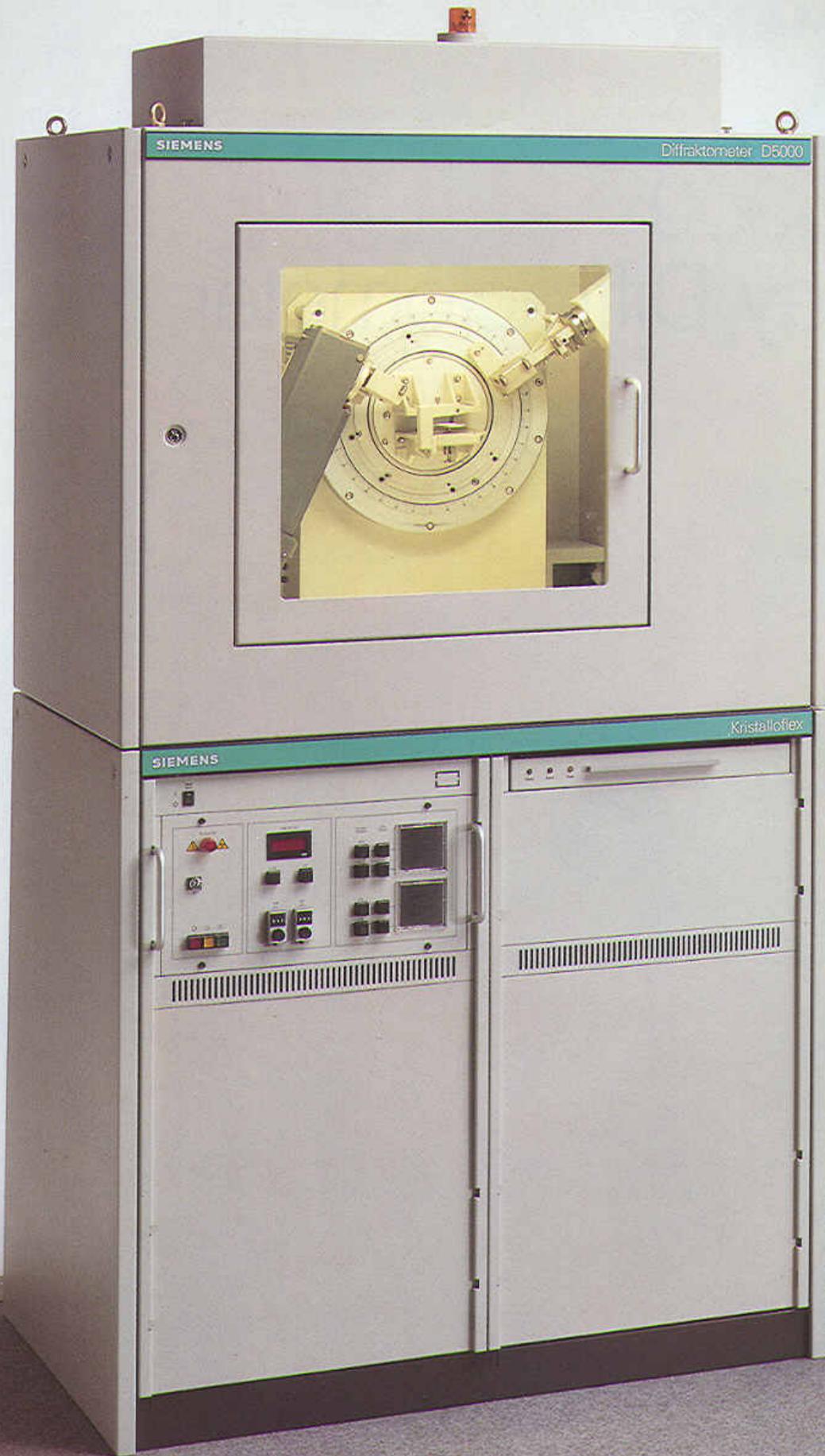


SIEMENS

D 5000
X-ray Diffractometer





SIEMENS

Diffraktometer D5000

SIEMENS

Kristalloflex

The D 5000 X-ray Diffractometer

Combining the latest technology in a modular design, the new D 5000 brings expanded capabilities to your lab, providing:

- High sensitivity with rugged construction;
- Fast results with high reproducibility;
- A modular design that can be optimized for specific applications with optional attachments.



The Heart of the D 5000:

A goniometer precisely engineered for exact measuring results, yet flexible to accommodate a variety of tasks.

