

ICDD Annual Spring Meetings

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The International Centre for Diffraction Data® (ICDD®) is a non-profit scientific organization dedicated to collecting, editing, publishing, and distributing powder diffraction data for the identification of crystalline materials.

ICDD had a successful 2015 Spring Meetings Week from 23 to 27 March 2015. There were 54 attending members from 12 countries.

Early on Tuesday, everyone enjoyed the Plenary talks by Bob B. He, from Bruker AXS, who gave a talk entitled *RECENT ADVANCES IN TWO-DIMENSIONAL XRD*. Brian Toby, from Argonne National Laboratory, Advanced Photon Source, chose the following topic of discussion: *WHY WE ARE WRITING GSAS-II IN PYTHON*. Jacco van de Streek, of the University of Copenhagen, Department of Pharmacy, spoke about *XRPD + DFT-D: VALIDATION, EXAMPLES AND FUTURE*. Amy Gindhart and Kai Zhong, both from the International Centre for Diffraction Data, explained the following: *INCORPORATION OF PHARMACEUTICAL API'S INTO THE PDF® DATABASES* (Figure 1).

This year's social event took place at the Delaware Museum of Natural History (DMNH), located in Wilmington, Delaware. The evening ended with a 100 person member dinner

and Poster Session at the nearby Mendenhall Inn. There were 29 posters, including 14 from ICDD contributing authors. Twenty ICDD employees authored or coauthored a poster (Figure 2).

Congratulations to Jim Kaduk, Illinois Inst. of Technology and Ekaterina Pomerantseva, Drexel University, for their third place tie; Winnie Wong-Ng, National Institute of Standards and Technology, for her second place poster, and Benjamin Trump, Johns Hopkins University, for his first place win for his poster "*SELECTIVE AREA ELECTRON DIFFRACTION AND SIMULATED ANNEALING OF POWDER X-RAY DIFFRACTION FOR THE DETERMINATION OF NEW IRIDATE CRYSTAL STRUCTURES*" (Figure 3).

Wednesday was well attended, with meetings of the many ICDD committees and subcommittees, including PDF Editorial Staff, Scholarship, X-ray Diffraction Methods, X-ray Fluorescence, Metals & Alloys, Micro and Meso, Education, Ceramics, Polymers, Minerals, Electron Diffraction, Synchrotron & Neutron Scattering Methods, Non-Ambient Diffraction, and Organic & Pharmaceutical.

During the New Product Developments meeting on Thursday morning, Justin Blanton, ICDD's Manager of Engineering and Design, unveiled the latest features of the



Figure 1. (Color online) Plenary Speakers – K. Zhong, B. Toby, A. Gindhart, B. He & J. van de Streek.



Figure 2. (Color online) H. McDonnell & D. Barry at DMNH.

PDF products. Tom Blanton, ICDD's Database Manager and Principal Scientist, demonstrated importing 2D patterns (Figure 4).

All the week's work came together with the Technical Committee Meeting, overseen by Matteo Leoni. Ben Trump gave the Ludo Frevel Crystallographic Scholarship Recipient Presentation. Also, there were reports from the Regional Co-chairs, and the technical subcommittees including motions

from the various subcommittees. These ultimately improve the ICDD database and enhance our software (Figure 5).

On Thursday afternoon, ICDD Chairman, Scott Misture, presided over the Annual Members Meeting. He was pleased to surprise Bob He with the *Fellow* award and John Faber with the *Distinguished Fellow* award. Brian Toby was named the 2015 Barrett Award recipient. Brian will accept the award during the Plenary Session at the Denver X-ray Conference in



Figure 3. (Color online) S. Misture & B. Trump.



Figure 4. (Color online) J. Blanton.

August. ICDD Executive Director, Tim Fawcett, reviewed Headquarters' Activities, and presented the Denver X-ray Conference Report. There were also reports of selected Board of Directors' committees.

Before the Report of the Finance Committee, David Taylor was given a beautiful specimen of petrified wood to honor 10 years of service as ICDD Treasurer. He is pictured

here with Tim Fawcett, and three chairmen of the Board of Directors with whom he served (Figure 6).

Full details of the ICDD meetings can be found at www.icdd.com/profile/march14.htm

Abstracts submitted for the poster session are given below. Further information about each abstract can be obtained by contacting the author whose name is underlined.

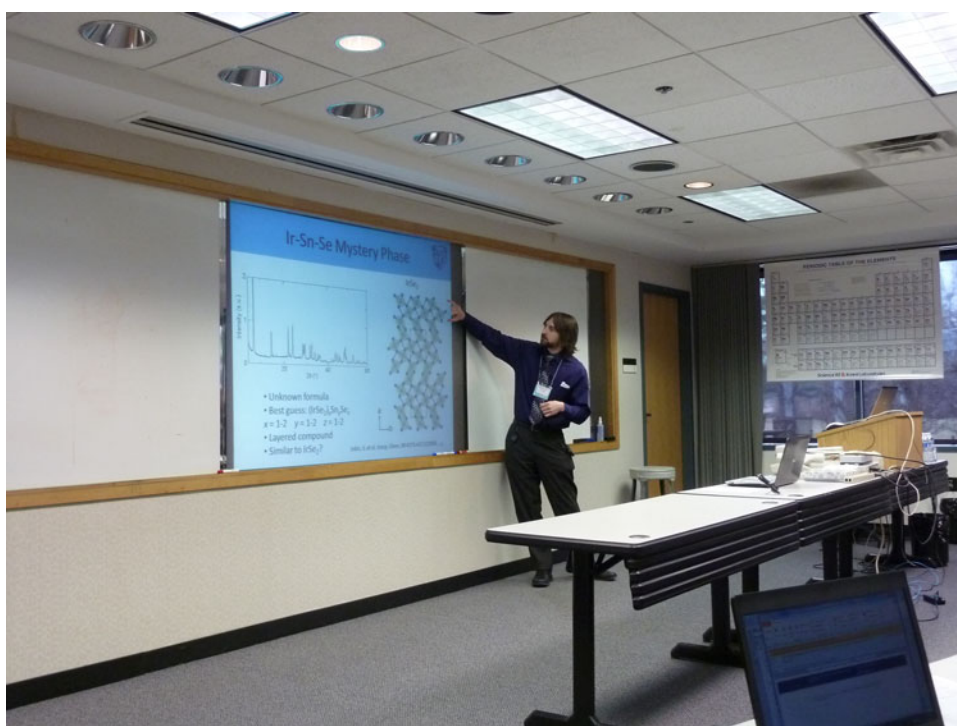


Figure 5. (Color online) B. Trump.



Figure 6. (Color online) T. Fawcett, S. Misture, D. Taylor, J. Kaduk & T. Blanton.

SUBFILE POPULATION IN 2015 PDF[®] RELEASES

V. Bosnic, S. Kabekkodu, L. Lanno, A. Gindhart, S. Gates, E. Foster, and M. Carr

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The classification of entries into various subfiles is important to enhance the effectiveness of search/match for phase identification using the Powder Diffraction File[™] (PDF[®]). The use of subfiles becomes obvious when considering there are over 900, 000 entries in the combined PDF-4+ and PDF-4/Organics databases. The identification and assignment of subfiles is accomplished in two ways: (i) manually, by the International Centre for Diffraction Data (ICDD)'s editorial team and task group members, (ii) semi-automatically by a query-based assignment that compares entries with matching chemistry and similar crystallographic parameters (with a user-defined tolerance factor). New subfile entries are reviewed by various editors and task group members for consistency. This poster is a report on the specialty subfile population in the upcoming 2015 PDF releases.

