

The 13<sup>th</sup>

# PHARMACEUTICAL POWDER X-RAY DIFFRACTION SYMPOSIUM

*XRD Training  
for the  
Pharmaceutical  
Scientist!*



# PROGRAM

**18-21 May 2015**

**Black Forest Panorama  
Schwarzwald Panorama,  
Bad Herrenalb, Germany**



*Sponsored by*



## WORKSHOP Monday (FULL DAY)

8:30AM – 5:00PM

### Quantitative Phase Analysis by XRPD

**Organizer & Instructors:**

**Arnt Kern\***, Bruker AXS GmbH, Karlsruhe, Germany, arnt.kern@bruker-axs.de

**Detlef Beckers**, PANalytical, Almelo, The Netherlands, detlef.beckers@panalytical.com

**Fabia Gozzo**, Excelsus Structural Solutions SPRL, Brussels, Belgium, fabia.gozzo@excelsus.us

**Robert Dinnebier**, Max-Planck Institute für Festkörperforschung, Stuttgart, Germany, r.dinnebier@fkf.mpg.de

**Arnaud Grandeur**, Novartis Pharma AG, Basel, Switzerland, arnaud.grandeur@novartis.com

This workshop will discuss the basis of quantitative phase analysis (QPA) for both single peak and whole pattern approaches. The focus will be on how to select and apply these methods, particularly in the context of the derivation of absolute phase abundances. The presence of poorly crystalline or amorphous content requires extension of the basic methods, but, once these are established, they can be shown to produce QPA to the similar lower limits of detection and accuracy as for crystalline phases.

*\*Not in attendance*

## SESSIONS Tuesday (MORNING) *\*indicates presenter*

### Patent Issues

Chair: **Julien Giovannini**, AstraZeneca R&D, Sweden, julien.giovannini@astrazeneca.com

8:45 **Welcoming Remarks from PPXRD-13 Organizing Committee Chairman**  
**Tom Blanton**, ICDD, Newtown Square, PA, USA

8:55 **Session Chair Opening Remarks**

9:00 Invited – P15

**Current Treatment of Crystalline Forms Under US and EP Patent Law**

**Jill K. MacAlpine\***, Finnegan, Henderson, Farabow, Garrett & Dunner, LLP, Washington, D.C., USA

10:00 **Break**

### Amorphous, Activated and Nano Materials

Chair: **Tim Fawcett**, International Centre for Diffraction Data, Newtown Square, PA, USA, fawcett@icdd.com

10:25 **Session Chair Opening Remarks**

10:30 Invited – P22

**The Relationship Between Stability and X-ray Powder Diffraction Profile for Non-crystalline Solid Forms**

**Simon Bates\***, **D. Jonaitis**, **T. Capozzi**, **P. Stahly**, Triclinic Labs, Lafayette, IN, USA

11:15 (P29) **Studying Early Phase Separation Warning Signs for Spray Dried Disperse Materials**

**Shawn Yin\***, **D. McNamara**, **D. Pan**, **G. Crull**, Bristol-Myers Squibb, New Brunswick, NJ, USA

**P. Timmins**, Bristol-Myers Squibb, Moreton, UK

11:45 (P24) **Analyzing Amorphous and Nanocrystalline Materials by Full Pattern Analyses**

