



# **X-ray Diffraction Laboratory**

**1/12/2018**

The operation and safety procedures outlined in this manual are to ensure compliance with the rules and regulations of Texas A & M University and to provide information concerning radiation safety. It is assumed that the users of this facility have attended the required University Training and are familiar with general radiation safety issues.

---

## **Radiation Sources in the X-ray Diffraction Laboratory**

There are presently nine (8) x-ray diffraction instruments located in rooms 2407 and 2409. These instruments produce 6KeV x-rays.

---

### **As Low As Reasonably Acceptable (ALARA)**

The instrumentation in the X-ray Diffraction Laboratory is equipped with safety interlocks in order to eliminate accidental exposure.

The goal of this facility is that one should receive **zero radiation exposure** during routine operation and alignment of the instrumentation. To achieve this goal the Laboratory will

- Periodically monitor radiation levels.
  - Provide sufficient shielding and distance separation.
  - Maintain adequate radiation safety records
  - Provide in-depth training
  - Provide laboratory security and limit access
-

## Normal Operating Procedure

- 1.1 All radiation producing devices are equipped with safety interlocks in order to minimize accidental radiation exposure.
  - 1.2 All panels and doors must be in place and secured before operation of the instrumentation.
  - 1.3 Visual inspection of the X-ray tube housing, collimator, water tubes and high voltage cables are required before operation of the instrument.
    - a) if any part has been displaced or damaged then close enclosure de-energized instrumentation and seek help
  - 1.4 Visual inspection of the X-ray generator is required before operation of the instrument.
    - a) if abnormal situation is seen then close enclosure de-energized instrument and seek help.
  - 1.5 Start the "APEX3" program.
  - 1.6 Close the enclosure doors and test the shutter operation
    - a distinct click noise will be heard and the shutter LEDs will change from green to red.
    - If abnormal operation is seen then de-energize x-rays and seek help
  - 1.7 Be sure shutter is closed before opening the enclosure doors.
  - 1.8 Visually inspect the enclosure safety switches.
    - shutter will not open with doors open
  - 1.9 Use survey equipment to test for stray radiation.
    - if excessive radiation is detected then cease activity, close enclosure, de-energize x-rays and seek help.
  - 1.10 With the "APEX3" program initialize drives.
    - visually inspect angles, if not a zero seek help.
  - 1.11 Ensure the shutter is closed and mount the sample in the holder
    - keep your hands well below the primary beam area.
  - 1.12 Visually check the detector position and generator settings
  - 1.13 Close enclosure door
  - 1.14 Enter information in the operational log book.
    - Enter the date, your name, your advisors name
    - Enter your project name and title.
    - Enter any unusual event or circumstance
    - Enter detector distance, generator settings
- 
- 2.0 Operation during data collection
    - 2.1 do not open enclosure doors during data collection
    - 2.2 do not remove any instrument panels during operation
    - 2.3 avoid movement in or around the instrument during data collection

## Shutter/Enclosure Safety features.



There are two X-ray shutter on the VENTURE system. The safety shutter that is behind the first slit apparatus attached to the x-ray tube (right hand side).

Normal operation :

- **AMBER** LED on light on the tube will be lit when X-rays are on.
- When the Safety shutter opens (loud click sound) and the red **LED** light will be seen on the tube housing.

Front/side/rear panels.

Normal operation :

- Yellow light  (radiation symbol) on front left panel will be on for normal operation.
- The solid green light will be on below the yellow light .
- The green light with door symbol will be on when door is open
- A solid **Red Light** and/or white light in either position indicates fault.
- Safety shutter will not open if the yellow or green lights are not lit.

## Power Connection and Disconnection


Depressing the **RED** button located on the wall next to the exit doors will disconnect the power to all diffractometers. This switch should be used in case of emergencies.

Depressing the **RED** button on the left-hand or right-hand panel of the diffractometer will disconnect the power to an individual unit.

## Radiation Hazard and Work Areas

An area of 4.5 meters (15ft.) extending in all directions from the X-ray diffractometer is designated as a HAZARDOUS OCCUPATION AREA. This area should only be occupied for short durations and only for the purpose of starting and ending an experiment.

## Energizing X-rays

- 1) All panels on the diffractometer must be in place and closed. All safety lights must be green.
- 2) Red safety button on generator panel (lower left) must be pulled out.
- 3) Depress the  to switch on generator panel left button.
- 4) X-ray will come on and ramp to KV 50 MA 1.

