

X-ray Diffraction Laboratory

10/23/2014

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 two x-ray diffraction instruments located in rooms 1420. 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
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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 "APEX2" 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 "APEX2" 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

There are two X-ray shutters on the APEX23 system. The first is the safety shutter that is behind the monochromator and the second is the rotary shutter in front of the monochromator.

Normal operation :

- **Green diode** on the top back rotary shutter is lit.
- **RED** X-ray on light on front panel will be lit.
- Yellow light will glow on generator (lower left) will be lit.
- Safety shutter opens (loud click sound) and the red **shutter open** light is seen on the front panel.
- Rotary shutter opens and the **red diodes** on the top front of the rotary shutter assembly will come on.

Front/side/rear panels.

Normal operation :

- **Green light** appears on front lower panel if panels are in place and closed.
- **Red light** appears for every upper panel that is in the open position.
- Safety shutter will not open if any red light on the front lower panel is lit.
- The *reset button* must be depressed if any panel has been opened.
- The safety key lock (located on the lower front panel) will override the Safety shutter interlock and **allow the shutter to operate when the panel(s) are open.**
- A load “beep” is heard and RED Safety Interlock Override is lit, when the key has been activated and the safety interlocks are OFF.
- The safety key lock is normally locked and the key removed.
- **Do not operate if safety key is in place and/or “beep” is active.**

Power Connection and Disconnection

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

Depressing the **RED** button on the front lower left-hand generator panel of the diffractometer will disconnect the power to the X-ray tube.

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 stand-by switch on generator panel (lower left).
- 4) A click is heard and panel indicator will say KV 20 MA 5.
- 5) Depress on switch. Yellow indicator and Red x-ray on will be lit.
- 6) Use program to ramp generator to KV 40 MA 40.

De-energizing X-rays

- 1) Depress stand-by switch on lower left-hand generator panel.
- 2) Yellow Light will not be lit.
- 3) Wait 5 mins (to allow tube to cool) and depress OFF button.

3.0 Radiation Safety Information

Radiation Units

- a) **Roentgen (R)** : amount of radiation that produces one unit of ions/cm³. (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>)

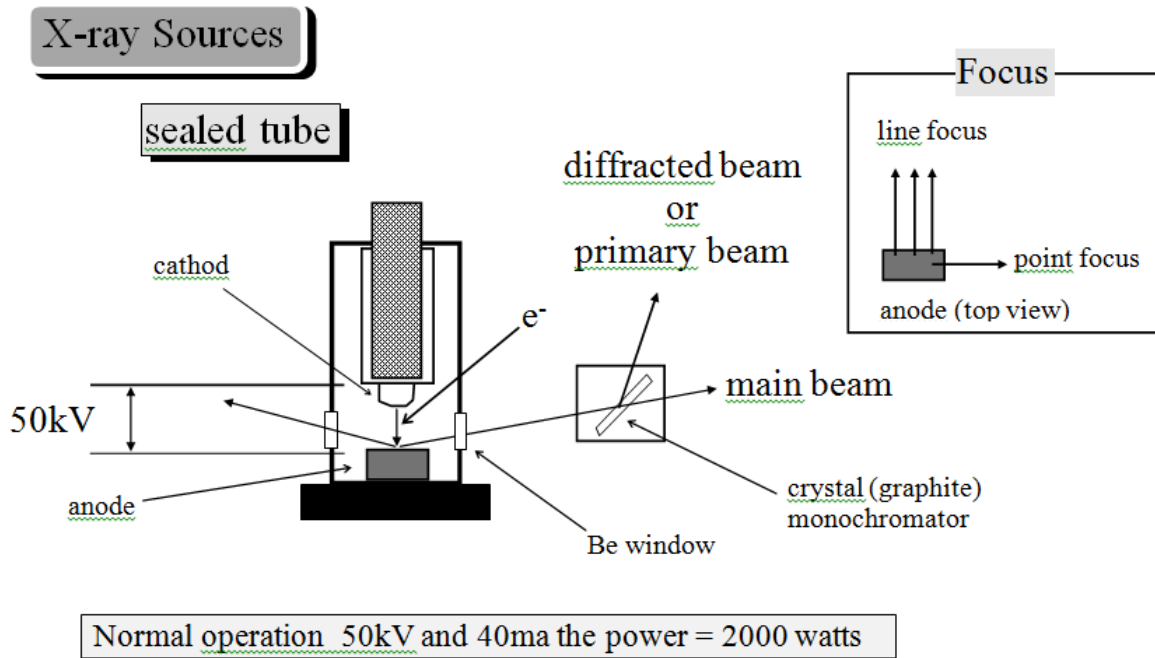
Whole body	5rems/yr. (elbows up; knees up)	2.5mR/hr
Long-term	(N-18)* 5rems	
Skin	15 rems/yr.	
Hands	75 rems/yr.	
Forearms	30 rems/yr.	