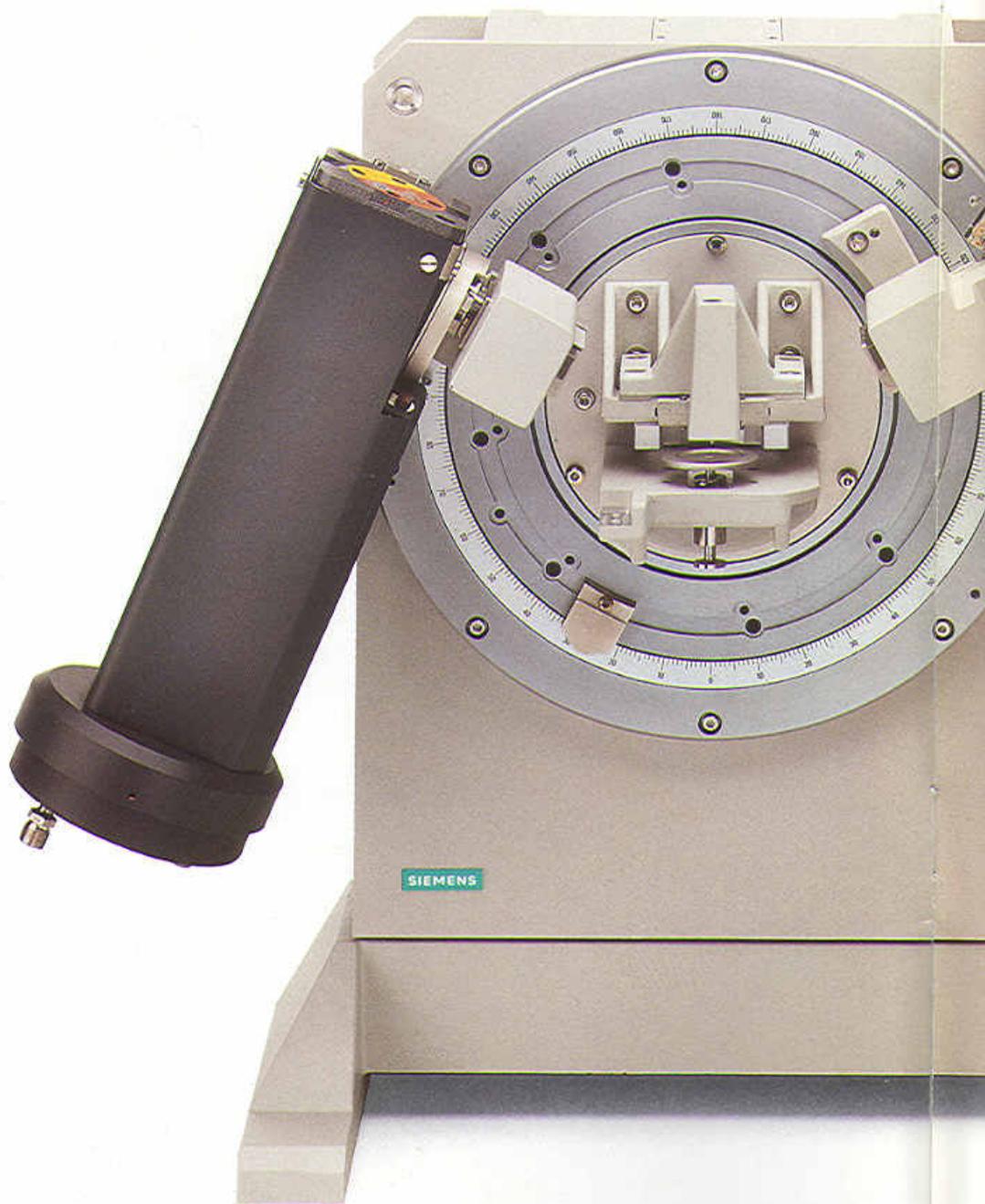
The basic module of the D 5000 is the goniometer. Its new, high-precision design features two independently controlled stepping motor drives. Representing the ultimate in flexibility, it operates in both horizontal and vertical modes (with the tube stand flanged to the goniometer housing for easy change-over), and in either Theta-2Theta or Theta-Theta geometry.

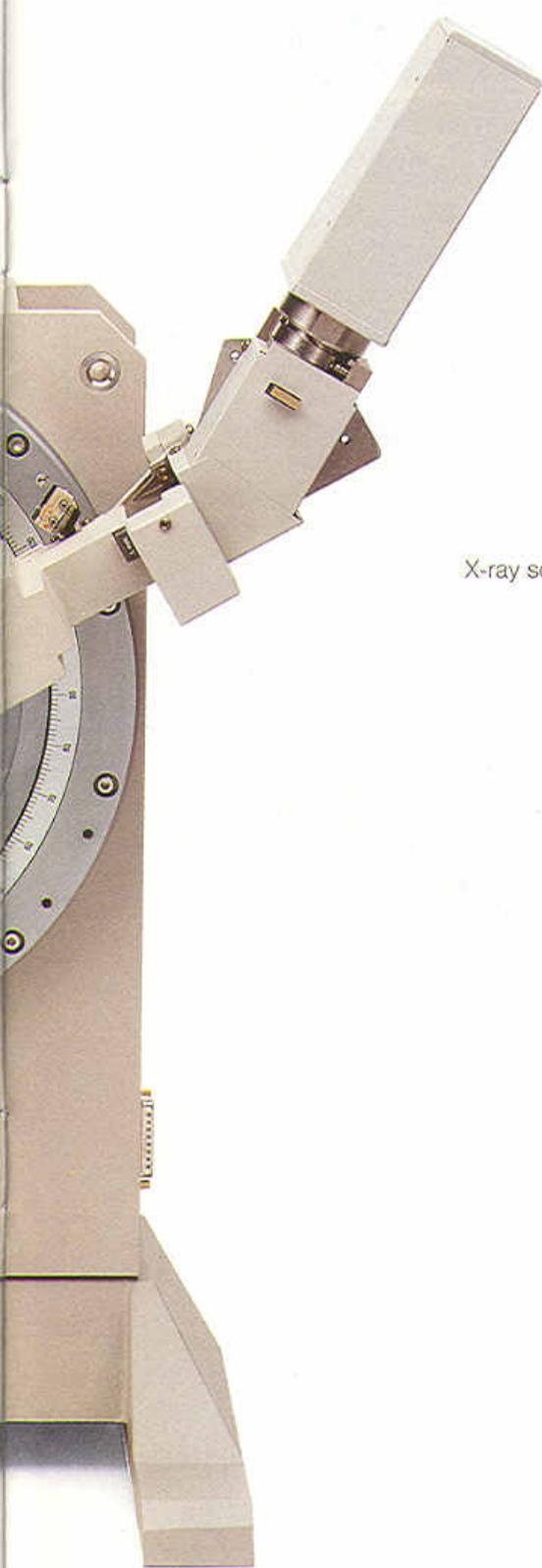
The open goniometer architecture forms the basis for a wide range of automation possibilities and supports a variety of applications. To adapt the unit to these applications, various attachments have been developed:

- Stepping-motor operating variable incident and diffracted beam slits,
- Automatic detector slit changer,
- Various measuring circle diameters, selectable from 401 to 600 mm,
- Reflection and transmission sample holder which can be freely rotated or set to specific positions,
- Automatic sample changer with sample rotation for up to 40 samples
- Focussing primary beam monochromators for transmission or reflection geometries,
- Focussing secondary beam monochromators,
- Various detector systems, including scintillation counters, proportional counters, PSD's (position sensitive detectors) and semi-conductor detectors.

For maximum reliability, the D 5000 is mechanically pre-aligned at the factory. After installation of your system only a computer assisted, automated final alignment that takes only a few minutes is required. Difficult alignments of the tube stand, sample changer and detector have been totally eli-

minated. Hence, the goniometer can also be used in a configuration with a vertical tube stand. Plus, the new zero-backlash sample changer interface permits the free exchange of accessories with reproducible exact positioning.



