished studies, that still may be required to fully understand the interesting and complex behavior of 3d cations in the solid state.

The $SrMnO_x$ system (2.25<x≤3) with the cubic-derived perovskite structure [1] is one of those “old” systems showing interesting oxygen-vacancy arrangements allowing for the observation of at least five different crystal structures for different values of x and, therefore, Mn formal charge. In this system a very strong association between Mn charge, coordination number and vacancy-ordering arrangement has been described that directs order-disorder and drives interesting magnetic structures. As an example, $Mn^{2+}$ found in the 2.25<x<2.5 region [2] is only present it $MnO_4$ tetrahedra while $Mn^{4+}$ present in the 2.5<x≤3 is only present in $MnO_6$ octahedra [1,3,4]. $Mn^{3+}$ being a pyramidal $MnO_5$ center when associated with vacancy order that could only be local for x=2.25 [2] or global [3-4]. $Mn^{III}O_5$ pyramids show a strong apical distortion suggesting orbital ordering even at room temperature defining complex magnetic structures.

In this presentation I will give an overview of oxygen-vacancy ordered and disordered phases in $SrMnO_x$ system showing some published [2-4] and unpublished results collected with X-rays, neutrons and electrons on powders of different compositions over the years, that were never put together in a systematic way. I hope I will highlight how the analysis of structural trends in these compounds give a better understanding of the behavior of Mn cations in perovskite oxides and the relationship between oxygen-vacancy, orbital, charge and spin ordering, as well as order-disorder relations based on these findings.

[1] R.S. Tichy, J. B. Goodenough, Solid State Sci. 2002, 4, 661-664.
[2] E. Dixon, J. Hadermann, M. Hayward, Chem. Mater, 2012, 24, 1486-1495.
[3] L. Suescun et al. (2007) J. Solid State Chem. 180, 1689-1707. [4] L. Suescun, B. Dabrowski, Acta Cryst. B, 2008, 64, 177-186.

**Keywords: Perovskite, Manganites, oxygen-vacancy ordering, magnetic ordering, powder diffraction**

*L.S. is thankful to Dr. J. D. Jorgensen of MSD-ANL, Prof. B. Dabrowski, Prof. O. Chmaissem, S. Kolesnik, J. Mais and staff of Physics Department – NIU, and J. Hadermann at EMAT-U. Antwerpen for data collection, analysis and helpful discussions.*

# *The JATOBÁ (Joint Analysis by TOtal and wide-angle X-ray scattering BeAmline) beamline for PDF analysis at SIRIUS/LNLS.*

***A. Schmidt, R. Szostak and H. C. N. Tolentino***

Brazilian Synchrotron Light Laboratory, Brazilian Center for Research in Energy and Materials (LNLS/CNPEM); Campinas, 13083-100, Brazil.

arianeeschmidt@gmail.com

The total scattering technique encompasses both Bragg diffraction from crystalline structures and diffuse scattering, which is related to short-range order effects, and without the need for crystalline ordering. The signal obtained from the total scattering experiment is used to obtain the function known as PDF (Pair Distribution Function). The JATOBÁ (Joint Analysis by TOtal and wide-angle X-ray scattering BeAmline) beamline will produce a high-energy beam in three different energies (41.2, 54.7, and 68.3 keV), high-photon flux (1-15 x 1012 ph/s/100 mA), and a beam focused on micrometric dimensions (around 20x50 mm^2 (H x V)). The beamline will be dedicated to the study of a wide range of materials using the full X-ray scattering and Pair Distribution Analysis (PDF) method. It will be equipped with several setups, allowing experiments ex-situ, in situ, and operando in different areas such as catalysis (electro-, photo-, thermo-), energy storage (batteries), energy conversion (solar cells), Metal Organic Frameworks (MOFS), nanoparticles, and other amorphous and low crystalline materials. The beamline will also have capabilities for 2D and tomography experiments, as well as grazing incidence. Beyond the total scattering, PDF, and grazing incidence PDF analysis, the beamline will also allow to perform Wide Angle X-ray Scattering (WAXS) and Grazing Incidence Wide Angle X-ray Scattering (GIWAXS) experiments. The beamline construction is underway and at a good pace and is expected to start commissioning in 2026.

**Keywords: Jatobá; Total Scattering; PDF; WAXS; GIWAXS.**

# *Real-time and in-situ Powder diffraction of mechanochemical synthesis of thermoelectric nanocrystalline materials*

***M. A. Malagutti[2], R. E. Camps[1], G. Hermogenes[1], R. Dinnebier[3], C. Weidenthaler[4], C. E. M. Campos[1]***

[1]Departamento de Física, Universidade Federal de Santa Catarina, Florianópolis – SC, Brasil.
[2]Department of Civil, Environmental and Mechanical Engineering, University of Trento, via Mesiano 77, 38123 Trento, Italy.
[3]Max Planck Institute for Solid State Research, Heisenbergstrasse 1, 170569, Stuttgart, Germany [4]Max-Planck-Institut fur Kohlenforschung, Heterogeneous Catalysis, Kaiser-Wilhelm-Platz 1, 45470 Mulheim, Germany
Email of communicating carlos.campos@ufsc.br

Thermoelectric materials have been intensively reinvestigated in recent decades due to their great potential as alternative energy generators and their recovery from waste heat sources [1]. Nanostructured semiconductors are in the spotlight to overcome challenges by improving the performance of thermoelectric devices, so to speak, to obtain a high thermoelectric merit index, ZT [2]. Mechanochemistry is an excellent green and easy route, seen as a fundamental step in the production of thermoelectric nanomaterials, allowing the increase of electrical conductivity and the reduction of thermal conductivity by the creation of phonon scattering centers in the complex microstructure of their products, containing nanometric crystalline domains [3]. With a better understanding of these physicochemical mechanisms driven by the mechanical energy of ball milling, we present a series of real-time and in-situ powder diffraction experiments using synchrotron radiation and a tailored milling setup that allows us to follow the chemical reactions between bismuth and tellurium from the first 10 seconds of milling [4]. The direct formation of binary nanocrystalline phases was revealed within the first minutes of milling, and fine-tuning of the phase composition was achieved simply by choosing the corresponding starting material. Although sequential fitting procedures are well established in the most popular Rietveld analysis systems [5], bismuth-tellurium crystallography is quite complex, mainly due to the predominance of hexagonalrhombohedral structures for all binary and even ternary alloys [6]. This presentation is dedicated to showing and discussing with the event audience the methodology, strategies and main results of our analysis of powder diffraction data aiming to implement more sophisticated modeling of anisotropic microdeformations, stacking faults, etc.

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**Keywords: Thermoelectricity; nanomaterials; mechanochemistry; real-time and in-situ powder diffraction; Rietveld**

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# *Micro-XPD analysis of archaeological materials using a laboratory SAXS/WAXS instrument*

***M. Landino[1]; M.C. Fuertes[2]; V. Palamarczuk[3]; L. Gheco[4]; E. Ahets Etcheberry[1]; M. Tascon[1]; F. Marte[1]; E.E. Asto Ramos[5]; C. Huck-Iriart[5,6]; D.G. Lamas[5]***

[1]Centro de Estudios sobre Patrimonios y Ambiente (CEPyA)-EAyP-EHyS, UNSAM-CONICET, San Martín, Buenos Aires, Argentina. [2]Gerencia Química e INN, Centro Atómico Constituyentes, CNEA, CONICET, San Martín, Buenos Aires, Argentina; [3]Instituto de las Culturas (IDECU), UBA–CONICET, Buenos Aires, Argentina. [4]IRES, CONICET-Universidad Nacional de Catamarca, Argentina. [5]Laboratorio de Cristalografía Aplicada, ITECA, CONICET-UNSAM, San Martín, Buenos Aires, Argentina. [6]ALBA Synchrotron Light Source, Cerdanyola del Vallès, Spain.

Email: dlamas@unsam.edu.ar

X-ray powder diffraction (XPD) is one of the most informative techniques for identifying the crystallographic phases present in various archaeological materials, such as ceramics and pigments. Due to the complexity of these materials high resolution and a collimated X-ray beam size are required. Besides, due to sampling restrictions associated with cultural heritage preservation, the available samples are usually extremely small (less than 1 mm²), and only nondestructive techniques can be used. Consequently, micro-XPD instrumentation is required for the analysis of these complex matrices; nevertheless, access to such facilities is limited in our region.

In this work, we present a micro-XRD approach using a laboratory simultaneous Small- and Wide-Angle X-ray Scattering (SAXS/WAXS) instrument as a viable alternative to synchrotronbased methods. To explore the applicability of this instrumentation, we studied several archaeological materials from the province of Catamarca, in the northwestern of Argentina, such as pottery fragments and rock painting micro-samples. The micro-diffraction studies were performed using a SAXS/WAXS XENOCS Xeuss 2.0 instrument located at the Laboratorio de Cristalografía Aplicada, ITECA institute, CONICET-UNSAM (Argentina). It is equipped with a GeniX3D Cu microfocus source (Cu-Kα X-ray beam) and two Dectris Pilatus3 R hybrid pixel photon counting detectors: 200K-A and 100K models for SAXS and WAXS studies, respectively, that opere simultaneously. Combining both detectors, data were collected within a 2θ range of 045°. A transmission geometry was used, with illuminated areas of about 500 μm × 500 μm.

As a first case study, we analyze black pigments used in Late Period (11th–17th centuries) painted pottery in the Yocavil Valley (Argentina). Changes and continuities in the composition of this material over an extended span were studed in regional sample of 47 potsherds from the Loma Rica, San José, and Santa María styles. Our results indicate the simultaneous presence of iron oxides (hematite) and solid solutions of manganese and iron oxides (jacobsite–magnetite). Besides, the paints were prepared by mixing the pigments with clay-based binding materials.

The second case study involves the characterization of rock painting samples from the archaeological sites of Oyola and La Candelaria (Catamarca, Argentina). The analysis of 28 micro-samples allowed us to identify several crystalline phases, primarily degradation products affecting the paintings. Considering the complex structure of these samples, further analysis using synchrotron-based techniques may be necessary to detect additional phases.

**Keywords: Micro-XPD; SAXS/WAXS; Archaeometry; Northwestern Argentina**

# *Solving crystal structures of pharmaceutical compounds with X-ray powder diffraction*

***F. F. Ferreira***

Centro de Ciências Naturais e Humanas (CCNH), Universidade Federal do ABC (UFABC), Av. dos Estados, 5001, Santo André, SP, 09210-580, Brazil fabio.furlan@ufabc.edu.br

X-ray powder diffraction (XRPD) is an essential technique for the structural characterization of pharmaceutical compounds. The Rietveld method, which involves refining parameters of a structural model to match the calculated X-ray powder diffraction pattern with the observed one, plays a crucial role in this process. This method allows for the refinement of crystal structures, quantification of both crystalline and amorphous phases, and other significant applications. Pharmaceutical formulations in solid form often show variations in their physicochemical properties, such as melting point, solubility, true density, and dissolution profile. These differences can affect the drug's efficacy and bioavailability. Many drugs are polycrystalline and can be effectively studied using X-ray diffraction. When the crystal structures are known, the Rietveld method is a valuable tool for structural analysis. However, when the structure is unknown, the first step is to efficiently collect X-ray diffraction data and apply computational methods to determine the crystal structure, followed by the use of the Rietveld method.

In this talk, I will demonstrate how powder diffraction can be used to determine the crystal structures of several candidate compounds for new drugs, combined with a simulated annealing approach.

**Keywords: Pharmaceuticals; X-ray powder diffraction; crystal structure determination; Rietveld method.**

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# MS 1 - Synchrotron X-ray Powder Diffraction

# Pressure-induced structural transitions in tantalum- and antimony-based trirutile oxides

**O. Canton[1*], N. P. Westin[1], R. Tartaglia[2], and E. Granado[1]**

[1]UNICAMP, IFGW, 13083-970, Campinas, Brazil; [2]CNPEM, LNLS, 13083-970, Campinas, Brazil

*canton@ifi.unicamp.br

High-pressure studies provide valuable insights into how atomic structures respond to external forces, revealing the mechanisms that govern material properties. These insights are especially crucial when examining compounds with complex structural and electronic behaviors, where even small changes in pressure can lead to significant alterations in their physical properties [1,2]. The trirutile $AB_2O_6$ compounds (where A = Ni, Co and B = Sb, Ta) exhibit unique magnetic properties [3,4], and a chemical structure conducive to gas sensing [5]. Given these characteristics, they o3er an intriguing platform for exploring how the ions at the A and B sites in4uence the stability of the trirutile structure, especially in comparison to the parent rutile structure, an example of which being $TiO_2$, that undergoes a clear symmetry-lowering phase transition at approximately 11 GPa [6].

In our study, we performed pressure-dependent X-ray powder diffraction experiments on these $ATa_2O_6$ (A = Ni, Co) compounds, as well as their Sb-based counterparts, at the EMA beamline of the Brazilian Synchrotron Light Source. Our results reveal a symmetry-lowering phase transition occurring around 13 GPa in all the compounds. Interestingly, the tantalum-based materials exhibit a subsequent symmetry-increasing transition near 25 GPa. Upon decompression, the antimonate compounds revert to their original phase, while the tantalate compounds retain the high-pressure phase even at ambient conditions. These findings highlight the differences between the Ta- and Sb-based compounds, demonstrating that tantalum introduces further structural instability. Additionally, our results suggest that cobalt at the A site helps stabilize the phase, as the transitions occur at higher pressures for the Co-containing samples.

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**Keywords: Transition Metal Oxides, High-Pressure, Phase Transitions**

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# *In situ synchrotron XRD evaluation of high-entropy perovskites: exploring redox cyclability for catalytic applications*

***S. Baúti[1,2], S. F. Moya[1], C. B. Rodella[2], J. M. Gonçalves[3], O. L. S. Ferreira[1], D. do Carmo[2], B. B. N. S. Brandão[1], H. Zanin[1]***

[1]School of Electrical and Computer Engineering, University of Campinas, Av. Albert Einstein 400, Campinas, SP – 13083-852, Brazil, [2]Brazilian Synchrotron Light Laboratory (LNLS), Brazilian Center for Research in Energy and Materials (CNPEM), Campinas, São Paulo 13083-970, Brazil, [3]Mackenzie Institute for Research in Graphene and Nanotechnologies (MackGraphe), Mackenzie Presbyterian Institute, 896, 01302-907 São Paulo, Brazil sofia.bauti@lnls.br, hzanin@unicamp.br

Entropy-driven materials find applications in catalysis from the perspective of clean energy production. Due to this context, high structural stability materials with the possibility of catalytic recovery are a cutting-edge challenge in this research field [1]. The perovskite structure thus emerges as a suitable option for incorporating catalyst elements and maintaining structural stability [2,3]. Conversely, high-entropy materials (HEMs) exhibit relevant catalytic properties such as enhanced catalytic activity, active phase tunability, and regeneration [4]. In this scenario, HEMs in a perovskite-type crystalline structure offer a compelling strategy to integrate the catalytic advantages of high-entropy systems with structural stability, phase control, and potential for cyclic exsolution processes that are intrinsic to perovskite-based materials [3]. High-entropy (La0.35Sr0.35Ba0.30)(Fe0.6NixCo(x-1))O3 (x=0, 0.1, 0,2) has been developed using the spray drying technique, which enables the formation of high surface microspherical particles, favorable for catalysis applications, and in-situ synchrotron X-ray diffraction measurements were performed during thermal cycling under reductive and oxidative atmospheres aiming to understand the time resolved perovskite structural stability and the exsolution process of this material. To conduct this work, the catalysts were heated in 3% $H_2$/He atmosphere up to 850 °C and subsequently cooled in synthetic air (80% $N_2$/20% $O_2$), both under a flow rate of 5 mL/min. This procedure simulated real catalytic operation within a 1 mm x 6 mm capillary catalytic bed. Diffraction patterns were acquired in transmission mode using a Pimega 450D detector. To minimize sample absorbance, the catalysts were diluted with 80% quartz wool. The use of synchrotron radiation enabled the detection of segregated Co and Ni nanoparticle phases at low concentrations during the heating cycles, as well as their subsequent reincorporation into the oxide structure under the oxidizing cooling atmosphere. Moreover, it was observed that nanoparticle exsolution occurs at different temperatures, depending on the nickel concentration in the original perovskite structure. In summary, in-situ synchrotron XRD measurements are essential to unveil the dynamic structural stability and time-resolved exsolution behavior of high-entropy perovskite catalysts under realistic redox conditions, enabling insights inaccessible through conventional techniques.

