

Powder Diffraction

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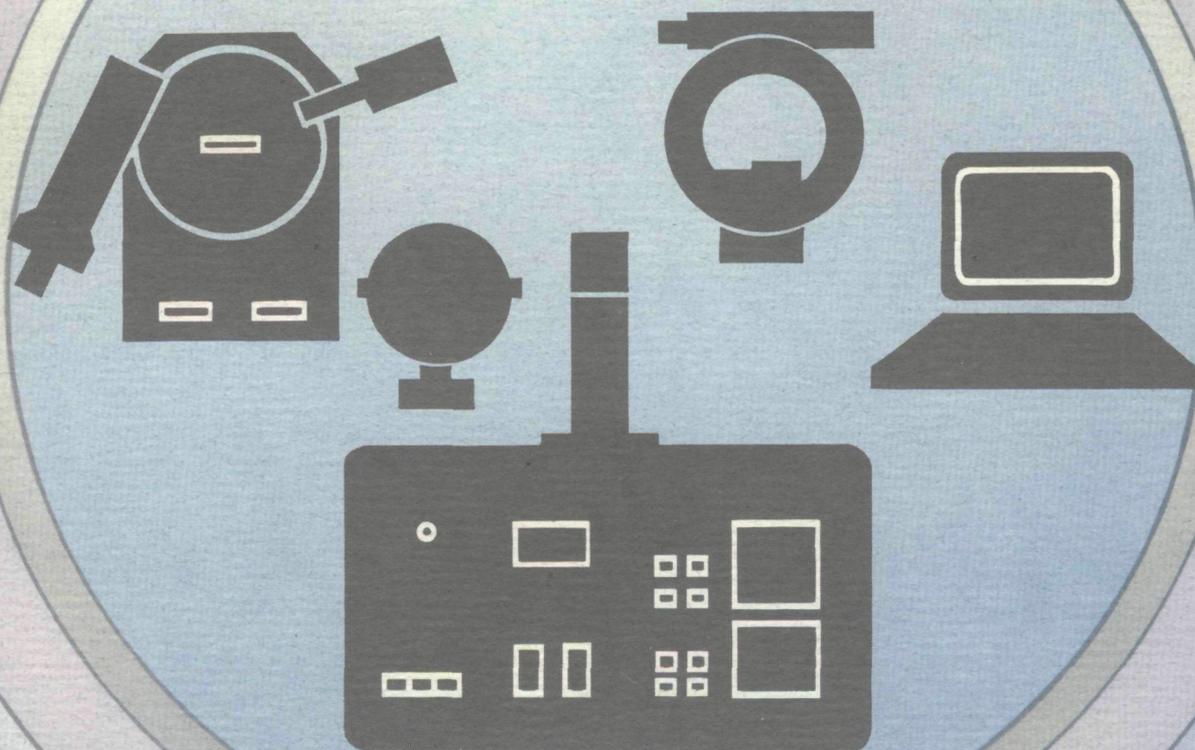
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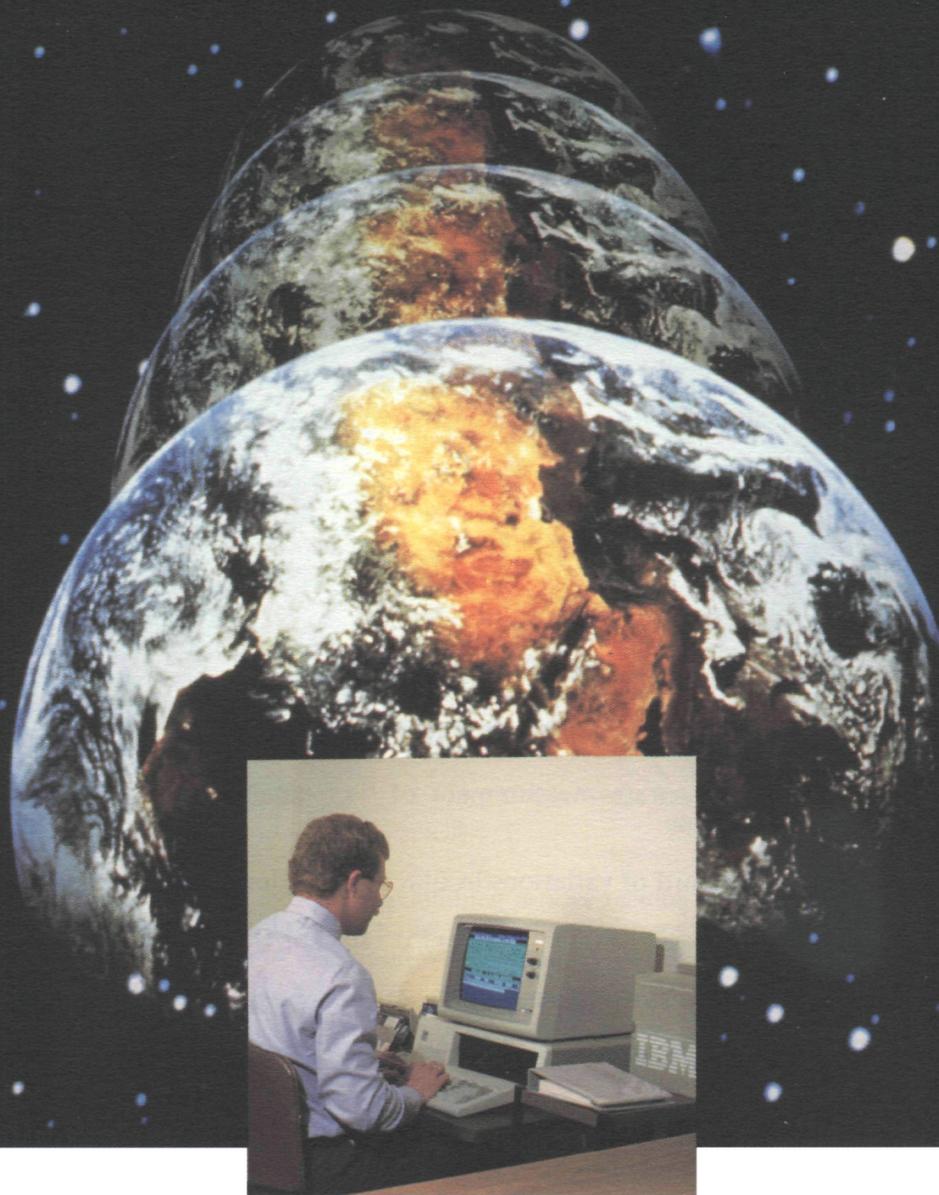
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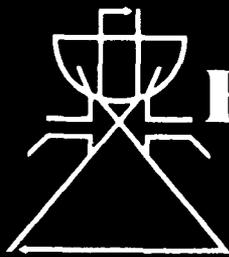
