COMMISSION ON POWDER DIFFRACTION

INTERNATIONAL UNION OF CRYSTALLOGRAPHY

NEWSLETTER No. 3

August 1989

REPORT ON THE IUCr WORKSHOP
ON THE RIETVELD METHOD

The workshop was held, 13 to 15 June 1989, at the site of the Netherlands Energy Research Foundation ECN, Petten, which is about 50 kilometers north of Amsterdem on the North Sea Coast. More than twenty years ago, Dr. H. M. Rietveld invented at Petten a method of the crystal structure refinement from the powder diffraction, pattern which is now the standard technique as "Rietveld method" for the structure refinement in the many science fields.

There were about 150 participants in the workshop (see photograph) from many countries. 18 invited oral presentations and 45 posters were contributed through three days. Besides them the round table discussion was prepared at the end of the session in every day.

The workshop started with the opening remark by Prof. H. H. van den Kroonenberg, Director of ECN and the introductory remarks by Prof. R. A. Young, Chairman of the CPD. The first presenter in the science session was Dr. H. M. Rietveld, who talked about "The early days: A retrospective view of the Rietveld method".

All participants appreciated the extremely meaningful lectures and fruitful discussions. A sight seeing boat tour with a buffet through the canals of Amsterdam was held at the evening June 14.

IUCr CPD expresses his gratitude to Dr. C. van Dijk, and the local organizing committee for the well organized meeting and is also grateful to Dr. A. W. Hewat and the international programme committee for the excellent arrangement.

Participants of the Rietveld Workshop at Petten.

FROM CHAIRMAN OF THE COMMITTEE
RIETVELD REFINEMENT AT ECN PETTEN

More than twenty years ago, what is now the standard technique fof refining the crystal structure of materials from their powder diffraction patterns, Rietveld Refinement, was invented at Petten. Many solid state chemists and physicists may not know much about neutron diffraction, but most of them have heard about Rietveld refinement. The Rietveld is widely recognized to be uniquely valuable for structural analysis of nearly all classes of crystalline materials not available as single crystals. Most recently it has proved essential in the determination of the new superconductor structures. In general, structure analysis of other heavymetal oxides, such as catalysts and battery electrodes, as well as hydrides and hydrocarbons in zeolites, clays, minerals, biological materials, polymers, and of course magnetic materials, are representative of the many important applications of the Rietveld method both with neutron and X-ray powder diffraction data.

It is appropriate then that the present of the technique should be celebrated by bringing such a distinguished group of specialists back to Petten, the home of Rietveld refinement, to look back at past achievements certainly, but more importantly to look ahead to what still needs to be done, and to the new opportunities now that Rietveld refinement is accepted as a fundamental technique in solid state science.

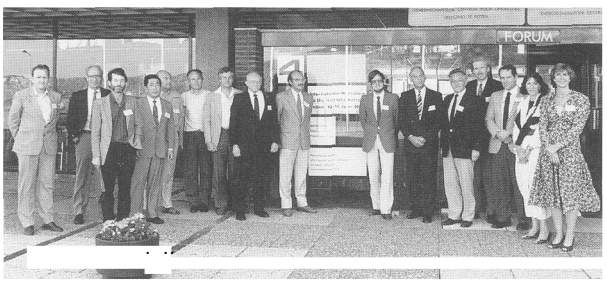
This letter consideration did the Programme Committee agree on a scientific programme of RW-89 with the following objectives:

RW-89 ought to emphasize the current status, problems and future development of the Rietveld method, rather than applications of the method to current scientific questions. Of course improved solutions to scientific problems, and not just the development of techniques per se, is the ultimate objective, and examples of current research will be used extensively to illustarate technical points.

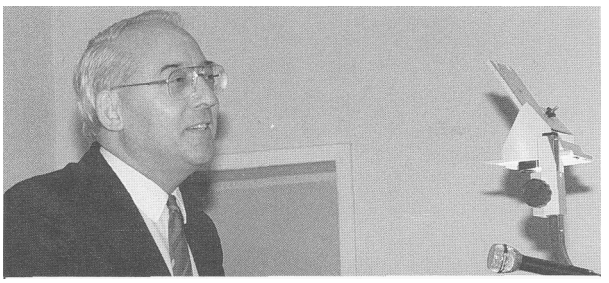
For the same reason, instrumentation and data collection techniques will be mentioned, as will ab initio structure solution, quantitative phase analysis, etc., but the meeting

will mainly concentrate on the Rietveld method. An important objective is that the workshop should provide the basis of a comprehensive book about the nature, implementations and applications of the method.

