CO MMISSION ON POWDER DIFFRACTION

INTERNATIONAL UNION OF CRYSTALLOGRAPHY

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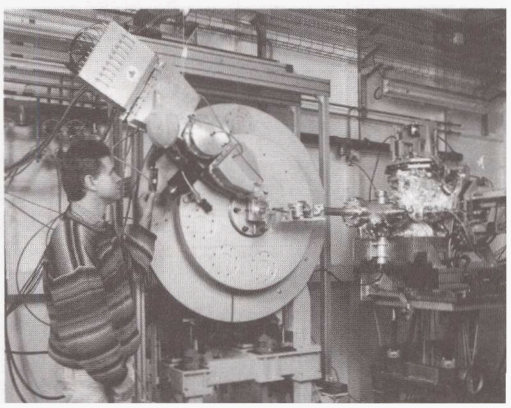
POWDER DIFFRACTION AT ESRF

by A.N. Fitch, ESRF, BP 220, 38043 Grenoble Cedex, France

The benefits of using synchrotron radiation for high quality powder diffraction studies have been emphatically demonstrated at national synchrotron radiation sources. Hence, it is not surprising to find intense interest in developing the technique at the recently constructed European Synchrotron Radiation Facility in Grenoble, the first of the world's third generation synchrotron radiation sources. There are now three beam lines with powder diffraction facilities operating on bending magnets, and a fourth is under construction. The bending magnets deliver powerful, vertically-collimated beams with a wide range of photon energies. These are suited to many applications, in particular diffraction and absorption spectroscopy. Three of the beam lines for powder diffraction are operated by externally-funded collaborating research groups (CRGs): the Swiss-Norwegian (SNBL), the Italian (GILDA), and the Dutch-Belgium (DUBBLE) beam lines. The latter is currently under construction, with its powder diffractometer due to become operational in 1998. The ESRF has itself built a dedicated powder diffraction beam line, BM-16, for high resolution powder diffraction studies (Figure 1). In addition, there are high-pressure facilities on two insertion devices, where powder specimens can be examined in a range of diamond anvil cells or large volume presses, at temperatures up to 1500°C.

Because of the very small source size at a third generation source, it is advantageous to focus the radiation, and much care is taken in the optical design of a beam line. The optics for BM-16 are typical for a bending-magnet beam line at the ESRF. The beam line can accept up to 4 mrad of radiation horizontally from the bending magnet. Thus, at the first mirror, 25 m from the source, the beam is 100 mm wide. The first mirror is 1.2 m long and is made from silicon, polished to a roughness of a few Å, and coated with rhodium. The mirror is bent by a pneumatic system to a radius of ca 30 km and set at an angle of grazing incidence to intercept a beam approximately 2 mm high. At this angle of incidence, only photons with a wavelength greater than 0.3 Å are transmitted. The curved mirror collimates even further the already highly collimated beam. After the mirror, the residual vertical divergence of the beam is ca 2-3 arc sec. If the beam passes directly to the sample without further vertical focussing, this high degree of collimation ensures the highest angular resolution for the diffraction pattern. Horizontal focussing by sagittal bending of the second crystal of the double-crystal monochromator can lead to a large increase in flux at the sample, and a second mirror can be used to focus the beam vertically onto the sample, gaining flux at the expense of poorer angular resolution.



The GILDA beam line has a 2-axis Seifert diffractometer equipped with two detection systems. The first is a 16-element Si(Li) semiconductor detector with an angular resolution of 0.2" and good energy resolution (300 eV at 5.9 keV) for anomalous scattering on weak scatterers like amorphous compounds or liquids. The second has an analyser crystal in the diffracted beam to enhance the angular and energy resolution for powder diffraction and to suppress fluorescence and Compton scattering. A high-temperature cell is also available.

The Swiss-Norwegian line has a high-accuracy, heavy-duty powder diffractometer manufactured by Airmatic Engineering, Bath, UK. The diffractometer is normally run in Debye-Schemer mode, scanning a narrow slit before a scintillation detector. Peak **FWHM** of 0.03°2θ are routinely available from good quality specimens. The diffractometer has been operating since January 1995, and a number of structures have been solved or refined from SNBL data For example, the novel microporous aluminophosphate UiO-7, (unit cell volume 3700Å^3, 30 independent atoms) [1] and the zincosilicate VPI-9 (unit cell volume 7195Å^3, *48* independent atoms) [2]. This is the most complex framework topology yet solved from powder diffraction data using automated procedures. Other compounds investigated include the stable and metastable

forms of $(CH_3)_2SBr_2$ [3]. The use of a MAR Research on-line image plate scanner has also proved valuable for powder diffraction experiments on less-well-crystallised materials such as fullerenes, which do not require high angular resolution, but do require high flux and a highly efficient detector system, because the scattering is weak. A useable 1-dimensional powder pattern could be obtained from a sample of $C_{59}N$ in 180 s by integrating around the Debye-Scherrer rings on the image plate [4].

BM-16, the ESRF powder diffraction beam line, welcomed its first users in May 1996, after nearly four and a half years of development and construction [5]. The beam line is aimed at a wide range of powder diffraction experiments needing very high angular or energy resolution, for structure solution and refinement, for microstructural characterisation and for anomalous scattering studies. The beam line is also well suited to general materials science applications, e.g. measurements of stress and strain in surface layers or in the bulk, studies of glasses, and studies at grazing incidence. With the acquisition of a position sensitive detector next year, dynamic experiments on the time scale of a few seconds will also be feasible. The operational wavelength range is 2.5 - 0.3 Å *(5* - 40 keV), making accessible a K or L absorption edge for the elements from Ti to U.

The diffractometer was developed in a joint project with the SNBL, and hence the two diffractometers have similar mechanical characteristics. On BM-16 a multianalyser stage [6] is used for routine operation in high-resolution mode. This comprises a bank of nine detectors, each behind a Ge 111 analyser crystal, with the nine crystals mounted on a single rotation stage. The separation between each channel is close to 2˚2θ. Data are collected in a continuous scanning mode, with the 2θ arm moving at a constant rate and the angular encoder and the electronic scalers for each channel read, without resetting, as often as desired (e.g. 1 to 20 Hz), depending on the scanning speed. This approach eliminates the dead time inherent in a step scan, which would be large for a high resolution diffraction pattern where many small steps are needed. Following data collection, the counts from the nine channels are rebinned, taking into account the exact separation between channels, the different detector efficiencies, and the decline in the beam current during the scan, to produce the equivalent normalised step scan, which is more suitable for analysis by standard programs. The data can be rebinned into whatever step is appropriate (typically 0.002-0.005˚2θ).

