X Ray Facility

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XRF Instrument Description

X-Ray Facility Instruments & Resources

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Main

Facility

The X-Ray Facility is located on the east side of the fourth floor of the newly renovated Kasha Laboratory of Biophysics, Institute of Molecular Biophysics, Florida State University, Tallahassee, FL 32306-4380. It occupies approximately 1350 ft^2 of space consisting of one large laboratory, one cold room, two smaller work areas and one office room. A schematic diagram of the X-Ray Facility is shown in Figure 1.

X-Ray Generators

Rotating Anode Generators

The X-Ray Facility has three rotating anode generators. Two of the three generators are manufactured by Rigaku Corporation of Japan and the third generator is manufactured by Elliott Neutron Division of England. All the generators are located in KLB 413 and are equipped with copper rotating anodes (x-ray output wavelength, λ=1.541Å). Rigaku generators (Model # RU-H2R) have a 0.3 x 3.0 mm^2 fine focal point projected on to the anode with the total load capacity of 5.4 kW. Elliott (Model # GX-20) has a 0.15 x 1.50 mm^2 fine focal point projected on to the anode with the total load capacity of 1.2kW. Rigaku generators were manufactured in 1993 and the Elliott in 1980.

X-Ray Detectors

Image Plate

X-Ray Facility has one image plate (IP) detector made by Rigaku Corporation of Japan (Model R-Axis IIc). It is an automated IP reader with two identical IPs positioned 180° from each other. The data acquisition is controlled via a dedicated Windows NT 4.0 dual 733 MHz Pentium III computer with 756 MB of memory. The detector capabilities are:

1) Reusable IPs are based on BaFBr: $Eu2^+$ Photo-Stimulable Luminescence (PSL),

2) The IP with 1900 x 1900 pixel (102 μm/pixel) active area allows continuous data collection with very little dead time between plate swapping,

3) Automated read and erasure of 16 bit/pixel x-ray diffraction data with HeNe laser and erase lamp,

4) Automated analog to digital conversion and export of data to host computer via SCSI interface,

5) High dynamic range (10^4 to 10^5, compared to 10^2 for photographic film), higher sensitivity (10^2 times more compared to photographic film) and linear response, and

6) The read-out time for 110 μm/pixel size is approximately eight (8) minutes.

Charge coupled device (CCD)

X-Ray Facility has one charge coupled device (Mar CCD165) detector manufactured by Mar-USA Inc. of Evanston, IL. It is an automated detector with a circular x-ray active area of 165 mm diameter with a very fast read-out of data. The capabilities of the CCD are:

1) A circular detector with a large (165 mm diameter) x-ray active area consisting of one single CCD chip with no gap between chips,

2) CCD chip is cooled to -70°C using peltier cooler to reduce background noise and dark current,

3) Fast read-out time of 3.5 s for a 2048 x 2048 pixel active area with 78 μm/pixel and 16 bit/pixel, and

4) Small fiber-optic taper (2.7:1) giving good demagnification of the image at the CCD

Auxiliaries

Focusing & Cryo Components

Mirror System

Both the Rigaku generators are coupled to confocal mirror systems manufactured by Osmic Inc, Troy, MI. Elliott generator is coupled to long Supper Mirrors manufactured by Charles Supper Inc, Natick, MA. The confocal mirror (CMF Confocal Max-Flux) Model CMF 12-38-Cu6 'Blue' mirror from Osmic is coupled to the R-Axis IIc IP detector. Model CMF 20-28-Cu6 'Purple' mirror from Osmic is coupled to marCCD 165 detector. Long supper double mirrors (Model # 7616) have adjustable focal distance. All mirror systems are purged with helium and are constantly tuned to maximize their output intensity. Advantages of the Confocal Max-Flux mirror system are:

1) It reduces the number of independent alignment parameters making the focusing process faster,

2) It also reduces the number of coupled alignment parameters,

3) Specialized multi-layer mirror coating reduces the contaminating radiation while maximizing the Copper K_α output, and

4) Maximized output flux compared to total reflection mirrors due to larger acceptance-angle and due to the side-by-side geometry of the two mirrors.

Cryo System

We have two cryo cooling systems capable of maintaining the samples at temperatures between 80° and 400° K, with a stability of ±0.1° K. The required temperature can be maintained for several days. The cryo cooling systems (Cryo Stream Model # 600 and 700) manufactured by Oxford CryoSystems of Oxford, England are state of the art instruments with very low liquid nitrogen consumption of 0.6 liter/hour. The details of the cryo system are:

1) Cryo Stream works at ambient pressure making it easier to replenish liquid nitrogen during the run,

2) Very low consumption of liquid nitrogen and the consumption is independent of set temperature.

