COMMISSION ON POWDER DIFFRACTION

INTERNATIONAL UNION OF CRYSTALLOGRAPHY

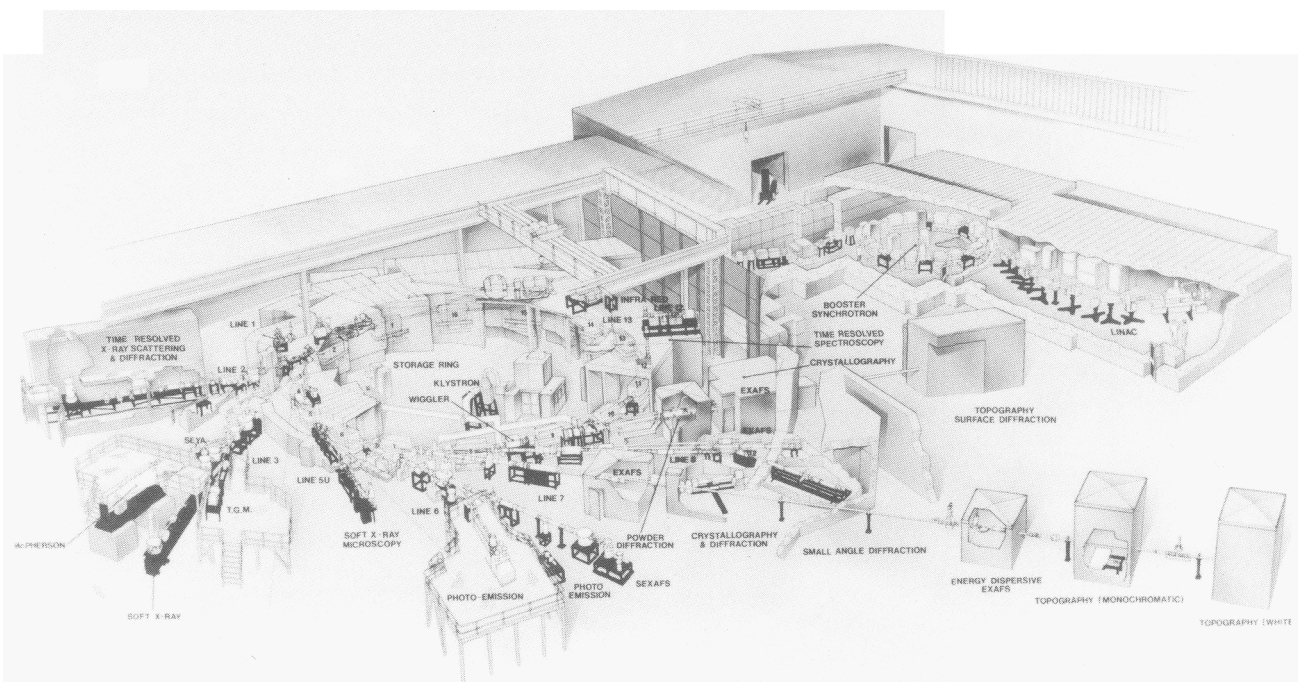
NEWSLETTER Nc. 5

October 1990

EDITORIAL

One aim of the CPD Newsletter is to inform the powder diffraction community of various aspects of the subject which are of topical interest, for example, features on PD facilities, notes of particularly important developments in instrumentation or technique and so on. In this issue Drs Bob Cemik and Phil Pattison highlight the pioneering work of the powder diffraction group at the Science and Engineering Council's Daresbury Laboratory synchrotron radiation source. This is followed by reports on recent ventures organised, sponsored or supported by the CPD. 1990 has been a particularly busy year for the Commission. Two microsymposia and a discussion session were arranged by the CPD at the XVth IUCr Congress at Bordeaux in July. Despite abnormally high temperatures - delegates consumed over 10,000 litres of mineral water during the Congress! - this first involvement of the CPD in the triennial IUCr conference attracted first rate speakers and was well supported. The Congress was preceded by the outstanding PD Satellite Meeting arranged by CPD at Toulouse, without doubt a milestone in the annals of PD. During August the CPD sponsored a Rietveld Method Summer School for beginners at Cieszyn, Poland, another successful venture, which was over-subscribed by a factor of two. The Chairman and Secretary contributed substantially to the International School on Powder Diffraction held in Cairo in January, at which an excellent introductory lecture was given by Professor Mario Nardelli, then President of the IUCr. The Rietveld Refinement Round Robin is nearing completion - a preliminary report was presented at the Toulouse meeting by Dr Rod Hill - and the book on the method, based on the proceedings of the 1989 Petten workshop, should soon be with the publishers. The first issue of the Information Exchange Bank for PD programs, compiled by Professor Deane Smith, will be available shortly. An update on these projects will appear in the next Newsletter.

