Powder Diffraction

Volume 3 Number 2 June 1988
Powder Diffraction An international journal of materials characterization

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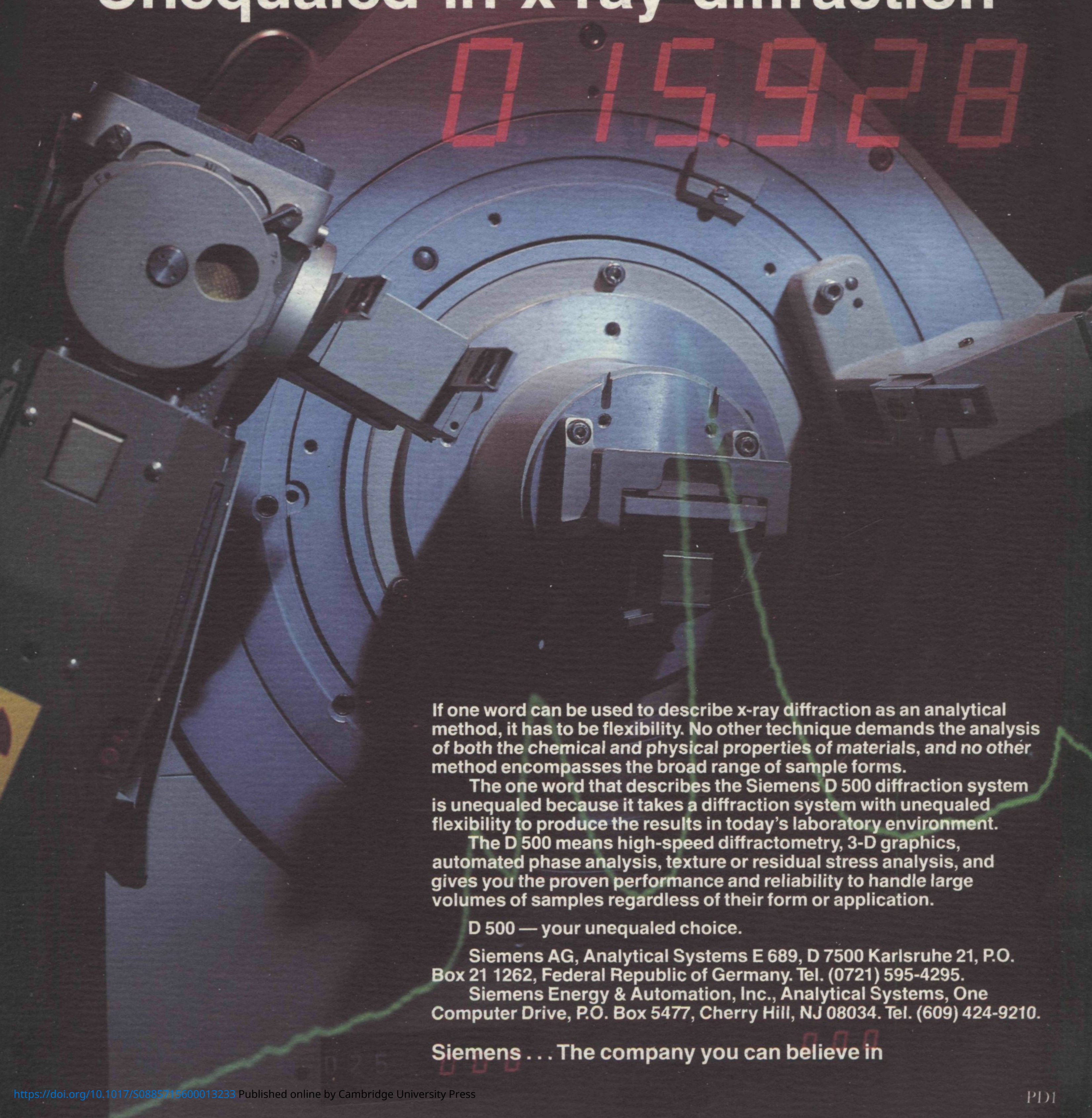