Alan Hewat



Organizing and Program Committee and CPD Member.


Dr. H. M. Rietveld giving a retrospective talk.

REPORT FROM IUCr COMMISSION ON POWDER DIFFRACTION

Meeting held in association with the Rietveld Method Workshop, Petten, the Netherlands, 13-15June 1989

The meeting was held at the Akersloot Motel, near Alkmaar. The current membership of the CPD is as follows:

Prof. R. A. Young	Chairman
Dr. J. I. Langford	Secretary
Prof. Z. Bojarski	
Dr. A. Hewat	
Dr. R. J. Hill	
Prof. P-E Werner	
Prof. T. Yamanaka	
Dr. L. Frevel	(JCPDS-ICDDRepresentative)
Dr. D. Louer	(Consultant)

This list is as in Appendix I of the notes on the Perth meetings (August 1989),with the addition of Dr. Louer, who was co-opted as a Consultant to the Commission on 15thJune 1988.

1. Rietveld Refinement Robin Project (Dr. Hill)

Dr. Hill reported that, as a result of several questionnaires sent to CPD members, the following general conditions had been agreed:

(a) Two samples to be distributed, with a combination of 'in house' and 'CPD recommended' conditions for data collection.

(b) The structures to be refined by using a small set of CPD-selected Rietveld programs but local codes could be used, if desired.

(c) The CPD to supply a standard dataset for 'in-house' refinement.

As regards (a), it was agreed that a sample of zirconia (M-ZrO_2, V=140.7Å, P21/c, sample 1)should be sent to all participants in the project. Prof. Yamanaka should adequate for 'in house' data collection was considered, but Dr. Hewat was of the opinion that a second, and more demanding, sample should be distributed. It was, therefore agreed that a stabilized zeolite (sample 2), to be supplied by Dr. Hewat, should also be circulated. All participants were to treat data from the zirconia, but treatment of the zeolite would be optional.

For the standard data (item (c) above) it was decided to obtain two datasets for $PbSO_4$ (sample *3*). one with $CuK\alpha$ radiation and a conventional diffractometer (Dr. Hill) and the other with fixed wavelength (~1.59Å) neutrons (Dr. Hewat). The step length should be 0.025° (2θ) and the data are to be collected over the full angular range of the instruments used. Both datasets are to be analyzed by using the program DBW3.2S-8804A before distribution.

After further discussion of item (a),it was not considered necessary for the CPD to advise on experimental strategy for samples 1 and 2. It was decided that all participants should submit both datasets to the CPD, who will use them to make independent refinements of the structures with program DBW3.2S-8804A.

2. Timetable for Round Robin

It is intended that samples will be available for distribution within two months, with a limit of six months for the return of results and datasets. This should allow sufficient time for a preliminary report to be presented (by Dr. Hill) at the 1990 Toulouse satellite meeting.

3. Rietveld-Method Workshop

(a) Report on Meeting

The International Workshop on the Rietveld Method, 13to 15June, the first meeting to be promoted by the CPD, was hosted by ECN and held at Petten, The Netherlands. The Commission was well satisfied with the organization and scientific content of the workshop. Therewere about 150participants, the maximum number which could be reasonably accommodated. In addition to the oral contributions, which were all by invitation, 45 posters were presented. A full accont of the meeting will be included in the next CPD Newsletter.

Dr. Hewat and his committee were thanked for arranging a comprehensive and stimulating programme. The Chairman undertook to thank Prof. van der Kroonenberg, Director of the Netherlands Energy Research Foundation, for allowing the workshop to be held at the Petten laboratory and to express the Commission's gratitude to Dr. van Dijk and his committee for the excellent local arrangements.

