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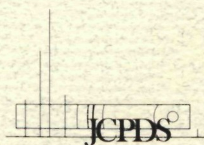

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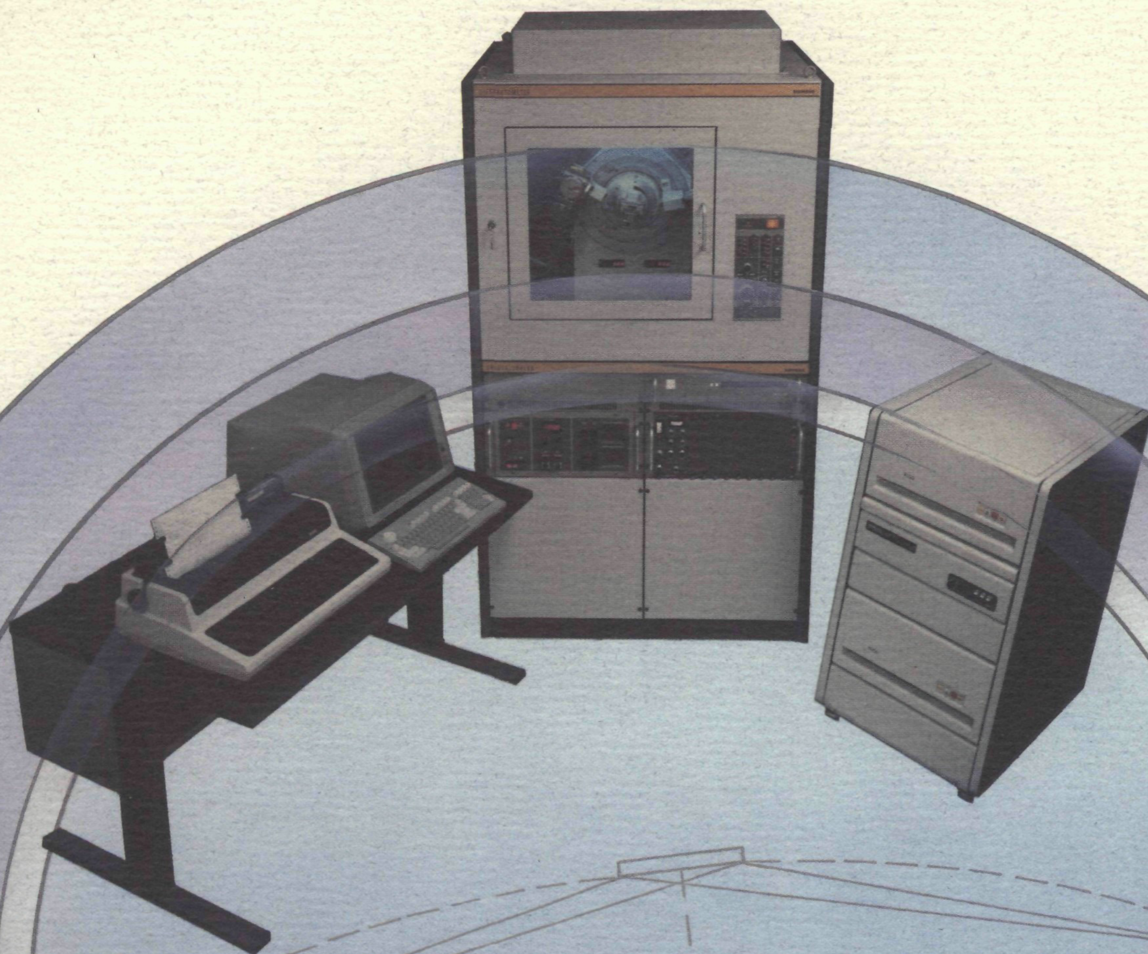