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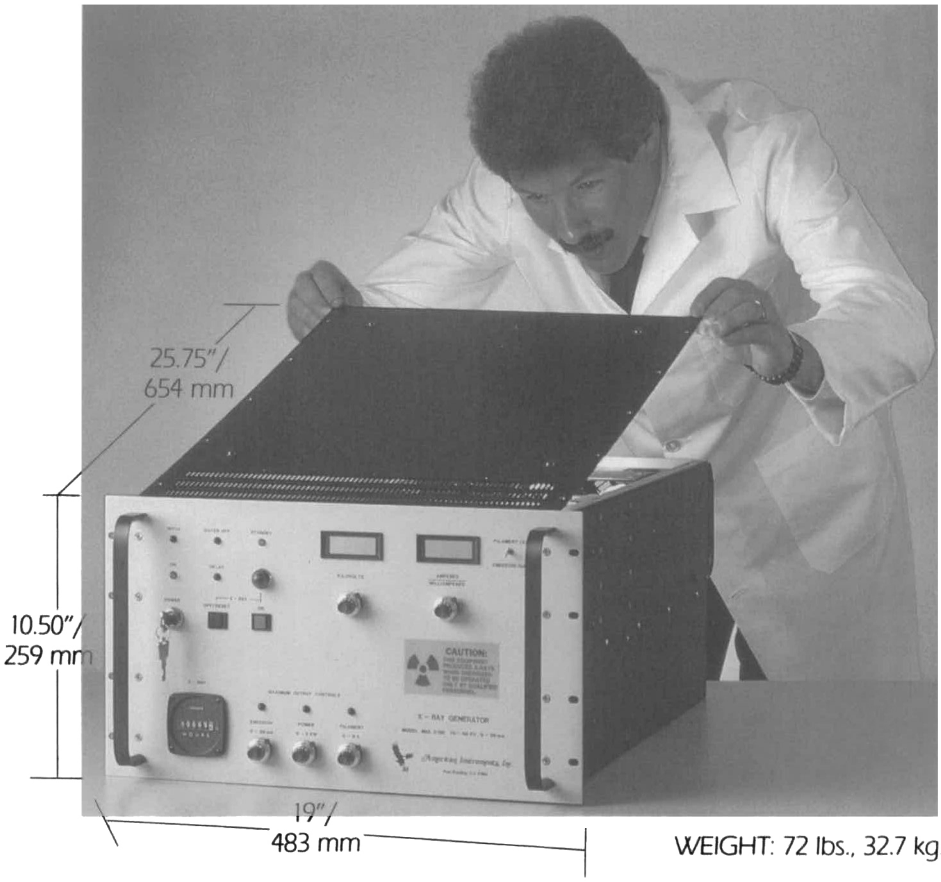

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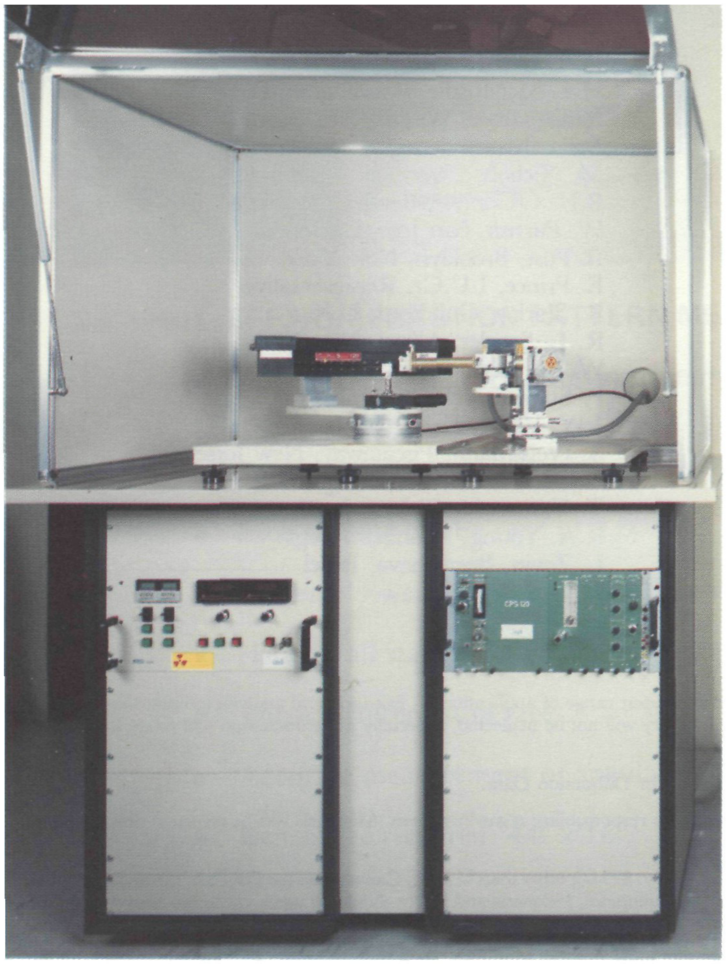