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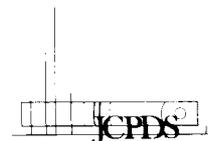
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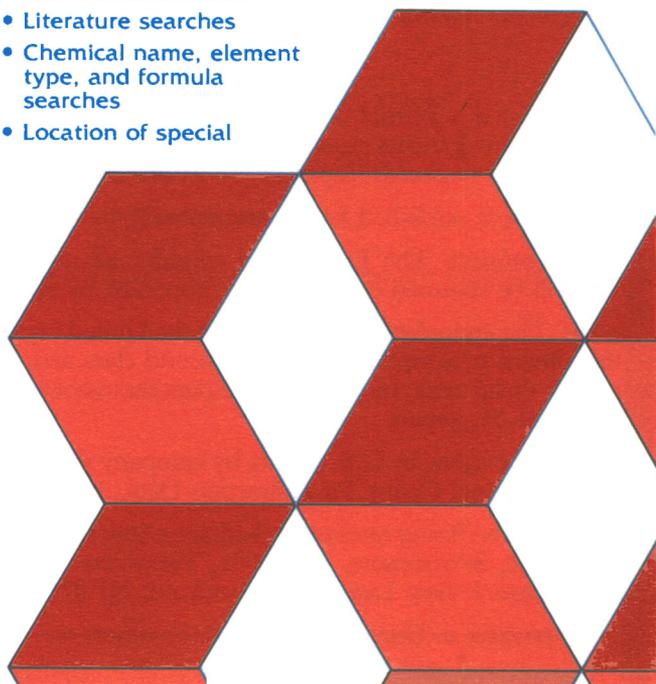
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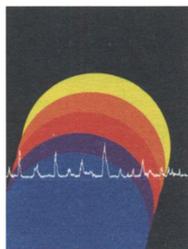
