

COMMISSION ON POWDER DIFFRACTION

INTERNATIONAL UNION OF CRYSTALLOGRAPHY

NEWSLETTER No 1

January 1988

ESTABLISHMENT OF THE NEW COMMISSION ON POWDER DIFFRACTION

At its 14th August 1987 meeting in Perth, Australia, the General Assembly (GA) of the IUCr established a Commission on Powder Diffraction (CPD). This is an action widely welcomed and considered by many to be long overdue.

In early 1986, the IUCr Executive Committee (EC) established an ad hoc committee to assess worldwide interest and, if it be sufficient, to prepare specific proposals for the GA to consider in determining whether to establish a CPD. The Terms of Reference proposed by the committee and the EC and accepted by the GA are:

- (i) To advise the IUCr in organizing or sponsoring meetings, schools and Congress sessions on powder diffraction and related subjects.
- (ii) To promote and coordinate scientific exchange between countries in the field of powder diffraction.
- (iii) To cooperate with other IUCr Commissions on matters concerning powder diffraction.
- (iv) To cooperate with other international bodies interested in powder diffraction and allied subjects.
- (v) To promote useful interactions of the IUCr with the large world-wide body of X-ray and neutron powder diffractionists.
- (vi) To promote the scientific growth and development of the field of powder diffraction.

Since the members of the ad hoc committee became the initial members of the Commission, they were able to meet three times during the Congress to make plans for CPD projects. Among those being given first consideration are:

- * Program exchange "bank"
- * Satellite meeting for the 1990 Bordeaux IUCr Congress
- * Sessions on powder diffraction at the Bordeaux Congress
- * Workshop on Rietveld refinement (to be held in May or June 1989)
- * Newsletter

- * Round-robin survey of the Rietveld method involving X-ray and neutron data and several samples
- * New book(s), possibly resulting from workshop(s)

The members of the new Commission on Powder Diffraction are:

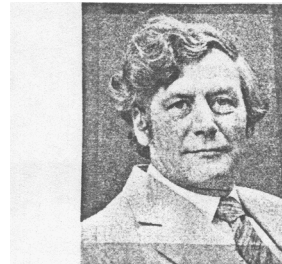
Z. Bojarski	Poland
R.J. Hill	Australia
A.W. Hewat	France
J.I. Langford	UK (Secretary)
P.E. Werner	Sweden
T. Yamanaka	Japan
R.A. Young	USA (Chairman)

In addition, Dr L.K. Frevel serves as the JCPDS appointed representative to the Commission.

R.A. Young



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CALL FOR CONTRIBUTIONS TO THE COMMISSION AND ITS NEWSLETTER

Members of the powder diffraction community are invited to contact any member of the Commission on Powder Diffraction with matters for possible consideration by the Commission and/or

inclusion in subsequent (biannual) Newsletters. A matter for which input from the diffraction community is certainly needed relates to the selection (if deemed desirable) of a logo for the Commission.

R.J. Hill
Editor, Newsletter No 1.

MEETING REPORT:

INTERNATIONAL SYMPOSIUM ON X-RAY POWDER
DIFFRACTOMETRY

Fremantle, Australia, August 20-22, 1987



Conference venue: The Esplanade Hotel, Fremantle.

Organizing Committee:

- B.H. O'Connor (Chairman)
- A.N. Bagshaw
- J. Graham
- E.H. Nickel

Program Committee:

- | | |
|---------------|----------------------|
| B.H. O'Connor | Australia (Chairman) |
| Z. Bojarski | Poland |
| H.F. Fan | China |
| J. Fiala | Czechoslovakia |
| H.E. Goebel | Fed Rep Germany |
| R.J. Hill | Australia |
| C.R. Hubbard | USA |
| H.M. Rietveld | The Netherlands |
| P.E. Werner | Sweden |



Brian O'Connor

Overview

The symposium was a satellite meeting of the XIVth Congress and General Assembly of the IUCr held in Perth, Western Australia from August 12-20, 1987. The meeting was designed to deal with data collection and analytical procedures, emphasizing the following topics:

