PPXRD-18

18th Pharmaceutical Powder X-ray Diffraction Symposium

ONSITE PROGRAM & BOOK OF ABSTRACTS

6–9 May 2025

The Cambridge Crystallography Data Centre (CCDC)
Cambridge, United Kingdom

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PPXRD-18 Organizing Committee

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PPXRD-18 Program

Table of Contents

Thank you to Merck Sharp & Dohme LLC, Rahway, NJ, USA for their support of PPXRD-18.

Exhibitor Information

Exhibition

Exhibits will be set in the common area at CCDC from Tuesday, 6 May until Friday, 9 May. Please visit with our exhibitors during coffee breaks and lunches. Exhibits will close at the conclusion of the symposium.

Exhibiting Companies

Anton Paar Ltd
www.anton-paar.com

Representative: *Nishil Malde*,
nishil.malde@anton-paar.com

Anton Paar was established in 1922 and headquartered in Graz, Austria. The company has over 4200 employees worldwide with established global sales, application and customer support network in more than 110 countries. Our dedicated UK operation was established in 1982 and currently has over 60 employees trained to support our growing customer needs.

Anton Paar is recognised as a pioneer and leader in the field of small-angle X-ray scattering (SAXS) and non-ambient X-ray diffraction (XRD), acquired over more than half a century. We focus on premium quality and superior performance, which is trusted by the global X-ray analytics community.

Bruker AXS
www.bruker.com

Representative: *Michael Carr,*
michael.carr@bruker.com

Bruker AXS is a global market and technology leader for structural analysis with X-ray and electron diffraction and elemental analysis using X-ray fluorescence, spark optical emission spectroscopy, and CS/ONH analysis. Bruker AXS provides advanced imaging solutions in X-ray and electron microscopy.

International Centre for Diffraction Data
www.icdd.com

Representative: *Suri Kabekkodu, kabekkodu@icdd.com*

ICDD, started in 1941, focuses on meeting the needs of the scientific community through the publication of the Powder Diffraction File™ (PDF®) and JADE® software. ICDD's carefully curated and edited material identification databases interface with diffractometers and analysis systems of the world's leading X-ray equipment manufacturers and software developers. Our products are available in a variety of licensing options. We also provide quality training through our educational courses. Visit us to discuss your analysis or training needs.

Malvern Panalytical
www.malvernpanalytical.com

Representative: *Paul O'Meara,*
paul.omeara@malvernpanalytical.com

Together we are a powerful and highly complementary combination of market leading technologies. We are the toolmakers for the world's most innovative companies, academic institutions, and government laboratories. More than 92,000 of our instruments are used every day in our customers' laboratories. Customers value us not only for the power of our analytical technologies, but also for the depth of our expertise. Malvern Panalytical instruments analyze the chemical, physical and structural nature of materials. Our leading technologies measure particle size, shape, concentration and zeta potential, biomolecular interactions and stability, elemental concentrations and crystallographic structure. Micromeritics manufactures systems for the characterization of particles, powders, and porous materials. Our leading technologies measure surface area, porosity, density, adsorption and particle activity. SciAps specializes in portable X-ray fluorescence (XRF), laser-based (LIBS) and near-infrared (NIR) analyzers to measure any element in any environment.

Rigaku
www.rigaku.com

Representative: *Christopher Morris,*
christopher.morris@rigaku.com

Founded in Tokyo in 1951, Rigaku is a leading global provider of analytical and industrial instruments. The company's broad product portfolio includes general X-ray diffraction, thin-film analysis, X-ray fluorescence spectrometry, small angle X-ray scattering, protein and small molecule X-ray crystallography, Raman spectroscopy, X-ray optics, semiconductor metrology, X-ray sources, computed tomography, non-destructive testing and thermal analysis. With more than 1,800 employees, the company serves industries such as pharmaceuticals, biotechnology, semiconductor manufacturing, and environmental sciences. Rigaku is committed to customer satisfaction through exceptional service, comprehensive training and global support.

STOE & Cie GmbH
www.stoe.com

Representative: *Michael Teck, teck@stoe.com*

STOE, founded in 1887, has been a pioneer in powder and single crystal X-ray diffraction since the 1960s. STOE invented the transmission geometry for Powder XRD and developed the first open Eulerian cradle XRD system with a pixel detector for single crystals. With in-house R&D, programming, electrical and mechanical engineering, and production, STOE offers standard and individual solutions, delivering unparalleled quality and precision. Thus, having become THE partner in X-Ray Diffraction for crystallographers worldwide!

Tuesday Workshops, 6 May 2025

CCDC Headquarters

ICDD Database and Software

Tom Blanton & Suri Kabekkodu, International Centre for Diffraction Data, USA

This workshop will present practical applications of X-ray powder diffraction as an analytical technique as it is currently used in today's laboratory. This knowledge and understanding are applicable whether the attendee works in academia, industry, or government laboratory. The workshop presentation will combine lectures and hands-on demonstrations covering database datamining and, if time is available, software analyses methods. *Participants in this workshop will have access to the ICDD PDF-5+ database and JADE Pro software through a cloud Remote Desktop Server. Attendees will be provided with an account to log into the RDS. Participation in this workshop will be most productive with access to a laptop computer.*

9:00am **CCDC Staff**: Health and Safety Presentation

9:05am **Session 1**: Datamining using the ICDD Powder Diffraction File (PDF) Databases

- PDF entry (card)
- Datamining search options including:
    - Periodic Table
    - Formula/Name
    - Classifications
    - Crystallography

10:30am Coffee Break & Exhibition

11:00am **Session 2**: Diffraction Analysis using JADE Pro Software

- User Interface
- Profile Fitting
- Phase Identification
- Quantitative Analysis
- Whole Pattern Fitting/Rietveld

12:30pm Lunch Break

Investigating Structures Using the CSD

Suzanna Ward, Andrew Peel, CCDC, United Kingdom

This workshop is suitable for both beginners and experienced users of the CSD Portfolio. Participants must bring a laptop with the CSD Portfolio installed. More information and a workshop license will be provided in advance.

1:30pm **Session 1**: Exploring the CSD and Generating Powder Patterns

- Introduction to the CSD
- How to search and visualize structures in ConQuest and Mercury, with a focus on powder diffraction data
- Generating and analyzing powder patterns, including the use of AutoFIDEL algorithms

3:00pm Coffee Break & Exhibition

3:30pm **Session 2**: Using the CSD to Help Assess Solid Form Stability

- Overview of the CSD-Materials suite for solid state analysis
- Understanding intermolecular interactions and assessing structural stability
- Industrial applications and case studies, particularly relevant to pharmaceuticals

5:00 pm *End of Day 1*

Wednesday Sessions, 7 May 2025

CCDC Headquarters

Plenary Session: Intellectual Property

Chair: Tom Blanton, International Centre for Diffraction Data, USA

9:00am		Opening Remarks **Tom Blanton**, PPXRD Organizing Committee Chair, International Centre for Diffraction Data, USA
9:10am	P14	Invited - Patenting Polymorphs at the European Patent Office **Andrew Pitts**, Mewburn Ellis LLP, United Kingdom and Germany
9:50am	P31	Invited - Patenting Solid Forms of Psilocybin **Leonard Chyall**, Improved Pharma, USA
10:30am		Coffee Break & Exhibition
11:00am	P21	Pharmaceutical Crystal/Salt/Co-crystal Form Patents - The Industry Perspectives **Shawn Yin**, Bristol-Myers Squibb, Co., USA
11:30am		Intellectual Property Round Table; Moderator: **Tom Blanton** *All attendees are welcome to participate in discussion.*
12:30pm		Lunch Break

Pharmaceutical Case Study of PXRD

Chair: **Fabia Gozzo**, Excelsus Structural Solutions sprl, Switzerland and Belgium

1:30pm	P22	Invited - When the Unaddressed Answers Come from Synchrotron XRPD: A Case Study According to Textbook! **Arnaud Grandeury**, Novartis Pharma, Switzerland

Exhibitor Introductions

2:10pm		Exhibitors will give a brief presentation on their products offered.
3:10pm		Coffee Break and Exhibition

Industrial Applications and Machine Learning

Chair: **Simon Bates**, Rigaku Americas, USA

3:30pm	P32	Invited – Physical and Computational Characterisation of Solid-form Landscapes of Pharmaceutical Materials **Ian Rosbottom*, L. Russo, A. Watt, M. Thornton, A. Wardell, M. Nicholls**, GSK, United Kingdom
4:10pm	P10	Characterization of Polymers Used in Pharmaceutical and Biomedical Applications **Thomas Blanton*, M. Rost, D. Bohnenberger**, International Centre for Diffraction Data, USA
4:40pm	P25	Identification of Active Pharmaceutical Ingredients and Excipients Using AI **Akito Sasaki*, T. Shibasaki, T. Ohta, A. Himeda**, Rigaku Corporation, Japan
5:10 pm	*End of Day 2*	