The CPD was formed in 1987 at the XlVth General Assembly and had grown into a lusty infant by the time of the Bordeaux meeting. The 'founder members' ably led (goaded?) by the



Synchrotron Radiation Facilities at Daresbury Laboratory

Chairman, Professor Ray Young were, however, not allowed to rest on their laurels; all were re-elected at the XVth General Assembly for the current triennium when, hopefully, the CPD will skip adolescence and move directly to maturity. To further this progress, approval was given to increase the membership from 6 to 9 and the Commission welcomes Dr David Cox (USA), Dr Jaroslav Fiala (Czechoslovakia) and Dr Daniel Louer (France, previously a consultant to the Commission). Professor Deane Smith was re-appointed as a consultant and Dr Rod Hill has taken over the role of Secretary.

While the intention is to maintain a degree of uniformity in successive newsletters, each issue inevitably reflects the individual approach of the member whose turn it is to be editor and whose task would be made easier if there were some input from you, the PD community. Short and pertinent articles, news items, a note of 'what's on' in powder diffraction for your area, ***etc***, are always welcome. Why not let the CPD know if you approve or disagree with the style of the Newsletter - or, for that matter, with the Commission's activities and projects in general? Contributions can be sent the Chairman or Secretary, whose addresses are given at the end of each issue. Some 'feedback' would be welcome - if only for reassurance that somewhere 'out there' a **PD** community exists and is aware **of** the CPD's exertions on its behalf!

J. Ian Langford
Editor, Newsletter No. 5
School of Physics & Space Research
University of Birmingham

As you may have deduced, the CPD's first Secretary, Dr J.I. Langford, has handed over his secretaryship. The CPD take this opportunity to express their deep appreciation of Dr Langford's unflagging attention to defining and fulfilling the role of CPD secretary and of his Olympian efforts in it, which have served the CPD in stellar fashion during the first triennium of its existence. All this he did with grace and wisdom, even though he particularly wanted NOT to be the Secretary in the first place.

R.A. Young,
Chairman

POWDER DIFFRACTION WITH SYNCHROTRON RADIATION AT DARESBURY LABORATORY

The Synchrotron Radiation Source (SRS) at the Daresbury Laboratory in Cheshire, UK, is the largest dedicated synchrotron radiation facility in Europe. X-rays are produced when the 2 GeV electrons in the storage ring are deflected through the magnetic fields of the 1.2 Tesla bending magnets or the 5 Tesla insertion device (Wiggler). The synchrotron beam produced from these devices has a broad spectrum of X-rays, peaking round a critical wavelength of 3.98, for the bending magnets and 0.938, for the Wiggler. Electron beam lifetimes of the SRS are typically around 24 hours and initial currents of 200-300mA are achieved.

The excellent angular collimation of synchrotron radiation (about 0.2 mrad vertical divergence), combined with the tunability and high brightness of the source, make it ideal for powder diffraction. At the Daresbury SRS, powder diffractometers have been installed on three experimental stations. Two of these stations are situated on the existing Wiggler beamline, where both monochromatic (angle-dispersive) and white-beam (energy-dispersive) diffractometers are operational. In addition, a new high resolution diffractometer has recently been built and commissioned on a bending-magnet beamline (1).