So far the beam line has been operated with a Si 111 monochromator without sagittal focussing, and the data in some cases have been spectacular. For a sample of silicon, a peak width of less than 0.007" was obtained. As an example of the quality of the data, the high angle range of the pattern for a sample of europium perchlorate measured at a wavelength of 0.55 Å is shown in Figure 2 [7]. The short wavelength means that a capillary of reasonable size (0.3 mm) can be used without absorption problems. The structure was solved and refined from these data. Other samples studied for structural characterisation include various microporous materials; a new aluminosilicate (only a few mg) synthesised under extreme hydrothermal conditions (possibly a constituent of the Earth's mantle); and solid phases of organic molecules, at both ambient and low temperatures. Two in-situ studies deserve mention: (1) a study of the chemical changes in the hydrothermal transformation of zeolites, which required steel capillaries and high photon energies for penetration, and (2) an investigation of the structural changes accompanying the charging-discharge cycle in various electrochemical cells.

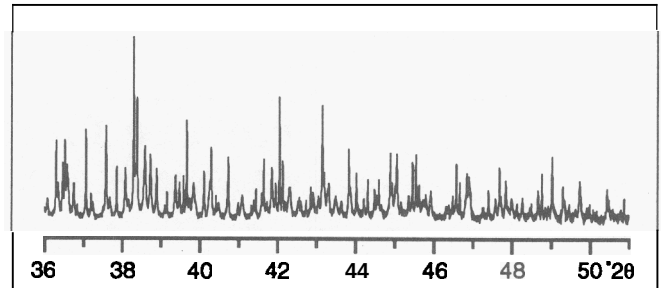


Figure 2. High angle data collected on a europium perchlorate sample at λ = 0.55 Å (d-spacings range from 0.89 to 0.64 Å)

Resonant scattering experiments have also been performed to determine the disordered cation arrangements in layered magnetoresistive perovskites, and in possible hydrogen-storage alloys. For the former, the high resolution data revealed a broadening of the 001 reflections, so the stacking faults caused by the insertion of extra perovskite layers between the double-perovskite layers of the ideal structure could be studied [8]. Small shifts in the position of an absorption edge are characteristic of different oxidation states for the absorbing ion, and this phenomenon was exploited to do some oxidation-state specific diffraction experiments. The edges probed have covered the full span of BM16's energy range, from the Mn K edge (6538 eV) to the La K edge (38934 eV). Strain measurements with high energy photons have been used to probe Ti alloy materials, and the distribution of stress through 10 mm thick plates of aluminium. Also, the depth at which the surface compressive stress is balanced by a tensile stress in peened alloys has been investigated.

The early experiments using the powder diffraction beam lines at ESRF have already been highly successful. Further enhancements, such as the implementation of sagittal focussing on BM-16 are planned for next year. The availability of highly-monochromatic, intense X-ray sources combined with instrumentation providing excellent angular and energy resolution are leading to the characterisation of bigger and more-complex structures, deeper penetration into or through materials, and the exploitation of anomalous-scattering over a wide energy range. The future is bright for powder diffraction studies at the ESRF. Information about obtaining beam time is available from The User Office, or via http://w3i.esrf.fr.

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[4] **C.M. Brown, L. Cristofolini, K. Kordatos, K. Prassides, C. Bellavia, R. Gonzalez, M. Keshavarz-K, F. Wudl, A. K. Cheetham, J.P. Zhang, W. Andreoni, A. Curioni, A.N. Fitch and P. Pattison, *Chem. Mater.*, In Press.**

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[6] J.L. Hodeau, P. Bordet, M. Anne, A. Prat, A. Fitch E. Dooryhee and G. Vaughan, Proc. 45th Annual Denver Conference.
[7] F. Favier and J.-L. Pascal, Exp. HC-323 on BM-16 at ESRF.
[81 R Seshadri, M. Hervieu, C. Martin, A. Maignan, B. Domenges, B. Raveau and A. Fitch, to be submitted to *Chem Muter.*

CHAIRMAN'S MESSAGE

The CPD was formed in 1987, and since then a large number of people have worked very hard on behalf of the Commission. I would like to thank everyone who has helped in this respect, especially Rod Hill, the out going chairman. Rod has done a great deal to put the Commission in its current healthy state and he will be a hard act to follow.

One of the most fundamental arguments for the formation of the commission was the unique position of international research using powder diffraction. In the field of crystallography represented by the main body of the IUCr there is more activity from academic institutions than from industry. This position is exactly reversed in the field of powder diffraction, and it is one of the major purposes of the commission to provide an information link between the very large industrial community and the more academic, less applied aspects of the subject. 'Ihe commission will continue to do this through the IUCr by endorsing large and small meetings, by encouraging collaboration between groups working in similar fields, by enhancing mobility of students to attend CPD events, and by undertaking specific projects designed to improve standards and research practices in powder diffraction. A particular aim of the CPD that I am very keen to continue is to encourage the teaching of powder diffraction in developing countries

The CPD has a mandate from the IUCr to collaborate with other commissions, this is especially important given the number of new and exiting areas of research using crystallography. I very keen to try to open up links that might be of interest to powder diffractionists in the areas of electron diffraction, XAFS, high pressure research, neutron and synchrotron research and small angle scattering.

The CPD has a very close relationship with the International Centre for Diffraction Data. The ICDD have over 15,000 people on their circulation list which illustrates the huge number of scientists engaged in powder diffraction. These people would not normally describe themselves as crystallographers but as materials scientists or chemists. It is the job of the CPD to draw together these groups, to encourage co-operation and collaboration, to accelerate the already phenomenal growth of the subject and to make new information available to everyone.

Bob Cernik

BOOK REVIEW

Introduction to X-ray Powder Diffraction

by Ron Jenkins and Robert L. Snyder

Ron Jenkins and Robert Snyder have written a comprehensive volume covering the use of X-ray powder diffraction using common laboratory instrumentation for phase identification and quantitation. In sequential chapters, the authors provide an introduction to X-ray radiation, diffraction theory and crystallography, X-ray generation, monochromation and detection, X-ray cameras and diffractometers, sample preparation, and use of the ICDD-JCPDS database for phase identification and quantitative analysis. While the book has virtually no coverage of non-routine instrumentation, it is rich in historical detail, practical experience and illustrative examples.

Of particular merit are the chapters on radiation production, monochromation and diffractometer design. While they are limited in scope to sealed-tube technologies, these chapters present details I have not seen elsewhere in print. For example, I found the description of gear designs in diffractometers both unique and very interesting. The chapter describing diffractometer alignment is also extremely detailed. While parts of the chapter are directly applicable to only older Philips instruments, it still provides a comprehensive overview to this very important process.

