Purpose
Analytical X-ray equipment is used for X-ray diffraction analysis, fluorescence analysis, or direct X-ray transmission analysis of materials. These analytical x-ray systems are comprised of components that utilize X-rays to determine elemental composition, or to examine the microstructure of materials. These analytical x-ray devices are used for non-medical purposes. The primary objective of this program is to keep occupational exposures to radiation as low as reasonably achievable (ALARA) while utilizing these types of equipment. This safety program establishes UVa procedures for the safe operation of analytical X-ray equipment and the associated potential radiation hazards.

Section 1: Regulations
The Virginia Department of Health, specifically the Bureau of Radiological Health (http://www.vdh.virginia.gov/Epidemiology/RadiologicalHealth/), regulates the use of all X-ray producing equipment. The following regulations pertain to analytical X-ray equipment.

12 VAC 5-481-2130. Personnel requirements.
A. Instruction.
No individual shall be permitted to operate or maintain analytical X-ray equipment unless such individual has received instruction in and demonstrated competence as to:
1. identification of radiation hazards associated with the use of the equipment;
2. significance of the various radiation warning, safety devices, and interlocks incorporated into the equipment, or the reasons they have not been installed on certain pieces of equipment and the extra precautions required in such cases;
3. proper operating procedures for the equipment;
4. recognition of symptoms of an acute localized exposure; and
5. proper procedures for reporting an actual or suspected exposure.

B. Personnel Monitoring.
1. Finger or wrist dosimetric devices shall be provided to and shall be used by:
a. analytical X-ray equipment workers using systems having an open-beam configuration and not equipped with a safety device; and
b. personnel maintaining analytical X-ray equipment if the maintenance procedures require the presence of a primary X-ray beam when any local component in the analytical X-ray system is disassembled or removed.

12VAC5-481-2250 Posting of notices to workers
You must post a copy of the equipment registration, “Notice to Employee”, regulations and operating procedures
Section 2: Requirements for Safe Operation

No individual shall be permitted to operate a particular x-ray diffraction machine until they have:

- Received an acceptable amount of training in radiation safety
- Demonstrated competence to use the machine and radiation survey instruments

- Only trained personnel, as approved by the Environmental Health & Safety Office, shall be permitted to **install, repair, or make other than routine modifications** to the X-ray generating apparatus and the tube housing apparatus complex.

- There may be an increase in radiation hazard following modification of the equipment. The equipment must be surveyed whenever a modification to the apparatus is made. Call EHS (2-4911) for radiation surveys and monitoring of any newly installed or relocated machines and especially when the machine has been modified for special experiments.

  A survey meter with a low-energy NaI detector, rather than a G-M detector, is the most appropriate detector to survey for the low-energy x-rays associated with x-ray diffraction work.

- Personnel shall not expose any parts of their bodies to the primary radiation beam

- 12VAC5-481-2120 - Normal operating procedures shall be written and available to all analytical X-ray equipment workers. No individual shall be permitted to operate analytical X-ray equipment in any manner other than that specified in the procedures unless such individual has obtained written approval of the radiation safety officer.

- Bypassing. No individual shall bypass a safety device or interlock unless such individual has obtained the approval of the radiation safety officer. Such approval shall be for a specified period of time. When a safety device or interlock has been bypassed, a readily discernible sign bearing the words "SAFETY DEVICE NOT WORKING", or words having a similar intent, shall be placed on the radiation source housing.

- Repair or modification of X-ray tube systems. Except as specified in 12VAC5-481-2450 B, no operation involving removal of covers, shielding materials or tube housings or modifications to shutters, collimators, or beam stops shall be performed without ascertaining that the tube is off and will remain off until safe conditions have been restored. The main switch, rather than interlocks, shall be used for routine shutdown in preparation for repairs.

- Radioactive source replacement, testing, or repair. Radioactive source housings shall be opened for source replacement, leak testing, or other maintenance or repair procedures only by individuals authorized to specifically conduct such procedures under a license issued by the Nuclear Regulatory Commission, an agreement state, or a licensing state.
**Section 3: Radiation Basics**

X-rays are known as ionizing radiation because X-rays possess sufficient energy to remove electrons from the atoms with which the X-rays interact. There are three quantities primarily used for describing the intensity of an x-ray beam:

- Exposure
- Absorbed Dose
- Dose Equivalent

**Exposure** is a quantity describing how much ionization is produced (i.e., how many electrons are produced as X-rays or gammas create ionization) in air by gamma- or X-rays. The unit of exposure is the **Roentgen**.