The experimental arrangement for high resolution powder diffraction is the Debye-Scherrer geometry with a scanning counter placed behind either a single slit or multiple slits. Both spinning-capillary and flat-plate specimens can be accommodated in the single-slit geometry, while the multiple slit is only used for flat-plate samples. In the capillary mode only a few milligrams of sample are required. Full pattern data collection with good counting statistics can be achieved in about 6-8 hours per sample. A minimum resolution width in Debye-Scherrer geometry of $0.03°(2\theta)$ (FWHM) has been measured for a BaF_2 sample, with all peaks being fitted well with a Voigt or pseudo-Voigt function. Typical peak count-rates for the (111) reflection from the NBS604b silicon powder standard are about 3.5×10^4 counts per second with a FWHM of less than $0.05°(2\theta)$. Over an angular range 0 to $120°(2\theta)$ the deviation between nominal and measured positions of all observable Si powder peaks is less than ± 1 mdeg. Furnaces and cryostats are available, covering the temperature range 7K - 1300K.

In the energy-dispersive technique, the diffraction angle is kept fixed and the energy distribution of the scattered white beam is measured with a solid-state detector. Although the energy resolution of this detector limits the number of diffraction peaks which can be resolved, lattice constants for relatively high symmetry materials can be determined to within a few parts in 10^4. The main advantages of the energy -dispersive technique are the fixed angle (useful for sample environment chambers with small windows), the simultaneous measurement of the whole pattern and the rapid data collection time. The majority of the development work has concentrated on two areas of research: kinetic experiments and high pressure diffraction. Much of the kinetics work is concerned with temperature-dependent phase transitions between various crystallographic phases and from amorphous to crystalline states. In some cases other techniques, such as differential scanning calorimetry, are combined with the collection of X-ray diffraction data in order to provide a more nearly complete thermodynamic description of the reaction. In the high pressure studies, the very small beam cross section used in white-beam work, typically less than 200 microns in diameter, is ideal for using diamond anvil cells. Pressures in excess of 500 kbar can readily be achieved with this type of cell, and a wide

range of inorganic compounds have already been studied at the **SRS.** The next stage is to combine high temperature and pressure for phase diagram determination.

Improvements in the quality of the data from laboratory and synchrotron sources has stimulated much interest in the solution and refinement of crystal structures from high resolution powder patterns. In a recent study (2) of an organic material (Cimetidine, $C_{10}H_{16}N_6S$), using only data collected at the SRS, the structure was successfully solved with direct methods using integrated intensities obtained from pattern decomposition. The final agreement factors in the subsequent refinement were much improved by including the hydrogen atoms, which illustrates the potential of the technique. *Ab-initio* structure solution using combined neutron and synchrotron powder diffraction data also opens up **a** particularly fruitful field of research. An example which nicely highlights the power of the combined data approach was published recently (3). Indexing of the powder pattern measured on the high resolution neutron diffractometer at the Institut Laue-Langevin, Grenoble, was the first step. Further structural analysis was then possible with the use of synchrotron data from the SRS to determine the atomic positions of Na and C from direct methods. The full structure was then derived and refined from the neutron data by means of the Rietveld method.

An important advantage, in addition to high intensity and resolution, is that the tunability of the source opens up the possibility of using anomalous scattering techniques. These increase the sensitivity of the experiment to a specific atomic species, or even different valence states of the same species. The intensity contrast afforded by the wavelength dependence of the atomic scattering factor for X-rays is generally complemented by the isotopic variation of scattering lengths in neutron diffraction. The combination of synchrotron and neutron diffraction data in **a** joint refinement is an improved approach and this line of development is attracting interest worldwide. The use of isotopic substitution in the neutron case, and wavelength tuning with synchrotron radiation, gives an atomic site specific approach to crystallography and crystal chemistry.

Additional information on the facilities available at the SRS Daresbury Laboratory can be obtained from the authors. Beamtime allocations are made twice yearly after a peer review process for which the closing dates are March 31st and September 30th each year. Details of the application procedures can be obtained from the following address:

SRS User Liaison Office,
SERC Daresbury Laboratory.
Warrington WA4 4AD, UK
Tel: 0925 603000
Fax: 0925 603174

R.J. Cemik and P. Pattison
X-ray Diffraction Project
Daresbury Laboratory

References

(l) R.J. Cemik, P.K. Murray, P. Pattison & **A.N.** Fitch, J. *Appl. Cryst.* **23** (1990), 292-296.