The authors state that they intend their book to be used as "an introduction for students of materials science, mineralogy, chemistry and physics." As such, they have missed their target. A course text should present a wider range of information and perhaps less detail. I cannot support teaching the diffractionists of the 21st century with a text that discusses strip-chart recorders and half-wave rectifiers but has only only oblique references to synchrotrons and less than a page on rotating anode generators. While the majority of X-ray diffraction measurements will continue to be made with conventional instruments, students need to see the broader picture so that they will know when to avail themselves of more advanced techniques such as high-resolution parallel beam optics, anomolous dispersion, neutron diffraction or many of the other topics not covered in this book.

On the other hand, the book is an excellent reference guide for practitioners of diffraction. If I had to pick one book that should be on the shelf next to every X-ray diffractometer, it would be this. While there is no coverage of crystal structure determination from powder diffraction, powder crystallographers who use conventional instruments will find much of value in the book, particularly the specimen preparation chapter.

Brian Toby

ROUND ROBIN ON QUANTITATIVE PHASE ANALYSIS

Announcement and Call for Expressions of Interest

Initiated and Organized by the IUCr Commission on Powder Diffraction

Aims of the Round Robin

The round robin will focus on the analysis of powder diffraction data specifically for the derivation of quantitative phase abundance. While the study will focus on the use of laboratory X-ray, synchrotron X-ray and neutron diffraction data, other methods may be used to validate the diffraction results (e.g. FTIR, normative analysis, etc.). The additional methods used will be at the discretion of the participant.

General aims

The general goals of the round robin will include the following:

- To document the methods & strategies commonly employed in Quantitative Phase Analysis (QPA), especially those involving powder diffraction
- To assess (i) levels of accuracy & precision, and (ii) lower limits of detection
- To identify specific problem areas & develop practical solutions
- To formulate recommended procedures for-QPA using diffraction data
- To create a standard set of samples for futurereference

Specific aims

The round robin will address the following analytical issues:

Type of analysis:

- diffraction (X-ray Neutron) vs non-diffraction internal std vs external std vs spiking etc standardless methods

Samplefeatures

- representivity & homogeneity
- particle & crystallite size
- statistics & microabsorption
- crystallinity & surface roughness
- preferred orientation, microabsorption & extinction

Data collection

- type of instrument / geometry
- sample preparation
- data range and wavelength

Data analysis

- integrated intensities vs full-profiie Rietveld vs data-base of observed patterns
- use of constraints and corrections
- software systems & methods
- complexity of the pattern - peak overlap

Proposed Samples

The samples used in the study will consist of mixtures of major and minor components covering a wide range of analytical complexity. All synthetic samples contain a minor impurity phase (spinel - $MgAl_2O_4$) to provide an ambiguity of chemistry as well as providing estimates of the accuracy and precision of trace phases.

Simple system

α-Al_2O_3 + Si + MgO + $MgAl_2O_4$ (minor)

Preferred orientation

$Mg(OH)_2$ + α-Al_2O_3 + MgO + $MgAl_2O_4$ (minor)

Crystallinity

α-Al_2O_3 + $MgCO_3$ + amorphous + $MgAl_2O_4$ (minor)

Microabsorption X-ray

α-Al_2O_3 + Fe_3O_4 + $ZrSiO_4$ + $MgAl_2O_4$ (minor)

Microabsorption neutron

α-Al_2O_3 + B_2O_3 + H_3BO_3 + $MgAl_2O_4$ (minor)

Complex system

(i) Granodiorite (quartz + 2 x feldspar + biotite)
(ii) Bauxite ore (gibbsite + hematite + kaolinite + etc)
(iii) Pharmaceutical system

Types of analyses

The involvement of participants in the round robin can vary depending on the amount of time available to each individual/laboratory. However, since the success of the round robin will depend on the number of results returned, full participation is encouraged.

Possible levels of participation will include the following:

Diffraction data only

Participant analyses 'standard' data sets supplied by the CPD and returns details of the analytical procedure plus the results.

Prepared samples

Participant collects own data for analysis from at least two of the samples supplied by the CPD ('simple' and 'preferred orientation')

Unprepared samples

Samples supplied by the CPD are representative of bulk, but may need additional preparation. Participant prepares samples for data collection and analysis.

Since the purpose of the round robin is to assess the methods of quantification, and not identification, the identity of each of the component phases will be supplied. For those participants using Rietveld based methods for the analysis, full structural information for each of the phases will also be supplied. Participants will be *asked* to return the results using 'standard' reporting forms. This should minimize the number of errors likely to occur during transcription of the data. In addition, it is requested that the diffraction data be returned to the CPD as an ASCII format file for re-analysis with a 'standard' Rietveld code.

Timeline

The following timeline for the round robin is proposed:

August / September1996

Final recommendations for mixtures and protocol
Formal approval from CPD

August 96 - March 97

Call for expressions of interest
Samples sourced & prepared for distribution
Collection of standard data
Questionnaire sent to all participants

June 97

Samples and data distributed to participants

December 97
Call-in of results
January-June 98
Analysis of results (including late submissions)
August 98
Preliminary results presented at EPDIC-6
August 99
Presentation of final results at Glasgow IUCr Congress

How **to participate**
Potential participants can register their interest in the round robin by-sending an Email message containing the full contact information to the address given below. Details should include Name, Affiliation, Postal address, Phone and FAX numbers (including country and area codes) and Email address. Respondents will be sent a questionnaire requesting information about which of the samples they wish to analyze plus the amount of sample required (i.e. are they an X-ray or neutron user).

Operating Team

IanMadsen	CSIRO Minerals, Australia
Rod Hill	CSIRO Minerals, Australia
Edward Groleau	Eli Lilly & Co, USA
Lachlan Cranswick	Melbourne, Australia

Advisory Team

Deane Smith (CPD)	Penn State University, USA
Jaroslav Fiala	Central Res. Inst., Czech Rep.

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Neutron Powder Diffraction **Beamtime** Available in Sweden

Since January 1,1996, the Neutron Research Laboratory (NFL) of Uppsala University, Sweden, has received financial support from the European Union, (EU). This means that all scientists from EU-countries can apply for beamtime and financial support for travel and living expenses during the data-collection. (Scientists outside EU can get beamtime but not financial support). The powderdiffractometerwas recently upgraded from 10 to 35 detectors. It can be used in two modes: high intensity or high resolution. Proposals can be sent in at any time of the year. For more information send an e-mail message to **Roland Tellgren** at roland.tellgren@kemi.uu.se (or use the address on page 12).

MEETING REPORTS

Keeping up-to-date on the many advances in powder diffraction instrumentation, data analysis and application is a difficult task. Just how active the powder diffraction community is, can be seen in the number of workshops, symposia and conferences that are devoted to the subject *each* year. To give our readers an impression of the current interests in powder diffraction, reports from various meetings, many of which were sponsored and/or organized by the CPD, are given below.