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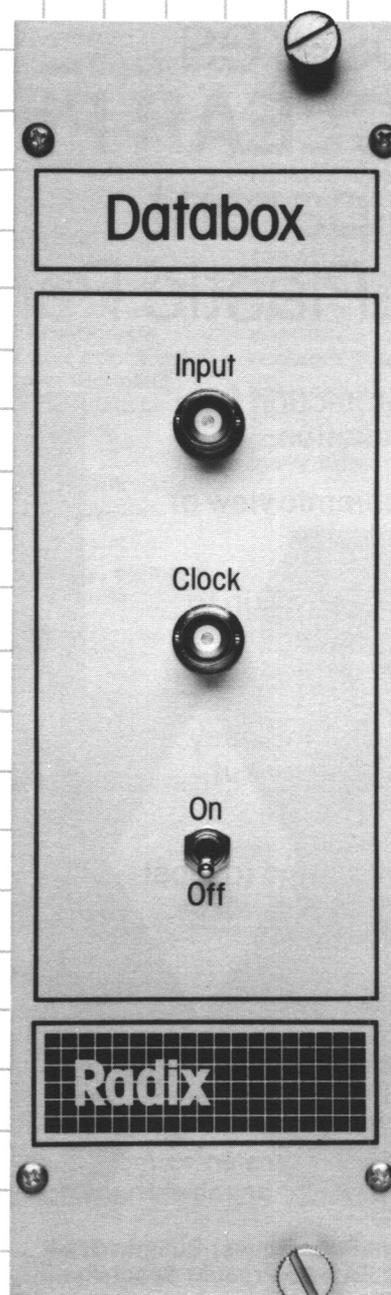
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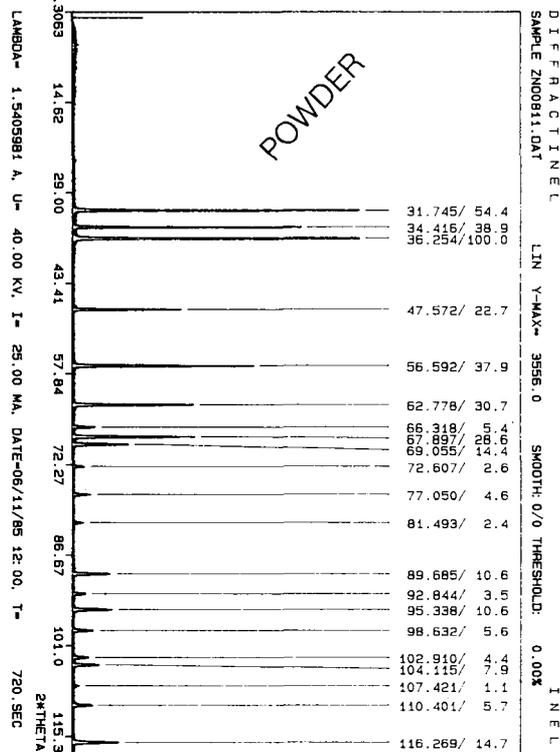
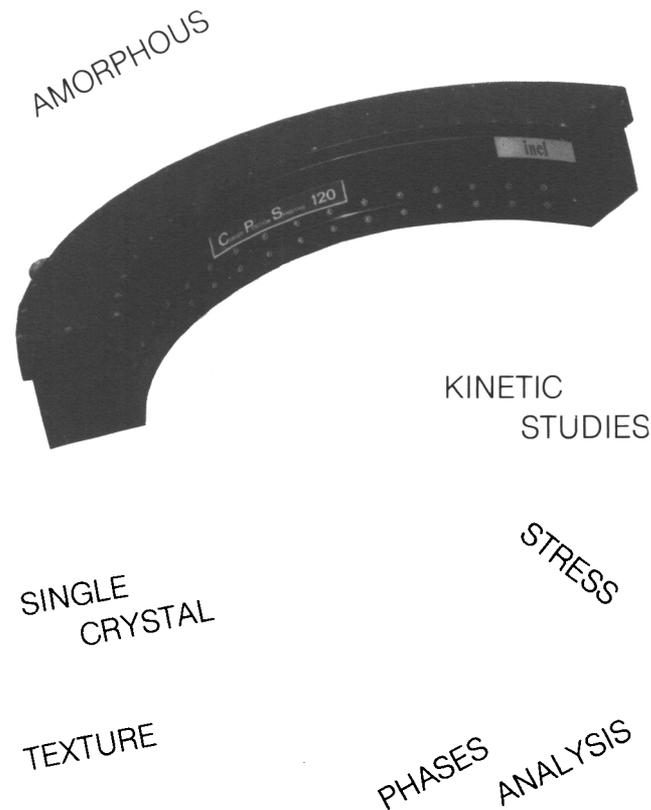
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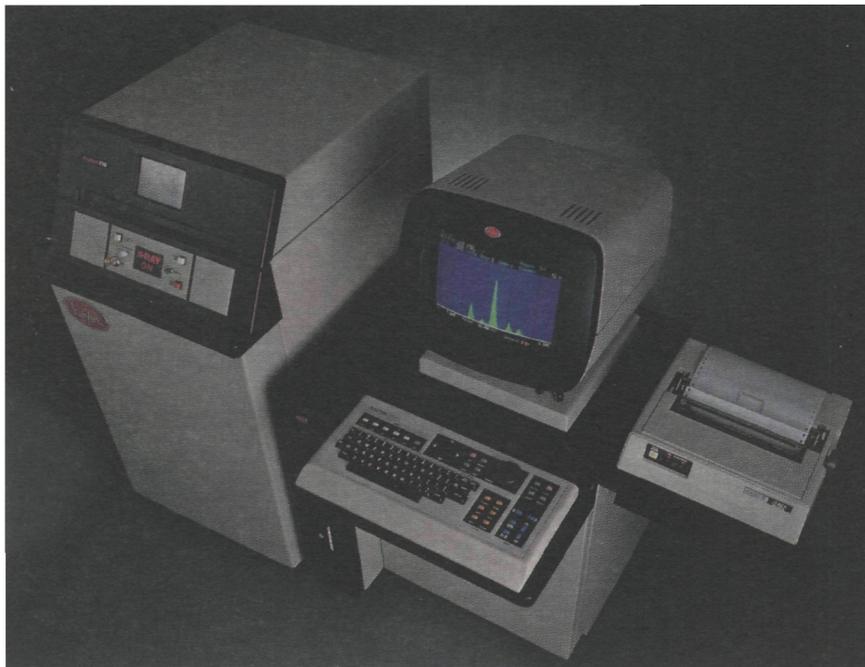