1 Roentgen (R) = 2.58 x 10^-4 Coulombs of charge produced per kg of air.

**Limitations:** Exposure applies only to x-rays and gamma rays and describes only the effect on air, not on tissue.

**Absorbed dose** is a quantity describing how much energy is deposited in a material by a beam of radiation and is not restricted to x-rays or gamma rays passing through air. The unit of absorbed dose is the **rad**.

1 rad = 100 ergs of energy deposited in one gram of material

**Limitations:** Absorbed dose does not indicate the effectiveness of various types of radiation in causing biological harm. Different types of radiation may deposit the same amount of absorbed dose but produce different effects and different levels of damage. For instance, charged massive alpha particles will interact more intensely and deposit energy over a shorter distance within a cell than uncharged massless gamma rays. Consequently, some radiations are more effective than other radiations at producing biological damage, even though equivalent amounts of energy are deposited overall. The quantity, dose equivalent, described in the next paragraph takes into account the abilities of differing radiations to cause damage.

**Dose equivalent** is a quantity derived by multiplying the absorbed dose by a quality factor (QF) which depends on the type of radiation being measured. Consequently, dose equivalent does reflect the ability of each type of radiation to cause damage. The unit of dose equivalent is the **rem**.

- Dose equivalent = Absorbed dose x QF
- QF = 1 for gamma rays, x-rays and most beta particles

For the remainder of this module, references to radiation intensity and dose will be expressed in terms of rems or millirems.
Section 4: Hazards Associated with Analytical X-Ray Equipment

Analytical X-ray equipment has become a major tool in research and quality control programs. Despite the advances in operating techniques and equipment design, the most common hazards are due to operator errors and equipment malfunctions. X-ray diffraction machines make use of beams of extremely high intensity and although the primary beam has a small diameter it can produce severe and permanent local injury from only momentary irradiation of the body. The greatest risk of acute accidental exposures occurs in manipulations of a sample to be irradiated by the direct beam in diffraction studies.

The potential exposure to the primary beam is a major concern when evaluating potential radiation exposures. Exposures to the primary beam in a typical analytical X-ray unit may be as great as 100,000 R/min.

Erythema (reddening of the skin) would be produced after an exposure of only 0.03 seconds, and in 0.1 second severe and permanent injury could occur. The fingers, of course, are the parts of the body most likely to receive these high exposures.

Generally, occupational workers are only exposed to scattered radiation which is principally from radiation interactions in the sample or surrounding equipment. These values are very general and will depend on the type of unit, amount of intervening shielding and location of the worker during operation of the unit.

Research (x-ray diffraction; spectrometers):

- **In Primary Beam:** 1000 to 100,000+ R/sec (small cross-sectional area)
- **From Scatter:** Negligible

In research, analytical X-ray units are used to irradiate samples. There is minimal radiation dose from scatter due to the inherent design of the equipment. With these types of units, it is imperative that safety interlocks operate according to their design. Any steps taken to modify the primary beam could result in unnecessary radiation exposure to the operator. Operators should be fully cognizant of the protective devices incorporated into their machines and the possibilities for failure or malfunction. Operators working with older machines must be especially careful of the possibilities of receiving excessive amounts of radiation.

X-Ray producing equipment can be dangerous to both the operator and persons in the immediate vicinity unless safety precautions are strictly observed.

Exposure to excessive quantities of X-radiation may be injurious to health. Therefore, users should avoid exposing any parts of their persons, not only to the direct beam, but also to secondary or scattered radiation which occurs when an X-ray beam strikes or passes through any material.

Human beings have no senses for detecting X-rays. Therefore, X-ray measuring instruments, like low energy X-ray Geiger counters, must be used to detect X-ray emission or leakage radiation.

X-Ray producing equipment is usually installed in a room or cabinet providing an adequate radiation barrier. The user should always be aware that the useful X-ray beam can constitute a distinct hazard if not employed in strict accordance with instructions composed to provide maximum safety for the operator.
The electrical circuits, although enclosed and interlocked for the protection of operators, must be considered a potential source of hazard, requiring strict observance of those portions of instructions pertaining to safety in operation and maintenance. Proper electrical grounding must always be in effect.

Consequently, adequate precautions should be taken to make it impossible for unauthorized or unqualified persons to operate this equipment or to expose themselves or others to its radiation or electrical dangers.

The maximum operating voltages and currents, or ranges of voltages or currents, are set at or established by the factory and should not be altered except as provided for in the manufacturer’s instructions. If established limitations are exceeded, the effectiveness of the incorporated shielding may be reduced to a point where the penetrating or emergent radiation may exceed safe values. If radiation shielding shows chemical or mechanical damage, service personnel should be notified immediately to prevent accidental radiation exposure.

