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 (KLB), Institute of Molecular Biophysics (IMB), Florida State University (FSU) in Tallahassee FL 32306-4380. It occupies approximately 1350 ft² 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, $\lambda=1.541\text{Å}$). Rigaku generators (Model # RU-H2R) have a 0.3 x 3.0 mm² 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 mm² fine focal point projected on to the anode with the total load capacity of 1.2 kW. Rigaku generators were manufactured in 1993 and the Elliott in 1980.

X-Ray Detectors

Image Plate (IP)

X-Ray Facility has one image plate (IP) detector made in 1992 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 dual 733 MHz Pentium III computer with 756 MB of memory running under Windows XP SP2 operating system. 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 in 2000 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 data acquisition is controlled via a dedicated single 651 MHz Pentium III computer with 512 MB of memory running under RedHat Linux 8.0 operating system. 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

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**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 Kα 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 XP SP2 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 8.0 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 for the processing of home and synchrotron x-ray diffraction data. This machine, (raccoon.sb.fsu.edu | 128.186.103.108) has an internal DDS-5 (aka DAT72) tape drive, an external FireWire DVD-R writer and an external SCSI DDS-3 tape drive. Another 733 MHz Pentium III machine running Red Hat 7.1 (neptune.sb.fsu.edu | 128.186.103.106) is used both for processing and archiving 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 variety of FireWire and USB2 Lacie DVD-R+RW drives and external hard drives from Maxtor.

We have two dedicated 3-D stereo-capable workstations for model building work. One machine (salmon.sb.fsu.edu | 128.186.102.125) runs under Windows XP operating system with 3.01 GHz Pentium IV processor and has StereoVision glasses and emitter. The other machine (jury.sb.fsu.edu | 128.186.103.135) runs under Ubuntu Linux operating system with 3.01 GHz Pentium IV processor and has StereoVision glasses and emitter.

In addition, the x-ray facility users have access to a dual-bootable (Linux or WinXP) IBM ThinkPad laptop computer for synchrotron trips. This laptop (whisper.sb.fsu.edu | 128.186.103.114) features a 1.8 GHz Pentium IV mobile 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.

**Software**

Essential and popular data processing and modeling software packages have been installed and licensed in several X-Ray Facility computers. Software packages such as HKL2000, CrystalClear, CCP4, CNS, O, SOLVE, RSREF, and USF are available both at the Facility and Computer Resources workstations. Several of these packages are available both under Linux and/or Windows platforms. Auxiliary software packages such as PYMOL, MOLSCRIPT, ADXV, and XtalView are also available.

**Archiving and Storage**

For short-term storing and processing of data sets from home as well as synchrotrons, XRF has a 1 tera byte data storage system (1TB Data Storage). The storage system consists of nine (9) Seagate Cheetah 10.K7 146 GB Ultra320 SCSI hard drives in RAID 5 (redundant array independent disks, level 5) configuration. Software RAID control, access and data quota are managed by a dedicated computer (radio.sb.fsu.edu | 128.186.103.112) running Ubuntu Linux. An Ultrium1 SCSI tape drive from HP with LT01 media is available for archiving large data sets. Maxtor and Iomega external hard drives with FireWire (aka, IEEE 1394 & I-Link) and USB2 interfaces are available for storing data sets at synchrotrons and quickly transporting the data sets back home. The
hot-swappable portable drives also serve as a fall back option in the event of corrupted or unreadable archived data. Several data-processing and archiving computers have FireWire and USB2 dual interface cards that allow hot-plugging and hot-swapping of external drives with data sets.

XRF offers several options for the long-term archiving and storing of the user’s data, ranging from magnetic (DDS & LT01 tapes) and optical (DVD-R and DVD+R) media under both Windows and Linux environments and are shown in Table 1.

