

X-Ray Powder Diffraction Statement of Work

Multipurpose research X-ray Powder Diffraction System to be able to configure to run several types of experiments in a multi-user environment with operators at various levels of experience. The basic functionality includes but not limited to the following:

- High resolution Bragg Brentano optics
- Parallel Beam Optics using a parabolic mirror with parallel plate collimator with diffracted beam monochromator.
- Able to run transmission and reflectance on same sample without reconfiguring
- Micro diffraction, manual with 0.3 mm Monocapillary and optical microscope
 - Upgradable to do micro diffraction area mapping optics with optimized 0.1 mm point focus mono-capillary with programmable x,y,z stage and video system
- High Speed strip Detector with monochromator
 - Upgradable to 2D and 3D imaging Capability
- Programmable slits
- Spinner stage for reflectance and transmission maintaining sample horizontal for both reflectance and transmission experiments
- Large specimen stage to hold samples up to 100 mm x 100mm and 5 pound sample
- Copper, Cu x-ray tube rotatable for line and point foci with auto detection
- 15 position sample holder
- Silicon low background holders for a minimum of 6 samples
- Powder sample holders for a minimum of six samples
- High precision machined optics and accessories for rapid experiment reconfiguration within 2 minutes without further alignment
- Cabinet shielding sufficient for hard (Silver) radiation without additional shielding
- Rietveld analysis
- Percent Crystallinity
- Crystallite size/micro-strain
- ICSD Data base with non-expiring license with use up to 10 computers
- PDF-2
- Computer with 22" monitor and printer
- Water chiller
- Three (3) days on-site training by an application chemist

Diffraction System

- Single instrument enclosure for HT generator, mains supply, electronics and tube tower and sufficient space for goniometer system and all accessories.
- Accessories precision machined of hardened tool steel to a tolerance of 3 micrometers for rapid instrument configuration of accessories with no need of further alignment.
- Total Enclosed system designed for hard radiation such as Silver without additional shielding. All direct radiation area 12 mm steel
- Fully Interlocked double sliding doors for normal access (=> 18mm lead acrylic)
- Back panels removable for system access and maintenance
- Solid State 3 kW x-ray generator

- Microprocessor for control of the goniometer, dc motors, stepper motors, 4 detectors and 3 shutters simultaneously.
- Internal cabinet mounting racks for accessories such as temperature controllers and detectors electronics
- Generator setting controlled by software including auto burn-in, ramping of tube power and standby modes after analysis. The kV setting must be adjustable to 15-60 output in 1 kV steps at a stability of < 0.01% per +/- 10 % mains variation. The mA setting must be adjustable to 5-60 mA output in 1 mA steps at a stability of less than +/- 0.01% at mains variation of +/- 10%.
- Real time display to include operating conditions and parameters of:
Angular position, counts, kV, mA settings

High Power X-Ray Tube and Generator

- Copper anode with ceramic insulation
- Long Fine focal spot 0.4 x 12 mm
- 2 exit beam ports with Be window one line focus one point focus to be rotated from line projection to point projection as required for maximum x-ray tube flux in either line or point modes.
- Manual rotation of x-ray tube without disconnecting water and power lines for configuring from Line to point focus with auto anode recognition of position in less than 5 minute by inexperienced users without the need for realignment of the tube, optics or goniometer manually or via computer.
- Tube must be manufacturer by the supplier of the Diffractometer with a two year non-pro-rated failure warranty. All future tubes will also be warranty also for a two year non-pro-rated period.
- The system must include X-Ray generator that is capable of 3.0kW output power with remote computer control and tube recognition and tube lifetime monitoring.
- Maximum power 60 kW , 55 mA, 3.0 kW

Parabolic x-ray mirror (parallel beam)

Divergence of parallel beam <0.04 degrees

Beam dimensions 1.2 x 20 mm

Goniometer

- Goniometer theta/theta optically positioned with a minimum radius of 240mm
- Able to be configured for vertical theta-theta or theta-2theta operation or horizontal theta-2theta operation.
- Radius variable from 130 to 320 mm vertical

Specifications:

Accuracy +/-0.0025 degrees

Reproducibility +/-0.0001 degrees

+/-0.001 degrees with attachments

Scan Speed 0.001 to 1.27 degrees / second

Slew speed 15 degrees/ second

Minimum step size 0.0001 degrees in both omega and two-theta

2theta range -40 to 167 degrees

Theta range -15 to 181 degrees

- The goniometer must use DC drive motors and Heidenhain encoders direct optical positioning sensing technology.