The specific hazards of analytical x-ray equipment can include:

- Exposure to an intense, localized primary x-ray beam
- Exposure to diffracted and/or scattered portions of the primary x-ray beam (includes x-ray leakage)

**Four Main Causes of Accidents**

- Poor equipment configuration, e.g., unused beam ports not covered
- Manipulation of equipment when energized, e.g., adjustment of samples or alignment of cameras when x-ray beam is on
- Equipment failure, e.g., shutter failure, warning light failure
- Inadequate training or violation of procedure, e.g., incorrect use of equipment, overriding interlocks

**Interlocks**

Interlock switches should be built into all access doors of rooms or cabinets. These switches should under no circumstances be tampered with and should be maintained in proper operating condition. In no case should they be defeated or wired out, since failure of automatic high voltage protection will then result.

**Maintenance**

All parts of the equipment, particularly interlock switches, should be carefully maintained for proper operation. Doors should close sufficiently to prevent access before interlock switches close. Tube operating voltage and current should be checked whenever the equipment is operated by service personnel.
Servicing Precaution
Before changing tubes or making any internal adjustments, the equipment shall be disconnected from the power supply to insure that no X-ray emission can occur. Care should be taken to assure that all high voltage condenser charges are removed using insulated grounding lead, before personal contact is established.

Supervision
X-ray Producing equipment should be used only under the guidance and supervision of a responsible qualified person. The instruction manual supplied with each unit clearly specifies the system’s intended operation. All equipment operators must be given adequate radiation safety instructions as specified by the Virginia state regulations.

Preventing Accidents
- Know location and/or presence of primary and diffracted beams AT ALL TIMES
- Provide and inspect shielding
- Do not perform maintenance without confirming that tube is not energized
- Perform check of safety devices at least once a month
- Survey unit whenever moved or reconfigured
- Cap off any unused ports
- Don’t put your body parts in the beam!
Section 5: Biological (Health) Effects of X-Ray Exposure

X-rays produced by X-ray diffraction equipment are normally too low in energy to be deeply penetrating. With X-ray diffraction equipment, skin effects are the effect of concern. There is no concern for genetic effects or prenatal radiation exposure for x-ray diffraction users.

Analytical X-ray equipment makes use of very narrow collimated X-ray beams of high intensity. Exposure of the skin to the primary X-ray beam may result in doses within fractions of a second that will result in severe radiation burns. These burns heal poorly, and on rare occasions have required amputation of fingers.

<table>
<thead>
<tr>
<th>Possible Radiation Intensity Near Analytical X-Ray Equipment</th>
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<tbody>
<tr>
<td><strong>Location</strong></td>
</tr>
<tr>
<td>Primary beam at tube port</td>
</tr>
<tr>
<td>Primary beam at end of 10 cm collimator</td>
</tr>
<tr>
<td>Scattered radiation near sample</td>
</tr>
</tbody>
</table>

Mechanisms of Damage

Injury to living tissue results from the transfer of energy to atoms and molecules in the cellular structure. Ionizing radiation causes atoms and molecules to become ionized or excited. These excitations and ionizations can:

- Produce free radicals.
- Break chemical bonds.
- Produce new chemical bonds and cross-linkage between macromolecules.
- Damage molecules that regulate vital cell processes (e.g. DNA, RNA, proteins).
- The cell can repair certain levels of cell damage. At low doses, such as that received every day from background radiation, cellular damage is rapidly repaired.

At higher levels, cell death results. At extremely high doses, cells cannot be replaced quickly enough, and tissues fail to function.

Prompt and Delayed Effects

Radiation effects can be categorized by when they appear.

**Prompt, acute effects** include effects such as skin reddening, hair loss and radiation burns, which develop soon after large doses of radiation (hundreds to thousands of rems) delivered over short periods of time (seconds to minutes).