**Timothy Fawcett\***, **S.N. Kabekkodu**, **K. Zhong**, **A.M. Gindhart**, **J.R. Blanton**, **T.N. Blanton**,

International Centre for Diffraction Data, Newtown Square, PA, USA

12:15 **Lunch**

## SESSION Tuesday (AFTERNOON) *\*indicates presenter*

### Qualitative and Quantitative Analysis

Chair: Tom Blanton, ICDD, Newtown Square, PA, USA, tblanton@icdd.com

- 1:55 **Session Chair Opening Remarks**
- 2:00 Invited – P6  
**Crystal Structures of Large-Volume Commercial Pharmaceuticals with  $Z' > 1$**   
**James Kaduk\***, *Illinois Institute of Technology, Chicago, IL, USA*  
**J.W. Reid**, *Canadian Light Source, Saskatoon, Canada*  
**R. Papoular**, *Leon Brillouin Lab., Saclay, France*  
**K. Zhong, A.M. Gindhart, T.N. Blanton**, *ICDD, Newtown Square PA, USA*
- 2:45 (P12) **Problems/solutions of Polymorphism in the Daily Work of a Self-employed Service Laboratory**  
**Martin Ermrich\***, *X-ray Laboratory Dr. Ermrich, Reinheim, Germany*
- 3:15 **Break**
- 3:45 Invited – P17  
**The Impact of Qualitative and Quantitative Synchrotron-XRPD Trace Analyses on the Characterization of Pharmaceuticals**  
**Fabia Gozzo\***, *Excelsus Structural Solutions SPRL, Brussels, Belgium*
- 4:30 (P-28) **ICDD Powder Diffraction File® Coverage of Polymers Used in Pharmaceutical and Biomedical Applications**  
**Tom Blanton\***, *S. Gates, ICDD, Newtown Square, PA, USA*
- 5:00 **End of Session**

## POSTER SESSION Tuesday (EVENING) *\*indicates presenter*

### 6:00 – 8:00PM

- P7 **In situ Formation of Pharmaceutical Cocrystal During Wet Granulation**  
**Ondřej Dammer\***, *G. Sedmak, Zentiva, k.s., Prague, Czech Republic*  
**V. Sládková, E. Škořepová**, *Institute of Chemical Technology Prague, Prague, Czech Republic*
- P8 **MHC-trans – Multi-Sample Humidity Chamber: Non-Ambient XRD for the Investigation of Temperature-Humidity Induced Structural Changes in Pharmaceuticals**  
**Barbara Pühr\***, *A. Pein, Anton Paar GmbH, Graz, Austria*
- P16 **Optimizing PDF Data Quality using a Laboratory Powder Diffractometer**  
**Thomas Hartmann\***, *STOE & Cie GmbH, Darmstadt, Germany*
- P26 **Piracetam/Poly ( $\epsilon$ -caprolactone) Blends to the Formulation of Functional Scaffolds for Neuronal Growth in 3D: Nano and Crystalline Structure**  
**Bonifacio Alvarado-Tenorio\***, *A. Martinez-Martinez, A. Diaz-Sanchez, R. Gonzalez-Fernandez, J. Valero-Galván, Universidad Autónoma de Ciudad Juárez, Chihuahua, Mexico*
- P27 **Quantification of Flupirtine Maleate Polymorphs using X-ray Powder Diffraction Technique**  
**Yumei Zhao\***, *Z. Zheng, S. Li, Beijing institute of Pharmacology and Toxicology, Beijing, China*
- P30 **ICDD Powder Diffraction File™ Coverage of Polymers Used in Pharmaceutical and Biomedical Applications**  
**Tom Blanton\***, *S. Gates, ICDD, Newtown Square, PA, USA*
- P31 **Incorporation of Pharmaceutical API's into the PDF® Databases**  
**A. Gindhart, K. Zhong, Tom Blanton\***, *ICDD, Newtown Square, PA, USA*
- P32 **PDF-4/Organics: Powder Diffraction File™**  
**A. Gindhart, Tom Blanton\***, *ICDD, Newtown Square, PA, USA*
- P33 **Accurate Material Identification Using Sieve+**  
**Tim Fawcett\***, *S. N. Kabekkodu, K. Zhong, J. Blanton, T. Blanton, ICDD, Newtown Square, PA, USA*



## SESSION Wednesday (MORNING) *\*indicates presenter*

### PXRD Techniques/Crystal Structure Prediction/Crystal Structure Verification

Chair: Kenneth Shankland, *Reading University*, Reading, U.K., k.shankland@reading.ac.uk

- 8:55 Session Chair Opening Remarks
- 9:00 Invited – P14  
**Verification of Crystal Structures Determined from PXRD— the Role of DFT-D**  
Jacco van de Streek\*, *University of Copenhagen*, Copenhagen, Denmark
- 9:45 (P18) **Molecular Complexes of Agomelatine-Phosphoric Acid: Crystal Structure Determination and Phase Transformation Kinetics by Non-Ambient Powder XRD**  
R.S. Voguri, R.R. Chirla, Archan Dey\*, *Dr. Reddy's Laboratories Limited*, Hyderabad, India
- 10:15 Break
- 10:45 (P10) **Crystal Structure Prediction as a Tool**  
Marcus Neumann\*, *Avant-garde Materials Simulation*, Freiburg, Germany
- 11:15 (P4) **Structural Characterization and Rationalization of Formation, Stability, and Transformations of Benperidol Solvates**  
Agris Bērziņš\*, *University of Latvia*, Riga, Latvia and *Durham University*, Durham, UK  
E. Skarbulis, A. Actiņš, *University of Latvia*, Riga, Latvia
- 11:45 (P34) **Expanding the Scope of PDF4/Organics in Pharmaceutical PXRD Analysis**  
S. Kabekkodu\*, T.G. Fawcett, K. Zhong, A.M. Gindhart, S. Gates, J.R. Blanton, T.N. Blanton, *ICDD*, Newtown Square, PA, USA
- 12:15 Lunch

