

CRYSTALLOGRAPHY TIMES

Rigaku

- For the first time in a few years, we held our annual European Users' Meeting in the UK. We had a strong turnout and an interesting program

RIGAKU TOPIQ WEBINARS

Rigaku has developed a series of 20-30 minute webinars that cover a broad range of topics in the

fields of X-ray and electron diffraction, X-ray fluorescence and X-ray imaging. You can watch recordings of our past

sessions here.

UPCOMING EVENTS: 80th Pittsburgh Diffraction Conference, Pittsburgh, PA, October 15-17, 2023.

SERMACS 2023, Durham, NC, October 25-28, 2023.

31st Protein Structure Determination in Industry,

Cambridge, UK, November 12-14, 2023. Rayons X et Matière 2023, Bordeaux, France, November 21-24, 2023.

CRYSTALLOGRAPHY IN THE NEWS

Research Institute report the structures several transition state analogs of Pd catalysts that activate *sp*³ C-H bonds and arylate free alcohols.

Scientists from China and the U.S. have characterized a Li-S battery

cell in situ using a EC-TEM holder

using both electron microscopy

Researchers from the Chinese

Academy of Sciences and UC

Berkeley have synthesized and characterized a stable Cu(III)

Scientists from UC Berkeley, UC Davis and F. Hoffmann-La Roche

have synthesized a novel Cu(II)

Morocco, a flood in Libya, a fire in

organizations like the International

Movement, so they can distribute

Maui and an ongoing war in

suffering, perhaps it is a good time to donate to larger relief

Red Cross and Red Crescent

FOLLOW US ON TWITTER

To keep up to date on the latest

relief as needed.

Ukraine. With some much

complex that initiates cross-

and electron diffraction.

September 8, 2023

September 8, 2023

complex.

Researchers from The Scripps

September 6, 2023

September 6, 2023

coupling reactions. **USEFUL LINKS** We have had an earthquake in

news and events from Rigaku Oxford Diffraction, follow our Twitter feed. **FOLLOW**

JOIN US ON LINKEDIN

Our LinkedIn group shares

information and fosters discussion

about X-ray crystallography and

SAXS topics. Connect with other

updates on how they use these

laboratories. You can also catch

up on the latest newsletter or

Rigaku Journal issue. We also

research groups and receive

techniques in their own

hope that you will share information about your own research and laboratory groups.

RIGAKU X-RAY FORUM

At rigakuxrayforum.com you can find discussions about software, general crystallography issues and more. It's also the place to download the latest version of Rigaku Oxford Diffraction's

JOIN HERE

JOIN HERE

CrysAlis^{Pro} software for single

crystal data processing.

Volume 15, No. 9, September 2023

INTRODUCTION

with a lot of innovative techniques and topics packed in. The meeting was organized in collaboration with the National Electron Diffraction Facility (NEDF) and co-sponsored by JEOL. The NEDF is a new facility offering

data collection and structure determination using electron diffraction in a similar way to that in which the National Crystallography Service (NCS) has been offering X-ray services for many years. The first day of our meeting was based around electron diffraction, with talks from invited speakers and NEDF staff to introduce the facility itself and the capabilities of electron diffraction in general. Speakers on the first

day included Simon Coles and David Walker (NEDF), Simon Parsons (Edinburgh), Jere Tidey and Daniel Rainer (NEDF), David Waterman (STFC), Seb Pike (Warwick), Andrea Laybourn (Nottingham) and Kasper Pederson (DTU). These talks all drove home the point that electron diffraction has arrived and is enabling scientists to break new ground and reach new targets. After the coffee break, we took our guests to see the facilities in Warwick, new electron diffractometer, the XtaLAB Synergy-ED. We also had a

both their existing XtaLAB Synergy X-ray diffractometers and their brandsession hosted by Mathias Meyer in which he spent some time explaining the history of our collaboration with JEOL.