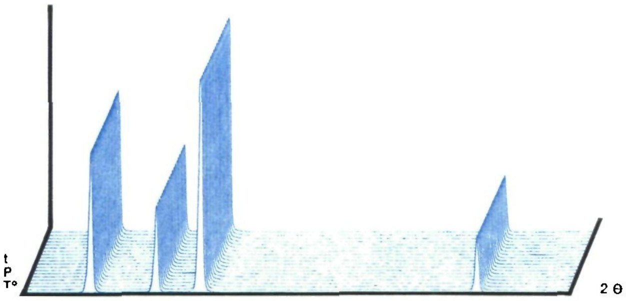
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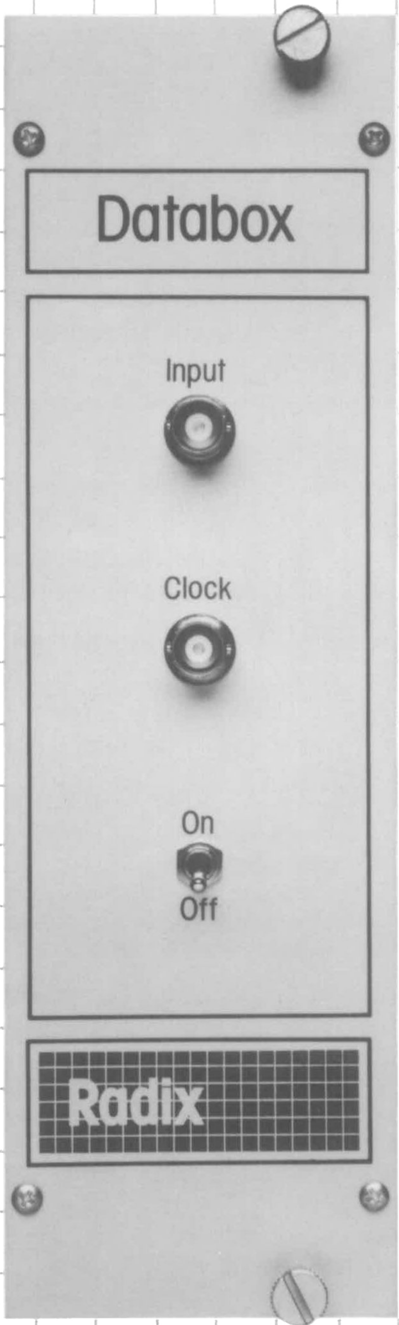
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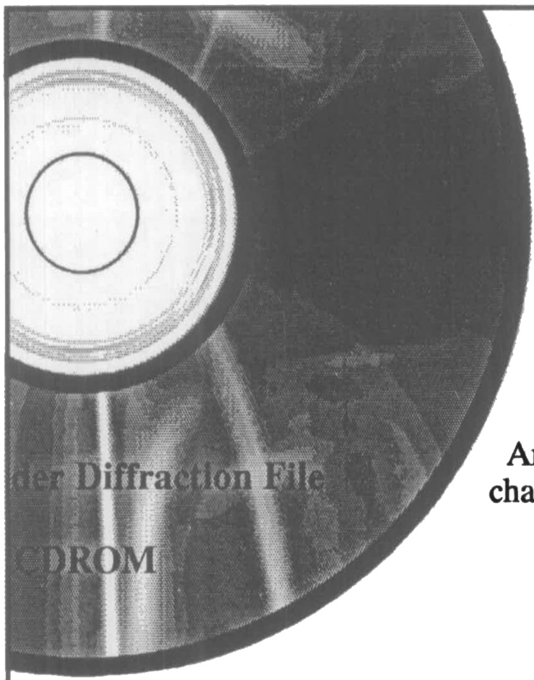
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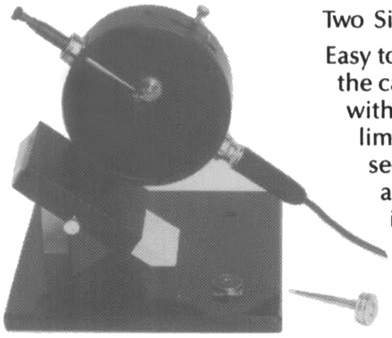