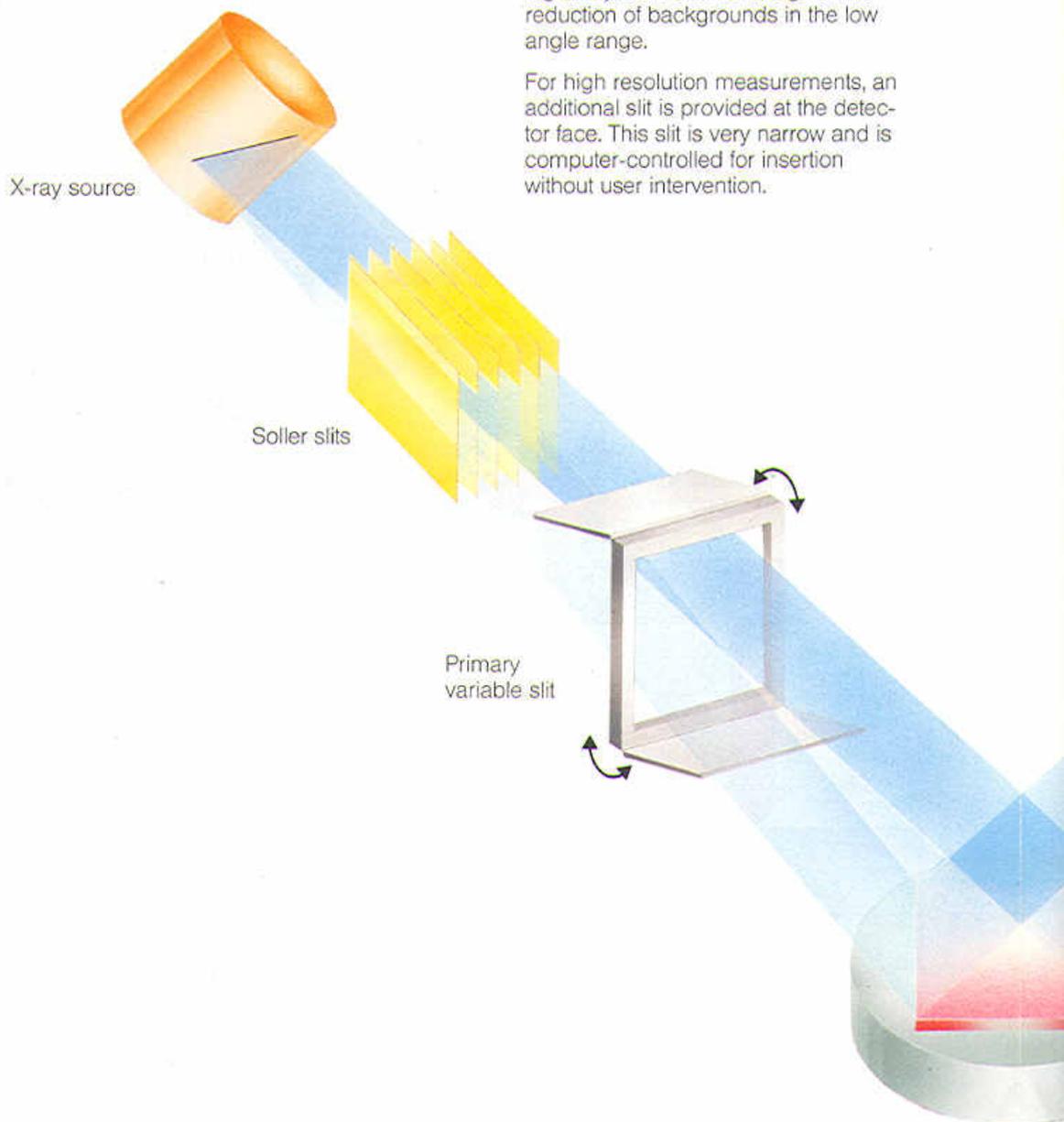


Add-on modules offer total flexibility in configuring the D 5000 for specific applications without compromising the integrity of design and ease of use of the basic system.

Automated Slit Assemblies

Variable aperture, stepping motor controlled incident- and diffracted-beam slits are the keys to optimum measuring results, providing rapid adjustment of incident beam divergence and collimation of the diffracted beam for reduction of scattering effects, all under computer control. These assemblies permit automatic Theta-coupled adjustment of the slits as well as fixed, computer-controlled width selection. This allows fast and easy adaptation of the goniometer to varying analytical conditions, e.g. for the reduction of backgrounds in the low angle range.

For high resolution measurements, an additional slit is provided at the detector face. This slit is very narrow and is computer-controlled for insertion without user intervention.



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Sample Mounts

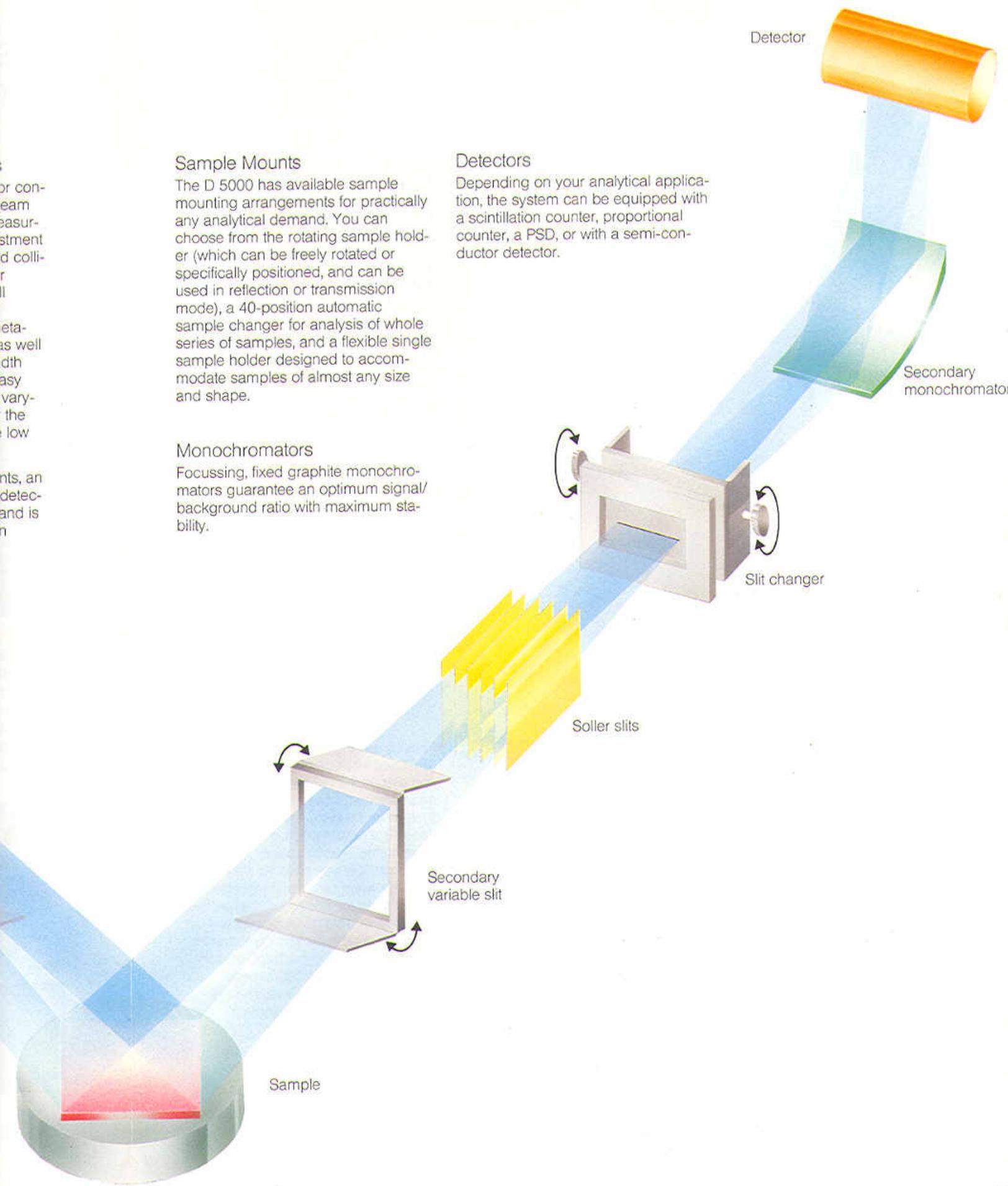
The D 5000 has available sample mounting arrangements for practically any analytical demand. You can choose from the rotating sample holder (which can be freely rotated or specifically positioned, and can be used in reflection or transmission mode), a 40-position automatic sample changer for analysis of whole series of samples, and a flexible single sample holder designed to accommodate samples of almost any size and shape.

