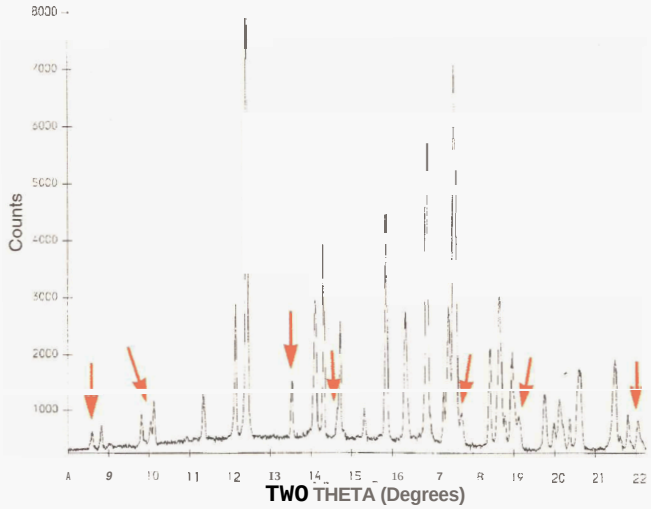
COMMISSION ON POWDER DIFFRACTION

INTERNATIONAL UNION OF CRYSTALLOGRAPHY

NEWSLETTER No. 13, OCTOBER 1994

DIFFRACTION FROM SINGLE POWDER GRAINS

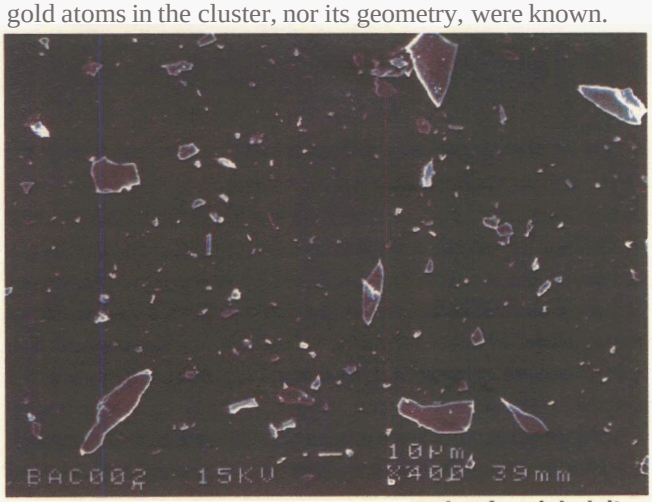
When single crystals of good quality and size can be obtained, single crystal X-ray diffraction will be the method of choice for the determination of atomic coordinates. Both structure solution and refinement are straightforward, and can normally give results of high accuracy with regard to molecular geometry, interatomic distances and temperature factors.



Powder diffraction pattern of aurichalcite

Sometimes only very small crystals of a compound of real interest are available, too small for conventional single crystal methods. In many cases, recrystallisation has been unsuccessful or is not applicable. Also, if the temperature or pressure of the crystal is varied, a phase transition may induce the crystal to shatter, leaving only very small fragments. Under these circumstances, the powder method has been employed to solve the crystal structures of materials with some notable successes. These include the structure of polymer electrolytes from conventional X-ray sources, the structure of fullerenes from neutron diffraction and the structure of drugs, zeolites and other minerals of industrial importance from synchrotron X-ray diffraction. Structure solution from powdered materials relies on data of excellent quality and high resolution: if impurities are present in the sample, as shown by the red arrows in the powder diagram above, the process of indexing and decomposition is considerably more complex. If the bulk structure of a material with multiple phases is required, as is often the case with naturally-occurring samples, some help in phase identification and starting structures is very helpful.

This help can be provided by removing what amounts to a single grain of a powder from the bulk sample and performing a single crystal structure analysis on this very small crystal. For these very small single crystals, monochromatised synchrotron radiation, often used with an area detector system, has allowed diffraction patterns to be recorded for very tiny crystals picked out of samples which look more like polycrystalline powders. Since these are single crystal diffraction patterns, determination of unit cell and indexing of all observable reflections is comparatively easy: all the reflection intensities can be measured, and the structure solved and refined. Structures established in this way include two minerals of interest in relation to their use as precursors in industrially important zinc/copper oxide catalysts, and organometallic compounds prepared in projects related to homogeneous catalysis. The structure of aurichalcite, $(Zn,Cu)_5(OH)_6(CO_3)_2$, was established from a flake of material ca. 5pm thick (an EM of the sample is shown below), and that of rosasite, $(Zn,Cu)_2(OH)_2CO_3$, from a fragment of dimensions 20x20x10 pm. The structure of the organometallic cluster, $Au_{10}(PPh_3)_7(S_2C_2(CN)_2)$ was established from a crystal of dimensions 30x10x10pm. Before the structure determination, neither the number of gold atoms in the cluster, nor its geometry, were known.