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**Keywords: perovskite structure; high-entropy materials; catalysts; synchrotron light XRD**

# MS 2 – Total Scattering Methods for Materials Characterization

# *Probing Oxygen Vacancy-Induced Transformations in Fe-doped $TiO_2$ Using X-ray Total Scattering*

***G. B. Strapasson[1,2,3], A. S. Arjona[3], J. E. McPeak[3], O. A. Frederiksen[3], A. F. Sapnik[3], N. L. Baun[3], H. N. Bordallo[4], C. B. Rodella[2], D. Zanchet[1], and K. M. Ø. Jensen[3]***

[1]Institute of Chemistry, University of Campinas, UNICAMP, Campinas, SP, Brazil, [2]Brazilian Synchrotron Light Laboratory, CNPEM, Campinas, SP, Brazil, [3]Department of Chemistry, University of Copenhagen, Universitetsparken 5, 2100 Copenhagen Ø, Denmark guilherme.strapasson@lnls.br

Oxygen vacancies and defect engineering in reducible metal oxides are key strategies for tuning structural, electronic, and catalytic properties [1]. Among these materials, titanium dioxide ($TiO_2$) stands out due to its structural flexibility and ability to accommodate dopants and defects that significantly alter its functional properties [2]. Understanding how oxygen vacancies drive structural evolution in doped $TiO_2$ systems is essential for advancing their design and application. In this work, we provide a comprehensive investigation into the structural evolution of Fe-doped $TiO_2$ nanomaterials, focusing on the role of oxygen vacancies in driving phase transformations and defect formation. By systematically varying the Fe loading and synthesis time, we demonstrated that Fe incorporation induces a sequence of structural transformations, from anatase at low Fe concentrations to a defect-rich rutile phase at higher loadings. These transformations are accompanied by the formation of disordered shear-plane-like defects, which emerge as a structural response to stabilize the oxygen-deficient lattice and enable dopant incorporation beyond conventional solid solubility limits. The structural analysis combining *ex situ* and *in situ* total scattering and Pair Distribution Function (PDF) analysis was crucial to capture the local to mediumrange distortions that are not resolved by conventional Bragg scattering. PDF analysis provided key insights into defects evolution, revealing the presence of extended disordered domains and their progression with Fe loading. *In situ* hydrothermal PDF analysis further allowed the identification of transient intermediates and the nucleation and crystallization pathways under synthesis conditions, highlighting the influence of Fe on particle formation mechanism. Together, these findings establish that the structural evolution of Fe-doped $TiO_2$ nanomaterials is governed by the interplay between dopant incorporation, oxygen vacancy generation, and extended defect formation. The detailed nanoscale picture provided by PDF analysis was critical to uncovering these mechanisms, reinforcing its relevance as a powerful tool for understanding complex structural dynamics in reducible metal oxide nanomaterials and offering new perspectives for defect-engineering strategies.

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**Keywords: nanomaterials; reducible metal oxides; pair distribution function**

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# *PDFxDDB: a database for pair distribution function patterns of amorphous drugs*

***M. C. Silva[1], A. C. Silva[2], V. D. N. Bezzon[2]***

[1]Center of Mathematics, Computing and Cognition, Federal University of ABC, Avenida dos Estados, 5001. Bairro Bangu - Santo André - SP - Brazil.

[2]Department of Physics, Federal University of Ouro Preto, Rua Quatro, Campus Universitário Morro do Cruzeiro, Ouro Preto - MG, Brazil.

Email of communicating vinicius.bezzon@ufop.edu.br

Active pharmaceutical ingredients (API) are usually used in crystalline state for drug formulations due to their stability in environmental conditions. However, many crystalline drugs face issues concerned to their low solubility, which can be improved through amorphization of the structure. The amorphous state has higher inner energy, and this can provide an increase in solubility [1-2].

In this case, the use of the Pair Distribution Function (PDF) method is highly useful for characterization of the local structure. The amorphous state can be generated by different routes (ball-milling, melt-cooling, spray-drying), therefore the PDF can be used to identify variations in the structure induced by the route. Although high-energy is suggested to obtain the X-ray total scattering data in PDF analysis, some authors have demonstrated that PDF patterns obtained using Mo and Ag radiation provided high-quality information of local structures for drug samples
[3].

Consequently, considering the importance of amorphous drugs in the field, the possibility of apply PDF method with medium-energy data in local amorphous structures, and thus to identify the influence of the amorphization route, we have been developing the PDFxDDB, a new PDF drug database that uses a Deep Neural Network to classify the input amorphous PDF pattern in the patterns obtained by different routes and available in the database. The purpose is creating an environment for medium-energy PDF patterns, especially from Mo and Ag radiation found in lab diffractometers, providing broad access for usual laboratories. The first results demonstrated the efficiency of the proposed algorithm of the PDFxDDB in classifying the input pattern in one of the patterns available in the database, validating in which route the sample was obtained [4]. We used and a proof-of-concept the flubendazole drug, obtaining the samples in the ball-milling and solvent-evaporation amorphization routes and the PDF patterns with Mo radiation.

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**Keywords: Amorphous drug; PDF method; Deep Neural Network; database**

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# *How the "nano" nature of the nanomaterials is revealed using X-Ray Powder Diffraction*

***J. M. Delgado***

Laboratorio de Cristalografía-LNDRX, Departamento de Química, Facultad de Ciencias Universidad de Los Andes, Mérida 5101, Venezuela.
Email of communicating jmdq2000@gmail.com

One of the most consequential developments in X-Ray Powder Diffraction (XRPD) has been the demonstration of the relationship between the broadening of the diffraction peaks and the size of the *Kristallchen* (crystallites, *cristalitas* or *cristalitos*) in materials by Paul Scherrer summarized in his well-known equation [1]. After more than 100 years of this equation, it is still widely used in spite of its serious limitations and the impressive advances experienced since then.

In general, in the study of nanomaterials the crystal structures of the materials are known. After a proper identification, the structural characterization of nanomaterials focuses primarily on the determination of the size, shape, and distribution of the crystalline domains and other aspects of their microstructure.

Following the pioneering work of Scherrer, the so-called *Line Profile Analysis* (LPA) was expanded by the seminal contributions of Williamson and Hall (WH) [2], and Warren and Averbach (WA) [3]. After the profile refinement method developed by Rietveld [4] (another great development in XRPD), the introduction of the Voigt function in XRPD by Langford [5], the pseudo-Voigt [6] and other functions, the *Whole Powder Pattern Fitting* (WPPF) method has been successfully implemented in improved versions of the WH and WA methods and in the Rietveld method itself in sophisticated programs, which include parameters associated with domain size and microstrain.

The use of physical models for the different microstructural aspects influencing the broadening of the diffraction peaks developed by Scardi and Leoni [7] constitutes the foundation of the *Whole Powder Pattern Modeling* (WPPM) method, an important advance in the characterization of the microstructure starting with, but not limited to, the determination of the size, shape, and distribution of the crystalline domains.

Total scattering methods, using the Debye equation and the Pair Distribution Function, are increasingly being employed in recent years in the study of nanomaterials.

In this short presentation, a revision of these fascinating developments will be presented highlighting some of the strengths and weaknesses of the different methods in the context of the characterization of nanomaterials.

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**Keywords: Nanomaterials; Microstructure; Domain size; Microstrain.**

# MS 3 – Multi-phase Identification and Quantification

# *Monitoring the formation pathway and cation ordering of a rocksalt battery material via in-situ X-ray diffraction*

***M. Azevêdo[1], A. Menezes[1,2], R. Mouta[3,4], C. Santos[1,2]***

*[1] Programa de Pós-graduação em Física, Universidade Federal do Maranhão, São Luís, MA, Brasil, CEP: 65080-805*

*[2] Departamento de Física, Universidade Federal do Maranhão, São Luís, MA, Brasil, CEP: 65080-805*

*[3] Programa de Pós-graduação em Física, Universidade Federal do Ceará, Fortaleza, CE, Brasil, CEP: 60440-900*

*[4] Departamento de Física, Universidade Federal do Ceará, Fortaleza, CE, Brasil, CEP: 60440900*

Email of communicating rodolpho@fisica.ufc.br

Rocksalt oxides with partial cation ordering are pivotal materials for promoting rapid Li-ion transport in protective cathode coatings for lithium-ion batteries (LIBs). When appropriately doped or partially substituted, members of this family can attain Li-ion conductivities on the order of 0.1 mS/cm, positioning them as candidates for solid electrolytes in all-solid-state LIBs. However, critical aspects of their synthesis–structure relationships remain insufficiently resolved: the detailed formation pathway, the relative proportions of cation-ordered and cation-disordered regions, and the characteristic ordering-domain size as functions of synthesis temperature are still poorly understood. In this work, we track the solid-state synthesis of a representative rocksalt oxide material for LIBs using in-situ powder X-ray diffraction, enabling us to (i) establish the sequence of intermediate phases, (ii) quantify their evolving phase fractions, and (iii) estimate the temperaturedependent ordering-domain size of the targeted product. These results provide a temperatureresolved picture that supply practical guidelines for the rational selection of synthesis parameters to deliver specified phase purity, degree of cation ordering, and ordering-domain size – specifications that, in turn, can exert a decisive influence on Li-ion transport performance.

**Keywords: Lithium-ion batteries; Coating; Solid electrolyte; In-situ X-ray diffraction; Solid-state synthesis**

# *Mineralogical Study of Ash from the Poás Volcano: Geological and Risk Implications*

***J. Saavedra-Arias[1], B. M. Villegas[1], L. E. M. Cambronero[2], G. Avard[3]***

[1]Department of Physics, Faculty of Exact and Natural Sciences, Universidad Nacional, Heredia, Costa Rica, [2]School of Chemistry, Faculty of Exact and Natural Sciences, Universidad Nacional. Heredia, Costa Rica, [3]OVSICORI, Faculty of Earth and Marine Sciences, Universidad Nacional, Heredia, Costa Rica jsaavedr@una.ac.cr

Poás Volcano is an active stratovolcano located in Costa Rica's Central Volcanic Range, within the Poás Volcano National Park. Its main crater, approximately 1.5 km in diameter and up to 300 meters deep, contains a turquoise acidic lagoon known as Laguna Caliente—one of the most acidic on Earth. The volcano's activity is directly related to the subduction of the Cocos Plate beneath the Caribbean Plate along the Middle America Trench [1]. Poás has exhibited a range of eruption types, including phreatic, phreatomagmatic, and magmatic events. Its eruptions are typically explosive due to magma interacting with the surface hydrothermal system, causing sudden releases of steam and gases. Nearby towns such as Alajuela, Grecia, and San Pedro de Poás lie within the potential impact zone of ashfall, gas emissions, and lahars. Notably, eruptions in April 2017 led to rock ejections and the temporary closure of the national park, highlighting the volcano's high threat level [2]. This status has resulted in continuous monitoring and strict access protocols, significantly impacting local communities reliant on tourism [3].

This study analyzes pyroclastic material collected from various eruptions between 2017 and 2025, a period marked by eruptive crises, degassing, and fluctuating volcanic activity. Elemental composition was determined through X-ray fluorescence (XRF), while mineralogical and morphological characterization was performed using X-ray diffraction (XRD) and scanning electron microscopy coupled with energy-dispersive X-ray spectroscopy
(SEM-EDS). Preliminary results reveal the presence of juvenile magma fragments (labradorite, tridymite), hydrothermal alteration products (natroalunite, calcium sulfate), and sublimation minerals (pyrite, sulfides). These findings suggest the coexistence of magmatic, hydrothermal, and sublimation processes, providing insight into the volcano's thermodynamic evolution, material fragmentation mechanisms, and emission dynamics. This contributes to volcanic hazard assessment, environmental geochemistry, and the enhancement of eruption forecasting models through correlation with seismic, gas, and deformation data. The samples also offer a valuable chronological record for future volcanological and environmental research.

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**Keywords: Mineral characterization; Volcanic ash; Poás Volcano.**

# *Systematic analysis of amorphous phase quantification in Rietveld refinement by different techniques*

***G. Darin Filho[1], M. M. Chaves[1,2], C. Ulsen[1]***

[1]Laboratório de Caracterização Tecnológica, Escola Politécnica Universidade de São Paulo,
[2]Instituto de Ciência e Tecnologia de Sorocaba, Universidade Estadual de São Paulo Júlio Mesquita Filho mauricio.chaves@unesp.br

The accurate amorphous matter quantification in polycrystalline samples is a fundamental part of the modern X-ray powder-diffraction practice. Applications range from mineralogical identification polymers, pharmaceutical compounds or new materials, the amount of structurally disordered material governs critical properties such as reactivity, solubility or mechanical performance. The classical Rietveld approach is not suited to accurately quantify the amorphous contribution because the method relies on crystal structure models to calculate diffracted intensities [1]. During the last three decades three complementary strategies have matured into routine protocols—the Internal Standard (IS) method [2], the External Standard (ES) approach and the Partial or No Known Crystal Structure formalism (PONKCS) [3]. This study provides a systematic, comparative discussion of those techniques, as well as their accuracy when applied to the Rietveld refinement, their implementation for different concentrations, and materials. Tests have been made with varying amorphous materials added during the grinding processes in known quantities, and the diffractograms have been analyzed.

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**Keywords: XRPD; Rietveld Method; Amorphous Component; PONKCS.**

# MS 4 – General Interest Powder Diffraction

# *XRDplayground: an interactive educational tool for teaching powder X-ray diffraction (PXRD)*

***F. R. Estrada*, A. Malfatti-Gasperini***

Brazilian Synchrotron Light Laboratory – CNPEM, Campinas, SP, Brazil

Email of communicating flavia.estrada@lnls.br

Powder X-ray diffraction (PXRD) is a widely used technique to investigate the structure and symmetry of materials. However, its teaching is often challenging due to the mathematical complexity and the difficulty in visualizing key concepts. To address these challenges, we developed XRDplayground [1], an open-source educational software written in Python, featuring an interactive graphical interface that allows simultaneous simulation of the unit cell and its PXRD pattern.

The tool enables real-time adjustment of structural parameters such as lattice constants, atomic positions, crystallite size, and X-ray beam energy, with immediate updates to the diffraction pattern. Its main goal is to make learning more visual and intuitive, helping users understand how symmetry and crystal structure influence PXRD patterns, without relying solely on complex mathematical treatments.

XRDplayground was developed within the context of the multidisciplinary school “Escola Ricardo Rodrigues de Luz Síncrotron (ER2LS)” and is aimed at graduate and undergraduate students, postdocs, and early-career professionals from diverse scientific backgrounds. The software allows users to build real or fictitious crystal models without requiring external input files and includes features such as comparison with experimental data, visualization of Miller planes, and control over multiple atoms in the unit cell.

As proof of concept, we present a simulation of some structures, diffraction pattern, demonstrating the tool’s ability to reproduce realistic results. Despite some performance limitations for complex structures, XRDplayground proves to be a powerful ally in crystallography education, promoting active exploration and deeper understanding of PXRD concepts.

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**Keywords: crystallography class; powder diffraction; structural model.**

# Copper Oxidation and the Formation Pathways of $Cs_2CuSbCl_6$ and $Cs_2CuSbBr_6$ Double Perovskites

***F. R. Praxedes, P. L. Forcellini, F. F. Ferreira***

Federal University of ABC – Center for Natural and Human Sciences, Avenida dos Estados 5001, Bangu, CEP: 09210-580, Santo André, SP, Brazil.
Email: fabiano.praxedes@ufabc.edu.br

Double perovskites are promising materials for solar energy applications, serving as an alternative to lead-based halide perovskites due to their improved stability under various conditions, including temperature, humidity, and oxidation [1]. Copper-based perovskites exhibit a low bandgap of approximately 1 eV, making them interesting for solar light harvesting [2]. However, the role of $Cu^+$ in the formation of $Cs_2CuSbCl_6$ and $Cs_2CuSbBr_6$ perovskites remains unclear. In this study, we investigate the synthesis of $Cs_2CuSbCl_6$ and $Cs_2CuSbBr_6$ through a chemical method. The perovskite powder was obtained using a precipitation technique from a stoichiometric acidic solution of precursor halide reagents. We also varied the synthesis process by using dimethyl sulfoxide (DMSO) as a polar solvent. The powders were precipitated in pre-refrigerated isopropanol under an ice bath, resulting in the immediate formation of a dark perovskite powder for $Cs_2CuSbCl_6$ and a green powder for $Cs_2CuSbBr_6$. The presence of a $Cs_4CuSb_2Cl_{12}$ phase, characterized by monoclinic symmetry and space group $C2/m$, was confirmed by X-ray diffraction, indicating high crystallinity. Additionally, small peaks corresponding to the $Cs_3SbCl_9$ (trigonal, $P\bar{3}m1$) phase were detected when the material was synthesized without DMSO. In contrast, when bromine was used instead of chlorine, there was no incorporation of copper into the double perovskite lattice; instead, the reaction favored the formation of the $Cs_3Sb_2Br_9$ phase. Even with DMSO present, the $Cs_3Sb_2Br_9$ phase remained the predominant one. From a crystallographic perspective, $Cs_4CuSb_2Cl_{12}$ is more likely to form than $Cs_2CuSbCl_6$ because the occupation of copper in an octahedral structure with cubic symmetry is unstable due to the inherent instability of $Cu^+$ oxidation and the difference in ionic radii (0.77 pm for $Cu^+$ compared to 0.73 pm for $Cu^{+2}$). The close tolerance factor between $Cu^+$ and $Cu^{+2}$ also impacts the formation of the perovskite phase. However, concerning bromide-based perovskites, the $Cs_3Sb_2Br_9$ phase is significantly more stable and favorable than $Cs_2CuSbBr_6$, which, even in the presence of DMSO, does not yield the expected $Cs_4CuSb_2Br_{12}$ phase. This outcome may be attributed to the high stabilization of bromine in a trigonal symmetry with space group $P\bar{3}m1$. Further details regarding Rietveld refinement, phase quantification, and structural analysis based on bond lengths, bond angles, and bond valences will be discussed.