(b) Young Scientist Grants

17 applications were received and 15 awards were made, the grants ranging from $240 to $600. Two recipients were unable to attend the meeting and the total amount disbursed was $4,125. The selection committee appointed by the CPD consisted of Drs. D. Cox, C. van Dijk and R. Hill.

(c) Publication

It was confirmed that the oral contributions of the workshop should from the basis of a book on the Rietveld Method, along the lines envisaged at the inaugural meeting of the CPD.

Review of Current Projects

(a) Newsletters

Two Newsletters had been issued since the Perth meeting. No. 1had been edited by Dr. Hill and No. 2 by Prof. Bojarski, and a high standard had been set. Prof. Yamanaka had offered to edit No. *3* (August) and Prof. Werner No. **4** (December/January). The Secretary reported that to data he had received about 350 requests for inclusion in the mailing list.

(b) Toulouse Satellite Meeting, 1990

Instytut Fizyki i Chemii Metali
Dr. Eugeniusz Lagiewka
ul. Bankowa 12

40-007 Katowice, Poland
Phone:59-69-29, Telex: 0315584 usk pl
or 315572 uskwz pl

NEWS FROM OTHER IUCr COMMISSIONS

CPD plants to keep a strong liaison with Commissions on Crystallographic Apparatus, Neutron Diffraction, and Electron Diffraction. The activities of the first two commissions were reported respectively in CPD Newsletters 1 and 2. In this Newsletter a number of items of interest to readers of the CPD Newsletter have been abstracted from the last two annual reports of the Commission on Electron Diffraction.

1987 Report by Professor C. J. Humphreys, past chairman of CED

The Commission on Electron Diffraction has five HEED members (high-energy electron diffraction), three GED members (gas electron diffraction) and two LEED members (low-energy electron diffraction). The Commission (CED) is responsible for advising Editors of the International Tables for Crystallography on sections dealing with electron diffraction and electron microscopy.

At the XIV Congress in Perth, the Open Commission Meeting included Dynamical Electron Diffraction in Transmission and Reflection (THEED, MEED, LEED, RHEED).

"Major discoveries have occurred in the last three years in which crystallography plays a leading role, and these have gripped the imagination of scientists throughout the world. First came the totally unexpected discovery of quasicrystals with fivefold symmetry, and the Commission (CED) has advised the inclusion of this topic in International Tables for Crystallography. Second came the news of the discovery of high-temperature superconductors. Members of CED are now actively studying the local crystallography of these materials using electron diffraction techniques."

1988 Report by Professor J. M. Cowley, present chairman of CED.

The Commission on Electron Diffraction has examined the desirability of publishing a book on Electron Diffraction Techniques. support for the proposal was sufficient to encourage CED to proceed with the planning of a multi-author volume for publication in the proposed IUCr/ Oxford University Press series. The editor will be J. M. Cowley and a publication date in 1990 is anticipated. "The emphasis will be on the experimental and interpretive techniques for high energy electron diffraction in the transmission, convergent beam and reflection modes, with brief sections on low energy electron diffraction and gas electron diffraction."

"It was proposed in the CED meeting at the 1987 Perth Congress that a survey should be conducted of the computer programs now being used for the calculation of intensities for high resolution electron microscope images and electron diffraction patterns. Dr. D. Van Dyck, Antwerp, has agreed to coordinate this project and will present proposals for discussion at the Symposium on Computer Simulation of Electron Microscope Diffraction and Images to be held at the Annual Meeting of the American Metallurgical Society in Lap Vagus, March 1989 and, organized by Dry. Whilom Creak and Mutual A. O'Keefe."

Members of the Commission on Electron Diffraction:
Chairman
J. M. Cowley (USA)
Elected members
C. Colliex (France)
I. Hargittai (Hungary)
R. L. Hilderbrandt (USA)
C. J. Humphreys (UK)
L. Kihlborg (Sweden)
K.-h Kuo (People's Republic of China)
H. Oberhammer (Federal Republic of Germany)
M. Prutton (UK)
M. Tanaka (Japan)
B. B. Zvyagin (USSR)