Thursday Sessions, 8 May 2025

CCDC Headquarters

Structure Solution, Crystal Structure Prediction

Chair: **Shawn Yin**, Bristol-Myers Squibb, Co., USA

9:00am	P6	Invited - Organic Crystal Structure Prediction: What is Needed for Confident Prediction of Polymorphs? **Sarah Price**, University College London, United Kingdom
9:40am	P7	Molecular Crystal Structure Prediction and Comparison **Erin Johnso**n, Dalhousie University & University of Cambridge, Canada & United Kingdom
10:10am		Coffee Break and Exhibition
11:00am	P8	The Synergy of Computational Modeling, Machine Learning, and Experiments in Pharmaceutical Solid-State Research and Development **R. Alex Mayo*, G. Sun, M. Bellucci, Z. Yang, W. Fu, J. Yuan, Q. Zeng**, XtalPi, Inc., USA
11:30am	P9	Structure Solution of Sulphonamides from Powder Diffraction Data – A Problematic Moeity? **Pamela Whitfield**, Excelsus Structural Solutions, Switzerland
12:00pm	P26	Crystallization of Molecular Glass: From Amorphous State to New Polymorphic Forms of Sodium Naproxen **Paul O'Meara*, M. Orlova, G. Nénert, L. Ding, N. Dadivanyan**, Malvern Panalytical, United Kingdom
12:30pm		Lunch

Qualitative / Quantitative Analysis, High Throughput XRD Methods

Chair: **Raj Suryanarayanan**, University of Minnesota, USA

1:30pm	P28	Invited - Exploring High-Throughput Synchrotron X-ray Powder Diffraction for Quantitative Phase Analysis of Pharmaceutical Mixtures **Fabia Gozzo*, M. Reinle-Schmitt, R. Widmer, M. Garnier, Th. Stoll**, Excelsus Structural Solutions, Switzerland
2:10pm	P18	High-Throughput Synthesis & Powder XRD Screening of Pharmaceutical Solid Forms with Modular Multiwell Devices **V. Nicholas Vukotic*, A. Dmitrienko, E.T. Douglas, J. Kobti**, University of Windsor, Canada
2:40pm	P29	Advantages of Curated Subfiles in Pharmaceutical Analysis **Soorya Kabekkodu*, J. Blanton, T. Blanton**, International Centre for Diffraction Data, USA
3:10pm		Coffee Break & Exhibition

Poster Session & Reception

3:30 – 5:00pm, CCDC Common Area

Chair: **Tom Blanton,** International Centre for Diffraction Data, USA

Posters can be set anytime before 3:30pm on Thursday, 8 May. Poster boards will be set around the CCDC common area and will not have specific designations. You may choose any open board for your poster. Velcro will be provided.

P4 Crystal Structures of Large-Volume Commercial Pharmaceuticals
James A. Kaduk*, North Central College, Illinois Institute of Technology, USA
A. Dosen, T. Blanton, ICDD, USA

P11 Exploring the Role of Non-Ambient X-Ray Diffraction (XRD) in the Pharmaceutical Industry
Barbara Puhr*, M. Kremer, B. Schrode, Anton Paar GmbH, Austria
N. Malde, Anton Paar Ltd., United Kingdom

P12 Use of Component Analysis and Direct Derivation Methods to Access Phase Behavior in Amorphous Solid Dispersions
Kasumi Kihara*, A. Sasaki, Rigaku Corporation, Japan
S. Bates, Rigaku Americas Corporation, USA

P15 Matching ROY Crystal Structures to High Throughput PXRD
Grace Sparrow*, E.R. Johnson, Dalhousie University, Canada
R.A. Mayo, University of Ottawa, Canada

P16 Driving XRD in Regulated Environments
Barbara Puhr*, F. Gürer, M. Loombard, B. Schrode, Anton Paar GmbH, Austria
N. Malde, Anton Paar Ltd., United Kingdom

P17 DIFFRAC.EVA, a Comprehensive Toolset for Characterizing Pharmaceutical Materials
Karsten Knorr, Bruker AXS, Germany

P24 Transmission XRPD for the Unravelling of Complex Solid Form Landscapes
Matthew Thornton*, I. Rosbottom, A. Wardell, D. Beddall, J. Harris, M. Nicholls, GSK, United Kingdom

P27 Detect and Control Polymorphism: Quick and Efficient Solid Form Analysis
Natalia Dadivanyan*, P. O'Meara, M. Orlova, G. Nénert, L. Ding, Malvern Panalytical, Germany

Friday Sessions, 9 May 2025

CCDC Headquarters

Complex Materials Analysis

Chair: **Natalia Dadivanyan**, Malvern Panalytical, Germany

9:00am	P19	Invited - Advanced Characterization of Lipid Nanoparticles in Formulation Development Using X-rays Scattering **Viktoriia Meklesh**, AstraZeneca, Sweden
9:40am	P5	Small-Angle X-Ray Scattering as Powerful Tool for Phase and Crystallinity Assessment of Monoclonal Antibody Crystallites in Support of Batch Crystallization **Patrick Larpent*, L. Codan**, MSD Werthenstein Biopharma GmbH, Switzerland **J.R. Bothe, L. Iuzzolino, S. Pabit, S. Gupta, T. Fischmann, Y. Su, P. Reichert, D. Stueber, A. Cote**, Merck & Co., Inc., USA
10:10am		Coffee Break and Exhibition

Instrumentation and Complementary Techniques

Chair: **Anisha Patel**, GSK, USA

10:40am	P13	Invited - Advancing Electron Diffraction for Beam-Sensitive Materials: Current Insights & Emerging Techniques **Partha Pratim Das*, A. Gomez-Perez, S. Nicolopoulos, A.S. Galanis, M. Fransen**, NanoMEGAS SPRL, Belgium **M.J. Rodríguez-Espinosa, J.M.B. Romero**, National Biotechnology Center, Spain **S. Plana-Ruiz**, Universitat Rovira i Virgili, Spain
11:20am	P3	Measurement of Dose in Mixed Gamma and Neutron Fields by X-Ray Diffraction in Alanine **Omololu O. Makinde*, U. Jain, N.D. Gallant, N. Diaz-Elsayed**, University of South Florida, USA **T.V. Holschuh II, M.A. Reichenberger**, Idaho National Laboratory, USA
11:50am		Closing Remarks **Tom Blanton**, PPXRD Organizing Committee Chair, International Centre for Diffraction Data, USA
12:00pm		*End of Symposium*

PPXRD-18 Plenary

Intellectual Property

- Speakers -

Dr. Andrew Pitts

Dr Andrew Pitts is a European and Chartered Patent Attorney working at the Munich office of the British patent firm Mewburn Ellis LLP. He has nearly a decade of experience managing small-molecule patent matters for multinational and medium-sized pharmaceutical companies. His work often includes bringing polymorph patent applications to grant and defending polymorph patents at the EPO.

Andrew has a Ph.D. in organic chemistry from the University of Cambridge, where his research focused on C–H functionalisation and natural product synthesis. He also has an MSci (Hons.) degree in medical and biological chemistry from the University of Nottingham, with a year spent working at AstraZeneca in Göteborg, Sweden.

Dr. Leonard Chyall

Dr. Leonard "Len" Chyall is an organic chemist and a consultant with Improved Pharma, located in Indiana, USA. He has extensive expertise with X-Ray powder diffraction as an analytical technique to characterize solid forms of pharmaceutical drug substances and drug products. Prior to joining Improved Pharma, Len worked for over a decade at SSCI, Inc. and then at Aptuit Consulting as a research chemist, project manager and business leader. He subsequently founded Chyall Pharma, where he continues to provide research and consulting services to the pharmaceutical industry. He received his Ph.D. in chemistry from the University of Minnesota and was a postdoctoral fellow in the chemistry department at Purdue University. Len has testified as an expert witness in patent litigation matters concerning XRPD analyses in United States courts and abroad.

Notes

PPXRD-18 Pharmaceutical Powder X-ray Diffraction Symposium

Patenting Polymorphs at the European Patent Office

Andrew K. Pitts, Ph.D.

Mewburn Ellis LLP

United Kingdom and Germany

andrew.pitts@mewburn.com

Polymorphs are separately patentable inventions that are often discovered later in the product development process. The patent term for these polymorphs can therefore extend beyond expiry of the first patent to the chemical in its first form. The European Patent Office (EPO) is commonly considered one of the more difficult offices to bring a polymorph patent application to grant. This is due to the large body of case law that has developed over the years setting out specific requirements for a polymorph to be found patentable. This presentation will explore important considerations when drafting a polymorph patent application and how to navigate the complex legal requirements at the EPO to bring such an application to grant. This will include an analysis of relevant EPO polymorph case law, such as seminal decision T 777/08 (which sets out technical effects that cannot be relied upon for inventive step of a crystalline form) and the more recent related decisions T 1994/22 and T 672/21 (which concern relying on new data after filing an application to support inventive step of a crystalline form).

Keywords: intellectual property, patents, European Patent Office, novelty, inventive step, case law, crystalline forms, IP strategy.

PPXRD-18
Pharmaceutical Powder
X-ray Diffraction Symposium

Patenting Solid Forms of Psilocybin

Leonard Chyall, Improved Pharma, United States, len.chyall@improvedpharma.com

New crystalline forms of existing compounds are frequently discovered and characterized by XRPD, and inventors often rely on XRPD peak positions to claim these new polymorphs in patent applications. But earlier published literature may cause a patent examiner to be concerned with whether the claimed invention is expressly or implicitly disclosed in those prior investigations. Patents may also be granted but then later found to be invalid though litigation, or in Inter Partes Review proceedings in the United States. A recent case in in front of US Patent Trial and Appeal Board concerned the hallucinogenic drug psilocybin, which was first isolated from mushrooms and characterized in 1958. A key scientific question was whether earlier literature XRPD data for psilocybin anticipates the claims covering solid forms of this compound in later issued patents. This presentation will focus on the scientific issues related to those proceedings.