Despite the fantastic evening meal, merriment, and an unwelcome fire alarm at around 1:30 am everyone still managed to get back to the meeting room for 9 am the next day, where we had talks from our staff and more invited speakers. Second-day speakers included, Mike Probert (Newcastle), Mathias Meyer, Christian Schürmann, Khai Nghi Truong and Jakub Wojciechowski (all Rigaku), Lauren Hatcher (Cardiff), Michael Bodensteiner (Olexsys/Regensburg), Tom Turner (Leeds) and Tom Fellowes (Liverpool). Thanks to our speakers, and attendees for making this meeting possible and making it such a fun event. We are fortunate to have a community of fantastic users.

Rigaku.com shortly. We are changing things up this month. Instead of the *Instrument in the* Spotlight and the Researcher in the Spotlight we will highlight a technique and provide a useful tip. This month we highlight the FlowXI developed by Tom Turner at Leeds, which provides state-of-the-art analysis of crystallization processes in situ using X-ray diffraction and Raman. Our tip of the month is provided by Fraser White, who explains how to generate precession images. Please let us know what you think of these changes. This month Jeanette reviews an important book titled Not Just for the Boys:

Why We Need More Women in Science. We also have the usual features:

Crystallography in the News and Useful Links.

TECHNIQUE IN THE SPOTLIGHT

Fraser White and Joe Ferrara

By popular demand, we have decided to run another Rigaku School for

Practical Crystallography in January 2024. The dates are January 15-19

and January 22-26. A syllabus and registration information will appear at

Flow crystallography Flow crystallography is a technique where crystallization processes can be studied *in situ* by passing a crystallization mixture through an X-ray beam and detecting diffraction from crystals and crystallites.

FlowXI is a new research facility at the University of Leeds that enables state-of-the-art analysis of crystallization processes in-situ through

The facility integrates a number of flow devices for studying crystallization

microfluidic devices, acoustic droplet levitator, variable humidity chamber

users to establish which experiments conducted initially at FlowXI require

beamtime at synchrotron sources such as Diamond. Current case studies at

pharmaceutical ingredient (API) hydrate species during a solution mediated

phase transformation to its anhydrate, tracking the solution supersaturation

and solid state phase changes during evaporative crystallization of an

processes, including a slurry recirculation loop for process reactors, droplet

and continuous flow crystallization devices. The goal of the facility is to provide researchers from industry and academia with an environment in which to design, develop, test and apply in-situ sample environments to the study of nucleation and growth of functional materials under fluid-flow or liquid environments. This facilitates new scientific discoveries and enables

the facility include probing the structural evolution of an active

combined Raman spectroscopy and powder X-ray diffraction.

inorganic salt in levitated droplets, and measuring the impact the of process conditions on the polymorphic selection of an API in continuous flow crystallization reactors.

detector with high dynamic range, fast readout speed and extremely low noise, allows for in-situ diffraction studies of samples in flow. Additionally, the set-up has a Raman spectrometer, a Labram HR Evolution from HORIBA Jobin Yvon, with both green (532 nm) and red (785 nm) lasers with respective ultralow frequency (ULF) modules allowing measurements in the sub-100 cm⁻¹ region, with measurements down to 10 cm⁻¹ routinely available. Two SuperHead fiber optic probes also facilitate insitu measurements at samples contained within the X-ray diffractometer enclosure. This combination allows the user to probe solution-state molecular detail through to structural phase identification and quantification of materials in liquid systems. Hydrate to Anhydrate **Phase Transformation** Non-crystalline recursor Phase 1200

FlowXI Facility inside the X-ray enclosure probing the crystallization of Na_2SO_4 inside a 10 μl acoustically levitated droplet using combined Raman spectroscopy and X-ray diffraction, the sample is in the center of the image where the laser of the Raman can be

The facility has a state-of-the-art Rigaku XtaLAB Synergy Custom X-ray diffractometer with an ultrahigh-brightness source, optimized towards data

collection from the weakest of diffracting samples. This, paired with a

seen hitting the droplet.