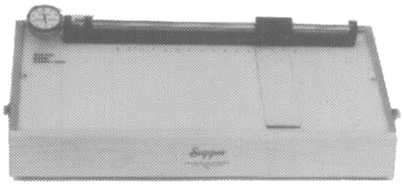

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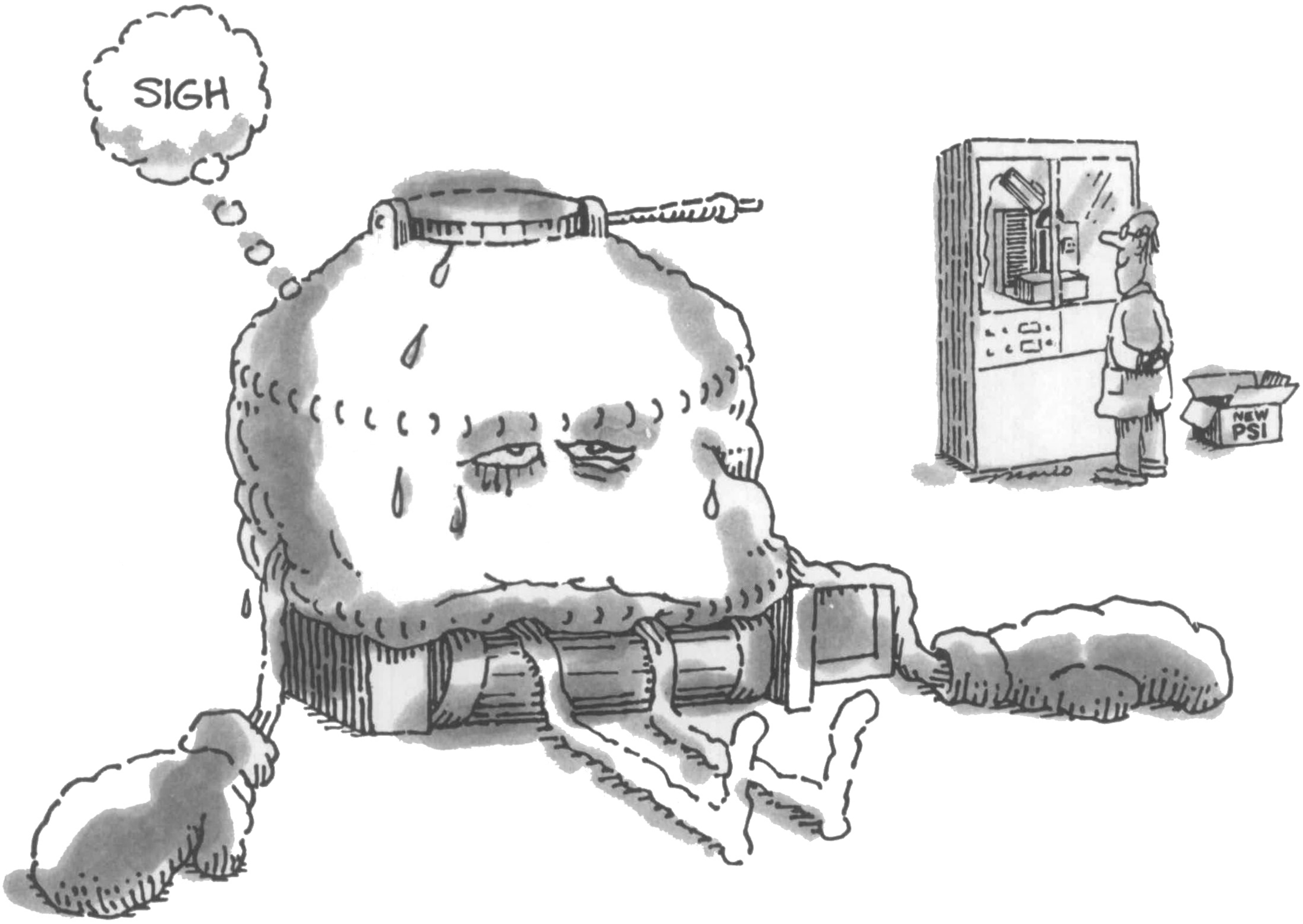

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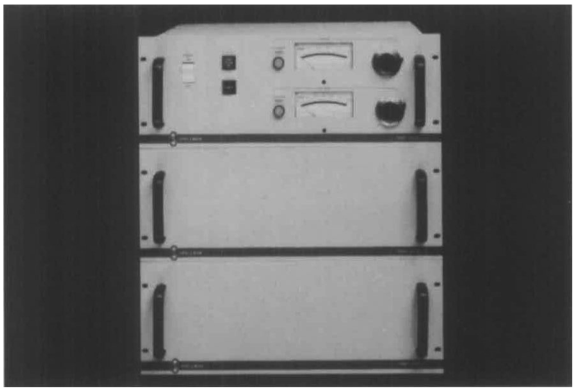