Powder Diffraction Course
Merida, Venezuela
May 13-18,1996

This course was held at the Centro Nacional de Difraccion de Rayos-X (Universidad de Los Andes), in Merida Venezuela. The invited lecturers were Prof. Robert L. Snyder of Ohio State University, Dr. Daniel Louer and Dr. Michele Louer of Universite de Rennes, and Dr. Rodolfo Vargas of IVIC. Profs. Graciela Diaz de Delgado, Miguel Delgado, Gerzon Delgado and Jose Antonio Henao, members of the CNDR-X, also participated as instructors.

The course began with general reviews of the fundamentals of X-Ray diffraction, sample preparation, data acquisition and qualitative analysis. In the more advanced part of the course, the emphasis was placed in the use of computer-based methods of data analysis for qualitative as well as quantitative phase analysis. Special attention was paid to crystal structure and microstructure analysis that can now be done even with conventional laboratory X-rays. The discussion on ***ab initio*** structure determination from powder data presented by Dr, Daniel Louer was particularly interesting. The lectures were conducted in the mornings, and the practical sessions in the afternoons. During the practical sessions, several PC computer programs dealing with the different aspects of powder X-ray diffraction, from indexing to the Rietveld method, were used. The number of participants was limited to 30 in order to ensure full access to the computing facilities of the CNDR-X and adequate supervision by the instructors during the practical sessions. Only graduate students and young scientist from Venezuela, Colombia and Cuba could be accomodated, although a number of requests fromother Latin American countries were received.

The CNDR-X organizes this type of course (given in even years), and one on single crystal X-Ray diffraction (given in odd years) to improve the level of crystallography in Venezuela and some of the nearby Latin American countries. Both coursesare given by internationally recognized lecturers covering both theoretical and practical aspects of modem X-ray diffraction techniques.

For more information on the activities of the CNDR-X, contact
Prof. Miguel Delgado, CNDR-X, Universidad de Los Andes, Facultad de Ciencias, Apdo. Postal 40, La Hechicera - Merida 5101, Venezuela, Phone: 58 74 401372, FAX: 58 74 401286, e-mail: cndrx@ciens.ula.ve

Materials Structure Characterization Techniques
Stark Hamry, Czech Republic
June 24-28,1996

In all, 32 oral contributions and 24posters were presented at this colloquium, organized by the Czech and Slovak Crystallographic Association (CSCA). A number of the papers were directly concerned with powder diffraction techniques: "Principles of Some Modem Deconvolution Methods" (M. Cemansky), "Diffraction Tensometric Characterization of Non-Conventional Methods of Materials Separation" (N. Ganev, 1. Kraus), "Application of X-ray Diffraction in Medicine" (D. Krausova), "X-ray Study of Chemical Reactions" (L. Benes et al.), "X-ray Diffraction Study of Silver Films Deposited by R.F. Reactive Sputtering" (P. Sutta et al.), "Notes on the Methods and Interpretation of Phase and Structure Analysis of Hydroxylapatite" (A. Buchal) and "X-ray Diffraction Profile Analysis for Polymer Mixtures" (M. Horky, J. Baldrian).

A number of other contributions also caught the interest of the participants: "Possibilities of Phase Analysis via the Interpretation of Electron Diffraction Patterns: (Z. Novy), "Possibilities of Modem Transmission Microscopy" (A. Orlova), "LEED in Surface Crystallography" (I. Bartos), "Use of Ion Back Scattering for Study of Crystal and Real Structure of Materials" (D. Machajdik), "Application of NMR Spectroscopy for Study of 3D-Structure of Biopolymers (V. Sklenar) and "X-ray Crystallography in the Czech Pharmaceutical Industry" (B. Kratochvil). Proceedings of the colloquium, edited by P. Kuzel, were published as two separate issues of the 3rd volume of the journal *Materials Structure in Chemistly, Biology, Physics and Technology* **(280 p**ages) and distributed to the participants upon registration.

The scientific instruments exhibition, the annual meeting of CSCA members, a trip to the surrounding Beskydy mountains, and a farewell party completed the program, which was enjoyed by approximately 80 participants (mostly from the Czech and Slovak republics). Both the organizing committee (chaired by D. Krausova and J. Kamenicek) and the program committee (chaired by J. Hasek and R. Kuzel) were congratulated for their efforts.

Jaroslav Fiala

CALL FOR CONTRIBUTIONS TO THE NEXT CPD NEWSLETTER

The next issue of the CPD Newsletter will be edited by Dr. Rob Delhez to appear in April of 1996. He would greatly appreciate contributions from readers on matters of interest to the powder diffraction community, e.g. meeting reports, future meetings, developments in instruments, techniques and computer programs and news of general interest. Please send articles and suggestions directly to **him** (address is given on page **12).**

Lynne McCusker, Editor, CPD Newsletter 17

45th Annual Denver X-ray Conference
Denver, Colorado, USA
August 3-8, 1996

Diffraction Peak Profile Analysis

The fist part of this CPD session was dedicated to the recent developments in *Model-based interpretation of diffraction line profiles*. Outstanding contributions were presented by three invited speakers who emphasized the interpretation of three major effects in diffraction line broadening: *the case of lattice distortion* by Pr E.J. Mittemeijer (Delft), *the determination of crystallite size and shape* by Dr J.I. Langford (Birmingham) and *strain broadening caused by dislocations* Pr T. Ungar (Budapest). The complementarity of the three talks was particularly appreciated. In the second part of the session, six oral presentations covered different aspects of diffraction line broadening, including the use of a probabilistic method for deconvolution (J. Cline, Gaithersburg), a convolution-fitting approach for the evaluation of strain broadening (R.W. Cheary, Sydney), a microstructural study of natural samples (G. Berti, Pisa), diffraction line-profile shape by synchrotron and laboratory X-ray sources (D. Balzar, Boulder) and applications of line broadening analysis (P. Quintana, Mexico; W.N. Weins, Lincoln).

Daniel Louer

Phase Quantification

The CPD organised several Special Sessions and Workshops at the Combined Powder Diffraction Satellite Meeting and **45th** Denver X-ray Conference in August, **1996.** Two of these focused on quantitative phase analysis using powder diffraction data.

