

# PPXRD-3

23-25 February 2004

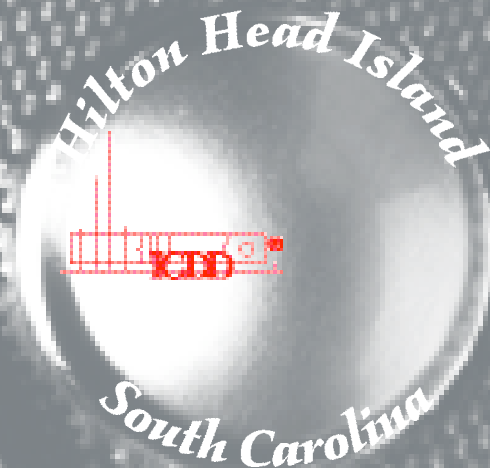
The International Centre for Diffraction Data

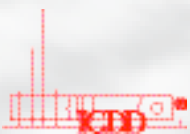
is pleased to announce....

## The 3<sup>rd</sup> Pharmaceutical Powder X-ray Diffraction Symposium Program

**Make the most of your XRPD data!**

- ▼ Secure fast development of drug candidates ▼
- ▼ Obtain reliable data for regulatory submissions ▼
- ▼ Provide the right data to support patent development ▼





# PPXRD-3

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## The 3<sup>rd</sup> Pharmaceutical Powder X-ray Diffraction Symposium 23-25 February 2004

**Crowne Plaza Resort  
Hilton Head Island  
South Carolina, USA**

**Experience all that Hilton Head Island has to offer ...**

- Spectacular island setting
- White-sand beaches
- Ocean-view dining
- Championship golf courses
- Lush tropical gardens

The PPXRD-3 Symposium has a special focus. We have organized topics to deal with making the most of your XRPD data. How do I determine crystallinity? How do I support the physiochemical team during solubility determinations? Is my material in a single crystalline phase? Can I use XRPD to determine stoichiometry during formulation processes? Do I need XRPD for stability studies? How can I prepare XRPD data for regulatory submissions? What XRPD data do I need to construct a good patent? The goal of PPXRD-3 is to create a venue to discuss and address these issues.

### **Scientific Organizing Committee**

**Dr. John Faber, Chair**, *International Centre for Diffraction Data*, Newtown Square, PA, USA

**Dr. Detlef Beckers**, *PANalytical*, Almelo, The Netherlands

**Dr. Harry G. Brittain**, *Center for Pharmaceutical Physics*, Milford, NJ, USA

**Dr. Julien Giovannini**, *AstraZeneca*, Lund, Sweden

**Dr. Jun Han**, *Abbott Laboratories*, Abbott Park, IL, USA

**Dr. Arnt Kern**, *Bruker AXS GmbH*, Karlsruhe, Germany

**Dr. Daniel Louër**, *University of Rennes I*, Rennes cedex, France

**Dr. Fangling Needham**, *International Centre for Diffraction Data*, Newtown Square, PA, USA

**Dr. Peter W. Stephens**, *State University of New York*, Stony Brook, NY, USA

**Dr. Gregory A. Stephenson**, *Eli Lilly and Company*, Indianapolis, IN, USA

**Dr. Raj Suryanarayanan**, *University of Minnesota*, Minneapolis, MN, USA

**Dr. Peter G. Varlashkin**, *GlaxoSmithKline*, Research Triangle Park, NC, USA

**Dr. Shawn Yin**, *Bristol-Myers Squibb Company*, New Brunswick, NJ, USA

Special **Thank You** to our coffee break sponsors:



