PPXRD-15 PROGRAM

Pharmaceutical Powder X-ray Diffraction Symposium

A satellite meeting to the 24th Congress & General Assembly
of the International Union of Crystallography - IUCr 2017

18-20 August 2017, Hyderabad, India

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FRIDAY WORKSHOP

18 August ▪ 08.30-17.30
Hyderabad International Convention Centre ▪ Meeting Rooms G01 & 02

Powder XRD Applications for Pharmaceutical Product Development

Organizers & Instructors:
R. Suryanarayanan, University of Minnesota, USA, surya001@umn.edu
T. Blanton, International Centre for Diffraction Data, USA, tblanton@icdd.com
G. Diaz de Delgado, University de Los Andes, Venezuela, diaz@ula.ve
D. Beckers, PANalytical B.V., The Netherlands, detlef.beckers@panalytical.com

In the context of preformulation studies, X-ray powder diffraction (XRD) has been extensively used to identify different physical forms of drugs and excipients. The application of XRD for pharmaceutical characterization will be described with several representative examples, followed by the use of XRD to identify and characterize multiple crystalline phases in a complex matrix. In drug products, XRD can be used to simultaneously characterize crystalline drugs and excipients and the application of this technique in different dosage forms including tablets and lyophilized solids will be demonstrated. During pharmaceutical processing, there is the potential for processing-induced phase transformations which can have profound implications on the final product performance. As representative examples, phase transitions during the manufacture of tablets and freeze-dried products will be discussed. Recent advances in instrumentation and software have enabled quantitative phase analyses in complex, multicomponent systems with enhanced limits of detection and quantification. Moreover, selective subtraction of the contribution of formulation components (for example, high concentration of excipients) can enhance analyte sensitivity and examples will be provided. The course participants will get a 30 day license of the ICDD PDF-4+ database and associated software for use during the workshop and later evaluation. There will be a demonstration of data mining, phase identification and other relevant searches of interest to the pharmaceutical community.

Schedule

08.30	Opening remarks from Professor **G.R. Desiraju,** Indian Institute of Science, India. Exhibitor Introductions by the Conference Chairman, **T. Blanton**, International Centre for Diffraction Data, USA.
09.00	Fundamentals and Overview of XRD **D. Beckers**, PANalytical B.V., The Netherlands
10.30	Break
11.00	XRD Tools and Capabilities / Characterization of Pharmaceutical Formulations **R. Suryanarayanan**, University of Minnesota, USA
12.30	Lunch
14.00	Data Mining & Phase Identification **T. Blanton**, International Centre for Diffraction Data, USA
15.30	Break
16.00	New Frontiers and Novel Approaches / Combining Structure – Powder XRD **G. Diaz de Delgado**, University de Los Andes, Venezuela
17.30	End

SATURDAY SESSIONS

19 August ▪ 09.00-19.00

Hyderabad International Convention Centre ▪ Meeting Rooms G01 & 02

*Signifies the speaker

09.00 Opening remarks from PPXRD-15 Local Organizing Committee Member, **T.N. Guru Row**, Indian Institute of Science, India

Welcoming remarks from PPXRD-15 Organizing Committee Chairman, **T. Blanton**, International Centre for Diffraction Data, USA

Qualitative Phase Analysis with XRPD

Co-chairs: **D. Beckers**, PANalytical B.V., The Netherlands, detlef.beckers@panalytical.com
F. Gozzo, Excelsus Structural Solutions sprl, Belgium, fabia.gozzo@excels.us

09.15 P39 *Invited:* Structure and Analysis of Amorphous Dispersions
S. Byrn*, **C. Benmore**, **G. deAraujo**, **Y. Song**, **H. Nie**, Purdue University and Argonne National Laboratory, USA

09.45 P32 Phase Identification and Microstructure Characterization of Pharmaceutical Formulations Using Excipient Reference Data in the Powder Diffraction File (PDF®)
T. Blanton*, **S. Gates-Rector**, **T. Fawcett**, International Centre for Diffraction Data, USA

10.05 P38 Application of the Powder Diffraction File™ in Pharmaceutical Analysis
S. Kabekkodu*, **J. Blanton**, **T. Fawcett**, **T. Blanton**, International Centre for Diffraction Data, USA

10.25 Break

Quantitative Phase Analysis: Single Peak, Whole-Pattern Methods and Beyond

Co-chairs: **F. Gozzo**, Excelsus Structural Solutions sprl, Belgium, fabia.gozzo@excels.us
D. Beckers, PANalytical B.V., The Netherlands, detlef.beckers@panalytical.com

11.00 P42 *Invited:* Quantitative Applications of X-ray Powder Diffractometry
R. Suryanarayanan*, University of Minnesota, Minneapolis, MN, USA

11.30 P18 The Appropriate Internal Standard for Pharmaceuticals: The Art of Dealing with Compromises
M.L. Reinle-Schmitt*, **R. Frison**, Excelsus Structural Solutions (Swiss) AG, Switzerland
P.P. Mazzeo, Excelsus Structural Solutions (Swiss) AG, Switzerland and University of Parma, Italy
F. Gozzo, Excelsus Structural Solutions (Swiss) AG, Switzerland and Excelsus Structural Solutions sprl, Belgium