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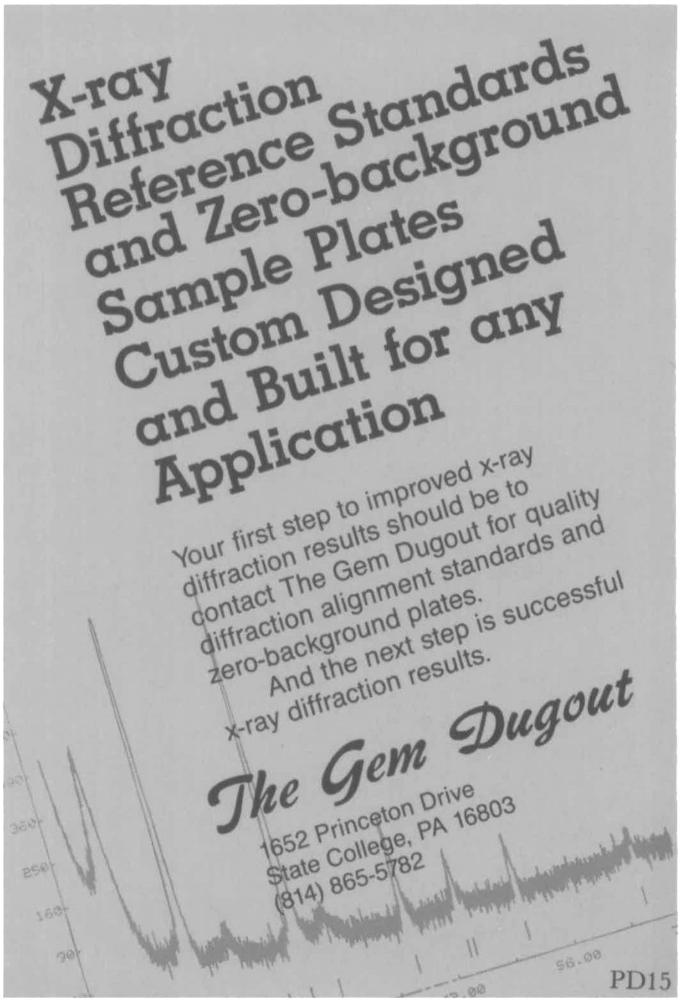

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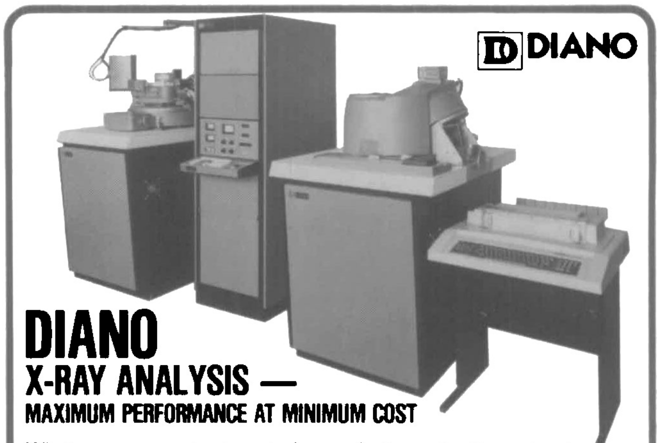

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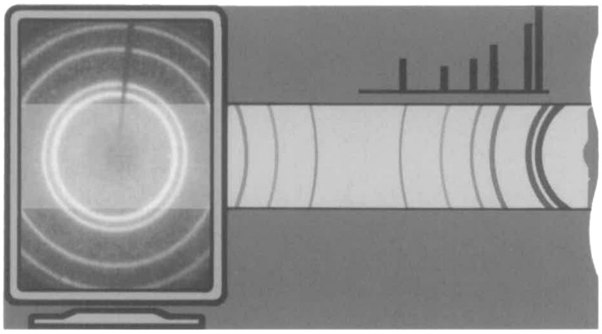