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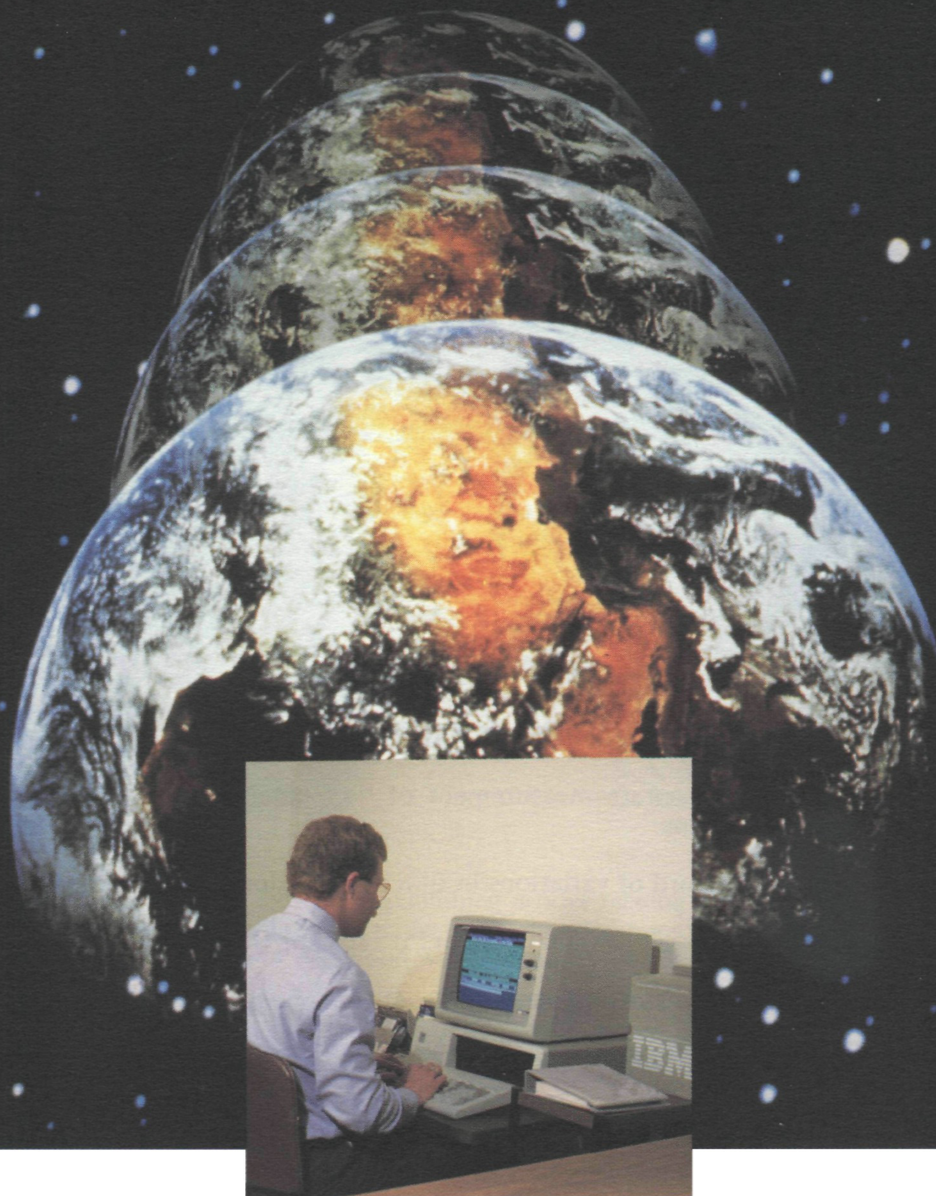

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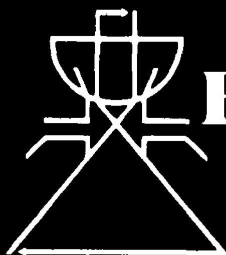

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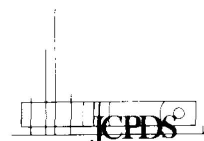

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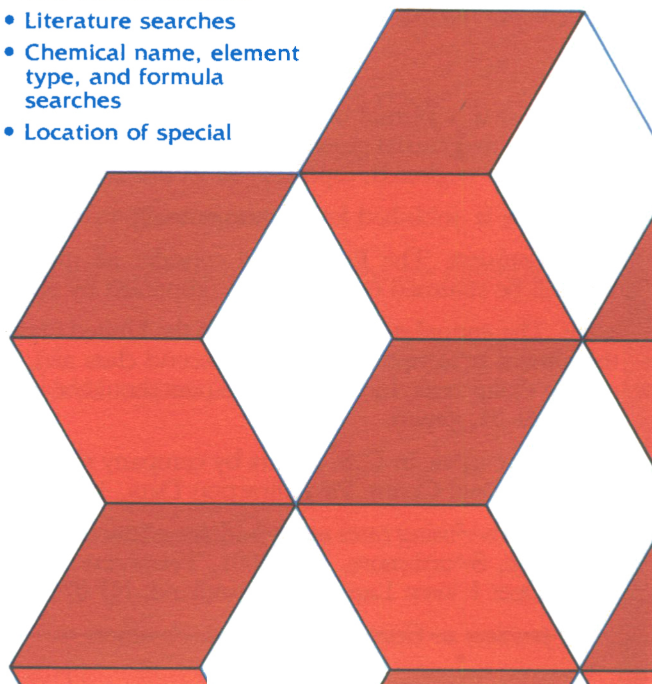