(2) R.J. Cemik, A.K. Cheetham, C.K. Prout, D.J. Watkin, A.P. Wilkinson & B.T.M. Willis, to be published.

(3) E, Weiss, S. Corbelin, J.K. Cockcroft & A.N. Fitch, *Angew. Chem.* **102** (l990), 728-729.

Local organisers and Programme Committee of Powder Diffraction Satellite Meeting, Toulouse, and CPD members

IUCr POWDER DIFFRACTION SATELLITE MEETING, TOULOUSE, 16-19 JULY 1990

The meeting coincided with a number of celebratory events, most notably the 200th anniversary of the French revolution and the 'Tourde France', which was billedin themeeting information as 'an important cycle race'. Neither event could be ignored! The conference was held in the Paul Sabatier University, near the southern outskirts of Toulouse, and was preceded by a JCPDS/ ICDD workshop on modem search-match procedures. The chairman of the organising committee was Dr Jean Galy, Director of CEMES-LOE CNRS, and the programme committee was chaired by Dr Daniel Louer. Lectures were held in a largish air-conditioned auditorium with a hall area outside reserved for poster presentation, reception and refreshments.

The meeting was organised into five topics:

1) Powder diffraction accuracy, high resolution, standards
2) Powder pattern fitting
3) Structure determination and refinement
4) Time- and temperature-resolved powder diffraction
5) Phase identification and databases

The first session started with a general review by D.E. Cox (Brookhaven, USA) and this was followed by a strong British contingent–R.J. Cemik, J.I. Langfordand W.I.F. David. Dr Cox drew attention to the continual element of change in powder diffraction experimentation and that, "with seven orders of magnitude at our control, we should fully exploit the intensity and resolution possibilities of our experiments". The papers which followed illustrated these points with studies of barium fluoride, cimetidine, copper oxide, high Tc superconductors, zeolites and NIST (NBS) standards.

The session on powder pattern fitting started with a preliminary report by R.J. Hill on around robin on structure refinement by the Rietveld method. This involved 34 invitations (worldwide) to analyse data for $PbSO_4$, mono-ZrO, and the zeolite ZSM5. **As** is usual with round robins, an embarrassingly wide range of differing end conclusions is the outcome to date. This session continued as expected, with various descriptions of mathematical functions and physical models to handle powder diffraction peaks: stacking faults, textures (approaching the single crystal case), strain due to inclusions, plastic deformation, dislocation density and deformation energy figured amongst the fitted parameters.

The structure determination and refinement topic brought a variety of materials and methods. Le Bail quoted a structure determination for NiV_2O_6 involving 1175 reflections and Clearfield used Patterson maps to derive positions of Zr and P in layered ion-exchanged $Zr.Na/KH(PO_4)_2.nH_2O$ systems and direct methods for anewly synthesised zeolite. Anisotropic temperature factors and maximum entropy methods were also discussed. **A** particularly provocative talk on indexing was given by P.E. Werner, with advice on general indexing strategies. The high rate of unindexed patterns (typically –40%) in the Powder Diffraction File (PDF) was lamented and an objective was stated "that indexing should always be available except for the rarest and best defended cases"!

To have a topic on time- and temperature-resolved powder diffraction at this meeting was clearly an important milestone. J. Pannetier (ILL) reviewed this fast developing field in which the timescale for data collection has been reduced to the millisecond range. The Birkbeck trio – Tarling, Barnes and Hausermann – brought in a range of examples and techniques (with X-rays and neutrons) to illustrate this point. Other examples followed for systems involving hydration, high temperature transformations in zirconia and Bi-Sr-Ca-Cu oxides.