Keywords: Psilocybin, XRPD, Patents; Polymorph; Anticipation

PPXRD-18 Pharmaceutical Powder X-ray Diffraction Symposium

Pharmaceutical Crystal/Salt/Co-crystal Form Patents

- The Industry Perspectives

Shawn Yin, Bristol-Myers Squibb, Co., New Brunswick, New Jersey, shawn.yin@bms.com

It is well known that Intellectual Property (IP) is one of the critical components in pharmaceutical industry. On top of the Composition of Matter (COM) patents, there are some key patentable innovations, benefiting patients, generated from the whole stages of pharmaceutical development. Those Patient Centered Innovations (PCI) include API crystal/salt/co-crystal form, drug product formulation, drug delivery and device, etc. These PCIs/patents are not only providing benefit to patients and may also be innovations that are patentable. The common pharma industry PCI/patent strategy on API form will be described with some well-known case studies. The critical role PXRD played in API form patents will also be discussed.

Keywords: Intellectual Property, Patient Centered Innovation, Patents, Crystal Form, PXRD

PPXRD-18 Pharmaceutical Powder X-ray Diffraction Symposium

When the unaddressed answers come from Synchrotron XRPD: A case study according to textbook!

Arnaud Grandeury, Leading Novartis Scientist, Materials Science, Technical Research and Development, Novartis Campus, Virchow 6.3.231, 4056 Basel, Switzerland

The aim of this study is to highlight the complementary nature of synchrotron-based techniques in comparison to state-of-the-art laboratory equipment, specifically X-ray powder diffractometers. Currently, limitations have been identified in the peak-to-peak resolution between different phases, impacting the evaluation of the purity of active pharmaceutical ingredients (APIs) as a quality attribute. Furthermore, the evaluation of phase purity in the drug product is hindered by a lack of specificity and low strength, which significantly affects sensitivity and the limit of detection. This case serves as a textbook example of how synchrotron techniques can address these challenges by leveraging improved brightness and an optimum choice of wavelength. The use of synchrotron-based approaches can effectively enhance selectivity, peak-to-peak resolution, and sensitivity. By utilizing synchrotron methods, researchers can overcome the limitations of traditional laboratory equipment and achieve more accurate and reliable results. This study demonstrates the significance of synchrotron-based approaches in pharmaceutical analysis, specifically in evaluating the purity of APIs and drug products. The unique capabilities of synchrotron techniques offer valuable insights into the characterization of pharmaceutical materials, ultimately contributing to the development of high-quality drugs and ensuring patient safety.

Keywords: polymorphism; phase purity; trace analysis; synchrotron XRD; ICH

PPXRD-18 Pharmaceutical Powder X-ray Diffraction Symposium

Physical and Computational Characterisation of Solid-form Landscapes of Pharmaceutical Materials

Ian Rosbottom[1], Luca Russo[1], Andrew Watt[1], Matthew Thornton[1], Aneesa Wardell[1], Mark Nicholls[1], Joe Harris[1,2]

1. *Materials Science, Drug Substance Development, Medicine Development and Supply, GSK, Gunnels Wood Road, Stevenage, SG1 2NY, UK*
2. *Materials Science, Manufacturing Science and Technology, Global Supply Chain, GSK, Priory Street, Ware, Hertfordshire, SG12 0DJ, UK*

Over the past 30 years, there has been a significant advancement in the understanding and risk assessment of pharmaceutical material polymorphs. This progress is largely due to past incidents of late-appearing polymorphs causing costly product recalls[1].

As the complexity of molecules and solid-form landscapes increases, including solvates and hydrates, targeted computational approaches are required to reduce the experimental burden of investigating solid-form landscapes. GSK employs a combination of physical and computational characterization to develop reliable solid-forms for these increasingly complex small molecules[2].

This presentation highlights the value of using complex, cost-effective computational modelling tools and optimizing data quality to explore solid-form landscapes. Key topics include:

- Optimizing XRPD data collection through geometry and optics adjustments
- Utilizing XRPD indexing and Rietveld refinement to maximize information extracted from XRPD data and validating structural models
- Employing complex structural modelling to enhance understanding of solid-form landscapes

These approaches are demonstrated through both generic and GSK-specific case studies, illustrating how they address laboratory challenges and expedite the delivery of medicines to patients. While computational advances are significant, they must be complemented by careful experimentation grounded in robust scientific and instrumentation knowledge.

Characterisation remains central to the Materials Science Tetrahedron in pharmaceutical development[3], but where possible should be complemented by computational and structural modelling to make the most of every point of experimental data. This modelling approach enhances scientists' understanding of the data that is gathered and enables effective responses when addressing anomalies, thereby potentially accelerating the development process of pharmaceutical products.

Keywords: powder diffraction, crystal structure prediction, modelling, indexing, solid-form landscapes

References

1. Bučar, D.-K., Lancaster, R.W. and Bernstein, J. (2015), Disappearing Polymorphs Revisited. Angew. Chem. Int. Ed., 54: 6972-6993.
2. Agarwal, P., Huckle, J., Newman, J., & Reid, D. L. (2022). Trends in small molecule drug properties: A developability molecule assessment perspective. Drug Discovery Today, 27(12), 103366.
3. Sun, C.C. (2009), Materials science tetrahedron—A useful tool for pharmaceutical research and development. J. Pharm. Sci., 98: 1671-1687.

PPXRD-18 Pharmaceutical Powder X-ray Diffraction Symposium

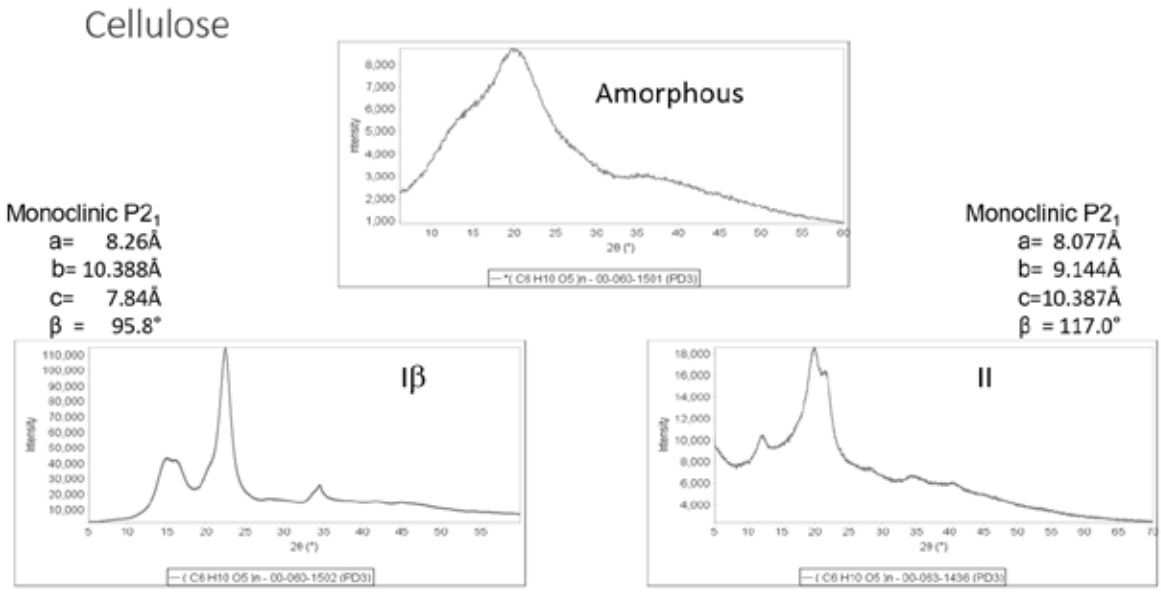
Characterization Of Polymers Used In Pharmaceutical And Biomedical Applications

Thomas Blanton, M. Rost, D. Bohnenberger, International Centre for Diffraction Data, Newtown Square, PA, USA, tblanton@icdd.com

Polymers show a range of order from amorphous to semi-crystalline. Traditional organic analytical techniques, such as infrared spectroscopy (IR), differential scanning calorimetry (DSC), thermal gravimetric analysis (TGA), and nuclear magnetic resonance (NMR), are typically used for polymer analysis. Though X-ray diffraction (XRD) is not commonly used as the primary technique for polymer characterization, XRD does provide unique information about a polymer particularly when assessing crystallinity and crystallite size. In medical applications, polymers are often used as excipients in pharmaceuticals, and the base material for delivery devices used in biomedical applications.

ICDD has been adding polymer diffraction data to the Powder Diffraction File (PDF®) with the focus on adding raw data diffraction patterns (1D and 2D) as part of the PDF entry. The inclusion of the raw data diffraction pattern is important in correctly identifying the polymer contribution to a composite material diffraction pattern. A traditional d-spacing/intensity stick pattern or simulated diffraction pattern is not capable of accounting for the full-pattern diffraction profile of polymers since all polymers have some amorphous component. This polymer project focuses on industrially important polymers with an added emphasis on polymers used in medical and biomedical applications. New entries resulting from this project will be presented along with phase identification analysis results for pharmaceutical formulations that include polymer excipients.