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**Keywords: Perovskite; crystal structure, X-ray diffraction.**

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# Diffractometric, spectroscopic, and thermal analysis characterization of valsartan - 4-aminobenzoic acid salt-solvate

**P. O. Ferreira[1,2], A. C. Almeida[2], L. T. Ferreira[3], G. P. Costa[2], G. Isquibola[3], M. E. S. Eusébio[4], R. A. E. Castro[4,5], F. J. Caires[2], F. F. Ferreira[1]**

[1]LCCEM/CCNH/UFABC, Santo André-BR, [2]FC/UNESP, Bauru-BR, [3]IQ/UNESP, Araraquara-BR, [4]CQC/IMS/UC, Coimbra-PT, [5]FFUC, Coimbra-PT patricia.osorio@ufabc.edu.br

Salt formation is the oldest and most successful strategy to improve solubility and dissolution rate of drugs. It is estimated that more than 50% of the commercialized drugs in actuality are pharmaceutical salts [1]. Valsartan (VAL), the chosen drug for this work, is an antihypertensive drug classified by the Biopharmaceutical Classification System as a class II drug, which means that VAL has low aqueous solubility and high intestinal permeability [2]. As a coformer, the 4-aminobenzoic acid (PABA) was chosen. PABA is a B-complex vitamin, vitamin B10, widely used in the pharmaceutical industry [3,4]. For this work, an adaptation of a synthetic method to the commercialized drug Entresto® [5] was used to obtain a new salt of VAL with PABA. For the synthesis method, drug and coformer ethanolic solution, in a 0.5 molar ratio (1:1 (n/n)) were left under agitation followed by slow evaporation at a desiccator. The VAL-PABA salt was characterized using powder X-ray diffractometry (PXRD), infrared spectroscopy (FTIR), thermogravimetry and differential thermal analysis (TG/DTG-DTA), and the evolved gas analysis (EGA).

The VALa-PABA (0.5) diffraction pattern presented a crystalline pattern with an amorphization halo. Compared with the PABA, and PABA sodium presented in literature [6], the system presented new peaks at $2\theta$ equal to 9.7°, 17.3°, 25.3°, 25.8°, 26.9°, 30.1° and 38.8°. The FTIR of the system VALa-PABA (0.5) had a broad band in the region from 3700 $cm^{-1}$ to 2700 $cm^{-1}$, which suggests the establishment of interactions between VAL and PABA, in addition to the disappearance of both carbonyl bands of VAL and appearance of the asymmetric and symmetric carboxylate stretching modes ($\nu_{asym}$ and $\nu_{sym}$ $COO^-$) bands in 1599 $cm^{-1}$ and 1407 $cm^{-1}$, which strongly suggest the formation of a new salt. This system presented high thermal stability, up to 300 °C. And the EGA showed that water was first liberated from the heating sample, and then close to 200 °C, ethanol was also liberated which, together with the TG curves, shows that a salt-solvate was formed.

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**Keywords: salt formation; diffractometry; solvate; thermal analysis.**

# MS 5 – Structure Determination and Refinement Using Powder Diffraction

# *Structural Determination by X-ray Powder Diffraction and Supramolecular Analysis of the Opiod Dihydrocodeine Bitartrate*

***V. Ordóñez Ulloa[1], C. G. Albarracín Caballero[1], A. Dugarte Dugarte[2], R. A. Toro Hernández[1], G. Díaz de Delgado[2], J. M. Delgado[2], J. A. Henao Martínez[1]***

[1]Grupo de Investigación en Química Estructural (GIQUE), Universidad Industrial de Santander, Bucaramanga, Colombia. [2]Laboratorio de Cristalografía-LNDRX, Universidad de Los Andes, Mérida, Venezuela.

valentina2210116@correo.uis.edu.co, jahenao@uis.edu.co

Dihydrocodeine bitartrate ($DHC^{+}\cdot Tar^{-}$) is an opioid used for the treatment of severe pain and dry cough. It has been compared to codeine although it has better biological potency. Accordingly, the World Health Organization considers it a second-tier drug for the treatment of cancer-related pain [1]. However, side effects and possible addiction have been a concern as they affect a large percentage of patients [2]. Current information on the drug is limited. The powder diffraction pattern or the crystal structure of the compound could not be found in crystallographic databases, articles, or patents. Therefore, we decided to undertake the structure determination of dihydrocodeine bitartrate. Further characterization was carried out by Infrared spectroscopy (ATR FT-IR) and thermal analysis (TGA-DSC). The X-ray powder diffraction pattern was registered, and a search/match procedure did not show coincidence with any reported patterns in the PDF-5+ database. Indexing of the pattern with DICVOL14, implemented in the PreDICT interface, suggests that the crystal system is triclinic and the final parameters obtained were $\boldsymbol{a}$ = 7.1687(10) Å, $\boldsymbol{b}$ = 8.0786(10) Å, $\boldsymbol{c}$ = 9.4312(12) Å, α = 76.127(8)°, β = 82.112(10)°, γ = 86.818(11)°, V = 525.09(12) Å³ with Z = 1. The analysis of the reflection conditions using EXPO suggested *P*1 as the space group. In the crystalline arrangement, the $DHC^{+}$ cation is connected to the counterion by strong NH···O hydrogen bonds. $DHC^{+}$ moieties pack into linear chains parallel to the *b* axis, alternating with chains of $Tar^{-}$ ions. Hirshfeld surface analysis, fingerprint plots, and intermolecular energy calculations using the 'UNI Intermolecular Potentials' within CSD-Materials in the Mercury interface were also performed.

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**Keywords: dihydrocodeine bitartrate; opioid; X-ray powder diffraction; crystal structure; structure determination.**

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# *Structure Determination of racemic Lomefloxacin Iodide using Laboratory X-Ray Powder Diffraction Data*

***A. Dugarte-Dugarte[1], G. Díaz de Delgado[1], A. N. Fitch[2], J. M. Delgado[1]***

[1]Laboratorio de Cristalografía-LNDRX, Departamento de Química, Facultad de Ciencias, Universidad de los Andes, Mérida 5101, Venezuela, [2]European Synchrotron Radiation Facility (ESRF), 71 Avenue des Martyrs, CS40220, Grenoble, Cedex 9 38043, France

Email of communicating analio@ula.ve

Lomefloxacin ($C_{17}H_{19}F_2N_3O_3$, *LOM*) is a second-generation fluroquinolone antibiotic with activity against a wide range of Gram-negative and Gram-positive organisms. Its hydrochloride form ($C_{17}H_{19}F_2N_3O_3 \cdot HCl$, CAS Number 98079-52-8) is marketed under the name Maxaquin™. It is formulated as tablets and as eye and ear drops [1]. There are several studies in the literature related to the structural characterization of this material and its derivatives. A search in the Cambridge Structural Database [2] (*CSD*) yields 16 entries. No iodide form ($C_{17}H_{19}F_2N_3O_3 \cdot HI$, *LOM·HI*) has been reported. Six cocrystals, eight metal derivatives, one monohydrate and one hydrochloride dihydrate form (*CSD* refcode LATPON) [3] are reported. The raw powder diffraction data of racemic Lomefloxacinium Iodide (*PDF* 00-058-1280) is contained in the PDF-5+ database [4]. Only the unit cell parameters and the monoclinic space group $P2_1/c$ are reported. The crystal structure was determined using the PDF-5+ data by Simulated Annealing with *EXPO* 2.3.8 [5]. The Rietveld refinement was carried out with the *Topas-Academic* software [6]. The structure was evaluated and optimized by dispersion corrected DFT calculations using *CASTEP* software [7] as implemented in the Biovia Materials Studio 22.1 program. This compound crystalizes in a monoclinic cell, space group $P2_1/c$, with unit cell parameters $a$ = 13.3716(7) Å, $b$ = 11.9978(10) Å, $c$ = 12.2140(8) Å, ! = 112.452(3)°, $V$ = 1811.0(2) Å$^3$ with $Z$ = 4. The Rietveld refinement converged to $R_e$ = 0.0482, $R_p$ = 0.0708, $R_{wp}$ = 0.0911, and *GoF* = 1.890. The crystal structure is a complex 3D arrangement of LOM·HI units held together by hydrogen bonds, π···π and C-H···π interactions. Hirshfeld surface analysis and fingerprint plots were performed.

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**Keywords: Lomefloxacin iodide; antibiotic drug; powder diffraction; structure determination; Rietveld method.**

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# Amorphous Forms of Efavirenz and Atorvastatin: Solubility Enhancement and PDF Based Structural Characterization

**L. D. Queiroz[1], V. D. N. Bezzon[2]**

[1]Graduate Program in Materials Science, Federal University of Ouro Preto, Ouro Preto, MG, Brazil.
[2]Department of Physics, Federal University of Ouro Preto, Ouro Preto, MG, Brazil
Email of communicating lucas.dq@aluno.ufop.edu.br

Low drug solubility is a recurring challenge in the development and efficacy of pharmaceuticals, as drugs must dissolve in biological fluids to be absorbed by the body [1]. Compounds with complex and nonpolar structures often exhibit this issue, compromising their bioavailability. A promising solution is amorphization, a process that transforms the drug from a crystalline state to an amorphous one, resulting in higher inner energy and, consequently, improved solubility and therapeutic performance [2].

This study focuses on the amorphization of two important drugs, calcium atorvastatin, used for hypercholesterolemia treatment and the prevention of cardiovascular diseases, and efavirenz, widely used in the treatment of human immunodeficiency virus (HIV-1) infection. Both drugs are known to have solubility issues [3, 4]. The research also involves the structural characterization of these compounds using powder X-ray diffraction. Traditional diffraction techniques are based on the analysis of crystalline materials, where the ordered repetition of unit cells produces welldefined diffraction peaks. However, when a substance is amorphized, this long-range order is lost, and the peaks disappear.

Nevertheless, short-range order remains in amorphous materials. To study them, the Pair Distribution Function (PDF) method is used, which analyzes the distribution of distances between pairs of atoms [5]. This technique generates a statistical profile unique to each atomic arrangement, with distinct peaks at short distances and a leveling off at longer distances. PDF analysis is ideal for disordered structures, whereas conventional diffraction is more suitable for crystalline systems. In the present case, since the materials will be amorphized, structural characterization will be performed using the PDF method.

A preliminary characterization of the raw materials of both drugs was carried out. Using the Rietveld [6] refinement with TOPAS software, we confirmed polymorph I for efavirenz, with an orthorhombic structure and space group $P2_12_12$ [7], and for atorvastatin, we identified calcium atorvastatin trihydrate, with a triclinic structure and space group $P\bar{1}$ [8]. Based on this preliminary characterization, we will carry out amorphization using milling and melt-extrusion techniques.

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**Keywords**: PDF; X-Ray Powder Diffraction; Atorvastatin; Efavirenz

# Posters

# *In situ investigations of the synthesis mechanism and stability under extreme pressures of a niobium-based oxide for rechargeable batteries*

***B. J. S. Mendes[1], M. Oliveira[2], A. S. de Menezes[3], R. Mouta[4], C. C. dos Santos[3]***

[1]Curso de Química Industrial - UFMA, São Luís, Maranhão, Brazil,

[2]Programa de pós-graduação em Física, Universidade Federal do Ceará - UFC, Fortaleza, Ceará, Brazil,

[3]Departamento de Física, Universidade Federal do Maranhão - UFMA, São Luís, Maranhão, Brazil,

[4]Departamento de Física, Universidade Federal do Ceará - UFC, Fortaleza, Ceará, Brazil

Brenda.j.s.mendes@gmail.com

The search for safer and more efficient lithium-ion batteries (LIBs) has driven the development of new anode materials, especially due to the risks associated with lithium dendrite formation under high charging rates on the commercially widespread graphite anode. In this work, we investigated the synthesis mechanism and stability at high pressures of a niobium-based oxide from the Wadsley-Roth family with promising properties for high-rate negative electrodes for LIBs. The material was synthesized by solid-state reaction and characterized by X-ray diffraction (XRD) and Raman spectroscopy. The results confirmed the material formation with high purity, and the in situ XRD measurements elucidated the phase evolution during the synthesis. Synchrotron X-ray diffraction and Raman spectroscopy measurements under pressure revealed an irreversible structural phase transition to a previously unknown polymorph of this anode material. From the pressure-dependent evolution of lattice parameters and unit cell volume, linear compressibilities of both phases and the bulk modulus of the original phase were obtained. These findings not only offer new insights into synthesis mechanism, as well as structural and mechanical behavior of the material, but also revealed a new phase that should be further investigated by searching possible synthesis routes to obtain it at gram scale and then evaluating its performance as a high-rate anode for LIBs.

**Keywords: Lithium-ion batteries; anode materials;**

# *Synthesis and Structural and Magnetic Characterization of Bismuth Ferrite ($BiFeO_3$) Obtained by the Proteic Sol-Gel Method*

***L. Q. Rodrigues[1]*, F. W. C. Vasconcelos[1], M. R. Araujo[1], G. F. Guimarães[2], J. S. Andrad[2], Y. Martinez-Camejo[3], J. M. Sasaki[3]***

[1]Federal University of Ceará, Postgraduate Program in Materials Science and Engineering, Fortaleza - Campus Universitario do Pici - Bloco 729, 60440-554, Brazil,

[2]Federal Institute of Education, Science, and Technology of Ceará, Postgraduate Program in Telecommunications Engineering, Photonics Laboratory, Fortaleza - Av. Treze de Maio, 2081 – Benfica 60040-531, Ceará, Brazil

[3]Federal University of Ceará, Physics Department, Rua Prof. Armando Farias, s/n, Campus do Pici, Fortaleza, 60440-760, Ceará, Brazil

*lidia.quirino@alu.ufc.br

This study aims to investigate the influence of rare-earth ion doping, particularly lanthanum ($La^{3+}$), on the structural and magnetic properties of bismuth ferrite ($BiFeO_3$) nanoparticles. This material belongs to the perovskite family ($ABO_3$) and is classified as multiferroic due to its ability to exhibit both ferroelectricity and antiferromagnetism simultaneously at room temperature. These characteristics make $BiFeO_3$ highly promising for technological applications such as magnetic field sensors, magnetoelectric devices, and solar cells. The samples were synthesized using the proteic-based sol-gel route, a technique that allows precise control over the chemical composition and morphology of the particles. For characterization, advanced techniques such as X-ray diffraction (XRD), X-ray fluorescence (XRF), vibrating sample magnetometry (VSM),were employed, enabling a detailed analysis of the crystalline phases, chemical composition, and magnetic behavior of the material.

From a structural point of view, $BiFeO_3$ crystallizes in a rhombohedral system with space group R3c, where $Bi^{3+}$ ions occupy the A-site (12-fold coordination) and $Fe^{3+}$ ions occupy the B-site (6-fold coordination). The partial substitution of $Bi^{3+}$ by $La^{3+}$ alters the bonding in the crystal lattice, directly affecting the electric polarization and magnetic ordering. This modification enhances the stability of the main phase, improves crystallinity, and strengthens the multiferroic performance of the compound. The incorporation of lanthanum also helps to reduce structural defects and may suppress lattice distortions associated with the presence of bismuth, promoting a G-type antiferromagnetic ordering, as described in the literature.

Despite improvements from $La^{3+}$ doping, Rietveld refinement showed that the secondary phase $Bi_2Fe_4O_9$ still persists due to partial bismuth volatilization during calcination at 550 °C. While doping enhances structural stability, it does not fully eliminate impurities.

Thus, further optimization of synthesis conditions is needed to obtain a pure, single-phase $BiFeO_3$ with better performance.

**Keywords: Bismuth ferrite; Lanthanum doping; Multiferroic materials; Proteic solgel; Perovskite structure; Magnetic properties.**

# Synthesis and characterization of Co-Mg-Al layered double hydroxides (LDH) with adiapate between the layers

**R. Medronho[1], S. Billinge[2], V. Rodrigues[3], A. Faro[3*], L. Palacio[1]**

[1]Universidade do Estado do Rio de Janeiro, Brazil, [2]Columbia University, USA,
[3]Universidade Federal do Rio de Janeiro, Brazil.*Retired lpalacio@uerj.br

This study explores the influence of varying metal concentrations in layered double hydroxides (LDHs), which serve as precursors for catalysts in the oxidative dehydrogenation of propane. The expected structural phase for these materials is hydrotalcite. Co-Mg-Al LDHs were synthesized via coprecipitation using adipate as the interlayer anion. The target formula of the synthesized LDHs is $Co_yMg_{1-y}Al_{0.5}(OH)_2(C_6H_{16}O_8)_{0.25}$, where y = 0.1, 0.2, 0.3, and 0.4.

Inductively coupled plasma (ICP) analysis confirmed that the metals were incorporated in the intended ratios. However, CHN elemental analysis revealed lower-than-expected carbon content, particularly in the sample with the highest cobalt concentration (Co40), suggesting a reduced amount of compensating adipate anions and, consequently, fewer trivalent cations in the LDH layers. Thermogravimetric analysis (TGA) showed similar mass losses across the samples, with Co40 exhibiting a notably lower loss, corroborating the CHN findings of diminished adipate presence.

Fourier-transform infrared spectroscopy (FTIR) displayed characteristic bands at 1560 $cm^{-1}$ and 1400 $cm^{-1}$, corresponding to the symmetric and asymmetric C=O stretching of carboxylate groups in adipate. Additional bands at 550 $cm^{-1}$ and 441 $cm^{-1}$ were attributed to Al–O and metal– oxygen stretching vibrations, respectively.

High-resolution X-ray diffraction (HR-XRD, λ = 0.1646 Å) confirmed the formation of the hydrotalcite phase with adipate intercalated between the layers. Bayerite impurities were detected, being more observable in samples with higher cobalt content (Co30 and Co40). Phase indexing using Checkcell [1] yielded a good fit to the R–3m space group. The increase in lattice parameters with cobalt enrichment suggests isomorphic substitution of magnesium by cobalt, consistent with larger ionic radius of cobalt.