1000

800

≥theta (degrees)

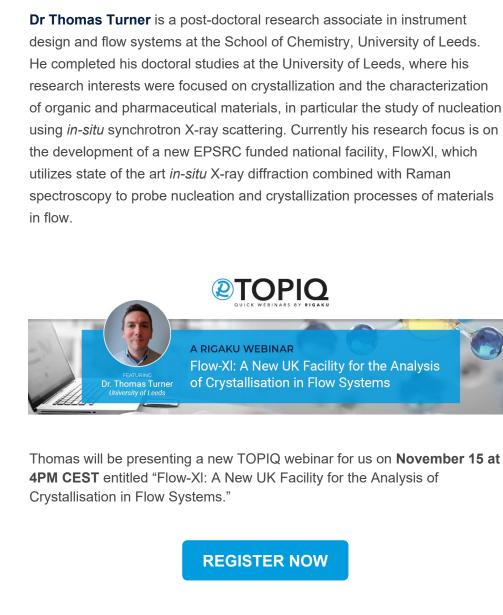
structural changes during evaporative crystallization.

Droplet Evaporation

RESEARCHER IN THE SPOTLIGHT

The diffraction data versus time for the levitated droplet experiment, highlighting the key

Relative Intensity



PRODUCT IN THE SPOTLIGHT

TIP OF THE MONTH **Generating Standard and Integrative Precession Images**

reciprocal space to better identify certain features.

structural chemistry program.

What are they?

Why should I use them?

How do I make them?

UNWARP: Add a reconstruction layer

L1 to the right and L2 po

Unwarp

Os 0.00 0.00 0.80

Oe: 0.00 0.00 1.2

Use L1, L2, 0/0s De as cartesian (pt e

Figure 1. Unwarp access.

"next."

L1: 100

reconstruction layer is determined by three vectors: L1 and L2, layer vectors and 0, the vector be non-collinear and will define the reconstruction plane:

e right-handed from there. Os and De are the origin vecto nputed at:R = I1*L1 + I2*L2 + Oi; where Oi is looping ("Int

Tip: Use 2d Laue averaging to fill your images due to missing coverage! But: It takes time!

Tip: Choose a meaningful name like "h k 0 with 0.8A". This helps to find your images back.

Use mirror plane

to the Lattice Wizard and find the correct cell

Choose which layers to generate:

before.

Examples

= 3.

XtaLAB Synergy-Custom

One of the advantages of Rigaku's rotating anode X-ray generators is the ability to utilize both ports of the generator simultaneously. This might

port dedicated to protein crystallography and a second goniometer

close to the goniometer. Thus, the versatility offered by the XtaLAB Synergy-Custom system provides the most flexibility possible for your

Reconstructed precession images (also called unwarped images) allow you to create any plane of reciprocal space using all the relevant frames from the data collection. Integrative precession images allow you to take a larger slice of

Precession images are very useful for visualizing the crystallinity of your sample.

incommensurate behavior as well as diffuse scatter. Integrative layers can be particularly useful for studying diffuse scatter, as you can choose a thicker slice of

• Click on the **Unwarping** button on the **Lattice Wizard** window (Figure 1).

They may help show weaker peaks due to twinning, superstructure or

reciprocal space with which to create a composite image.

Lattice wizard

involve having two single crystal goniometers mounted on the generator or it might involve having one goniometer with a sample changing robot on one

dedicated to chemical crystallography. In order to accommodate the various configurations that a researcher might envision, Rigaku offers the XtaLAB Synergy-Custom, a system that can be configured with either a MicroMax-007 HF microfocus rotating anode source or the highest-flux in-house X-ray source, the FR-X. Additionally, the XtaLAB Synergy-Custom comes in a specially designed enclosure with your choice of table size to accommodate accessory equipment, such as an ACTOR 2 robotic system or a microscope

and choose the order and the resolution. For example, typing "2 0.8" will generate the -2kl, -1kl, 0kl, 1kl, 2kl images out to 0.8 Å, as well as the corresponding images along the k and I directions. • To generate a **single layer**, click on "New layer", choose "One pixel layer" for a standard precession image then choose the

would set O = 0 4 0, L1 = 1 0 0, and L2 = 0 0 1.