Keywords: XRD; polymer; pharmaceutical; biomedical



PPXRD-18
Pharmaceutical Powder
X-ray Diffraction Symposium

Identification of Active Pharmaceutical Ingredients and Excipients Using AI

Akito Sasaki, **T. Shibasaki**, **T. Ohta, A. Himeda**

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Pharmaceutical tablets contain active pharmaceutical ingredients (APIs) and various functional excipients that govern the drug product performance. Traditionally, many of these components were crystalline, allowing for easy identification and quantification using powder X-ray diffraction (PXRD). However, modern formulations increasingly include amorphous! substances, making conventional PXRD analysis more challenging. While crystalline! components can be identified through characteristic diffraction patterns, amorphous materials! produce diffuse patterns with subtle variations, making their identification more difficult.

In this study, we applied artificial intelligence (AI) to improve the identification of amorphous pharmaceutical components. Specifically, we developed an AI model based on Vision Transformer (ViT), which treats PXRD patterns as images, enabling it to capture subtle differences in diffraction patterns. We trained our model using simulated diffraction patterns of pharmaceutical mixtures with varying component ratios and compared its performance against conventional search/match methods. Our results demonstrate that the AI model significantly improves the accuracy of component identification. Furthermore, the application of transfer learning played a crucial role in enhancing the model's ability to generalize to new data.

At the conference, we will present the methodology, model development, and evaluation results, highlighting the advantages of using ViT for PXRD-based pharmaceutical analysis.

Keywords: AI-based phase identification, Vision Transformer, APIs, excipients, amorphous

PPXRD-18 Pharmaceutical Powder X-ray Diffraction Symposium

Organic Crystal Structure Prediction: What is Needed for Confident Prediction of Polymorphs?

Sarah Price, University College London, UK, s.l.price@ucl.ac.uk

Computational crystal structure prediction (CSP) codes seek to find the most thermodynamically stable crystal structure from the molecular diagram. This has been so successful that CSP is now widely used in industry.[1, 2] Indeed, in principle, CSP could be the first stage in the digital design of the crystallisation process though using the predicted crystal structure to estimate the morphologies, growth rates and solubilities in different solvates.[3] However, polymorphism considerably complicates our understanding of crystallisation. Most metastable polymorphs are also generated in a CSP study as local minima, and indeed a CSP generated structure can provide a starting point for solving structures from powder diffraction or solid state NMR data. However, a CSP study based on lattice energies usually suggests that there could be far more polymorphs than are actually found.[4, 5] This lecture will illustrate the challenges and progress towards developing an understanding of crystallisation that could be predict the polymorphs of an organic molecule. [6]

Keywords: Organic Polymorphs; Crystal Structure Prediction; Computational modelling; Crystallization; Nucleation

References

1. Hunnisett, L. M. et al. The seventh blind test of crystal structure prediction: structure generation methods. *Acta Cryst. B* **2024**, *80* (6), 517-547. doi:10.1107/S2052520624007492
2. Hunnisett, L. M.. The seventh blind test of crystal structure prediction: structure ranking methods. *Acta Cryst. B* **2024**, *80* (6), 548-574. doi:10.1107/S2052520624008679
3. Burcham, C. L.; Doherty, M. F.; Peters, B. G.; Price, S. L.; Salvalaglio, M.; Reutzel-Edens, S. M.; Price, L. S.; Addula, R. K. R.; Francia, N.; Khanna, V.; et al. Pharmaceutical Digital Design: From Chemical Structure through Crystal Polymorph to Conceptual Crystallization Process. *Crystal Growth & Design* **2024**, *24* (13), 5417-5438. doi: 10.1021/acs.cgd.3c01390.
4. Price, S. L. Control and prediction of the organic solid state: a challenge to theory and experiment. *Proc Roy Soc A* **2018**, *474* (2217), 20180351. doi: 10.1098/rspa.2018.0351.
5. Price, S. L. Why don't we find more polymorphs? *Acta Cryst.* **2013**, *69*, 313-328. doi: 10.1107/S2052519213018861.
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PPXRD-18 Pharmaceutical Powder X-ray Diffraction Symposium

Molecular Crystal Structure Prediction and Comparison

Erin R. Johnson, Department of Chemistry, Dalhousie University, Halifax, NS, Canada and Yusuf Hamied Department of Chemistry, University of Cambridge, Cambridge, UK, erin.johnson@dal.ca

Accurate and efficient computation of relative energies of molecular crystal polymorphs is of central importance for solid-state pharmaceuticals and in development of new molecular materials. In this talk, I will discuss results for the crystal structure prediction (CSP) blind tests obtained using hybrid density functionals paired with the exchange-hole dipole moment dispersion correction. I will also present our variable-cell powder difference (VC-PWDF) method, which allows rapid and reliable identification of matches to experimental polymorphs from computed crystal-energy landscapes. VC-PWDF has also been extended to allow direct comparison of collected powder X-ray diffractograms of unknown polymorphs to both experimental and computationally generated crystal structures. This method is shown to correctly identify the most similar crystal structure to both moderate- and low-quality experimental diffractograms for a set of 7 representative organic compounds and should allow rapid identification of new polymorphs from solid-form screening studies, without requiring single-crystal analysis.

Keywords: Crystal structure prediction; density-functional theory; powder X-ray diffraction

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PPXRD-18 Pharmaceutical Powder X-ray Diffraction Symposium

The Synergy of Computational Modeling, Machine Learning, and Experiments in Pharmaceutical Solid-State Research and Development

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Computational modeling and machine learning (ML) have achieved recognition as vital technologies in drug discovery. Using these technologies, XtalPi has developed a series of methods to guide and enhance experimental workflows for drug development:

- Virtual multicomponent screening to predict the likely salts, cocrystals, and solvates of a compound and recommend solvents for formation experiments. These virtual screenings can be used to reduce the number of wet-lab experiments performed and increase the likelihood of generating the desired result.
- Crystal structure prediction (CSP) predicts all possible polymorphs of a compound and ranks them by thermodynamic stability. The CSP structure-energy landscape can reveal if the most stable polymorph has been discovered and complements experimental XRPD, SC-XRD, and MicroED techniques for crystal structure determination.
- AI-enhanced crystallization (Xtal^2) is a machine learning model—constructed from more than 100k virtual and 10k experimental data—used to recommend crystallization strategies based on molecular structure information. Combining Xtal2 with autonomous workstations allows intelligently designed experiments to be run 24x7.
- Morphology prediction calculations reveal how variables like solvent and additives affect the particle shape of a crystallized compound. These calculations reveal if an undesirable morphology (e.g. needles) is expected and can be avoided by crystallization conditions, or if engineering solutions (e.g. milling) will be required.

Keywords: CSP, computational modeling; polymorphs; particle morphology; automation; ML

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Structure Solution of Sulphonamides from Powder Diffraction Data – A Problematic Moeity?

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Sulphonamides and sulphones frequently exhibit anomalously long bond-lengths after Density Functional Theory (DFT) optimizations integrated into an otherwise highly effective structure solution workflow for powder data. Hypervalent sulphur appears to be problematic as targeted Mogul geometry checks often flag bonds surrounding S(VI) as suspicious. Results from periodic DFT are also highly sensitive to the quality of the pseudopotentials, adding another variable into the pot.

Many common DFT functionals handle simultaneous weak and strong interactions poorly. Higher-order meta-GGA DFT functionals are more effective at modelling complex bonding environments, whilst less resource and time-intensive than higher level "hybrid" functionals.

Meta-GGA SCAN-rVV10 is tested in CUDA-accelerated Quantum Espresso (QE) with a series of high-quality RT SXD sulphonamide structures. Figure 1 shows average S=O length from five sulphonamides from SXD results, and optimized structures via different dispersion-corrected functionals, including SCAN-rVV10.

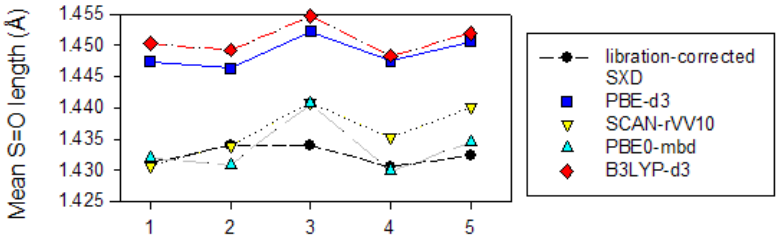

Figure 1. Mean S=O bond distances from the SXD CIF files and a selection of DFT functionals.

Real-world problems? The ICDD Pharmaceuticals Project includes sulphonamides, so matching structure and synchrotron data are available, along with a recent literature RT SXD structure. With SCAN-rVV10 in Quantum Espresso, S(VI) bond lengths for tamsulosin hydrochloride pass Mogul geometry tests at 2σ level (only fractionally > 1σ for S-N). These include those flagged as suspect after CRYSTAL14 DFT optimization in the 2021 ICDD paper[1] (Table 1). Further details of methodologies and results will be presented.