NIST STANDARD REFERENCE MATERIALS FOR X-RAY METROLOGY

J.P. Cline, M. Mendenhall, D. Windover, and A. Henins
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The powder diffraction method has an exceptionally broad range of applications owing to its relative ease of use and its applicability to any crystalline material. Furthermore, powder diffraction can access a considerable breadth of sample characteristics: qualitative and quantitative phase analysis as well as crystal- and micro-structure determinations.

Each application presents a specific challenge with regards to the development of a standard, both in the context of the character of the standard itself and the method by which it is certified.

The overall technical approach can be divided into a few subtopics: (1) microstructure engineering and/or evaluation to obtain standard reference material (SRM) feedstock suitable to address the measurement issue at hand, (2) the design and commissioning of the equipment that embodies the unique features required for SI traceable measurements, and (3) the design of experiments, measurement methods, and data analysis strategies that allow for a valid assessment of systematic uncertainties of the measurement quantities. A last component of the program entails the examination of the techniques and data analysis methods by which SRMs are used for the calibration of instruments in the field.

The success of an SRM is often dependent on obtaining a feedstock of the appropriate microstructure. Several years were expended to develop the preparation process used for the feedstock of the new nano-crystallite size SRM 1979. The process that was used consisted of the decomposition of zinc oxalate in a custom NIST-built vacuum furnace to yield two powders with median crystallite sizes of about 15 and 60 nm. We have now collected two sets of data on this material on a machine equipped with an incident beam monochromator and either a fixed slit and scintillation detector or a silicon-strip position-sensitive detector. We will present the data analysis from this material, showing how a combination of the fundamental parameters approach and extraction of the Fourier transforms of isolated peaks allows computation of the necessary parameters in Fourier space, where the detailed theory for particle size distributions is derived.

Recent line position SRMs as has been previously reported, have been certified with respect to lattice parameter

in an SI traceable manner using the NIST-built divergent beam diffractometer. While this divergent beam diffractometer includes several unique features that enable it to provide SI traceable measurements, we have also commissioned a parallel beam diffractometer that includes additional features which will yield measurements with reduced error bounds. We have now realized calibration of the optical angular encoders on this machine; with the determination of both short range and long-range corrections, to yield an angular measurement accuracy of ± 0.035 arc-seconds. We also report on the results of a double crystal experiment that was successfully used to examine the $\text{CuK}\alpha$ emission spectrum. This experiment will be repeated after the graded parabolic optic is installed as it is known that this optic distorts the spectrum to some extent.

THE POWDER DIFFRACTION PATTERN AND THE CRYSTAL STRUCTURE OF A NEW ZN-FLUCONAZOLE COMPLEX

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Fluconazole is a well-known antifungal belonging to the azole family of compounds. It is administered topically, orally, and intravenously to treat common infections such as *tinea pedis* (athlete's foot) and *tinea cruris* (jock itch). As part of the work being carried out in our laboratory to explore the possible formation of polymorphs of some highly used drugs and some of their metal complexes, we have synthesized a new zinc complex of fluconazole. Reaction of fluconazole and $\text{ZnSO}_4 \cdot 7\text{H}_2\text{O}$ in a 1:1 molar ratio in water produced over a period of 3 months crystalline colorless material. The powder diffraction pattern was recorded on a Bruker D8 Advance diffractometer working in the Bragg-Brentano geometry, using $\text{CuK}\alpha$ radiation, operating at 40 kV and 30 mA. The data were collected in steps of 0.01526° (2θ), from 5° to 70° at 1.5 s step⁻¹. The diffractometer is equipped with primary and secondary Soller slits of 2.5° , a divergence slit of 0.2 mm, Ni filter of 0.02 mm, and a LynxEye detector. After the peak positions were established with FullProf, the indexing of the pattern was successfully performed with Dicvol06 in a triclinic unit cell. The analysis of the 89 diffraction maxima registered, carried out with NBS*AIDS83, led to the following parameters: $a = 10.955(2)$, $b = 13.693(1)$, $c = 17.113(2)$ Å, $\alpha = 104.31(1)^\circ$, $\beta = 90.17(1)^\circ$, $\gamma = 90.28(2)^\circ$ with $V = 2487.5(4)$ Å³. The figure of merits were $M20 = 34.4$ and $F30 = 90.3$ (0.0050; 67). The single-crystal structure determination confirmed that the unit cell was correct. A search in the Cambridge Structural Database (CSD) indicated that this was a new compound. This material was also characterized by FT-IR and TGA/DTA analysis. In the present contribution, the powder diffraction pattern and the most relevant structural features of the Zn-fluconazole complex obtained will be presented.

ACKNOWLEDGEMENTS

This work has been supported by a grant (no. LAB-97000821) from FONACITVenezuela for Laboratorio de Cristalografía-LNDRX and a grant for Laboratorio de Difracción de Rayos X, PTG, Universidad Industrial de Santander, Bucaramanga, Colombia. The authors thank M.Sc. José Luis Pinto for technical assistance with the powder diffractometer.

POWDER DIFFRACTION JOURNAL IN REVIEW 2014

N. Ernst Boris

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This past year *Powder Diffraction* had both editorial changes and additional content for our readers. The Australian X-ray Analytical Association (AXAA) proceedings were published and a special issue on XRD Software was published in addition to our regular four issues. We had a good number of submissions and a high interest in our content as shown by the number of downloads and top articles viewed. International Centre for Diffraction Data (ICDD) was also pleased to announce that Camden Hubbard became the Editor-in-Chief of the journal. In 2015 we will once again be publishing the European Powder Diffraction Conference proceedings (EPDIC14). Finally, the author notes have been updated to reflect changes, such as the addition of Data Reports as a manuscript category and updates to the New Diffraction Data and Rapid Communications sections.

STUDY OF SRM 660B USING NEUTRON TIME-OF-FLIGHT DIFFRACTION DATA FROM POWGEN AT THE SPALLATION NEUTRON SOURCE (SNS)

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In a recent synchrotron X-ray diffraction study of SRM 660A, Faber and Suchomel showed that approximately 680 ppm(wt.) of ZrB₂ was present in SRM 660A. A detailed analysis of the data suggested that the second phase altered the stoichiometry of the Standard Reference Material (SRM). A new SRM 660B has been formulated and issued by National Institute of Standards and Technology (NIST, Black et al. 2011). The SRM was prepared using 11B isotopically enriched precursor material. This isotopically enriched material was designed to meet the needs of the neutron scattering community (low absorption and incoherent cross-sections). In contrast to X-ray scattering, the scattering length for 11B = 0.665 cm^{-12} whereas that for La = 0.824 cm^{-12} . Moreover, 11B enriched LaB₆ is used as a lattice parameter standard for neutron diffraction powder diffraction instrumentation. In this paper, we report the results of neutron diffraction experiments using the high-resolution neutron powder diffractometer on BL-11 at the spallation neutron source (SNS). GSAS-II (Toby and Von Dreele, 2013) was used to analyze the diffraction data. Within the sensitivity of the results, no

second phase was detected in these experiments. The crystallographic model used to analyze the data will be discussed in detail. Of particular interest is the occupation of 11B among equivalent crystallographic sites. The derived composition of the sample is LaB₆.000 (3). The conclusion is that SRM-660B is stoichiometric. This strengthens our conclusions about SRM 660A behavior. We will compare the neutron TOF results with those from X-ray experiments from BM-11 at the APS.