Pair distribution function (PDF) analyses further validated cobalt incorporation, evidenced by shifts to higher r (Å) values and increased peak intensities in cobalt-rich samples. These materials exhibit a nanometric domain size, with smaller structural domains inferred for samples containing less magnesium. Simulations using PDFgui [2] are ongoing.

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**Keywords: Layered double hydroxides, hydrotalcite, catalysts, high resolution powder X-ray diffraction, pair distribution function**

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# *Structural and phase evolution of Al-doped CuO catalysts*

***T. Gomes[1], M. W. Terban[2], F. Zotin[1], L. Palacio[1]***

[1]Universidade do Estado do Rio de Janeiro, Brazil, [2] Momentum Transfer GmbH, 22547 Hamburg, Germany lpalacio@uerj.br

In this study, catalysts for the reduction of nitric oxide (NO) by carbon monoxide (CO) were investigated. This reaction targets the conversion of automotive pollutants (NO and CO) into less harmful gases. The catalysts were synthesized via the precipitation method using an acidic solution containing $Cu(NO_3)_2 \cdot 3H_2O$ and $Al(NO_3)_3 \cdot 9H_2O$, and a basic solution of NaOH and/or $Na_2CO_3$. After precipitation, the solids were vacuum-filtered and dried in a convection oven at 100 °C for 1 h. Calcination was performed at 600 °C for 3 h with a heating rate of 5 °C/min.

The nominal formula of the synthesized materials was $Cu_{1-x}Al_xO_{1+x/2}$, with x values of 0, 0.05, 0.1, 0.2, and 1. X-ray fluorescence (FRX) confirmed successful incorporation of aluminum into the solid structure.

The precursors (pre-calcination) were analyzed via conventional X-ray diffraction (XRD, λ = 1.5406 Å) and thermogravimetric analysis (TGA). With increasing Al content, $Cu(OH)_2$ phase progressively transformed into a Cu–Al hydrotalcite-like phase, becoming more defined at x = 0.2. At x = 1, $Al(OH)_3$ was formed. XRD revealed broad peaks typical of nanoparticulate materials. TGA showed that mass loss up to 1000 °C increased with Al content. XRD analysis of the residue of TGA indicated the formation of CuO and $CuAl_2O_4$ for bimetallic compositions, while monometallic samples produced CuO and $Cu_2O$ (x = 0) or a highly amorphous phase (x = 1).

The calcined catalysts were characterized using high-resolution XRD (HR-XRD, λ = 0.1652 Å) and pair distribution function (PDF) analysis. Samples with x ranging from 0 to 0.2 exhibited a pure CuO crystalline phase, with no detectable $Al_2O_3$ impurities. Even with just 5% Al incorporation, a notable increase in peak broadening and a decrease in intensity were observed. Peak shifts to higher 2θ values suggested Al incorporation into the CuO lattice. PDF comparisons between undoped and Al-doped CuO confirmed the nanoparticulate nature of the catalysts. Al doping significantly reduced the crystallite size of CuO. Simulations with PDFgui [1] provided a good fit for the CuO structure. Further refinements for the doped materials are in progress.

Fourier Transform Infrared (FTIR) spectroscopy was employed to characterize the calcined solids. The spectra revealed distinct absorption bands corresponding to the H–O–H bending vibrations of water molecules, observed near 1635 $cm^{-1}$; the vibrational modes of carbonate ions, detected around 1500 $cm^{-1}$ and 1380 $cm^{-1}$; and the metal–oxygen bonding, indicated by a band at approximately 800 $cm^{-1}$.

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**Keywords: Copper oxide, automotive catalysts, high resolution powder X-ray diffraction, pair distribution function**

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# *In-situ synchrotron X-ray diffraction and mass spectroscopy investigation of the thermal stability and decomposition mechanism of a novel cathode for safer Li-ion batteries*

***M. Oliveira[1], J. V. de S. Neto[2], C. C. dos Santos[2], A. S. de Menezes[2], A. A. Tanaka[2], R. Mouta[1]***

[1]Programa de Pós-graduação em Física, Universidade Federal do Ceará - UFC,

[2]Universidade Federal do Maranhão – UFMA

*e-mail:mariana.oliveira@fisica.ufc.br*

An important step in the energy transition is the decarbonization of the vehicle fleet through the large-scale adoption of electric vehicles. To achieve this, advancements are still needed in the safety, sustainability, and cost of lithium-ion batteries (LIBs) that power these vehicles. In this work, we investigate a novel cathode material with the potential to improve battery safety without compromising sustainability, cost, or energy density. Specifically, we aim to elucidate its thermal degradation mechanism through ex-situ and in-situ X-ray diffraction measurements, including synchrotron radiation and simultaneous in-situ mass spectrometry. In this initial stage of the study, we identified the cathode's decomposition products and found that thermal degradation begins at around 600 °C – significantly higher than the degradation temperature of commercial cathodes. Further analysis of the results, combined with complementary techniques such as X-ray absorption, transmission electron microscopy, and thermodynamic calculations, is expected to guide the development of safer cathode materials for LIBs.

**Keywords**: Lithium-ion batteries; Cathode materials; Thermal stability; X-ray diffraction; Synchrotron radiation

# *Charge density correlations of underdoped $YBa_2Cu_3O_{7-\delta}$ thin films probed by Soft X-ray Resonant Scattering*

***N. P. Westin[1,2,3], K. S. Alcântara[1,2], O. Canton[1], V. P. Gastaldo[2], P. Schio[2], T. Mori[3], T. Rocha[3], and E. Granado[1]***

*[1] Instituto de Física Gleb Wataghin (IFGW), Universidade Estadual de Campinas (UNICAMP), [2] Laboratório Nacional de Nanotecnologia (LNNano), Centro Nacional de Pesquisa em Energia e Materiais (CNPEM), [3] Laboratório Nacional de Luz Síncrotron (LNLS), Centro Nacional de Pesquisa em Energia e Materiais (CNPEM) nwestin@ifi.unicamp.br*

Strongly correlated systems have attracted continuous attention, with complex phase diagrams. In cuprates, the strong Coulomb repulsion has significant impacts on the electronic distribution in the $CuO_2$ planes, where Cooper pairs appear for a given range of temperature and electronic doping. Additionally, the superconductor dome is surrounded by exotic charge ordered phases, most notably 3D or 2D charge density waves (CDW) that may be correlated with superconductivity and nearby states such as the so-called strange-metal and pseudogap regimes. In particular, much work has been done to understand the spontaneous two-dimensional charge-density wave (2D-CDW) order and fluctuations at (H,0) in the reciprocal space with H ~ 0.3 r.l.u. [1,2,3]. These charge orderings are the focus of the present work, where an epitaxial thin film of $YBa_2Cu_3O_{7-\delta}$ (YBCO) in the underdoped regime is analyzed. In order to discern between the X-ray quasi-elastic scattering of CDW orderings and the strong inelastic signal, we performed Resonant Inelastic X-Ray Scattering (RIXS) measurements at the Cu $L_3$-edge. By evaluating the quasi-elastic scattering region, we found a relatively sharp peak at H = 0.31 r.l.u. due to the static 2D-CDW order, and a second broader component centered at H ~ 0.30 r.l.u., attributed to dynamic short-range CDW correlations. This broad feature persists at least up to T ~ 400 K, with an apparent change of behavior at T ~ 300 K. A more systematic study is being planned to possibly correlate the temperature dependence of the RIXS data with the emergence of the exotic electronic phases of cuprates.

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**Keywords: Superconductivity, Charge Density Waves, Soft X-Ray Scattering.**

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# *Pressure-induced structural transitions in tantalum- and antimony-based trirutile oxides*

***O. Canton[1*], N. P. Westin[1], R. Tartaglia[2], and E. Granado[1]***

*[1]UNICAMP, IFGW, 13083-970, Campinas, Brazil; [2]CNPEM, LNLS, 13083-970, Campinas, Brazil.*

*canton@ifi.unicamp.br

High-pressure studies provide valuable insights into how atomic structures respond to external forces, revealing the mechanisms that govern material properties. These insights are especially crucial when examining compounds with complex structural and electronic behaviors, where even small changes in pressure can lead to significant alterations in their physical properties [1,2]. The trirutile $AB_2O_6$ compounds (where A = Ni, Co and B = Sb, Ta) exhibit unique magnetic properties [3,4], and a chemical structure conducive to gas sensing [5]. Given these characteristics, they over an intriguing platform for exploring how the ions at the A and B sites influence the stability of the trirutile structure, especially in comparison to the parent rutile structure, an example of which being $TiO_2$, that undergoes a clear symmetry-lowering phase transition at approximately 11 GPa [6].

In our study, we performed pressure-dependent X-ray powder diffraction experiments on these $ATa_2O_6$ (A = Ni, Co) compounds, as well as their Sb-based counterparts, at the EMA beamline of the Brazilian Synchrotron Light Source. Our results reveal a symmetry-lowering phase transition occurring around 13 GPa in all the compounds. Interestingly, the tantalum-based materials exhibit a subsequent symmetry-increasing transition near 25 GPa. Upon decompression, the antimonate compounds revert to their original phase, while the tantalate compounds retain the high-pressure phase even at ambient conditions. These findings highlight the differences between the Ta- and Sb-based compounds, demonstrating that tantalum introduces further structural instability. Additionally, our results suggest that cobalt at the A site helps stabilize the phase, as the transitions occur at higher pressures for the Co-containing samples.

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***Keywords: Transition Metal Oxides, High-Pressure, Phase Transitions***

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# *Dispersivity and crystallinity of magnetite nanoparticles by x-ray scattering metrology*

***Valeria K. C. Molina[1], Társis M. Germano[1], Bruna P. Lima[1], Giancarlo E. S. Brito[1], S. L. Morelhão[1]***

[1]*Institute of Physics, University of São Paulo, Brazil* valeria.camargo@usp.br

Control of shape and size dispersivity and crystallinity of nanoparticles (NPs) has been a challenge in identifying these parameters' role in the physical and chemical properties of NPs. There are many examples, such as improved catalytic performance of $CeO_2$ NPs by adjusting size, morphology, and lattice perfection [1,2], tuned nonlinear optical response in $BiFeO_3$ nanocrystals for biomedical imaging applications by narrowing phase and size dispersivity [3,4], and self-stabilized catalysts based on NPs with crystalline core and amorphous shell [5]. The need for reliable quantitative tools for analyzing the dispersivity and crystallinity of NPs is a considerable problem in optimizing scalable synthesis routes capable of controlling NP properties. The most common tools are electron microscopy and X-ray scattering techniques. However, each technique has different susceptibility to these parameters, implying that more than one technique is necessary to characterize NP systems with minimum reliability. Wide-angle X-ray scattering (WAXS) is mandatory to access information on crystallinity. In contrast, electron microscopy (EM) or small-angle X-ray scattering (SAXS) is required to access information on the whole NP sizes. EM provides average values on relatively small ensembles compared to bulk values accessed by X-ray techniques. Besides the fact of SAXS and WAXS weight size distribution differently [6], SAXS is easily affected by NP-NP interactions. Addressing the long-standing challenge of cross-analyzing data from techniques with different sensitivities to nanoparticle parameters, we now present an improved SAXS/WAXS-based methodology, building upon our previous work [7], to provide reliable quantitative results for dispersivity and crystallinity in magnetite NPs.

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**Keywords: Crystalline nanoparticles; Size distribution; Magnetite; SAXS; WAXS.**

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# *Study of the crystallization process of $La_2(WO_4)_3$ ceramic via in situ High-Temperature X-ray Diffraction*

***A. L. Pinheiro Neto[1,2], T. E. R. Fiuza[2], T. M. Perfecto[2], E. R. Leite[2,3], I. C. Nogueira[1]***

[1]Federal University of Amazonas (UFAM), [2]Nacional Center of Research in Energy and Materials (CNPEM), [3]Feferal University of São Carlos (UFSCar).
e–mail: antoniolopes@ufam.edu.br

The study of the crystallization process is essential in field of materials science and nuclear industry [1,2]. Although widely investigated, there are still gaps in the physicochemical understanding of this phenomenon, especially when dealing with amorphous, non-glassy precursors. The formation of metal oxides from amorphous phases is particularly relevant in the field of nanotechnology, as it requires precise control over growth and nucleation rates to define the size, shape, and distribution of the particles.

In this work, lanthanum tungstate, $La_2(WO_4)_3$, was synthesized by the co-precipitation method in aqueous medium using $Na_2WO_4$ and $La_2(CO_3)_3$ as precursors. This synthesis route is simple and potentially sustainable, as it employs water as a solvent and operates at low or moderate temperatures, reducing the use of toxic solvents and energy consumption. The method produces an amorphous material that crystallizes directly into monoclinic $La_2(WO_4)_3$ upon thermal treatment, without the formationof intermediate phases [3]. This transformation was observed through thermal analyses—Thermogravimetric Analysis (TGA) and Differential Scanning Calorimetry (DSC) and structural analyse such as X-ray Diffraction (XRD).

The crystallization evolution was studied using in situ high-temperature X-ray diffraction (HTXRD) in the temperature range of 500 to 700 °C, with a heating rate of 10 °C/min, resolution of 0.02°, step time of 0.6s, and exposure time of 5 min, in the 2θ range of 20 to 35°. Crystallization of $La_2(WO_4)_3$ initiated at 610 °C with about 12.3% and progressed to around 72.1% at 700 °C.
Additionally, an isothermal analysis was performed at 580 °C, with data collected every 10 min from 0 to 120 min under the same experimental conditions. In this analysis, crystallization started at 45 min with approximately 1.6% and reached about 18% at 120 min. These results indicate that the thermal treatment time directly influences the phase transformation kinetics.

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**Keywords: Coprecipitation, in situ high-temperature X-ray diffraction; crystallization process.**

*Acknowledgements: CAPES, FAPESP, CNPEM and UFAM*

# *Li-ion conductivity engineering of partially cation-disordered rocksalt oxides via non-stoichiometry*

***M. A. de Oliveira[1], M. Oliveira[2], R. Mouta[3]***

[1]Bacharelado em Física, Universidade Federal do Ceará - UFC, Fortaleza, Ceará, Brazil
[2]Programa de Pós-graduação em Física, Universidade Federal do Ceará - UFC, Fortaleza, Ceará, Brazil
[3]Departamento de Física, Universidade Federal do Ceará - UFC, Fortaleza, Ceará, Brazil
[1]mateus.andrade@fisica.ufc.br, [2]mariana.oliveira@fisica.ufc.br, [3]rodolpho@fisica.ufc.br,

Lithium-ion batteries (LIBs) face safety challenges due to flammable liquid electrolytes. Although there are non-flammable solid-state alternatives with very competitive ionic conductivities (> 1 mS/cm), they suffer from poor air-stability and must be synthesized and/or handled/stored under inert atmosphere. Since this makes their potential large-scale production more challenging and costly, much effort has gone into understanding and trying to mitigate their reactions with CO2 and H2O in the air. Here, we explored the reverse approach: we started from an air-stable but very poor Li+ conductor ($\sigma\sigma$ ~ 10-10 mS/cm at room temperature) rocksalt oxide system and engineered its Li+ conductivity. This was done via nonstoichiometry, which simultaneously introduces a high concentration of charge carriers (Li+ interstitials), changes the energy barrier landscape for Li+ migration though partial cation disorder, and favors percolation due to Li+ occupying a greater fraction of cations sites. These partially cation-disordered oxides were synthesized by solid-state reaction preceded by wet milling and characterized by X-ray powder diffraction (XRD) and impedance spectroscopy. The XRD patterns revealed a selective peak broadening, where the superstructure ones - associated only with the cation-ordered phased - are much broader than those that are common to both phases. This indicates the presence of cation-ordered nanodomains embedded in crystallites that are otherwise cation-disordered, to be confirmed soon via techniques such as transmission electron microscopy and selected area electron diffraction. We observed that shorter dwelling times during the synthesis and higher non-stoichiometry were both parameters that contributed to broaden the superstructure peaks, indicating they result in shorter cation-ordering nanodomain sizes. Impedance results demonstrated that non-stoichiometry indeed increased the conductivity by several orders of magnitude, up to ~10-3 mS/cm at RT. They also showed a correlation between the conductivity gain and the cation-ordering nanodomain sizes. To better understand the impact of cation order/disorder to the conductivity in this non-stoichiometric system, we plan to probe also the local cation arrangements by applying Pair Distribution Function (PDF) analysis to data to be collected either from total scattering measurements using either synchrotron X-rays or neutrons.

**Keywords: Solid-state electrolytes, Ionic conductivity, Disordered rocksalt oxides, Powder diffraction, Nonstoichiometry**

# Synthesis of zeolites from municipal solid waste as a low-cost raw material

***N. P. Garcia[1], W. Aguilar[1]***

*[1]Departamento de Quimica, Facultad de Ciencias y Tecnologia, Universidad Mayor de San Simon.Cbba- Bolivia* pereyra.neysa@gmail.com

Nowadays, environmental regulations promote the valuation of solid waste and encourage its use as raw material to produce value-added materials, instead of said waste being deposited in landfills, which can subsequently generate environmental liabilities. Zeolites are materials that have a microporous structure that gives them properties which give them potential application in different areas such as catalysis, adsorption and ion exchange processes [1], [2].

