Volume 13, No. 8, August 2021

WELCOME

The last month has seen an incredible amount of activity: the ACA held its annual meeting (virtual) and workshops, and the Canadian National Committee for Crystallography (CNCCr) held its annual chemical crystallography workshop (also virtual).

As I write this, the 25th IUCr Congress (hybrid) is winding down. The General Assembly met yesterday to begin selection of new members of the Executive Committee and decide on the venue for the 27th IUCr Congress and General Assembly. I am pleased to report that Hanna Dabkowska has been chosen as the new President, Santiago Garcia Grande, the Vice President, and Bo Brummerstedt Iversen, the General Secretary and Treasurer. A lone, joint bid by the CNCCr, the USNC/Cr and ACA for Calgary, Canada was affirmed as the winning bid for the 27th Congress. Elections for ordinary members of the Executive Committee are ongoing as I write but should be completed by the time you read this.

The summer conferences were dominated by three main themes: the crystallographic community's response to the pandemic, a celebration of the PDB's 50th anniversary and 3DED/MicroED. Other interesting topics included quantum crystallography, magnetic structures, and total scattering. ACA and IUCr both conducted workshops on 3DED/MicroED.

I look forward to seeing you in Portland next year, Baltimore and Melbourne in 2023.

Stay positive and test negative,

Joe

RIGAKU'S VIRTUAL MEETING POSTERS

Rigaku's scientific single crystal staff produced several posters for the August ACA and IUCr meetings. In case you missed these posters at these virtual meetings, we are dedicating the August issue of Crystallography Times to sharing this information with our readership.

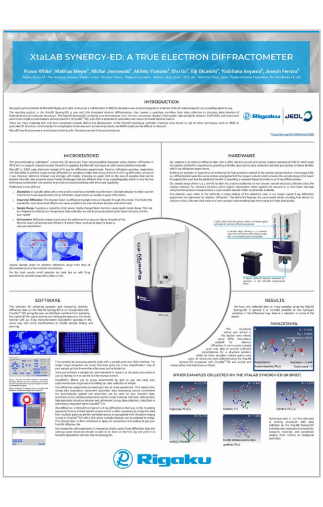
INTRODUCING THE XtaLAB Synergy Flow

The XtaLAB Synergy Flow turns any Synergy cabinet diffractometer into an automated, high-throughput machine by incorporating a 6-axis UR3 Universal Robot and a 3-puck dewar. The XtaLAB Synergy Flow system can automatically screen and collect 48 crystal samples with minimal human intervention. CrysAlis^{Pro} has been upgraded with tools to control all aspects of robotics and sample queuing. A unique X-ray safe dewar-drawer system allows loading and unloading of pucks without opening the X-ray enclosure or disturbing data collection. Ultimately, the XtaLAB Synergy Flow system is the perfect solution to allow full-time use of your diffractometer during a time when human interaction and contamination must be minimized.
Read More >

PDF ANALYSIS OF GLASSY MATERIALS ON A HOME LABORATORY DIFFRACTOMETER

There is no question that atomic Pair Distribution Function analysis has had a profound impact on the analysis of crystalline and amorphous materials. As a complement to the use of synchrotron sources for collecting PDF data, we have explored the use of home laboratory-based single crystal diffractometers to analyze amorphous organic materials. In order to generate the most useful reduced radial distribution functions, *G(r)*, we have found it necessary to modify existing code in CrysAlis^{Pro} and develop new code to generate *G(r)* data for refinement and analysis. In this presentation we will explore the collection and analysis of total scattering data glassy materials with wavelengths readily available to home laboratory systems.
Read More >

XtaLAB Synergy-ED: A TRUE ELECTRON DIFFRACTOMETER



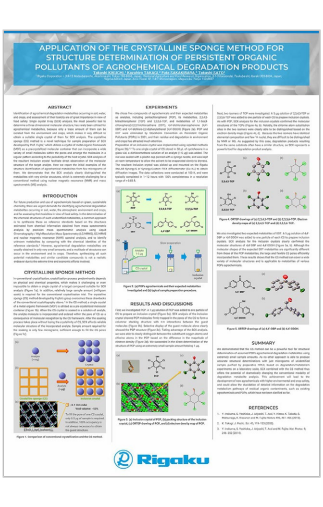
Recognizing the potential of MicroED, Rigaku and JEOL announced a collaboration in 2020 to develop a new product designed in a fashion that will make it easy for any crystallographer to use.