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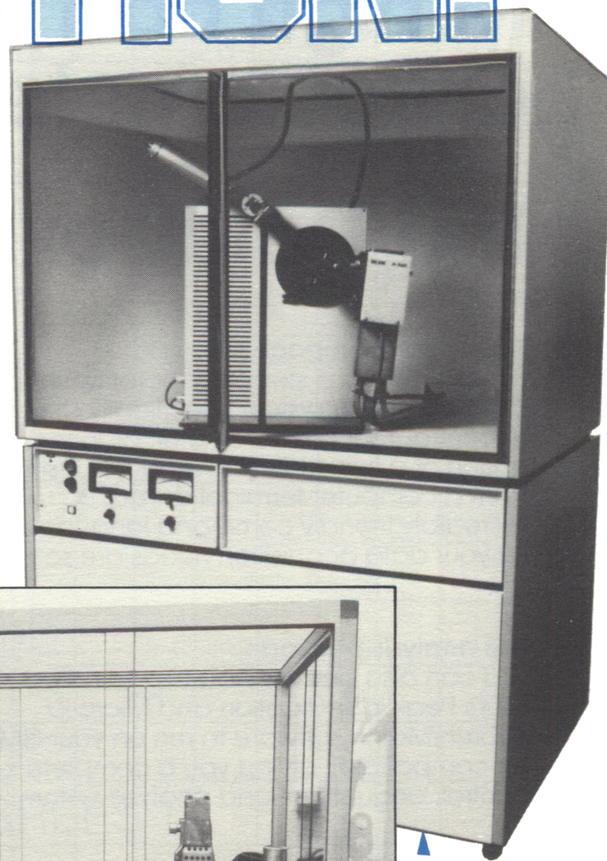

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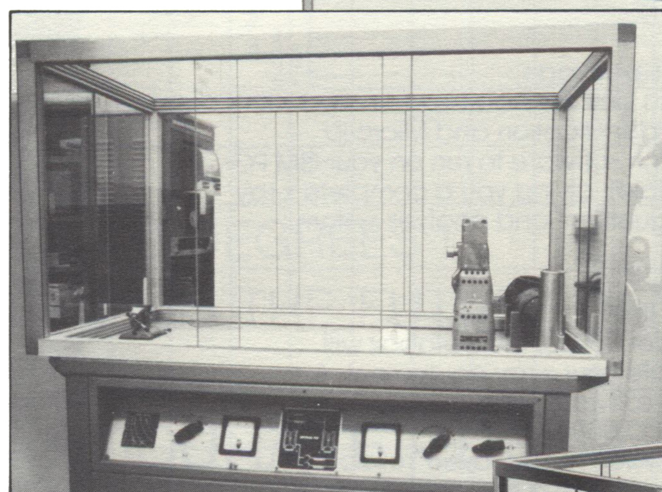

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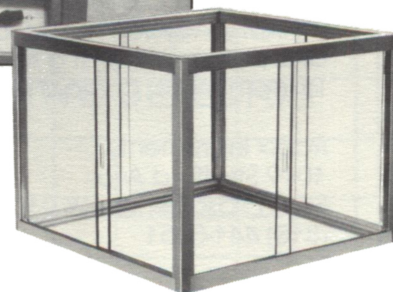

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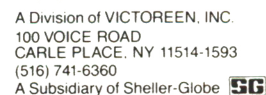

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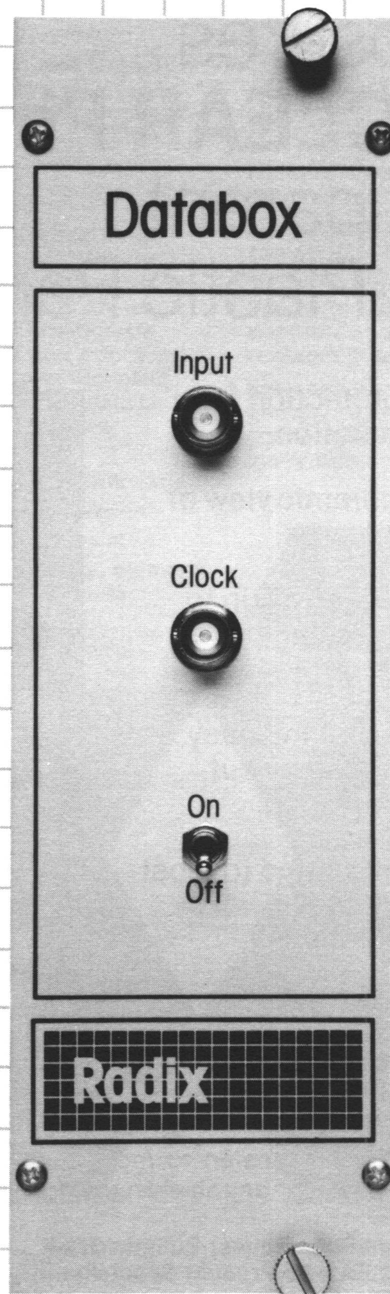

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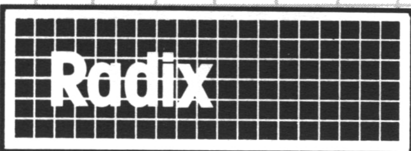