Small crystals from the powder sample of aurichalcite

The diffraction data for all three of these compounds was recorded at the SRS, Daresbury Laboratory; that for rosasite by the Laue method (full 'white' X-ray beam, stationary crystal, several photographs in different orientations), and the other two on the FAST diffractometer with monochromatic radiation of wavelength ca 0.9 Å. The number of reflection intensities measured was adequate for structure solution and some refinement, but the accuracy of interatomic distances was not as good as in a high quality structure determination with a good crystal. This information is very useful as a starting point for a bulk refinement from the powder material.

This much has been achieved with present second generation synchrotron sources and experimental arrangements

...continued on back page

NEWS FROM ICRD

Members of ICDD were greatly saddened by the death of Professor Benjamin Post on May 4 and the passing of Professor Sigmund Weissmann on May 16, 1994. Both of these scientists made invaluable contributions to the Editorial Process of The Powder Diffraction File. Accordingly, this year's ICDD publications will be dedicated in their honour.

Sigmund Weissmann's association with the International Centre dates back 41 years. He was active in the Centre's technical activities; Sig had been the Editor for Metals, Alloys and Certain Inorganic Substances of the Powder Diffraction File since the early 1950s. Of special note was Sig's untiring and almost individual effort in bringing forth the publication of the 1978 Metals and Alloys File. He took great pride in seeing his effort come to fruition. He was honoured as a Distinguished Fellow of ICDD.

Ben Post had been the Editor for Organic and Inorganic Substances of the Powder Diffraction File since the early 1950s and during that time, always put forth tremendous effort and took great pride in fulfilling this role. It was the Centre's privilege to have Ben actively participate in its activities. He served on many of the technical subcommittees and, from 1982 to 1986, was a member of the Board of Directors. Although Ben was recognized throughout the world for his crystallographic achievements - his numerous awards included the 1982 Warren award by the American Crystallographic Association - he most proudly treasured the ICDD Distinguished Fellow Award. Ben exemplified the meaning of this award.

ICDD BULLETIN

The ICDD has created a bulletin board to contain information of interest to the Powder Diffraction community. The first item on the Bulletin board is the table of contents for Powder Diffraction, Vol. 8. We are currently adding the table of contents for Vols. 1-7. The table of contents for each issue can be found in files named PDv-i. TOC, where v is the volume and i is the issue number. Each table of contents file is in ASCII and can be searched using any text editor. The file README.1ST is a road map to the information on the bulletin board.

The ICDD bulletin board is accessed through the Internet using anonymous FTP. To connect to the bulletin board, FTP to ICDD.COM, login as *anonymous* and use your Internet address as your password. The README.1ST file can be downloaded to your computer at this point by typing *get readme.1st.*. The table of contents, located in PD subdirectory, can be reached by typing the command *cd pd.* Type *dir* to display the files. To download all of the files, type mget*.toc. To download a specific file, for example Vol. 8 issue 1, type *get pd8. 1*

ICDD X-RAY CLINICS

The annual ICDD X-ray Clinics were held in June at the ICDD Headquarters in Newtown Square, Pennsylvania. Sessions were offered in both diffraction and fluorescence.
The ICDD Clinic on X-ray Powder Diffraction was held in two week-long sessions as follows:
Session I: Fundamentals of X-ray Powder Diffraction - Session II: Advanced Methods in X-ray Powder Diffraction Twenty attendees participated in Session I and thirty participated in Session II. The faculty included Tom Blanton, Paden Dismore, Mario Fornoff, Mark Holomany, Sue Hoyle, Ron Jenkins, Gerry Johnson, Earle Ryba, Deane Smith, and Dan Sundeen.

NEW PRODUCTS FROM ICDD

Set 44 of the Powder Diffraction File is now available for distribution. It contains 1500 inorganic and 500 organic entries and is published on a wide variety of media. Sets 1-44 PDF-2 database, distributed on CD-ROM, contains 59,847 active entries.

PC-PDF for Windows is a new data retrieval software package available as an option for those users of the database who have at least an 80386 processor and are operating in the Windows environment. This new program features all of the functionality of the current DOS retrieval software with the addition of a few new features. The DOS retrieval software, which has been frozen in its current form, will continue to be supplied with the PDF-2 database, free of charge.

PC Search Index is a new (DOS) program which is designed to fulfil the role of a "computer-readable search manual". It employs classical, systematic approaches such as the "Hanawalt" and "Fink" methods, which have been developed over the years to assist the user to personally allocate lines in the experimental pattern to phases of his choice, as "suggested" by the search scheme.

The Dow Polymer Pattern Collection is being made available to the public for the first time in an ICDD softcover publication. Collected by the research laboratories of the Dow Chemical Company, diffraction patterns of polymer species are reproduced in the form of diffractometer traces. The booklet also features short discussions on diffraction theory and on the experimental procedure used to obtain the patterns.