	DFT wall-time (mins)	RMS non-H bond lengths vs SXD (Å)	S=O length < 1σ / < 2σ	S-N length < 1σ / < 2σ	R_{wp}
CRYSTAL14 (1)	~34560 (CPU)	0.02882	× / ×	× / ×	0.0925
PBE-d3 (QE)	156	0.02031	× / ✓	× / ✓	0.0830
B97-3c (QE)	124	0.02561	✓ / ✓	× / ×	0.0862
SCAN-rVV10 (QE)	874*	0.01777	✓ / ✓	× / ✓	0.0817
PBE0-mbd (QE)	2678*	0.01960	✓ / ✓	✓ / ✓	0.0831

Table 1. Summary of results for tamsulosin hydrochloride. Pass/fail compared to mean values via Mogul geometry check. (* two-step using PBE-d3 coordinates)

Keywords: structure solution; density functional theory; complimentary techniques

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Crystallization of Molecular Glass: From Amorphous State to New Polymorphic Forms of Sodium Naproxen

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The solid form of an active pharmaceutical ingredient (API) is usually governing its material properties such as solubility or tableting characteristics. Several strategies have been put in place to improve solubility as this is often a major issue. Naproxen is a poorly soluble API, but its solubility can be improved by synthetizing corresponding sodium salt. Another alternative to improve solubility is to stabilize an amorphous state [1], however it has been shown that naproxen is a typical non glass former [2].
For ceramic materials, it has been demonstrated that crystallization from melt can be a successful approach [3] to stabilize new polymorphic forms of technological relevance. In this contribution, we have been applying a similar approach investigating sodium naproxen as an example. When heating up sodium naproxen above its melting point, various phases can be stabilized upon cooling at room temperature depending on the heat treatment: a new polymorph, or a glass phase. This is in significant contrast to behavior of pure naproxen. In Figure 1 the temperature dependence of this substance is shown as function of temperature. Upon consecutive heating, the initial amorphous state (1) evolves towards a second amorphous state (2) before recrystallizing into previously unreported crystalline polymorphic forms of sodium naproxen (regions (3) and (4)). For instance, the crystal structure present in the region (4) of the isoline plot can be stabilized at room temperature and varies from the initially reported crystal structure of sodium naproxen [4] only by the orientation of the methoxy group (Figure 1 b).

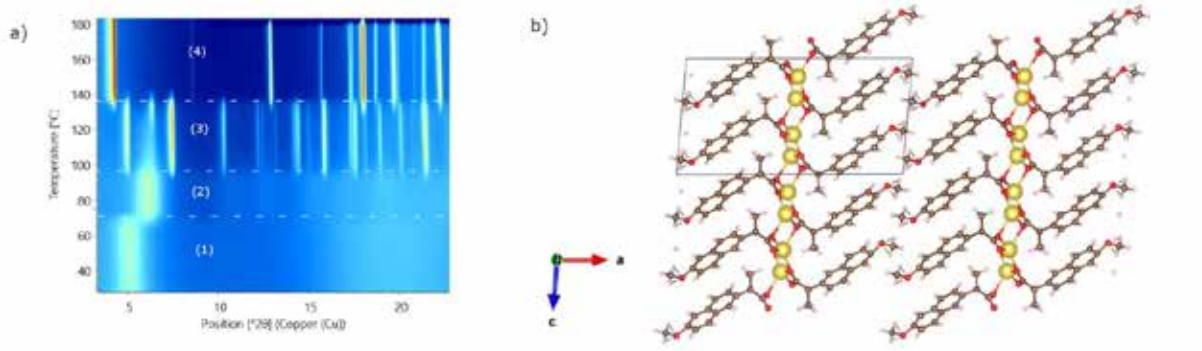

Figure 1. a) Isoline plot of the recrystallization of amorphous sodium naproxen as function of temperature and b) crystal structure of the new polymorph of sodium naproxen appearing at high temperature (section 4).

While molecular glasses are investigated to attempt stabilizing amorphous phases of APIs with the goal of bioavailability-enhancing formulations, this work demonstrates that their crystallization can be an alternative route for the stabilization of new polymorphic forms of this poorly soluble API.

Keywords: small molecule pharmaceuticals; X-ray diffraction; polymorphism; amorphous API, solubility

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Exploring High-Throughput Synchrotron X-ray Powder Diffraction for Quantitative Phase Analysis of Pharmaceutical Mixtures

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Industrial X-ray powder diffraction (XRPD) measurements at synchrotron facilities (S-XRPD) are primarily employed for research and development as well as advanced troubleshooting. This is largely due to the operational complexity, limited accessibility, and higher costs associated with synchrotron experiments compared to laboratory-based instrumentation. However, high-throughput synchrotron XRPD systems are poised to bridge this gap by offering a compelling alternative that combines the benefits of synchrotron-quality data with increased efficiency and reduced costs.

Crucially, high-throughput S-XRPD extends beyond rapid and automated data collection—it also incorporates streamlined sample preparation and efficient data analysis, significantly enhancing overall workflow efficiency. We have recently demonstrated that high-throughput data acquisition, requiring only a few seconds, yields XRPD data of a quality comparable to, if not superior to, the best laboratory-based XRPD data collected over more than 10 hours[1]. While the accessibility of laboratory XRPD ensures its continued role as an invaluable in-house technique, the increasing availability of high-throughput synchrotron XRPD may soon position it as a powerful and complementary tool.

In this presentation, we explore the applications of high-throughput S-XRPD in pharmaceutical characterization, extending beyond high-resolution qualitative phase analysis to quantitative phase assessments (QPA). We address key experimental parameters that must be optimized to achieve robust quantification, discuss the expected accuracy of QPA using high-throughput systems, and compare these results with state-of-the-art quantification methodologies. We will present our latest advancements, highlight the progress achieved, and outline ongoing developments in this rapidly evolving field.

Keywords: Synchrotron X-Ray Powder Diffraction; Quantitative Phase Analysis; High-Throughput; Pharmaceuticals; Structural Analysis

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PPXRD-18 Pharmaceutical Powder X-ray Diffraction Symposium

High-Throughput Synthesis & Powder XRD Screening of Pharmaceutical Solid Forms with Modular Multiwell Devices

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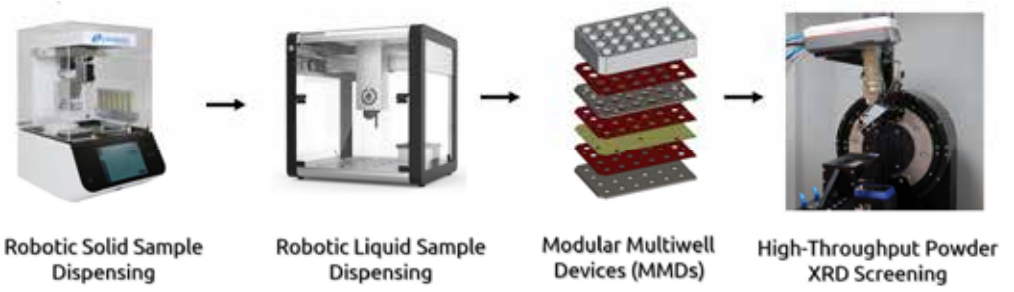

The rapid discovery of new materials requires advancements in automation tools and high-throughput experimental methods to complement the growing capabilities of digital tools such as artificial intelligence and machine learning. While solution-based chemical synthesis has benefited from flow systems coupled with in-situ spectroscopic analysis, solid-state materials present unique challenges in accelerating synthesis and characterization.

To address this gap, we have developed a series of patented Modular Multiwell Devices (MMDs) and associated methodologies that significantly enhance the speed of synthesizing and screening solid-state crystalline materials, including pharmaceutical solid forms, using powder X-ray diffraction (XRD). These devices streamline characterization for solution-based methods, such as reaction crystallization, while also enabling parallel synthesis in 24-, 48-, and 96-well plate formats for solid-state methods, including mechanochemical transformations and melt crystallization, followed by in-situ powder XRD analysis.

These versatile tools accelerate traditional time-consuming processes, with broad applications in drug discovery and materials science. By expediting the identification of novel materials, MMDs hold significant promise for integration into fully automated workflows, further advancing scientific and technological innovation. Their effectiveness will be demonstrated through the synthesis of pharmaceutical co-crystals, rapid powder XRD characterization, and cluster analysis in JADE.

Keywords: High-Throughput XRD, Pharmaceutical Solid Forms, Solid-State Chemistry, Reaction Crystallization, Mechanochemistry, Melt Crystallization, In-situ Characterization

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PPXRD-18 Pharmaceutical Powder X-ray Diffraction Symposium

Advantages of Curated Subfiles in Pharmaceutical Analysis

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The phase identification of pharmaceutical specimens using powder X-ray diffraction (PXRD) can be challenging due to various factors such as peak overlap, preferred orientation, specimen transparency, active pharmaceutical ingredient (API) in minor weight fractions and the presence of non-crystalline phases (Fawcett et. al, 2019). The International Centre for Diffraction Data's (ICDD®) Powder Diffraction File™ (PDF®) has been the gold standard for materials characterization using PXRD for several decades (Kabekkodu et. al, 2024). The PDF-5+ 2025 database contains more than one million entries. It is expected to come across false positives during a search/match phase identification carried out on a million entries without using proper database search filters. For pharmaceutical analysis, curated subfiles (such as Bioactive, Pharmaceuticals, Minerals, etc.) play a vital role in minimizing false positives during search/match. ICDD's editors and member scientists with specific field expertise continuously review these subfiles to maintain their quality. Pharmaceutical specimens often contain non-crystalline phases and the availability of raw PXRD data in PDF-5+ is essential in carrying out in-depth phase identification. Continued growth of pharmaceutical and related subfiles in each release of PDF-5+ is maintained by targeted data acquisition, including raw data. ICDD's editorial process employs rigorous data curation and structural and chemical classifications to optimize pattern search/match and characterization methods. Every entry in the Powder Diffraction File has an editorially assigned quality mark. An editorial comment will describe the reason an entry does not meet the top-quality mark. This presentation will have two parts: The first part will emphasize the various aspects of the editorial process, data curation, results of cluster analysis and the second part will demonstrate the features of PDF-5+ that are beneficial in phase identification of pharmaceuticals.