The resulting product is the XtaLAB Synergy-ED, a new and fully integrated electron diffractometer, that creates a seamless workflow from data collection to structure determination of three-dimensional molecular structures. The XtaLAB Synergy-ED combines core technologies from the two companies: Rigaku's high-speed, high-sensitivity detector (HyPix-ED), and instrument control and single crystal analysis software platform (CrysAlis^{Pro} ED), and JEOL's expertise in generation and control of stable electron beams.

There are many materials that only form nanosized crystals. Before the development of the MicroED technique, synthetic chemists were forced to rely on other techniques, such as NMR, to postulate 3D structure. Unfortunately, for complicated molecules such as natural products, the NMR results can be difficult to interpret.

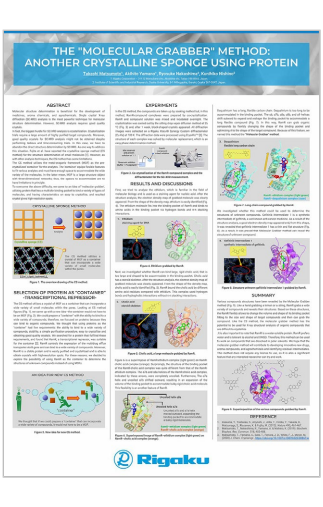
MicroED has thus become a revolutionary technique for the advancement of structural science.
Read More >

APPLICATION OF THE CRYSTALLINE SPONGE METHOD FOR STRUCTURE DETERMINATION OF PERSISTENT ORGANIC POLLUTANTS OF AGROCHEMICAL DEGRADATION PRODUCTS



Identification of agrochemical degradation metabolites occurring in soil, water, and crops, and assessment of their toxicity are of great importance in view of food safety. Single crystal X-ray (SCX) analysis, the most powerful tool to determine a three-dimensional molecular structure, has rarely been utilized for agrochemical metabolites, because only a trace amount of them can be isolated from the environment and crops, which makes it very difficult to obtain a suitable single crystal of them for SCX analysis. The crystalline sponge (CS) method is a novel technique for sample preparation of SCX developed by Prof. Fujita, which utilizes a crystal of metal-organic framework (MOF) as a pre-crystallized molecular container that can incorporate a wide variety of small molecules within the pores and arrange the molecules in a regular pattern according to the periodicity of the host crystal. SCX analysis of the resultant inclusion crystal facilitate direct observation of the molecular structure of the target analyte. Here we report the initial examples of the structure identification of agrochemical metabolites from only a few micrograms of them. We demonstrate that the SCX analysis clearly distinguished the metabolites with very similar structures, which is extremely challenging for a conventional method using nuclear magnetic resonance (NMR) and mass spectrometric (MS) analysis.
Read More >

THE MOLECULAR GRABBER METHOD: ANOTHER CRYSTALLINE SPONGE USING PROTEIN



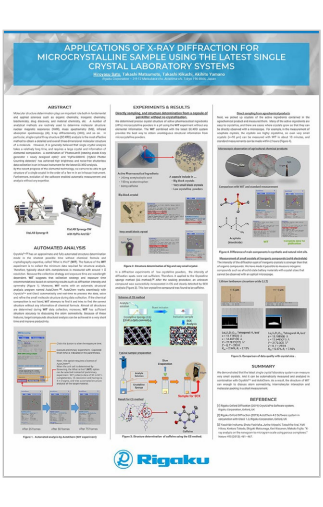
Molecular structure determination is beneficial for the development of medicines, aroma chemicals, and agrochemicals. Single crystal X-ray diffraction (SC-XRD) analysis is the most powerful technique for molecular structure determination. However, SC-XRD analysis requires good quality crystals.

In fact, the biggest hurdle for SC-XRD analysis is crystallization. Crystallization trials require a large amount of highly purified target compounds. Moreover, good quality crystals for SC-XRD analysis might not be obtained despite performing tedious and time-consuming trials. In this case, we have to abandon the direct structure determination by SC-XRD. As one way to address this situation, Fujita et al. have reported the crystalline sponge method (CS method) for the structure determination of small molecules. However, as with other analysis techniques, the CS method has some limitations.

The CS method utilizes a metal-organic framework (MOF) as the pre-crystallized "container" for the analytes. The "container" is equipped with flexible features to fit various analytes and must have enough space to accommodate a wide variety of molecules. A MOF is a large structure object with three-dimensional networks; thus, the spaces to accommodate molecules are to have limitations in principle.