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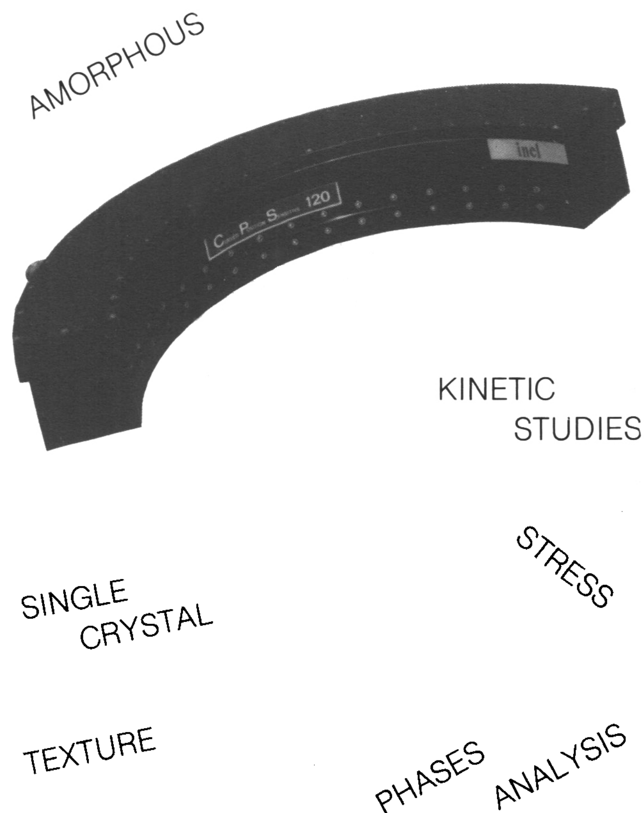

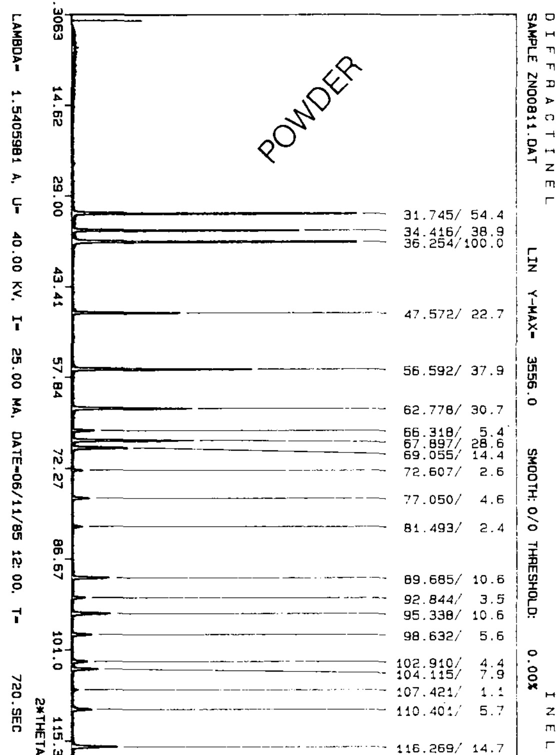

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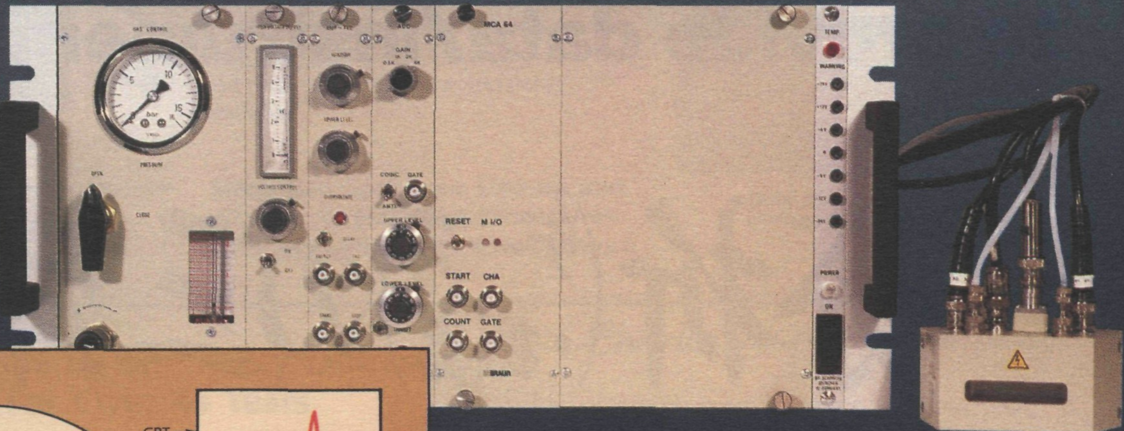