Participants of the Powder Diffraction Satellite Meeting at the Paul Sabatier University, Toulouse

The final session, on phase identification and databases, introduced some re-thinking on databases and analysis routines generally. One idea stated was to set up a distribution centre for powder diffraction programs. (Contact Deane K. Smith with any offers.) Another idea discussed at length was that of creating a database of complete powder patterns. The need to re-determine many of the old *(eg* 30 years or more) patterns, particularly oxides, was argued. A timely talk by R.J. Hill, on the half dozen or so most important problems in quantitative analysis using the Rietveld method, rounded off this session.

The clear impression from the meeting was that powder diffraction is still growing in importance and in its number of practitioners. With 255 participants registered, it was a somewhat more formal meeting than the previous one at Freemantle in 1987. Perhaps this is the inevitable price of a growing and successful subject.

Paul Barnes.

CPD MICROSYMPOSIA AT THE XVth IUCr CONGRESS, BORDEAUX, 19-28 JULY 1990

ADVANCES IN STRUCTURE DETERMINATION FROM POWDER X-RAY DIFFRACTION DATA

An open meeting of the CPD on Advances in Structure Determination from Powder X-ray Diffraction Data was held during the morning of Friday, 20 July. The microsymposium was well attended, with an audience of approximately 350, and featured recent progress and emerging methods in structure determination by powder diffraction.

The session was opened by Professor A.K.Cheetham (UK), chairman of the microsymposium, who gave a brief survey of recent results and new trends in *ab initio* structure determination and then introduced the six speakers. The experimental aspects of structure determination from X-ray powder data were covered by Dr.D.E.Cox (USA). The exceptional features of high resolution diffractometers with synchrotron sources - instrumental resolution functions with a minimum of 0.015-0.020" (2θ), well defined peak shapes and measurement of peak position with an accuracy of 0.002° - provide favourable conditions for indexing powder patterns and collecting a large number of integrated intensities for structure determination. Examples were described, including the application of anomalous scattering to probe cation distributions and oxidation states. In the next presentation, Dr.D.Louër (France) described a comparison between a structure determination from single crystal data and from powder data collected with a conventional diffractometer and monochromatic X-rays. The results obtained at each stage of the analysis were compared. The fundamental problem of the evaluation of the area of overlapping peaks was discussed by Dr.J.Jansen (The Netherlands), who spoke on intensity determination by using improved procedures for fitting and on direct methods.

New developments in techniques for structure determination from powder data were presented during the microsymposium. Dr C.J.Gilmore (UK) reported on the powerful maximum entropy method, applied to the phase problem in the case of incomplete datasets. Examples based on datasets with and without overlapping reflections were discussed. Professor C. Giacovazzo (Italy) gave an account of the application of direct and Patterson methods to limited datasets and also discussed the problem of overlapped peaks. Results obtained from the application of a new direct-methods package were reported. Dr J.M.Newsam (USA) spoke on the determination of the framework structures of zeolites and related materials by computer modelling. In this approach, the configuration of the framework tetrahedra species is optimized with respect to a 'cost' function based on constraints - distance, angles, coordination number, *etc* - by means of simulated annealing methods. The potential of this procedure was illustrated by examples and its application in complex cases was discussed.

In addition, 16 posters were displayed during the session of Saturday afternoon, 21 July. Of these half were directly devoted to examples of *ab initio* structure determination or related problems. Instrumental aspects, including time-resolved diffraction, fitting applications, and programs for powder diffraction analyses, were presented in the second half of the session.

D.Louër and A.K.Cheetham

POWDER DIFFRACTION STUDIES OF FIBROUS, POLYMERIC AND SIMILARLY IMPERFECTLY ORDERED STRUCTURES

One of the three co-chairmen, Professor R.A.Young, introduced the session, held on Sunday 22 July, by explaining that 'Powder Diffraction' included 'polycrystalline diffraction' and the contributions, about materials as disparate as silicates, surgical sutures and tennis-racket strings, reflected this wide range of interest. Common themes, running through many of the contributions, included the need to use all available information, from other experiments as well as utilizing the whole diffraction pattern, the calculation of scattering from proposed structures using computers and the advantages of synchrotron radiation.