ACKNOWLEDGEMENTS

A portion of this research at ORNL's Spallation Neutron Source was sponsored by the Scientific User Facilities Division, Office of Basic Energy Sciences, US Department of Energy.

Black, D. R., Windover, D., Heinins, A., Filliben, J., and Cline, J. P. (2011). *Powder Diffraction* 26, Special Edition 02, 155–158.

Toby, B. H., and Von Dreele, R. B. (2013). *J. Appl. Crystallogr.* 46, 544–549.

NEW PROJECT TEAM ADVANCEMENTS AT ICDD

E. Foster, S. Gates, D. Sagnella, M. Rost, M. Hammock, and S. Jennings
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In September, the International Centre for Diffraction Data (ICDD) New Project Team was formed. Continuing on from the PDF-4/Organics 2015 product, current publications are routinely monitored for single crystal organic data. This process results in cutting-edge materials being added to our database. From these journals alone, there are approximately 2400 papers that are ready to be abstracted for insertion to the database. A new vision for the PDF-4/Organics 2016 release is currently being realized. Rather than concentrating on getting as many patterns into the database as possible, our focus has shifted to getting as many special interest materials into the database as possible. This is achieved by maintaining our stringent editorial system that all powder diffraction file (PDF) products go through as well spending more time seeking data for our commercially valuable sub-files: pharmaceuticals, bioactive compounds, excipients, forensic compounds, pigments, and dyes.

Outside of the Organics Project, the New Project Team is also focusing on collecting diffraction data on both nanomaterials and polymers. Unlike patterns for crystalline materials, the ICDD is primarily focused on specific 2θ ranges for both nanomaterial and polymer patterns. Thus, a modified "Writes" letter has been drafted for these materials. The "Writes" letter asks the author for much more specific criteria when it comes to submitting their data. This is also facilitated by having members of the New Project Team investigate the literature for evidence that the experimenters have conducted a well-defined experiment and collected the data that the ICDD requires.

Though Genie was not a new concept for 2014, it was the first year that Genie became used much more widely outside of the ICDD community. The process of receiving Genie patterns has become much more streamlined over the past year. Currently, the ICDD will accept organic single crystal or powder diffraction data through Genie to go into our PDF products. In 2014, 74 usable patterns were submitted through the Genie web application. In 2014, four patterns were submitted by ICDD members that were published in *Powder Diffraction*. An additional, unsolicited pattern was submitted for publication and is currently being processed. In addition to the processing of Genie patterns becoming more streamlined, a paper was published this year describing the functionality of the application (Sagnella and Foster, 2014).

Sagnella, D. E. and Foster, E. (2014). Genie: new software for rapid submission of powder diffraction data to the ICDD powder diffraction file™. *Powder Diffr.* 29, S31–S34. doi: 10.1017/S0885715614000864.

ADVANCES IN 2D DIFFRACTION: INCORPORATING 2D DATA INTO THE PDF™

S. Gates, K. Zhong, T. Blanton, and J. Blanton
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Annually, updates are made to the Powder Diffraction File™ (PDF) in support of our mission statement focused on being the world center for quality diffraction and related data, to meet the needs of the technical community. These updates include the incorporation of new diffraction data entries, as well as implementation of various new features. One of our recent PDF-4 initiatives focuses on the incorporation of two-dimensional (2D) diffraction data into the PDF. Over the years, small sample sizes have become more attractive in scientific research. As a result, a need for more advanced methods for analyzing micro- and nano-sized samples has increased. Currently, the preferred/ideal method of collecting 2D XRD data requires the use of a micro-diffractometer and 2D detector. As a result, we are developing methods to read 2D diffraction data directly into the PDF for use in phase identification in order to accommodate the growing need of the diffraction community. Additionally, we are investigating methods to abstract 2D data published in literature. In carrying out these tasks, various factors need to be addressed, and this presentation discusses some of the benefits of incorporating this new feature, as well as some obstacles encountered along the way.

PDF-4+ TOOLS AND CAPABILITIES: CHARACTERIZATION OF MINERALS

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This presentation was shown at Geological Society of America (GSA) 2014 meeting, and demonstrates the various tools in PDF-4+, and their usefulness in characterizing

mineralogical samples. As an example a rock sample was investigated using powder X-ray diffraction, and the resulting diffraction data are characterized using SIEve+ to carry out phase identification. Once the appropriate phases are identified, various simulation tools are utilized to provide the user with additional information about the sample.

IDENTIFICATION AND ANALYSIS OF CLAY MINERALS USING THE POWDER DIFFRACTION FILE

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Clay minerals, a varied group of hydrous phyllosilicates occur widely in many rocks and soils but their precise identification by X-ray powder diffraction is more difficult than most other groups of minerals. These difficulties stem mainly from the facts that (1) many clay minerals are sensitive to environmental conditions and can imbibe a variety of polar and other molecules into their layer structure (interlayer) which may alter the positions and profiles of their diagnostic basal (00 l) spacing's, (2) a variety of both ordered and disordered layer displacements common in clay minerals may be expressed as different polytypes or may demodulate some non-basal (hkl) peaks to varying degrees into broad two-dimensional (hk) diffraction bands, often spanning a wide 2θ range and (3) mixed-layering is common in many clay minerals whereby variable proportions and intimate layer arrangement of two or more different layer types determine the positions of basal peaks and profiles observed.

There are a large amount of data in the powder diffraction file (PDF) pertaining to common clay minerals [291 entries retrieved PDF4 2013 (v4.1302) for smectites, kaolins, illites, glauconites, palygorskite, sepiolite, and mixed-layer clay minerals] but the aforementioned difficulties of clay mineral identification combined with the variable quality of PDF entries severely limit the utility of the PDF for clay mineral identification, even for specialist clay mineralogists.

A cursory review of the data is presented for montmorillonite [(all names contains words "montmorillonite") retrieves 50 entries] to illustrate the challenges that face analysts using the PDF data as a tool for clay mineral identification. The results suggest that a more detailed systematic review of clay mineral data in the PDF may be warranted to explore ways to assist in the use of the PDF for the identification of clay minerals in rocks and soils.

ICDD PDF-4 PRODUCT DEVELOPMENTS FOR RELEASE 2015

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The PDF-4 2015 product line will contain numerous improvements and several added capabilities for release in September. The data mining search has been completely

revamped to include "always visible" common International Centre for Diffraction Data (ICDD) filters; searches rearranged into more logical categories; color coded Quality Marks based on relative quality; and a dynamic search description that is automatically displayed as data mining criteria are selected. Users will be able to create custom "user-defined subfiles", which are personal sets of powder diffraction file (PDF) entries used for data mining, display, and analysis. The atomic coordinates search and display field have been updated to always include cross-referenced entries. In addition, a toggle has been added to temperature series graphs, allowing the user a convenient way to remove PDF entries where data collection occurred at room temperature.

New graphing and pattern simulation features have also been added to the PDF-4 2015 product line. All simulated diffraction patterns can now be plotted using square root intensities, as well as display the pattern's wavelength directly on the graph. Custom sets of simulated profile preferences can now be saved by the user for different X-ray/neutron/electron/synchrotron machine configurations. Debye-Scherrer geometry functionality has been extended to simulations that use non-characteristic X-ray wavelengths. An orientation distribution function (ODF) can now be applied to all ring pattern simulations to simulate 1D preferred orientation. Additionally, the d -spacings and intensities of an imported image pattern can now be calculated.