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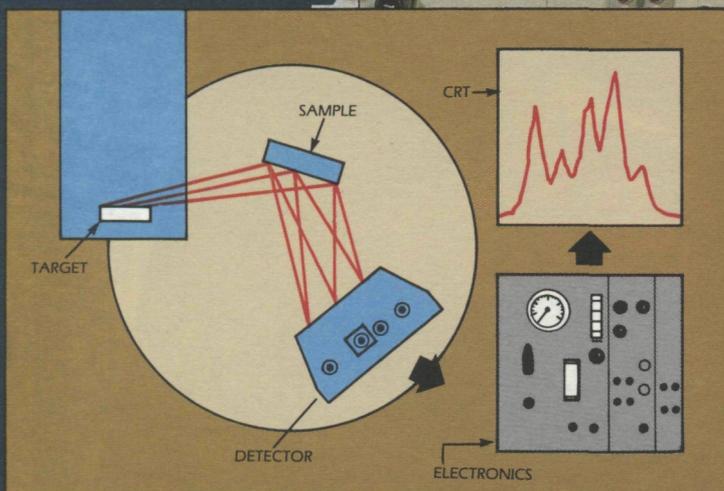
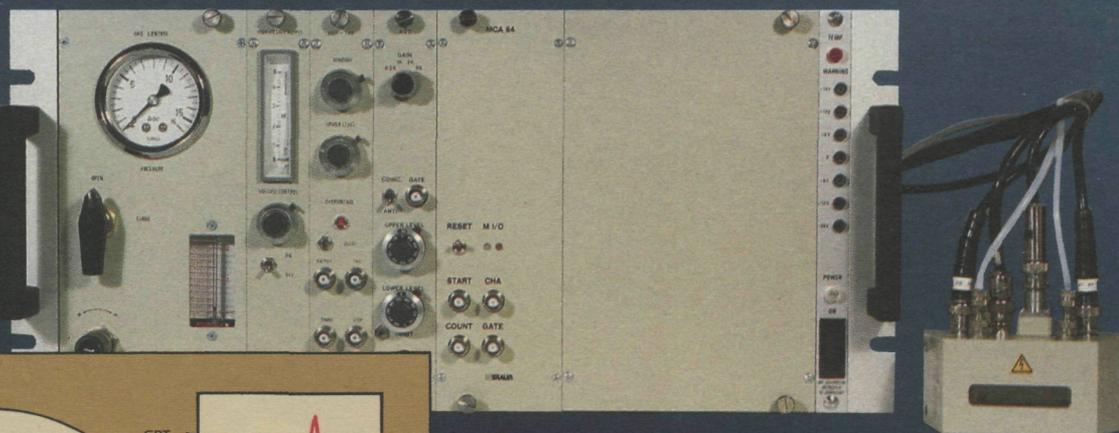
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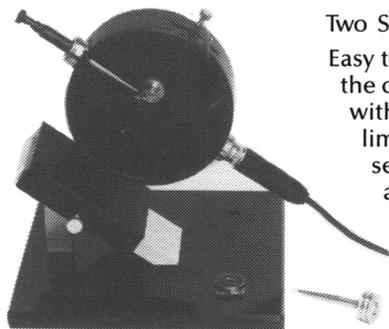
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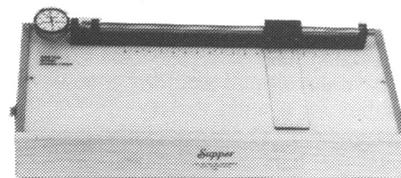