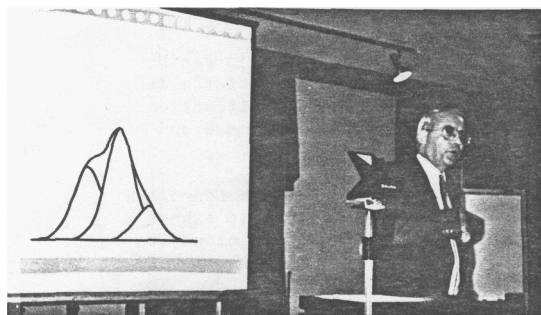
- (i) Data acquisition
- (ii) Profile characteristics and applications
- (iii) Quantitative studies of materials, including the Rietveld method

The text of most of the Keynote and Invited papers from the symposium will be published in a special volume of the *Aust.J.Physics*.

Dr Hugo Rietveld of The Netherlands was Guest-of-Honour for the symposium. The other feature of the meeting was its selection by the Board of the JCPDS International Centre for Diffraction Data for presentation of the second Hanawalt Award and its associated lecture; Dr William Parrish, San Jose, USA received the award.

Guest-of-Honour: Dr H.M. Rietveld

Dr Rietveld is now Head, Department of Scientific and Technical Information, Netherlands Energy Research Foundation. His contribution to the field of X-ray powder diffraction has been of major importance in information technology as it relates to materials. He first described the Rietveld refinement method at the IUCr Congress in Moscow in 1966 and subsequently published two major papers on the subject in 1967 and 1969. The rapid acceptance of the method may be gauged from the statistic that the number of citations for the Rietveld method increased from 5 in 1969, to 52 in 1978 and 120 in 1986.



Dr Hugo Rietveld during his keynote address

The Second Hanawalt Award Recipient: Dr W. Parrish

The Hanawalt Award is presented every three years for an important contribution to the field of powder diffraction. The Award was first presented in 1983 to Dr Ludo Frevel at the Denver X-ray Conference.

Symposium participants were saddened to hear of the passing of Dr J.D. Hanawalt shortly before the meeting. Dr Hanawalt's influence on the X-ray powder diffraction field was prodigious. In 1936 he published with the late Sid Rimm their classic paper *Identification of Crystalline Materials - Classification and Use of X-ray Diffraction Patterns* in the Analytical Edition of *Industrial and Engineering Chemistry*. And so the term Hanawalt Method became a by-word in powder diffraction. The fiftieth anniversary of this contribution was marked by its re-publication as the first paper in the first issue of *Powder Diffraction*.

Dr Parrish's accomplishments are described in a contribution from Dr D.K. Smith later in the Newsletter.



Presentation of the Hanawalt Award to Dr William Parrish (right) by Dr D.K. Smith of the Pennsylvania State University

B.H. O'Connor

XIIIth CONFERENCE ON APPLIED CRYSTALLOGRAPHY:
Silesian University, Cieszyn, Poland, August 23-
27, 1988

The conference is being organized by the Institute of Physics and Chemistry of Metals at the Silesian University in Katowice, Poland, jointly with the Institute of Ferrous Metallurgy in Gliwice and with the support of the Crystallography Committee of the Polish Academy of Sciences. For further information, notification of participation and submission of papers, address all correspondence to:

XIIIth Conference on Applied Crystallography
Uniwersytet Skaski,
Instytut Fizyki i Chemii Metali
Dr Eugeniusz Lagiewka, ul. Bankowa 12
40-007 Katowice, POLAND

ELEVENTH EUROPEAN CRYSTALLOGRAPHIC MEETING:

Technical Univ of Vienna, August 28 - September 2,
1988

The conference will include invited general lectures, microsymbiosia and poster sessions. The main topics are:

fundamental crystallography * crystallography
and biology crystallography and chemistry
crystallography and mineralogy crystallography
and materials science crystallography and
physics .