The present study focuses on the synthesis of zeolitic material derived from glass waste generated within urban waste as a low-cost raw material. It began by applying an alkaline treatment to the raw material. The process was carried out under different conditions, varying the temperature, time and the NaOH:solid waste ratio. The precursors for the synthesis of the zeolitic material were prepared through a hydrothermal reaction, varying the temperature and crystallization time, to optimize the synthesis.

The optimal conditions for the different stages were the following: The optimal temperature and time of the alkaline treatment were 550 °C, 2 h and the NaOH : solid waste ratio was 1:1.5, then the crystallization conditions, both time and temperature, were 100 ºC and 6 hours. Subsequently, the raw material and the zeolitic material were characterized by different methods: to determine the phases present, X-ray diffraction was used and to determine the ion exchange capacity, the ion-selective electrode method was used. The results obtained demonstrate that the products obtained was faujasite zeolites and had a cation exchange capacity of 120 meq/100 g.

The present study revealed that waste-derived zeolitic materials are a promising field for the development of profitable and sustainable zeolites.

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**Keywords: Recycling; alkaline treatment; hydrothermal synthesis; zeolites; sustainable materials.**

# Influence of Phase Coexistence on Ionic Conductivity in Composite Cathode Materials for Lithium-Ion Batteries

**V. S. Paiva[1], R. Mouta[2], C. C. dos Santos[3]**

[1]*Programa de Pós-graduação em Física, Universidade Federal do Maranhão - UFMA, São Luís, Maranhão,Brazil,* [2]*Departamento de Física, Universidade Federal do Ceará - UFC, Fortaleza, Ceará, Brazil,* [3]*Departamento de Física, Universidade Federal do Maranhão - UFMA, São Luís, Maranhão, Brazil* virna.paiva@discente.ufma.br

Enhancing the performance and safety of lithium-ion batteries is crucial for applications in electric vehicles and energy storage systems. One of the challenges in this field involves mitigating the reactivity of cathode materials with the electrolyte, which can lead to capacity loss and thermal instability. In this study, the impact of phase coexistence on ionic conductivity was investigated. The materials were synthesized via solid-state reaction and characterized using various analytical techniques, including X-ray diffraction, Raman spectroscopy, scanning electron microscopy, and impedance spectroscopy. X-ray diffraction (XRD) was fundamental in identifying and quantifying the crystalline phases present in the samples, serving as a key tool to verify the formation and stability of the targeted composite structures. The analysis of the diffraction patterns confirmed the coexistence of multiple crystalline phases and preserved structural order, which are essential parameters for interpreting the transport properties and ionic conduction mechanisms within the material. The synthesis process yielded phase fractions closely aligned with the targeted compositions. Conductivity measurements at different temperatures, along with activation enthalpies, indicated that composite materials exhibited superior conductivity and lower activation energy compared to single-phase counterparts. These improvements are attributed to chemical potential differences that induce defect formation, leading to enhanced ionic transport properties. The findings contribute to a deeper understanding of the mechanisms that govern conductivity improvements, with potential implications for optimizing battery electrode materials.

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**Keywords: Lithium-Ion Batteries; ionic conductivity; phase-coexistence; solid-state synthesis**

# *Microstructural and Thermal Characterization of $Li_{1.3}Al_{0.3}Ti_{1.7}(PO_4)_3$ Glass – ceramic with controlled disorder: Toward enhanced Li+ Mobility*

***A. Quispe[1], G. Garcá[1], F. Akhtar[2]***

[1]Department of Chemistry, Faculty of Pure and Natural Sciences, Universidad Mayor de San Andrés, UMSA, La Paz, Bolivia. [2]Division of Materials Science, Luleå University of Technology, 97187 Luleå, Sweden

alejandra.rossio.quispe.medina@associated.ltu.se

NASICON-type $Li_{1.3}Al_{0.3}Ti_{1.7}(PO_4)_3$ (LATP) is a promising solid electrolyte for lithium batteries due to its high thermal and chemical stability, although its relatively low ionic conductivity remains a challenge. [1] In this work, we address this limitation by introducing intrinsic structural defects to create disorder and promote Li+ diffusion. LATP powders were synthesized via a conventional solid-state route; intrinsic defects were then generated by varying the duration of high-energy ball milling. Milling times from 0 to 12 hours reduced average crystallite sizes from ~400 nm to ~40 nm and significantly increased dislocation density, as confirmed by X-ray diffraction. SEM revealed morphological refinement, while Raman spectroscopy and Fourier electron-density mapping provided insights into short-range order and potential $Li^+$ migration pathways. [2,3] Thermal analysis (DSC/TGA) demonstrated enhanced thermal stability and revealed slight transitions indicative of increased amorphous character.

Although ionic conductivity was not directly measured in this study, the structural and thermal analyses support the hypothesis that the introduction of controlled disorder and intrinsic defects may create more favorable conditions for lithium-ion transport. These findings contribute to a deeper understanding of the defect chemistry in LATP and suggest a potential route to improve its performance as a glass-ceramic electrolyte.

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**Keywords: mechanochemical; thermal analysis; structural disorders; glass-ceramic, NASICON**

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# Structural Properties and Exchange Extension as Functions of Chain Length for Layered Hexaniobate Intercalated with N,N'-Dialkylimidazolium

**A. Duarte[1,2], C. L. P. Oliveira[2], V. R. L. Constantino[1]**

[1]Instituto de Química da Universidade de São Paulo – IQUSP
[2]Instituto de Física da Universidade de São Paulo – IFUSP São Paulo-SP-Brazil.
alfredoduarte@usp.br

Potassium hexaniobate, $K_4Nb_6O_{17}$, is a highly crystalline, synthetic, inorganic layered material, whose structure belongs to the orthorhombic system and $P2_1nb$ space group, with unit cell dimensions (Å) *a* = 7,83, *b* = 33,21 and *c* = 6,46, comprising densely negative charged layers (25,3 Å$^2$/charge) orthogonally stacked along the *b* axis and composed of arrangements of bounded [$NbO_6$] octahedral units, containing $K^+$ cations in the interlayer region [1]. Two different interlayer regions are present, whereas that named **I** exhibits higher hydration and intercalation reactivity than **II**. To allow organophilization of layers' surfaces, intercalation reactions through exchange of $K^+$ by organic cations may be carried out, leading to increased basal spacing into those hybrid inorganic-organic particles [2]. In this work, a set of composites were obtained from $K_4Nb_6O_{17}$ intercalated with several different **N'-alkyl**-N-methyl-imidazolium cations (**R**-MIm$^+$; **R** = $H^+$, Benzyl or linear-$C_n$ : $2 \leq n_{even} \leq 16$) from R-MImCl aqueous solutions or molten salts media. The extension of intercalation and resulting materials' structural properties were evaluated as functions of R-MIm$^+$ alkyl chain lengths.

X ray diffraction (XRD), thermal (TGA-DSC-MS) and elemental (CHN) analysis data suggest that significant degree of intercalation only occurs when reactions were conducted in aqueous R-MIm$^+$ solutions. When varying R-MIm$^+$ sizes and ratios towards interlayer exchangeable $K^+$, beyond that significant degree of intercalation only occurs from alkyl chain ≥ octyl on, data also suggest that the increase in alkyl chain length promotes exchange of higher cation amounts comparing the same R-MIm$^+$/$K^+$ molar ratio used. XRD data showed a tendency for primary expression of a preferential phase in most materials, where a perpendicular monolayer of organic cations intercalated only into interlayer I is observed. Regardless, besides the monolayer, some products simultaneously presented other phases, which may be attributed to perpendicular bilayers and/or other kind of arrangements. Concerning that, higher perpendicular monolayer phase purity and crystallinity of the materials were achieved and less differences in their structure and composition were observed due to R-MIm$^+$/$K^+$ ratio increase.

Since the positive charge delocalization at the imidazolium cation head induces attenuation effects in their electrostatic attraction towards hexaniobate negative layers, while compared to the original $K^+$, it is suggested that hydrophobic interactions, due to alkyl chains, prevail as the driving forces of for imidazolium confinement between layers. Thermal stabilities of intercalated R-MIm$^+$ are around 200 °C, i.e. about 80 °C higher than samples intercalated with hexadecyltrimethylammonium – an usual surfactant for layered inorganic materials organophilization. Thermally stable layered hybrids are required for production of nanocomposites with organic polymers by melting processes.

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**Keywords: layered niobates; intercalation chemistry; ionic exchange; quaternary ammonium; hybrid materials.**

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# Screening of coformers for co-crystal formation of LassBio 1735 anhydrous

***C. C. S. Batista, F. R. Praxedes, F. F. Ferreira***

Centro de Ciências Naturais e Humanas (CCNH), Universidade Federal do ABC (UFABC), Av. dos Estados, 5001, Santo André, SP, 09280-560, Brazil e-mail: carin.cristina@ufabc.edu.br

The World Health Organization (WHO) classifies cancer as one of the leading causes of death worldwide. Scientists are committed to research focused on the ongoing treatment of various types of cancer.[1] In this context, the main objective of this work is to improve the performance of the drug candidate LASSBio-1735, both by developing new crystalline forms and by investigating their biological interactions. Our research group determined the crystal structure of LASSBio-1735, an N-acylhydrazone derivative that exhibited in vitro antiproliferative activity against the cell lines HL-60 (human leukemia), SF-295 (human glioblastoma), MDA-MB-435 (melanoma), and HCT-8 (ileocecal adenocarcinoma).[2] This molecule served as a prototype for the synthesis and investigation of the physicochemical properties of new crystalline forms, including salts, hydrates, solvates, and/or cocrystals. Initially, the selection of potential coformers was based on theoretical studies from the Mercury® program. However, the selected classical candidates did not form cocrystals. Therefore, we chose to study other candidates, such as βcyclodextin, PEG (polyethylene glycol), and folic acid, taking into account qualities such as functional groups, water solubility, and photosensitivity. The methodology used to obtain the new crystalline forms involved mechanochemistry, using a ball mill and manual grinding in a mortar and pestle, varying the concentrations of the compounds.[3] We studied the interaction of LASSBio-1735 with the three candidates and analyzed the formation of potential cocrystals by powder X-ray diffraction (PXRD) and Fourier transform infrared spectroscopy (FTIR). Additional analyses are ongoing.

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**Keywords: LASSBio-1735; Cocrystals; X-ray diffraction (XRD); Fourier transform infrared spectroscopy (FTIR); Mechanochemistry;**

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# *Retention of the cubic phase in $Gd_2O_3$ co-doped $Sc_2O_3$-stabilized $ZrO_2$ ceramics*

***V. C. Contini[1], A. L. Larralde[2], G. Suarez[3], D. G. Lamas[4]***

[1]Instituto de Geología y Recursos Naturales (INGeReN), UNLaR, La Rioja Capital, Provincia de La Rioja, Argentina. [2]CONICET/Departamento de Almacenamiento de la Energía, Subgerencia de Energía y Movilidad, Gerencia de Desarrollo Tecnológico e Innovación, INTI, San Martín, Provincia de Buenos Aires, Argentina.[3]Centro de Tecnología de Recursos Minerales y Cerámica (CETMIC), CONICET-UNLP-CIC, M.B. Gonnet, Provincia de Buenos Aires, Argentina.

[4]Laboratorio de Cristalografía Aplicada, Instituto de Tecnologías Emergentes y Ciencias Aplicadas (ITECA), CONICET-UNSAM, San Martín, Provincia de Buenos Aires, Argentina.

Email: dlamas@unsam.edu.ar

Scandia-stabilized zirconia (ScSZ) ceramics, obtained by doping $ZrO_2$ with $ScO_3$, exhibits the highest ionic conductivity among zirconia-based electrolytes at temperatures above 600 °C. For this reason, ScSZ ceramics are considered as promising candidates for solid electrolytes in solidoxide cells. The optimum ionic conductivity corresponds to $Sc_2O_3$ contents between 9 and 11 mol%. Unfortunately, a rhombohedral phase (known as the -phase) of poor electrical properties, which has not been observed in other zirconia-based materials, exists at low-temperatures for this compositional range. The β-phase transforms to the high-conductivity cubic one on heating at temperatures above 600°C, retransforming on cooling at about 500°C. This transition is undesirable for applications because the volume change involved can cause the deterioration of the electrolyte. Therefore, from a technological point of view, it is important to avoid the  ↔ c transition without an important loss in the ionic conductivity. For this purpose, the introduction of co-dopants that promote the retention of the c-phase, such as $Y_2O_3$ or $CeO_2$, has been proposed.

Among the many oxides reported in the literature for stabilizing the cubic phase in ScSZ ceramics, $Gd_2O_3$ has been scarcely studied. Besides, the influence of the fabrication process has not been evaluated in detail. Most of the authors only considered a conventional solid-state reaction process under limited processing conditions. For these reasons, this work evaluates $Gd_2O_3$-codoped ScSZ ceramics with composition of $ZrO_2$-10 mol%$Sc_2O_3$-1 mol% $Gd_2O_3$ prepared via mechanochemical-assisted solid-state reaction. The influence of sintering temperature (ranging from 1100 to 1500 °C) and milling time during high-energy milling on the stabilization of the cubic polymorph was systematically studied by conventional (laboratory) X-ray powder diffraction. Our study demonstrated that the content of the cubic phase increased with increasing milling time or sintering temperature and the complete retention of this phase was reached for milling times of at least 30 min and sintering temperatures of 1400 °C or higher. The properties of other codopants, such as $Y_2O_3$ and $Sm_2O_3$, were also analyzed for the purpose of comparison.

**Keywords: ScSZ; Zirconia ceramics; Solid electrolytes**

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# *Evolution of crystalline phases in Layered Double Hydroxide precursors and derived catalysts containing Fe, Al, Zn and Mg*

***G. Nunes[1], F. Zotin[1], L. Palacio[1]***

[1]Universidade do Estado do Rio de Janeiro, RJ, Brazil nunes.gabriel@graduacao.uerj.br

Propylene is the second most important petrochemical product and is used to produce many valuable end products, including fibers, plastics, rubbers, polymers, and paints. Its growing demand is not met by current industrial processes. Oxidative dehydrogenation of propane assisted by $CO_2$ ($CO_2$-ODHP) is a promising alternative route. $CO_2$ offers two major advantages: it is a mild oxidant, which prevents complete oxidation of both propane and propylene, and it is a greenhouse gas that can be utilized from fixed-source emissions [1]. We obtained promising results for this reaction using catalysts based on the Zn-Al-Fe and Mg-Al-Fe systems, derived from layered double hydroxides (LDH).

LDHs consist of layers of positively charged mixed hydroxides, compensated by interlayer anions and accompanied by water [2]. Oxides derived from LDHs exhibit properties of interest in the field of catalysis, such as high thermal stability and moderately high surface area [3]. In this study, LDH precursors were synthesized via a pH-controlled co-precipitation method, using molar ratios of y = Fe/(Fe + Al) = 0.5 and x = (Fe + Al)/(MII + Fe + Al) = 0.25, where MII = Zn or Mg. The precursors were calcined in a furnace with at 20 °C/min up to 600 °C for 3 h to obtain the catalysts. XRD analysis revealed that pure LDH phases were obtained for both Mg and Zn precursors, with diffraction peaks matching the hydrotalcite pattern (PDF 00-048-1021). FTIR, ICP, and TGA complemented precursor characterization. FTIR confirmed the presence of carbonate species within the interlayer regions. ICP results showed that the compositions of Zn, Fe, Al, and Mg were approximately equal to the intended ratios. TGA analysis showed two main stages of mass loss: the first associated with interlayer water removal, and the second with the decomposition of carbonates and hydroxyl groups, stabilizing after 600 °C.

In-situ XRD results showed the collapse of the hydrotalcite structure at 200 °C and the crystallization of oxide phases (ZnO or MgO) at 400 °C. XRD of the final catalysts identified $Zn(FeO_2)_2$ (PDF 01-079-1500) and ZnO (PDF 00-036-1451) phases in the Zn-Al-Fe sample, and $Mg_{0.963}OFe_{0.037}$ (PDF 01-076-3013) in the Mg-Al-Fe sample. Post-reaction XRD of spent catalysts indicated the presence of magnetite in the Zn-Al-Fe catalyst.

Catalysts were further characterized using XPS and SEM. The Fe2p region of XPS analysis revealed that all surface iron was in the $Fe^{3+}$ oxidation state. The O1s region showed a predominance of M–O bonds over M–OH and C=O species. SEM-FEG analysis showed that the Zn-Al-Fe catalyst presented a more defined morphology than the Mg-Al-Fe catalyst.

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**Keywords: Layered Double Hydroxide; Mg-Fe-Al, Zn-Fe-Al, Oxidative Dehydrogenation of propane with $CO_2$**

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# *Tailoring $TiO_2$ Crystallinity through Alcohol Selection in the Pechini Route*

***P. Aoyague[1], E. A. Almeida[1], J. L. Andade[1] and G. T. Tractz[1]***

[1]Universidade Tecnológica Federal do Paraná, Campus Campo Mourão- PR, Brazil
gideatractz@utfpr.edu.br

Titanium dioxide ($TiO_2$) is a widely used oxide material due to its stability, low cost, and photocatalytic properties. The Pechini method offers a simple and effective route for synthesizing nanostructured $TiO_2$ with controlled phase composition, where parameters like the choice of alcohol influence the final crystalline structure [1]. The Pechini method is a polymeric precursor route that involves the formation of metal–chelate complexes with citric acid and their subsequent polyesterification with an alcohol group. This process ensures homogeneous mixing at the molecular level, leading to uniform oxide materials upon calcination. Due to the calcination step, the Pechini method may lead to the formation of particle agglomerates, as organic removal and crystallization promote particle coalescence [2].