The fist was a Workshop on *Quantitative Techniques and Errors in XRPD,* co-chaired and co-organised by Prof R.L. Snyder of Ohio State University and Dr R.J. Hill (CPD) of CSIRO Minerals, Melbourne, Australia. Bob Snyder started proceedings with an elegant and comprehensive description of the origins and mathematical derivation of quantitative phase analysis using the traditional reference intensity ratio method since its initial definition by Visser and de Wolff in 1966**.** This was followed by Rod Hill, who worked through the history and development of the full-pattern database and Rietveld methods, with an emphasis on their practical use and relative advantages in a variety of complex analytical situations. Giora Kimmel (NRCN, Beer Sheva, Israel) then described some of the pitfalls of phase analysis using single-peak methods, Dirk Reefman (Philips Research Lab., The Netherlands), described the relationship between scale factors and the ODF in the phase analysis of textured materials, and Jeff Dann (Osram Sylvania Inc, Pennsylvania), presented an eye-opening paper on the use of zone control charts and 'run rules' for the determination of natural variation and outlier results in process analysis and control. Dave Bish (LANL, Los

Alamos, USA) presented a review of recent work on the analysis of materials that lack perfect 3D order and warried of the dangers of using simple diffraction models for the analysis of clay minerals and stacking-disordered materials. The workshop concluded with Julius Schneider (Univ. Munich, Germany), who gave an exhaustive review of the wide variety of powder diffraction software packages that are now available in the public domain for phase analysis; this talk was interspersed with many comments and advice from the authors of the programs which was of great interest to members of the audience trying to wade their way through the many options.

The second CPD contribution was a Special Session on ***Phase Quantification,*** co-organised by Prof D.K. Smith (CPD) of Pennsylvania State University and Dr D.L. Bish of LANL, Los Alamos, New Mexico, and co-chaired by Dr Bish and Dr J. Schneider of Munich University, Germany. This session began with a review of the Rietveld and database methods of full-pattern phase analysis by Rod Hill, who demonstrated that the two methods were rapidly coming into closer alignment through (i) the use of observed patterns in Rietveld analysis as an adjunct to the calculated patterns normally utilised, and (ii) the increased co-option of calculated patterns (with peak modelling) in the databese method to complement the observed patterns usually used. Julius Schneider then reviewed the software now available for phase analysis, as in his workshop presentation above. G.E. Braun (BJ Services Co., Texas) provided an interesting description of the use of quadratic goal programming applied to chemical and X-ray powder data, and James Cline (NIST, Maryland) comprehensively brought us up to date with the pitfalls and inaccuracies in the measurement of amorphous content. Jim successfully convinced us that we are far more confident than we should be about these techniques! Then followed presentations by J.P. Nicolich (Technical Univ, Darmstadt) on the analysis of silicon nitride and silicon carbide mixtures, G.A. Cooke (Solutions Int. Inc., Pennsylvania) on practical applications of PD for phase analysis, and A.R. Hölzel (Ober-Olm, Germany) on modifications to the PDF-2 for searched using chemical analysis data.

All-in-all, a very productive, enjoyable and worthwhile two sessions that substantially enhanced our knowledge of quantitative phase analysis.

Rod Hill

New Developments in Detectors and Other Instrumentation

This session, chaired by H. Toraya (Nagoya Inst. Tech.) and J. I. Langford (Birmingham Univ.), consisted of 10 oral (two invited) and 3 poster presentations and attracted many participants.

The session started with a presentation by D.J. Cookson (Australian Nucl. Sci. Tech. Org.) reviewing recent results of synchrotron radiation powder diffraction experiments using imaging plates on the Australian beam-line at the Photon Factory. M. Shah (Princeton Instrum. Inc.) discussed the performance of high-resolution CCD camera systems for synchrotron radiation and laboratory sources. S. A. Belmonte (Univ. Edinburgh) spoke about the application of a two-dimensional detector using an imaging plate to the analyses of micro-structural effects in powders. A. N. Fitch **(ESRF)** described the optics and diffractometer designs of the high-resolution powder diffraction station on a bending magnet beam-line at the ESRF, and J. L. Hodeau (ESRF) spoke about the nine-crystal multiple detector system installed on that diffractometer. H. Toraya described the latest results obtained with the multiple-detector system at the Photon Factory. H. F. Poulsen (Riso Nat. Lab.) dealt with the nondestructive local texture study using high energy (60-300keV) synchrotron radiation and a two-dimensional detector. B. H. Toby (NIST) discussed the optimization of the instrumental resolution of a neutron powder diffractometer with three different monochromators at NIST for various types of experiments. R. D. Deslattes (NIST) discribed the diffractometer system equipped with graded multilayer collimators and long parallel-slits to be used for the accurate measurement of unit-cell parameters. C. A. MacDonald (Univ. Albany) discussed the diffraction applications of polycapillary x-ray optics.

About half of the topics were concerned with synchrotron radiation, and the another half with two-dimensional detectors of both CCD cameras and imaging plates. A quarter of the topics were on parallel-beam optics installed on laboratory systems. Overall, the session topics were of a high standard and provided informative views on recent developments in powder diffraction instrumentation.

Hideo Toraya

Thin Films and Multilayers

The steadily-growing interest in thin films and multilayers was reflected not only in the two sessions on this topic but also in the theme chosen for the plenary session, which was devoted to grazing-incidence x-ray analysis. About 20% of the invited and submitted papers fell into these categories, and it is clear that both synchrotron-radiation sources and the development of parallel-beam graded multilayer optics are having a major impact in this area. As emphasized in the three plenary talks by T. Huang (IBM), H. Goebel (Siemens) and D. de Boer (Philips), specular and diffuse x-ray reflectometry is a powerful tool for the determination of layer thickness, density and interfacial roughness, for example, while grazing-incidence diffraction is a valuable technique for the analysis of epitaxial films and depth-profiling of residual stress and strain. The latter topic was the subject of several papers dealing with a variety of technogically-important materials suchas wear-resistant, thermal-barrier and metallized coatings, and optoelectronic thin films. In the two oral sessions on thin fimls and multilayers, very interesting and topical invited talks were given by V. Valvoda (Charles U.), who described the analysis of magnetoresistive Ag/NiFe multilayers by a combination of low-angle and high-angle x-ray diffraction, by U. Pietsch (U. Potsdam) on the domain structure of Langmuir- Blodgett multilayers prepared from fatty-acid salts using both x-ray and neutron

scattering techniques, and by H. Wenk (Berkeley) on the quantitative characterization of texture in thin f i s of a variety of materials, including superconducting YBCO deposited on several substrates. Other talks in the session dealt with the characterization of a number of metallic multilayers such as Fe/Ir, CuNi and Co/Cu, and grazing-incidence measurements on electronic materials and wear-resistant coatings. Overall, these sessions provided a thorough overview of the rapid progress and increasingly sophisticated techniques and methods of data analysis in this important area of materials research.

