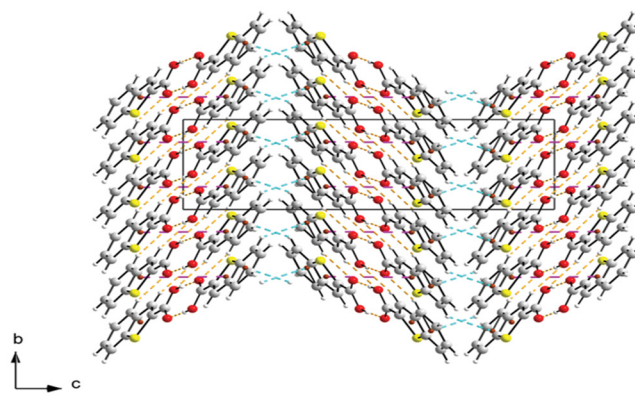
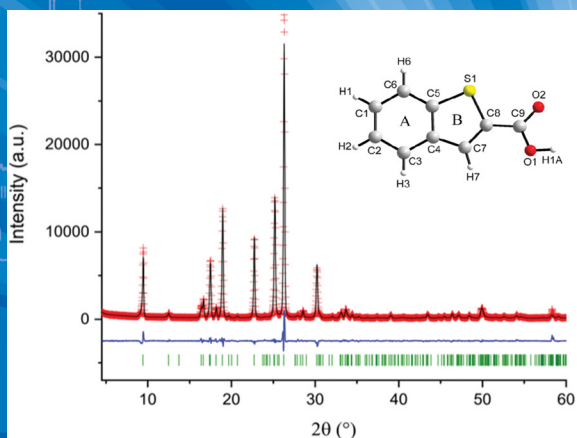
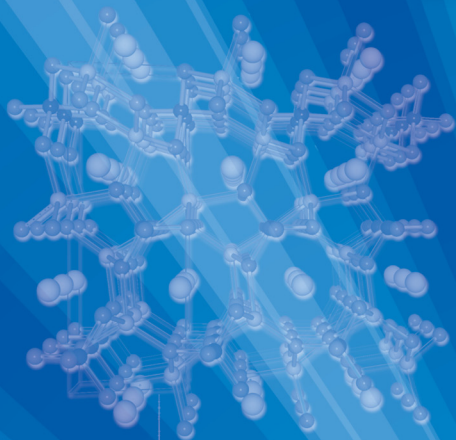


Powder Diffraction PDJ

Journal of Materials Characterization



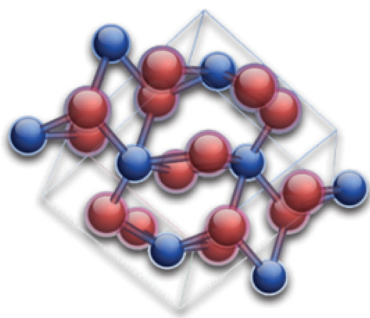
WORKING TOGETHER FOR YOU

With over 1,000,000 patterns available at your fingertips, ICDD's PDF databases and MDI's JADE software work together to make your life easier. Let the software work for the science and take your results further.



ABOUT US

For over 30 years, The ICDD and MDI have worked together in a complementary manner since MDI began in 1987. The XRD community has trusted MDI to provide unbiased results and help interpret both the everyday and the difficult XRD data. We are proud of our products and the daily effort we put forth towards advancing the science of XRD. Materials Data creates XRD software applications to collect, analyze, and simulate XRD data. These products are here to help solve issues in an enormous array of materials science projects, and may be found in labs around the world with data collected on virtually every brand of XRD equipment.



OUR PRODUCTS



PDF-4+

Phase Identification and Quantitation

The world's largest sources of inorganic diffraction data from crystals and powders in a single database featuring 426,000+ entries, including 323,900+ entries with atomic coordinates



PDF-4+/Web

Data on the Go

Provides portability to the PDF-4+ database via the internet.



PDF-4/Axiom

Quality Plus Value

Phase identification and quantitation that requires diffraction equipment manufacturer or vendor software.



PDF-2

Phase Identification + Value

Quality and subfile filters combined with 68 different searches and 53 display fields enable you to target your results for more accurate identification.



PDF-4/Organics

Solve Difficult Problems, Get Better Results

Designed to solve difficult problems that are analyzed by powder diffraction analysis for a multitude of applications in the pharmaceutical, regulatory, specialty chemical, biomaterial, and forensic fields.



PDF-4/Minerals

Comprehensive Mineral Collection

Ninety-seven percent of all known mineral types, as defined by the International Mineralogical Association (IMA), are represented in the database, as well as many unclassified minerals.