For the phase identification software SIEve/SIEve+, ICDD has significantly improved the importing and data-processing features available to the user. Experimental data file formats for Rigaku (*.ras) and FullProf (*.xyy/*.xy) software, as well as CIF's with pd_proc data, can now be imported into SIEve/SIEve+. Images of two-dimensional (2D) diffraction (ring) patterns can be integrated to create a 1D diffraction pattern used for phase identification. Also, the user can now create custom sets of data processing parameters for background removal, smoothing, $K\alpha_2$ stripping, and peak finding, which can be conveniently saved and retrieved. Lookup fields have been internally added for the 2nd Pass Filter algorithm (used for removing false positive matches), resulting in a 700% search performance speed increase.

These are just some of the many value-added developments designed to make the ICDD PDF-4 products more functional and powerful for 2015.

CRYSTAL STRUCTURES OF LARGE-VOLUME COMMERCIAL PHARMACEUTICALS

J. A. Kaduk,¹ J. W. Reid,² R. J. Papoular,³ K. Zhong,⁴
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Continuing the project to determine the crystal structures and to provide high-quality reference powder diffraction data of commercial pharmaceuticals, several new crystal structures have been determined:

Name	a (Å) α (°)	b (Å) β (°)	c (Å) γ (°)	V (Å ³)	Space group	Z
Atazanavir hydrogen sulfate	15.33431(8)	5.90392(3) 96.2922(4)	21.56869(9)	1940.91(2)	$P2_1$	2
Bortezomib	19.6961(8)	10.7089(6) 104.320(4)	14.8652(6)	3037.99(24)	$P2_1$	6
Citalopram HBr	10.76632(8)	33.0703(2) 90.8535(4)	10.89320(7)	3878.04(4)	$P2_1/c$	8
Methylprednisolone acetate	8.17607(2)	9.67945(3)	26.35168(7)	2085.470(9)	$P2_12_12_1$	4
Metoprolol tartrate	9.74907(8)	45.6066(4)	8.47632(10)	3768.76(7)	$Pnma$	4
Pantoprazole Na (H ₂ O) ₂	33.4766(6)	17.2934(1)	13.5592(1)	7848.74(15)	$Pbca$	16
Salmeterol xinafoate	9.1725(2) 82.273(2)	9.4814(2) 85.247(2)	21.3527(4) 62.143(1)	1626.48(6)	$P\bar{1}$	2
Solifenacin hydrogen succinate	6.47703(2)	7.83095(2) 90.2373(3)	23.84872(7)	1209.63(1)	$P2_1$	2
Telaprevir	37.6255(4)	19.3859(2)	10.5602(1)	7702.64(14)	$P2_12_12_1$	8
Tenofovir disoproxil fumarate	18.2396(10)	35.4932(18)	9.74575(27)	6309.2(4)	$P2_12_12_1$	4

A number of these structures have more than one molecule in the asymmetric unit ($Z' > 1$) of large unit cells, and have been challenging to solve and refine. The multiple molecules provide a way of exploring conformational space, and provide additional insight into the crystal energies. The hydrogen bonding will be discussed in as much detail as the progress of Density Functional Theory (DFT) calculations permits. Additional structures in progress may also be presented (Figure 7).

ICDD: EMBRACING SOCIAL MEDIA

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The ICDD.COM website strives to meet the information and communication needs of International Centre for Diffraction

Data (ICDD) members, customers, scholars, professionals, Grant-in-Aid participants, developers, and students. In the past 2 years, ICDD has embraced social media networks – creating and nurturing six different social profiles (Facebook, YouTube, Twitter, LinkedIn, Weibo, Google+). Our goal is to engage people who might not seek out information on ICDD.COM regularly. This presentation will describe ICDD's social media evolution, and how we plan to appeal to Internet users in the future.

WINPSSP: A REVAMP OF THE CRYSTAL STRUCTURE DETERMINATION PROGRAM PSSP WITH EDUCATIONAL PERSPECTIVES

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Figure 7. (Color online) J. Kaduk.

Crystal structure solution from powder diffraction is increasingly used in academic and industrial research. The automation of the structure solution process from powders using multi-module software suites is currently a very attractive option, requiring a steep learning curve because of the extent of the necessary pre- and post-processing tasks.

Powder diffraction specialists often use more than one software package for crystal structure determination, implementing different algorithms (direct-space methods, charge flipping, direct methods, etc.) depending on the material studied and the quality of the diffraction data. Direct-space methods using global optimization algorithms must find a set of parameters among a large number of trial structural models, which minimize the difference between the observed and calculated powder diffraction data, while optimizing the structural model (Cerny and Favre-Nicolin, 2007 and Pagola and Stephens, 2010).

The computer program PSSP (Pagola and Stephens, 2010) (direct-space methods) uses the simulated annealing algorithm for the location of the asymmetric unit in the crystal lattice of known space group symmetry and lattice parameters. PSSP currently runs in MS-DOS mode, and its scope is towards experienced scientists in the field of powder diffraction. We propose to develop a new computer program for crystal structure solution from powders based on the open source code of PSSP, with a new intuitive user interface suitable for familiarizing undergraduate college students to the topic, but also with advanced features for research purposes. Our work has three major goals: (i) to optimize the code for faster and smoother runs, (ii) to enrich the working environment with a Graphical User Interface (GUI) working in windows-based OS, and (iii) to facilitate additional educational characteristics to introduce the crystallographic and diffraction nomenclature to undergraduate research projects, undergraduate courses, and laboratory experiments.

Cerny, R. and Favre-Nicolin, V. (2007). "Direct space methods of structure determination from powder diffraction: principles, guidelines and perspectives", *Z. Kristallogr.* **222**, 105–113.

Pagola, S. and Stephens, P. W. (2010). "PSSP, a computer program for the crystal structure solution of molecular materials from X-ray powder diffraction data", *J. Appl. Crystallogr.* **43**, 370–376.

ICDD'S COMMITMENT TO EDUCATION: THE PERSONAL SIDE!

E. Jennings, S. Jennings, T. Maguire, and D. Zulli
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In compliance with the requirements of Pennsylvania's Nonprofit Corporation Law, International Centre for Diffraction Data (ICDD) is organized exclusively for charitable, educational, and scientific purposes. With that charter in mind, ICDD focuses many of its philanthropic efforts in supporting students – the next generation of X-ray analysts.

ICDD offers many incentives, grants, and scholarships to help students advance their education and further their career.

For example, the Ludo Frevel Crystallography Scholarship Program has been supporting student research since 1991. Since its inception, the program has awarded \$404,750 in support of 171 scholarships.

ICDD offers tuition waivers for several of its annual training events: the X-ray fluorescence (XRF) Clinic, the X-ray powder diffraction (XRD) I and II Clinics, and the Rietveld Refinement and Indexing Workshop. Established to promote the education of the scientific community in X-ray materials analysis, the waivers are targeted to professors and graduate students to encourage them to incorporate XRD–XRF topics into their curriculum. The waivers are named for pioneers in the industry: Eugene Bertin, Ron Jenkins, Frank McClune, and Deane Smith.