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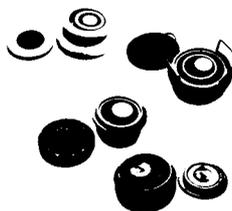
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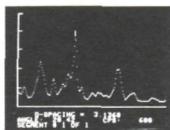
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SUMMARY OF PEAKS FOUND				
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51.63	1.7483	514.12	199	
24.55	3.5536	411.2	98	
65.71	1.4193	318.75	62	
37.86	2.3736	272.48	52	
28.13	3.1493	154.23	38	
41.7	1.9815	87.4	17	
43.82	2.8627	82.26	16	
24.99	3.5592	82.26	16	
76.4	1.2183	64.83	12	

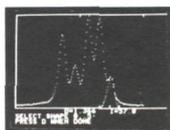
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ANALYSIS OF FILE SYNROC-E						
SEARCH PARAMETERS:						
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			OFFSET ANGLE: 0 DEGREES.			
PHASES FOUND:	LINE	FIGURES	OF	HERIT	UNIQUESS	
	FOUND	INTEN-	POSI-	OFF-	LINE	INT-
		SITY:	TION:	SET:	FOUND	RATIO
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GAZTR1207	18	.94	.72	.00	14	.20
CAT103	17	.89	.61	.00	12	.28
AL2O3	10	.75	.32	.02	6	.10x
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A high value (close to 1.0) indicates that most major lines matched.

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A low figure may indicate too wide a search window.

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,

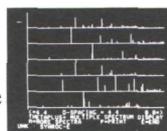


FIGURE 1



FIGURE 2

you can interactively "take apart" a pattern to determine the compounds present in the unknown.

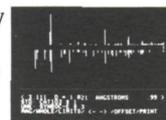
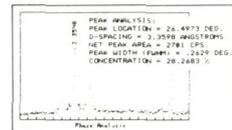