Keywords: Phase Identification; Powder Diffraction File; Raw Data; Data Curation; PDF-5+

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Crystal Structures of Large-Volume Commercial Pharmaceuticals

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As part of a continuing project, the room-temperature crystal structures of 17 commercial pharmaceutical APIs have been solved and refined using synchrotron X-ray powder diffraction data (11-BM at APS and Wiggler Low Energy Beamline at CLS), and optimized using density functional techniques. These include: delamanid $C_{25}H_{25}F_3N_4O_6$ (Deltyba®), diroximel fumarate $C_{11}H_{13}NO_6$ (Vumerity®), sparsentan $C_{32}H_{40}N_4O_5S$ (Filspari®), trametinib dimethyl sulfoxide $C_{26}H_{23}FIN_5O_4(C_2H_6OS)$ (Mekinist®), etrasimod $C_{26}H_{26}F_3NO_3$ (Velsipity®), anisomycin $C_{14}H_{19}NO_4$, iprodione $C_{13}H_{13}Cl_2N_3O_3$ (Rovral®), flumethasone $C_{22}H_{28}F_2O_5$, givinostat hydrochloride monohydrate Form I $C_{24}H_{28}N_3O_4Cl(H_2O)$ (Duvyzat™), aprocitentan Form A $C_{16}H_{14}Br_2N_6O_4S$ (Tryvio™), ethynodiol diacetate $C_{24}H_{32}O_4$ (Ovulen), repotrectinib $C_{18}H_{18}FN_5O_2$ (Augtyro™), fruquintinib Form I $C_{21}H_{19}N_3O_5$ (Fruzaqla®), quizartinib hydrate $C_{29}H_{32}N_6O_4S(H_2O)_{1/3}$ (Vanflyta®), cabotegravir $C_{19}H_{17}F_2N_3O_5$ (Vocabria), pirtobrutinib Forms 1 and 2 $C_{22}H_{21}F_4N_5O_3$ (Jaypirca®), and protriptyline hydrochloride $C_{19}H_{22}NCl$ (Vivactil®). Other new structures may be presented as they become available.

Keywords: crystal structure; Rietveld refinement; density functional theory

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EXPLORING THE ROLE OF NON-AMBIENT X-RAY DIFFRACTION (XRD) IN THE PHARMACEUTICAL INDUSTRY

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Nishil Malde, Anton Paar Ltd., United Kingdom

This abstract is not available for online viewing. It will be available in the printed program.

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Use of Component Analysis and Direct Derivation Methods to Access Phase Behavior in Amorphous Solid Dispersions

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With the increasing molecular complexity of active pharmaceutical ingredients (API), the use of amorphous solid dispersions (ASD) to enhance bioavailability and manufacturability continues to grow. While the importance of ASD formulations increases, the challenges of characterizing the solid-phase behavior remain. For example, how much of the API is fully dispersed at a molecular level, and how does this change over time or through using different manufacturing conditions. More importantly, how do these solid-phase changes impact the re-crystallization propensity and bioavailability of the drug.

The most frequently utilized ASD formulations make use of API solubility into various polymers to achieve molecular level dispersions. The presence of any specific drug-polymer interactions will likely maintain this molecularly dispersed solid-phase over time despite the general metastability of ASDs. This makes the stability and effectiveness of ASDs very polymer specific. Previous studies have evaluated the compatibility between APIs and different polymers using XRD, solid-state NMR, FT-IR, etc., but the information obtained from these general methods is typically limited and not suitable for a QA/QC method.

In this presentation, we will discuss a novel Component Analysis and Direct Derivation approach to qualitative and quantitative solid-phase characterizations of ASDs. The benefits of this approach for a QA/QC evaluation of production material will be demonstrated utilizing the binary ASD of indomethacin and polyvinylpyrrolidone (PVP).

Keywords: Amorphous solid dispersions; Direct Derivation method; Component analysis

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Matching ROY Crystal Structures to High Throughput PXRD

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The ability of a compound to form different crystalline structures is known as polymorphism, where polymorphs will possess different chemical and physical properties. To identify the isolable polymorphs of a compound, extensive experimental screening of crystallization conditions is often carried out in a high throughput fashion, where only powder X-ray diffractograms (PXRD) are obtainable. To identify if a particular solid form is a new or pre-existing polymorph, the room-temperature PXRD must be compared to low temperature single crystal X-ray structures, such as from the Cambridge structural database (CSD). This comparison is problematic because the diffractogram peak positions shift substantially with temperature. To address this challenge, the variable-cell experimental powder difference (VC-xPWDF) method was developed and allows for reliable comparison of experimental PXRD to simulated diffractograms of known crystal structures. This work assesses the performance of VC-xPWDF for high-throughput polymorph screening for the test case of 5-Methyl-2-[(2-nitrophenyl)amino]-3-thiophenecarbonitrile, also known as ROY, which is a prolific polymorph former. The method is shown to be successful for comparison of PXRD to both experimental crystal structures from the CSD and computationally generated structures obtained from Beran's previous crystal structure prediction study. The experimental PXRD quality was shown to not affect the results significantly, except for errors occurring due to preferential orientation, which could potentially be reduced by some minimal grinding of the samples prior to making the PXRD measurements. Overall, this work demonstrates the utility of VC-xPWDF to solve crystal structures from PXRD data generated during high-throughput polymorph screening.

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Driving XRD in Regulated Environments

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This abstract is not available for online viewing. It will be available in the printed program.

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DIFFRAC.EVA, a Comprehensive Toolset for Characterizing Pharmaceutical Materials

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Powder X-ray diffraction (PXRD) is a cornerstone technique in pharmaceutical research, playing a critical role in drug discovery and development. Its applications span phase identification, structure solution and refinement, crystallinity assessment, crystallite size determination, amorphous phase characterization, in-situ monitoring of phase transformations or chemical reactions, and high-throughput screening. PXRD can analyze a variety of sample forms, including solids, liquids, gels, and powders.

DIFFRAC.EVA, a comprehensive software toolset available with each Bruker XRD system, including a 21 CFR Part 11 compliant version, has seen significant advancements in recent years. This presentation will highlight the latest features and improvements of DIFFRAC.EVA, focusing on:

- **Automated Phase Identification**: Enhancing the efficiency and accuracy of identifying different phases within a sample.
- **Quantitative Phase and Microstructure Analysis**: Providing detailed insights into the composition and microstructural properties of pharmaceutical materials.
- **Automated Data Evaluation Using Workflows**: Streamlining the data analysis process through customizable workflows, reducing manual intervention and increasing reproducibility.
- **Analysis of Pair Distribution Functions (PDF)**: Offering advanced capabilities for studying the local structural environment of materials.
- **Use of Databases Such as PDF5+**: Leveraging extensive databases for phase identification, quantification, and data mining, facilitating comprehensive and accurate analysis.

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Transmission XRPD for the Unravelling of Complex Solid Form Landscapes

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Solid form landscape understanding is critical to deliver a stable, phase-pure and efficacious active pharmaceutical ingredient (API) in the final drug product. This study shows two compounds where transmission X-Ray Powder Diffraction (XRPD) delivered important understanding of the solid form landscape during drug substance development.

For example, Form 2 of compound A shows a shoulder in transmission XRPD that was previously not observed in reflection mode XRPD. This was identified as being associated with a solvate that formed during the crystallisation process. Drying studies of compound A using transmission XRPD showed that two solvates could nucleate in the crystallisation process, one which desolvates to the target Form 2 and one which desolvates to another undesired solid form. This understanding delivered the design space where the desired Form 2 anhydrate will be isolated phase pure.

Compound B is delivered as a hydrated form. Drying of the material could produce the anhydrous form of compound B. Transmission XRPD was used, alongside the crystal structures of the hydrated and anhydrous forms, to estimate the amount of anhydrate produced during certain drying conditions using Rietveld Refinement (RR). Spikes of anhydrate in the hydrated form were prepared and excellent agreement between the theoretical and RR calculated amounts of the solid forms was found, validating the estimations of form content in the isolated material.

This work shows the value of transmission XRPD to deliver understanding and robust control to the solid form landscape during drug substance development.

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Detect and control polymorphism: quick and efficient solid form analysis

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Aeris, a compact XRD system designed to make solid-state analysis simple and efficient. Its plug-and-play simplicity makes it accessible to even non-crystallographers. Aeris delivers fast, high-quality data – providing a full pattern within five minutes – with exceptional sensitivity for detecting low impurity quantities.
1Der, the high energy resolution detector improves detection limits for polymorphic contaminants, while reducing background level caused by air scattering or coming from fluorescent elements like iron or lanthanum that can sometimes be present in drug substances and/or products.
Further simplification of XRD analysis can be supported by the analytical software package HighScore Suite. It allows you to run various types of analysis starting from pattern matching to limit testing, quantification of multiple amorphous and crystalline phases, and cluster analysis to identify similar XRD patterns and hence predict which polymorphs are likely to have comparable physical properties (solubility, bioavailability, etc.) and crystallinity/amorphicity assessment. Due to the Smart Batches feature most of this information can be extracted in an automated or semi-automated way, providing push-button solutions for a quick, yet reliable analysis to anyone from chemists to crystallographers – independent of the depth of XRD knowledge and experience.
Finally, OmniTrust solution makes working in regulated environments easy and time efficient. OmniTrust allows you to restrict access according to user profiles and it provides a reliable and comprehensive way of accessing and reviewing system audit trail data.