# Symposium Program

**Monday, 23 February**

**Morning**

- 8:30–9:00 **Welcoming Remarks & Introductions: Dr. John Faber, ICDD**  
**ACQUISITION and USE OF X-RAY POWDER DIFFRACTION DATA**  
**Session Chair: Dr. Detlef Beckers**, PANalytical B.V., Almelo, The Netherlands
- 9:00–9:45 ACCURACY IN POWDER DIFFRACTION MEASUREMENTS—Invited  
**Robert L. Snyder**, Georgia Institute of Technology, Atlanta, GA, USA
- 9:45–10:10 IN-SITU VARIABLE TEMPERATURE POWDER X-RAY DIFFRACTION AND THERMAL ANALYSIS  
**Shawn X. Yin, Raymond P. Scaringe, Jack Z. Gougoutas**, Bristol-Myers Squibb Company, New Brunswick, NJ, USA
- 10:10–10:35 GENERATION OF ACCURATE X-RAY POWDER DIFFRACTION DATA OF A PHARMACEUTICAL COMPOUND FOR INCLUSION INTO THE INTERNATIONAL CENTRE FOR DIFFRACTION DATA (ICDD) POWDER DIFFRACTION FILE (PDF)  
**Gerald Monger, Peter Varlashkin**, GlaxoSmithKline, Research Triangle Park, NC, USA
- 10:35–11:05 **BREAK**
- 11:05–11:30 FORMULATION ANALYSES OF OFF THE SHELF PHARMACEUTICALS  
**Timothy G. Fawcett, John Faber**, International Centre for Diffraction Data, Newtown Square, PA, USA  
**C.R. Hubbard**, Oak Ridge National Laboratory, Oak Ridge, TN, USA
- 11:30–11:55 RAPID COMBINATORIAL SCREENING BY TWO-DIMENSIONAL XRD  
**Bob B. He, Lutz Brügemann, Uwe Preckwinkel, Kingsley L. Smith**, Bruker AXS, Inc., Madison, WI, USA
- 11:55–1:25 **LUNCH**

**Afternoon: Continued: ACQUISITION and USE OF X-RAY POWDER DIFFRACTION DATA**

- 1:25–1:50 ON THE OPTIMUM OPTICAL SETUP FOR MEASURING PHARMACEUTICAL SUBSTANCES  
**Martijn Fransen, Detlef Beckers**, PANalytical B.V., Almelo, The Netherlands  
(Detlef Beckers will be speaking on behalf of Martijn Fransen)
- 1:50–2:15 NEW DETECTOR TECHNOLOGY FOR SUPER SPEED X-RAY DIFFRACTION  
**Lutz Brügemann, A. Kern, H.-G. Krane**, Bruker AXS GmbH, Karlsruhe, Germany  
**R. Durst, B. He, Y. Diawara**, Bruker AXS, Inc., Madison, WI, USA
- POWDER PATTERN INDEXING AND PATTERN MATCHING**  
**Session Chair: Dr. Daniel Louër**, University of Rennes, Rennes cedex, France
- 2:15–3:00 INDEXING: ALGORITHMS, ACCURACY, AND AB INITIO—Invited  
**James A. Kaduk**, BP Chemicals, Naperville, IL, USA
- 3:00–3:30 **BREAK**
- 3:30–3:55 ADVANCES IN INDEXING OF POWDER DIFFRACTION PATTERNS: ITERATIVE USE OF LEAST SQUARES AND MONTE-CARLO BASED WHOLE POWDER PATTERN DECOMPOSITION  
**A.A. Coelho, Arnt Kern**, Bruker AXS GmbH, Karlsruhe, Germany  
(Arnt Kern will be speaking on behalf of A.A. Coelho)
- 3:55–4:20 INDEXING POWDER DIFFRACTION PATTERNS WITH THE DICHOTOMY ALGORITHM  
**Daniel Louër**, University of Rennes, Rennes cedex, France  
**A. Boultif**, Mentouri University, Constantine, Algeria

# Symposium Program

**Tuesday, 24 February**

**Morning**

**STRUCTURE DETERMINATION, RIETVELD REFINEMENT AND MOLECULAR MODELING**

**Session Chair: Dr. Peter Stephens**, State University of New York, Stony Brook, NY, USA

8:30–9:15

IDENTIFICATION AND CRYSTAL STRUCTURE SOLUTION OF STABLE AND METASTABLE PHARMACEUTICAL COMPOUNDS USING X-RAY POWDER DIFFRACTION DATA—Invited

**W.I.F. David**, **K. Shankland**, CCLRC Rutherford Appleton Laboratory, Oxon, England, U.K.  
**C.R. Pulham**, The University of Edinburgh, Edinburgh, Scotland, U.K.

9:15–10:00

CRYSTAL STRUCTURE SOLUTION AND REFINEMENT OF A SERIES OF D<sub>2</sub>/□<sub>2</sub> RECEPTOR AGONISTS USING LABORATORY X-RAY POWDER DIFFRACTION DATA—Invited

**A. Johnston**, **A.J. Florence**, **N. Shankland**, Strathclyde University, Glasgow, Scotland, U.K.  
**K. Shankland**, **A.J. Markvardsen**, W.I.F. David, CCLRC Rutherford Appleton Lab., Oxon, England, U.K.