Three of the papers used the Rietveld method. Andrews and Colquhoun studied the structure of poly (ether ethyl ketone), using the known structure of a 5-ring homologue to model that of a 7-ring, and refining this using the Rietveld method, assuming a profile shape. Gardner and Harlow applied the method to polyactide structures. Their starting model was derived from a single chain whose helix dimension and stereo-chemistry were consistent with known information.

Iannelli and Immirizi extended the method to handle the 2-dimensional diffraction from partially-oriented polycrystalline polymeric fibres. They used functions to describe the profile along the lines of constant 20 and in an orthogonal direction. Pearson VII functions were used successfully, but others, related to the microstructure, are also being investigated. Hall and Somashekar adopted a different approach to explain the profile shape for these materials, calculating that from models which

comprised a range of crystallite sizes and a paracrystalline lattice, and successfully comparing it with the experimental profiles.

Two other approaches to structure determination were also described. The meridional scattering from wholly aromatic copolyamides is aperiodic, and Blackwell and Schneider showed that this followed from the random sequence of comonomers. The disordering of the lamellar structure of silicates was discussed by Drits and Besson, who constructed models containing different types and content of defects and determined their diffraction patterns.

The program was varied and stimulating and, although the audience of about 90 looked a little sparse in the very large lecture theatre, it was appreciative and keen to join in discussion.

I.H.Hall

CPD MICROSYMPOSIA DISCUSSION SESSION

The CPD organised one of several Discussion Sessions at the Congress.

The theme was 'New Challenges for Powder Diffraction', with particular reference being made to the content of the CPD microsymposia posters. Short oral presentations were given by J.M.Newsam (USA) on *Large Unit Cell Materials,* J.I.Langford (UK) on *Disordered and Low Crystallinity Materials,* R.J,Nelmes (UK) on *Studies at High Pressure and Temperature,* H.Fuess (Germany) on *Studies with Small Samples and / or at Short Times,* A.W.Hewat (France) on *The Current Status of Instrumental Resolution,* A.N.Christensen (Denmark) on *The Maximum Number of Refinahle Parameters* and *G.* Will (Germany) on *The Relative Status of Rietveld and Pattern-Fitting Methods of Analysis.* The Session was moderated by R.J.HiI1.

Following the short invited contributions, the Session was thrown open for further discussion on the poster presentations not covered in the talks and on any other subject of interest. One particular point raised, both during this session and at other times during the Congress, was the problem of software propagation, some of it in the commercial area, and the ways in which due credit can be given to the original and/or current authors of the code. Other subjects of lively debate were the current status of powder pattern indexing programs, the real or perceived advantages of ultra-high resolution with synchrotron sources and the most efficient means of gaining access to the new generation of synchrotron and neutron beams.

R.J.HiII

SUMMER SCHOOL FOR BEGINNERS WITH THE RIETVELD METHOD

The Summer School for Beginners with the Rietveld Method was held 9- 11 August 1990 in Cieszyn, Poland, in facilities belonging to the Silesian University. Hugo Rietveld took part in the opening ceremony, to declare the School officially in session. The local organiser was Professor Bojarski. I was the academic organiser (Course Director). In so far as we know, this was the first-ever summer school or short course on the Rietveld method involving hands-on experience with the method on microcomputers (PCs).

There were some 58 registered. The majority were from Eastern Europe, but there were many from Western Europe *(eg* The Netherlands, West Germany, Italy, Spain). There were about twice as many applicants as could be accommodated.

The general plan of the School was one of lectures in the morning, with the afternoon being devoted to hands-on work with PC-type computers, on set Rietveld-method problems, plus tutorials. As Professor Bojarski and his staff had managed to make available I5 PCs of AT and XT style, most with coprocessors, about half the students could work comfortably on the computers while the other half went to the current tutorial session. The students were invited to make their own free copies, to take home with them, of the Rietveld-refinement program used in the School *(DBWS-9006PC,* release 13.7.90) A large number did.