JADE Pro

JADE Works The Way You Do

Automate your analysis with JADE. In so many cases, it's just one click and you're done. Often these results are better than those an experienced analyst could report and in a more timely manner.

For more information contact ICDD at marketing@icdd.com

EDITORIAL

| | | |
|----------------|-----------|---|
| Camden Hubbard | Editorial | 1 |
|----------------|-----------|---|

TECHNICAL ARTICLES

| | | |
|---|---|----|
| Anelio J. Dugarte-Dugarte, Jacco van de Streek, Graciela Díaz de Delgado, Alicja Rafalska-Lasocha and José Miguel Delgado | Crystal structure from laboratory X-ray powder diffraction data, DFT-D calculations, Hirshfeld surface analysis, and energy frameworks of a new polymorph of 1-benzothiophene-2-carboxylic acid | 2 |
| P. Mikula, M. Vrána, J. Šaroun and V. Ryukhtin | Observation of multiple Bragg reflections accompanying forbidden Si(002) reflection in bent-perfect Si crystal | 14 |

NEW DIFFRACTION DATA

| | | |
|---|---|----|
| José H. Quintana Mendoza, Andrea P. Aparicio and J. A. Henao | Powder diffraction data and preliminary spectroscopic and thermal characterization of pinaverium bromide, a drug used for functional gastrointestinal disorders | 20 |
| Ji Yang, Zhi Hua Liu, Rui Zhi Zhu, Neng Jun Xiang, Shi Yun Tang, Pei He, Zi Li Suo and Hui Li | X-ray powder diffraction data for nicotine 3,5-dihydroxybenzoate dihydrate, $C_{10}H_{15}N_2 \cdot C_7H_5O_4 \cdot 2H_2O$ | 25 |
| Mariana M. V. M. Souza, Alex Maza and Pablo V. Tuza | X-ray powder diffraction data of $LaNi_{0.5}Ti_{0.45}Co_{0.05}O_3$, $LaNi_{0.45}Co_{0.05}Ti_{0.5}O_3$, and $LaNi_{0.5}Ti_{0.5}O_3$ perovskites | 29 |
| Shivang Bhaskar, Joseph T. Golab, James A. Kaduk, Amy M. Gindhart and Thomas N. Blanton | Crystal structure of pimecrolimus Form B, $C_{43}H_{68}ClNO_{11}$ | 35 |
| Jerry Hong, Joseph T. Golab, James A. Kaduk, Amy M. Gindhart and Thomas N. Blanton | Crystal structure of (<i>E</i>)-doxepin hydrochloride, $C_{19}H_{22}NOCl$ | 43 |
| James A. Kaduk, Amy M. Gindhart and Thomas N. Blanton | Crystal structure of loteprednol etabonate Form II, $C_{24}H_{31}ClO_7$ | 50 |
| James A. Kaduk, Amy M. Gindhart and Thomas N. Blanton | Crystal structure of tezacaftor Form A, $C_{26}H_{27}F_3N_2O_6$ | 56 |

DATA REPORTS

| | | |
|---|---|----|
| James A. Kaduk, Amy M. Gindhart and Thomas N. Blanton | Powder X-ray diffraction of azelastine hydrochloride, $C_{22}H_{25}ClN_3O \cdot Cl$ | 63 |
|---|---|----|

| | | |
|--|--|----|
| Jingwen Fan, Zhicheng Zha, Qing Wang and Shoujun Zheng | X-ray powder diffraction data for norethisterone enanthate, $C_{27}H_{38}O_3$ | 65 |
| James A. Kaduk, Amy M. Gindhart and Thomas N. Blanton | Powder X-ray diffraction of escitalopram oxalate oxalic acid hydrate, $(C_{20}H_{21}FN_2O)_2(C_2O_4)(H_2C_2O_4)(H_2O)_{0.16}$ | 68 |

CALENDARS OF MEETINGS, SHORT COURSES AND WORKSHOPS

| | | |
|-----------|---|----|
| Gang Wang | Calendar of Forthcoming Meetings | 70 |
| Gang Wang | Calendar of Short Courses and Workshops | 71 |

ERRATUM

| | | |
|--------------------------|--|----|
| “On the Cover” — Erratum | | 72 |
|--------------------------|--|----|

Powder Diffraction

An International Journal of Materials Characterization

Editor-in-Chief

Camden Hubbard
Applied Diffraction Services, U.S.A.
camden.hubbard@me.com

Managing Editor

Nicole M. Ernst Boris
International Centre for Diffraction Data, U.S.A.
boris@icdd.com

Production Editor

Kayla Riddleberger
Cambridge University Press, U.S.A.
kriddleberger@cambridge.org