To overcome the above difficulty, we came up with the idea of a "molecular grabber," utilizing a protein that has a multisite binding pocket to bind a variety of types of molecules, and having characteristics of being easy to crystallize, and the resulting crystal gives high-resolution spots.
Read More >

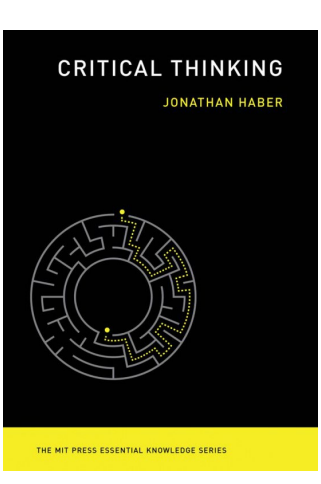
APPLICATIONS OF X-RAY DIFFRACTION FOR MICROCRYSTALLINE SAMPLES USING THE LATEST SINGLE CRYSTAL LABORATORY SYSTEMS



Molecular structure determination plays an important role both in fundamental and applied sciences such as organic chemistry, inorganic chemistry, biochemistry, drug discovery, and material chemistry, etc. A number of analytical methods are routinely used to determine molecular structure: nuclear magnetic resonance (NMR), mass spectrometry (MS), infrared absorption spectroscopy (IR), X-ray diffractometry (XRD), and so on. In particular, single-crystal X-ray structure (SC-XRD) analysis is the most effective method to obtain a detailed and overall three-dimensional molecular structure of a molecule. However, it is generally believed that single crystal analysis takes a relatively long time and requires a large crystal and information of elemental composition. A combination of "PhotonJet-R (rotating anode X-ray generator + newly designed optic)" and "HyPix-6000HE (Hybrid Photon Counting detector)" has achieved high brightness and noise-free shutterless data collection in an in-house instrument for the latest SC-XRD analysis.

Recent progress in this fundamental technology allows us to get the structure of a single crystal in the order of a few minutes in an in-house instrument. Furthermore, evolution of the software enabled automatic measurement and analysis without any expertise.
Read More >

BOOK REVIEW



Critical Thinking
By Jonathan Haber
ISBN 978-0262538282

Jonathan Haber's *Critical Thinking* is the latest installment in the MIT Press Essential Knowledge Series. Per the foreword, it's one of the "accessible, concise, beautifully-produced pocket-sized books on topics of current interest" in the series.

The back cover declares its purpose: "how the concept of critical thinking emerged, how it has been defined, and how critical thinking skills can be taught."

It's a clear summary of Haber s approach to critical thinking, although it excludes the introductory context Haber provides in the preface why we should care. Haber illuminates the importance of critical thinking in an advanced society, as well as what may be considered the decline of critical thinking or the apparent decline of critical thinking in the United States in the past few years. Critical thinking skills, or the lack thereof, can contribute to a person's ability or inability to parse information particularly information of a false nature.

Haber starts with a history of critical thinking, largely attributing it to the practice of philosophy, the study of logic and rhetoric, and the concept of syllogisms. He traces a social approach to critical thinking in education, explaining how the concept has been defined and taught over the generations.

But perhaps most importantly, Haber ends his book with a chapter titled "Where Do We Go from Here?" In this final chapter, Haber outlines the requisite steps for both educators and families to respectfully stimulate and cultivate critical thinking skills in their students and children, but also in themselves. Critical thinking is not like riding a bike. It's not a skill set you learn one time that you carry with you for the rest of your life. It's one that must be learned and practiced every day, by approaching the world around you with an approach based on logical reasoning and fact, not one based on emotions and fear-based falsehoods.

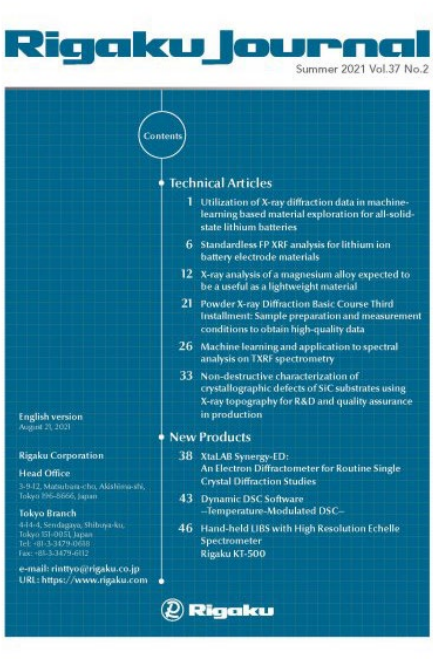
The design and presentation of the book is indeed beautiful as well as delightfully minimalist, as one might expect from a work advertised as "pocket-sized." And the subject matter is conveyed in an accessible and concise manner. All in all, it hits the mark.