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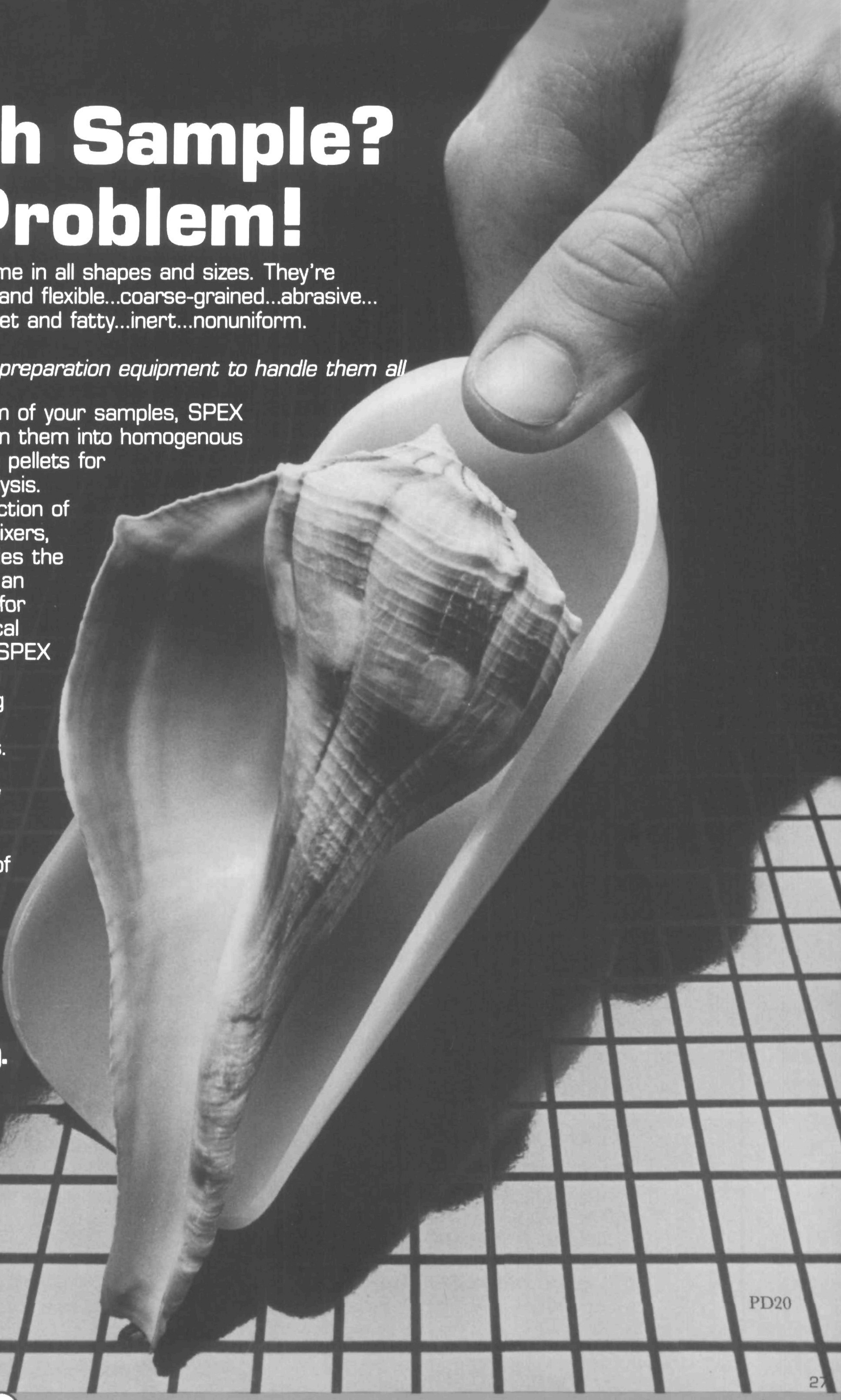

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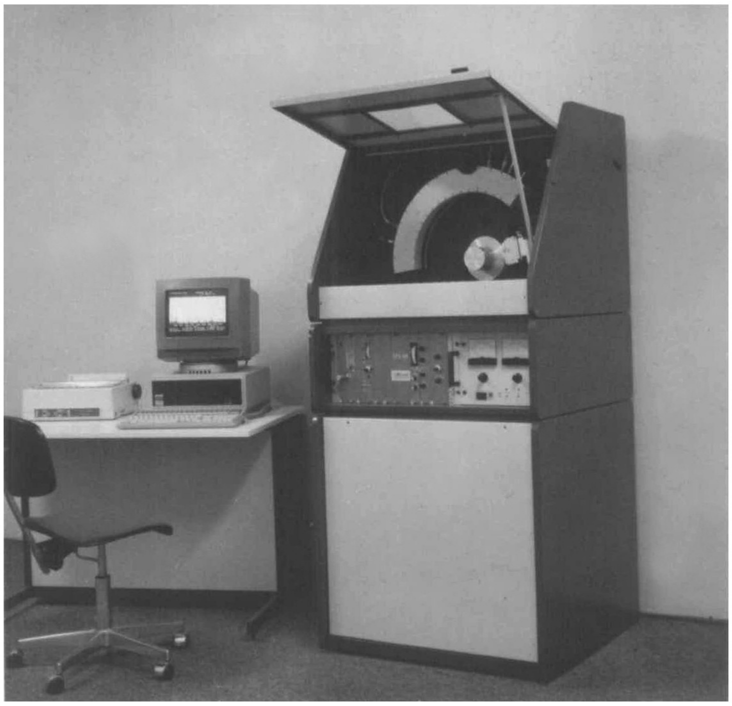