11.50 P21 Quantification of Multiple Amorphous and Crystalline Phases
A. Adibhatla*, PANalytical, Inc., USA
D. Beckers, **T. Degen**, PANalytical B.V., The Netherlands

12.10 P29 Importance of Solid State Characteristics of Impurities in Phase Analysis of Active Pharmaceutical Ingredients
R. Narsimhamurthy*, **M. Sundaram**, **P. Chodon**, **P. Kameshwar**, **A. Dikundwar**, **S. Pal**, **H. Bhutani**, Biocon Bristol-Myers Squibb Research & Development Center, Syngene International Limited, India

12.30 Lunch

Formulation and Product Development

Chair: **T.N. Guru Row**, Indian Institute of Science, India

14.00 *Invited:* Solid Form Screening and Selection: Challenges in Generic Pharmaceutical Development
V. Peddy*, Dr. Reddy's Laboratories Ltd., India

14.30 P31 Use of SXRD to Study Spatial Heterogeneity in Phase Composition in Co-Lyophilized Binary Systems
S. Thakral*, **S. Koranne**, **R. Suryanarayanan**, University of Minnesota, USA

Polymorph, Salt and Co-crystal Screening

Chair: **G. Diaz de Delgado**, University de Los Andes, Venezuela, diaz@ula.ve

14.50 P40 *Invited:* XRD in the Screening and Characterization of Pharmaceutical Co-crystals
A.K. Nangia*, University of Hyderabad and CSIR-National Chemical Laboratory, India

15.20 P44 *Invited:* Why Polymorphism? An Evaluation using Experimental Charge Densities Analysis
T.N. Guru Row*, Indian Institute of Science, India

15.50 Break

Non-Ambient Powder X-ray Diffraction

Chair: **T. Blanton**, International Centre for Diffraction Data, USA

16.20 P46 *Invited:* Non-Ambient Applications of X-ray Diffractometry for the Characterization of Pharmaceutical Systems
R. Suryanarayanan*, University of Minnesota, USA

SATURDAY EVENING POSTER SESSION AND RECEPTION

19 August ▪ 17.00-19.00
Hyderabad International Convention Centre ▪ Meeting Rooms G01 & 02

*Signifies the speaker

P12 Nanocrystal Formation of Docetaxel via an Amorphous Precursor
I. Choi, **J. Ham**, **J.H. Jeong**, **I.W. Kim***, Soongsil University, South Korea
K.S. Kim, BioSynectics, South Korea

P15 High-throughput Screening with a Well Plate Changer
M. Evans, **C. Drathen***, **A. Kern**, **M. Zimmermann**, Bruker AXS GmbH, Germany

P16 STOE STADI P and the Multi-MYTHEN – The Solution if One Dectris MYTHEN 1K is Not Fast Enough
T. Hartmann*, Stoe & Cie GmbH, Germany

P20 X-ray Diffraction Study on Ultrasonically Levitated Droplets
Y.I. Kim*, **K.B. Kim**, **Y.H. Lee**, KRISS, South Korea
K.M. Nam, Mokpo National University, South Korea

P22 Atomic Pair Distribution Function (PDF) and X-ray Scattering Methods to Assess Amorphous Organic Compounds
D. Beckers*, **M. Gateshki**, PANalytical B.V., The Netherlands
A. Adibhatla, PANalytical, Inc., USA

P24 Structural Characterization of Flunixin, a Non-steroidal Anti-inflammatory Drug of Veterinary Use by Powder X-ray Diffraction
M.C. Dávila, **A.J. Dugarte**, **J.E. Contreras**, **J.M. Delgado**, **G. Díaz de Delgado***, Universidad de Los Andes, Venezuela

P25 Detailed Investigation of Crystalline Phases of Pharmaceutical Drugs Found by XRD-DSC by Employing a Thermal Analysis System having a Sample Observation Camera and Raman Spectroscopy
Y. Shiramata*, **A. Miyayama**, **A. Yamano**, Rigaku Corporation, Japan

P28 Synthesis and Characterization by Single Crystal X-ray Diffraction of a New Copper Derivative of Ciprofloxacin
M.A. Ramírez, **J.M. Delgado**, **G. Díaz de Delgado***, Universidad de Los Andes, Venezuela
H.A. Camargo, **J.A. Henao**, Universidad Industrial de Santander, Colombia

P33 Powder Diffraction File™ Coverage of Polymers used in Pharmaceutical and Biomedical Applications
T. Blanton*, **S. Gates-Rector**, **T. Fawcett**, International Centre for Diffraction Data, USA
S. Misture, Alfred University, USA

P34 Crystal Structures of Large-Volume Commercial Pharmaceuticals
J.A. Kaduk*, Illinois Institute of Technology and North Central College, USA
A.M. Gindhart, **T. Blanton**, International Centre for Diffraction Data, USA

P43 Rapid Screening and Manufacturing of Co-crystals via Thermal Ink Jet Printing for Pharmaceutical Applications
S. Raviteja*, **T.N. Guru Row**, Indian Institute of Science, India