**Delayed effects** include effects such as cataract formation and cancer induction that may occur months or years after a radiation exposure.
Prompt Effects

The following information is adapted from the Acute Radiation Syndrome Fact Sheet for Physicians, published by the Center for Disease Control:

*When the skin receives a high dose of radiation, the primary damage occurs to hair follicles, basal (dividing) cells of the outer skin layer, and small blood vessels.*

*When the basal cell layer of the skin is damaged by radiation, inflammation, erythema (skin reddening), and dry or moist desquamation (shedding of the outer layers of the skin) can occur. Also, hair follicles may be damaged causing epilation (loss of hair). Within a few hours after irradiation a transient and inconsistent erythema (associated with itching) can occur. Then, there may be a latent phase that lasts from a few days up to several weeks, when intense reddening, blistering and ulceration of the irradiated site is visible. In most cases healing occurs by regenerative means; however, very large skin doses can cause permanent hair loss, damaged sebaceous and sweat glands, atrophy, fibrosis, decreased or increased skin pigmentation, and ulceration or necrosis of the exposed tissue.*

At doses in excess of two thousand rems, destruction of the skin occurs due to direct skin cell death, loss of basal cells, or reduced blood flow due to destruction of small blood vessels. Pain is associated with loss of the integrity of the skin because nerves can become exposed to the air, die due to loss of blood flow, or be affected by infection of the damaged skin.

Although irradiation of fingers or hands with x-rays at energies of about 5 - 30 keV does not seem to result in significant damage to blood-forming tissue, at high exposures some general somatic effects to the skin can occur. Very high exposures may necessitate skin grafting or amputation of the affected extremity.

The medical response to these acute effects involves administering drugs to prevent infection, to control pain and to improve blood flow, but in the case of tissue necrosis, it is generally necessary to resort to skin grafting or amputation of the necrotic portion.

The photos below show damage to the hands from high-dose x-ray exposure
The following table summarizes the effects and the threshold dose required to produce these acute effects:

<table>
<thead>
<tr>
<th>Effect</th>
<th>Threshold Dose</th>
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</thead>
<tbody>
<tr>
<td>Erythema (skin reddening)</td>
<td>24-48 hrs</td>
</tr>
<tr>
<td></td>
<td>300 - 500 rem</td>
</tr>
<tr>
<td>Temporary hair loss</td>
<td>24-48 hrs</td>
</tr>
<tr>
<td></td>
<td>300 - 500 rem</td>
</tr>
<tr>
<td>Permanent hair loss</td>
<td></td>
</tr>
<tr>
<td></td>
<td>700 rem</td>
</tr>
<tr>
<td>Transepidermal injury (skin burns)</td>
<td></td>
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<tr>
<td></td>
<td>1000 rem</td>
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<tr>
<td>Dermal radionecrosis (tissue death)</td>
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</tr>
<tr>
<td></td>
<td>2000 - 3000 rem</td>
</tr>
</tbody>
</table>

Additional notes concerning prompt effects:

- These acute effects will develop within hours, days or weeks, depending on the size of the dose. The larger the dose, the sooner a given effect will occur. For example, at doses of 300 rems, it may take 1-3 weeks for erythema to develop, but, at doses of thousands of rems, erythema may develop within hours to days.

- These acute effects are limited to the site of exposure. For example, if a portion of the hand is exposed to a very large dose, skin reddening or burns is limited to the section of the hand that received the high dose.

Example of Radiation Damage

Erythema Of Right Hand 4th Day, 17th Day, 3 ½ Months, Five Months, Seven Months After Accident
Delayed Effects of Radiation Exposure

Cataracts

Cataracts, or clouding of the lens of the eye, are induced when a dose exceeding approximately 500 rems is delivered to the lens of the eye. Radiation-induced cataracts may take many months to years to appear. It is extremely unlikely to receive a substantial dose to the lens of the eye when working with some types of cabinet type XRDs. It would be very difficult to place one's head in a position near enough to the primary beam to receive a dose sufficient to induce cataracts.

Cancer

Studies of people exposed to high doses of radiation (on the order of hundreds to thousands of rem) have shown that there is a risk of cancer induction associated with high doses. These studies demonstrate that cancer risk is linearly proportional to the dose in the high dose region. The specific type of cancer associated with low-energy x-ray exposure is skin cancer. Radiation-induced cancers may take 10 - 15 years or more to appear. There may be a risk of cancer at low doses as well. The following material discusses the risk of cancer at lower doses.
Section 6: Radiation Protection

There are three general principles of radiation protection: *time, distance, and shielding*.

**Time**
*Decreasing the amount of time* spent in the vicinity of the source of radiation will *decrease* the amount of radiation exposure. Radiation doses are approximately directly proportional to the time spent in a radiation field. Although reducing time in the radiation field to reduce exposure is a very simple concept, it is a very effective concept as well.

**Distance**
*Increasing the distance* from a source of radiation will *decrease* the amount of radiation exposure. Radiation doses will decrease approximately as the inverse square of the distance from the radiation source.