Monochromators

Focussing, fixed graphite monochromators guarantee an optimum signal/background ratio with maximum stability.

Detectors

Depending on your analytical application, the system can be equipped with a scintillation counter, proportional counter, a PSD, or with a semi-conductor detector.





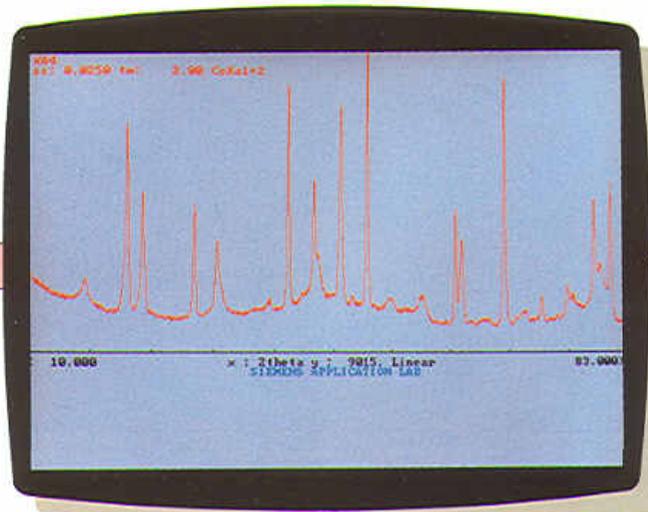
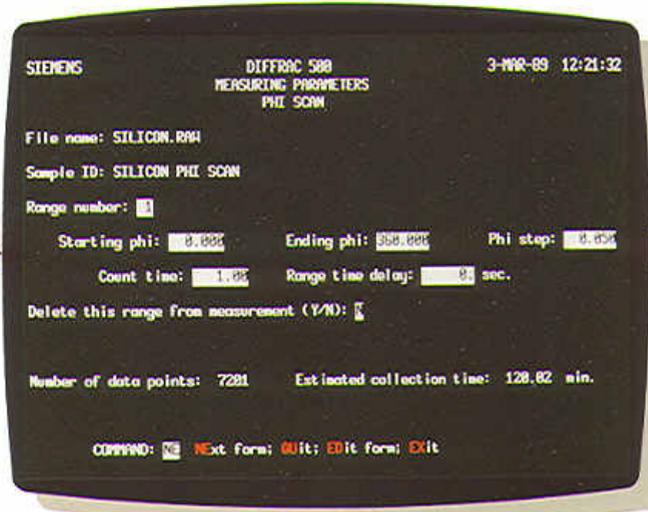
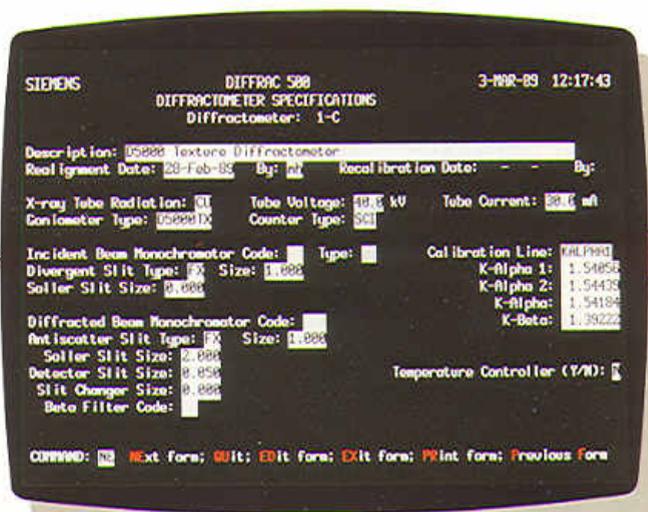
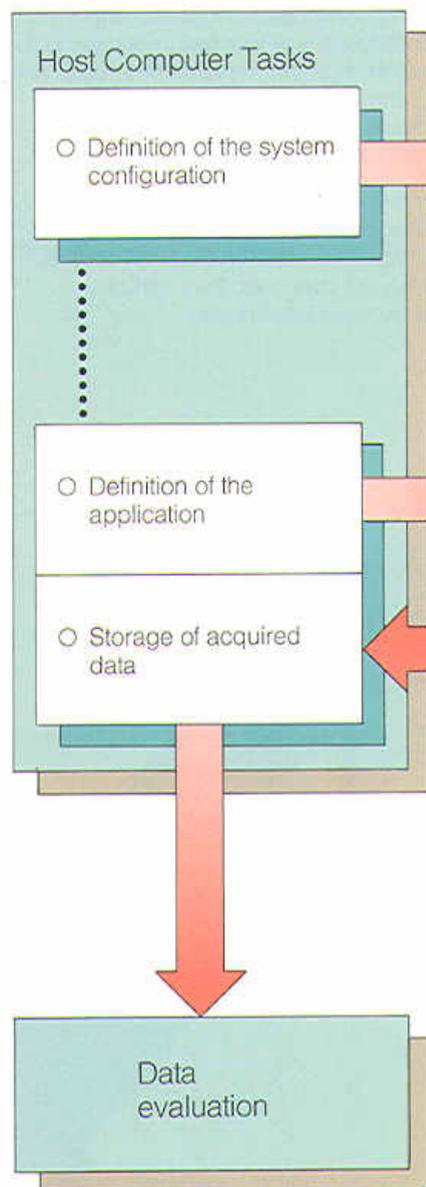
The D 5000 has the flexibility to meet any analytical challenge. Whether you want to work in horizontal or vertical mode, in Theta-2Theta or in Theta-Theta mode, the D 5000 supports all these different demands in only one system. With only a few accessories and in very little time, you can change the basic Theta-2Theta goniometer into a Theta-Theta unit right in your lab. This gives your lab the capability of handling measurements of liquid or loose powder samples, or even bulky samples (e.g. for stress measurements or for high temperature studies).