FIGURE 3

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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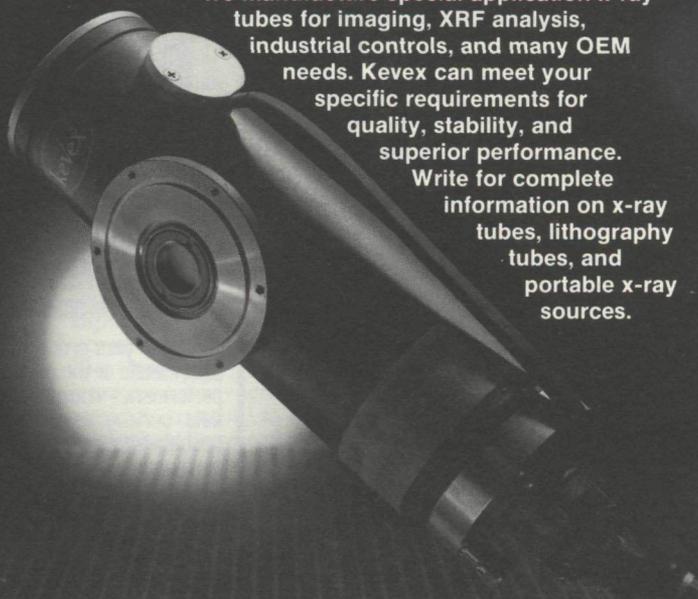
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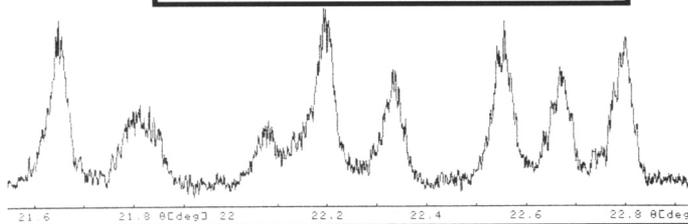
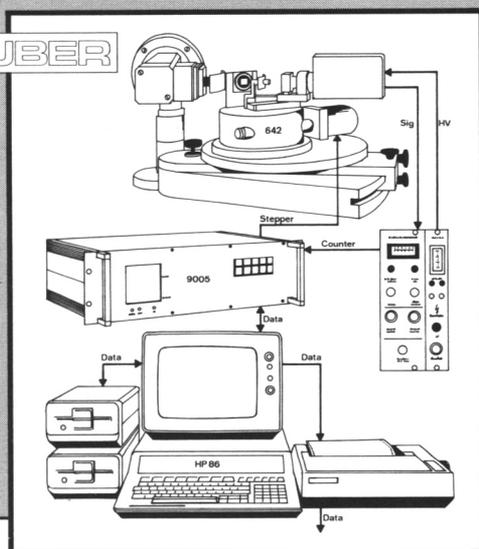
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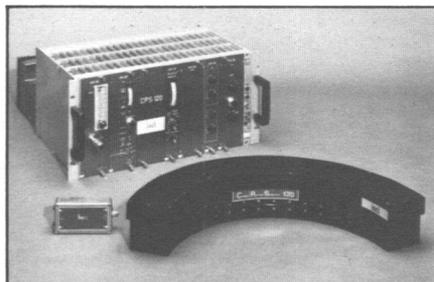
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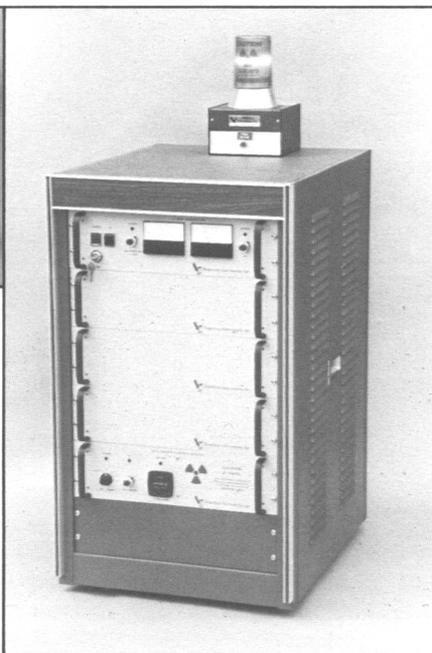
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PD21

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PD21

Powder Diffraction, Vol. 1, No. 2, June 1986

XVI

Editorial

The Status of Powder Diffraction Data

One of the incentives for initiating *Powder Diffraction* was to encourage the continuing collection and archiving of new and revised reference powder diffraction patterns. The reader may ask the question "Is the state of our powder diffraction data really a problem?" The answer is both yes and no. There are large amounts of good data available, and considerably more of moderate quality which are very useful for the identification of compounds. However, most of the latter group are relatively inaccurate and need upgrading, and there are still large numbers of known compounds whose powder data have not yet been recorded.

The Powder Diffraction File (PDF) is the largest database of powder diffraction patterns. Including set 35, the PDF contains 42299 data sets of which 30376 (72%) are classified as inorganic and 11923 (28%) are classified as organic. The inorganic number is impressive but still represents only a fraction of the known inorganic compounds. The organic coverage is an even smaller proportion of the known compounds.