Titanium dioxide ($TiO_2$) powders were synthesized via the Pechini method using ethylene glycol and Tween 80 as esterifying agents. The resin was calcinated at 550 °C for 6 hours, with the powders characterized by XRD measurement

XRD and Rietveld refinement revealed anatase–rutile mixtures, with phase ratios strongly influenced by the alcohol used [2]. Ethylene glycol favored rutile (56.5%), while Tween 80 promoted anatase (74.3%). These results highlight how precursor selection affects $TiO_2$ crystallinity through steric and chemical interactions.

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**Keywords: nanostructured oxides; anatase; rutile; alcohol precursor**

# Exploring Metal Variation in IR-MOF-74 III: Synthesis, structural and optical characterizations

***J. A. de Oliveira Neto[1], B. T. D. Procópio[2], W. X. C. Oliveira[2], P. H. M. Andrade[1], C. B. Pinheiro[1]***

[1]Department of Physics, Federal University of Minas Gerais, Belo Horizonte, Brazil.
[2]Department of Chemistry, Federal University of Minas Gerais, Belo Horizonte,Brazil. joao2712@ufmg.br

Metal-organic frameworks (MOFs) are porous materials that have emerged as promising solutions for addressing various environmental challenges [1]. Their unique architecture constructed from one or more metal ions coordinated with diverse organic linkers allows for precise control over pore size, surface chemistry, and other key properties [2]. One of the defining features of MOFs is their exceptionally high surface area, which enhances their ability to interact with and capture specific molecules. As a result, MOFs have shown great potential for selective gas capture and sensing applications [3].

This work focuses on synthesizing and characterizing isoreticular MOF-74 (IR-MOF-74) using various divalent metals via the solvothermal method. Deng et al. first reported the synthesis of a large - pore variant of this framework by incorporating aromatic rings into the 2,5dihydroxyterephthalic acid ligand with $Mg^{2+}$, preserving the original topology while expanding the pore size from ~11Å in MOF-74 to ~98Å in IR-MOF-74 XI [4]. Based on this protocol, the IRMOF-74 III was synthesized using the LINK-III ligand obtained via the Suzuki–Miyaura crosscoupling approach and precursor of $Zn^{2+}$, $Co^{2+}$, $Ni^{2+}$, and $Mn^{2+}$ cations. Characterization techniques, including XRD, SEM, UV-Vis, FT-IR, Raman spectroscopy, and thermal analyses, were used to enhance the understanding of these new structures. The results demonstrated that similar to MOF-74 [5], varying the coordinating metal in IR-MOF-74 III preserves the framework topology while potentially influencing its functional properties.

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**Keywords: Crystallograph, X- ray diffraction, MOFs, characterization materials**

# Synthesis and characterization of polycrystalline materials with ionic conductivity and cationic disorder for lithium-ion battery applications

***M. E. C. Diniz*[1]*, A. S. de Menezes*[2]*, R. Mouta*[3]*, C. C. dos Santos*[4]**

[1]Bacharelado em Física, Universidade Federal do Maranhão - UFMA, São Luís, Maranhão, Brazil

[2]Departamento de Física, Universidade Federal do Maranhão - UFMA, São Luís, Maranhão, Brazil

[3,4] Departamento de Física, Universidade Federal do Ceará - UFC, Fortaleza, Ceará, Brazil

[1]mec.diniz@discente.ufma.br, [2]alan.menezes@ufma.br, [3]rodolpho@fisica.ufc.br, [4]clenilton.cs@ufma.br

The demand for rechargeable lithium-ion batteries (LIBs) is growing exponentially, driven by the expansion of electric vehicles and mobile electronic devices, fostering a market that exceeds USD 120 billion. A critical challenge lies in the flammability of conventional liquid electrolytes, which undermines thermal safety. Ceramic solid electrolytes have emerged as a promising alternative to overcome this limitation, offering enhanced safety alongside the potential for higher energy density.This study investigates novel materials for application as solid electrolytes, employing a partial ionic substitution strategy to increase cationic disorder in polycrystalline materials, with the goal of enhancing $Li^+$ mobility and, consequently, ionic conductivity. The crystals were synthesized via solid-state reaction at temperatures ranging from 800 to 950 °C. Structural and phase characterization was performed by room-temperature X-ray diffraction (XRD), while substitution-induced changes were probed using Raman spectroscopy. XRD patterns of samples synthesized at lower temperatures exhibited broader, more Lorentzian peak shapes and decreased intensity in reflections associated with cation ordering, indicating a significant increase in cationic disorder. Raman spectra revealed broadening of optical modes, further corroborating the enhanced structural disorder induced by partial substitution. Preliminary impedance spectroscopy results demonstrated that this increased disorder led to a substantial improvement in $Li^+$ conductivity, with enhancements of several orders of magnitude compared to the pristine material. Future work will involve Pair Distribution Function (PDF) analyses to investigate potential short-range cation ordering, which is expected to play a critical role in $Li^+$ conductivity in disordered rocksalt-structured oxides and oxyfluorides.

Keywords: Solid electrolyte; Lithium-ion batteries; X-ray diffraction; Raman spectroscopy; Cationic disorder.

# *Synthesis and characterization of $Nd^{3+}$ doped nickel ferrites via the Proteic sol-gel method*

**M. R. Araujo[1,*], F. W. C. Vasconcelos[1], L. Q. Rodrigues[1], Y. Martinez-Camejo[2], J. S. Andrade[3], E. F. Chagas[4], J. M. Sasaki[2], G. F. Guimarães[3]**

[1]Federal University of Ceará, Postgraduate Program in Materials Science and Engineering, Fortaleza - Campus Universitario do Pici - Bloco 729, 60440-554, Ceará, Brazil,

[2]Federal University of Ceará, Physics Department, Rua Prof. Armando Farias, s/n, Campus do Pici, Fortaleza, 60440-760, Ceará, Brazil,

[3]Federal Institute of Education, Science, and Technology of Ceará, Postgraduate Program in Telecommunications Engineering, Photonics Laboratory, Fortaleza - Av. Treze de Maio, 2081 – Benfica 60040-531, Ceará, Brazil

[4]Federal University of Mato Grosso, Institute of Physics, Avenida Fernando Corrêa da Costa, s/n, Cidade Universitária – Bloco F, Cuiabá, 78060-900, Mato Grosso, Brazil,

*matheus.rodrigues.araujo@alu.ufc.br

Rare earth elements have gained prominence in science and technology due to their magnetic, optical, and electronic properties, playing a key role in applications such as high-performance magnets, LEDs, sensors, catalysts, and biomedical contrast agents. In this work, we investigate the synthesis and characterization of neodymium ($Nd^{3+}$) doped nickel ferrites ($NiFe_2O_4$), produced using the protein-based sol-gel method developed and patented by our group, which employs gelatin as an organic precursor. This method offers advantages such as low cost, environmental sustainability, fast processing, and precise control over nanoparticle size, making it effective for doping studies in spinel-type ferrite systems. $Nd^{3+}$ was chosen as a dopant due to its ionic radius, electronic configuration, and magnetic moment, which influence its occupation of tetrahedral or octahedral sites within the spinel structure, allowing the investigation of the relationship between cation distribution, inversion degree, and magnetic properties. Samples with different $Nd^{3+}$ concentrations were prepared to analyze the structural and magnetic evolution of the material.

Structural characterization was performed using X-ray diffraction (XRD), confirming the formation of single-phase structures and efficient incorporation of $Nd^{3+}$ without secondary phases. Peak shifts and a reduction in crystallite size were observed, calculated using the Scherrer and WilliamsonHall equations, indicating progressive lattice distortions due to doping. X-ray fluorescence (XRF) analyses corroborated the presence and quantification of the dopant in the samples. Magnetic properties were investigated using vibrating sample magnetometry (VSM), revealing significant variations in the magnetic parameters as a function of $Nd^{3+}$ content, confirming that rare-earth doping considerably influences the magnetic behavior of ferrites. Changes in saturation magnetization and coercivity were observed, indicating the material's potential for applications in magnetic sensors and magneto-optical devices.

The results demonstrate that the protein-based sol-gel method is an effective route for the controlled synthesis of $Nd^{3+}$ doped ferrite nanoparticles, enabling fine-tuning of their structural and magnetic properties through doping, with perspectives for advanced technological applications. This study contributes to understanding the effects of rare earths on spinel-type ferrites and the development of functional materials with optimized performance for magnetic and magneto-optical devices.

**Keywords: nickel ferrite, neodymium, proteic sol-gel, magnetic nanoparticles, structural and magnetic properties**

# Comparative study of multicomponent system of RS-ofloxacin and levofloxacin toward diastereomeric salt resolution

***P. S. Carvalho Jr[1*], L. F. Diniz[2], C. Fernandes[2] and R. Diniz[3]***

[1] Institute of Physics, Federal University of Mato Grosso do Sul, 79070-900, Campo Grande, MS, Brazil. 2 Faculty of Pharmacy, Federal University of Minas Gerais, 31270-901, Belo Horizonte, Brazil. [3] Departamento de Química, Instituto de Ciências Exatas (ICEx), Universidade Federal de Minas Gerais, 31270-901-Belo Horizonte, MG, Brazil.

*e-mail: paulo.sousa@ufms.br

Identifying effective resolving agents is essential for the efficient separation of enantiomers. Ofloxacin (Ofx), a widely used chiral fluoroquinolone antibiotic, is marketed as a racemic mixture (RS-Ofx). To explore potential resolution strategies, we screened combinations of RS-Ofx and pure Levofloxacin (Levo) with various chiral acids, including (D)- and (L)-mandelic acids, (L)- and (D)-ditoluyltartaric acids, (L)-dibenzoyltartaric acid, and (L)-bromomethylbutyric acid. These experiments have yielded a range of distinct crystalline phases. Single-crystal and poweder X-ray diffraction, thermal analysis (DSC/TGA), and chiral chromatography were employed to characterize these forms. Notably, Ofx salts with formed with (L)-ditoluyltartaric acid and (L)dibenzoyltartaric acid, exhibited solid solution behavior. Additionally, the Levo-(L)bromomethylbutyrate salt was identified as a salt-cocrystal, in which the anionic component plays a critical role in dictating the supramolecular architecture of the ionic pairs. Successful diastereomeric resolution was achieved with both enantiomers of mandelic acid. A comparative structural analysis of racemic and enantiomerically pure salts highlights characteristics associated to chiral resolution. These findings provide valuable insights into the structural basis of chiral recognition in Ofx systems and lay the groundwork for the design of targeted salt and cocrystal formation strategies for this and other chiral pharmaceutical compounds.

**Keywords: Chiral resolution, Crystallization, Ofloxacin, crystal engineering**

# *Structural Characterization of $NdFeO_3$ under High Pressures: Investigation of Short-Range Order via Rietveld Refinement and Pair Distribution Function*

***Q. H. F. Rebelo[1], T. Puccinelli[2], D. I. S. Lacerda[3], C. M.Ruzo[4], C. C.Pinto[5], D. M. Trichês[3], S. M. de Souza[3]***

[1]Institute of Engineering and Geosciences, Federal University of Western Pará (UFOPA), Santarém, PA, Brazil. [2]Institute of Physics, University of São Paulo (USP), São Paulo, SP, Brazil. [3]Department of Materials Physics, Federal University of Amazonas (UFAM), Manaus, AM, Brazil. [4]Postgraduate Program in Physics (PPGFIS), Federal University of Amazonas - UFAM, Manaus, AM, Brazil. [5]Federal Institute of Amazonas (IFAM), Presidente Figueiredo, AM, Brazil.

Email of communicating author: aquerem@gmail.com

The study of ferroelectric materials under high-pressure conditions has proven to be a promising approach for uncovering structural and functional properties that remain hidden under ambient conditions. Among these materials, $ABO_3$-type perovskites stand out due to their broad applicability in advanced technologies, such as ultrasonic transducers, actuators, and information storage devices. The application of hydrostatic pressure enables the investigation of crystalline phase stability, the identification of subtle structural transitions, and access to regimes of piezoelectric anisotropy that are not observable under normal conditions.

In this context, $NdFeO_3$ particles were synthesized via high-energy ball milling followed by thermal treatment [1], aiming to investigate the effect of pressures up to 45 GPa on their structure. A diamond anvil cell was assembled at room temperature using neon gas as the pressuretransmitting medium to ensure hydrostatic compression. The experiments were carried out using synchrotron radiation at the EMA beamline at the Sirius facility, and pressure measurements were determined based on the ruby fluorescence shift. X-ray diffraction (XRD) patterns, refined using the Rietveld method, show that increasing pressure compresses the crystal structure, leading to a reduction in interatomic distances and a shift of the diffraction peaks toward higher angles. The broadening of the peaks also suggests a reduction in grain size and/or crystalline domain size. The experimental results show excellent agreement with available theoretical data.

Additionally, the Cowley-Warren parameter, associated with the chemical short-range order within the first coordination sphere, was determined. This parameter was obtained from the pair distribution function, derived by normalizing the XRD patterns using the analytical function proposed by Cromer and Mann and Rietveld refinements [2,3].

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**Keywords: perovskites; X-ray diffraction; Rietveld refinement; pair distribution function.**

# *Study of Lanthanum-Doped Cerium Oxide via Combustion Synthesis for Use in Solid Oxide Cells*

**S. Pioli[1], R. Moras[1], L. Suescun[1]**

[1]Cryssmat-Lab, Faculty of Chemistry, UdelaR, Montevideo, Uruguay spioli@fq.edu.uy

Solid oxide electrolysis cells (SOECs) offer a promising route for green hydrogen production. They consist of a solid electrolyte that conducts oxygen ions ($O^{2-}$) or protons ($H^+$), flanked by two solid electrodes. These electrodes, which are crucial for facilitating the electro-decomposition of water into $H_2$ (at the fuel electrode) and $O_2$ (at the oxygen electrode), require mixed ionicelectronic conductivity and catalytic activity.

At Cryssmat-Lab, we have implemented a combustion synthesis method that allows for a reduction in the preparation temperature of nanostructured fluorite- and perovskite-type materials. This improves the microstructure of electrodes and electrolytes in all types of solid oxide cells (SOCs).

During the one-step combustion synthesis of biphasic oxygen electrodes $La_{0.6}Sr_{0.4}Fe_{0.8}Cu_{0.2}O_{3-\delta}$ with $CeO_2$ (LSFCu-$CeO_2$), partial doping of the $CeO_2$ phase with La has been detected, modifying the structure and properties of this component [1], resulting in a synergistic effect that benefits the material's activity. There is a report on the phase behavior of La-doped $CeO_2$ prepared by solid-state synthesis [2], but there is no literature available on methods to estimate this behavior for $Ce_{1-x}La_xO_{2-x}/_2$ nanoparticles like those obtained in our work. We have prepared $Ce_{1-x}La_xO_2$ (x = 0.05, 0.1, 0.15, and 0.2) under the same synthetic conditions used for preparing the LSFCu-$CeO_2$ oxygen electrode.

We will present the results of the structural characterization by powder X-ray diffraction and Rietveld analysis of the materials obtained. These results have allowed us to produce a calibration curve to determine the level of La doping in $CeO_2$ within the LSFCu-$CeO_2$ composites with different $CeO_2$ proportions, and thereby estimate the La vacancies in the perovskite phase $La_{0.6}Sr_{0.4}Fe_{0.8}Cu_{0.2}O_{3-\delta}$.

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**Keywords: LAPDIC; GREEN HYDROGEN; PEROVSKITES**

## *Controlled Growth and Characterization of MOF-74 Thin Films on Metallic Substrates for Electrochemical Applications*

***V. E. G. Gomes, J. A P. de Figueiredo, P. H. M. Andrade, C. B. Pinheiro***

Universidade Federal De Minas Gerais Belo Horizonte, Minas Gerais victore@ufmg.br, cbpinheiro@ufmg.br.

The progress of society is intrinsically linked to the development of new materials that meet the growing demands of advanced technologies, particularly in energy storage. In this context, Metal–Organic Frameworks (MOFs) have attracted great attention due to their high porosity, large surface areas, crystallinity, and structural versatility, enabling applications in gas storage, catalysis, sensing, drug delivery, and electrochemical devices.

Among the 12,000+ MOF structures in the Cambridge Structural Database, the MOF-74 family stands out for its one-dimensional pore architecture (1.5–9.0 nm) and secondary building units composed of metals such as Zn, Cu, Ni, Co, Fe, Mn, and Mg. These features grant MOF-74 high stability and suitability for fuel cells and electrochemical applications.

In this project, we propose the synthesis and characterization of MOF-74 thin films with metals Me = Ni, Co, Zn, and Cu, using the linker 3,3''-dihydroxyterphthalic acid. Thin film growth is achieved through a controlled dip-coating process, in which metallic substrates are sequentially immersed in linker and metal ion solutions. This approach allows room-temperature growth with precise control of film thickness.

We adapted this strategy to achieve controlled growth of Zn-MOF-74 films and subsequent deposition of Co-MOF layers directly onto Zn and Co metallic substrates. The growth process occurs simply by immersing the substrates in a resting solution containing the organic linker, enabling bottom-up fabrication of functional thin films for electrochemical devices.

