# COMMISSION ON POWDER DIFFRACTION

## INTERNATIONAL UNION OF CRYSTALLOGRAPHY

## NEWSLETTER No. 4

**January 1990**

### POWDER DIFFRACTION AT ISIS

ISIS is currently the most powerful pulsed spallation neutron source in the world, operating with an average current of 100μA. Neutrons are produced every 20ms by a high intensity 750MeV proton beam in sharp bursts that are under-moderated to produce a polychromatic pulse. The neutron pulse width and time of flight are both proportional to wavelength and thus the resulting ratio, the resolution, is essentially independent of wavelength and improves linearly with the length of flight path. Different neutron wavelengths may be discriminated by measuring the time of flight from moderator to sample to detector, and thus a complete diffraction pattern may be collected at a single Bragg angle. Additionally, the high epithermal flux below wavelengths of 1Å permits d-spacings as low as 0.2Å to be observed.

The High Resolution Powder Diffractometer (HRPD) at ISIS is situated at the end of a 100m neutron guide. The highest resolution ($\Delta d/d \approx 4 \times 10^{-4}$) is obtained in backscattering ($170° < 2\theta < 178°$) where geometrical contributions are negligible. This high resolution is essentially independent of d-spacing and thus HRPD is well-suited for the study of phase transitions and subtle symmetry changes; peak splittings are not confiied to one small 'high resolution' section of the pattern but occur with roughly equal magnitude across the diffraction pattern. High resolution has proved invaluable for the profile refinement using data from crystals of complex molecules with large unit cells and ab-initio structure determination. In the latter case an advantage of time-of-flight powder diffraction experiments is the ease of lattice parameter determination from first principles when the highest d-spacing information is available. Indeed individual d-spacing

The spallation Neutron Source, ISIS, at Rutherford Appleton Laboratory. A long neutron channel, seen somewhat below and to the right of the centre of the photo, connects the diffractometer, in the smaller building, to the neutron source.

up to 6Å may be measured with an accuracy of ~1 in $10^5$. The narrow instrumental peak shape, although complicated, is well known, thus permitting the observation and study of line broadening effects. Recent successes in this field include the modelling of chemical concentration gradients in samples, and the study of effects due to anisotropic strain broadening and antiphase domains.

Medium resolution high intensity neutron diffraction is also available at ISIS on the POLARIS diffractometer. POLARIS is complementary to HRPD providing a resolution of $\Delta d/d \approx 5 \times 10^{-3}$ but with increased flux because of its 10 m primary flightpath. Recent experiments on POLARIS include kinetic studies involving rapid experimental collection time of ~5 minutes and the study of powders at high pressures up to 50kbar. With suitable collimation the fixed 90° detector geometry permitted by the time-of-flight method, allows full diffraction patterns to be collected at elevated pressures without corruption of data by diffraction peaks from the pressure cell itself.

A suite of data analysis programs, developed and supported at ISIS are available for use by the user community.

Informal enquiries should be made to the authors. Beam time is allocated twice yearly following a peer review selection procedure. The closing dates for proposals are April 16 and October 16 each year. Application details can be obtained at the following address:

University Liaison Secretariat, R3
The ISIS Facility
Rutherford Appleton Laboratory
Chilton, Didcot
Oxfordshire OX11 0QX U.K.

Tel: 0235-44592 Fax: 0235-445720
Telex: 83159 RUTHLB G

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## COMMENTS ON SEARCH/MATCH PROCEDURES

In September 1989, the International Centre for Diffraction Data (ICDD) submitted a questionnaire to its members to survey the interests and concerns of industrial diffractionists. One recurrent concern is the efficient and reliable identification of multiphase powder patterns in regard to cost-effectiveness and turnaround time. Search procedures fall into two categories; namely, manual searching in Search Manuals and computer searching on large mainframe computers or on PC's. Although computer searching has been practiced since 1965. manual searching is still actively continued as evidenced by the undiminished demand for updated Hanawalt Search Manuals. However, with the annual addition of 2000 new powder-pattern to the Powder Diffraction File (PDF), the future search manuals will become excessively bulky and costly. Search routines will have to be devised and tested that are more efficient and compact. Substantial condensation of the 1987 Alphabetical Index could be achieved by eliminating multiple listings of fluorides (3277), hydroxides (2069). chlorides (1907) and other common ions or radicals inasmuch as the particular fluorides, hydroxides, etc. can be located more readily under the element name of the fluoride. hydroxide. etc. For example erbium fluoride would be located only under erbium.