3) Wide range of cooling and heating rates (360° K/hour to 0.1° K/hour), and

4) Easily programmable to achieve conditions like cycling and holding.

Two-theta Stage

The R-Axis IIc detector sits on top of a variable two-theta stage. This stage allows a wide selection of sample-detector distances anywhere between 65 and 455 mm for a zero 2-theta angle. Non-zero 2-theta angles between -18.00° and +30.00° are achievable for sample-detector distance of at least 120 mm and larger.

Mar CCD detector sits on top of Mar base and allows sample-detector distance anywhere between of 35 and 384 mm for a zero 2-theta angle. Several but limited non-zero 2-theta angles are achievable with this stage for all the sample-detector distances. Mar CCD distance can be controlled remotely with the help of the software.

Auto Refill System

Oxford CryoStream 700 cryo cooler on the Mar CCD detector has an automated liquid nitrogen refill system. This Liquid Level Sensor (Model # 186) manufactured by American Magnetics Inc. of Oak Ridge, TN automatically refills the experimental cryo dewar, when liquid nitrogen falls below a pre-set level, from an attached supply dewar (Cryofab 160 liter tank). Auto-refill system helps the user with a worry-free cryo crystallographic data collection extending for several days including weekends.

Computer Resources

Computers & Peripherals

A dedicated Pentium III 733 MHz dual processor Dell computer with 756 MB of memory, 40 GB of hard drive, 21" monitor, running under Windows NT 4.0 operating system controls the data collection and processing of R-Axis IIc detector. This machine (`anaconda.sb.fsu.edu | 128.186.103.102`) has a floppy disk, an internal CD-ROM drive, an internal DVD-R+RW drive, and an internal DDS-4 tape drive connected via a SCSI controller for archiving data. A dedicated Linux machine (`spruce.sb.fsu.edu |`

128.186.103.109), running under RedHat Linux 7.0 operating system, controls the data collection and processing of the Mar CCD165. This Linux machine has a floppy disk, two 36 GB internal disks, an internal DDS-4 tape drive, and an internal CD-ROM drive connected via a SCSI controller.

We have a 933 MHz Pentium III Linux machine with 21" monitor, 256 MB memory and 40-gigabyte disk space running Red Hat 7.0 solely for the processing of IP and CCD data. This machine, (raccoon.sb.fsu.edu | 128.186.103.108) has an external DDS-3 tape drive, internal DVD-ROM and CD-RW Writer connected via a SCSI controller. Another 733 MHz Pentium III machine (neptune.sb.fsu.edu | 128.186.103.106) has recently been added for processing data. It has a 19" monitor, 256 MB memory and 20 GB hard disk, a DVD-R+RW drive and a FireWire drive. Both neptune and raccoon have FireWire (aka IEEE 1394 or i-Link) and USB2 interfaces. Using these interfaces they connect to a Lacie FireWire DVD-R+RW drive and an 80 GB Maxtor Fire Wire External Harddrive (DV3000).

In addition, the users have access to a dual-bootable (Linux or WinXP) Dell Latitude Cpx laptop during synchrotron trips. This laptop (tampa.sb.fsu.edu | 128.186.103.110) features a 497 MHz Pentium III processor, a DVD-ROM/SuperDisk drive, USB port, Ethernet interface and a modem interface. All the XRF computers are behind firewall and run sshd, so only secure ssh2 protocols are allowed to access these machines.

Archiving and Storage

XRF offers several options for archiving the user's data, ranging from tapes, CDs, and DVDs both under Windows and Linux environments and are shown in Table 1 below:

Manufacturer & Model	Computer	Operating System	Interface	Media	Capacity (native | compressed)
Seagate STD624000N	raccoon.sb.fsu.edu (128.186.103.108)	Linux	External SCSI	DDS-3 tape	12 GB | 24 GB
Seagate STD1401LW	spruce.sb.fsu.edu (128.186.103.109)	Linux	Internal SCSI	DDS-4 tape	20 GB | 40 GB
Seagate STD1401LW	anaconda.sb.fsu.edu (128.186.103.102)	Windows NT 4.0	Internal SCSI	DDS-4 tape	20 GB | 40 GB
HP 9500 CD-Writer Plus	raccoon.sb.fsu.edu (128.186.103.108)	Linux	Internal SCSI	CD-R & CD-RW	~ 650-700 MB
Pioneer DVR-A04	neptune.sb.fsu.edu (128.186.103.106)	Linux	External FireWire	DVD-R & DVD-RW	~4.5 – 4.7 GB
Pioneer DVR-A04	anaconda.sb.fsu.edu (128.186.103.102)	Windows NT 4.0	Internal SCSI	DVD-R & DVD-RW	~4.5 – 4.7 GB