Please address all correspondence to:

ECM-11, Institut für Mineral, Kristallogr und
Strukturchemie
TU-Wien, Getreidemarkt 9, A-16060 WIEN,
Austria

*3rd INTERNATIONAL CONFERENCE ON SYNCHROTRON
RADIATION INSTRUMENTATION*

Tsukuba Science City, Japan, August 29 -
September 2, 1988

SRI-88 is being organized under the auspices of the Photon Factory, National Laboratory for High Energy Physics. It will follow through on the theme of previous conferences held at Stanford, USA (SRI-85) and at Hamburg, FRG (SRI-82), and focus on recent developments in synchrotron

radiation instrumentation, specifically those with the potential to overcome current limitations. The scientific program may include the following topics:

* machine operation and electron beam cha- *
racteristics * undulators, wigglers and FEL *
detectors * optical elements for synchrotron
radiation * apparatus for beam line stations
new scientific opportunities using synchrotron
radiation * small storage rings for industrial
use new concepts and designs * plans for the
next generation of storage rings * powder dif-
fraction analysis.

Please address all enquiries and corres-
pondence to:

Secretariat for SRI-88
c/o Simul International Inc,
Kowa Bldg No 9
8-10, Akasaka 1-chrome, Minato-ku,
Tokyo 107, JAPAN

*FIFTEENTH CONGRESS AND GENERAL ASSEMBLY OF THE
IUCr:*

Bordeaux, France, July 19-28, 1990

Planned satellite meetings include:

- Local order by X-ray and neutron diffuse scattering and spectroscopy. Orsay-Paris, July 16-18
- * Application of synchrotron and neutron radiation to the diffraction analysis of nuclear and magnetic structures. Grenoble, July 29-31
- * Summer school on crystallographic computing. Strasbourg, July 29 - August 4.

In addition, the CPD hopes to receive approval to organize a satellite on Powder Diffraction Analysis at another location in France, probably just prior to the Bordeaux Congress.

For further information please contact:

Dr M. Hospital,
Laboratoire de Cristallographie
Univ de Bordeaux I,
33405 TALENCE CEDEX, FRANCE

NEWS FROM OTHER IUCr COMMISSIONS

The Commission on Powder Diffraction plans to keep a strong liaison with the Commissions on Crystallographic Apparatus, Neutron Diffraction, and Electron Diffraction because they have interests and responsibilities most closely allied to those of the CPD. In line with this policy, a brief news item from the CCA is reproduced below.

COMMISSION ON CRYSTALLOGRAPHIC APPARATUS

Aims and objectives

The Commission on Crystallographic Apparatus (CCA) is responsible to the Executive Committee (EC) of the IUCr for the following:

- (i) All aspects of the design, construction and development of equipment used in crystallography (which ranges from that involved in X-rays to Mossbauer and NMR spectroscopy) ;
- (ii) The provision of advice on the use of this apparatus (including investigations into sources of error in their use);

- (iii) The study of the physical properties of the radiations employed in the crystallographic experiments and in the nature of their interaction with matter, and
- (iv) Collaboration with other Commissions in the dissemination of the information gained from the studies mentioned in (i)-(iii), perhaps involving collaboration on projects of mutual interest.

Members

B. Batterman	USA
D.C. Creagh	Chairman Australia
R. Fourme	France
H. Hashizume	Japan
P. Kierkegaard	Sweden
Liang Jin Kui	PRC
G. Materlik	FRG
T. Paakkari	Finland

In addition, there are two consultants:
G. de Titta and S. Martinez-Carrera who are responsible for the operation of the CCA's project (i) below.

ROUNE-ROBIN SURVEY OF RIETVELD REFINEMENT

One of the first tasks of the new Commission on Powder Diffraction is to respond to requests to undertake an intercomparison of Rietveld data collection and analysis methods. A similar survey has been on the agenda of the Commission on Crystallographic Apparatus for some time and will now become the responsibility of the new Commission on PD. This task is urgently needed in view of the plethora of Rietveld programs that are in current use and under active distributed around the world. With several notable exceptions, few of these programs have been subjected to vigorous and independent testing by anyone other than those in the laboratory from whence they came.