Many opportunities for student development are available at the annual Denver X-ray Conference (DXC). In addition to reduced registration fees and reduced housing costs, the conference also offers a limited number of travel grants, named in honor of Robert L. Snyder, to students who participate in the technical program with an oral or poster presentation. Approximately ten grants are awarded in the amount of \$500 for undergraduate or graduate students living within the USA, and \$1000 for those living outside of the USA. Recipients also receive free registration to attend the DXC. Further opportunities are available through the Jerome B. Cohen Student Award, which recognizes outstanding achievements of student research in X-ray analysis. Graduate and undergraduate students are encouraged to submit a technical paper describing their work, several weeks prior to the conference. The recipient is selected by a committee of researchers in the field, and receives a \$1000 award. More accolades are available to students who submit poster presentations. Two of the poster prizes at each of the XRF and XRD Poster Sessions are reserved for student submissions. Finally, ICDD sponsors a student lunch or breakfast on the first day of the conference. This gathering serves as an introduction to the conference, its Organizing Committee, available networking opportunities, and overall, provides helpful hints to make the most of their DXC experience.

See some of the faces of those who benefited from ICDD's support in the past year in this poster presentation!

THE SURPRISING *AB INITIO* STRUCTURE SOLUTION OF A NEW METAL-ORGANIC COMPOUND USING TWO X-RAY DIFFRACTION UNCONVENTIONAL INSTRUMENTS

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New metal–organic/anionic–cationic compounds, such as [Cu(C₁₂H₁₀N₂)₂]²⁺, PF₆[−]], are currently being synthesized in the context of developing new compounds for CO₂ manipulation and utilization.

The *ab initio* structure solution of the aforementioned compound was consistently obtained twice and independently

by making use of two diffractometers seldom used for this task: a benchtop Bruker AXS D2 Phaser powder diffractometer predominantly used for phase identification, and a Bruker AXS Prospector usually geared toward X-ray single-crystal structure determination. In the latter case, the powder sample was glued using a tiny drop of oil and then mounted in a loop, the usual sample holder for a single crystal. Even though a regular capillary could have been used, it was not in our instance in order (1) to avoid the small-angle scattering because of the latter and (2) to check the feasibility of an *ab initio* structure solution from the ensuing data.

ACKNOWLEDGEMENTS

Last but not least, both X-ray experiments were carried out at room temperature and during the 2013 ACA Summer Course of Chemical Crystallography. Both the course organizers and IMSERC at Northwestern University are most gratefully acknowledged.

THE NATURAL MAGNETIC MINERAL THAT WAS NOT

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A beautifully glittering material bought as Hematite on Vulcano Island (Sicily) turns out to be plain fabricated SiC. First correctly identified at mere sight by an expert mineralogist, Jolyon Ralph, a few years ago, our sample was then and quite independently characterized using neutron and X-ray powder diffraction (XRPD) coupled with the use of the ICDD PDF-4 database and its associated SIEVE+ search tool (PDF-4 User's Guide to Synchrotron Data Analysis and Neutron Data Analysis, 2014). So were minor impurity phases added to the sample during its grinding into a powder suitable for both the neutron powder diffraction (NPD) and XRPD measurements.

In more detail, a neutron experiment was first run on the G41 powder diffractometer at the CEA-Saclay / Orpheus Reactor. The absence of a magnetic diffraction signal at low temperature (around $T = 4$ K) raised suspicion about the true nature of our sample.

The latter was then further measured with X-rays using both a Bruker AXS D8 powder diffractometer (University of Ioannina) and a Bruker AXS Prospector single crystal diffractometer endowed with Helios MX optics and an APEX II CDD detector allowing for powder diffraction measurements (Northwestern University), thus providing us with the additional powder diffractograms used in our present study.

ACKNOWLEDGEMENT

We warmly thank Jolyon Ralph [www.mindat.org], whose invaluable help is most gratefully acknowledged.

PDF-4 User's Guide to Synchrotron Data Analysis and Neutron Data Analysis, ICDD publication (2014).

CRYSTAL STRUCTURE MODIFICATION AND ENHANCED ELECTROCHEMICAL PERFORMANCE IN α -MnO₂ NANOWIRES VIA ACID-LEACHING

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Despite increased interest in energy storage technologies in recent years, there have been only few discoveries of new materials for metal-ion batteries. Therefore, it is important to develop methods to modify and improve the materials known to reversibly intercalate charge-carrying ions (e.g. Li⁺ or Na⁺). In this work, we propose acid-leaching as a post-synthesis treatment to chemically modify battery electrode materials and improve their electrochemical performance. Because this method is being studied for intercalation hosts, it is important to determine both the effects acid-leaching has on the crystal structure of materials and the relationship of those changes to the properties of the material. The goal of this study is to monitor changes in crystal structure of intercalation host and correlate these changes with electrochemical performance.

As a model material to investigate the effects of acid-leaching, we synthesized α -MnO₂ (K_{0.11}MnO₂) nanowires using hydrothermal methods. This material crystallizes in a body-centered tetragonal crystal structure belonging to the space group *I4/m*. The structure is composed of MnO₆ octahedra units arranged two by two to form a tunnel structure with side lengths of 4.6 Å. The tunnels, which run along the nanowire growth direction, are stabilized by

K⁺ ions. α -MnO₂ nanowires were immersed in concentrated nitric acid for 2.5–27 h, and X-ray powder diffraction was utilized to evaluate changes in the material's structure. It was confirmed that the tetragonal crystal structure was maintained for all samples, but the lattice parameter *a* was found to decrease from the pristine sample (no acid-leaching) to a sample acid-leached for 2.5 h. Further reduction in the lattice parameter *a* was seen for a sample acid-leached for 27 h. The lattice parameter *c* remained almost unchanged. These results indicate that acid-leaching causes an anisotropic inward shrinkage of the tunnel structure, caused by the removal of the stabilizing K⁺ ions from the tunnels. This is supported by the fact that the calculated K occupancy inside of the tunnels decreased with increase in acid-leaching time, in agreement with EDS data that showed a decreasing K concentration with longer acid-leaching. TEM images of nanowires acid-leached for 27 h demonstrated formation of the amorphous material on the surface of the wires and defects in the structure. Electrochemical testing revealed that the specific capacity of the α -MnO₂ nanowires in Li-ion cells increased with increasing acid-leaching time up until 15 h of acid treatment. Samples immersed in acid for 27 h showed poorer electrochemical performance compared with those acid-leached for 15 h, likely because of amorphous material formed on the surface of the nanowires that is inactive toward lithium intercalation. In conclusion, these results show that by carefully controlling post-synthesis acid-leaching parameters it is