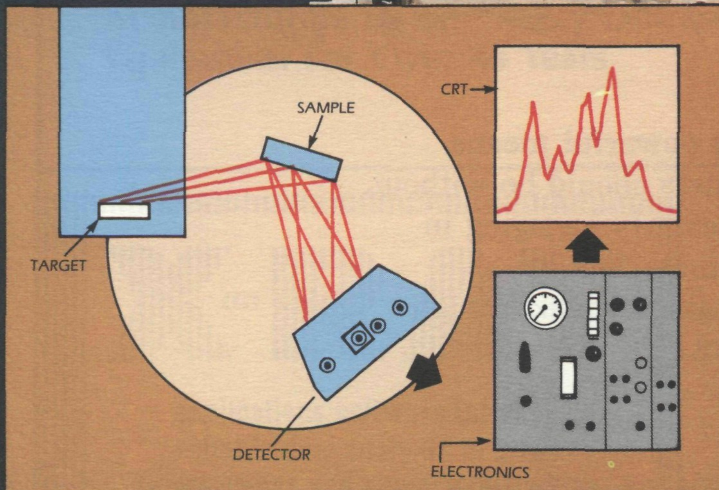

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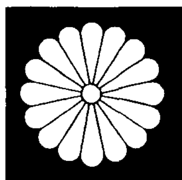
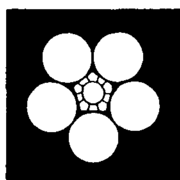

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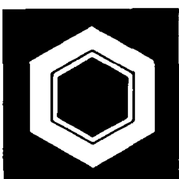
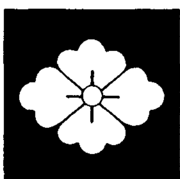

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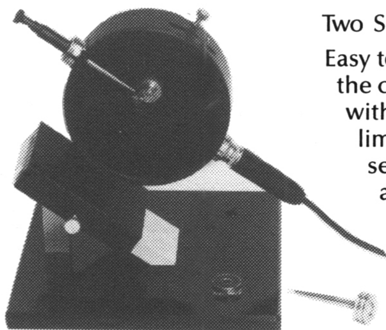

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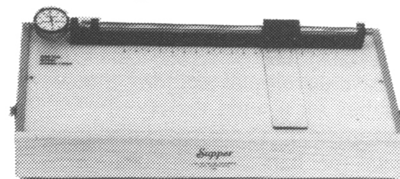

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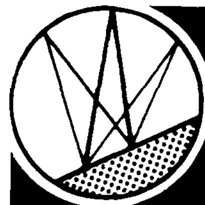

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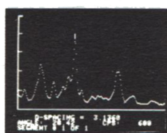
Automatic Peak Search

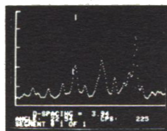
SUMMARY OF PEAKS FOUND

TWO-THETA ANGLE	D-SPACING	NET INTENS.	RELATIVE HT.
51.63	1.7683	514.12	100
26.55	3.3536	411.3	80
65.71	1.4193	318.75	62
37.86	2.3736	272.48	53
28.3	3.1493	154.23	30
61.7	1.5015	87.4	17
43.82	2.0637	82.26	16
24.99	3.5592	82.26	16
78.4	1.2183	66.83	13

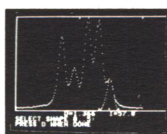
The second is **manual:** just position the cursor at the top of a peak and press "P".



The third method does a **parabolic fit:** all you do is position the cursor anywhere within a peak and press "F".



And the fourth performs **deconvolution,** by allowing you to subtract the background and then fit the peak with any of six stored profiles. This method is especially useful in low two-theta regions, where abnormal background and abnormally shaped peaks can otherwise make analysis difficult.



ThetaPlus+ also offers you two different methods of identifying unknowns:

1. Time saving search/match of most likely standards

Just set a few parameters, and ThetaPlus+ gives you a printout of the most likely phases, the number of lines uniquely matched for each, and three different figures of merit.

SEARCH MATCH RESULTS

ANALYSIS OF FILE SYNROC-E

SEARCH PARAMETERS:

MINIMUM LINES FOR POSITIVE MATCH/TOTAL LINES SCANNED: 7/20
ERROR-WINDOW MULTIPLIER: .15 OFFSET ANGLE: 0 DEGREES.

		FIGURES - OF - MERIT			/UNIQUENESS:/	
PHASES FOUND:	LINES FOUND	INTEN- SITY:	POSI- TION:	OFF- SET:	LINES FOUND	INT. RATIO
HOLLANDITE	19	.97	.76	.00	12	.77
CAZRTI207	18	.94	.72	.00	14	.20
CATI03	17	.89	.61	.00	12	.28
AL203	10	.75	.32	.02	6	.06
TI02 (R)	7	.38	.18	.00	4	.05

A high value (close to 1.0) indicates that most major lines matched.

A low figure may indicate too wide a search window.

A high value warns you of a systematic error in measurement.

2. Visual comparison of diffraction patterns

ThetaPlus+ lets you compare the unknown with up to 5 standards at a time (Figure 1), subtract any one of the standards (Figure 2), then compare the residual with a second standard (Figure 3), and so forth. In this way, you can interactively "take apart" a pattern to determine the compounds present in the unknown.