The details of the round-robin have yet to be decided completely, but it is the intention to follow the format used by the JCPDS International Centre for Diffraction Data in powder diffraction analysis, and by several Commissions of the IUCr in single crystal analysis, charge density, X-ray attenuation, etc. A small number of samples will be selected that offer the potential to test the resolution of X-ray and neutron powder diffraction instruments (of all kinds), together with the accuracy and precision of the associated parameters derived from the analytical code. The samples will be distributed to a group of laboratories and their data and in-house results will be assessed by a sub-committee appointed by the Commission on

Powder Diffraction. The assessment by the CPD will probably include reanalysis of the raw diffraction data with a 'standard' program.

Although the survey will not be executed until late 1989, the Commission wishes to ensure maximum input from all interested powder diffractionists well in advance and now invites suggestions on:

- (i) The recommended data collection procedure (step width, step intensity, wavelength, scan range, etc)
- (ii) The particular aspects of Rietveld analysis that are most urgently in need of testing/comparison.
- (iii) Samples that are best suited to test these specific aspects.
- (iv) Laboratories that would be willing to participate in the survey.

Please send your ideas/recommendations/offers-of-help to:

Dr R.J. Hill,
CSIRO Division of Mineral Products,
PO Box 124, Port Melbourne, Victoria 3207,
Australia
Telex AA34349 MINRL
Fax (61) 3 647 0395
ACSNET rodh@dmpmela.oz

FORTHCOMING MEETINGS OF INTEREST TO POWDER DIFFRACTIONISTS

WORKSHOP ON RIETVELD ANALYSIS:
NERF, Petten, The Netherlands, May or June 1989.

Although not yet formally approved by the IUCr, it is intended that this meeting will be organized under the auspices of the IUCr Commission on Powder Diffraction. The members of the CPD are delighted, and very enthusiastic, about the prospect of holding this workshop at Dr H.M. Rietveld's Institution, the 'Mecca' of powder diffraction analysis by the Rietveld refinement method.

It is intended, at this stage, that the scientific program will operate on two levels: introductory 'workshoptype' sessions for newcomers to the field, and a full program of oral and poster sessions concentrating on new developments in Rietveld analysis. The topics to be covered will probably include:

* modelling of preferred orientation * criteria of fit quantitative phase analysis
extraction of crystal size and strain characteristics * extension to two dimensions (fibre diagrams) * modelling of amorphous components * modelling of faults and defects (layered and intercalate structures) * treatment and effects of background * correction procedures for specimen and instrument aberrations * reflection profile functions * refinement limits * treatment of false minima and correlations * meaning of esd's.

It is planned that the proceedings will be published in coherent, internally consistent book form, providing solutions to problems rather than a maze of controversy. For further information please contact Dr J.I. Langford (address on page 1) or:

Dr A.W. Hewat,
Institute Laue-Langevin, 156X
Grenoble Cedex 38042, France

AMERICAN CRYSTALLOGRAPHIC ASSOCIATION ANNUAL MEETING:
Philadelphia, Pennsylvania, USA, June 27 - July 1, 1988

For further information, contact:

Dr D.H. Voet,
Dept of Chemistry, Univ Pennsylvania
Philadelphia, PA 19104, USA

DENVER CONFERENCE ON APPLICATIONS OF X-RAY ANALYSIS:
Denver, Colorado, USA, August 1-5, 1988

For further information contact:

Lynne Bonno, Conference Secretary,
Dept of Engineering
Univ of Denver, 2390 South York St,
Denver, CO 80208, USA

7th NATIONAL CONFERENCE OF THE AUSTRALIAN X-RAY ANALYTICAL ASSOCIATION
Univ Western Australia, Perth, Western Australia,
August 14-18, 1988

Under the title "New Horizons in Analytical Science", AXAA-88 will provide a forum for discussion across the full range of XRD, XRF and Surface Analysis. In addition, three consecutive schools/workshops on each of these topics will be held and there will be a busy social programme and post-conference tours. For further information, please contact:

Dr B.H. O'Connor, Dept of Physics
Curtin University of Technology,
GPO Box U1987
Perth 6001, Western Australia.