Keywords: small molecule pharmaceuticals; X-ray diffraction; polymorphism; detection limits; regulated environment

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Advanced Characterization of Lipid Nanoparticles in Formulation Development Using X-Rays Scattering

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The past decade has seen the accelerated development of lipid nanoparticles (LNP) in the vaccine and therapeutic areas of medicine. Following the success of the first FDA-approved LNP based drug for the treatment of transthyretin-induced amyloidosis (Onpattro®, 2018), the two mRNA-LNP vaccines have emerged as breakthrough solutions in the fight against Covid-19 (Pfizer/BioNTech, 2021 and Moderna, 2022) [1,2]. Despite their success, the nanostructure of these complex multi-component delivery systems remains poorly understood.

Several conceptual models have been proposed to explain the distribution of RNA inside the LNP. Such models include encapsulation of RNA into inverted micelles; in between the bilayers in a multilamellar core; or into amorphous lipid matrix, etc. [1-4]. Among the variety of methods that were used to address this problem, the studies involving characterization of LNPs using small-angle X-ray and neutron scattering (SAXS, SANS) have been the most informative [3,4]. SAXS is a powerful technique for elucidating the nanostructure of LNPs, offering insights into their size, shape, stability and phase behavior. LNPs can be studied using a benchtop laboratory instrument, or with higher resolution using a synchrotron light source. However, the data interpretation is more challenging and requires an extensive set of samples, along with the application of complementary techniques, such as cryo-electron transmission microscopy (cryo-TEM).

In this study, we explore how formulation conditions, composition, and methods influence the structure of LNPs. Utilizing a robust set of SAXS data we compared the properties of LNPs to their efficacy *in vitro* and *in vivo.* Moreover, cryogenic transmission electron microscopy (cryo-TEM) provided insights into particle size distributions and the presence of structural irregularities. By employing these advanced methods, we demonstrated that the size of RNA and formulation methods are crucial parameters of the structural arrangement of LNPs. The ability to link structural characteristics with functional outcomes facilitates the rational design of LNPs to enhance therapeutic efficacy.

Keywords: LNP; structure; formulation; SAXS; cryo-TEM

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PPXRD-18 Pharmaceutical Powder X-ray Diffraction Symposium

Small-Angle X-Ray Scattering as Powerful Tool for Phase and Crystallinity Assessment of Monoclonal Antibody Crystallites in Support of Batch Crystallization

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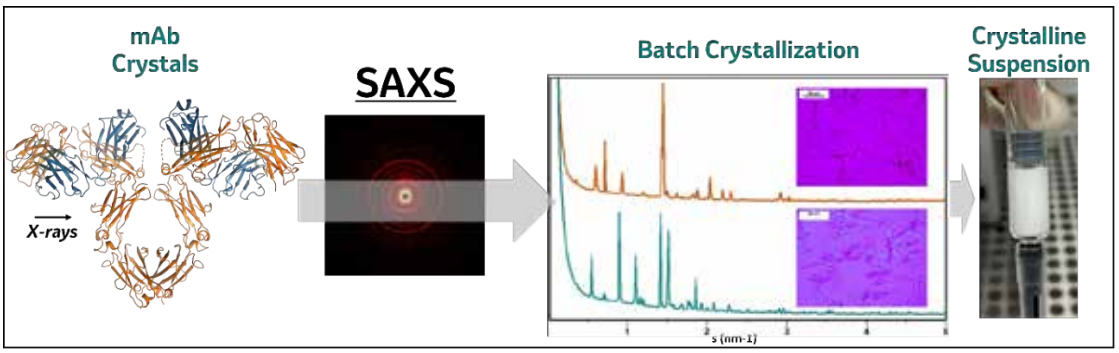

Crystalline suspensions of monoclonal antibodies (mAbs) have great potential to improve drug substance isolation and purification at a large scale and to be used for drug delivery via high concentration formulations. Crystalline mAb suspensions are expected to have enhanced chemical and physical properties relative to mAb solutions delivered intravenously, making them attractive candidate for subcutaneous delivery. In contrast to small molecules, development of protein crystalline suspensions is not a widely used approach in the pharmaceutical industry. This is mainly due to the challenges in finding crystalline hits and the suboptimal physical properties of the resulting crystallites when hits are found. Modern advances in instrumentation and increased knowledge in mAb crystallization have, however, resulted in higher probabilities of discovering crystal forms and improving their particle properties and characterization. In this regard, physical analytical characterization plays a central role in the initial steps of understanding and later optimizing the crystallization of mAbs and requires careful selection of the appropriate tools. This contribution describes a novel crystal structure of the antibody pembrolizumab and demonstrates the usefulness of Small Angle X-Ray Scattering (SAXS) for characterizing its crystalline suspensions. It illustrates the advantages of SAXS when used to (i) confirm crystallinity and crystal phase of crystallites produced in batch-mode; (ii) confirm crystallinity under various conditions and detect variations in crystal phases, enabling fine-tuning of the crystallizations for phase control across multiple batches; (iii) monitor the physical response and stability of the crystallites in suspension with regards to filtration and washing; and (iv) monitor the physical stability of the crystallites upon drying. Overall, this work highlights how SAXS is an essential tool for mAb crystallization characterization.

Keywords: Small-angle x-ray scattering, monoclonal antibody, crystalline suspension, crystal phase, protein crystal, pembrolizumab, batch crystallization

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Larpent, P.; Codan, L.; Bothe, J. R.; Iuzzolino, L.; Pabit, S.; Gupta, S.; Fischmann, T.; Su, Y.; Reichert, P.; Stueber, D.; Cote, A., Small-Angle X-ray Scattering as a Powerful Tool for Phase and Crystallinity Assessment of Monoclonal Antibody Crystallites in Support of Batch Crystallization. Mol Pharm 2024, 21 (8), 4024-4037.
doi :10.1021/acs.molpharmaceut.4c00418

PPXRD-18 Pharmaceutical Powder X-ray Diffraction Symposium

Advancing Electron Diffraction for Beam-Sensitive Materials: Current Insights & Emerging Techniques

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Ab initio structure solution from single-crystal X-ray diffraction data is generally limited by the requirement for crystals that are at least a few micrometers in size. Powder diffraction inherently provides limited information and requires many randomly oriented crystals. 3D Electron Diffraction (3DED) / MicroED enables structure determination from individual nanometer-sized (50–100 nm) crystals. For successful 3DED data collection from beam-sensitive materials (e.g., COFs, MOFs, pharmaceuticals, etc.), the key parameters are using a lower electron dose (for many active pharmaceutical ingredients (APIs), the critical electron dose is typically below 5 $e^{-}/Å^{2}$) and employing high-sensitivity cameras. This can be achieved with new-generation direct electron detection cameras. Using this methodology, we have successfully solved the structures of several pharmaceuticals, including both known and previously unknown structures [1]. Examples from our previous work will be presented, highlighting different challenges. Data collection can be performed using different protocols, either through stepwise precession or continuous rotation of the crystal, with or without beam precession. In stepwise precession, where the total data collection time is longer, the total electron dose can be controlled by using beam blanking between tilting steps of the crystal.

3DED data collection is typically performed using standard TEM grids. Additionally, during 3DED/MicroED experiments, crystals can be encapsulated between two graphene layers to enhance stability and reduce radiation damage. A few examples from our recent study involving pharmaceuticals with known structures, such as carbamazepine and trimethylamine N-oxide dihydrate, will be presented.

Furthermore, we are developing a Serial Electron Diffraction data collection approach that involves collecting electron diffraction data from multiple crystals, obtaining one diffraction pattern per crystal, and merging the datasets for structure solution. With this approach, it would be possible to avoid tilting of crystals, thereby minimizing the electron dose per crystal. As proof of concept, an example using a structure with a known unit cell will be presented, demonstrating how this method can be extended to unknown structures with previously undetermined unit cells.

Keywords: Electron Diffraction, Pharmaceuticals, Graphene

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PPXRD-18 Pharmaceutical Powder X-ray Diffraction Symposium

Measurement of Dose in Mixed Gamma and Neutron Fields by X-Ray Diffraction in Alanine)

Omololu O. Makinde, **Urvi Jain**, **Dr. Nathan D. Gallant**, **Dr. Nancy Diaz-Elsayed**, University of South Florida, Mechanical Engineering Department, USA, makindeo@usf.edu,

Dr. Thomas V. Holschuh II, **Dr. Michael A. Reichenberger**, Idaho National Laboratory, USA

This abstract is not available for online viewing. It will be available in the printed program.