10:00–10:30

**BREAK**

10:30–10:55

ANALYSIS OF SHORT RANGE ORDER IN X-RAY AMORPHOUS INDOMETHACIN USING X-RAY POWDER DIFFRACTION AND PAIR-WISE DISTRIBUTION FUNCTIONS

**Simon Bates**, SSCI, Inc., West Lafayette, IN, USA  
**George Zograf**, University of Wisconsin-Madison, Madison, WI, USA  
**Kieran Crowley**, Cardinal Health Inc., Somerset, NJ, USA

10:55–11:20

RECENT ADVANCES IN STRUCTURE SOLUTION FROM POWDER DIFFRACTION DATA

**Cikui Liang**, Accelrys, Inc., San Diego, CA, USA

11:20–11:45

CRYSTAL STRUCTURE OF D-MANNITOL HEMIHYDRATE

**Cristian E. Botez**, **Peter W. Stephens**, Stony Brook University, Stony Brook, NY, USA and Brookhaven National Laboratory, Upton, NY, USA  
**Cletus Nunes**, **Raj Suryanarayanan**, University of Minnesota, Minneapolis, MN, USA

11:45–1:15

**LUNCH**

**Afternoon**

**Continued: STRUCTURE DETERMINATION, RIETVELD REFINEMENT AND MOLECULAR MODELING**

1:15–1:40

STRUCTURE DETERMINATION OF AMBUIC ACID WITH SYNCHROTRON POWDER DIFFRACTION AND SOLID-STATE NMR

**Yuegang Zhang**, **Peter L. Lee**, **Robert Von Dreele**, Argonne National Laboratory, Argonne, IL, USA  
**James K. Harper**, **David M. Grant**, University of Utah, Salt Lake City, UT, USA

1:40–2:05

THE CRYSTAL STRUCTURE OF GUAIFENESIN, 3-(2-METHOXYPHENOXY)-1,2-PROPANEDIOL

**James A. Kaduk**, BP Chemicals, Naperville, IL, USA

**PROCESS ANALYTICAL TECHNOLOGY**

**Session Chair: Dr. Peter Varlashkin**, GlaxoSmithKline, Research Triangle Park, NC, USA

2:05–2:50

USE OF X-RAY POWDER DIFFRACTOMETRY TO MONITOR PHASE TRANSITIONS DURING PHARMACEUTICAL PROCESSING—Invited

**Raj Suryanarayanan**, University of Minnesota, Minneapolis, MN, USA

2:50–3:20

**BREAK**

3:20 - 3:45

POLYCAPILLARY X-RAY OPTICS ENABLED DIFFRACTION-BASED PROCESS DIAGNOSTICS AND CONTROL

**Walter M. Gibson**, **Huapeng Huang**, X-ray Optical Systems, Inc., East Greenbush, NY, USA

3:45 - 4:10

IN SITU XRPD OF COMPACTS AS A POTENTIAL NON-DESTRUCTIVE ANALYTICAL TECHNIQUE FOR DETECTING PROCESS INDUCED PHASE CHANGES

**Peter L.D. Wildfong**, **Nicole A. Morley**, **Kenneth R. Morris**, Purdue University, West Lafayette, IN, USA

# Symposium Program

**Wednesday, 25 February**

**Morning**

**POLYMORPH CHARACTERIZATION BY XRPD**

**Session Chair: Dr. Shawn Yin, Bristol-Myers Squibb**, New Brunswick, NJ, USA

8:30-9:15

POLYMORPHS AND OTHER CRYSTAL FORMS: WHAT'S IN THAT POWDER?—Invited  
**Raymond P. Scaringe**, Bristol-Myers Squibb, New Brunswick, NJ, USA

9:15-9:40

PHARMACEUTICAL POWDER X-RAY DIFFRACTION: FROM DESIGN TO FINAL SELECTION OF A DEVELOPABLE MATERIAL  
**Julien Giovannini**, AstraZeneca R&D, Lund, Sweden

9:40-10:05

IDENTIFICATION OF POLYMORPH TYPE AND PREDICTION OF MATERIALS PROPERTIES FROM LABORATORY X-RAY POWDER PATTERNS USING INDEXING AND ELECTRON DENSITY CALCULATIONS

**Simon Bates, Igor Ivanisevic, Barbara Stahly**, SSCI, Inc., West Lafayette, Indiana, USA

10:05-10:35

**BREAK**

10:35-11:00

POLYMORPHIC FORM SCREENING USING X-RAY POWDER DIFFRACTION

**Gregory A. Stephenson, Gregory S. Clanton, Benjamin A. Diserod**, Eli Lilly and Company, Indianapolis, IN, USA

11:00-11:25

INFLUENCE OF EXPERIMENTAL CONDITIONS ON THE PHASE TRANSITIONS OF  $\alpha$ ,  $\beta$ -TREHALOSE  
**D. Beckers, S. Prugovečki**, PANalytical B.V., Almelo, The Netherlands