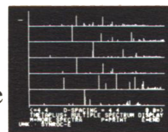
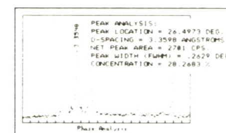


FIGURE 1

FIGURE 2

FIGURE 3

For rapid **quantitative analysis,** ThetaPlus+ will compare a single peak intensity against stored calibration curves to yield percent concentrations. Alternatively, you can compare the sum of the intensity integrals from up to 10 regions in the unknown with the sum of the corresponding integrals in the reference pattern. This increases precision and minimizes errors due to inhomogeneities and preferred-orientation effects.



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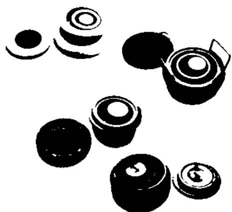

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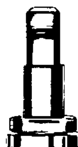

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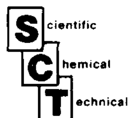

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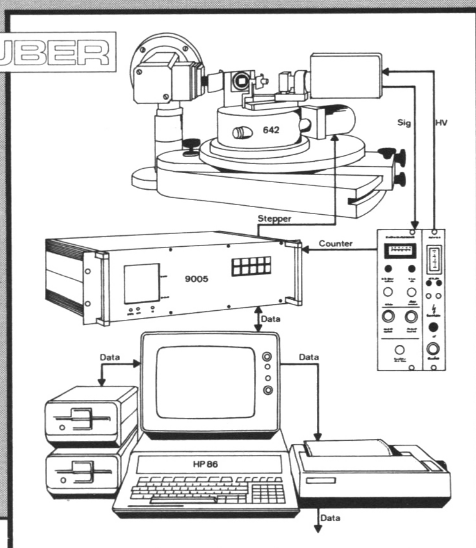
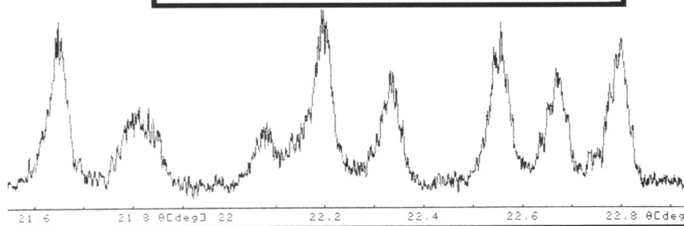
GUINIER DIFFRACTOMETER
FOR POWDER AND THIN FILM TECHNIQUE





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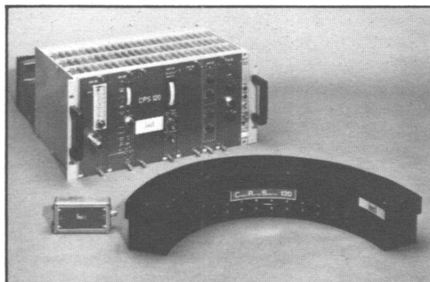

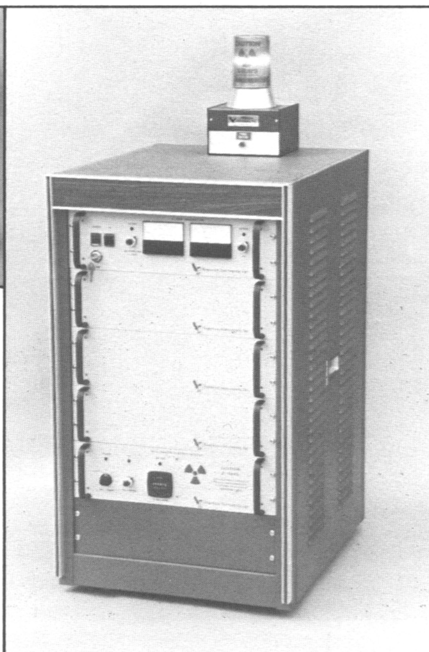

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The CPS 120 is a curved position sensitive detector system with angular range of 120° two theta. It has the capability of giving a live display of the entire two theta diffractogram on a multi channel analyzer. The output from the multi channel analyzer can then be directed to a plotter or computer for data reduction. No movement of the sample or the detector is required. This detector system can be retrofitted to an existing diffractometer or purchased as a complete x-ray diffraction system. With a spacial resolution of better than .02° and high quantum efficiency, this detector system can be used for all phases of diffraction investigations, powder, texture, kinetic study, single crystal and stress.

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Editorial

The possibility for publishing the journal, *Powder Diffraction,* was first conceived in 1981 during a meeting of the Long-Range Planning Committee of the JCPDS — International Centre for Diffraction Data. The microcomputer had made a dramatic effect in the powder diffraction field in the mid to late 1970's leading to a resurgence of interest in the use of the technique for materials analysis. Use of computer-based internal and external calibration techniques had led in turn to more accurate experimental and reference data. Because of the increasing cost of publishing, the trend was beginning among technical journals to archive primary data tables rather than include data with the published paper. At a time when there was an increasing demand for new, accurate diffraction data, these data were becoming increasingly more difficult to disseminate rapidly. It was also recognized by the JCPDS-ICDD that there was no central journal serving as a forum for developments and applications of powder diffraction methods. A survey indicated that such papers were distributed in over 300 different publications. The need for a single journal devoted to powder diffraction was apparent.