SUNDAY SESSIONS

20 August ▪ 09.00-16.10

Hyderabad International Convention Centre ▪ Meeting Rooms G01 & 02

*Signifies the speaker

Amorphous, Mesomorphous and Nano Materials

Chair: **A. Kern**, Bruker AXS GmbH, Germany, arnt.kern@bruker.com

09.00 P13 *Invited:* X-ray Total Scattering of Amorphous, Mesomorphous and Nanocrystalline Materials: The Pharmaceutical Challenge
R. Frison*, Excelsus Structural Solutions sprl, Belgium and University of Zurich, Switzerland
P. Mazzeo, University of Parma, Italy
M. Reinle-Schmitt, Excelsus Structural Solutions (Swiss) AG, Switzerland
A. Cervellino, Paul Scherrer Institut, Switzerland
A. Prodi, Excelsus Structural Solutions sprl, Belgium

09.30 P8 *Invited:* Does an Accurate Modelling of the Peak Shape Matter?
M. Leoni*, DICAM University of Trento, Italy

10.00 P14 Fast PDF Screening of Amorphous Pharmaceuticals
C. Drathen*, **A. Kern**, **M. Evans**, Bruker AXS GmbH, Germany

10.20 Break

Crystal Structure Prediction, Elucidation and Verification from Powder Diffraction Data

Chair: **J. Kaduk**, Poly Crystallography, Inc. & Illinois Institute of Technology, USA, kaduk@polycrystallography.com

10.50 P19 *Invited:* Crystal Morphology Prediction of Structures Determined by X-ray Powder Diffraction
F.F. Ferreira*, Federal University of ABC (UFABC), Brazil
F. Punzo, Università degli Studi di Catania, Italy

11.20 P3 Lessons Learned from 25 Commercial Pharmaceutical Crystal Structure Prediction Studies
J. van de Streek*, **M.A. Neumann**, Avant-garde Materials Simulation, Germany

11.40 P6 Structure Determination and Rietveld Refinement Study of Coordination Polymers
M.A. Haque*, **M.A. Haque**, **L.J. Paliwal**, **U.A. Palikundwar**, Rashtrasant Tukadoji Maharaj Nagpur University, India

12.00 Lunch

New Developments/Complementary Techniques

Chair: **K. Saito**, Rigaku Europe SE, Germany, keisuke.saito@rigaku.com

13.30 P9 *Invited:* A New Method for Quantitative Phase Analysis Using Observed Integrated Intensities and Chemical Composition Data of Individual Crystalline Phases
H. Toraya*, Rigaku Corporation, Japan

14.00 P11 Appropriate Restraint Settings for Crystal Structure Refinement
A. Sasaki*, **H. Konaka**, Rigaku Corporation, Japan

14.20 P23 Characterizing Biological Macromolecules using SAXS on a Multi-Purpose Laboratory X-ray Diffraction System
D. Beckers*, **J. Bolze**, **B. Schierbeek**, PANalytical, B.V., The Netherlands
A. Adibhatla, PANalytical, Inc., USA

14.40 Break

15.10 P27 TEM 3D Precession Electron Diffraction Tomography to Solve Pharmaceutical API Structures
P.P. Das*, **S. Nicolopoulos**, NanoMEGAS sprl, Belgium

15.30 P30 Strengthening of Powder Pattern Information with Complementary Crystal Structure Determination Methods
C.W. Lehmann*, Max-Planck-Institut fuer Kohlenforschung, Germany

15.50 Summary and Closing Remarks

16.10 End of Symposium

PPXRD-15 Program-at-a-Glance

Workshop, Sessions and Exhibits will be held at the Hyderabad International Convention Centre
Meeting Rooms G01 & 02

Friday August 18	08:30-10:30	Workshop: Powder XRD Applications for Pharmaceutical Product Development
	10:30-11:00	Break
	11:00-12:30	Workshop continued
	12:30-14:00	Lunch
	14:00-15:30	Workshop continued
	15:30-16:00	Break
	16:00-17:30	Workshop continued
Saturday August 19	09:00-10:25	Session: Qualitative Phase Analysis with XRPD
	10:25-11:00	Break
	11:00-12:30	Session: Quantitative Phase Analysis: Single Peak, Whole-Pattern Methods and Beyond
	12:30-14:00	Lunch
	14:00-14:50	Session: Formulation and Product Development
	14:50-15:50	Session: Polymorph, Salt and Co-crystal Screening
	15:50-16:20	Break
	16:20-16:50	Session: Non-Ambient Powder X-ray Diffraction
	17:00-19:00	Evening Poster Session and Reception
Sunday August 20	09:00-10:20	Session: Amorphous, Mesomorphous and Nano Materials
	10:20-10:50	Break
	10:50-12:00	Session: Crystal Structure Prediction, Elucidation and Verification from Powder Diffraction Data
	12:00-13:30	Lunch
	13:30-14:40	Session: New Developments/Complementary Techniques
	14:40-15:10	Break
	15:10-15:50	Session: New Developments/Complementary Techniques - continued
	15:50-16:10	Summary and Closing Remarks