Editors for New Diffraction Data

Soorya Kabekkodu
International Centre for Diffraction Data, U.S.A.
kabekkodu@icdd.com

Stacy Gates-Rector

International Centre for Diffraction Data, U.S.A.
gates-rector@icdd.com

Associate Editor for New Diffraction Data

Frank J. Rotella
Argonne National Laboratory (Retired), U.S.A.
fjrotella1949@gmail.com

Editors

Xiaolong Chen
Institute of Physics, Chinese Academy of Sciences, China
xlchen@iphy.ac.cn

José Miguel Delgado
Universidad de Los Andes, Venezuela
miguel@ula.ve

Norberto Masciocchi
Università dell'Insubria, Italy
norberto.masciocchi@uninsubria.it

Editors for Crystallography Education

James Kaduk
Poly Crystallography Inc., U.S.A.
Kaduk@polycrystallography.com

Brian H. Toby
Argonne National Laboratory, U.S.A.
brian.toby@anl.gov

International Reports Editor

Winnie Wong-Ng
National Institute of Standards and Technology, U.S.A.
winnie.wong-ng@nist.gov

Calendar of Meetings and Workshops Editor

Gang Wang
Institute of Physics, Chinese Academy of Sciences, China
gangwang@iphy.ac.cn

The Advisory Board is served by the International Centre for Diffraction Data's Regional Co-Chairs and representatives from Cambridge University Press.

Advisory Board

| | |
|---------------------|---|
| Mark Rodriguez | Sandia National Labs, U.S.A |
| Takashi Ida | Nagoya Institute of Technology, Japan |
| Matteo Leoni | University of Trento, Italy |
| T.N. Guru Row | Indian Institute of Science, India |
| Vanessa Peterson | Australian Nuclear Science and Technology Organisation, Australia |
| Evgeny Antipov | Moscow State University, Russia |
| Xiaolong Chen | Chinese Academy of Sciences, People's Republic of China |
| Jose Miguel Delgado | University de Los Andes, Venezuela |
| Steve Hillier | The James Hutton Institute, Scotland, UK |
| Sara Yanny-Tillar | Cambridge University Press, U.S.A. |

On the Cover: The cover figures come from the manuscript "Crystal structure from laboratory X-ray powder diffraction data, DFT+D calculations, Hirshfeld surface analysis, and energy frameworks of 1-Benzothiophene-2-carboxylic acid" (BTCA) published in this issue of Powder Diffraction by Analio J. Dugarte-Dugarte, Jacco van de Streek, Graciela Diaz de Delgado, Alicja Rafalska Lasocha, and José Miguel Delgado. This study used state-of-the-art PXRD data collection, performed both Rietveld structure solution and then applied DFT+D calculations to optimize the structure. The structure is a complex 3D arrangement involving hydrogen bonded dimers of the BTCA molecules.

The cover shows the PXRD data, Rietveld fit, atomic arrangement and molecular packing within the crystal viewed down the a-axis. The combined use of DFT optimization methods following Rietveld structure refinement is increasing in use, particularly for obtaining the best model of many solid state crystalline organic materials. These combined Rietveld and DFT optimization tools are becoming more widely used for analysis of compounds with important pharmacological activity.

Powder Diffraction is a journal of practical technique, publishing articles relating to the widest range of application—from materials analysis to epitaxial growth of thin films and to the latest advances in software. Although practice will be emphasized, theory will not be neglected, especially as its discussion will relate to better understanding of technique.

Submit manuscripts online at <http://mc.manuscriptcentral.com/pdj>. See the instructions on submitting your manuscript linked on that page. The editors will consider all manuscripts received, but assume no responsibility regarding them. There is no publication charge.

Most proofs are handled via email at kriddleberger@cambridge.org. Please include the job number in all correspondence.

For advertising rates and schedules contact M.J. Mrvica Associates, 2 West Taunton Avenue, Berlin, NJ 08009; Phone: 856-768-9360; Fax: 856-753-0064; Email: mjmrvica@mrvica.com

Subscription Prices 2021

| | Print & Online | Online |
|------------------------------------|----------------|--------|
| Individual (U.S. & Canada) | \$269 | \$190 |
| Individual (outside U.S. & Canada) | £208 | £146 |
| Student | N/A | \$42 |
| Institutional or Library | \$536 | \$324 |

Subscription rates to Eastern Hemisphere include air freight service.

Back-Number Prices. 2020 single copies: \$158.