THE 1987 J.D. HANAWALT AWARD
FOR CONTRIBUTIONS IN POWDER DIFFRACTION ANALYSIS

The 1987 Hanawalt award for significant contributions to the field of powder diffraction analysis was presented to Dr William Parrish at the IUCr Symposium on Powder Diffractometry in Fremantle, Western Australia on August 21, 1987. Although this award is presented for recent work in the field, it is fitting that the recipient has had a long career as one of the leaders in powder diffraction.

William Parrish first studied crystallography at the Massachusetts Institute of Technology and received his PhD in 1940. He was a Research Associate at MIT and an Instructor in Mineralogy and Crystallography at the Pennsylvania State University from 1939 to 1942. In 1942, he was appointed Chief Technologist in the War Department to take charge of the technical development of methods for manufacturing quartz oscillator plates for radio frequency control for the US Armed Forces. It was necessary to control accurately the sawing angles for cutting the plates, and he developed unique X-ray diffraction methods for this alignment which were made an integral part of the production process. The program could then produce large quantities of crystals, and Parrish was awarded a War Department Citation for his contribution.

In 1943, Parrish joined Philips Laboratories, and became Chief of the X-ray and Crystallography Section. He was responsible for developing many of the instruments and methods which were marketed by Philips Electronics Instruments and widely used by laboratories around the world. His most important achievement was the invention in 1947 of the X-ray diffractometer using Bragg-Brentano optical geometry which yielded high intensity and resolution with a good line shape. This design is basic to most diffractometers in use today, and it is estimated that there are around 12000-15000 such instruments throughout the world, making it the most widely used X-ray crystallographic instrument. An extensive systematic research program at Philips produced many important devices to enhance the utility of the diffractometer, including the introduction of the scintillation and proportional counters with electronic filtering to improve the intensity response, linearity, and peak-to-background ratio of the diffraction peaks. Other devices included the specimen rotator, alignment tools, transmission diffractometers and vacuum chambers. Parrish considers the powder diffractometer as his most important and satisfying achievement. The Fremantle meeting marked the 40th anniversary of this invention.

In the early 60's, Parrish became interested in the space program and put a proposal to the National Aeronautics and Space Administration to construct a low power, compact powder diffractometer to analyze the lunar surface by remote control. The tests were successful, but the rapid progress in the manned Apollo program relegated the X-ray studies of the lunar samples to the biological containment compartments at the Lunar Receiving Laboratory of the Johnson Space Centre. In 1968, Parrish was appointed Chief of the Materials Characterization Branch, NASA Electronics Research Center, to develop structural methods for analyzing electronic materials.

Parrish joined the IBM Research Division in San Jose, California in 1970. As Manager of the Crystallography and Microstructure Department, he set up diffraction and fluorescence methods for characterizing new materials, including thin films. Improvements in the accuracy of structural and chemical characterization required new developments in instrumental methods, including the introduction of the computer in both data acquisition and data analysis. A large number of analytical programs were developed which were marketed by IBM and recognized with an IBM Outstanding Contribution Award.

Programs included a routine for profile fitting which could separate unresolved peaks in a complex profile. Other routines located peaks and simulated the Powder Diffraction File reference patterns for pattern matching.

In 1977, Parrish became interested in the new storage ring radiation sources and began studies at the Stanford Synchrotron Radiation Laboratory with Professor Michael Hart of Bristol, England. Diffraction topography was used to study garnet films grown on garnet substrates for magnetic bubble memory devices. Laue patterns were obtained which showed considerable detail, and double crystal topographs made it possible to separate the film and the substrate components of the sample to reveal their individual properties. In 1983, the synchrotron studies were extended to powder diffraction. Parallel beam optics and silicon channel monochromators allowed high resolution, high peak-to-background and symmetric profiles which, coupled with the wavelength selectivity, produced remarkable diffraction patterns. Patterns could be obtained in the conventional 2:1 scanning mode, in transmission mode, or in energy dispersive mode at a fixed diffraction angle. This latter method allowed patterns to be obtained in a very short time for time-resolved studies. The new techniques were applied to crystal structure refinements, anomalous scattering, texture analysis, precision lattice parameter determination, quantitative analysis, and depth profiling of thin films.