L. K. Frevel

COMING INTERNATIONAL AND LOCAL MEETING ON THE POWDER DIFFRACTION

February 15, 1990	A one-day workshop on 'Microstructural properties from line-profile analysis', at IcI, Runcorn, Cheshire. Organizer-Dr. S.Fletcher, R & T Dept, ICI plc, Chemical & Polymers Group, The Heath, Runcorn, Cheshire WA7 4QD.
February 16-18, 1990	3-day course & workshop on search/ match procedure, arranged in association with JCPDS-ICDD. At Daresbury Laboratory, Cheshire. Organizer-Mr. J. W. Harding, Room 131B Brunel House, British Rail Research, London Road, Derby DE2 8UP.
December 7, 1989	BCA Industrial Group Workshop: "Powder X-ray Diffraction in Special Environments" BIRKBECK COLLEGE, LONDON [Dr. S. E. Tarling, Industrial Materials Group, Crystallography Dept., Birkbeck College, Malet Street, London WClE7XH]
April 22-29, 1990	"17th Course on Electron Crystallography"ERICE, ITALY [Prof. L. Riva di Sanseverino, Dip. De Scienze Mineralogia, Piazza Porta San Donato 1, 1-40126, Bologna, Italy]
July 12-17, 1990	15th General Meeting of the International Mineralogical Association. BEIJING, CHINA [Prof. H. Yunhui, Organising Committee of IMA 1990. c/o Institute of Mineral Deposits, Chinese Academy of Geological Fuchengmenwai, Beijing, People's Republic of China]
July 19-28, 1990	International Union of Crystallography:"15th General Assembly and International Congress" BORDEAUX, FRANCE [M. Hospital, Lab be Cristallographie, Universite de Bordeaux 1, 351 Cours de la Liberation, F-33405 Talence, France.]

graphic community, for the CPD to define a standard format for the input of data to PD programs, along the lines of the JCAMP-DX format. The Chairman pointed out that the JCPDS-ICDD had already embarked on such a project and it was felt inappropriate for the CPD to become involved at this stage.

8. Awards

The IUCr Executive Committee had asked the CPD to recommend nominations for the award of the Ewald Prize. Various possible nominees were discussed and it was agreed unanimously that the CPD nomination should be Professor Guinier. The Chairman undertook to inform the Executive Secretary of this decision.

J. Ian Langford

Appendix

Areas of interest of industrial crystallographers engaged in powder diffraction, conveyed to the CPD by Dr. Frevel.

1. The efficient and reliable identification of multi-phase patterns (3 or more phases).

Cost-effectiveness and turn-around time for X-ray powder diffraction analysis are of obvious importance to the managers of industrial laboratories.

2. The detection and measurement of preferred orientation.
3. Sample preparation techniques.
4. Deconvolution of overlapping peaks.
5. Quantitative assay of crystalline phases (analysis time, accuracy).
6. Two-dimensional aspects of polycrystalline diffraction.
7. Computational aids such as POWDER12.
8. Degree of crystallinity (organic polymers; hydroxy oxides such as FeO(OH), MnO(OH), and AIO(0H); heterogeneous catalysts, etc.)
9. Determination of minor phases; e.g., β-Sic, metallic silicides, and quartz in commercial silicon (98% assay).
10. accuracy of PDF data.
11. Crystallite-size distributions, strain distributions.
12. Textural properties of fabricated products (polymers, metals, ceramics, composites).

RIETVELD SUMMER SCHOOL
FOR BEGINNERS RSSB-90
Cieszyn, Poland, August 9-11, 1990

The School is organized by the Commission on Powder Diffraction IUCr, Silesian University (Katowice, Poland), Institute of Ferrous Metallurgy (Gliwice, Poland) with the financial support of the International Union of Crystallography. It will be held just after closing 14th Conference on Applied Crystallography.

The School is to give the opportunity to acquire the basic features and present applications of the Rietveld method, and will consist of lectures and practical training.

Deadline for preliminary notification of participation: December 31, 1989

The number of participants is pestricted to 50 persons.
The registration and accommodation fee is 2OO$US.