Plus, by adding one of our proven, application-specific accessories, you can extend the diffractometer to form an integrated, special measurement system for residual stresses and textures. The combination of our open asymmetric Eulerian cradle with the X-Y-Z sample tray ensures the highest flexibility when mounting samples. The stepping motor controlled X-translation provides sample oscillation as well as selection of specific sample areas for analysis. Following the basic design philosophy of the D 5000, these modules have been developed for easy installation and alignment.

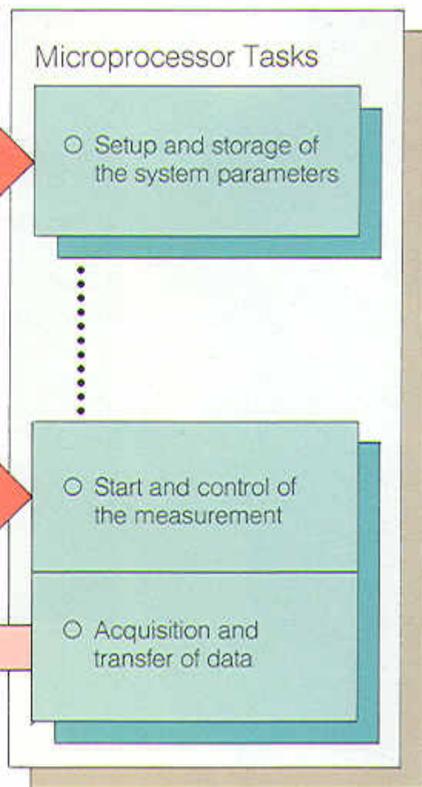
The Host Computer and Microprocessor Controller

The D 5000 uses a dedicated microprocessor controller to handle real-time operation of the diffractometer. The microprocessor operates from commands issued by the host computer, which is normally used for high-level system control and data evaluation. Add-in slots in the microprocessor accept control boards for the diffractometer accessories.



Besides the control and supply electronics for the goniometer and accessories, the microprocessor's chassis also accommodates the complete counting chain and high voltage supply for a scintillation or proportional detector. All parameters for the D 5000 -system are set from the host computer.

For service and alignment, the microprocessor can be manually activated (e.g. in order to check the goniometer functions or to test the status of the system independent on the host computer).



During normal operation the user enters experimental parameters at the host computer, which transfers the instructions as high level code to the microprocessor over a serial interface. The microprocessor interprets the code into operation and control functions, which it stores in its memory for execution, then transfers the resulting data back to the host computer for evaluation.

Detailed status messages can be displayed at any time on the host computer. Plus, in case of system malfunction, the host computer is programmed to permit modem transmission of the system status for fast, efficient trouble-shooting by telephone diagnosis.

An Integrated System with Maximum Flexibility

By installing one of our special purpose modules and the related application optimized software package, you can convert the basic D 5000 into an automated high temperature diffractometer, or into a texture measurement system, or even into a diffractometer with Theta-Theta geometry.

D 5000 HS – High Speed Data Collection

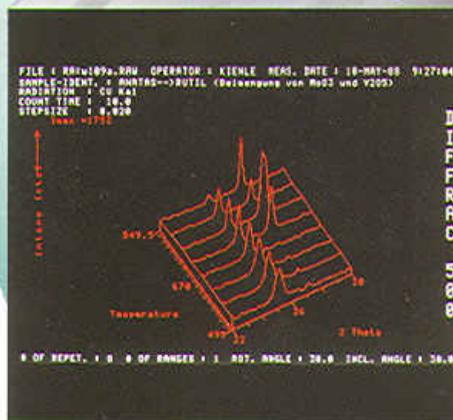
Patented high speed diffractometer with a position sensitive proportional counter (PSD) for drastically reduced measurement times, allowing the preparation of diffractograms in seconds. It is especially suited for examinations of phase changes and reactions under physical influences such as pressure and temperature as well as for time resolved studies.

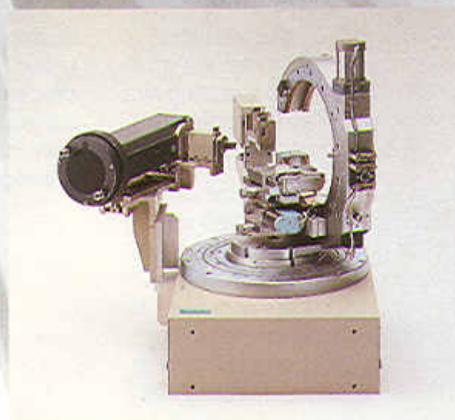
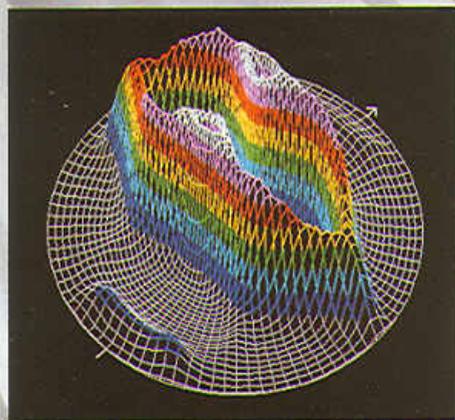
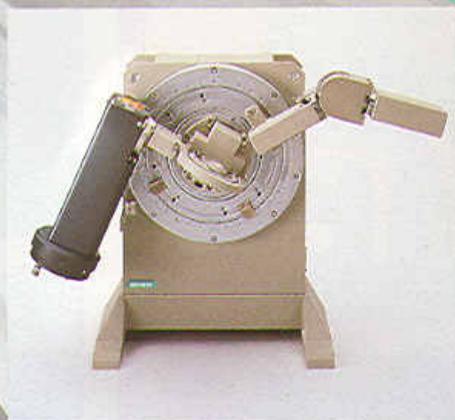
D 5000 HT – High Temperature