The first open discussions relative to publishing a journal devoted to powder diffraction took place at the 1981 International Union of Crystallography Congress in Ottawa, Ontario, Canada. Further discussions took place at the 1983 combined meeting for the American Crystallographic Association and the Denver Analytical X-ray Conference in Snowmass, Colorado. At this time questionnaires also were circulated, and the summary of these questionnaires supported clearly the development of such a journal. Finally, a meeting with the Commission on Journals of IUCr and an open organizational meeting were held at the IUCr Congress in Hamburg, Germany in 1984. These meetings led to the formalization of *Powder Diffraction* and the realization that the journal should expand its original goals beyond the idea of being a forum for data dissemination by including technical articles on the use of powder diffraction analysis in the characterization of crystalline materials.

The final stages in establishing the goals and scope of *Powder Diffraction* were done in close consultation with the IUCr Executive Committee and the Commission on Journals, especially S. C. Abrahams and H. L. Yakel. These discussions established a close liaison between *Powder Diffraction* and the *Journal of Applied Crystallography,* the closest technical journal in subject matter. As a result of this liaison H. L. Yakel will represent IUCr and the *Journal of Applied Crystallography* at *Powder Diffraction* editorial meetings. This close liaison is also one of the reasons why *Powder Diffraction* elected to follow the IUCr guidelines for authors with only minor modifications to emphasize the powder aspects.

The scope of *Powder Diffraction,* as stated on the title page of this issue, is to cover the many fields of powder diffraction as applied to the studies of materials. This scope includes neutron and electron diffraction as well as X-ray diffraction. The primary audience is intended to be the laboratory specialist who is involved in the diffraction experiments. Articles will range from detailed papers on the applications of powder methods (including theory, laboratory techniques, new instrumentation, and new data) to items of news in powder diffraction such as meetings and reports of JCPDS-ICDD activities. From time to time, articles of historic interest will be reprinted, partly to remind readers where our roots lie and partly to show how significant these papers are even now in our present level of knowledge. (The first reprint is the introduction of the classic method for identifying compounds by Hanawalt and Rinn in 1936.)

The relationship of *Powder Diffraction* to JCPDS-ICDD requires some explanation. As mentioned previously, the idea for the journal originated at JCPDS-ICDD meetings. All members of the organizing committee are members of JCPDS-ICDD. The JCPDS-ICDD has also provided the initial funds necessary to cover the early development and publishing costs of the journal. This support should diminish as the journal becomes self supporting through its subscriptions and advertising. The goals of *Powder Diffraction* are, however, quite distinct from the JCPDS-ICDD in that the journal is for the effective dissemination of new ideas, both in practice and theory, of the methods of powder diffraction as well the improvement of the quality of data characterizing compounds. It is inevitable that many of the members of the Editorial Advisory Board are also members of JCPDS-ICDD. However, a major effort was made to include on this Board other active diffractionists and crystallographers both from inside and outside the United States who are not members of JCPDS-ICDD. Because powder diffraction is used all over the world, it is also necessary that the journal coverage reflect this universality in the papers published. This first issue has several authors from outside North America including Japan and Europe, and more such papers are in review. Initially, the editorial offices of *Powder Diffraction* will be based at JCPDS-ICDD headquarters, and there are no plans to change this arrangement. The journal will serve both the readers and the JCPDS-ICDD by including reports of JCPDS-ICDD committee activities and announcements of new products. (Announcements of new products of all advertisers will be included.) The management of the journal, however, will be maintained independently of the management of JCPDS-ICDD such that the technical contents, editorials and other reports will reflect the ideas of the authors subject only to peer review when appropriate.

The primary goal of *Powder Diffraction* is to meet the needs of the readers. Comments from readers especially on how we can meet these needs more effectively will always be welcome.