• Table 1 Available options for data archiving at IMB XRF

The data collected at the Facility is currently archived in DDS-3 and DDS-4 tapes. Earlier archives were stored in one of the following formats: 4mm DDS tapes, 8mm Exabyte tapes (mostly 8500 or 8500c), ISO 1990 CD-R, and 4mm DDS-3 tapes. In addition to the drives mentioned in Table 1, the facility has access to DLT, ZIP-100, ZIP-250 drives. Due

to random-access capability, media reliability, prolonged shelf life, and wide availability, the XRF is encouraging the users to select DVD-R media instead of tapes for future data storage and transport, even though the Facility will continue to support tape drives for several more years.

Maxtor Personal Storage DV3000, an 80 GB FireWire (aka, IEEE 1394 & I-link) drive is added recently for collecting data at synchrotrons and quickly transporting the whole data set back home. The hot-swappable FireWire drive will serve as a fall back option in the event of corrupted or unreadable archived data.

Accessories

Microscopes

The facility has several optical and stereo zoom microscopes for sample screening and mounting. The Olympus Model # SZ 60 has 10-63X zoom and a polarizer. A Nikon Optiphot-2 microscope with polarizer, photo documentation attachment is available also available. Two more light microscopes are also available for crystal manipulation.

Crystal Documentation

In 2004 we have added a Leica stereo microscope with a Motic digital camera for crystal documentation. The Leica S8 APO stereo zoom apochromatic microscope has been interfaced with a 1.3 mega pixel FireWire Motic digital camera and a Windows 2000 computer (`phe.sb.fsu.edu | 128.186.103.107`). Motic Advanced Imaging 3.2 software allows automated capture and documentation of crystal images from the microscope. `Phe.sb.fsu.edu` is under \\IMB2 domain and can be accessed by anyone who has an account in the Windows platform.

Crystal manipulation

A vibration-free constant temperature crystallization chamber is available in room KLB 412 for growing crystals at temperatures different from the ambient and cold room. It has an interior volume of 8 ft^3 with wide range of selectable temperature settings (6-25°C).

Cold room dedicated for growing and mounting crystals is located in room KLB 411 adjacent to the Facility.

Dry transport dewar (Model # S/C 4/2 V) manufactured by Chart Biomed is available to safely transport pre-frozen crystals at 80°K either using over-night shipper or by plane. Storage dewar (Model # XT10; aka 10XT) manufactured by Taylor-Wharton is available for long-term storage of pre-frozen crystals.