Ludo Frevel, ICDD Representative

USING CIF FOR POWDER DIFFRACTION

Most crystallographers are aware of the IUCr's efforts to define a standardized format for crystallographic information known as the Crystallographic Information File (CIF). Those crystallographers who are not may wish to review the box, "A Very Brief Introduction to CIF", or consult the enclosed references. The initial defmitions developed for CIF were designed to suit the needs of the small-molecule single-crystal crystallographic communitybutthe importance of efforts to broaden the applicalibility of CIF to suit the needs of the macromolecular and the powder diffraction community were recognised earty.

Over the last few years, a large group of individuals, most notably Syd Hall, Ian Langford and I, with the assistance of many people, have been working on expanding the definitions in CIF for powder diffraction applications. These efforts have been assisted by the members of the Commission on Powder Diffraction (CPD) and the Commission on CIF (COMGIFS). A draft of these additions has been completed in the form of a supplementary dictionary for powder diffraction, The draft dictionary is available to the crystallographic community for review prior to its adoption as a standard (see the box "On-line Access;to Information About CIF"). Those crystallographers who do not have access to electronic mail may contact the Research and Development Officer of the IUCr, Brian Mc Mahon for printed information (see box for address). The assistance of the powder diffraction community is needed to ensure that definitions used in the dictionary conform to proper usage and are sufficiently comprehensive to include the vast majority of powder diffraction applications. It should be noted that powder diffraction CIF files are likely to use definitions in both the original (core) CIF dictionary and the powder CIF dictionary, so both dictionaries should be reviewed together. Comments about the draft dictionary may be addressed to the chair of COMClFS, I.David Brown (lDBrown@mcmaster.ca) or Brian McMahon. Questions may be addressed to the principal author of the powder dictionary, Brian Toby (tobybh@ttown.apci.com)

While the goal of the initial CIF effort was to define a file format to contain information typically found in a single-crystal structure manuscript (and supplementary materials), the powder CIF effort had the additional goals of documenting and archiving experimental data. It is intended that powder CIF be used for communication of completed studies and also for data exchange between laboratories. Since a wide variety in powder diffraction instrumentation is available and because there are many different types of analyses performed using powder data, a large number of definitions were created. The dictionary was written with the goal of recording data from conventional X-ray diffractometers, as well from synchrotoron CW and TOF neutron and energy dispersive instruments. Due to lack of familiarity, a more cursory attempt was made for film instruments. Due to difficulties in storing 2-Drrays, we have detayed addressing requirements for image plates and area detector instruments.

A powder CIF may contain descriptions of:

(1) the experimental configuration;
(2) sample preparation, characterization and mounting details;
(3) references to calibration information;
(4) the experimental (raw) diffiction dataset;
(5) the processed diffraction dataset;
(6) a calculated diffractogram;
(7) a peak table;
(8) reflection assignments and intensities
(9) derived structural results

A powder CIF can also document diffraction data where multiple phases are indexed or refined. It is also possible to document a Rietveld refinement that incorporates multiple datasets.

..continued on page 4

On-line Access to Information About CIF

There are a number of methods for accessing C IF-related information via Internet. Much of the information is independent of the method used for access, but World-Wide Web (WWW) and gopher offer a more friendly user interface and more introductory material.

WWW (using Mosaic. Lynx....)

* Connect to URL http:Nwww.iucr.ac.ukelcome.html and follow references to bring you to the CIF home page.

Gopher

* Point your favourite rodent to gopher.iucr.ac.uk (port 70). Look for the section titled "CIF Dictionary files." Of most interest: the "draft powder"'and "draft revised core" dictionaries.
* Also see "General CIF-related" files for some software (ciftbx, ciftex, quasar and cyclops).

Anonymous.ftp

* Connect to ftp.iucr.ac.uk. Look in directory cifdics. Files of interest are: cifdic.c94 (core dictionary) and cifdic.p94 (powder dictionary). Also note the PostScript versions of these files with the added suffix .ps
* Also, see directory pub for some sofware (ciftbx, ciftex, quasar and cyclops).

E-mail

* Send a message to SENDCIF@IUCR.AC.UK containing the single word "help." Most of the files listed above are available through SENDCIF.

If all else fails, contact Brian McMahon, Research and Development Officer, IUCr, 5 Abbey Square, Chester CHI 2HU, England. (tel: +44 244 342878, fax:+44 244 314888, or e-mail bm@iucr.ac.uk).

A VERY BRIEF INTRODUCTION TO CIF

The Crystallographic information File (CIF) is a mechanismforstoring crystallographic information in a computer-readableASCIIfile. The formatisintendedto be human-readableand editable. The syntax of CIF is a subset of the self-defining text archive and retrieval (STAR) standard.

A CIF file consists of a series of data items (entries) and corresponding values e.g.:

```
_cell_length_a      5.959(1)
```

A data item may also have a series of values associated with it by preceding the data item with "loop," . The following examples shows a list of four symmetry operators associated with a single data item:

```
loop-  _symmetry_equiv_pos-as-xyz
       +x,+y,+z
       1/2-x,-y, 1/2 +z
       1/2+x, 1/2-y,-z
       -x, 1/2+y, 1/2-z
```

It is also possible to group a series of values together, in this case the coordinates and atomic labels:

```
loop,
_atom_site_label
_atom_site_fract_x
_atom_site_fract_y
_atom_site_flact_z
C1  .4154(4)   .5699(1)   3026(3)
C2  .5630(5)   .5087(2)   .3246(1)
C3  .5330(5)   .5350(5)   .3997(1)
```

Rotated entries are grouped together in a block. The beginning of a block is designated by the string "data-".