Automatic high/low temperature diffractometer with computer-control of temperature values and temperature profile. For measurement of processes dependent on temperature such as crystallization, lattice changes, etc., with special software for complex measurements and threedimensional plots of results. For analysis of time-critical changes, a PSD is the ideal companion to this accessory.

D 5000 BA – Bath Analyzer

Diffractometer for the analysis of the electrolytic bath in the aluminium industry. Addition of a proportional counter permits quantitative determination of the calcium content of samples by x-ray fluorescence. Together with the ALFLUX software package, this permits exact determination of the entire phase composition giving results that can be used for optimizing the electrolytic process by adding primary material.





D 5000 TF – Thin Film Analyzer

For measurements of polycrystalline layers and films with a thickness down to a few Ångströms. With this attachment you can obtain, by adjusting the grazing angle of the incident beam, information from the sample surfaces as well as from layers close to the surface.

Besides characterization of the sample structure, it is also possible to perform depth profile analysis and to measure the different layers of multi-layer samples.

D 5000 TX – Texture Analyzer

Texture diffractometer for measurement of preferred orientations in crystalline materials.

This configuration uses a polefigure program for data collection in reflection/transmission mode and for graphic display of measurement results. Additionally, the orientation distribution function (ODF) can be calculated either from complete (reflection and transmission measurements) or from incomplete pole figures (reflection measurement only).

D 5000 ST – Stress Texture Analyzer

While the standard D 5000 permits stress Analysis, the combined stress/texture diffractometer provides for determination of residual stresses and preferred orientations.

The combination of an open Eulerian cradle with an XYZ-adjustable sample tray supports extensive material characterization of samples of different geometries and allows access to many different spots on the sample surface.

Flexible Software Solutions for Your Applications

Data Acquisition

- Selection of all goniometer movements.
- Application of different measurement strategies.

Special Methods

- Profile analysis.
- Indexing and lattice parameter refinement.
- Crystallite size and micro-stress measurement.
- Raw data simulation from crystal structure.
- Automatic high temperature analysis.

TEX/ODF

- Pole figure measurement and graphic display of the pole figure.
- Calculation of the orientation distribution function (ODF).

DIFFRAC

Data Evaluation

- Interactive graphics.
- Data interpretation.
- Qualitative and quantitative phase analysis.
- Output of diagrams on color plotter or printer.

Auxiliary Programs

- Goniometer calibration.
- System configuration.
- Data administration.
- On-line documentation and help.

STRESS

- Determination of the residual stress according to the $\sin^2\psi$ method.
- Calculation of the entire stress tensor.

ALFLUX

- Determination of the bath ratio in electrolytic aluminium production.

RETAIN

- Determination of the retained austenite content in steels.

A wide variety of application-specific software packages is available for the D 5000 to optimize its performance for your experimental requirements.

Software packages are offered for Digital VAX computers under the multi-tasking/multi-user VMS* operating system, and also for AT-compatible 16 and 32 bit personal computers.

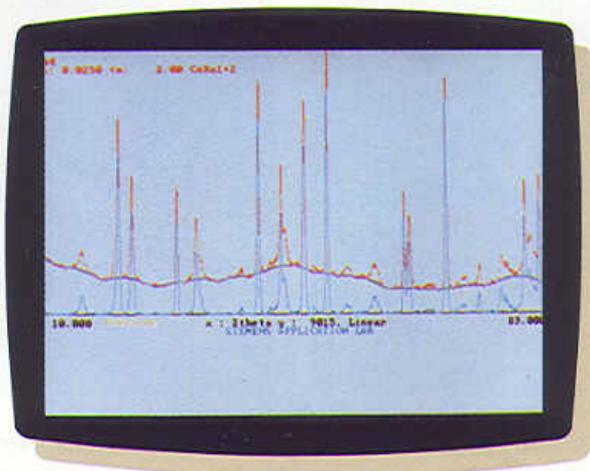
All software is menu-based for user friendly operation. The concept of modularity is consistently maintained. Consequently, a feature of the extensive selection of software packages is its great flexibility. Measurement and evaluation programs can be linked to each other like a chain, enabling fast

routine operation without user intervention. When infrequently used applications are run, full user interaction is supported with graphically-oriented user interfaces at virtually every stage of program implementation.

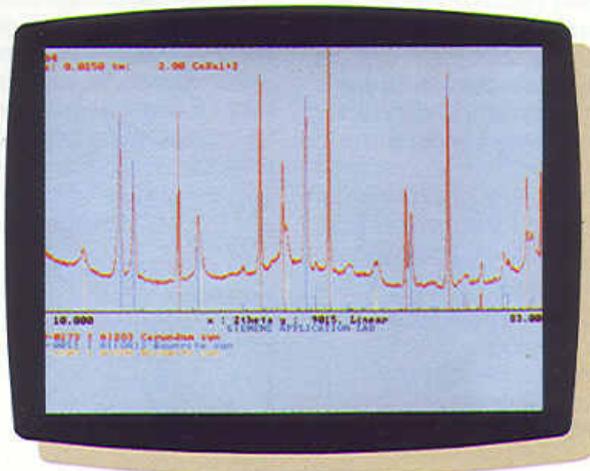
*Trademark Digital Equipment Co.