## De-energizing X-rays

- 1) Depress yellow switch  on left-hand panel.
- 2) Wait for yellow Light to change to green .

## 3.0 Radiation Safety Information

### Radiation Units

- a) Roentgen (R)** : amount of radiation that produces one unit of ions/cm<sup>3</sup>.  
(measure of X-rays) 1mR/hr is considered a low rate. 100mR/hr is considered high.
- b) Radiation Absorbed Dose (rad)** [SI unit : gray (Gy) where 1Gy = 100 rad]:  
Energy imparted to matter in volume (V) divided by the mass. 1 rad = 100 ergs/gram (measure of any radiation) 100 rad/sec = 1 Watt/kg
- c) Radiation Equivalent Dose (rem)** [SI unit sievert (Sv) where 1Sv=100 rem]:  
Product of the absorbed dose and the relative biological effect (RBE) necessary to express on a common scale. rad x (RBE) = rem for X-rays the RBE = 1.0 and for neutrons = 10.0 (measure of radiation effect on humans)

for X-rays :

$$\begin{array}{ll} 1 \text{ rad} = 1 \text{ rem} & 1 \text{ R} \\ 3.6 \times 10^5 \text{ R/hr} & 1 \text{ Watt/kg} \end{array}$$

- d) Maximum Permissible Dose:**  
(<https://www.osha.gov/SLTC/radiationionizing/introtoionizing/ionizingattachmentsix.html>)

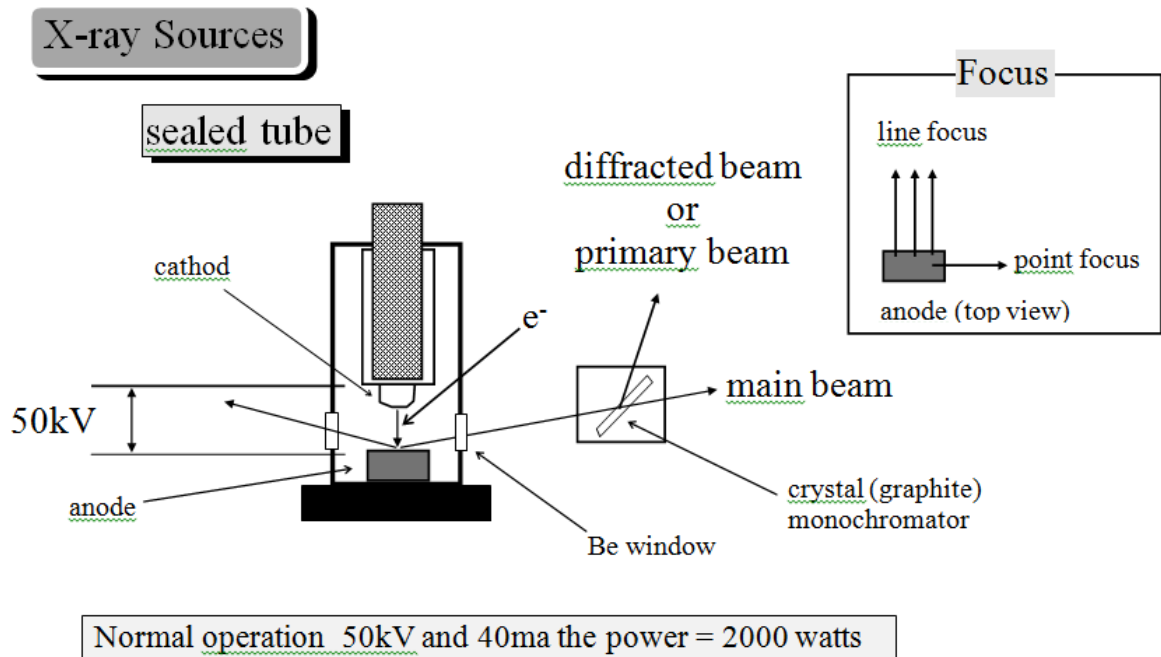
Whole body	5rems/yr. (elbows up; knees up)	2.5mR/hr
Long-term	(N-18)* 5rems	
Skin	15 rems/yr.	
Hands	75 rems/yr.	
Forearms	30 rems/yr.	
non-occupational	0.17 rem/yr.	
minors	0.17 rem/yr	0.01 mR/hr
pregnant (declared)	0. 5 rem/9-month (must be declared in writing)	

e) **Background Radiation**

	Highest (world)	5 rem/yr.	Kerala, India
	Highest (U. S.A)	0.2 rem/yr	Leadville,
Colorado	Average(world)	0.09 rem/yr	
	Lowest(U.S.A)	0.07 rem/yr	Atlantic/Gulf

coast

X-ray Source.