Data items can be recognisedbecause they always begin with an underscore (_)and values are delimited by spaces

pairs of lines beginning with a semicolon. Quotation marks are thos used to indicate a value that contains spaces, but do not span lines

```
_chemical_formula_sum      'C18H25 N 03"
```

and semicolons are used for values that span more than one line:

```
_publ_author_address
;
Research School of Chemistry
Australian National University
GPOBox 4
Canberra, ACT
Australia 2601
;
```

Thus, CIF is largely free format. Two restrictions were implemented to allow facile transmission of CIFs by e-mail: lines may not exceed 80 characters and only printable ASCII characters may be used.

Data items used in CIF are described in a dictionary, which defines the meaning and usage. For example the dictionary entry far cell_lengtha specifies that the value wilt be a number in Angstroms and that an ESD is allowed, The dictionary is itself a STAR file where the dictionary syntax is defined in a separate Dictionary Definition Language (DDL).

References;

CIF: S Hall, F. Allen &I.D. Brown, Acta *Cryst. A47* (1991), 655-685

STAR: S. Hall & N. Spaccini, J Chem lnf Comput. Sci. 34 (1994), 505-508

Brian H. Toby, Air Products and Chemicals

NEW BOOKS

A NEW BOOK ON POWDER DIFFRACTION BY CZECH AND SLOVAK CRYSTALLOGRAPHERS

This book has been published under the title "Difrakcia na polykrystalickych latkach("Diffraction by Polycrystalline Materials", 458 pp., ed. by.Lubos Smrcok, ISBN -80-P85488-01-9 R&D Print, Bratislava, Slovakia, 1994. Chapters have been written by A. Buchal, P Capkova, I. Cerven, S. Durovic, P Fejdi, I. Fiala, N. Ganev, M. Jergel, I. Kraus, R. Kuzel, D. Louer, D. Rafaja, M. Rieder, L. Smrcok and Z. Weiss. They cover the fundamentals of crystallography, principles of the diffraction of X-rays by crystals, instrumentation, experimental procedures, texture analysis, indexing, identification and phase abundance analysis, structure solution and refinement, stress measurement, profile fitting, thin films investigation, radial distribution analysis and synchrotron utilization.

Authors of individual chapters met at a summer school on July 18-21 1994 and presented lectures on their subjects to some 60 attendees to teach good powder diffraction practice to young Czech and Slovak crystallographers. In the presence of Daniel Louer, secretary of the Commission on Powder Diffraction of the IUCr, the meeting took place in Liptovsky Mikulas, a beautiful town in the middle of Slovak mountains, where Regional (Czech and Stovak) Power Diffraction Conferences are organized regularly (the next one October 3-7 1994).

Jaroslav Fiala

Report on the International Conference Powder Diffraction and Crystal Chemistry'' Saint Petersburg, 20-23 June 1994

The conference was organised by the Commission on Powder Diffraction of the lUCr, the Russian Association "Powder Crystallography", firma Bourevestnik (the main Russian producer of x-ray equipment) and the Department of Crystallography of St. Petersburg University (Prof S. K. Filatov, chairman of the Organizing Committee) at the campus of St. Petersburg University, 20 - 23 June, 1994.

The scientific program of the conference was devoted mainly to the new developments in traditional powder diffraction methods, achievements in crystal chemistry on the basis of powder data, high temperature and high pressure diffraction, and the use of neutron, electron and synchrotron x-ray diffraction in crystal chemistry.

40 lectures and 70 posters were presented to 165 particpants from 20 countries along with a JCPDS-ICDD workshop run by Dr. J. W. Visser and Prof. R. L. Snyder and an exhibition of scientific instruments and software products. A volume of collected abstracts (121 pages) was provided to attendees of the conference upon registration. There were many remarkable contributions, especially "Crystal structures of the high temperature superconductors" (R.L.Snyder), "Angle dispersive time-of-flight powder diffraction" (G.Will), "X-ray powder diffraction line broadening analysis as a characterisation tool for oxide ceramics"(G. Kimmel), "Structure systematics of solids under strong compression'' (W.B.Holzapfel), "Structural disorder in Fe-layer silicates by simulation of X-ray powder diffraction patterns"(E.A. Goilo), "Powder X-ray diffraction in situ studies of structural phase transformations in framework aluminosilicates at high pressures in DAC" (B.A.Fursenko) and "Determination of actual crystal structure of minerals by simulation method of powder diffraction patterns" (B.A.Sakharov).

The social program included - among many attractive items - a skip voyage along the Neva river, excursion to St. Petersburg and a visitto a theatre and the Hermitage, letting the participants appreciate the wonderful atmosphere of St. Petersburg where crystallography has a long and glorious tradition.