Deane K. Smith
Editor-in-Chief

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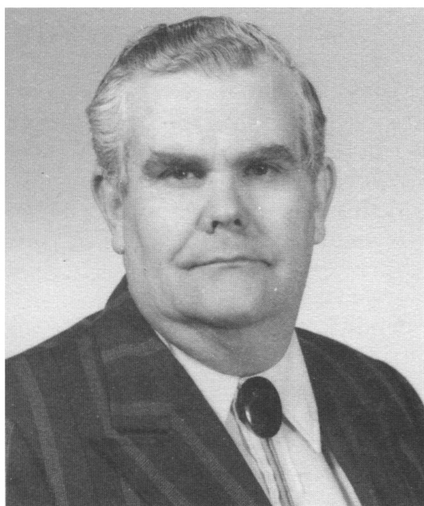
Editorial Board

Deane K. Smith
Editor in Chief

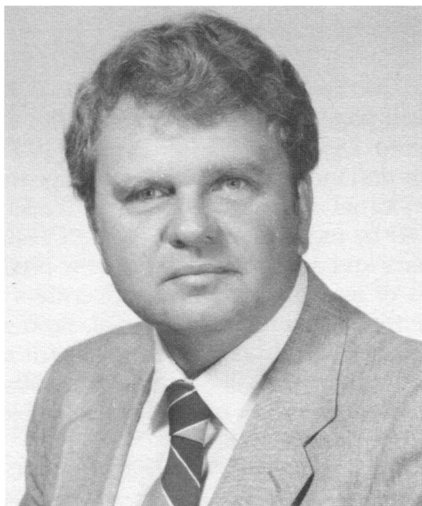


Deane Smith is Professor of Mineralogy, Department of Geosciences, at The Pennsylvania State University. He has over 30 years experience in X-ray powder diffraction of minerals, ceramics and related materials. He has published over 80 papers in fields related to X-ray diffraction. His fields of research include studies at high temperature, modeling crystal structures for powder diffraction studies and the organization of mineral X-ray data.

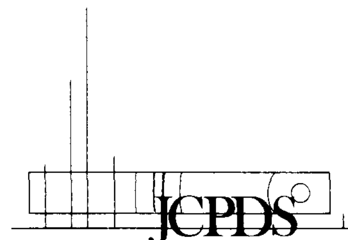
Ron Jenkins
Managing Editor

Ron Jenkins is the Principal Scientist at the JCPDS-International Centre for Diffraction Data. He has worked in the fields of X-ray diffraction and X-ray spectrometry for more than 25 years and has published over 120 papers and seven books related to these fields. He has served as Editor-in-Chief of the X-Ray Spectrometry Journal since its inception in 1972. His research and development interests have centered mainly around the development of instrumentation for X-ray analysis and he holds many patents in this area.

Ron Anderson
Departments Editor



Ron Anderson is an Advisory Physicist with IBM with over 25 years experience as a practising electron microscopist and X-ray diffractionist. His primary research interests include analysis of ceramics, thin metals films and semiconductor devices. Beside authoring numerous scientific papers, primarily in the field of electron microscopy, he recently completed a four year term as Editor-in-Chief of the EMSA Bulletin.



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Historical Development of the Powder Method

It is now fifty years since the publication of the classic paper by Don Hanawalt and Sid Rinn, describing the classification and use of a file of single phase powder diffraction patterns for the identification of mixtures of crystalline materials. This paper was first published in the 1936 Analytical Edition of 'Industrial and Engineering Chemistry', and was followed up two years later by a second paper by Hanawalt, Rinn and Frevel on 'Chemical Analysis by X-Ray Diffraction'. Although these papers were not the first to suggest that the powder method had potential as a qualitative phase analysis tool, they provide the foundation of the search/match techniques which we still employ today. Sid Rinn passed away several years ago, but both Don Hanawalt and Ludo Frevel are still very active in the diffraction field today and have both contributed enormously over the years to the development of this powerful method.

The particular volume of I&EC which carried the first Hanawalt/Rinn paper is now a collectors item, but if you are able to obtain a copy it makes interesting but sobering reading. In the same volume one finds a paper by Kothoff on the use of acid-base indicators. Over the next 20 years this work would develop into our modern techniques of electro-analytical chemistry. A paper by Vanselow and Laurance describes the use of a spectrographic determination of zinc in plant materials. Their published method was able to determine zinc at the tens of parts per million and one determination took the best part of a day. One wonders how these authors would react to the modern X-Ray Fluorescence and Ultra-Violet Emission Spectrometers, capable of analysing 30–40 elements in concentration ranges from tens of one percent to fractions of a part per million, all in less than two minutes. A paper by Marvin and Schumb describes the determination of Selenium in 18–8 type steels. They state in their summary 'the technique is fairly rapid — a series of five samples was analysed simultaneously in 1.5 hours'. Again, one wonders what the modern melt shop foreman would say if you told him to wait one and a half hours for an analysis!

In comparison with these early beginnings for some analytical methods, however, the methods described by Hanawalt and his coworkers have not changed so dramatically over the past fifty years. While it is true that our techniques have been refined and to a large extent automated, the same basic concepts proposed all those years ago have not changed, surely a tribute to the creativity and foresight of the Dow team.

In recognition of the fiftieth anniversary of the publication of the Hanawalt/Rinn paper, the Editorial Board of Powder Diffraction is pleased to reprint the paper, in its original form in this first issue of the Journal. We are even more pleased that Dr. Hanawalt has kindly consented to write a '50 years later' paper which is found in this issue of Powder Diffraction, following the I&CE reprint.

Ron Jenkins
Managing Editor

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