Substantive improvements in computer search-routines would be much appreciated by powder diffractionists. General acceptance of computer search-match methods has been slow for several reasons: lack of uniqueness in the identification of an unknown mixture of phases (too many alternative matching candidates); lack of fast, reliable, automated digitization of precision powder diffraction data (obtained with Bragg-Brentano parafocusing diffractometers), and questionable cost benefits over manual search procedures. It is generally conceded that utilization of elemental data greatly reduces the number of pseudo-matches. Selective restrictions of the PDF data base would be conducive to speeding up the various search/match routines. There are about a dozen computer programs that have been tested and are beeing used. However, all their algorithms have some limitations. It would be a challenge to devise an efficient cost-effective program which would embody the combined merits of the separate algorithms. A critical review of the appended pertinent references should serv to stimulate renewed efforts in meeting the needs of the world-wide diffraction community engaged in powder difffactometry. It is also proposed by CPD to sponsor a workshop on phase identification to address the above problems.

## SELECTED REFERENCES IN CHRONOLOGICAL ORDER

(1) Hanawalt, J. D., Rinn, H.W., and Frevel, L.K. (1938) Chemical Analysis by X-ray Diffraction - Classification and Use of Diffraction Patterns. Ind. Eng. Chem. Anal. Ed **10,** 457-512.

(2) Frevel, L.K.(1944) Chemical Analysis by Powder Diffraction. Ind, Eng. Chem. Anal. Ed. **16,** 209-218.

(3) "Index (Inorganic to the Powder Diffraction File (1963))" ASTM Special Technical Publication 48-M2, Philadelphia, PA.

**(4)** **Smith** D.K. (1963) "A Fortran Program for Calculating Powder Pattems from Atom Coordinates" Am. Cryst. Assoc. Annual Meeting, March 28-30; paper E1 1.

*(5)* Frevel, L.K. (1965) Computational Aids for Identifying Crystalline Phases by Powder Diffraction, Analyt. Chemistry 37,471-482.

(6) Nichols, M. C. (1966) A fortran 11 Program for the Identification of X-ray Powder Diffraction Patterns, UCRL-70078, Lawrence Livermore Laboratory.

similarly disordered materials. Registrants for the satellite meeting are encouraged to attend the main congress and transport between Toulouse and Bordeaux will be provided.

Papers will be presented as oral contributions or posters and the invited speakers are:

***Accuracy in Data Collection***
D.E. Cox (USA)
W.I.F. David (UK)

***Recent Applications***
T.C. Huang (USA)
J. Pannetier (France)

***Line Profile** and Sample **Characteristics***
H. Toraya (Japan)
E.J. Mittemeijer (Netherlands)

***Identifcation** and **Quantitative Analysis***
D.K. Smith (USA)
R.J. Hill (Australia)

***Structure Analysis***
A. Le Bail (France)
M. Sakata (Japan)

Other oral contributions will be selected from submitted abstracts by the programme committee (chairman Dr. D. Louër). The deadline for abstracts is 15 February and that for registration is 1 May. Further information and copies of the second circular can be obtained from Dr. J. Galy (chairman of the organizing committee), CEMES-LOE CNRS, Powder Diffraction, 29 rue Jeanne Marvig, BP4347, 31055 Toulouse Cédex, France.

Chairman of the program committee, Daniel Louër.

Limited financial support is available from the IUCr for young scientists who wish to attend the Bordeaux Congress and/or its satellite meetings. Further information and copies of the second circular for the Bordeaux meeting can be obtained from Dr. M. Hospital, Laboratoire de Cristallographie, Université Bordeaux, 1, 351 Cours de la Libération, 33405 Talence Cédex, France.

## OTHER FORTHCOMING **MEETINGS** OF INTEREST TO POWDER DIFFRACTIONISTS

March 14-16, 1990 — "Neutron Scattering Data Analysis" RUTHERFORD APPLETON LABORATORY (Dr M.W. Johnson, Rutherford Appleton Laboratory, Chilton, Didcot, Oxon OX11 0QX)

April 22-29, 1990 — 17th Course on Electron Crystallography. ERICE, Italy (prof. L. Riva di Sanseverino, Dip. De Scienze Mineralogia, Piazza Porta San Donato 1, I-40126, Bologna, Italy)

July 12-17, 1990 — 15th General Meeting of the International Mineralogical Association. BEIJING, CHINA (prof. H.G. Yunhui. Organizing Committee of IMA 1990, c/o Institute of Mineral Deposits, Chinese Academy of Geological Fuchengmenwai. Beijing, People's Republic of China)

July 29-31, 1990 — Symposium on Complementary Applications of Diffraction by Neutrons and by X-Ray Synchrotron Radiation. Alpe d'Huez, near Grenoble. Organizing Chairman: M. Marezio, Programme Chairman: C. Vettier. Requests for the Second Circular: IUCr XVth Satellite Meeting Secretariat, Laboratoire de Cristallographie, CNRS, 166X, 38042 GRENOBLE CEDEX, France.