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
 Filtered
 collimated beams

- reduces the radiation level ($\sim 10^5$)
 - reduces the radiation level ($\sim 10^1$)
 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.

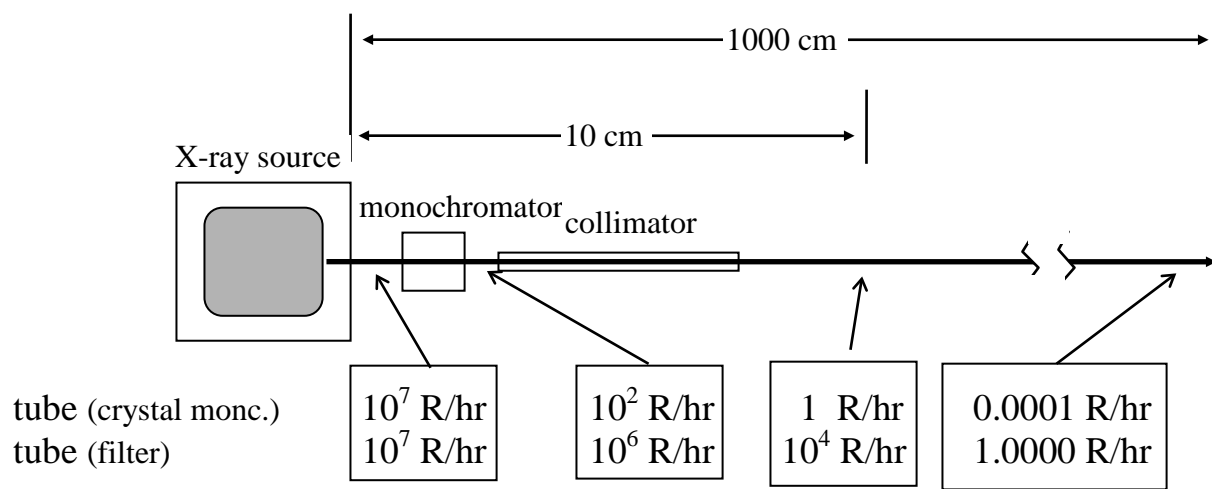
Stray X-ray beam directions tend to be at large angles away from the direction of the primary beam.

Adventitious Radiation.

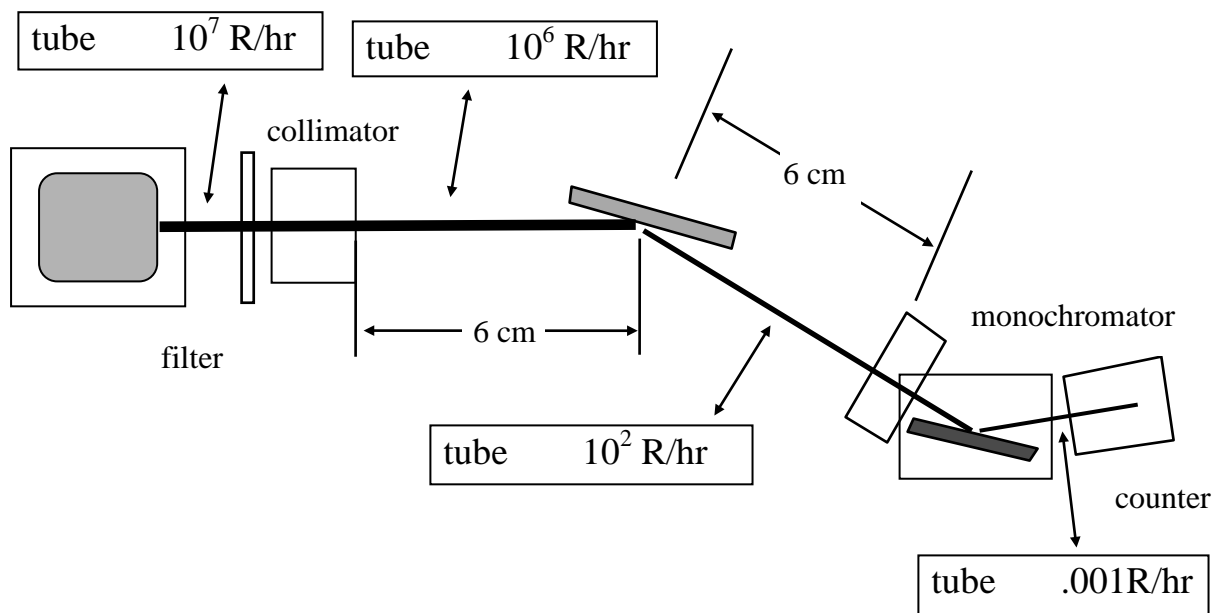
High voltage rectifiers. (an X-ray tube is a rectifier)