<table>
<thead>
<tr>
<th>Manufacturer &amp; Model</th>
<th>Computer</th>
<th>Operating System</th>
<th>Interface</th>
<th>Media</th>
<th>Capacity (native</th>
<th>compressed)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Seagate STD624000N</td>
<td>raccoon.sb.fsu.edu (128.186.103.108)</td>
<td>Linux</td>
<td>External SCSI</td>
<td>DDS-3 tape</td>
<td>12 GB</td>
<td>24 GB</td>
</tr>
<tr>
<td>Seagate STD1401LW</td>
<td>spruce.sb.fsu.edu (128.186.103.109)</td>
<td>Linux</td>
<td>Internal SCSI</td>
<td>DDS-4 tape</td>
<td>20 GB</td>
<td>40 GB</td>
</tr>
<tr>
<td>Seagate STD1401LW</td>
<td>anaconda.sb.fsu.edu (128.186.103.102)</td>
<td>Windows XP SP2</td>
<td>Internal SCSI</td>
<td>DDS-4 tape</td>
<td>20 GB</td>
<td>40 GB</td>
</tr>
<tr>
<td>Certance CD72LWH-SS</td>
<td>raccoon.sb.fsu.edu (128.186.103.108)</td>
<td>Linux</td>
<td>Internal SCSI</td>
<td>DDS-5</td>
<td>36 GB</td>
<td>72 GB</td>
</tr>
<tr>
<td>Pioneer DVR-A04</td>
<td>Neptune.sb.fsu.edu (128.186.103.106)</td>
<td>Linux</td>
<td>External FireWire</td>
<td>DVD-R &amp; DVD-RW</td>
<td>~4.5 – 4.7 GB</td>
<td></td>
</tr>
<tr>
<td>Pioneer DVR-A04</td>
<td>anaconda.sb.fsu.edu (128.186.103.102)</td>
<td>Windows XP SP2</td>
<td>Internal SCSI</td>
<td>DVD-R &amp; DVD-RW</td>
<td>~4.5 – 4.7 GB</td>
<td></td>
</tr>
<tr>
<td>HP Ultrium 215</td>
<td>Radio.sb.fsu.edu (128.186.103.112)</td>
<td>Linux</td>
<td>External SCSI</td>
<td>LTO-1</td>
<td>100</td>
<td>200 GB</td>
</tr>
</tbody>
</table>

- Table 1 Data archiving options available at IMB XRF

The data collected at the Facility is currently archived in DDS-4 and DAT72 tapes. Earlier archives were stored in one of the following formats: 4mm DAT tapes, 8mm Exabyte tapes (mostly 8500 or 8500c), ISO 1990 DVD-R, and 4mm DDS-3 tapes.

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## 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. The Carl Zeiss Model Stemi DV4 has 8-32X zoom with Model C stand with transmitted and reflected light source. Two more light microscopes are also available for crystal manipulation.
Crystal Documentation

In addition to above mentioned stereo microscopes, we also have 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 \XRAY 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 ft³ 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 ft² split between the main x-ray lab (KLB 413: 700 ft²), crystal growth room (KLB 412: 270 ft²), cold room (KLB 411: 170 ft²), and office (KLB 414: 140 ft²). 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

Single crystal x-ray diffraction data collection can be carried out in the newly renovated and dedicated 1350 ft² x-ray facility using either an automated image plate or a CCD detector. Each detector is coupled to a 5.4 kW copper rotating anode Rigaku generator with an Osmic Confocal Max-Flux mirror system. The detector’s two-theta stage allows for the data collection at variety of sample-detector distances as well as offset detector configuration for high-resolution studies. Special accessories
allow for the data collection at wide range of temperature settings different from the ambient, including cryo temperatures. Dedicated vibration-free crystallization chambers and cold room is available for crystal growth and manipulation at ambient, 4°C, and non-ambient temperatures. Dedicated Linux and Windows based computers are available for automated data collection and processing of diffraction data. Essential data manipulation software packages (HKL, CCP4, O, & CNS) are installed and licensed. Short- and long-term storage of diffraction data using magnetic (DDS and LT0 tapes) and optical media (CD-R and DVD ± R) are supported under different operating systems (Linux and Windows). Several mono- and stereo- light microscopes, microscope with digital camera, cryo dewars and accessories are available at the Facility for sample handling, documentation and storage. Membership in SER-CAT Beamline 22 in the Advanced Photon Source allows us to collect data using the synchrotron radiation several times a year.

Figure 1. Schematic drawing of x-ray facility and the East side of fourth floor of IMB (KLB).