Examples

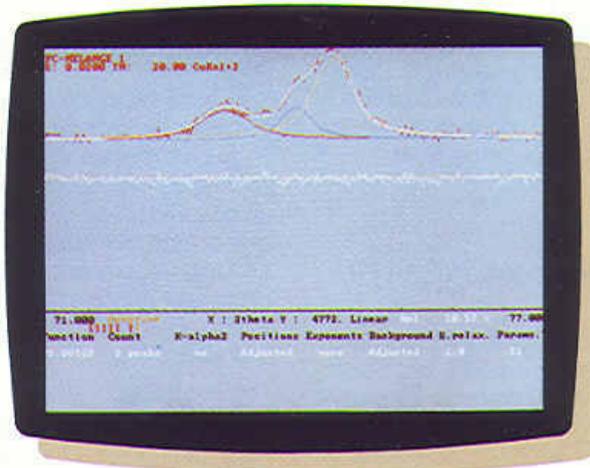
The graphic-oriented user friendly interface makes data processing easy. The results of $K\alpha_2$ correction, data smoothing, peak search and background subtraction, etc. are graphically displayed and can be accepted or rejected. The obtained data can be stored for further evaluation.



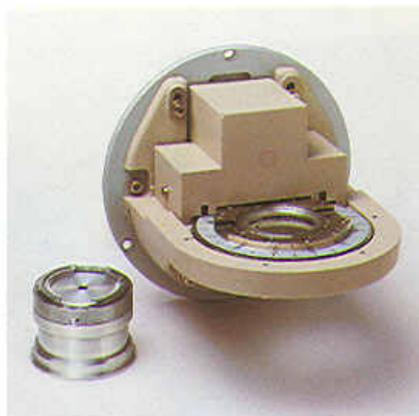
Qualitative analysis (SEARCH/MATCH) of a phase mixture or a pure phase: the data are compared with the standards stored in the JCPDS files or user-created databases. In addition to the classical method (using d and I values of the measured diagrams for the SEARCH/MATCH procedure) a new technique using the complete digitally stored raw data has been developed. This method minimizes user intervention during the search process, eliminates mistakes occurring during peak location. So complete phase identification can be carried out within a few minutes.



Using mathematical profile functions, overlapping diffraction lines can be deconvoluted and all line parameters, such as intensity, location, half-widths and asymmetries can be calculated. The results can, for example, be processed for the determination of crystallite size distributions, micro-stress or lattice parameter determinations.



Attachments and Accessories



Rotating Sample Holder for Reflection and Transmission

For reflection and transmission configurations: this stage offers stepping motor controlled sample rotation during measurements. It has been developed for analyses of coarse crystalline or inhomogeneous materials, as well as for the setting of sample orientations and the execution of phi scans.

Technical data:

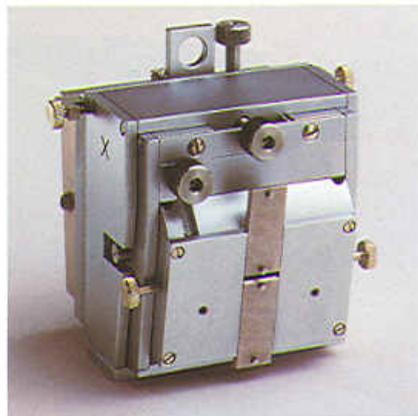
Max. sample size:
50 mm dia. by 30 mm thick for reflection sample holders.
50 mm dia. by 2 mm thick for transmission sample holders.
Masks for sample cups: 23 mm, 34 mm, 42 mm diameter.
Rotation speeds: 0, 15, 30, 60 and 120 rpm.
Positioning: 0...360°
Smallest step width: 0.28°

Standard Sample Holder

A fixed, non-rotating sample holder designed to accommodate samples of different sizes and volumes. A special holder for mounting large, bulky samples is also included.

Automatic 40 position Sample Changer

For automatic serial analysis, this unit features a linear magazine that holds up to 40 individual samples for analysis. Sample rotation can be selected individually for each measurement.



Primary Beam Monochromator

Removes Bremsstrahlung and undesired radiation (including the $K\alpha_2$ line) before they reach the sample. Equipped with a high resolution crystal, this device focusses radiation according to the Johannson principle. Depending on the requested application, it is available with a short ($b = 211$ mm) or long ($b = 355$ mm) focal length.

Technical data:

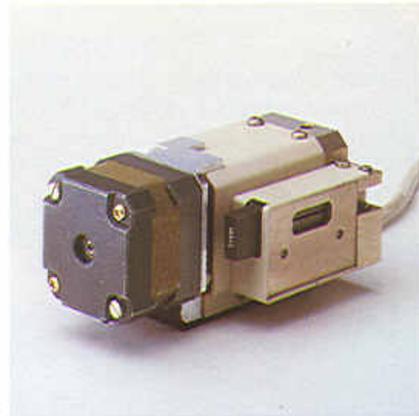
Crystals:
With short focal length for reflection geometry
Cr $K\alpha_1$ radiation Quartz
Co $K\alpha_1$ radiation Quartz
Fe $K\alpha_1$ radiation Quartz
Cu $K\alpha_1$ radiation Quartz, Ge
Mo $K\alpha_1$ radiation Quartz, Si
With long focal length for transmission geometry
Co $K\alpha_1$ radiation Quartz
Cu $K\alpha_1$ radiation Ge
Mo $K\alpha_1$ radiation Quartz

Secondary Monochromator

For cases when the fluorescent radiation from the sample is to be eliminated to give a better signal/noise ratio, a secondary monochromator provides an effective means. A secondary monochromator is easier to mount and align than a primary beam monochromator and is usually preferred for those cases where $K\alpha_2$ stripping is not required.

Technical Data:

Bent graphite crystal (Johann principle), adjusted for Cr-, Co-, Fe-, Cu- or Mo- $K\alpha$ radiation.



Variable Slits

Stepping motor controlled slits to be mounted in the primary or secondary beam path. During analysis, they can be coupled with the Theta movement in order to keep the irradiated area sample surface constant (very important for small diffraction angles), using computer controlled positioning. Variable slits can also be used as computer controlled fixed slits.

Technical Data:

Irradiated sample surface with Theta coupling:
Optional 6.6 or 20 mm length,
Fixed settings: 0.1 to 3°

Detector Slit Changer

Mounted in front of the detector window, this device allows selection of a narrow slit (0.06 mm) for high resolution diffractograms.



Eulerian Cradles

These attachments provides two additional circles (chi and phi) so that the sample can be freely oriented with respect to the primary beam.

Closed Eulerian Cradle

For texture measurement in reflection and transmission mode.