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**Keywords: Metal–Organic Frameworks (MOFs) thin films, energy storage, electrochemical devices, fuel cells, dip-coating method, porosity, surface area, crystallinity, cobalt MOF-74, substrate immersion, controlled growth**

# *Synthesis and characterization of $La_2(WO_4)_3$ ceramic obtained via coprecipitation method.*

***A. L. Pinheiro Neto[1,2], T. E. R. Fiuza[2], T. M. Perfecto[2], E. R. Leite[2,3], I. C. Nogueira[1].***

[1]Federal University of Amazonas, Manaus, AM, Brazil, [2]Brazilian Center for Research in Energy and Materials, Cmapinas, SP, Brazil, [3]Feferal University of São Carlos (UFSCar), São Carlos, SP, Brazil.
e–mail: antoniolopes@ufam.edu.br

Lanthanum tungstate, $La_2(WO_4)_3$, is a metal oxide from the $A_2(MO_4)_3$ family, which has gained significant attention due to its physical and chemical properties, making it ideal for various technological applications, such as photocatalytic activity [1], electrocatalysts in electrochemical sensors [2], and others. However, a deeper understanding of this material is necessary, as there is limited detailed information about its structure, emphasizing the need for further studies to optimize its use in advanced ceramics.

In this study, $La_2(WO_4)_3$ was synthesized using the coprecipitation method, a simple and environmentally friendly approach. This method uses water as the solvent and requires only low to moderate temperatures, reducing the need for toxic solvents and minimizing energy consumption. The thermal behavior of the amorphous material showed an exothermic peak corresponding to its crystallization at 587 °C. Based on this, thermal treatments were performed at 550, 600, and 800 °C/2h. The material remained amorphous at 550 °C/2h, while at 600 and 800 °C/2h, the crystalline phase of $La_2(WO_4)_3$ was identified. Rietveld refinement confirmed the monoclinic structure with space group C2/c (Nº 15) [3].

Raman spectroscopy showed that $La_2(WO_4)_3$ transitions from an amorphous state to a crystalline material as the temperature increases. At 600 °C, sharp Raman bands indicating crystallinity appeared. At 800 °C, the material retained the crystalline structure with more defined bands, signaling better structural order. The vibrational modes of the $W^{I}O_4$ and $W^{II}O_4$ units identified included: symmetric stretching ($A_1(v_1)$, 900–1000 cm⁻¹), antisymmetric stretching ($F_2(v_3)$, 700– 850 cm⁻¹), and bending modes ($E(v_2)$ and $F_2(v_4)$, 300–550 cm⁻¹). Transmission Electron Microscopy (TEM) images revealed agglomerated nanoparticles with good morphological homogeneity and a pseudo-spherical shape. Agglomeration is characteristic of the synthesis method used.

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**Keywords: Lanthanum tungstate; coprecipitation; monoclinic.**

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# *Crystal Structure by X-ray Powder Diffraction and Hirshfeld Surface analysis for polymorph I of the Hydrocodone Bitartrate*

***B. S. Santos García[1], C. G. Albarracín Caballero[1], R. A. Toro Hernández[1], J. A. Henao Martínez[1]***

[1]Grupo de Investigación en Química Estructural (GIQUE), Universidad Industrial de Santander, Bucaramanga, Colombia.

Brayan2202858@correo.uis.edu.co, jahenaop@uis.edu.co

Prescription opioid abuse has increased at an alarming rate and is now considered an epidemic [1]. This crisis underscores the urgent need for strategies to mitigate opioid misuse, including the development of safer pharmaceutical formulations. Understanding the solid-state properties of active pharmaceutical ingredients (APIs) is critical, as they influence manufacturability, stability, and bioavailability, and can be key to designing formulations that deter abuse. This research focuses on the structural characterization of Hydrocodone Bitartrate ($HCD^{+}$•$Tar^{-}$) using powder Xray diffraction. Crystallographic information on this active ingredient (API) is scarce. With this study, we aim to provide structural data relevant to quality control, formulation optimization, and other applications. Initial characterization involved Infrared spectroscopy (ATR FT-IR), confirming characteristic functional groups of the molecule. Thermal analysis (TGA-DSC) revealed a weight loss consistent with two water molecules, indicating the API is a dihydrate, and identified a melting point of 116.31 °C, corresponding to polymorph I as reported in patent US 7,625,918 B2 [2]. The powder diffraction pattern matches the unindexed and incomplete pattern of polymorph I reported in the PDF-5+ (PDF: 00-064-1551). Indexing with the DICVOL program, implemented in the PreDICT graphical interface, indicates that the API crystallizes in a unit cell orthorhombic with parameters: ***a*** = 34.141(14) Å, ***b*** = 19.420(39) Å, ***c*** = 7.0855(17) Å, *V*=4698(2) Å³, and Z=8 with Figures of Merit of $M_{(20)}$=31.5 and $F_{(20)}$=96.4. Reflection condition analysis using EXPO suggested $P2_12_12_1$ as a possible space group. The crystal structure is dominated by hydrogen bonds. The hydrocodone cation connects to the tartrate via the N-H···O contact. The $HCD^{+}$ molecules extend in the form of helical chains along the b axis, alternating with motifs of the $Tar^{-}$ ion. Water molecules connect the $HCD^{+}$ and $Tar^{-}$ chains throughout the crystal lattice.

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**Keywords: hydrocodone bitartrate; powder X-ray diffraction; crystal structure; opioid; polymorphism**

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# *Structural phase transitions at high-temperature in double perovskite $Sr_2GdRuO_6$*

***D. A. Landinez Tellez[1], J. Roa-Rojas[1], C. A. Triana[1]***

[1]Grupo de Fisica de Nuevos Materiales, Departamento de Fisica, Universidad Nacional de Colombia, Bogota, Colombia.

Email: dalandinezt@unal.edu.co

The crystal structure evolution of the $Sr_2GdRuO_6$ complex perovskite at high temperature has been investigated over a wide temperature range between 298K ≤ T ≤1273 K. Powder X-ray diffraction measurements at room temperature and Rietveld analysis show that this compounds crystallizes in a monoclinic perovskite-type structure with $P2_1/n$ (#14) space group and the 1:1 ordered arrangement of $Ru^{5+}$ and $Gd^{3+}$ cations over the six-coordinate M sites, with lattice parameters a = 5.81032(8) Å, b = 5.82341(4) Å, c = 8.21939(7) Å, V = 278.11(6) $Å^3$ and angle β = 90.311(2)º. The high-temperature analysis shows that this material suffers a two-phase transition. At 373 K it adopts a monoclinic perovskite structure with *I*2/*m* space group, and lattice parameters a = 5.81383(2) Å, b = 5.82526(4) Å, c = 8.22486(1) Å, V =2 78.56(2) $Å^3$ and angle β = 90.28(2)º. Above 773 K, it suffers a phase transition from monoclinic *I*2/*m* to tetragonal *I*4/*m*, with lattice parameters a = 5.84779(1) Å, c = 8.27261(1) Å, V = 282.89(5) $Å^3$ and angle β = 90.02(9)º. The high-temperature phase transition from monoclinic *I*2/*m* to tetragonal *I*4/*m* is characterized by strongly anisotropic displacements of the anions.

**Keywords: Perovskite, phase transition**

# Investigation of the Structural Feature of Hierarchical Hydrogels Derived from the Crystallization of Urea Adducts with Cationic Surfactants

***E. R. Peçanha[1,2], F. F. Ferreira[2], E. Sabadini[1]***

[1]State University of Campinas, Department of Physical-Chemistry, Institute of Chemistry, Cidade Universitária Zeferino Vaz - Barão Geraldo, CEP: 13083-970, Campinas, SP, Brazil.
[2] Federal University of ABC – Center for Natural and Human Sciences, Avenida dos Estados 5001, Bangu, CEP: 09210-580, Santo André, SP, Brazil.
Email: elaynne.rp@gmail.com

Urea adducts are aperiodic organic crystals formed by the helical organization of urea molecules around a long-chain hydrocarbon [1]. Recently, a hydrogel was discovered that results from the inclusion of cationic surfactant molecules within urea channels [2]. Subsequently, the universality of this system across different classes of surfactants was demonstrated [3]. In conventional urea inclusion compounds, there is an infinite stacking of hydrocarbons along the c-axis. When the guest molecule is a surfactant, stacking in the –chain-head-chain-head-····-chain-head– direction is impeded by the bulky size of the surfactant head, leading to steric hindrance that restricts adduct growth. Crystalline urea is confined to the surfactant tails, and lateral stacking of the inclusion complex results in a lamellar layer comprising a surfactant bilayer. Additionally, the nature of the surfactant head group permits water molecules to occupy the space between polar sites, forming an aqueous layer in the presence of counterions and potential urea molecules that do not crystallize.

In this study, the mesoscale organization of urea in the presence of hexadecyltrimethylammonium bromide (CTAB) was investigated using small-angle X-ray scattering (SAXS). For the first time, Rietveld refinement was applied to urea inclusion compounds containing surfactants. We report here a case in which it was necessary to refine contributions related to crystallite size and microstrain anisotropy. The optimal urea:CTAB ratio and the corresponding lattice parameters were determined for various ratios. In all cases, the urea adduct lattice conformed to the hexagonal space group $P6_122$.

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**Keywords: Urea adduct; Hierarchical organization; Lamellar hydrogel; Rietveld refinement.**

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# *Structural determination of a novel anti-inflammatory N-methyl-Nacylhydrazonic derivative*

***I. T. S. Bastos[1], F. N. Costa[2], A. E. Kümmerle[3], E. J. Barreiro†[4], F. F. Ferreira[2], R. C. Barroso[5]***

[1]Departament of Physics, State University of Feira de Santana, 44036-900, Feira de Santana, BA, Brazil, [2]Center for Natural and Human Sciences, Federal University of ABC, Santo André 09210-580, SP, Brazil, [3]Department of Chemistry, Federal Rural University of Rio de Janeiro, Seropédica,Brazil, [4]Laboratório de Avaliação e Síntese de Substâncias Bioativas (LASSBio), Instituto de Ciências Biomédicas, Universidade Federal do Rio de Janeiro, 21941-902, Rio de Janeiro, RJ, Brazil, [5]LabFisMed, State University of Rio de Janeiro, Physics Institute, Rio de Janeiro 20550-900, RJ, Brazil itsbastos@uefs.br

Studies leading to a greater comprehension of biologically active compounds in the solid state play a key role in the synthesis of new drugs. As important as the design and synthesis of new drugs is their structural characterization; exploring the arrangements of molecules in crystals provides a path to the design of new compounds and to the elucidation of processes in which a molecule recognizes a particular biological target [1–2]. In this work, we determined the crystal structure of LASSBio-1366 with X-ray powder diffraction data. This compound was planned and synthesized in the Laboratory of Evaluation and Synthesis of Bioactive Substances – LASSBio® of the Federal University do Rio de Janeiro (UFRJ) as part of a research program to develop a series of compounds with anti-inflammatory properties [2]. LASSBio-1366 is a *N*-methyl-*N*acylhydrazonic derivative inhibitor of PDE4 and TNF-α in vitro and also active in LPS-induced inflammation models in vivo, thus it can be highlighted as anti-inflammatory prototype compound inhibitor of PDE4 [2]. The crystal structure determination procedure of the LASSBio-1366 was performed both by indexing and decomposing the powder pattern using the *Topas Academic v.5.0* software program [3] via a Pawley fit [4] and the space group was determined as being *Pbca*. The cell volume found in the indexing procedure suggested eight molecules per unit cell (Z = 8) and one molecule in the asymmetric unit (*Z´* = 1). Subsequently, the Rietveld refinement [5] of the final crystal structure was conducted using the *Topas Academic v.5.0* software program. The Rietveld method was used to refine the crystal structure and the goodness-of-fit indicator as well as R-factors were $\chi^2$ = 1.516, $R_{Bragg}$ = 1.974%, $R_{wp}$ = 2.475% and $R_{exp}$= 1.632%. The crystal structure of LASSBio-1366 allowed us to show structural aspects of the composite solid phase allowing the characterization of the relative configuration *E* of the imine double bond.

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**Keywords: structure determination; *N*-methyl-*N*-acylhydrazonic; phosphodiesterase-4 Inhibitor**

# Coordination Polymers Based on Aliphatic Organic Ligands: Self-Assembly Synthesis and Structural Characterization

**J. P. Silva[1], C. H. Franco[2], G. R. Castro[1], F. F. Ferreira[1]**

[1]Center for Natural and Human Sciences (CCNH), Federal University of ABC (UFABC),
[2]MINDlab: Molecular Design & Innovation Laboratory, Centro de Química Estrutural, Instituto Superior Técnico, Universidade de Lisboa. silva.j@ufabc.edu.br

Metal-Organic Frameworks (MOFs) have emerged as promising platforms for developing new antimicrobial agents, especially in the face of the growing challenge of multi-drug bacterial resistance (MDR) [1]. Metal ions (such as silver (Ag) and copper (Cu)) display biocidal properties, high antimicrobial efficacy, and low toxicity to human cells, making them attractive targets for synthesis and characterization [2]. A previous analysis carried out at the Cambridge Crystallographic Data Centre (CCDC) showed that crystal structures of metal complexes involving aliphatic carboxylic acids are significantly less reported than those based on aromatic dicarboxylic acids. Thus, the structural determination of aliphatic carboxylic acids still poses a challenge due to their low tendency to form single crystals, which reinforces the need for powder X-ray diffraction
[3].

The present work presents the crystal structure of a copper-based compound obtained via selfassembly synthesis. The synthesis process and resulting cooper coordination polymer with pimelic acid were thoroughly investigated using IR spectroscopy, powder, and single-crystal X-ray diffraction. The data indicate that the self-assembly method yields a cooper-pimelic complex that is stable at room temperature and readily crystallizes from aqueous solution. The crystal structure reveals the formation of a two-dimensional coordination polymer (2D-CP) in the centrosymmetric monoclinic system, space group $P2_1/c$, with the molecular formula $\{[Cu(pim)]\cdot 2H_2O\}_n$ (pim = pimelic acid). The central Cu(II) ion is five-coordinated in a $\{CuO_5\}$ environment, adopting a geometry close to square pyramidal, with oxygen atoms from the bridging pim ligands occupying the coordination sites. Although the coordination polymer has been successfully synthesized, process optimization is still required to control the crystalline phase obtained. Additionally, the potential incorporation of auxiliary ligands and biological assays will be explored as application and experimental efforts progress.

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**Keywords: MOFs; Coordination Compounds; Aliphatic Ligands; Powder X-ray Diffraction, SelfAssembly Synthesis.**

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# *Synthesis and characterization of $MgAl_2O_4$ spinel: application of the Rietveld method in structural refinement*

***K. L. L. Marinho[1], Y. M. Camejo[1], J. M. Sasaki[1]***

[1] Federal University of Ceará, Fortaleza, CE, Brazil kassiamrnh@gmail.com

Magnesium aluminate spinel ($MgAl_2O_4$) is a refractory ceramic material widely used in industrial applications due to its excellent thermal stability, mechanical strength, and chemical inertness. It finds application in high-temperature environments such as thermal barrier coatings, cement kilns, and steel furnaces, as well as in catalysts and electronic components, owing to its structural versatility and chemical resistance [1].

Among the different synthesis routes, the solid-state reaction is one of the most common methods due to its simplicity and scalability [2]. However, parameters such as precursor mixing, reaction temperature, and processing time have a significant influence on the formation of the spinel phase and the resulting microstructural properties [3].

In this study, the synthesis of $MgAl_2O_4$ using residues from the steel industry containing MgO and $Al_2O_3$ is investigated, contributing to sustainable practices and industrial waste valorization. The experimental approach involves the use of the solid-state reaction [3,4] and other alternative routes, such as hydrothermal synthesis [5], aiming to evaluate how different processing conditions affect the crystallinity, morphology, and thermal stability of the final material.

Structural characterization was conducted using X-ray diffraction, and the crystal structure was refined through the Rietveld method. The results showed that the adopted methodology effectively led to the formation of a highly crystalline spinel phase, with features that make it a viable candidate for various technological and industrial applications.

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**Keywords: Magnesium aluminate spinel; refractory material; X-ray diffraction; Rietveld method.**

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# *Novel crystal structure, magnetic and electrical properties of $TmFe_{1-x}Co_xO_3$ perovskites*

***M. V. Arellano[1], F. E. Lurgo[2], J. M. De Paoli[1], R. D. Sánchez[2] and R. E. Carbonio[1]***

[1]INFIQC (Consejo Nacional de Investigaciones Científicas y Técnicas [CONICET]–Universidad Nacional de Córdoba), Departamento de Fisicoquímica, Facultad de Ciencias Químicas, Universidad Nacional de Córdoba, Haya de la Torre Esq. Medina Allende, Ciudad Universitaria, X5000HUA Córdoba, Argentina. [2]Instituto de Nanociencia y Nanotecnología (CNEA-CONICET). Centro Atómico Bariloche, Comisión Nacional de Energía Atómica and Instituto Balseiro, Universidad Nacional de Cuyo, (8400) San Carlos de Bariloche (RN), Argentina. manuel.arellano@unc.edu.ar

Perovskite-type oxides with the general formula $ABO_3$ exhibit a wide variety of properties, making them ideal systems for exploring structure–property relationships in solid-state chemistry. In particular, $RMO_3$ compounds (R = rare-earth, M = first-row transition metal) are known for their complex magnetic interactions involving 3d and 4f electrons, as seen in well-studied orthoferrites ($RFeO_3$) [1] and orthocobaltites ($RCoO_3$) [2].