Parrish is the author of about 300 papers in virtually all aspects of powder diffraction. He organized the first World Directory of X-ray Crystallographers and was Chairman of the IUCr Committee to set up the Journal of Applied Crystallography. He was Secretary-Treasurer of the American Crystallographic Association and a member of the USA National Committee of Crystallography. He has also served on committees of the IUCr and the ACA.

Although the Hanawalt Award was presented to Parrish for his work employing synchrotron radiation in powder diffraction, it is difficult to separate his remarkable career from the most recent work. William Parrish is a young 73 and can expect to remain active for many more years. It was a pleasure for me as the Chairman of the JCPDS International Center for Diffraction Data to present the 1987 J.D. Hanawalt Award to this deserving recipient.

Deane K. Smith,
Chairman, JCPDS-ICDD

Organization

The organization of the CCA was changed somewhat at the Perth IUCr Congress to include responsibilities for the fields of variable temperature and pressure and synchrotron radiation and its uses. The EC has yet to formally ratify these changes, so the following information must be regarded as only tentative.

Consultative Committees

A small consultative committee will be set up to address problems which would be considered to have been of interest to the now defunct Commission on High Temperatures and Pressures. This group, "The Variable Temperatures and Pressures Group", will consist of the following members: Prof H. Schultz, Chairman (FRG), Dr R. Nelmes (UK), Prof L. Vettier (France) and Dr L. Finger (USA).

A second consultative committee is to be formed to consider problems associated with the use of synchrotron radiation sources. This committee is shared with the Commission on Neutron Diffraction, and each Commission will supply two members.

Projects of the commission

The CCA has a long and successful history of problem solving projects. Currently it has a number of projects on its books, some of which are in the planning stage:

(i) Experimental methods for measuring single crystal lattice constants using diffractometers (S. Martinez-Carrers, G. de Titta).

This is a project suggested by the Commission on Journals which is despondent about the manner in which crystal structure data has been presented to the journals by authors. It aims at providing advice to scientists, manufacturers, etc. concerning the proper use of data collected by diffractometers.

(ii) Rietveld refinement (no convenor)

This project was originally suggested by Prof J. Cermak and was intended to study the techniques used by laboratories for diffraction profile fitting and structure refinement. The Commission on Powder Diffraction will take over the organization of this project.

(iii) X-ray attenuation near an absorption edge (G. Materlik, D.C. Creagh)

This project is an extension of an earlier project on X-ray attenuation by D.C. Creagh and J.H. Hubbell. The work would be undertaken at a number of synchrotron radiation sources using the local experimental and analysis techniques to examine sets of standard samples. A number of experimental techniques could be employed (e.g. standard EXAFS, fluorescence EXAFS, X-ray interferometry).

(iv) Some CCA members have been heavily involved in the preparation of International Tables for Crystallography : Volume C.

(v) Planning has commenced for an Open CCA Microsymposium at the Bordeaux IUCr Conference (D.C. Creagh).

D.C. Creagh
Chairman, CCA

MAILING LIST FOR FUTURE NEWSLETTERS

If you wish to receive a copy of future CPD Newsletters please complete the following coupon, or its copy, and return to Dr Langford at the address below:

To the IUCr Commission on Powder Diffraction:

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

_____ Please add the following interested person to your mailing list.

Name : _____
(Please type or print in block letters)

Position/Title: _____

Address : _____

Return this form, filled out, to:

Dr J.I. Langford
CPD Secretary, Department of Physics
University of Birmingham
Birmingham B15 2TT
ENGLAND