The tutorial sessions were very popular, no doubt because they were handled by the team of A.K. 'Tony' Cheetham and Robert Von Dreele. The general, loose structure was that of selection of a particular topic ***(eg*** X rays *vis-a-vis* neutrons for Rietveld-method studies, or synchrotron X-rays and time-of-flight neutrons) to which Bob and Tony each spoke briefly and extemporaneously, followed by questions from and general discussion with the audience. The discussions could be far ranging and the questions were often of a very high calibre. (The 'students' all had substantial scientific background, most with PhDs.)

The number of lecturers was deliberately kept small, in keeping with the thought that it was a school, not a workshop. All but one of the lectures were given by the three senior lecturers (Cheetham, Von Dreele and Young), of whom the first named two are outstandingly able to give lucid, interesting and highly informative lectures perfectly geared to the task in hand. The fourth lecturer, Paul Attfield, assumed duties as *defacto* master of the practicals, making sure that appropriate problems were set and adequately presented to the students with all necessary files each day. He then became their principal individual advisor about how to run the problems.

The computer room was kept open after supper until 10 o'clock for the students to work more or less on their own, should they wish. They wished; there were always students in the room, often more than half the total group. We took this show of interest and enthusiasm as a mark of real success of the school.

R.A.Young

NEWS FROM THE JCPDS-INTERNATIONAL CENTRE FOR DIFFRACTION DATA

The officers and directors for 1990/91 are:

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The IUCr representative to the JCPDS-ICDD is Prof. R.A.Young.

CALENDAR: MEETINGS OF INTEREST TO POWDER DIFFRACTIONISTS

14-16 March 1991	First European Powder Diffraction Conference (EPDICI), Munich, Germany. (Dr H.E.Gobe1, Siemens AG, ZFE ME AMF12, Otto Hahn Ring 6, PO Box 830953, D-8000 München, Germany)
19-21 March 1991	JCPDS-ICDD Spring Technical Meetings and Annual Meeting, Concordville, Pennsylvania, USA (Josephine Felizzi, JCPDS-ICDD, 1601 Park Lane, Swarthmore, PA 19081-2389, USA)
25-28 March 1991	British Crystallographic Association Spring Meeting, Sheffield, UK (Dr A.J.Smith, Department of Chemistry, University of Sheffield, Sheffield S3 7HF, UK)
14-20 July 1991	14th International Conference on Synchrotron Radiation Instrumentation (SRI'91), Chester, UK (SRI'91 Conference Office, SERC Daresbury Laboratory, Warrington WA4 4AD, UK)
21-26 July 1991	ACA meeting, Toledo, Ohio, USA (Alan Pinkerton, Department of Chemistry, University of Toledo, OH 43606, USA)
12-16 August 1991	Pacific-International Congress on X-ray Analytical Methods (PICXAM), Honolulu, Hawaii Organized by the Australian X-ray Analytical Association, The Denver X-ray Conference and X-ray Analysis Group and the Japanese Society for Analytical Chemistry (Lynne Bonno, Department of Engineering, University of Denver, Denver, CO 80208, USA)
20-30 August 1991	13th European Crystallographic Meeting, Ljubljana, Yugoslavia (Prof. L. Golic, Department of Chemistry, **E.** Kardelj University, PO Box 537,61001 Ljubljana, Yugoslavia)
27-30 August 1991	International Conference on Neutron Scattering, Oxford, UK (Mrs M Sherwen, Rutherford Appleton Laboratory, Chilton, Didcot, Oxon OX11 OQX, UK)
2-3 September 1991	Synchrotron Radiation in Crystallography, Trieste, Italy (L.Randaccio, Dipartimento di Scienze Chimiche, Universitadi Trieste, Piazzale Europa 1, I-34127 Trieste, Italy)
18-29 September 1991	Summer School on Neutron Scattering, Oxford, UK (Prof. B.T.M.Willis, Chemical Crystallography Laboratory, 9 Parks Road, Oxford OX1 3PD, UK)

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My address has changed and is now as given below.

☐ Please add the name of the following person to the mailing list.

Name ..... Title .....

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Return to: Dr. R.J.Hill, Division of Mineral Chemistry, CSIRO, P.O.Box 124, Port Melbourne, Victoria 3207, Australia

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