11:25-12:55

**LUNCH**

**Afternoon**

**REGULATORY AND PATENT ISSUES**

**Session Chair: Dr. Julien Giovannini**, AstraZeneca R&D, Lund, Sweden

12:55-1:40

APPLICATIONS OF X-RAY POWDER DIFFRACTION TO REGULATORY ISSUES—Invited  
**Stephen R. Byrn**, Purdue University and SSCI, Inc., West Lafayette, IN, USA

1:40-2:25

PHARMACEUTICAL SOLID POLYMORPHISM—THE FDA VIEW—Invited

**Andre S. Raw, Lawrence X. Yu**, Food and Drug Administration—Center for Drug Evaluation and Search, Rockville, MD, USA

2:25-2:50

21CFR PART11—WHAT'S LEFT (FOR XRPD)?

**Arnt Kern**, Bruker AXS GmbH, Karlsruhe, Germany

2:50-3:10

**BREAK**

3:10-3:55

POLYMORPHS AND PURITY AND PATENTS; OH MY—Invited

**Michael H. Teschner, Esq.**, Lerner, David, Littenberg, Krumholz & Mentlik, Westfield, NJ, USA

3:55-4:15

**Q&A/Closing Remarks**



# Poster Session, Reception and Exhibits

23 February 2004  
Crowne Plaza Resort  
Manufacturers' Exhibits and Poster Session  
6–9 pm

## PREPARATION AND CHARACTERIZATION OF GLIMEPIRIDE SOLID DISPERSIONS BY POWDER X-RAY DIFFRACTION AND DIFFERENTIAL SCANNING CALORIMETRY

S. M. Al-Saidan, Y.S.R. Krishnaiah, Kuwait University, Safat, Kuwait

## DETERMINING THE RELATIVE SENSITIVITY OF X-RAY POWDER DIFFRACTION FOR IDENTIFYING AND MONITORING CRYSTAL FORM CHANGES OF ACTIVES IN SOLID DOSAGE FORMULATIONS

Gerald Monger, GlaxoSmithKline, Research Triangle Park, NC, USA

## ANALYSIS OF MANNITOL X-RAY DIFFRACTION PATTERNS

J. Faber, T.G. Fawcett, F. Needham, International Centre for Diffraction Data, Newtown Square, PA, USA

J. Kaduk, BP Chemicals, Naperville, IL, USA

A. Kern, Bruker AXS GmbH, Karlsruhe, Germany

D. Louër, University of Rennes, Rennes cedex, France

R.B. Ortega, Rigaku/MSC, Inc., The Woodlands, TX, USA

P.W. Stephens, State University of New York at Stony Brook, Stony Brook, NY, USA

G. Stephenson, Eli Lilly and Company, Indianapolis, IN, USA

P.G. Varlashkin, GlaxoSmithKline, Research Triangle Park, NC, USA

## AUTOMATIC PROCESSING AND PERFORMANCE QUALIFICATION (PQ) IN PHARMACEUTICAL APPLICATIONS

Jeffrey P. Nicolich, PANalytical, Natick, MA, USA

Wenjin Cao, AMGEN, Thousand Oaks, CA, USA

## SINGLE CRYSTAL STUDIES OF MEFENAMIC ACID AS A TOOL TO UNDERSTAND POLYMORPHISM

Vrushali Waknis, Neeraj Kumar, P.V. Bharatam, C.L. Kaul, Ramesh Panchagnula, National Institute of Pharmaceutical Education & Research (NIPER), Punjab, India

T.N. Guru Row, Deepak, Solid State Structural Chemistry, IISc, Bangalore, India

## SAXSess—AN ANALYTICAL TOOL FOR BIOMATERIALS

Heimo Schnablegger, Anton-Paar GmbH, Graz, Austria

Alexander Bergmann, Otto Glatter, University of Graz, Graz, Austria

## X-RAY POWDER DIFFRACTION AND SORPTION-DESORPTION INVESTIGATIONS OF INDAPAMIDE

Matej Smrkolj, Andrej Gartner, Marija Grčman, Krka, d.d., Novo Mesto, Slovenia

Anton Meden, University of Ljubljana, Ljubljana, Slovenia

## APPLICATION OF HIGH THROUGHPUT SCREENING TO THE STUDY OF THE CONFORMATIONAL POLYMORPHS OF "ROY"

G.A. Stephenson, G.S. Clanton, B.A. Diseroad, R.M. Goodwin, Eli Lilly and Company, Indianapolis, IN, USA