David Cox

Precision and accuracy in structure refinement from powder data

The cause of precision and accuracy in the field was substantially uplifted in this session by presentations of both theory and experiment. State of the art theory and the recommendations set forth in the report on Statistical Descriptors in Crystallography (see http://www.unige.ch /crystal/astat/preface.html) were presented and their relevance to powder diffraction pointed out by James A. Kaduk (Amoco). He made clear the logic of replacing 'standard deviation' by the term 'standard uncertainty', the underlying mathematics, and the roles of the Bayesian and frequentist approaches. Turning to experiment, he first focused on the quartz in Rietveld refinements (RR) of 26 zeolite samples with quartz as an internal standard. After having first determined that the errors were, essentially, normally distributed, he determined the best values of the a and *c* lattice parameters of quartz to be **4.4138(8)** and 5.4054(4) Å, respectively. Again with attention to the normal distribution of errors, he compared the 26 sets of RR results for bond distances and angles with 20 single crystal sets in the ICSD (Inorganic Crystal Structure Database from Fachinformationszentrum, Karlsruhe). He concluded that one can reasonably expect a precision and accuracy of **0.01** Å for bond distances and 0.5" for bond angles in well done Rietveld refinements of small structures based on laboratory X-ray data. He then posed the provocative question of the physical meaning of measuring a bond length to such accurasy when the r.m.s. vibrational amplitude is ten times that.

A novel assessment of accuracy was presented by R. B. Von Dreele Los Alamos National Laboratory). Using (i) multiple data sets from different settings of the same calcite powder sample beset by preferred orientation, (ii) the simultaneous refinement capability of the GSAS program and (iii) his new preferred orientation model involving spherical harmonics, he showed first that the structural results were in excellent agreement with single crystal results, i.e., his preferred orientation model served very well in spite of the high degree of preferred orientation. His procedure produces a result akin to an inverse pole figure. The direct pole figure derived from it was in excellent agreement with a pole figure prepared in the standard way with rotating samples.

W. I. F. David (ISIS, Daresbury) discussed strategies for data collection which improve the precision and accuracy of the Rietveld refinement results. He first noted with approval the approach recently advocated by Hill and Madsen of increasing counting times at high scattering angles to reduce the 'impact and overbearing importance of the first few Bragg peaks' by compensating for x-ray scattering factor fall off and atom displacement (thermal vibration') effects on intensities. He then pointed out that one can go beyond that to determining, in advance of f i i data collection, which points/regions in the powder pattern are most sensitive to the structural parameters of interest and which are the least useful for the purpose. Describing these as 'leverage' concepts and advocating their use in structure refinements with powder diffraction data, David noted that they are well known in the single crystal area.

D. E. Cox (NSLS, BNL) shared important portions of the rich lode of lore he has built up during his many years of concern with questions of precision and accuracy in RR based on neutron and X-ray data from various sources. Comments such as 'beware the effect of state of ionization on the refined site occupancies' and 'the intensity effect of absorption in a cylindrical specimen has a two-theta dependence similar to that of temperature factors [random atom displacements] if the absorption is small and similar to that of the scattering factors if the absorption is large' are near homilies which bear repeating. For indicators of the quality of the structural results, Cox advocates the use of a weighted residual, wR_I, based on the Bragg intensities and a goodness-of-fit, S_I, based on them rather than the currently most used quantities, wR_P and **S,** based on the point by point intensities. He pointed out that this goodness-of-fit, S_I, yields more realistic values for the 'estimated standard deviations' than does the **S** based on counting statistics at each measured point. To get the best values of the integrated intensities with which to work, he recommends pattern decomposition by the Le Bail method followed by RR with the profile parameters fixed at those found in the Le Bail extraction.

In their paper on structural peculiarities of phase transitions in lithium-doped $KTaO_3$, **S.A.** Ivanov and V.V. Zhurov of Moscow with H. Rundlof and R. Tellgren of Sweden gave some examples of the need for high precision in order to determine the important small differences that lead to large effects.

A different, but useful, kind of assessment of precision and accuracy is one that tests the computer programs themselves. The last two papers in the session presented a collection of test data sets attributed to L. D. Calvert. These test a powder-pattern calculation program by presenting structural data for the same structures (e.g. Mg and Na) as successively reset in lower symmetry. All point groups and about 80 space groups are covered by these test data sets. These two papers were authored by a group whose special scientific bond in this case is their volunteer work on behalf of the International Center for Diffraction Data: L. D. Calved, P. L. Wallace, T. C. Huang, James A. Kaduk, J. N. Dann, M.H. Mueller and **A.C.** Roberts.

R.A. Young

† **now *deceased***

Modern Powder Diffraction in Material Science

Keynote lecture by Daniel Louer

The IUCr Commission on Powder Diffraction and the Seattle programme committee gave Daniel Louer one of the most difficult tasks of the congress when they asked him to summarise the history and current developments in powder diffraction for a general audience. Daniel led us through the last one hundred years from the discovery of X-rays in 1895 to the application of X-ray and neutron diffraction to the solution of crystal structure from powder diffraction data. On this tour, notable stops included the development of the Debye-Scherrer-Hull technique, the application of Fourier methods by Warren, the work of the late Arthur Wilson throughout the 1950s to the 1960s when Hugo Rietveld published his seminal work on total pattern fitting. The powder method really began to take a new direction from here. Developments in autoindexing and much better sources of radiation, such as synchrotrons and neutrons, enabled crystal structures to be solved ab-*initio* from the powder diffraction data Daniel discussed the growth of this enterprise over the last 20 years, and showed that the solution of structures from good quality powder data, although never routine, is quite possible.

Daniel emphasised the impact of the technique in materials science, and presented examples ranging through high T_c superconductors, microporous materials, fullerenes, pharmaceuticals, electrolytes, non ambient studies for studying reaction kinetics and nanocrystalline materials.

The power of synchrotron radiation was illustrated by a 2D image from Nelmes and McMahon on InSb at 5.1 GPa. High energy radiation near the In edge enabled both anomalous enhancement of certain reflections as well as a 30 degree angular spread of data from the pressure cell. Sufficient data have been extracted from this and other samples to be able to identify new phases and also to be able to refine high resolution structures at elevated pressures.

An example of the work of Chabre and Pannetier during the electrochemicalreduction of γ-MnO_2 demonstrated how monochromatic neutron diffraction can follow the path of a reaction ending with pyrochroite and groutite. The complimentary nature of X-ray and neutrons was also mentioned with reference to quantitative phase analysis, combined refmement of data sets, and accurasy of co-ordinates.

One of the most exciting developments in phase identification is the use of whole patterns rather than lists of d spacings and approximate intensities. There is a very high success rate with this method even when a significant amount of preferred orientation is present.

In addition to better methods of data collection and better sources, the technique of structure solution from powder data has benefited from many advances in data analysis. These include improvements in direct methods optimized for powders, improved and sharpened Patterson maps, maximum entropy methods, Fourier recycling, simulated annealing, Monte Carlo methods and atom-atom potential methods. Examples of structure determination included C_{60} $Br_{24}(Br_2)_2$ and a new titanosilicate with 5-coordinated Ti published recently in ***Nature***. The use of Monte Carlo methods was illustrated with reference to p-methoxy-benzoic acid. Over 4000 iterations produced several distinct minima, and the lowest of these yielded the correct solution.