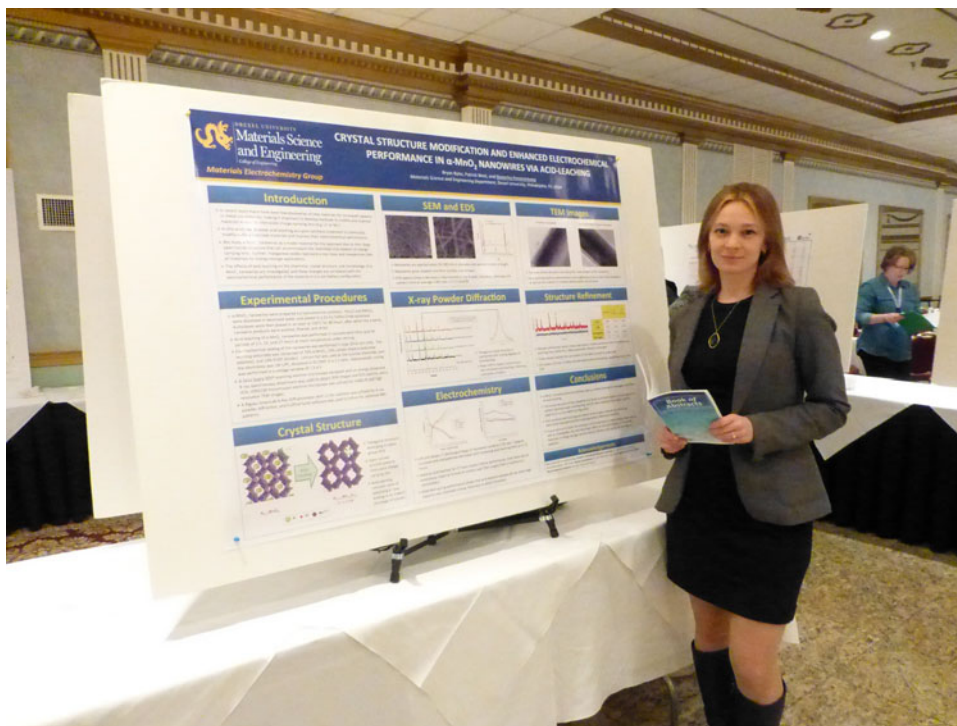


Figure 8. (Color online) E. Pomerantseva.

possible to improve electrochemical performance of metal-ion battery electrode materials (Figure 8).

X-RAY DIFFRACTION LABORATORY AT THE CENTRO DE FÍSICA APLICADA Y TECNOLOGÍA AVANZADA, UNIVERSIDAD NACIONAL AUTÓNOMA DE MÉXICO. (ISO 9001:2008 CERTIFIED)

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The aim of the X-ray Diffraction Laboratory at the *Centro de Física Aplicada y Tecnología Avanzada* (Center of Applied Physics and Advanced Technology) in the *Universidad Nacional Autónoma de México (UNAM)* is focused on the study of the structure of many materials, such as metals, minerals, polymers, catalysts, pharmaceutical products, liquid crystals, organic materials, thin films, ceramics, semiconductors, etc., through several techniques related to X-ray diffraction. With a unique configuration, the equipment has an ultra-fast solid-state detector, which allows us to implement, among others, the following techniques: phase identification and phase quantitative analysis, structural refinement, microstructural analysis (crystallite size and micro strains), residual stress, thin-film analysis (reflectometry), pole figures, small-angle X-ray scattering (SAXS), textural analysis, non-environmental condition analysis (high-temperature chamber that can reach 1500 °C), etc. These techniques are useful for research in basic science and have great applications in different industries such as pharmaceutical, mining, metal-mechanical, automotive, food, construction, electronics, ceramics, nanotechnology, catalysis, among others. In this poster, examples of the

different configurations and applications that are being exploited in the laboratory are shown.

STRUCTURE REFINEMENT AND POWDER X-RAY DIFFRACTION DATA FOR METASTUDTITE (UO_2) $\text{O}_2(\text{H}_2\text{O})^2$

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There has been some confusion in the published literature concerning the structure of Metastudtite (UO_2) $\text{O}_2(\text{H}_2\text{O})^2$ where differing unit cells and space groups have been cited for this compound. Because of the absence of a refined structure for Metastudtite, Weck *et al.* (*Dalton Trans.*, 2012, **41**, 9748) have documented a first-principles study of Metastudtite using density functional theory (DFT). Their model presents the structure of Metastudtite as an orthorhombic (space group $Pnma$) structure with lattice parameters of $a = 8.45$, $b = 8.72$, $c = 6.75$ Å. A PDF database entry has been allocated for this hypothetical Metastudtite phase based on the DFT modeling (see 01-081-9133) and aforementioned *Dalton Trans.* manuscript. Recently, we have obtained phase pure powder X-ray diffraction data for Metastudtite and have confirmed the model of Weck *et al.* via Rietveld refinement. We present the structural refinement of this powder diffraction dataset along with refined structural parameters derived from the fitting of the raw data. We compare the refined structure with that of the first-principles model derived from DFT. Tabulated powder diffraction data (d 's and I 's) for the Metastudtite have been derived from the refined model. These data are also presented.

Sandia National Laboratories is a multiprogram laboratory managed and operated by Sandia Corporation, a

wholly owned subsidiary of Lockheed Martin Corporation, for the United States Department of Energy's National Nuclear Security Administration under contract DE-AC04-94AL85000.

“THE FINAL COUNTDOWN”: POPULATING HISTORIC ENTRIES IN PDF-4/ORGANICS

M. Rost, M. Hammock, S. Gates, and E. Foster
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As the historic project comes to an end, over 10,000 articles from major and minor publishers were reviewed for quality crystal data. A large percentage of entries initially passed the quality assurance review. However, as each pattern goes through our editorial process, strict standards are maintained to ensure only high-quality data populates our subfiles. A total of 6383 entries were updated with atomic coordinates in the 2015 PDF-4 Organic release. Additionally, the quality of various previously published entries was improved through methods of complete re-abstraction into the ICDD calculated crystal database set (05).

When abstracting atomic coordinates for the historic project we frequently encountered errors stemming from a number of different sources, which often resulted in an entry being placed “on hold.” The most common errors we experience included missing coordinates, atomic coordinates that are inconsistent with the chemical formula, atom disorder or with partial occupancy inconsistencies. The editorial team reviews these issues and attempts to resolve as many as possible to ensure that only quality data enters into the product. With the PDF-4 Organics 2016 release, this project will be completed.

A NEW, HIGHLY FLEXIBLE, EASILY MODIFIABLE AIDS PROGRAM

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International Centre for Diffraction Data, Newtown Square, Pennsylvania

The program NBS AIDS is the cornerstone of the editorial work performed at the International Centre of Diffraction Data (ICDD). The program is used to ensure consistency and quality in each of the patterns published in the Powder Diffraction File (PDF). Among the functions of NBS AIDS are to check indexed reflections, improve cell parameters, check formulas, names, and densities. The key to running the AIDS program is the creation of an AIDS input file. This file has a strictly defined structure based on the concept of “PDF cards” which places strict limits on the number of reflections, the magnitude of intensities, the length of names, etc. For many years, these limits were more than adequate. However, with the ever increasing sensitivity of detectors, more complex materials systems, and more supporting data, it is clear that the needs of the scientific community have been changing. As a result these limits have begun to serve more as restrictions. Therefore, work has begun to remove the card dependence of the AIDS program. The poster to be presented will outline the need for a new AIDS program, what has been done to achieve

independence from PDF cards and the inherent benefits in doing so.

WHY DO WE NEED TO OPEN INTERNET PORTS? WebPDF-4+ 2014

J. Sunzeri
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- WebPDF-4+ provides the needed portability for accessing the PDF-4+ database via the Internet. It enables full functionality of the PDF-4+ database using a high-speed Internet connection. For more information about WebPDF-4+ visit: http://www.icdd.com/products/flyers/WebPDF4_Flyer.pdf
- Internet ports allow Internet applications to communicate with one another over the Internet. The most common example, your web browser uses port 80 to communicate based on <http://> in the URL or port 443 based on <https://>.
- Firewalls are the best ways for one to open or close Internet ports. There are two types of Firewalls: software and hardware. Firewalls are used to monitor, route (allow), or restrict communication between Internet applications. They can be found in operating systems like Windows®, in routers, and as stand-alone appliances. Home users have little, if any restrictions. Business and government institutions usually have more restrictions and will likely require changes to the firewall. If there are no restrictions, these ports are not blocked by your firewall.
- You may need to explain the WebPDF-4 ports to your I.T. Manager so these ports can be opened.