**Primary Beam Dosimetry**

**Radiation Level**

Inverse Rate Law

$$R/hr = 2432 * [(kV * mA)/cm^2] * Z \text{ atomic number of the target}$$

X-rays - **'Soft'** radiation - easily attenuated **but** never attenuated to **zero**.

$1/d^2$  decrease (standard temperature and pressure)

X-ray diffraction -

monochromatic

- reduces the radiation level ( $\sim 10^5$ )

Filtered

- reduces the radiation level ( $\sim 10^1$ )

collimated beams

directional

separate user from the source

reduce the primary radiation area

### **Leakage Dosimetry**

Normally very low for X-ray Diffractometers unless collimator and/or monochromator misaligned. (labyrinth design, well fitting joints)

Almost negligible leakage from tube housing.

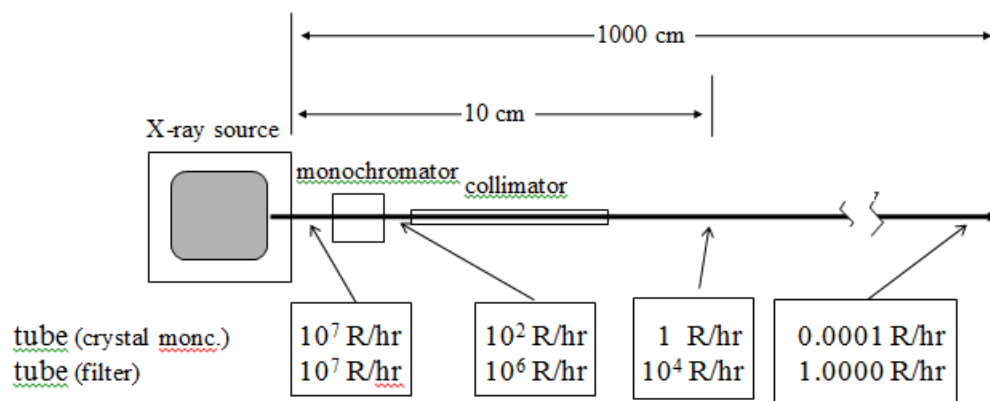
Stray X-ray beam directions tend to be at large angles away from the direction of the primary beam.

### **Adventitious Radiation.**

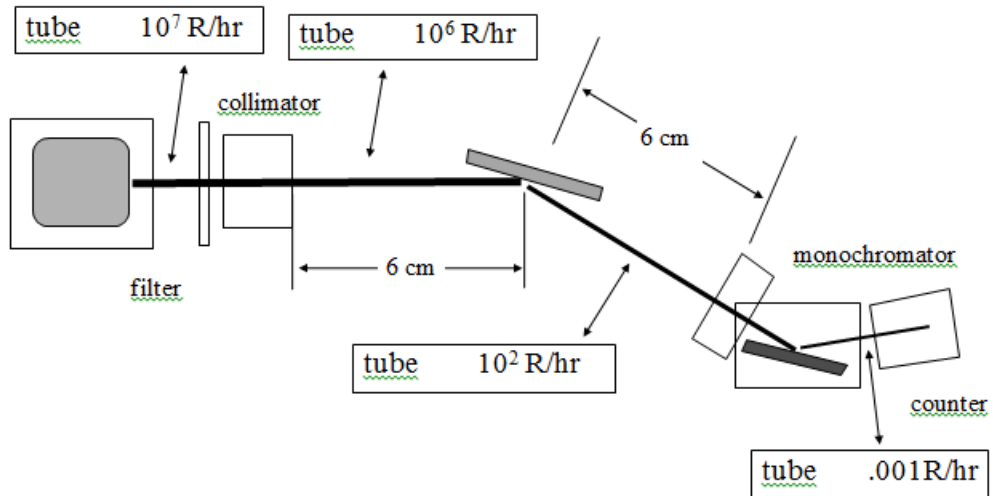
High voltage rectifiers. (an X-ray tube is a rectifier)

## Radiation Safety

### single-crystal diffractometer



### powder diffractometer





## Radiation Measurement

### Personal Dosimetry

(not employed at this location)

a) types

-LiF Thermoluminescent dosimetry **TLD** Pocket and extremity badges. (lowest detectable dose 2-3 mrad ; highest 105 rad) -Film badge