Let us mind ourselves that it was here in St. Petersburg, where Ye. S. Fedorov discovered the 230 space groups in 1890, which opened the door to modern crystallography. Prof. Fedorov founded the first Department of Crystallography in Russia, here at the Mining Institute, and his pupil, Professor O.A. Anshelis, founded the second Department of Crystallography in St. Petersburg at the University of St.Petersburg in 1924. It is this Department which served as the local organising body of the present Conference (and the todays chief df the Department took the role of the Chairman of the Organizing Committee). The broad contribution of Ye. S. Fedorov to crystallography (let us recall here his design of the universal stage for petrography) has produced a wide spectrum of works by his pupils in structure analysis, crystal growth, optical crystallography and mineralogy - today's Fedorov school of crystaltography. It is hoped that this successful international conference of Powder Diffraction and Crystal Chemistry will stimulate the further development of both Russian science and international crystallography.

Czech-Slovak Summer School on Diffraction by Polycrystalline Materials,Liptovsky Mikulas, Slovakia, July 17-21, 1994

A Summer School on Diffraction by Polycrystalline Materials was held at Liptovsky Mikulas (Slovakia) from 17-21 July 1994. This course was organized by Dr. L. Smrcok from the Slovak Academy of Sciences (Bratislava). The local organizer was Dr P. Sutta and the course took place in facilities belonging to the Military Academy. More than 50 registered students from the Czech and Slovak republics attended the School. The course programme was well prepared and Dr.L. Smrcok edited a book from the contributions presented by the invited lecturers. This 458-gage book was distributed to the attendees and provided efficient support for the students: it was a key factor in the School's success. The book contains 16 Chapters written in the Slovak language, except for Chapter 9, which is in English.

The programm covered a broad spectrum of crystallography: basic principles on lattices and symmetry in crystalls (S. Durovic, Chapter 1; P. Fejdi, Chapter 2), theory of diffraction and experimental methods for the study of crystals and powders (I.Cerven ,Chapter 3; S. Durovic, Chapter 4; M. Rieder Chapter 5), detectors (N. Ganev and I. Kraus, Chapter 6), diffracted intensity by powders (D. Rafaja, Chapter 7), texture of polycrystalline materials (P. Capkova, Chapter 8), N. Ganev and I. Kraus, Chapter 12), powder pattern indexing (D.Louer, Chapter 9), qualitative and quantitative analyses (J. Fiala, Chapter10), the Rietveld method (L. Smrcok, Chapter 11), microstructural properties (A. Buchal, Chapter 13, R. Kuzel, Chapter 14), electronic radial distributions (M. Jergel, Chapter 15).

...continued on page 6

APPLIED CRYSTALLOGRAPHY in CIESZYN, Poland

For four days in August 1994, the Cieszyn branchof the University of Silesia was host to the XVI Conference of Applied Crystallography, organised by the Institute of Physics and Chemistry of Metals. These conferences are organised biannually and started in 1964. This time the conference was sponsored by the Polish Ministry of Education, Committee of Research, Committee of Material Science of Polish Academy of Sciences and by Philips Analytical and JEOL Ltd. As at previous conferences, themain aim of the organisers was to enable scientists from West and East to meet, discuss and exchange knowledge, opinions and experience. This was possible due to the site of the Conference and reasonably low price of the fees. 138 scientistsparticipated, from such countries as Argentina, Austria, Bulgaria, the Czech Republic, Greece, France, Holland, Spain, Japan, Germany, Russia, Ukraine, USA and, of course, from Poland.

During 4 days the participants were exposed to 34 lectures and 81 posters. The conference subjects included phase characterization using diffraction patterns, powder diffraction methods in phase transformation studies, correction factors in the powder diffraction method, application of the Rietveld method, substructure analysis in textured materials, texture inhomogeneity and its determination, new X-ray diffraction techniques, application of position sensitive detectors software tools in applied crystallography, small angle X-ray scattering, stress analysis and its application to different materials, phase transition studies and special crystallography and peculiarities of the reversible martensitic transformation structure of non-crystalline materials and their crystallization and structure and properties of some new materials. Moreover, one could visit stalls of the JEOL, PHILIPS and SEIFERT companies where their representatives presented the newest products. There were several social events during the conference e.g. a tamb barbecue in Brenna where the participants could try toride a horse or to ... sing. The XV II Conference on Applied Crystallography will be held in 1997.

Danuta Stroz

Czech-Slovak Summer School, continued

synchrotron radiation (M. Jergel, Chapter 16). Basic mathematics used in crystallography were presented in two annexes (I. Cerven and Z. Weiss). In addition to the dense programme covered by the lectures, tutorial and practical sessions were organized with a few exercises carried out on personal computers

The school sites were fascinating Liptovsky Mikulás is situated between the mountains, the High and Low Tatras. A break was arranged by the organizers on Wednesday afternoon for a hike organized by Dr P. Fejdi at Prosiecka Dolina. All participants enjoyed this walk in the mountains which also contributed to the success of the School.