Technical Data:

2Theta max:	125°
Rotation in chi:	0 to 360°
Rotation in phi:	0 to 360°
Diameter of cradle:	250 mm
Measuring circle diameter:	500 or 600 mm

Open Asymmetric Eulerian Cradle

For stress and texture measurements provided with an XYZ sample stage which permits analysis of large, heavy samples due to the possibility of exact positioning in all three dimensions.

The gap in the Eulerian cradle allows measurements up to 170° in 2Theta, thus both stress and texture measurements can be carried out on the same attachment. Chi, phi, and X are motor driven.

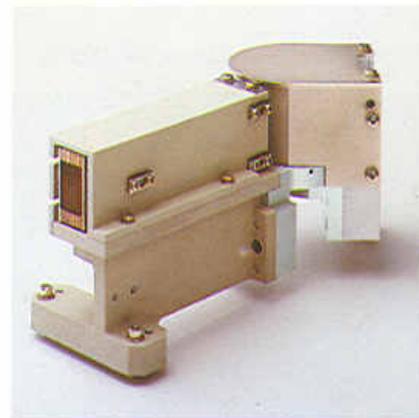
Technical Data:

2Theta max:	170°
Rotation in chi:	-91 to + 65°
Rotation in phi:	0 to 360°
Translation in X:	-30 to +30 mm
Translation in Y:	-25 to +25 mm
Translation in Z:	0 to +13 mm
Diameter of cradle:	250 mm
Measuring circle diameter:	500 or 600 mm



Temperature Attachments

With these attachments, diffraction measurements can be carried out at temperatures from -180 up to an extreme of +2,700°C. Measurements can be done in normal atmosphere, inert gas or vacuum. All attachments are equipped with a temperature stabilizer; furthermore, the high temperature attachment is available with a computerized temperature control which enables free selection and execution of temperature programs.



Thin Film Attachment

By using a special detector holder, secondary soller slits and a plane crystal monochromator, this attachment converts the geometry of the D 5000 to parallel beam optics. In conjunction with adjustment of the grazing angle of the incident beam this provides an ideal method for the analysis of thin films, multi-layer samples and sample surfaces. This attachment works with all sample holders and sample changers, and can be interchanged with the standard detector mount in minutes without realignment of the system.

Technical Data:

Soller slit:	0.15 or 0.4 degrees
Secondary monochromator:	LiF (100) flat crystal
Radiation:	Ag, Mo, Cu, Fe, Co, or Cr
Configuration:	Parallel or anti-parallel beam path with monochromator; also usable without monochromator.

Technical Data

Goniometer Specifications

Operational modes:	Horizontal or vertical, Theta-2Theta and Theta-Theta geometries.
Measuring circle diameter:	Variable; 401, 435, 500 and 600 mm.
Central opening in Theta plane:	155 mm.
Angular range in Theta:	360° (without accessories).
Angular range in 2Theta:	-100 to 168° (depending on accessories).
Smallest selectable step width (Theta and 2Theta):	0.001°
Maximum speed:	1000 deg./min.
Reproducibility (Theta/2Theta):	±0.0005°
Accuracy (Theta/2Theta):	±0.005°
Stability of the rotation rings:	500 N axial; 10 Nm momentum vertical to the axis.
Theta/2Theta motors:	Independent stepping motors.
Monochromators:	Incident or diffracted beam.
Fixed slits:	6, 2, 1, 0.6, 0.2, 0.1, 0.05 mm.
Variable slits:	Variable slits for incident and diffracted beam, both stepping motor driven, computer control for fixed setting or Theta-coupled size variation. Computer controlled detector slit changer with small (0,06 mm) aperture.
Soller slits:	2° soller slit block, to be used in primary or secondary beam.
K β filters:	To be used in primary or secondary beam, available in Ni, Zr, Mn, Fe and V.
Detectors:	Scintillation counter Proportional counter Position sensitive detector Semi-conductor detectors

Product Overview

X-ray Analytical Systems



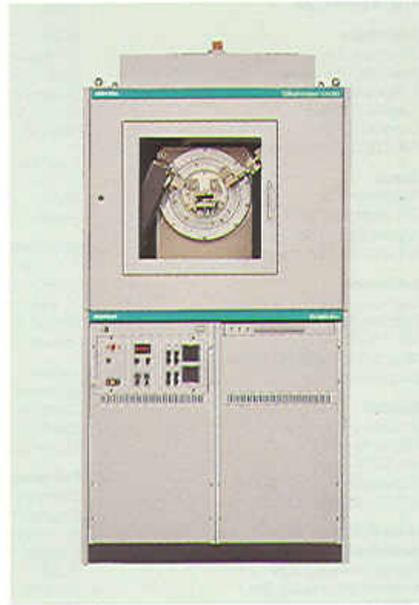
SRS 303 Sequential X-ray Spectrometer

Universal x-ray fluorescence spectrometer for qualitative and quantitative elemental analysis of liquid, solid and powder samples from the ppm range to 100%.



MRS 404 Multi-channel X-ray Spectrometer

X-ray fluorescence spectrometer for simultaneous quantitative analysis of up to 28 elements. This design has been optimized for fast process control applications, e.g. in steel and cement industries.



D 5000 X-ray Diffractometer

Versatile x-ray diffractometer system for qualitative and quantitative analysis of polycrystalline materials, texture, stress, high temperature and high speed measurements.



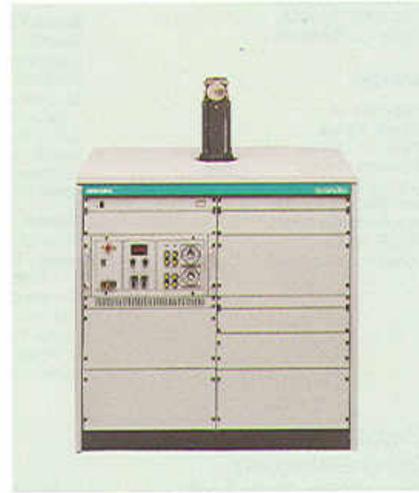
P3/R3 Series II Single Crystal Diffractometer

Four-circle x-ray diffractometer for data collection and structure determination of single crystal small molecules.



X100/X200 Area Detector Systems

Two-dimensional imaging x-ray detector for data collection from single crystal macro-molecules and proteins, microdiffraction studies of single and polycrystalline samples, and for high speed polymer/fiber characterization.



X-ray Generators

A variety of x-ray generators for all applications in x-ray diffraction analysis. Compact K710 medium frequency generators (2 and 3 kW) and high brilliance rotating anode generators up to a maximum capacity of 18 kW.