Examining the patterns in the PDF on the basis of quality reveals a more accurate picture of the status of the data. Since 1945, PDF Editors have assigned quality categories for each experimental data set which are identified as *, i, b, and o in order of decreasing quality. Initially, these assignments were subjective; but since 1980, the assignments have been made objectively for patterns which are indexed. The quality evaluation is based on the deviations of the experimental diffraction angles compared with the best fit values based on the refined cell coupled with an evaluation of the intensity measurements. For the inorganic portion of the PDF, 75% are indexed and can be evaluated objectively. Around 35% are classified as * or i which is the level of accuracy generally needed for good search results using computer assisted methods. The other 65% may lead to good identifications but are harder to recognize and confirm, due to poorer figures-of-merit in the computer evaluations. For organic compounds only 16% meet the * or i quality requirements.

The statistics for the most recent PDF set (35) are also enlightening. For the inorganic patterns, 79% are indexed and 52% meet the * and i criteria. For the organic patterns 17% meet these requirements. These figures indicate an improvement over the earlier sets, but there is still considerable room for increasing these numbers even higher.

There are many ways to improve the status of powder data, but it will require the efforts of all diffractionists. The

PDF relies mostly on data which appear in the open literature supplemented by patterns submitted directly to the International Centre of Diffraction Data. Some data collecting efforts are supported by Grants-in-Aid from the ICDD. With the incorporation of more and more automated powder diffractometers in analytical and research laboratories, it is now feasible for these laboratories to collect and process very representative data sets for new compounds and to improve the data for old compounds. Unfortunately, many of these patterns do not find their way into open reports or publications which reach the public domain and ultimately the PDF. Diffractionists need to obtain the support of their employers to complete the descriptions of these data, and then need to submit them for publication. *Powder Diffraction* can help by acting as a publication medium. This is an appeal to all diffractionists to clean out your closets and old files and write up the data for a short note or add the documentation and prepare a paper on a group of related materials.

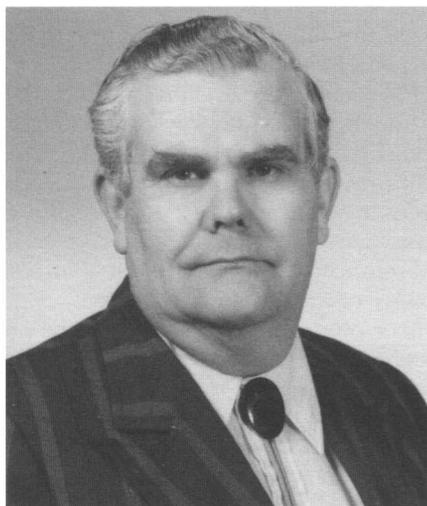
Guidelines for documentation and presentation of data were prepared by a subcommittee of the American Crystallographic Association and were accepted by the International Union of Crystallography for use in their Journals. These guidelines are included in the Notes for Authors (*Powder Diffraction* 1[i], 64, 1986). These guidelines indicate the data required to fully document the material and experimental conditions and the manner of presentation of the data. Where the materials allow this documentation, the information should be provided; but not all materials are so cooperative. For compounds such as corrosion products, precipitates in alloys, rare minerals and compounds which do not crystallize well, patterns which meet the * or i quality may not be obtainable. We have to use lower quality data for these materials. Remember that the many patterns in the PDF which do not meet the high quality standards are still the best available for those compounds. Thus, the quality should not be a deterrent to submission of the data if it is the best obtainable. Of course, the documentation should indicate the difficulties encountered.

Perhaps the proportion of good data has stabilized at around 50%, but this proportion may be an artifact of our complacency in accepting the status quo. Authors, pick up your pens and start sending in those hidden data sets.

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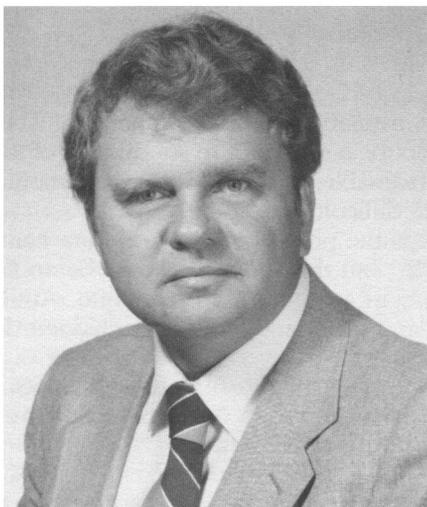
Deane Smith is Professor of Mineralogy, Department of Geosciences, at The Pennsylvania State University. He has over 30 years experience in X-ray powder diffraction of minerals, ceramics and related materials. He has published over 80 papers in fields related to X-ray diffraction. His fields of research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