August 9-11, 1990 — Rietveld Summer School for Beginners RSSB 90. CIESZYN, POLAND (Summer School RSSB-90, Uniwerytet Slaski, Instytut Fizyki Chemii Metals, Dr. Eugeniusz Lagiewka, ul. Bankowa 12, 40-407 Katowice, Poland)

August 20-24, 1990 — Advanced Methods in X-Ray and Neutron Structure Analysis of Materials. (Ph.Dr. Ludmila Chráštanská, Dum techniky CSVTS, Plzenská 2/1, 370 21 CESKÉ BUDEJOVICE, Czechoslovakia, tel: 26250, 26251; telex: 144364 cvts c.)

August 12-16, 1991 — PICXAM. Pacific-International Congress on X-ray Analytical Methods. HONOLULU, HAWAII. Organized by: The Australian X-ray Analytical Association. The Denver X-ray Conference and X-ray Analysis Group/Japan Society for Analytical Chemistry. (Lynne Bonno, Conference Secretary, Department of Engineering, University of Denver, Denver, CO 80208. U.S.A.)

If any reader wishes to participate in the survey and has not yet registered their interest, please write to:

Dr. R.J. Hill, CSIRO Division of Mineral Products, PO Box 124. Port Melbourne, Victoria 3207. Australia

## IUCr XVIth GENERAL ASSEMBLY & CONGRESS, 1993

At Perth in August 1987 the General Assembly of the IUCr gave preliminary acceptance to the invitation from the Chinese National Committee for Crystallography (CNCC) and the China Association for Science and Technology (CAST) to hold the 1993 meeting in Beijing. However, subsequent to the event in China last June, the following resolution was approved by the Executive Committee on 19 July.

> The Executive Committee invites the Chinese National Committee to accept that the US Adhering Body be asked to organize the 1993 General Assembly and Congress, and that the General Assembly and Congress in Beijing be postponed to 1996.

The Resolution was discussed at length at an Extraordinary General Assembly in London on 19 December. At the Assembly, Professor You-Qi Tang, Vice-President of the Union and Chairman of the CNCC, transmitted an assurance from CAST that the ICSU requirements for the free circulation of ***bona-fide*** scientists would be upheld. This would apply equally to Chinese students who had left the country, about whose welfare concern had been expressed.

The delegates present at the Assembly voted on behalf of their respective national committees and the majority of votes cast were against the resolution. The original decision taken at Perth therefore stands and the provisional venue for the 1993 meeting is still Beijing, to be formally confirmed in Bordeaux, 1990.

J. Ian Langford
CPD Secretary

## XVth CONGRESS AND GENERAL ASSEMBLY OF THE INTERNATIONAL UNION OF CRYSTALLOGRAPHY

Bordeaux, France, July, 19-26, 1990

Participation is open to all persons interested in Crystallography. Of special interest to powder diffractionists:

(i) Microsymposium: Powder Diffraction Studies of Fibrous, Polymeric and Similarly Imperfectly Ordered Materials.

(ii) Open Commission Meeting: Advances in Structure Determination from Powder X-Ray Diffraction Data (Commission on Powder Diffraction).

| Program Committee Chairman | Organizing Committee Chairman |
|---|---|
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Deadline for abstracts - February 15, 1990
Deadline for registration: May 15, 1990

Registration fees (All fees quoted in French Francs)

| | By May 15 | Later on |
|---|---|---|
| Regular members | 1400 FF | 1800 FF |
| Young scientists | 700 FF | 900 FF |
| Accompanying persons | 700 FF | 900 FF |

Hotel reservations should be made prior to May 15.

## POWDER DIFFRACTION MEETING, TOULOUSE 16-19 JULY, 1990

The IUCr Commission on Powder Diffraction is arranging a satellite meeting on powder diffraction in Toulouse, to take place immediately before the XVth Congress and General Assembly at Bordeaux. The theme will be recent theory. instrumentation and contributions to materials science and the programme will be based on the following topics:

- Accuracy in data collection; high resolution diffraction, standard reference materials.
- Sample characteristics from powder data by means of pattern fitting and other methods.
- Practical aspects of structure determination from powder data; indexing, structure solution, refmement.
- Recent applications, particularly time- and temperature-resolved studies of materials.
- Advances in phase identification and quantitative analysis; crystallographic data bases.