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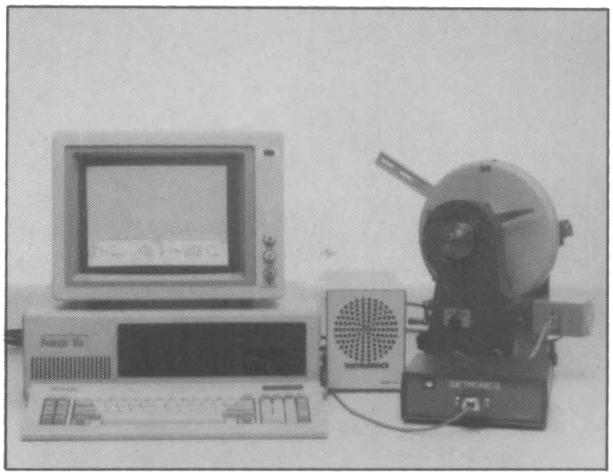

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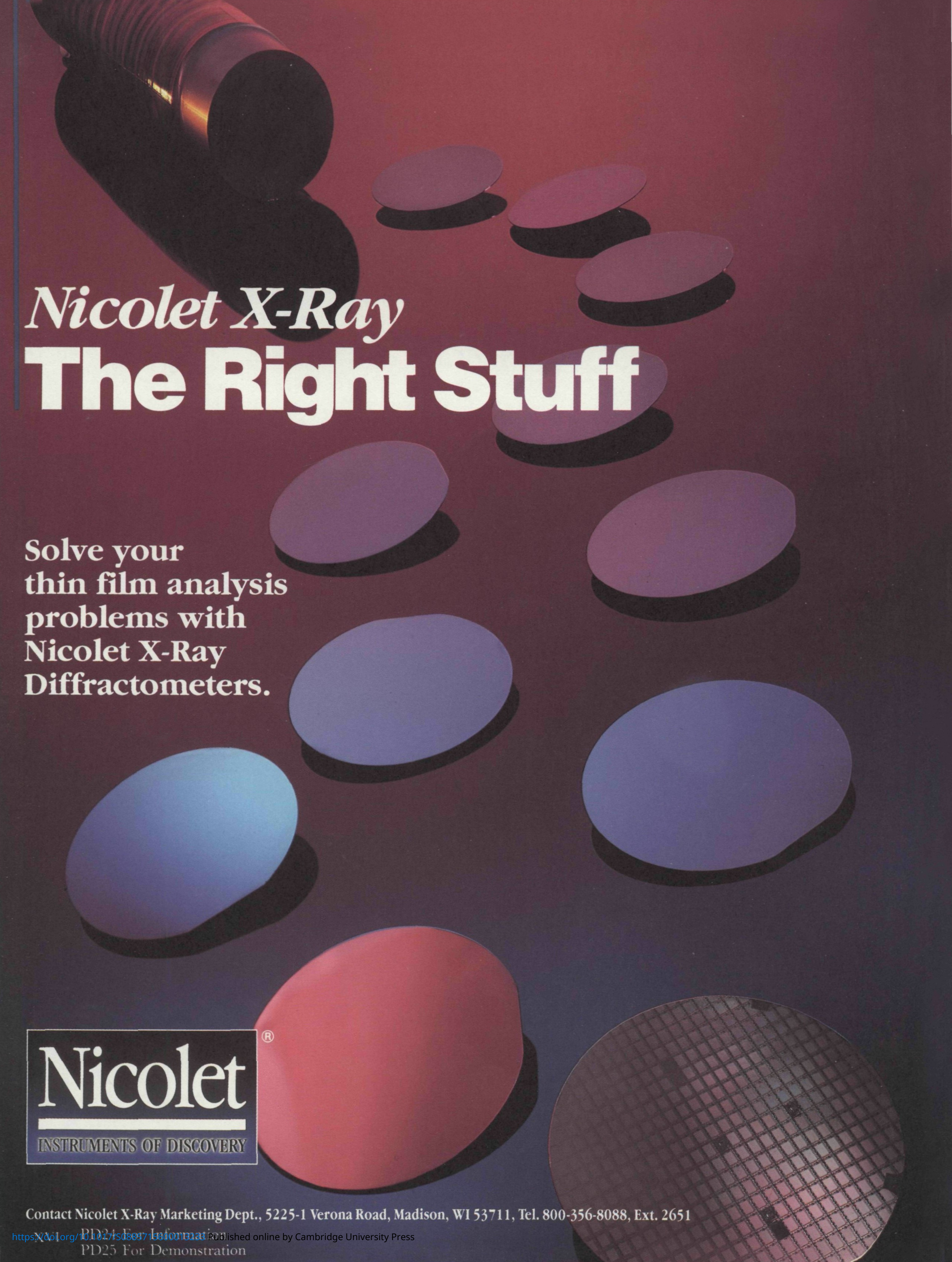