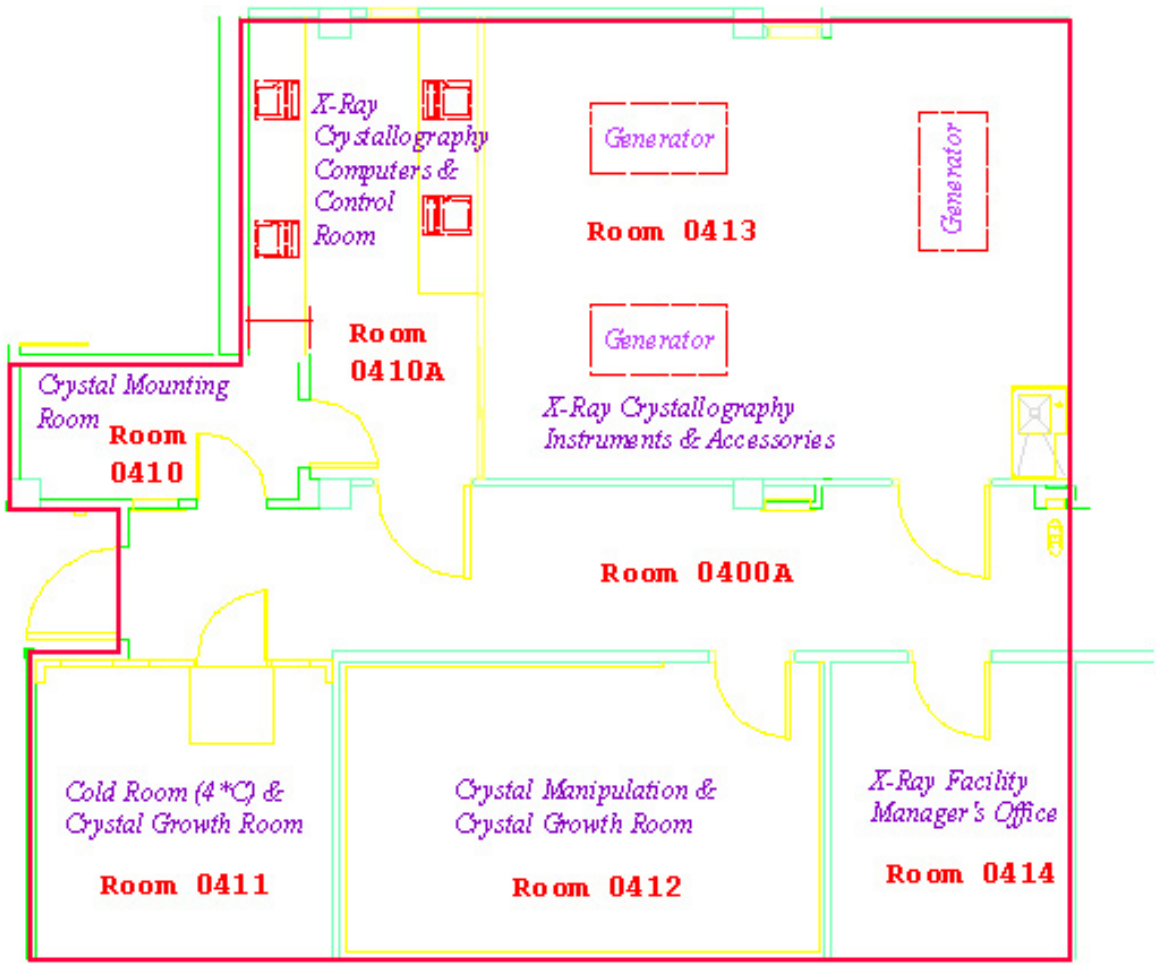
Laboratory Space & Amenities

X-ray facility occupies 1350 ft^2 split between the main x-ray lab (KLB 413: 700 ft^2), crystal growth room (KLB 412: 270 ft^2), cold room (KLB 411; 170 ft^2), and office (KLB 414: 140 ft^2). The laboratory has access to compressed air, vacuum, and chilled water and UPS power source. The generators are located inside lead glass separators.

We have initiated and installed two internet based devices that constantly monitor the temperature and relative humidity (T & RH) of both the crystallization cold room and the detector area. These networked environmental units from APC allow the Facility users to monitor the current conditions of T & RH of their crystallization areas and warn them via e-mail if the conditions deviate from the set thresholds. We intend to add two more devices to monitor other areas of the XRF.

NIH/NSF style description

X-ray diffraction data collection and processing can be carried out in the newly renovated dedicated 1350 ft^2 x-ray facility using either an automated image plate detector or a CCD detector. The detectors are coupled to two different 5.4 kW copper rotating anode Rigaku generators with Osmic Confocal Max-Flux mirrors. The detector two-theta stage allows for data collection at variety of sample-detector distances as well as offset detector configuration for high-resolution studies. Facilities exist to collect the data at wide range of temperature settings different from the ambient, including cryo temperatures. Specialized vibration-free crystallization chambers and dedicated cold room are available for exclusively for crystal growth and manipulation at ambient, 4°C, and non-ambient temperatures. Dedicated Linux and Windows NT based computers are available for automated data collection and processing of diffraction data. Variety of data archiving, storage, and retrieval options using tape, CD-R, & DVD-R media are available under different operating systems (Linux, Windows NT, and HP-Tru64 UNIX) and interfaces (SCSI, USB, and FireWire). Several mono- and stereo- light microscopes, microscopes with digital cameras, cryo dewars and accessories are available at the Facility for sample handling documentation and storage. Facility has dedicated computers with popular software for data processing and analysis and Institute's shared Linux and Windows workstation configured for 3-d modeling can be used for graphic work. Numerically intensive work can be carried out using Institute's shared HP-Alpha servers with 2.0 Gbytes of memory.



• Figure 1. Schematic drawing of x-ray facility and the east side of fourth floor IMB.