Check the orientation matrix is correct and click "next." If it is not, go back

• Choose the runs to generate the images (the more, the better) and click

To quickly generate multiple layers, click on "generate layers"

origin (O) and the two vectors (L1 & L2), as well as the output name for the image. For example, to generate the h4l plane you

• For integrative layers, repeat the above, choosing "Integrative layer" instead. Instead of an origin, you must now enter start and end origin vectors (Os & Oe) to define the thickness of the layer and integration units (number of slices) along with L1 and L2 as

 In either case, if portions of a layer are missing, you can apply 2D symmetry averaging to generate a full image of the plane.

 In the next few windows you can choose to apply various corrections to the images (background, convolution etc.) and apply a scale factor to make sure

Figure 2. Honeycomb.

- Review: Not Just for the Boys: Why We Need More Women in Science By Athene Donald ISBN 9780192893406
- As a Professor Emerita of Experimental Physics at the University of Cambridge, Donald certainly has her own breadth of experience navigating science and academia as a woman. While she does draw on that experience, much of Not Just for the Boys includes numerous anecdotes and personal stories from women across scientific disciplines, as well as explanations of research findings from studies designed to determine if gender gaps really do exist in the sciences, both at the professional and student level (spoiler alert: they do). Donald begins with a chapter entitled "What's the Problem?" in which she outlines and addresses the issues at hand—namely, after decades of initiatives to increase the number of women in science and the fact that the population of the world is roughly 50% women, women do not account
- for 50% of scientists. She then proceeds to delve into the issue as deeply as she can in a book of a little over 200 pages, starting with how scientists are perceived in society (mostly as men) and how that impacts young women's perceptions of what they can be. She also digs into what can keep young women from pursuing roles in scientific careers even if they have a proven interest, and what obstacles women can face once they've already made their career in the sciences. At the end of the day, it's still a man's world, and science is no exception. Donald argues that science, like any field, benefits from the diversity of those pursuing it. She acknowledges that, as a white woman, she still garners certain privileges not afforded to colleagues of color, particularly women of color. But Donald also acknowledges that as a white woman, she is not the right person to be writing a book about intersectionality in

weak features are visible (typically a value of 20 is used). Once generated, you use all standard image analysis tools, including the information (i) button to check peak indices, and you can also overlay the reciprocal lattice grid (drop-down arrow next to the RED button). The images are stored in the "unwarp" folder of the main experiment folder, and

Below are two examples of crystals that showed interesting diffuse scatter features. In Figure 2 you can see a "honeycomb" diffuse pattern, while in Figure 3 you can see that there is no Bragg intensity present, only signal from diffuse scatter! Both images were hk3 planes, with $Os = (0 \ 0 \ 2.8)$, $Oe = (0 \ 0 \ 3.2)$, and integration unit

can be accessed via the **Image list** button.

Figure 3. Diffuse rings.

BOOK REVIEW

ATHENE DONALD

Science is a concise but compelling read for anyone and everyone. It's a book for women who have pursued a career in science, regardless of where they are in that career. It's also a book for men in the same position. It's for anyone who has a young girl or young woman in their life with an interest in science and math. But it's also a book for anyone who has a young girl or young woman in their life who doesn't have an interest in science or math. Indeed, although Donald is writing about the importance of women in science and detailing the historical, present, and

Athene Donald's Not Just for the Boys: Why We Need More Women in

future impediments that can keep them from reaching their desired position in their respective fields, much of Donald's argument can be

applied to any field in which men outnumber women.

the sciences. While Donald's book will certainly resonate for any woman in

the sciences, if you're looking for a read about women of color in the sciences, Jasmine Brown's Twice as Hard is an excellent choice.

Review by Jeanette S. Ferrara, MFA

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