Address for correspondence:
Summer School RSSB-90
Uniwersytet Slaski
Instytut Fizyki i Chemii Metali
Dr. Eugeniusz Lagiewka
ul. Bankowa 12
40-007 Katowice, Poland
Phone:59-69-29,Telex:0315584 usk pl
or 315572 uskwzpl

The International Union of Crystallography has granted a financial support for students and young scientists to allow them to pay the participation and accommodation fees. Anyone who wants to compete for the grant should send an application which must contain:

- a short statement of how the participation in the School will help in the applicant's work
- a short curriculum vitae
- a supporting letter from the applicant's supervisor.

The applications should be send until December 31,1989 to:
Prof. Dr. Zbigniew Bojarski
Uniwersytet Slaski
Instytut Fizyki i Chemii Metali
ul. Bankowa 12
40-007 Katowice, Poland
Phone:59-69-29Telex:0315584 usk pl
or:315572 uskwzpl

The programme of the School and further information will be send in 2nd circular before February 28,1990 to everybody who will apply for participation in the school.

Dr. Eugeniusz Lagiewka

XIVth CONFERENCE
ON APPLIED CRYSTALLOGRAPHY
Cieszyn, Poland, August 5-8, 1990

The Conference is organized by the Institute of Physics and Chemistry of Metals at the Silesian University in Katowice jointly with the Institute of Ferrous Metallurgy in Gliwice and with the support of the Crystallography Committee of Polish Academy of Sciences. The scientific programme includes following topics:

- Real structure of materials (metals, ceramics, polymers, etc.)
- Research methods and equipments (phase identification, precession measurements of lattice constants, determination of crystallites sized and distortions, texture, high temperature X-ray techniques, small-angle scattering X-ray topography, etc.).

Deadline for notification of paricipation and subject of paper-February 1, 1990
Deadline for submitting papers
- March 1. 1990

The registration and accommodation fees:
for participants -2OO$US
for accompanying person - 11O$US

Fees for registration and accommodation should be paid to:
Uniwersytet Slaski, Bank Slaski, Oddazial VII,
Katowice, nr 312608-5018, Poland
(Applied Crystallography) by May 31, 1990.

Address for correspondence:
14th Conference on Applied Crystallography
Uniwersytet Slaski

The request from the CPD to hold a satellite meeting of the 15th IUCr Congress at Bordeaux, 1990, had been approved. The meeting, entitled 'Powder Diffraction', will be held at Toulouse, 16-19 July. The Chairman reported that Dr. Louer had agreed to be chairman of the programme committee and that Dr. J. Galy should be the local organizer. The following had accepted an invitation to serve on the programme committee:

Prof. A. K. Cheetham	(CND representative)
Dr. J. Fiala	(Czechoslovakia)
Dr. J. I. Langford	(UK)
Prof. D. K. Smith	(USA)
Dr. H. Toraya	(Japan)
Prof. P-E Werner	(Sweden)

In addition, Dr. Galy would be a member of the committee *ex officio.*

Dr. Louer presented a draft proposal for the scientific programme and a timetable for the meeting, which had been considered earlier in the day at an *ad* hoc meeting of five members of the programme committee. The first circular was due for circulation later in the summer, with the second circular to follow in October or November. The deadline for the submission of abstracts is to be 15 February 1990. It is interded that most sessions will be 'open', with only a limited number of invited contributions.

(c) Powder Sessions at the Bordeaux Congress

The CPD has already recommeded that a microsymposium entitled 'Scientific advances from structure determinations with powder diffraction data' should be included in the Bordeaux programme, with Prof. A. K. Cheetham as chairman. Various other topics were discussed and it was eventually agreed that the CPDs second suggestion should be 'Powder diffraction studies of fibrous, polymeric and similarly imperfectly ordered materials', with Dr. W. R. Busing as chairman.

(d) Program Information Exchange 'Bank'

The Chairman reported that Prof. Deane Smith had produced a compendium of programs relating to most aspects of powder diffraction. Copies of this had been made available to CPD members. In order to avoid duplication of effort, it was agreed unanimously that, subject to the approval of the President of the IUCr, Professor Smith should be co-opted as a consultant to develop further and to operate the 'Bank'. It was noted that the present compendium, excellent and welcome though it is, is short on pattern decomposition and profile analysis (not Rietveld) programs.