ICDD POWDER DIFFRACTION FILE® COVERAGE OF POLYMERS USED IN PHARMACEUTICAL AND BIOMEDICAL APPLICATIONS

T. Blanton, and S. Gates
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When one thinks of components used in medications and supplements administered as pills and capsules, the active pharmaceutical ingredient (API) is what initially comes to mind. For example, if you mention a pain reliever such as Tylenol®, the API is acetaminophen. However the API is often a small fraction of the actual pill or capsule. The bulk of most oral medications are referred to as excipients, including polymers. These polymers are not conventional commodity materials such as polyolefins, nylons, polyesters, etc., but rather they are biocompatible polymers such as cellulose, hydrocolloids, starches, etc. Polymers also find application in biomedical applications such as transdermal patches, medical delivery equipment, adhesives, petri dishes, contact lenses, etc.

Whether it is pharmaceutical or biomedical materials, phase analysis of their components requires high-quality reference diffraction patterns. The Powder Diffraction File®

(PDF[®]) databases have targeted subfiles for pharmaceuticals, bioactives, excipients, and polymers that are used for qualitative and quantitative phase identification. We will provide a review of polymer entries in the database, and plans for adding new polymer entries to enhance characterization of pharmaceuticals and biomedical as well as enhance the overall polymer subfile.

SELECTIVE AREA ELECTRON DIFFRACTION AND SIMULATED ANNEALING OF POWDER X-RAY DIFFRACTION FOR THE DETERMINATION OF NEW IRIDATE CRYSTAL STRUCTURES

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Determining both the unit cell and atomic positions for new materials is a challenging and demanding process. Here we report using a combination of electron and powder X-ray diffraction for rigorous experimental approaches in resolving new crystal structures containing the rare element iridium. Selective area electron diffraction (SAED) is used to give direct evidence of unit cell parameters, while simulated-annealing of powder X-ray diffraction data provides an exhaustive and accurate search of atomic positions. Together with chemical intuition these methods provide a rigorous approach for determining the structure of new materials for compounds that are difficult to synthesize as single crystals, a particular challenge in the Ir–Sn–Se system. Other systems may be investigated as well (Figure 9).

COMPUTATIONAL PHARMACEUTICAL MATERIALS SCIENCE: BEYOND STATIC STRUCTURES

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The five Crystal-Structure Prediction (CSP) Blind Tests have shown that molecular-mechanics force fields are not accurate enough for crystal structure prediction (Day *et al.*, 2009). The first – and only – method to successfully predict all four target crystal structures of one of the CSP Blind Tests was dispersion-corrected Density Functional Theory (DFT-D), and this is what we use for our work.

However, quantum-mechanical methods (such as DFT-D), are too slow to allow simulations that include the effects of time and temperature, certainly for the size of molecules that are common in pharmaceutical industry. Including the effects of time and temperature therefore still requires molecular dynamics (MD) with less accurate force fields.

In order to combine the accuracy of the successful DFT-D method with the speed of a force field to enable MD, our group uses Tailor-Made Force Fields (TMFFs) as described by Neumann (2008). In Neumann's TMFF approach, the force field for each chemical compound of interest is parameterized from scratch against reference data from DFT-D calculations; in other words, the TMFF is fitted to mimic the DFT-D energy potential.

Combining CSP with DFT-D followed by MD with the TMFF allows us to calculate, e.g. the temperature-dependent unit-cell expansion of each predicted polymorph, as well as possible temperature-dependent disorder. This is relevant for

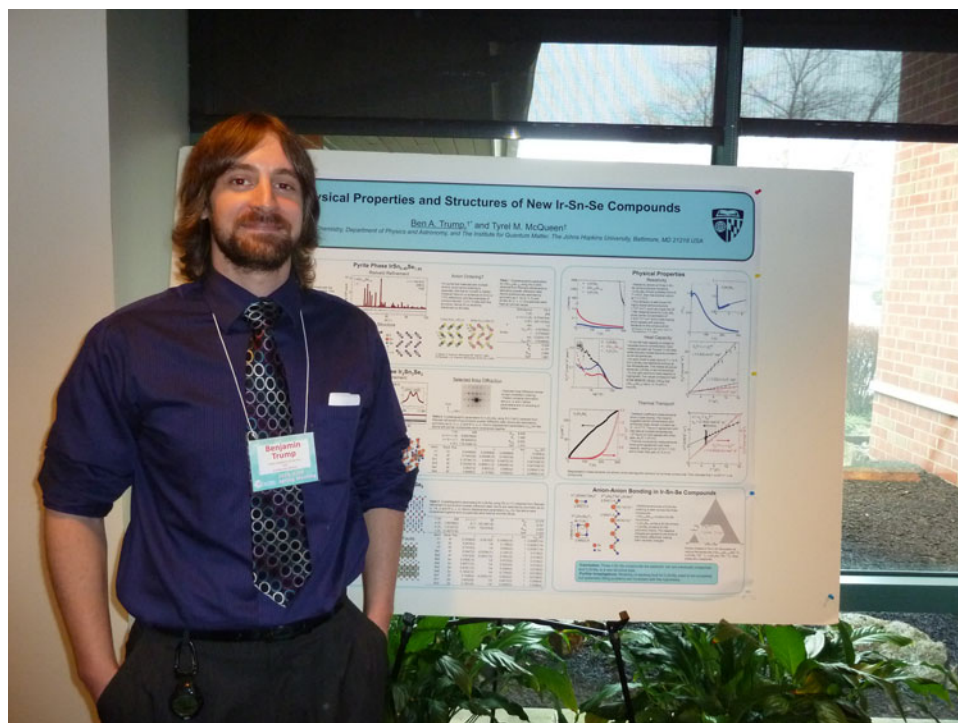


Figure 9. (Color online) B. Trump.

example when comparing the simulated X-ray powder diffraction patterns of the predicted crystal structures against experimental data.

Day, G. M. *et al.* (2009). *Acta Crystallogr. B* **65**, 107–125.
Neumann, M. A. (2008). *J. Phys. Chem. B* **112**, 9810–9829.

TIME-SLICING NON-AMBIENT POWGEN DATA – TEASING OUT DETAIL AFTER-THE-FACT

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Setting up non-ambient experiments at central facilities can be problematic because of various uncertainties. For example, temperature calibration errors and uncertain transition temperatures can create some risks when selecting temperature profiles. Studying reaction kinetics using a limited number of samples at a neutron facility such as SNS only adds to the quandary when deciding the length of each experiment and dataset in the experiment. Choose a temperature too low and the reaction will not finish, but too high and it may all be over before you know it.

The event-mode data collection on POWGEN gives the option of slicing up datasets after-the-fact into finer time slices or even other variables such as temperature. Re-reducing original event data to extract the information you wanted as opposed to making do with what the full dataset gave you has the potential to revolutionize how people carry out kinetic and other non-ambient experiments at POWGEN. To take advantage of the capability, a data acquisition mode has been developed to provide data with near-constant counting statistics across the temperature range even in case of accelerator trips or varying beam power. Examples of both ‘post-mortem’ re-reduction and deliberate application of the method will be shown.