Daniel Louer, Universite de Rennes

WHO'S WHO: THE IUCr COMMISSION ON POWDER DIFFRACTION

CHAIRMAN

Dr. R. J. Hill (Rod), Division of Mineral Products, CSIRO, P. O. Box 124, Port Melbourne, Victoria 3207, Australia. Tel: + 61-3-647-0208: FAX: + 61-3-646-3223; E-mail: RODH@DMP.CSIRO.AU.

MEMBERS

Dr. R. J. Cernik (Bob), Daresbury Laboratory, Daresbury, Warrington, WA4 4AD, UK. (FAX. + 44-925-603174 or 100)(CERNIK@DARESBURY.AC.UK).

Dr. D.E. Cox (Dave), Physics Department, Brookhaven National Laboratory, Upton, N.Y. 11973, USA. (FAX: +1-516-282-2739), COX@BNLX7A:NSLS.BNL.COV).

Dr. J. Fiata (Jaraslov), Department of Metallurgy, Central Research Institute SKODA, Tylova 46, 31600 Pizen, Czech Republic. (FAX: + 42-19-220-762)

Dr. D. Louer (Daniel) (Secretary), Laboratoire de Cristallochimie, Chimie de Solide et Inorganic Molecule, Universite de Rennes 1, 35042 Rennes, Cedex, France. (FAX: +33-99-38-34-87), (DANIEL.LOUER@UNIV-RENNES1.FR).

Dr. L. B. McCusker (Lynne), Institut fur Kristallographie und Petrolographie, ETH Zentrum, CH-8092 Zurich, Switzerland. (FAX:+41-1-632-1133) (LYNNE.MCCUSKER@ KRISTALL.ERDW.ETHZ.CH).

Prof. LIN Shao-Fan (Shao-Fan), Test and Computation Centre, Central Laboratory, Nankai University, Tianjin 300071, PR China (FAX: +86-22-337-1555), **(FENGCB@BEPC2.IHEP.AC.CN)**

Prof. D. K. Smith (Deane), 239 Deike Building, Department of Geosciences, The Pennsylvania State University, University Park, PA 16803, USA. (FAX: + 1-814-863-7845), (SMITH@ VAX1.MRL.PSU.EDU).

Prof. I. C. R. Tellgren (Roland), Institute of Chemistry, Uppsala University, Box 531, S-75121, Uppsala, Sweden. (FAX: +46-18-508-542 or 111-853), (ROLAND.TELLGREN@ KEMI.UU.SE).

Dr. H. Toraya (Hideo), Ceramics Research Laboratory, Nagoya Institute of Technology, Asahigaoka, Tajimi 507, Japan. (FAX:+81-572-27-6812), (H43517@SINET.AD.JP).

ICDD REPRESENTATIVE

Dr. L. K. Frevel (Ludo), 1205 W. Park Drive, Midland, MI 48640, USA. (FAX: +1-610-325-9823).

CONSULTANTS

Dr. J. I. Langford (Ian), School of Physics and Space Research, Birmingham University, P.O. Box 363, Birmingham, B15 2TT, UK. (FAX: + 44-21-414-6709 or 4577), (XT-I@IBM.PHYSICS .BIRMINGHAM.AC.UK).

Dr. R. A. Young (Ray), School of Physics, Georgia Institute of Technology, Atlanta, GA 30332-0430, USA. (FAX:+1-404-853-9958), (PH268RY@ GITVM1.GATECH.EDU).

WHAT'S ON?

20-22 March 1995

Fourth Workshop on Powder Diffraction Techniques. Gargnano (Brescia), Northern Italy. The subjects of the 1995 school will be the Contribution of Powder Diffraction Techniques to Structural Crystallography and ab-initio and Rietveld Techniques. Contact Norberto Masiocchi, University of Milano, lstituto di Chimica Strutturistica Inorganica, via venezian 21, I 20133 Milano, Italy. Tel: +39-2-70635 120. Fax: 39-2-70635288. E-mail: **norbert@stinch0.csmtbo.mi.cnr.it**

1-3 May 1995

Rietveld Method Short Course. Atlanta, USA. A commercially-run course with a fee of $695. This 3-day course will be conducted by Professors Von Dreele and Ray Young. The course content will deal with the principles and techniques of Rietveld analysis. There will be lectures and hands-on tutorials catering for both beginners and the moderately experienced. Contact Department of Continuing Education, Georgia lnstitute of Technology, Atlanta, Georgia 30332-0385, USA. Tel: 4041894 2547

10-15 July 1995

EPDIC **IV.** Chester College, UK. The 3-day meeting will cover all aspects of powder diffraction, with particular emphasis on structure solution and refinement, dynamic studies of structures under change and the industrial importance of the technique. The scope of the meeting will be as broad as possible and will be covered by a series of plenary and invited speakers. There will be comprehensive poster sessions in all areas of powder diffraction Contact: Dr. R. J. Cernik, Daresbury Laboratory, Daresbury, Warrington, WA4 4AD, UK. Fax: +44 925 603195; E-mail: cernik@daresbury.ac.uk