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XA1 PD24 For Information
PD25 For Demonstration

Editorial

Phase Identification by Electron Diffraction

Analytical electron microscopy (AEM) is a fertile field for the further application of technology developed in the X-ray laboratory by the extension of phase identification methods and resources to smaller crystals. Selected area diffraction, microdiffraction, and convergent beam electron diffraction (CBED) provide the means to obtain d-spacings, angles, and symmetry information from objects ranging in size from 10 Å to more than 10,000 Å. Concurrent X-ray fluorescence analysis is possible to the 100 Å scale. With a goniometer stage capable of tilting ± 45°, it is convenient to observe large segments of reciprocal space and locate prominent zones or to measure interzonal angles in very small crystals. The size scale accessible by the AEM is ideally suited for important commercial, scientific, and forensic enterprises. The information obtained is often complementary to that obtainable by X-ray diffraction and the combination results in a very complete understanding of a microstructure, a materials system, a mineral, or a device.

Many of the principles of compound identification by search/match methods developed for X-ray powder diffraction and single-crystal analysis can be carried forward to this new domain, but there are important differences that must be addressed before successful, objective identifications become routine and efficient. The precision of electron diffraction measurements is relatively low, and intensities are not often useful for identifying unknowns. Multiple diffraction effects can often obscure characteristic systematic extinctions in some patterns from some crystals. Significant improvements are unlikely because the limitations spring from basic physics. The prospects for identification are far from hopeless, however, because there are compensating strengths to the technique as well. Mixtures rarely need to be considered, because the spatial resolution of the technique is high. High quality compositional information as well as crystallographic data can be obtained from the same crystal, greatly reducing the number of unknowns that must be closely considered during a search process. Essentially all of the reciprocal space of a crystal can be examined, providing easy access to the most diagnostic, low index reflections. Interplanar angular information is immediately available from a typical zone axis pattern, and 3-D information can be obtained from related pairs of zone axis patterns or wide angle CBED patterns. The fine structure of CBED patterns can also be used to obtain point group and space group information.

Extensive, critically evaluated, and up-to-date databases of both powder data and single crystal data have been developed over the years to support the X-ray diffraction community. Great effort has been invested in developing manual and machine methods for searching through these resources in order to match the characteristics of an unknown phase to a previously reported phase or else to know with confidence that the unknown is new to science. These same resources can also be of great value to the AEM community.

During the past year, the NBS/Sandia/ICDD Electron Diffraction Database was derived from both the JCPDS/ICDD PDF-2 and NBS CRYSTAL DATA, and augmented by computations at NBS, to provide a single, comprehensive, computer based resource for phase identification, tailored specifically to electron microscopy applications. The Max-d Alphabetical Index now in preparation is further derived from the NSI database and will provide a resource for manual search/match methods of the same type. Both benefit from the careful editing and dedicated database management of the parent databases, without burdening either parent with format extensions or changes. A unique data format allows the compression of data for over 71,000 entries into ~8.5 Mbyte, making the NSI database applicable to common laboratory computing equipment. Rudimentary FORTRAN source code developed at Sandia National Laboratories to implement our novel electron diffraction search/match algorithm is distributed freely with the database to illustrate basic access operations, in the hope that it will seed development of many, varied search programs. The data contained in the NSI database will facilitate searching on the basis of composition, diffraction data, unit cell data, and reduced cell data, and/or symmetry information. The JCPDS/ICDD will announce the availability of these new resources directly to the AEM community at the joint meeting of the Electron Microscopy Society of America, the Microscopical Society of Canada, and the Microbeam Analysis Society in Milwaukee on August 7-12, 1988.

The creation of these new resources is the result of hard work by individuals from government laboratories, corporations, and universities, all working through the Technical Committee of the JCPDS-ICDD, but the work is far from finished. The challenge remains to nurture them, to bring them to the attention of the users who can benefit from them. Many search/match techniques are possible but not implemented in the demonstration software, since it is the policy of the JCPDS to concentrate on the quality of the database itself and to leave the development of specific techniques and software to the academic, industrial, and commercial users who are in the best position to explore and to evolve that which best suits their needs. As with any new tool, the possibility for unforeseen applications is real. The Phase Identification by Electron Diffraction Subcommittee, a standing unit of the JCPDS Technical Committee, exists to guide the development of these new resources and respond to the needs of users as they become known. The subcommittee is actively recruiting members and invites anyone with a professional interest in the scientific, educational, or commercial opportunities to participate.

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