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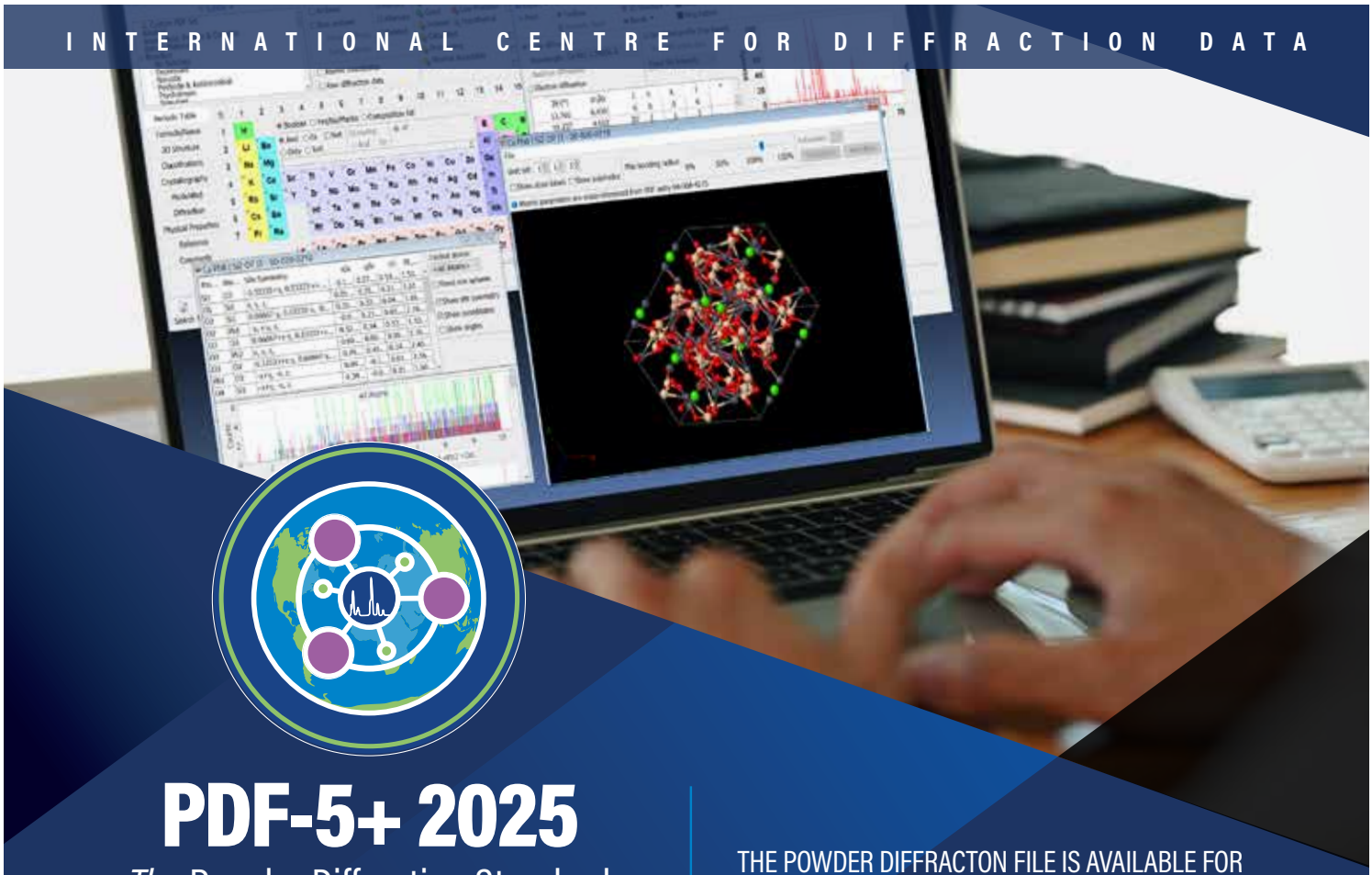

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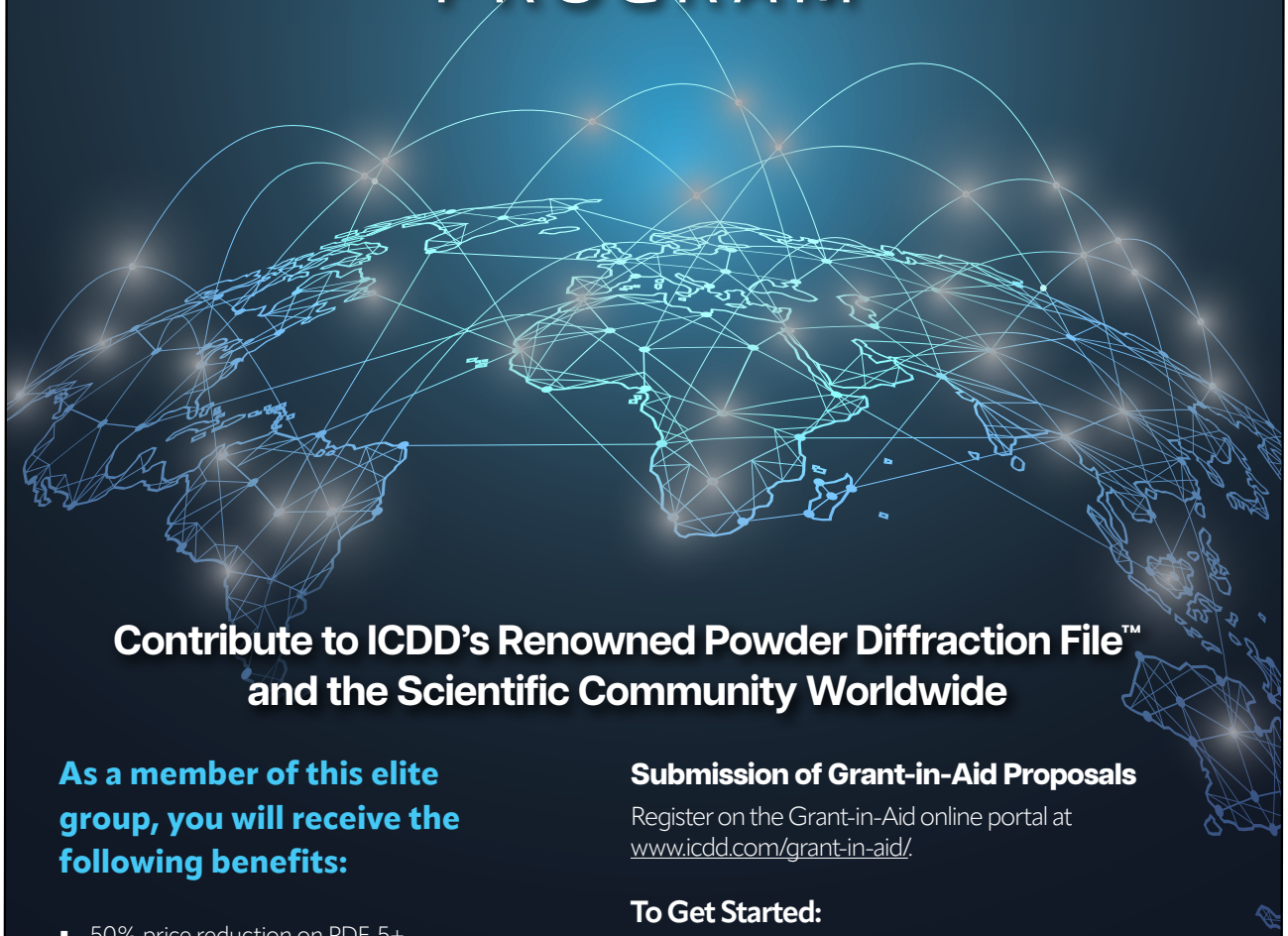

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Notes

PPXRD-18
Pharmaceutical Powder X-ray Diffraction Symposium

6 - 9 May 2025
The Cambridge Crystallography Data Centre (CCDC)
Cambridge, United Kingdom

PPXRD-18 Program-at-a-Glance

DAY	TIME	EVENT
Tuesday 6 May Workshop	8:30am	Attendee Check-in
	9:00am	*Health and Safety Presentation*; **CCDC Staff**
	9:05am	Workshop: ICDD Database and Software; Instructors: **Tom Blanton; Suri Kabekkodu**
	10:30am	Coffee Break & Exhibition
	11:00am	Workshop (con't): ICDD Database and Software
	12:30pm	Lunch Break
	1:30pm	Workshop: Investigating Structures Using the CSD; Instructors: **Suzanna Ward; Andrew Peel**
	3:00pm	Coffee Break & Exhibition
	3:30pm	Workshop (con't): Investigating Structures Using the CSD *(ends at 5:00pm)*
Wednesday 7 May Sessions	9:00am	Plenary Session: Intellectual Property; Chair: **Tom Blanton** *Opening Remarks*; Organizing Committee Chair: **Tom Blanton**
	10:30am	Coffee Break & Exhibition
	11:00am	Plenary Session (con't): Intellectual Property
	11:30am	Intellectual Property Round Table
	12:30pm	Lunch
	1:30pm	Pharmaceutical Case Study of PXRD; Chair: **Fabia Gozzo**
	2:10pm	Exhibitor Introductions
	3:10pm	Coffee Break & Exhibition
	3:30pm	Industrial Applications and Machine Learning; Chair: **Simon Bates** *(ends at 5:10pm)*
Thursday 8 May Sessions	9:00am	Structure Solution, Crystal Structure Prediction; Chair: **Shawn Yin**
	10:10am	Coffee Break & Exhibition
	11:00am	Structure Solution, Crystal Structure Prediction (con't)
	12:30pm	Lunch
	1:30pm	Qualitative / Quantitative Analysis, High Throughput XRD Methods; Chair: R**aj Suryanarayanan**
	3:10pm	Coffee Break & Exhibition
	3:30pm	Poster Session & Reception; Chair: **Tom Blanton** *(ends at 5:00pm)*
Friday 9 May Sessions	9:00am	Complex Materials Analysis; Chair: **Natalia Dadivanyan**
	10:10am	Coffee Break & Exhibition
	10:40am	Instrumentation and Complementary Techniques; Chair: **Anisha Patel**
	11:50am	*Closing Remarks*; Organizing Committee Chair: **Tom Blanton** *(Symposium closes at 12:00pm)*

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