## LAYERED STRUCTURE OF A MULTIPLE HYDRATE-FORMING PHARMACEUTICAL COMPOUND

Y.-H. Kiang, Wei Xu, Laura A. DeBusi, Merck and Co., Inc., West Point, PA, USA

Nancy Tsou, Richard G. Ball, Nobuyoshi Yasuda, Merck and Co., Rahway, NJ, USA

Peter W. Stephens, State University of New York, Stony Brook, NY, USA

# Instrument Calibration & Indexing Workshop

*Thursday, 26 February, 8am–4pm*

*Does your instrument produce good data?  
Instrument calibration and indexing will be covered in this hands-on workshop.*

To participate in the hands-on exercises, all workshop attendees must bring their own laptop computer, Windows®-based with Windows® 98 or later, to the workshop. **PPXRD-3** will provide the data analysis software for use on your laptop computer throughout the workshop.

## Exhibitor Information

*Exhibits will be located in Salon D  
Crowne Plaza Resort*

### **Exhibit Hours:**

Monday, 23 February 10 am–9 pm  
Tuesday, 24 February 10 am–3:30 pm

*A diagram of the exhibit locations will be available in the Book of Abstracts.*

### **Exhibitors:**

Accelrys  
Bruker AXS, Inc.  
Hecus X-ray Systems  
INEL, Inc.  
International Centre for Diffraction Data (ICDD)  
PANalytical (formerly Philips Analytical)  
Rigaku/MSK, Inc.

# Accommodations & Travel

The PPXRD Symposium will be held at the **Crowne Plaza Resort**,  
Hilton Head Island, South Carolina, USA

## *Hotel Information*

Crowne Plaza Resort  
130 Shipyard Drive, Shipyard Plantation  
Hilton Head Island, South Carolina 29928, USA  
Web site: [www.cphiltonhead.com](http://www.cphiltonhead.com)

To make your reservation see options below. Please reference the International Centre for Diffraction Data or PPXRD group to receive the reduced group rate of \$110, plus resort fee (currently \$8), and 10% tax. The deadline for reservations is 1 February 2004. You will not be guaranteed a room after that time. Please contact the hotel for the policy on cancellation of reservations. For your convenience, reservations can be made at the Crowne Plaza Resort with these easy options:

Internet: <a href="http://www.cphiltonhead.com">www.cphiltonhead.com</a>	Group Booking Code: ICD
Telephone: 1.800.334.1881 or +843.842.2400	Group Name: International Centre for Diffraction Data or PPXRD
Fax: +843.842.6503	

**Reservation deadline for group rate: 1 February 2004**

## **General Information**

Attire: Attire is informal for most activities  
Climate: The average temperature in February is 61°F  
Time Zone: EST  
Airports: Savannah International Airport, Savannah, Georgia, USA  
Hilton Head Island Airport, South Carolina, USA



# 2004 PPXRD-3 Symposium Registration Form

- |   |                     |               |             |
|---|---------------------|---------------|-------------|
| <input type="checkbox"/> Symposium only                   | 23-25 February 2004 | PPXRD04       | \$ 1,200.00 |
| <input type="checkbox"/> Symposium & Workshop             | 23-26 February 2004 | PPXRDWS04     | \$ 1,500.00 |
| <input type="checkbox"/> Symposium only Student fee       | 23-25 February 2004 | PPXRDSTUD04   | \$ 400.00   |
| <input type="checkbox"/> Symposium & Workshop Student fee | 23-26 February 2004 | PPXRDWSSTUD04 | \$ 700.00   |

Total: \_\_\_\_\_

## The easiest way to register



### Online at:

www.icdd.com/ppxrd



### Fax to:

+610.325.9823



### Mail to: ICDD, Attn: Leah Mooney

12 Campus Boulevard  
Newtown Square, PA 19073-3273 USA

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First Name \_\_\_\_\_ Middle Initial \_\_\_\_\_ Last Name \_\_\_\_\_

Preferred Badge Name (if different) \_\_\_\_\_

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City \_\_\_\_\_ State \_\_\_\_\_ Zip \_\_\_\_\_ Country \_\_\_\_\_

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Enclosed is a check for \$ \_\_\_\_\_, payable to ICDD in U.S. dollars and drawn on a U.S. bank

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**Please note any needs related to disabilities.**

Special needs \_\_\_\_\_



## ***Cancellation Policy***

Cancellations must be submitted in writing to ICDD. A full refund will be issued, less a \$50 processing fee, if the cancellation is received at least two weeks before the symposium (Monday, 9 February 2004). No refunds will be issued for cancellations received after 9 February 2004.