## SESSION Wednesday (AFTERNOON) *\*indicates presenter*

### New Frontiers in X-ray Diffraction for Pharmaceutical Research and Development

Chair: Detlef Beckers, *PANalytical*, Almelo, The Netherlands, detlef.beckers@panalytical.com

- 1:55 Session Chair Opening Remarks
- 2:00 Invited – P2  
**The Power of Powder: Protein-based Drug Screening**  
Irene Margiolaki\*, *University of Patras*, Patras, Greece
- 2:45 (P11) **Characterization and Structural Assessment of Pharmaceutical Solids by Total Scattering**  
Shuang Chen\*, *AbbVie, Inc.*, Chicago, IL, USA
- 3:15 Break
- 3:45 (P21) **Study of Biological Macromolecules with a Laboratory X-ray Diffraction System**  
Detlef Beckers\*, T. Degen, G. Nénert, J. Bolze, *PANalytical B.V.*, Almelo, The Netherlands  
S. Saslis, S. Logotheti, I. Margiolaki, *University of Patras*, Patras, Greece  
V. Kogan, *Dannalab*, Enschede, The Netherlands
- 4:15 (P13) **Ultrafast Crystal Structure Determination of Pharmaceutical Compounds using TEM Electron Diffraction without Cooling Techniques**  
Partha Pratim Das\*, *NanoMEGAS SPRL*, Brussels, Belgium & *Electron Crystallography Solutions*, Madrid, Spain  
E. Van Genderen, J.P. Abrahams, *University of Leiden*, Leiden, Netherlands  
S. Nicolopoulos, *NanoMEGAS SPRL*, Brussels, Belgium
- 4:45 (P25) **Instrumentation for Tackling Current and Future Challenges in Pharmaceutical R&D**  
Dubravka Šišak Jung\*, *DECTRIS Ltd*, Baden, Switzerland  
T. Hartmann, *STOE & Cie GmbH*, Darmstadt, Germany
- 5:15 End of Session

## SESSION Thursday (MORNING) *\*indicates presenter*

### Complementary Techniques/Formulation & Product Development

Chairs: Julien Giovannini, *AstraZeneca R&D, Sweden, julien.giovannini@astrazeneca.com*

Shawn Yin, *Bristol-Myers Squibb Company, New Brunswick, NJ, USA, shawn.yin@bms.com*

- 8:55      **Session Chair Opening Remarks**
- 9:00      Invited – P23  
**Small Angle X-ray and Neutron Scattering (SAXS, SANS) Applied to the Investigation of Self-Assembled Systems**  
*Michael Gradzielski\**, *Technische Universität Berlin, Berlin, Germany*
- 9:45      **Break**
- 10:15     (P5) **The Use of the Atomic Pair-Wise Distribution Function (PDF) for Assessing the Stability and Comparability of an Amorphous Drug Prepared by Different Spray Drying Processes: A Comparison Between Conventional X-ray Diffraction Versus High Energy Synchrotron Radiation**  
*Hector Novoa de Armas\**, *M. Brewster, Johnson & Johnson Pharmaceutical Research & Development, a division of Janssen Pharmaceutical NV, Beerse, Belgium*  
*D. Beckers, M. Gateshki, PANalytical B.V, Almelo, The Netherlands*  
*C. Benmore, Argonne National Laboratory, Argonne, IL, USA*  
*S. Byrn, Perdue University, West Lafayette, IN, USA*
- 10:45     (P9) **Characterization of Pharmaceutical Tablet Samples by Using Various Analysis Techniques**  
*Atsushi Ohbuchi\**, *C. Sato, T. Konya, A. Yamano, Rigaku Corporation, Tokyo, Japan*
- 11:15     **Round Table Discussion & Closing Remarks**
- NOON      **End of Symposium**