Subscription, renewals, and address changes should be addressed to Subscription Fulfillment, *Powder Diffraction*, Cambridge University Press, One Liberty Plaza, 20th floor New York, NY 10006-1435 (for U.S.A., Canada, and Mexico); or Cambridge University Press, The Edinburgh Building, Shaftsbury Road, Cambridge, CB2 8RU, Cambridge, England (for UK and elsewhere). Allow at least six weeks advance notice. For address changes please send both old and new addresses and, if possible, include a mailing label from the wrapper of a recent issue.

Claims, Single Copy Replacement, Back Volumes, and Reprints: Missing issue requests will be honored only if received within six months of publication date (nine months for Australia and Asia). Single copies of a journal may be ordered and back volumes are available in print or microform. Individual subscribers please contact Subscription Fulfillment, *Powder Diffraction*, One Liberty Plaza, 20th floor New York, NY 10006-1435. Phone: 845-353-7500; Toll free: 800-872-7423; Fax: 845-353-4141. Email: subscriptions_newyork@cambridge.org.

Powder Diffraction (ISSN: 0885-7156) is published quarterly (4X annually) by the JCPDS-International Centre for Diffraction Data through Cambridge University Press, One Liberty Plaza, 20th floor, New York, NY 10006. Periodicals postage rate paid at New York, NY, and at additional mailing offices. POSTMASTER: Send address changes in the USA, Canada, and Mexico to: Powder Diffraction, Cambridge University Press, Journals Fulfillment Department, One Liberty Plaza, 20th floor, New York, NY 10006. Send address changes elsewhere to Powder Diffraction, Cambridge University Press, Journals Fulfillment Department, UPH, Shaftsbury Road, Cambridge CB2 8BS, England.

Permission for Other Use: Permission is granted to quote from the journal with the customary acknowledgment of the source. To reprint a figure, table, or other excerpt requires the consent of one of the authors and notification to Cambridge University Press.

Requests for Permission: No part of this publication may be reproduced in any forms or by any means, electronic, photocopying, or otherwise, without permission in writing from Cambridge University Press. Policies, request forms, and contacts are available at: <http://www.cambridge.org/about-us/rights-permissions/>. Permission to copy (for users in the U.S.A.) is available from Copyright Clearance Center: <http://www.copyright.com>. Email: info@copyright.com.

Document Delivery and Online Availability: Abstracts of journal articles published by Cambridge University Press are available from Cambridge Core (<https://www.cambridge.org/pdj>).

Copyright © 2021 JCPDS- International Centre for Diffraction Data, 12 Campus Blvd., Newtown Square, PA 19073-3273, U.S.A. All rights reserved. www.icdd.com/products/journals.htm

PDF-4/Axiom 2021 Quality + Value

Cost effective database designed for
phase identification and quantitation.



Coming this Fall!

**Get your PDF-4/Axiom 2021 by
downloading from the cloud***

Visit www.icdd.com/pdf-4-axiom for
more information

*Also available on USB & DVD

ABOUT US

DIFFRACTION DATABASES YOU CAN TRUST

The Powder Diffraction File™ (PDF®) is the only crystallographic database that is specifically designed for material identification and characterization. It is an analysis system that is comprised of crystallographic and diffraction data. These data with embedded data mining and analysis software have been through a quality and classification editorial review system.

ISO CERTIFIED

The only crystallographic database organization in the world with its Quality Management System ISO 9001:2015 certified by DEKRA.



KEY POINTS

-  Featuring 97,500+ Entries, including 69,500+ Entries with Atomic Coordinates
-  Combines Powder & Single Crystal Data
-  Cost Effective 3 Year License*
*Contact us for our 6-year and 9-year license packages
-  Two Additional Seats - Low Cost
-  ICDD's data mining software is included!*
*Requires Vendor Software for phase identification and quantitation

ABOUT PDF-4/Axiom

PDF-4/Axiom 2021 is designed for phase identification and quantitation. This cost effective database includes 97,500+ selected data entries and 69,500+ entries with atomic coordinates selected for benchtop users. PDF-4/Axiom features 70,000+ entries with I/I_c values for quantitative phase analysis by Reference Intensity Ratio. PDF-4/Axiom is useful for routine phase identification and quantitation (both by Rietveld and RIR method). PDF-4/Axiom data is focused on ambient entries.

ICDD's search-identification software, Sleve+, is not included (available with PDF-4+). PDF-4/Axiom requires diffraction equipment manufacturer or vendor software for phase identification and quantitation. If you are using OEM/developer software that depends on PDF-4/Axiom, check that the OEM/developer applications function properly with the new database, and then uninstall the previous version of the PDF-4/Axiom.

PDF-4/Axiom offers an option to purchase two additional seats licensed for three years.

For more information contact ICDD at marketing@icdd.com