ICDD GRANT-IN-AID PROGRAM

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International Centre for Diffraction Data, Newtown Square,
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One of the International Centre for Diffraction Data (ICDD)’s main objectives is to expand the range of material references by archiving or producing high-quality diffraction patterns. To further this effort, the ICDD supports a well-developed program of grants to researchers around the world to provide high-quality reference diffraction data. Each year the ICDD extends financial support to qualified investigators, in the form of Grants-in-Aid awarded on a competing proposal basis, for the preparation of reference X-ray powder diffraction data.

The duration of a Grant-in-Aid is 12 months with two grant cycles per year. Cycle I begins 1 April and Cycle II begins 1 October.

Benefits offered to grantees are:

- Financial support to aid current research
Publication of pattern(s) in the Powder Diffraction File. (PDF[®]) if accepted by the Editorial Department
- Receive calibration standards
- Purchase certain products at reduced prices
Web-based access to the list of compounds in the ICDD master database - includes published patterns as well as patterns still in the editorial process
- First-time grantees receive a complimentary one-year subscription to *Powder Diffraction*

To date, 28,600+ data entries have been added to the Powder Diffraction File[™] (PDF[®]) as a result of the ICDD Grant-in-Aid program. In addition, a new targeted grant for pharmaceuticals was initiated in 2013 resulting in 30 new data entries that were published in the PDF[®] 2014 Release. For Release 2015, we have received 29 submissions.

STRUCTURAL/PROPERTY STUDIES OF *p*-type UNFILLED TERNARY SKUTTERUDITE, FeSb₂ + *x*Te_{1-x}

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The structural/property studies of the *p*-type unfilled skutterudite solid solution, Fe(Sb₂ + *x*Te_{1-x}) has been carried out. The Fe(Sb₂ + *x*Te_{1-x}) compounds crystallize in the cubic space group *Im* $\bar{3}$. The unit cell can be viewed as built from eight smaller cubes with the Fe atoms residing in the cube vertices. Six of these cubes are filled with rectangular planar rings formed by the Sb/Te atoms. Te was found to randomly substitute in the Sb site. Since the metallic radius of Sb is greater than that of Te, a trend of increasing lattice parameters has been observed as the *x* value in Fe(Sb₂ + *x*Te_{1-x}) increases [cell parameters range from 9.10432(4) to 9.11120(3) Å for *x* = 0.0–0.2, respectively]. The Fe–(Sb,Te) bond distance also increases progressively [from 2.5358(4) to 2.5388(4) Å] as the Te content decreases, while the average (Sb,Te)–(Sb,Te) bond distances of the four-membered rectangular rings essentially remain constant (2.877 and 3.070 Å). The substitution of Te by Sb generates more holes without noticeably affecting the band structure, while markedly improving the electrical conductivity and retaining a high Seebeck coefficient, therefore dramatically increased power factors. The heat-carrying phonons are strongly scattered with the increasing *x* value because of the formation of solid solutions between two end members: □FeSb₂Te and □FeSb₃ (where □ represents a vacancy). Consequently, high thermoelectric figures of merit (ZT) were achieved in the FeSb₂ + *x*Te_{1-x} compounds, with the largest ZT value reaching ~0.65 for the sample with *x* = 0.2. Reference X-ray diffraction patterns

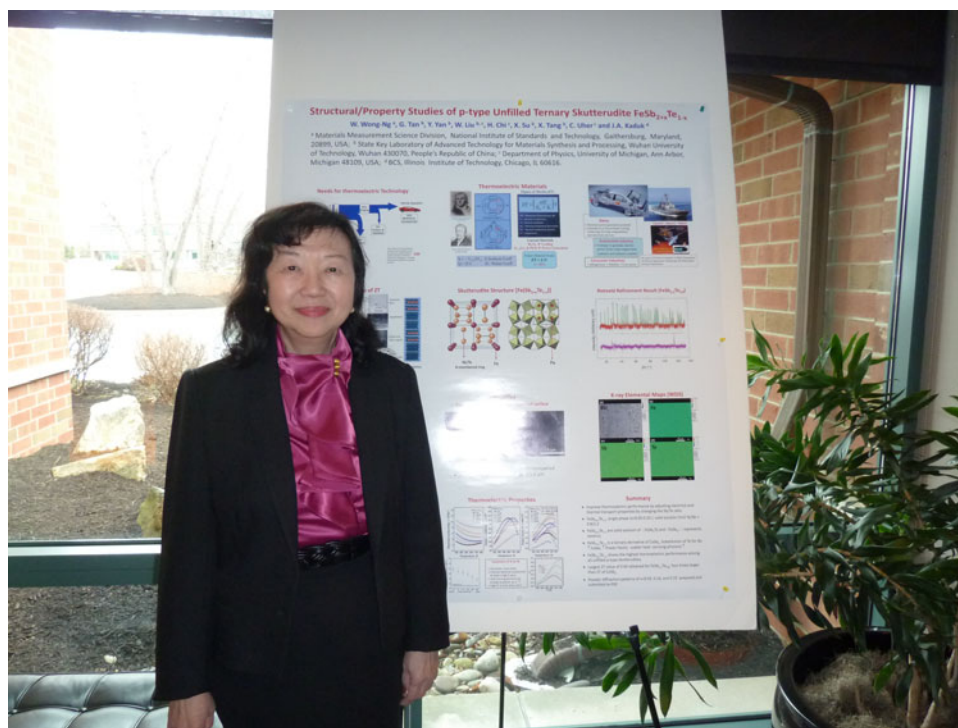


Figure 10. (Color online) W. Wong-Ng.

of the phases with $x = 0.05, 0.10,$ and 0.20 have been submitted to be included in the Powder Diffraction File (Figure 10).

INCORPORATION OF PHARMACEUTICAL API'S INTO THE PDF[®] DATABASES

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The International Centre for Diffraction Data[®] (ICDD[®]) continues to extend the coverage of all the Powder Diffraction File[™] (PDF[®]) databases demonstrated by more than 80,000 new entries being added to the PDF-4+ database since 2008. The comprehensive data and subfile systems greatly enhance the database analysis capabilities. As the X-ray diffraction method is more actively used in the pharmaceutical area, an even more enriched database is required for the analysis of pharmaceutical materials. The “Pharmaceutical Project” grew out of the need to acquire more pharmaceutical diffraction patterns and crystal structures in the

PDF databases for phase identification and quantitative analysis, particularly for analysis at room temperature. After reviewing the lists of the top 200 pharmaceutical drugs by US sales in 2011 and 2012, ICDD found 97 pharmaceutical API materials had not been analyzed by X-ray diffraction based on a search of published literature. A collaboration was proposed between ICDD-Argonne National Laboratories-Illinois Institute of Technology to collect synchrotron diffraction data for these materials. Once collected, the diffraction data were analyzed by Jim Kaduk for attempts at structure elucidation, and all pharmaceutical diffraction patterns were analyzed at ICDD using a strict three tier editorial review before their incorporation into the PDF products. To date 30 entries have been published in the PDF database with 29 additional entries to be published in Release 2015. This research program has also enhanced the understanding of pharmaceutical materials. Seven papers have been published in *Powder Diffraction*, with additional manuscripts submitted. Highlights of the technical results of this project will be discussed including an expansion of the targeted materials to include the top 500 pharmaceuticals by sales.