-Pocket ion chamber (pencil dosimeters; show immediate response)

- usage

-badges should be worn in such a way as to maximize the possibility of exposure in case of accident. (line of sight)

- advantages

usually first sign that something has gone wrong. extremity dosimetry is the most useful legal aspects

- disadvantages

-directionality of the primary beam and scattered radiation. monitoring devices must sustain a direct hit a 1/ 10000 chance for pocket and 1 /100 chance for extremity badges -nuisance (if too troublesome it won't be used) -expensive

- dosimetry exclusions

-workers who are likely not to exceed the max. yearly limits. -dosimeters will be distributed on a rotating basis.

## Accidental Exposure

### Rate

1 accident per 100 machines per year in 1968 and

1 accident per 200 machines per year in 1974.

for 10 X-ray diffractometers 1 accident every 10 years. 75% of those accident are with **XRD** instruments. **Relative User Risk** (increasing risk)

- routine users are the safest (lowest risk)
- advanced users (a little knowledge is dangerous)
- managers and service personal (highest risk)

## Exposure of Radiation to Flesh (localized) : Symptoms ( $10^3$ rad)

-fingers, arms, eyes etc.(finger/hand exposure is the highest risk) -depth of exposure of 10-20 keV X-rays 1.3-4.3 mm ( $t_{1/2}$ ) -non-stochastic (skin reaction)

0-1 hr.	tingling
1-7 days	swelling, blistering, pain, erythema, hair loss (epilation), skin loss (desquamation)
7-30 days	ulcers (sores that will not heal), gangrene
30-300 days	loss of digits.
300+ days:	cataracts, skin grafts

As dose increases the time for symptoms to appear decreases

### Other doses

500-1000 rad	erythema and epilation
300-500 rad	mild erythema, some epilation
300 rad	lowest visible detection of skin damage
1-300 rad	no visible skin damage
0.1-1 rad	significant dose (over-exposure)

As beam area decreases the dose required to produce skin damage increases.

stochastic (radiation-induced mutation : cell damage : cancer)

- No known threshold dose!
- risk is roughly proportional to the dose<sup>2</sup>
- >100 rad is considered significant
- >3000 rad cause cell sterilization - highest risk is between 2500-3000 rad
- known case for exposure above 2000 rad
- most cancers take more than 10 years to manifest symptoms
- for workers exposed to 100rem/life show only a statistical decrease in life expectancy of 1%. (70 days for 70 years life expectancy)
- statistically one day of life loss for each rem of exposure.
- exposure is accumulative (rem/life)

## Accident Detection

- X-rays are invisible (no sight, sound or taste)
- X-rays do not generate heat in tissue. (no touch)  
400 rad will raise the skin temperature by only 0.001° C
- X-rays do not produce detectable amounts Of O<sub>3</sub>, NO etc. (no smell).
- Personal Dosimetry
- Biological/Health Effect

## Bibliography

"Procedures Manual for Use of Radioisotopes and Radiation Producing Devices",  
Office of Radiological Safety, Texas A & M University

"A Guide to the Safe Use of X-ray Diffraction and Spectrometry Equipment", Martin,  
E., Science Reviews Ltd., Ash Drive, Leeds, LS 17 8RA U.K.

"A Case History of Severe Radiation Burns from 50 kVC X-rays", Steidley, K.,  
Stabile, R. & Santillippo, L. *Health Physics* (1981). 40 399-405.

"Analytical X-ray Hazards: A Continuing Problem" Lubenau, J., Davis, J., McDonald,  
D. & Gerusky, T. *Health Physics* (1969). 16, 739-746.

"Occupational Hazards in X-ray Analytical Work" Lindel, B. *Health Physics* (1968).  
15, 481-486.

"Incidence, Detection and Monitoring of Radiation from X-ray Analytical  
Instrumentation" Jenkins, R. & Haas, D. *X-ray Spectrom* (1975). 4, 33-39.

"Protection Against Radiation Injury" Cook, J. & Oosterkamp, W. *International Tables  
for Crystallography* (1962). 333 -338.

"Characteristic X-ray Flux from Sealed Cr, Cu, Mo, Ag, W Tubes" Honkimski, V.,  
Sleight, J. & Suortti, P. *J Appl. Cryst.* (1990). 23, 412-417.