Even with the significant advances made over the last 20 years, the powder method is being pushed still further. The limits of data collection sensitivity have measured the first purely magnetic peak from a powdered sample of UO, using synchrotron radiation. Combined X-ray and neutron analyses are improving structural models including the location of hydrogen atoms. In addition to intensity data, peak shape analysis is being pushed to its limits. Stacking faults, sequences, dislocations, particle sizes and shapes can all be obtained with data of sufficiently good quality.

Daniel ended by referring to a recent and very comprehensive review of powder diffraction (Langford and Louer) which gives an insight into how far the subject has progressed in a very short time. Although Daniel's given task was really impossible in one hour, he gave an excellent and very lively account of the renaissance in the subject and totally held the attention of his audience for the duration of this lively and very informative presentation. Students and people new to the field cannot fail to have been impressed by the progress the subject has made and by the potential of this rapidly growing field.

Bob Cernik

Materials VIII - Powder Diffraction

Chairpersons: R.J. Hill and J.B. Cohen

This Microsymposium was organized by the IUCr Commission on Powder Diffraction and consisted of four invitedlong talks together with four short talks selected from the submitted abstracts. The contributors were from six countries, and their lectures spanned topics from limits on precision and accursy, to data collection protocols, uses of magnetic neutron diffraction and anomalous X-ray dispersion, characterisation of multilayers, depth profiling, and the determination of polymer electrolyte structures. A highlight was the presentation by Bill David of the RAL, UK, who described the achievement of markedly superior fits of observed and calculated diffraction profiles in Rietveld refinement through the use of independent peak shape and width parameters. The method very effectively deals with the issue of anisotropic peak characteristics and allows the determination of structural results that are independent of (and therefore unbiased by) the peak shape model. Evidence for the 'saturation' of ead's at long counting times was provided, along with a strong

Grants-in-Aid from the International Centre for Diffraction Data

"Let us help each other"

The ICDD (International Centre for Diffraction Data) needs more high quality powder difraction patterns to add to its database, the PDF, which is used worldwide. Perhaps you could use a little additional financing. If you had that small financial help, could you produce some high quality X-ray powder diffraction patterns, in the needed format, for materials not now represented in the PDF? Then you might be interested in applying for an ICDD Grand-in-Aid.

The ICDD offers Grants-in-Aid which can be used most effectively as supplements to existing funded projects involving the preparation and powder XRD characterization of new materials. Grant-in-Aid proposals will be considered, on a competitive basis, from any qualified investigator (academic, government, or industry), anywhere in the world, who *can* demonstrate expertise in the preparation of high quality powder diffraction patterns. Proposals addressing current opportunities to extend and improve the usefulness of the PDF are given highest priority.

The duration of a Grant-in-Aid is 12 months. Renewal for additional 12-month periods can be considered, again on a competitive basis, if progress has been satisfactory. Grant recipients are required to submit biannual progress reports. The powder diffraction pattern data should be submitted in PDF-ready format.

Deadlines for receipt of Grand-in-Aid proposals at ICDD Headquarters are 31 December, 1996, 31 July 1997 and then 31 January and 31 July thereafter. Detailed guidelines for the proposals and proposal forms are available from Ms. Therese Mauchline, International Centre for Diffraction Data, 12 Campus Blvd., Newtown Square, PA 19073-3273, USA (Tel: (610) 325 9814, Fax: (610) 325 9823, E-mail: Mauchline@ICDD.com)

recommendation for the use of variable step counting time protocols during data collection. **A** method of decoupling the structure and peak contributions was described by Dave Cox of BNL, USA, this time through the fixing of peak parameters at the values determined during a pre-Rietveld LeBail-type refinement. Mikhail Kovalchuk of the Russian Academy of Sciences, Moscow, presented a comprehensive description of the use of X-ray standing waves as a sensitive probe for the characterisation of multilayers on single crystal surfaces, while Francoise Bouree of CEA-CNRS Saclay gave a comprehensive description of the key steps in the determination of magnetic structures from powder neutron diffraction data, Philip Lightfoot of St Andrews, UK, presented an elegant and visually stunning description of the *ab initio* determination of the crystal structures of polymeric materials through the use of Monte Car1o and constrained Rietveld refinement methods.

Rod Hill

Structure Determination using Powder Data

Chairpersons: A. Clearfield and W.I.F. David

The contributions to this session covered a wide range of applications from supramolecular structures to solutions from limited datasets. R.E. Dinnebier, University of Bayreuth, showed that very complicated organic and organometallic structures could be solved, and that the newly proposed pseudo-atom method proved to be a very efficient tool for solving structures containing well defined molecular fragments. D.M. Poojary and A. Clearfield, Texas A&M University, demonstrated that Cu Kα radiation in combination with direct and heavy atom methods can be very successful even when the powder samples are poorly crystalline with diffraction peak intensities falling off very rapidly.

The presentation of the new features in the EXTRA program by C. Giacovazzo and his team was another contribution which aroused great interest among the participants. EXTRA is now incorporated in the latest SIRPOW versions and is able to take information from Patterson maps, pseudo translational symmetry, preferred orientation and molecular fragments. For those who are interested, there will be a workshop on this matter in December '96 in Bari.

Roland Tellgren

5th Italian School on Diffraction from Polycrystalline Materials

Frascati (Rome), Italy

October 2-5,1996

The school was organised by G. Cappuccio and M.L. Terranova, and was sponsored by the Italian Association of Crystallography, the National Council of Research, the National Institute of Nuclear Physics, University of Rome 'Tor Vergata', Ital Structures, Philips analytical, Rich Seifert, Siars, Sistec and Web Power.

The aim of this school series is to promote the use of modem diffraction techniques, with special emphasis on the characterisation of polycrystalline materials. Previous schools have been held in Modena (1991), Pisa (1992), Trento (1994) and Gargnano (1995). The subject of the 1996 school was 'Thin film characterisation by advanced X-ray diffraction techniques'.

The introductory lectures on basic X-ray diffraction theory were presented by C. Giacovazzo, then V. Valvoda, P. Scardi, M. Leoni, B. Gilles, G.A. Battiston, P. Imperatori, G. Cappuccio, **S.I.** Zheludeva, **S.** Lagomarsino and **S.** Di Fonzo lectured on specific methodologies for the study of thin films. A. Balerna and R.J. Cernik gave lectures on synchrotron radiation application to powder diffraction and news from the facilities in Daresbury and Grenoble. Further contributions were presented by G. Berti (status of

the European project on standardisation of XRD techniques), A. Morone (Pulsed Laser Ablation deposition of thin films), C. Veroli (open discussion on XRD experimental methods) and A. Haase (XRD optics).