Ron Jenkins
Managing Editor

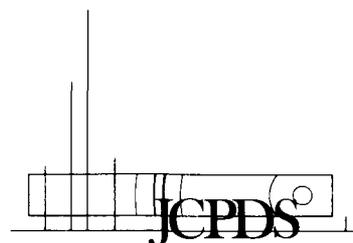


Ron Jenkins is the Principal Scientist at the JCPDS-International Centre for Diffraction Data. He has worked in the fields of X-ray diffraction and X-ray spectrometry for more than 25 years and has published over 120 papers and seven books related to these fields. He has served as Editor-in-Chief of 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.



Editor's Note and Introduction to Chemical Analysis by X-Ray Diffraction

The selection of the historical article to reprint in this issue of *Powder Diffraction* is a natural sequel to the one selected for the first issue. This paper is probably the better known of the two articles as it marked the beginning of what is now the *Powder Diffraction File*. It is also a tribute to *Industrial and Engineering Chemistry* and the Dow Chemical Company who had the foresight to financially support the publication so the 1000 recorded diffraction patterns could be distributed to other laboratories and the scientific community. Up to 1938, diffraction films were retained as the permanent record of the pattern characteristic of the compound, but this paper introduced the presentation of the data in table form (a form which is still used today.) Reprints were quickly exhausted, and the General Electric Company sponsored a second reprinting of 500 copies. Even this reprinting was insufficient to meet the demand, and in 1941 the American Society for Testing and Materials reissued the data sets on 3" × 5" cards. This reissue became Set 1 of the PDF.

The 1938 publication also provided the nucleus for the formation of a Joint Committee on Chemical Analysis by Powder Diffraction Methods. This "Joint Committee" was initially formed in 1939 under the auspices of ASTM Committee E-4 and the National Research Council. The British Institute of Physics soon joined these two organizations, and the American Society for X-ray and Electron Diffraction (now the American Crystallographic Association) replaced the National Research Council. Subsequently, many other scientific and industrial organizations have affiliated with this "Joint Committee".

The initial role of the Joint Committee was to approach industrial and scientific organizations for support to continue the publication of diffraction data and to initiate a structure for overseeing this activity. W. P. Davey established the first editorial office at the Pennsylvania State University in 1940. This operation subsequently moved to downtown State College, Pennsylvania, then to ASTM headquarters in Philadelphia. In 1969, the operation incorporated as the Joint Committee on Powder Diffraction Standards which was located first in Philadelphia and presently in Swarthmore, Pennsylvania. This non-profit corporation is presently known as the JCPDS-International Centre for Diffraction Data.

The authors of this paper have all been very important in Joint Committee activities. J. D. Hanawalt was always analyzing and improving the search methods and remains to this day one of the best at using the PDF for compound identification. He was a member of the Board of Directors of JCPDS for many years and was its Chairman from 1975 to 1978. Even today he maintains his interest in the search procedures, and it was only in 1982 that he stepped down as chairman of the Search Procedures Subcommittee. His continued interest is illustrated by his paper in the first issue of *Powder Diffraction*. H. W. "Sid" Rinn was JCPDS Chairman from 1956 to 1961 and retained an active interest until his death in 1967. L. K. Frevel is currently a Member-at-large on the JCPDS Board of Directors. He received the first J. D. Hanawalt Award in 1983 for his outstanding contribution to powder diffraction analysis.

The significance of the 1938 paper and the quality of the data is indicated by the status of the present PDF. The *Hanawalt Search Manual* still uses the basic concepts described in the 1936 and 1938 papers, employing the three strongest diffraction lines for the search procedure. Many suggestions have been offered over the years to improve the efficiency of searching, but they are minor compared to the initial concept of grouping the data to minimize the number of steps of finding the appropriate data set. The data were also quite good especially considering that they were all obtained using film techniques. Almost one half of the 1000-Set 1 patterns are still active patterns in 1986. Their average figure-of-merit is essentially the same as for the later sets. One has only to read the paper to realize the care that was used in obtaining these data.

Powder Diffraction has reprinted only the text and the table of substances from the original document. For those diffractionists who are familiar with this paper, its significance is already acknowledged. Those who have never read this article before will quickly recognize its role as one of the really important contributions to the field of powder diffraction analysis.

Deane K. Smith
Editor-in-Chief