Radiation Hazard

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 dose²
- >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)

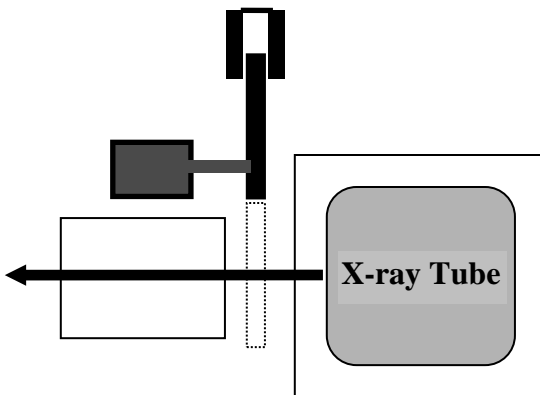
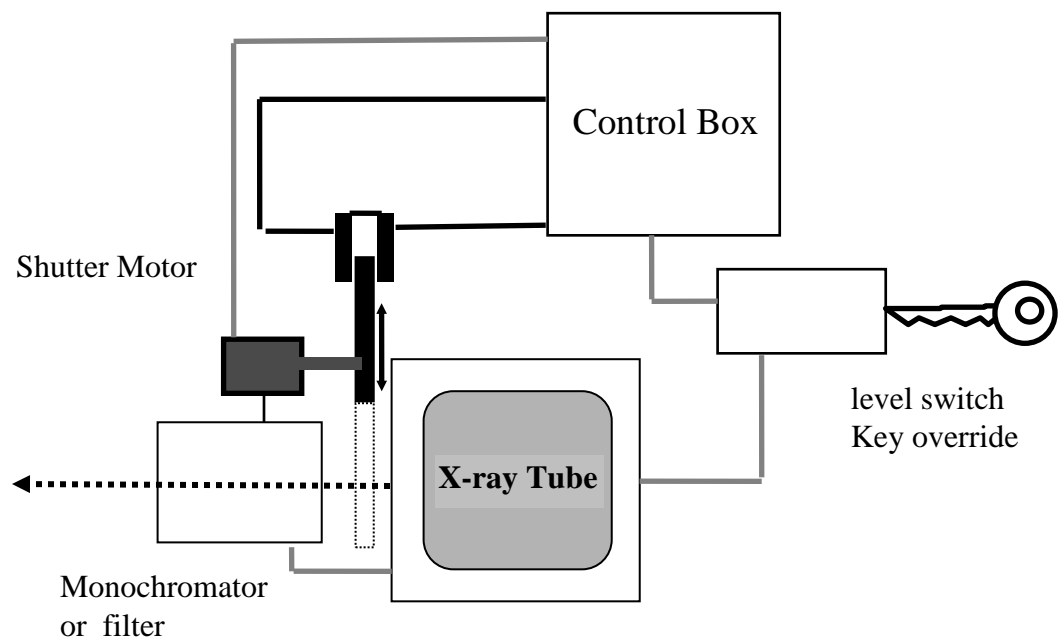
Accident Detection

- 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 O₃, NO etc. (no smell).
- Personal Dosimetry
- Biological/Health Effect

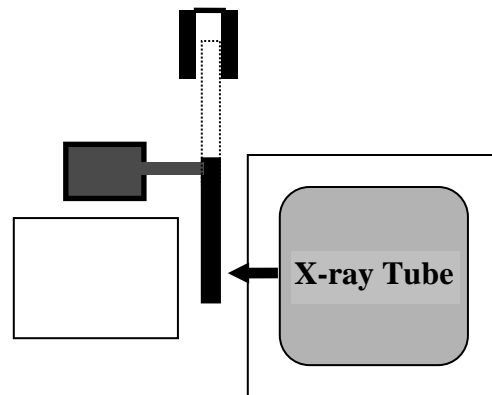
Shutter Safety Interlock

Instrument : Bruker X-ray diffractometer

Location : Room 1420 Chemistry



Shutter Open



Shutter Closed

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