16-20 July 1995

Ab-lnitio Structure Determination Using Powder Diffraction Techniques. Oxford, UK. Speakers will include Christian Baerlocher, Dave Cox, Andy Fitch, Chris Gilmore, Lynne McCusker, Kenneth Shankland, Gerard Bricogne, Bill David, Clive Freeman, Rod Hill, John Newsome, Devinder Sivia, Tony Cheetham, Michael Estermann, Carmello Giacovazzo, Daniel Louer Rene Pescher and Per-Erik Werner. Topics to be covered include optimal data collection strategies; auto-indexing; Bayesian methods; intensity extraction; direct methods; Rietveld refinement; maximum entropy/likelihood; prediction; computer modelling. Places limited to about 100. Contact: Dr. W. I. F. David. (e-mail: wifd@isise.rl.ac.uk) or Kenneth Shankland.

23-28 July 1995

American Crystallographic Association Meeting. Montreal, Quebec, Canada. Contact: Dr. Y. LePage, Program Chairman, NRC of Canada, Chemistry Department, Ottawa, Ontario, KIA OR6, Canada. Tel: +I-613-993-2527; Fax: +I-613-952-1275; E-mail: **yvon@iecems.lan.nrc.ca.**

6-11 August 1995

16th European Crystallographic Meeting. Lund, Sweden. Contact: Dr. Ake Oskarsson (Chairman), Department of lnorganic Chemistry I, Chemical Center, Lund University, P. **O.** Box 124, S-221 00 und, Sweden. Tel: +46-46-108102; E-mail: ake.oskarsson@inorgkl**.lu.se.**

21-29 August 1995

International Conference on X-ray Powder Diffraction Analysis of Size/Strain, Macrostress, and Texture. Liptofsky Mikulas, Slovakia. Contact: Dr. J. Fiala, Department of Metallurgy, Central Research Institute SKODA, Tylova 46, *3* 1600 Pizen, Czech Republic. Fax: +42-19-220-762.

3-9 August 1996

Satellite Meeting on Powder Diffraction associated with the IUCr Congress on Crystallography and Denver X-ray Conference. Denver, Colorado. Contact: Prof. Paul K. Predecki, Department of Engineering, University of Denver, Denver, CO 80208, USA. Fax: +I-303-87 1-4450; E-mail: denxcon@diana.cair.du.edu

18-17 August 1996

17th IUCr General Assembly and International Congress of Crystallography. Seattle, Washington, USA. Contact: Prof. R. F. Bryan, Department of Chemistry, University of Virginia, Charlottesville, VA 22903, USA.

12-21 September 1995

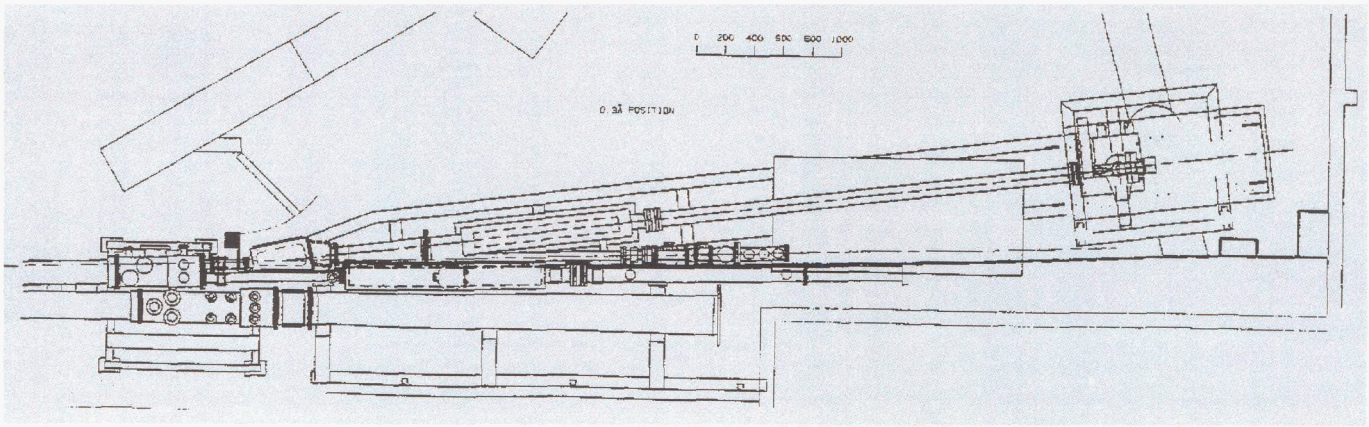
4th Oxford Summer School on Neutron Scattering. Oxford, UK. Sponsored by IUCr, Contact: Professor B T M Willis, Chemical Crystallography Laboratory, 9 Parks Road, Oxford, OX1 3PD, UK. Fax: +44 865 272960.