The intention is to complement powder diffraction sessions at Bordaux, which will include microsymposia on structure determination from PD data and PD studies of fibrous, polymeric and

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(9) Young, R.A. Mackie. P.E., and Von Dreele, RB. (1977) Application of the Pattern-Fitting Structure-Refinement Method to X-ray Powder Diffractometer Pattems, J. Appl. Cryst. 10, 262-269.

(10) Marquardt, R.G., Katsnelson, I.. Milne G.W.A.. Heller, S.R. ,Johnson G. G., Jr.. and Jenkins, R. (1979) Search-Match System for X-ray Powder Data, J. Appl. Cryst. 12,629-634.

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(13) Huang.T.G., Parrish, W. and Post., B. (1983) Computer Search/Match of Standards Containing a Small Number of Reflections, Adv. in X-ray Analysis26, 98.

(14) Cherukuri, S.C. and Synder, R.L.(1983) Comparison of the Hanawalt and Johnson-Vand Computer Search/ Match Strategies, Adv. in X-ray Analysis 26, 99-104.

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(16) Lin,T.H., Zhang, SZ.,Chen, L.J., and Cai, X.X. (1983), An Improved Program for Searching and Matching X-ray Powder Diffraction Patterns, J. Appl. Cryst. 16, 150-154.

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(18) **Skrobian. M.** and Havlik. T. (1986), Searching Matching of X-ray Powder Diffraction Pattems Using a Programmable Calculator, Powder Diffraction 1,235-239.

(19) Marquart, R.G. (1986) µPDSM Mainframe Search/ Match on IBM PC, Powder Diffraction L34-39.

(20) Jenkins. R. and Holomany, M. (1987) "PC-PDF": A Search/Display System Utilizing the CD-ROM and the Complete Powder Diffraction File, Powder Diffraction 2 215-219.

L.K. Frevel

## PROGRAM INFORMATION EXCHANGE BANK

D.K. Smith has been appointed Consultant to the CPD. His primary role as a consultant will be that of the development and operation of a Program Information Exchange Bank, where 'program' means computer programs for powder diffraction analysis and collection. A fist write up of the Program Data Bank has appeared as a chapter in the book Modem Powder Diffraction, which is available from the Mineralogical Society of America (20 US$).

At the planning stage is a compendium of program descriptions that can be maintained at some central location where users can dial in for current information on any programs or category of programs. It is envisioned that a central computer facility could put the list where it could be reached via dial-in and be queried for the updated information desired.

The Exhange Bank project will be further discussed at the Powder Diffraction Meeting in Toulouse, 1990 (see separate announcement in this Newsletter).

Anyone who has developed or modified a program suitable for the program bank is urgently requested to communicate th to Prof. D.K. Smith (address, see last page of this Newsletter).

## RIETVELD REFINEMENT ROUND ROBIN: **UPDATE**

Round Robin packages have been circulated to participants in 28 X-ray and neutron powder diffraction laboratories around the world. These packages contain:

(i) a detailed set of instructions.
(ii) forms for documenting the refinement and data collection procedures and associated Rietveld refinement results,
two powder samples (one simple, and one complex) for in-house data collection and crystal structure analysis, and
(iv) a magnetic tape containing two "standard" X-ray and neutron powder diffraction data sets for in-house analysis (also to be used for the return of data sets collected on the above samples).

The crystal structure/profile refinement results and diffraction data returned by participants over the next few months will be surveyed by the CPD and a preliminary report will be presented at the Powder Diffraction Satellite Meeting of the XVth Congress of the IUCr in Toulouse, France, July 16-19,1990.

***continued on next page***

## CALL FOR CONTRIBUTION TO THE COMMISSION AND ITS NEWSLETTER

Members of the powder diffraction community are invited to contact any member of the Commission on Powder Diffraction with matters for possible consideration by the Commission and/or inclusion in subsequent (biannual) Newsletters. A matter for which input form the diffraction community is certainly needed relates to the selection (if deemed desirable) of a logo for the Commission.

P.-E. Werner
Editor, Newsletter No 4

## MAILING LIST FOR FUTURE NEWSLETTERS

If you wish to receive a copy of future CPD Newsletters, and are not already on our mailing list, please complete the following coupon, or its copy, and return to Dr. Langford at the address below:

To the IUCr Commission on Powder Diffraction:

Please keep me on the mailing list for future issues of the CPD Newsletter.

Please add the following interested person to you mailing list.

Name: _____

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Return this form to: Dr J.L. Langford, CPD Secretary, School of Physics & Space Research, University of Birmingham, Birmingham B15 2TT, England.

**Addresses of the CPD members:**

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