Jeanette S. Ferrara, MFA

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 diffraction, X-ray fluorescence and X-ray imaging. You can register here and also watch recordings if you cannot attend live sessions.

USEFUL LINKS



SUMMER 2021, VOL.37, NO.2

The new edition of the Rigaku Journal has been released. You can read any of the articles by clicking on a title below.

Utilization of X-ray diffraction data in machine-learning based material exploration for all-solid-state lithium batteries
Kota Suzuki, Masaaki Hirayama, and Ryoji Kanno

Lithium-ion batteries are secondary (rechargeable) batteries that are used for a wide range of applications, from mobile devices to electric vehicles, as they combine both high energy density and excellent power characteristics. In recent years, research has been conducted toward the realization of an all-solid-state lithium battery...

Standardless FP XRF analysis for lithium ion battery electrode materials
Hikari Takahara and Hironori Kobayashi

Standardless FP X-ray fluorescence analysis is a quantification method using theoretical calculations including fundamental parameters. The analysis method has been widely used in the electronics and petrochemical industries, among others, since it can simply and quickly quantify sample compositions from spectral peak intensities without preparing calibration curves.

X-ray analysis of a magnesium alloy expected to be a useful lightweight material
Akimitsu Nezu, Wataru Matsuda, and Junichi Sato

Weight saving is an important challenge for various industries, including transportation (automotive, aeronautical, or bullet-train manufacturing), electronic devices, and intelligent robotics. Finding lighter-weight materials is, therefore, a popular research subject because of its potential impact on peoples' daily life. This is especially true in the modern automotive industry...

Powder X-ray Diffraction Basic Course | Third Installment: Sample preparation and measurement conditions to obtain high-quality data
Masashi Omori

In the second installment of the powder X-ray diffraction (PXRD) basic course, how to select instrument configurations to obtain high-quality data was described. This third installment provides information on how to prepare samples and determine the best measurement conditions to obtain high-quality data.

Machine learning and application to spectral analysis on TXRF spectrometry
Makoto Doi and Shinya Kikuta

Total Reflection X-ray Fluorescence (TXRF) analysis is a non-destructive and surface-sensitive analysis method using X-rays, in which incident X-rays are irradiated on a sample at an extremely low grazing angle (about 0.1°) and the fluorescent X-rays from the sample generated by the incident X-rays are measured with extremely low background.

Non-destructive characterization of crystallographic defects of SiC substrates using X-ray topography for R&D and quality assurance in production
Christian Reimann and Christian Kranert

One of the major technical challenges of this decade are energy efficient technologies, which is among others, comparable in its importance to Artificial Intelligence, 5G and IoT. Innovative silicon carbide (SiC) technology and components will contribute significantly towards the goal of a greener, energy efficient and sustainable economy.

XtaLAB Synergy-ED: An Electron Diffractometer for Routine Single Crystal Diffraction Studies

Recognizing the potential of MicroED, Rigaku and JEOL announced a collaboration in 2020 to develop a new product designed in a fashion that will make it easy for any crystallographer to use. The resulting product is the XtaLAB Synergy-ED, a new and fully integrated electron diffractometer...

Dynamic DSC Software Temperature-Modulated DSC

Differential scanning calorimetry (DSC) is a thermal analysis technique that measures the change in heat capacity of a sample, or endothermic/exothermic reactions, based on the difference in temperature between a sample and a reference material that are both heated/cooled at a predetermined constant rate.

Hand-held LIBS with High-Resolution Echelle Spectrometer Rigaku KT-500

Rapid Analysis of Carbon in Steel and High Performance Analysis of Stainless and High Temperature Alloys.

The Rigaku KT-500 hand-held analyzer represents the next advancement in handheld laser induced breakdown spectroscopy (LIBS).

JOIN US ON LINKEDIN

Our LinkedIn group shares information and fosters discussion about X-ray crystallography and SAXS topics. Connect with other research groups and receive updates on how they use these techniques in their own laboratories. You can also catch up on the latest newsletter or *Rigaku Journal* issue. We also hope that you will share information about your own research and laboratory groups.

JOIN HERE

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 CrysAlis^{Pro} software for single crystal data processing.

JOIN HERE