THE POWDER DIFFRACTION BEAMLINE AT THE ESRF

In the last newsletter, it was intimated that Pekka Suortti was responsible for the design of the powder instruments at the ESRF. Although Pekka has a long-standing interest in powder diffraction, he is responsible for the high energy beamline (beamline 5) and is not directly involved in the powder diffraction project on beamline 15. This is being constructed by the diffraction group. led by Ake Kvick, and designed by Andy Fitch. We regret any confusion that may have been caused.

MAILING LIST FOR NEWSLETTERS

If you would like to be added to the mailing list for the Commission on Powder Diffraction, or you have changed your address, please contact the CPD Secretary, Dr. Daniel Louer at the address shown on page 6.

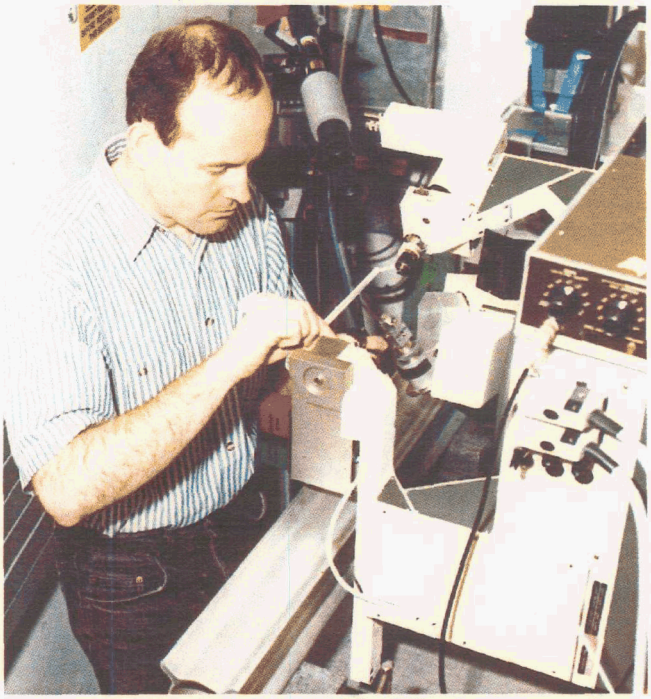


Layout of the new high-flux X-ray diffraction station at Daresbury

not fully optimised for these very small crystals. Several new developments are expected in the near future, including a new workstation at Daresbury Laboratory with intense focused monochromatic radiation, highly appropriate for very small single crystals.

So which will be appropriate for structure determination - powder diffraction or single crystal diffraction? There are a variety of considerations, and one warning! If, despite repeated attempts at recrystallisation, no good sized crystals can be obtained, the small crystals are probably far from perfect and may have stacking faults or other disorder. Single crystal methods for data collection will be preferable provided an adequate number of reflection intensities can be properly measured; still smaller crystals will need even more intense X-ray beams and better signal to noise discrimination. It will obviously be wise to check, using the powder diffraction pattern, that the single crystal used is typical of the bulk sample. Good powder diffraction data can be recorded even when the individual crystallites are very small. Multiphase Rietveld refinement can then be employed to solve the whole structure. Structure solution from powder diffraction patterns may be difficult for the more complex compounds, and can also be made difficult or impossible by the presence of impurities. For many problems there is a case for the use of both methods - unit cell and approximate structure from single crystal data, followed by Rietveld refinement with the powder data to give the bulk structure.

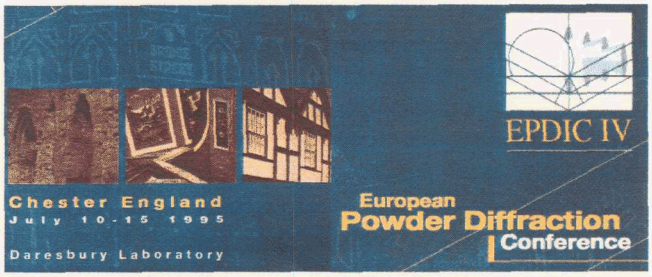
The plans for the new station at Daresbury are shown at the top of the page, and work is progressing on the installation of the new diffractometer (above right). This will provide ideal facilities for small crystals. However, new developments on third generation sources, such as the micro focus beamline at the ESRF, will allow crystals as small as 1μm, or even less, to be studied.



The installation of a four-circle diffractometer on the new beamline at Daresbury

These new developments highlight the complementary nature of small single-crystal and powder diffraction. This combination promises to be a powerful tool in solving crystal structures.

Marjorie Harding and Bob Cernik



CALL FOR CONTRIBUTIONS TO THE NEXT CPD NEWSLETTER

The next issue of the CPD Newsletter will be edited by Dr R Tellgren to appear in April of 1995. He would greatly appreciate contributions from readers on matters of interest to the powder diffraction community, eg meeting reports, future meetings, developments in instruments, techniques and computer programs and news of general interest. Please send articles and suggestions directly to him.

R J Cernik, Editor, CPD Newsletter 13

This newsletter was produced by Scientific Administration, Daresbury Laboratory, for the Commission on Powder Diffraction