(e) Rietveld Summer School, 1990

Professor Bojarski informed the Commission that preliminary arrangements had been made to hold a 'Rietveld Summer Scholl for Beginners' at Cieszyn, Poland, 9-11 August 1990. immediately after the 14th Conference on Applied Crystallography. This was being organized jointly by the CPD, the Silesian University (Katowice) and the Polish Institute of Ferrous Metallurgy (Gliwice). As a result of the CPDs formal application and vigorous support for such a Summer School, the IUCr had agreed to contribute $3,500 towards the travel costs of lecturers and a grant of $4,000 for Young Scientist Awards. The appointment of Prof. Young as Course Director was approved. CPD members were invited to recommend possible lecturers for the course.

5. Report from JCPDS-ICDD Representative

Dr. Frevel reported on a survey he had conducted to ascertain the interests and concerns of industrial crystallographers engaged in powder diffraction. He based his assessment contacts he had has with experienced diffractionists at Dow Chemical Company, Cow Corning Corporation, Pennsylvania State University, Dupont Central Research Laboratories, Sandia Laboratories and the Michigan Molecular Institute. The results of this survey are given in the appendix.

Dr. Frevel said that item 1, the efficient and reliable identification of multi-phase patterns, is generally regarded as being of fundamental importance, but there is no particular ordering of the remaining items. Dr. Frevel was thanked for his report and it was agreed that it should be passed to the Programme Committee of the Toulouse meeting for further consideration. See also items 6(a) and 7(b).

6. CPD Self Assessment

(a) Service to powder-diffractionists

It was clear from Dr. Frevel's report that we could serve our industrial 'constituency' more than at present. [See item 7(b)l .

(b) Awareness of powder-diffraction activities

It was felt that the CPD was not fully aware of PD activities worldwide. Members of the Commission were requested to send to the Secretary information on all PD meetings brought to their notice. This should then be transmitted to the Newsletter editors. Dr. Frevel pointed out that a useful source of information on crystallographic activities is Powder Diffraction and this could form the basis of a list in the Newsletter.

(c) Awareness of CPD activities

At present worldwide awareness of CPD activiities is patchy and a more widespread distribution of the Newsletter, through regional or national crystallographic bodies, is desirable. Dr. Frevel proposed, on behalf of the JCPDS/ICDD, that a request for ideas to improve search/match procedures be included in the Newsletter. He agreed to provide a suitable note for insertion in Newsletter No. **4.**

(d) Coverage of CPD activities

Areas of powder diffraction to which the CPD had hitherto given little attention were discussed, in conjunction with Dr. Frevel's report (item 6(a)). It was agreed that phase identification should be a suitable topic for a future workshop.

(e) Worldwide attention by CPD

It was agreed that members should inform the Secretary of any areas of the world which were not being given fair attention by the CPD.

7. CPD and its Activities in the Next-Triennium

(a) Membership

A list of possible members of the CPD for next triennium. received from national committees, was noted. Prof. Bojarski undertook to find a suitable candidate from the USSR. Suggestions from the CPD for future membership of the Commission, including the post of Secretary, were to be submitted to the Chairman by January, 1990, for transmission to the Executive Council. Recommendations for the post of Chairman should be sent to the Secretary.

(b) Intercongress Topical Meeting

It was noted that two major PD meetings will be held in 1991, an international meeting in Hawaii for the USA and the Far East and the first European PD meeting, to be held in Munich. It was agreed that it would be opportune for the CPD to arrange an intercongress meeting in 1992, possibly another workshop, along the lines of the recent meeting at Petten. Suggested topics were 'Effective Phase identification and quantitative analysis', 'Powderdiffraction in materials science' and 'Neutron and electron powder diffraction'. Recommendations for the topic and nature of the meeting, and a suitable venue, should be sent to the Secretary by January, 1990.

(c) New Projects

A request had been received from Mr. J. L. C. Daams of Philips, Eindoven, on behalf of the Dutch crystallo-

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.

T. Yamanaka
Editor, Newsletter No. 3

MAILING LIST FOR FUTURE NEWSLETTERS

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

To the IUCr Commission on Powder Diffraction:

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

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

Name: _____

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

Addresses of the CPD members:

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