There were about 50 participants, most of them from Italy but some from other European countries. All lectures were delivered in English. The proceedings (also in English), edited by G. Cappuccio and M.L. Terranova, include all lectures and have been published as a special issue of the INFN-LNF (Frascati National Laboratories) periodical publications LNF- 96/049 (IR)). Copies can be obtained from: SIS - Pubblicazioni Laboratori Nazionali di Frascati, P.O. Box 13, I-00044, Frascati, Italy.

The next (6th) school will be held in Bari, presumably in the Spring of 1998. The subject of that school, organised by C. Giacovazzo and co-workers, will be *'Ab initio and* Rietveld techniques', and will include a tutorial on quantitative phase analysis. As was the case for this 5th school, the CPD will also endorse the 1998 school.

Paolo Scardi

International Conference on Neutron Scattering (ICNS 97)
Toronto, Canada, August 17-21,1997

As a member of the Program Committee for the forthcoming International Conference on Neutron Scattering, I would like to encourage powder diffractionists who use neutrons to submit abstracts for this meeting. The scope will include many topics of interest to the powder community, such as magnetism, structures (including non-crystalline systems), chemistry (including surfaces and catalysis), soft materials (including biological systems), industrial applications, and new instruments and techniques (including data analysis). The deadline for abstracts is April 15th, 1997. To receive further announcements, send your name, address, fax number and e-mail address to the address given below. I hope that powder diffraction will be strongly represented at this meeting, and I would be particularly grateful to receive suggestions for sessions and/or invited speakers.
D.E. Cox (Brookhaven National Laboratory; E-mail: **cox@bnlx7a.nsls.bnl.gov;** Fax: 516-344-2739)

NEWS FROM ICDD

The International Centre for Diffraction Data is pleased to announce its new web site at www.icdd.com. The site contains links to many other useful diffraction and diffraction-related sites. Visit us on the web for the latest information about ICDD's products, educational activities and Grants-in-Aid program. We hope to expand this cite to be a central meeting place for all powder diffraction related activities. Please contact Bob Snyder with any suggestions or requests for use. We also propose to use this cite to minor any other PD sites to speed web access during prime time.

The ICDD announced the release of Set 46 annual products in September. The Sets 1-46 PDF-2 database contain 63,557 entries. Among these entries are 18,747 organic, 46,035 inorganic, 158 explosives, 265 superconducting materials, 787 zeolite, 3,638 forensic, and 12,150 metals & alloys entries. For more information regarding ICDD products, please contact the Business Manager (e-mail: info@icdd.com).

The International Centre is holding a Grants Workshop on November 11 and 12 in Prague. Approximately twenty-five scientists are expected to attend this Grants Workshop. Since the Prague workshop was over subscribed, the ICDD is planning to hold another workshop in Spring 1997 in England. For more information about the ICDD Grants-in-Aid program or upcoming Grants Workshops, please contact the Data Acquisitions Manager at ICDD (e-mail: Mauchline@ICDD.com).

Bob Snyder

WHAT'S ON

4-6 March 1997
Structure and Properties of Crystalline Materials, Dubna, Russia
Contact: Dr. V.V. Sikolenko, E-mail: sikolen@nf.jinr.ru

10-12 March 1997
***5.* Jahrestagung der Deutschen Gesellschaft für** Kristallographie, Hamburg, Germany.
Contact: Frau I. Bauer, Mineralogisch-Petrographisches Institut, Universität Hamburg, D-20146 Hamburg, Germany; Fax: +49(40) 4123 2422
E-mail: DGK97@mineralogie.uni-hamburg.de
WWW: http://www.rrz.uni-hamburg.de/mpi/DGK97/

14-18 April 1997
British Crystallographic Association Annual Conference(BCA 97) Leeds, UK.
Contact: Dr. M. Thomton-Pett, School of Chemistry, University of Leeds, **Leeds** LS2 9JT, UK
Fax:+44 (113) 233 6565
E-mail: marktp@chem.leeds.ac.uk
www:http://www.chem.leeds.ac.uk./conferences/BCA97/

25-28 May 1997
5th European Powder Diffraction Conference (EPDIC V), Parma, Italy.
Contact: Prof. G. Artioli, Dipartimento di **Scienze della Terra,** Universita di Milano, Via Botticelli 23, 1-20133 Milano, Italy; Fax: +39 (2706) 38681
E-mail:artioli@iunmix.terra.unimi.it
WWW: **http:/liummix.terra.un~i.it/-epdic/epdic5.html.**

4-8 August 1997
46th Annual Denver X-ray Conference, Steamboat Springs, Colorado, USA.
Contact Dr. Paul Predecki, Engineering Department, University of Denver, Denver, CO 80208, USA
E-mail: denxrcon@du.edu

17-21 August 1997
International Conference on Neutron Scattering (ICNS 97), Toronto, Canada.
Contact: Phyllis Green, Solid State Division, Oak Ridge Nat'l Laboratory, POB 2008, Oak Ridge, TN 37831-6033
Fax: 423-574-4143; E-mail: phg@oml.gov

24-28 August 1997
17th European Crystallographic Meeting (ECM-17), Lisbon, Portugal
Contact: ECM 17 Secretariat, Departamento de Engenharia Quimica, Instituto Superior Tecnico, Av. Rovisco Pais, 1096 Lisbon, Portugal; Fax: +351 (1) 442 1161
E-mail: qteresa@beta.ist.utl.ptor romao@itqb.unl.pt.
WWW:http://alfa.ist.utl.pt/-ecm-17/

31 August - 4 September 1997
17th Conference on Applied Crystallography, and *4-7 September 1997*
3rd Rietveld Summer School
Wisla-Jawomik, Hotel "Stok", Poland
Contact: Prof. H. Morawiec, Institute of Physics and Chemistry of Metals, University of Silesia, Bankowa 12, 40-007 Katowice, Poland; Tel/Fax: +48 (32) 59 69 29
E-mail: dana@usctouxl.cto.us.edu.pl

16-22 August 1998
18th European Crystallographic Meeting (ECM-18), Prague, Czechoslovakia
E-mail: hasek@imc.cas.cz

? 1998
6th European Powder Diffraction Conference (EPDIC-6), Budapest, Hungary
Contact: ?

4-13 August 1999
18th Congress and General Assembly of the International Union of Crystallography, Glasgow Scotland, UK
Contact: Dr. C. Gilmore, Department of Chemistry, University of Glasgow, Glasgow G12 8QQ, UK
Fax: +44 (41) 330 4888; E-mail: iucr99@chem.gla.ac.uk
WWW:http://www.chem.gla.ac.uk/iucr99/

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