Incident and diffracted beam optics

- The incident beam optics must incorporate precision machined pre-aligned, fast interchange optics modules for the incident x-ray beam, with each module attached to identical incident beam sub frames (individual). The optics mounting frames must be made of hardened tool steel and remounting of optics must be reproducible to < 3 microns. Interchange of each optics assembly must occur in under 2 minutes by persons who have no knowledge or experience in the alignment of x-ray diffraction optics and without the need for realignment of the goniometer or optics modules.

Spinner sample stage for reflection and transmission samples

- Spin rates 0.5, 1, 2, 4, 8 sec/revolution

Sample changer, 15 positions for both reflection and transmission

Parallel Plate Collimator, 0.27 degrees

- Diffracted Beam Angular divergence of the diffracted beam to 0.27 degrees.
- 0.1 mm slit for reflectivity measurements
- User can change parallel plate collimator without any realignment.

Programmable Divergence slits

- Automatic variable and fixed slits modes of operation
- Fixed slit mode 1/32, 1/16, 1/8, 1/4, 1/2, 1, 2 and 4 degree
- Continuously variable mode controlled from 0.5 to 20 mm in 0.5 mm increments
- Beam mask of 5, 10, 15 and 20 mm included

Programmable Anti-scatter slits

- Automatic variable and fixed slits modes of operation
- Fixed slit mode 1/32, 1/16, 1/8, 1/4, 1/2, 1, 2 and 4 degree
- Continuously variable mode controlled from 0.5 to 20 mm in 0.5 mm increments

Micro diffraction

- Monocapillary optic for micro diffraction applications. Converges and collimates beam size based on total internal reflectance of the X-ray beam inside the monocapillary. Length = 135mm, diameter = 0.3mm (300 micron).
- Optical alignment microscope with cross-hair, precision mounted with a viewing diameter 6 mm, resolution 50 micrometer / division.

High Speed Detector

- System must include a high speed pixilated solid state detector with a maximum pixel dimension of 55 micron.
- The detector must include the following modes operation, 0D where all pixels are added up to give one intensity, 1D where all pixels in one column are added up to form a position-sensitive detector in one direction (static or scanning measurement modes).
- 256 x 256 pixel array
- Minimum peak linearity of 50,000 counts/sec per pixel or 12,800 cpc per strip
- Faster scan speeds up to 120,000 counts/sec
- The detector must include a 97% linear count rate per square strip: 12.8 million photons/second/mm² background noise
- No detector gas operates at room temp

Graphite monochromator

- Diffracted beam monochromator with flat pyrolytic graphite crystal, adjustable for different wavelengths. Monochromator must incorporate precision machined pre-aligned, fast interchange module when needed. Must be reproducible to < 3 microns without alignment required.

SAMPLE STAGES

The sample stages must incorporate pre-aligned, fast interchange sample stages. Interchange between sample stages must occur in under 5 minutes without the need for realignment. The following stages must be included:

- Reflection / Transmission Sample spinner which utilizes top referencing of the sample holder mounting for accurate positioning for measurements in either reflection or transmission geometry.
- Large Sample holder to hold samples up to 100 mm x 100mm and 5 pound sample

SAMPLE CHANGER 15-POSITION The sample changer includes one (1) sample magazine for 15 samples Can be moved to its parking position inside the cabinet when not in use. Includes three separate positions for monitor/reference samples. Up to 2 additional magazines can be added for a total of 45 sample positions.

Computer

High performance computer windows 7
Intel core 2.33 GHz
8 GB, SDRAM memory
22 inch monitor
Dual hard drives 320 GB
Office Professional
16 x DVD and 16 X DVD+/-RW
22 inch monitor
Printer with cable

XRD Data collection and Analytical software

- Experiment set up and data collection and analysis with Word report formats.
- Automatic execution of user batch programs
- Must contain interactive routines to provide the following functionality for all the optics configuration modes of the instruments:
- Data files stored in open architecture yet secure XML file format to be compatible with historical stored data.
- Automation Software: Included with the Data Collection module shall be a batch scripting program to automate data collection, data analysis and report generation.
- Analysis package: Single software analysis program to include Search Match, Rietveld structure analysis, statical cluster analysis and Full pattern Quantification by Bish and Chipera method.
- Principal components analysis must be carried out as a separate and independent method to visualize and judge the quality of the clustering.
- Crystallographic analysis module to assist in indexing, lattice refinement and Rietveld calculations to include quantitative phase analysis, crystallite size and micro-strain determination running in either automatic, semiautomatic or manual modes. Module must also contain a crystal structure viewer module.
- High Score 10 licenses (for off line data analysis)

Databases

ICSD Database
ICDD PDF-2

On Site Training (3 days) (2days + 1 additional day). On-site training must be by an application scientist. This training shall be separate from any operations training by the service/installation engineer at the time of installation.