In this context, we synthesized and characterized the $TmFe_{1-x}Co_xO_3$ series, focusing on their crystal structure, magnetic properties, and electrical transport behavior. Structural refinement using powder X-ray diffraction (PXRD) confirmed that all samples crystallize in the orthorhombic *Pbnm* structure. The crystal structure refined by the Rietveld method implemented through FullProf program. A pseudo-Voigt function was employed to model the peak shapes. The refined parameters included background, scale factor, zero shift, unit cell parameters, atomic positions, isotropic atomic displacement parameters, and standard profile parameters associated with peak shape description. Magnetization measurements (5 – 300 K, up to 5 KOe) reveal predominantly antiferromagnetic interactions and spin reorientation transitions near 70 K in Fe-rich samples. Hightemperature susceptibility follows Curie–Weiss behavior with negative Weiss constants. Electrical resistivity measurements as a function of temperature show semiconducting behavior for all compositions, except for $TmCoO_3$ (x = 1), which exhibits semimetallic features. Most samples follow the Mott variable-range hopping model [3], whereas $TmCoO_3$ fits the Efros–Shklovskii model [4], indicating Coulomb gap effects due to electron–electron interactions.

These results underscore the correlation between crystal structure, magnetism, and charge transport in $TmFe_{1-x}Co_xO_3$, highlighting the tunability of physical properties via B-site substitution.

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**Keywords: Poder X-Ray diffraction; perovskite; magnetism**

# Structural, polymorphic and supramolecular study of Biperidene Chloride by Laboratory and Synchrotron X-Ray Powder Diffraction

**N. D. Pimiento Santos[1], A. Dugarte-Dugarte[2], C. G. Albarracín Caballero[1], R. A. Toro Hernández[1], G. Díaz de Delgado[2], J. M. Delgado[2], A. N. Fitch[3], C. Dejoie[3], J. A. Henao Martínez[1]**

[1]Grupo de Investigación en Química Estructural (GIQUE), Universidad Industrial de Santander, Bucaramanga, Colombia. [2]Laboratorio de Cristalografía-LNDRX, Universidad de Los Andes, Mérida, Venezuela. [3]European Synchrotron Radiation Facility (ESRF), Grenoble, France.
nelson2210115@correo.uis.edu.co

The limited bioavailability of essential medicines, identified by the WHO as a growing threat to global public health, has driven increased interest in the structural characterization, polymorphism, and supramolecular chemistry of active pharmaceutical ingredients (APIs). This study focuses on the solid-state characterization of biperiden hydrochloride (BPR-Cl), an antiparkinsonian drug with an unreported crystal structure [1-2]. Structural determination of BPR-Cl was performed by X-ray powder diffraction. Additional characterization was carried out by infrared spectroscopy and TGADSC thermal analysis. The diffraction pattern is consistent with the unindexed pattern reported in the PDF-5+ database (00-043-1743) [3]. Indexing with the program Conograph suggests an orthorhombic unit cell and space group *Fdd*2 with parameters ***a*** = 21.690 Å, ***b*** = 58.398 Å, ***c*** = 5.9924 Å, V= 7590.2 Å$^3$, Z = 16, and Figures of Merit $M_{20}$ = 50.601, Mwu = 41.400, Mrev = 43.375, and Msym = 219.48. IR spectra confirmed the characteristic bands of the functional groups present in the compound. Thermally, BPR-Cl showed a mass loss of 83.487% between 140 °C - 315 °C, and melting point with decomposition at 294.13 °C. Recrystallization under different conditions produced crystals of the same orthorhombic phase. The structure was determined using laboratory and synchrotron radiation data with EXPO14 and refined using the Rietveld method with Topas. The packing is governed by strong hydrogen bonds of BPR+ with the chloride ion forming linear chains parallel to the *c*-axis. The π-contacts form discrete dimers extending linearly along the *a*axis and in zig-zag fashion parallel to *b*-axis. Hirshfeld surface analysis corroborates the strong hydrogen bonds, and fingerprint plots reveal that the most contributing interactions are H···H, indicating that dispersion forces are important.

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**Keywords: biperiden chloride; crystal structure; supramolecular; X-Ray diffraction; pharmaceutical characterization.**

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# *Synthesis and structural characterization of organic-inorganic halide perovskites*

***F. R. Praxedes[1], P. L. Forcellini[1], F. F. Ferreira[1]***

[1]Federal University of ABC – Center for Natural and Human Sciences, Avenida dos Estados 5001, Bangu, CEP: 09210-580, Santo André, SP, Brazil.
Email :p.forcellini@ufabc.edu.br

Hybrid organic-inorganic perovskites are essential materials for solar energy conversion, owing to their outstanding photovoltaic characteristics. These compounds show significant potential for use in energy technologies, especially for solar cells [1]. Nevertheless, a complete understanding of the link between their crystal lattice and optoelectronic behavior is still lacking. This study reports on the preparation and analysis of a series of organic-inorganic halide perovskites: HMDAPbCl4, HMDAPbBr4, and HMDAPbI4. The perovskite powders were prepared using a precipitation method from a stoichiometric acidic solution of halide precursors. This process involved introducing the solution into chilled isopropanol within an ice bath, resulting in the instantaneous formation of powders with distinct colors: white (HMDAPbCl4), yellowish (HMDAPbBr4), and orange (HMDAPbI4). The prepared substances were analyzed by powder X-ray diffraction (PXRD), and their crystallographic details were determined with the Rietveld refinement method. Notably, the diffraction patterns from these materials did not correspond to any known structures in existing literature. This observation points to the creation of a novel crystalline phase that has not been documented before. For a more thorough understanding, we also examined the perovskite precursors—specifically, the 1,6-hexamethylenediamine halide salts (HMDACl2, HMDABr2, and HMDAI2). It is established that these salts exhibit temperature-induced phase changes [2], a characteristic that might also be present in the final perovskite structures.

**Keywords: Hybrid Perovskite; hexamethylenediamine; lead halides; crystalline structure**

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# *Rietveld refinement applied to a $Ni_2Ti_{1.68}Hf_{0.32}$ mol alloy*

***R. L. Soares[1], W. B. de Castro[2]***

[1]Federal University of Campina Grande (UAEP), [2]Federal University of Campina Grande (UAEM)
Email of communicating ronieter@gmail.com

Shape memory alloys are materials that enable phase changes in the solid state through the application of specific stress or thermal conditions, resulting in the formation of phases called austenite (during heating) and martensite (during cooling) [1,2]. A phase is characterized by being a crystalline cell that has certain physical and chemical configurations. The austenitic phase is considered stable, being cubic, while the martensitic phase, non-cubic, is known as unstable. This differentiation in atomic organization means that unit cells can present themselves as one of the 14 Bravais lattices [3] and therefore as any of the 230 space groups [4,5].

In this work, the $Ni_{50}Ti_{42}Hf_8$ .at% (2/1.68/0.32 mol) alloy, obtained by electric arc melting, was submitted to the Shimadzu XRD-6000 diffractometer at room temperature and, subsequently, the respective experimental results were submitted to the Rietveld structure refinement method [6]. The X'Pert HighScore Plus 3.0 software from PanAnalytical B. V. 2009, under license 32001273, was used in automatic mode to determine the quantitative and qualitative properties of the predominant unstable phase (B19'). The aim of this application was to characterize the monoclinic structure (space group number 11 → P 1 21/ m 1) detected from crystallographic chart No. 160485ICSD (CIF reference file 99-509-0003) [7].

The results collected indicated a preferential direction 001 present in the sample, and values were obtained for various structural data such as: network parameters (a b c), Wyckoff positions (x y z), interaxial angles (α γ β), mass fraction (%), density ($g/cm^3$), volume ($10^6$ $pm^3$), F(000) and Miller indices (h k l) in the 10 reflected peaks. In addition, it was possible to compile the coefficients of the polynomial equation adopted in the background (baseline), the Caglioti function (FWHM), the profile of the Pseudo-Voigt function and the statistical fit values R(expected) = 20.00, R(profile) = 19.03, R(weighted profile) = 25.44, d-statistic = 1.00 and GOF = 1.27.

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**Keywords: Rietveld refinement; X'Pert HighScore Plus; Ni-Ti-Hf; Ternary alloy; XRD.**

# *Structural Determination by X-ray Powder Diffraction and Supramolecular Analysis of the Opiod Dihydrocodeine Bitartrate*

***V. Ordóñez Ulloa[1], C. G. Albarracín Caballero[1], A. Dugarte Dugarte[2], R. A. Toro Hernández[1], G. Díaz de Delgado[2], J. M. Delgado[2], J. A. Henao Martínez[1]***

[1]Grupo de Investigación en Química Estructural (GIQUE), Universidad Industrial de Santander, Bucaramanga, Colombia. [2]Laboratorio de Cristalografía-LNDRX, Universidad de Los Andes, Mérida, Venezuela.

valentina2210116@correo.uis.edu.co, jahenao@uis.edu.co

Dihydrocodeine bitartrate ($DHC^{+}\cdot Tar^{-}$) is an opioid used for the treatment of severe pain and dry cough. It has been compared to codeine although it has better biological potency. Accordingly, the World Health Organization considers it a second-tier drug for the treatment of cancer-related pain [1]. However, side effects and possible addiction have been a concern as they affect a large percentage of patients [2]. Current information on the drug is limited. The powder diffraction pattern or the crystal structure of the compound could not be found in crystallographic databases, articles, or patents. Therefore, we decided to undertake the structure determination of dihydrocodeine bitartrate. Further characterization was carried out by Infrared spectroscopy (ATR FT-IR) and thermal analysis (TGA-DSC). The X-ray powder diffraction pattern was registered, and a search/match procedure did not show coincidence with any reported patterns in the PDF-5+ database. Indexing of the pattern with DICVOL14, implemented in the PreDICT interface, suggests that the crystal system is triclinic and the final parameters obtained were ***a*** = 7.1687(10) Å, ***b*** = 8.0786(10) Å, ***c*** = 9.4312(12) Å, α = 76.127(8)º, β = 82.112(10)º, γ = 86.818(11)°, V = 525.09(12) Å³ with Z = 1. The analysis of the reflection conditions using EXPO suggested *P*1 as the space group. In the crystalline arrangement, the $DHC^{+}$ cation is connected to the counterion by strong NH···O hydrogen bonds. $DHC^{+}$ moieties pack into linear chains parallel to the *b* axis, alternating with chains of $Tar^{-}$ ions. Hirshfeld surface analysis, fingerprint plots, and intermolecular energy calculations using the 'UNI Intermolecular Potentials' within CSD-Materials in the Mercury interface were also performed.

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**Keywords: dihydrocodeine bitartrate; opioid; X-ray powder diffraction; crystal structure; structure determination.**

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# *Identification and Quantification of Crystalline Phases in Inorganic Solids with Potential Application in the Study of Petroleum Emulsions by X-ray Diffraction*

***Victoria P. Polonini[1], Maximiliano J. M. Zapata[1], Cristina M. S. Sad[1], Marcos A. Ribeiro[1]***

[1]Universidade Federal do Espírito Santo, Centro de Ciências Exatas – Programa de PósGraduação em Química, Av. Fernando Ferrari, 514, Goiabeiras, Vitória – ES, Brasil. victoria.polonini@edu.ufes.br

Fine inorganic solids present in petroleum formation water play a key role in the stability of waterin-oil (W/O) emulsions, as they can increase oil viscosity and promote the formation of scale deposits. Among these solids, calcium carbonate ($CaCO_3$) stands out as a precursor to scaling, with its crystalline nature directly influencing interfacial phenomena and deposition in pipelines. [1]

Among these solids, calcium carbonate ($CaCO_3$) is often identified as a precursor to these incrustations, and its crystalline nature directly influences interfacial phenomena and pipeline scaling. Between $CaCO_3$ polymorphs, calcite is the most thermodynamically stable form, (rhombohedral structure); aragonite (orthorhombic) and vaterite (hexagonal) are less stable. The presence of magnesium in the formation of water may result in the formation of magnesian calcite, altering crystallization pathways and interfacial adsorption behavior, thus affecting emulsion stability.[2-3]

To gain insights into the crystallization behavior of fine inorganic solids, this study aims to characterize powdered mixtures of $CaCO_3$ and NaCl via X-ray diffraction (XRD) and the Rietveld method (RM). The objective of this work is to investigate the structural evolution and phase interactions in binary systems, as a reference pattern to a real solid found in petroleum production. Mixtures were prepared in mass ratios from 0% to 100% $CaCO_3$, at 12.5% intervals and analyzed using a Bruker D8 Advance diffractometer (15° - 90° range - 2 Theta -, 0.02° step size). Diffraction patterns confirmed the presence of the calcite phase and revealed intensity shifts and peak broadening correlated to NaCl concentration, suggesting changes in crystallinity.

This study highlights the relevance of XRD in identifying and quantifying crystalline phases in multicomponent inorganic systems, providing insights into fine inorganic solids contributions to petroleum production challenges, such as scale formation and emulsion stabilization.

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**Keywords: XRD; W/O emulsion; fine inorganic solids; calcium carbonate.**

# *Crystal structure, magnetic properties and elementary excitations of copper(II) oxide*

***V. Zaramella[1], N. P. Westin[1], E. Granado[1]***

[1]*Instituto de Física "Gleb Wataghin" (IFGW), Universidade Estadual de Campinas (Unicamp)*

*v195463@dac.unicamp.br*

Copper(II) oxide, considered a building block of high-temperature superconductors, exhibits remarkable electrical and magnetic properties, such as multiferroic behavior at low temperatures [1]. It also hosts various magnetic phases and hybrid excitations, such as electromagnons, which were recently reported through terahertz measurements [2] in a spectral range that has not yet been explored by vibrational spectroscopy. In this work, ceramic samples were produced in order to investigate the structural, magnetic, and vibrational properties of CuO, with emphasis on the low-frequency vibrational region. The results revealed, in addition to the known vibrational modes, an additional mode around 240 $cm^{-1}$. This mode had previously been reported as originating from strong spin-phonon coupling [3], but disappears at temperatures near 160 K, where a possible structural transition is assumed to occur [4]. Low-frequency excitations related to electromagnons were not detected. Based on these preliminary results, new measurements using CuO single crystals may contribute to a deeper investigation of the material's elementary excitations, aiming to clarify open questions regarding its physical properties.

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**Keywords: Copper(II) oxide; Multiferroic materials; Spin-phonon coupling; Electromagnons; Vibrational spectroscopy.**

# Sponsors

# *STOE Transmission Geometry Enhanced by Fully Automated Alignment*

***J. Kollath[1]***

[1]STOE & Cie GmbH, Hilpertstraße 10, 64295 Darmstadt, Germany
Kollath@stoe.com

The Transmission/Debye–Scherrer geometry, developed and patented by STOE & Cie GmbH, is a hybrid diffraction setup that combines elements of classical Scherrer and Guinier camera systems. It centers the sample within the detector circle (as in Scherrer geometry) while incorporating focusing optics (as in Guinier cameras), enabling high angular resolution over a wide 2θ range. STOE's transmission geometry is therefore especially suitable for high-quality powder diffraction analyses, Rietveld refinements, and studies requiring accurate intensities, sharp peaks, and minimal systematic errors.

In this contribution, we introduce the Transmission/Debye–Scherrer geometry and discuss its key advantages, including constant sample volume, no sample height displacement, no peak broadening, and reduced preferred orientation effects. We will present our newest feature, which is capable of fully automated alignment. This will ensure that all users always have access to the highest quality data. It handles all tasks, including realignment due to tube ageing, installing a new tube, and changing wavelengths between experiments.

**Keywords: LACA-ABCr; LAPDiC; Transmission Geometry; STOE; Diffraction; XRPD; Automated Alignment.**

# *Latest Advancements in Laboratory Powder XRD instrumentation.*

**J.-L. Brousseau**

Anton Paar, Switzerland jean-luc.brousseau@anton-paar.com

**Abstract**

Multi-user X-ray diffraction facilities require versatile systems capable of delivering high-quality data across diverse crystallographic applications. The XRDynamic 500 automated powder diffractometer addresses this need by integrating grazing incidence X-ray diffraction (GIXRD), variable-temperature XRD, small-angle X-ray scattering (SAXS), and pair distribution function (PDF) analysis, also known as total scattering, within a single platform. Automatic alignment capabilities also provide flexibility for exchanging X-ray tube of different energies without requiring manual realignment.

Application examples will highlight solutions to typical challenges, such as the detection of minor phases, alongside advanced methods like non-ambient XRD and SAXS, together with a presentation of the instrument’s technical features.

# *Powder X-Ray Diffraction: The Importance of Data Quality and Recent Advances in Instrumentation and Software – Redefining Benchtop XRD*

***F. João***

Essencis Technologies / Bruker joao.fiori@essencistech.com.br.org

**Abstract:**

The presentation will discuss the critical factors affecting data quality in Powder X-Ray Diffraction, such as fluorescence, white radiation, Kβ radiation, background reduction, and the inherent challenges in low-angle measurements. It will also address recent advances in instrumentation and software that have significantly improved overall data quality, enabling modern benchtop diffractometers to achieve performance levels once restricted to large-scale systems.

**Keywords: LACA-ABCr; Powder X-Ray Diffraction; white radiation; benchtop diffractometers Kβ radiation; background**