**Shielding**
*Increasing the amount of shielding* around a source of radiation will *decrease* the amount of radiation exposure. Shielding for analytical x-ray units can range from the use of leaded glass to enclosures constructed of tin-impregnated polycarbonate.

**Precautions and Guidelines for Use of Analytical X-Ray Equipment**

The operator of the X-ray diffraction or spectrographic equipment shall be responsible for all operations associated with that equipment, including radiation safety.

- **Maintain an Operators List** which indicates that individuals listed understand the safety procedures and techniques factors for the equipment that they are assigned to operate
- **Keep radiation exposure to you and others as low as is reasonably achievable** (ALARA).
- **Personnel shall not expose any parts of their bodies to the primary radiation beam**
- **Be familiar with safety procedures as they apply to each machine you operate**
- **Wear personnel monitoring devices, if applicable**
- **Have Emergency Instructions posted nearby**
- **In the event of a known or possible exposure to the beam, notify EHS immediately**
  - Arrange for medical examination, being sure to notify the examining physician that exposure to low energy X-rays may have occurred
- **Equipment failures that have been reported as resulting in radiation injury have involved defective shutters over the tube head ports. Even when shutters are provided, they must be inspected and monitored regularly.**
  - Shutter interlocks should be used to cut off the beam when samples are changed.
- **No repair, cleaning work on shutters and shielding material or other non-routine work that could result in exposing anyone to the primary beam should be allowed unless it has been positively ascertained that the tube is completely de-energized.**
- **Well-planned formal training and sessions should be conducted to ensure all users fully training in use of equipments**
- **Equipment should be secured so it cannot be used or approached by unauthorized personnel**
• The analytical x-ray equipment should be placed in a separate room from other work areas whenever practical.

• Properly installed permanent shields should be used in preference to temporary shielding. When temporary shielding is necessary, it must be securely fastened.

• Set-up procedures will be carried out with the x-ray beam off or with shutters closed as much as possible. If the latter, a survey shall be performed before starting set-up.

• Under normal operating conditions, always turn off the machine high voltage before opening the enclosure (for enclosed beam systems) or before taking any action which could expose the primary beam path (for open beam systems).

• Each day before using the machine, open and close the shutter a few times to check that the shutter is functioning properly.

• Never assume that the unit was left in a safe working condition by the previous user. Check the shielding and interlock status before turning the unit on.

• Do not bypass any safety device or interlock without the approval of the person responsible for the machine. When any portion of the safety devices are disabled, post a conspicuous sign stating the date, your name and listing what has been disabled. Return the machine to its unmodified state with all interlocks and safety devices operational as soon as possible.

• Do not work near the open, unshielded beam. However, if it is necessary to work near the unshielded beam (e.g., during system alignment):
  o Reduce the beam current and high voltage to the lowest possible settings to reduce exposure rates.
  o Keep hands and body as far as possible from the beam by using appropriate alignment tools.
  o You are in a potentially hazardous situation. Think before each step.

• Know what you are doing and where to expect problems. Be aware of the dangers. Do not work in a hurry or allow yourself to become distracted.

• Before energizing the x-ray tube, all ports will be closed or fitted with an approved apparatus.

• Unused ports on radiation source housings shall be secured in the closed position in a manner that will prevent casual opening.

• Shutters should be used that can not remain open unless a collimator is in position.

• An easily visible warning light labeled with the words “X-RAY ON”, or words having a similar intent, shall be located near any switch that energizes the x-ray tube an shall be illuminated only when the tube is energized or in the case of a radioactive source, near any switch that opens a housing shutter and shall be illuminated only when the shutter is open.

• Warning devices shall be labeled so that their purpose is easily identified. On equipment installed after September 20, 2006, warning devices shall have fail-safe characteristics.

• All safety devices such as interlocks, shutters, warning light, etc. will be tested weekly or upon each use (if use is less frequent) to ensure proper operation.

• Safety interlocks will not be routinely used to deactivate the x-ray beam.

• The unit will be positioned so that the primary x-ray beam is completely attenuated by the beam catcher. If this is not possible for an experimental set-up, or if the accessible scatter dose rate to personnel exceeds 2.0 mR/hr, then either the entire unit will be shielded, or entry to the room must be restricted. The room must be locked and appropriately posted with “CAUTION – RADIATION AREA”. 
• Each radioactive source housing or port cover or each X-ray tube housing shall be so constructed that, with all shutters closed, the radiation measured at a distance of five centimeters from its surface is not capable of producing a dose in excess of 2.5 millirems (0.025 mSv) in one hour. For systems utilizing X-ray tubes, this limit shall be met at any specified tube rating.

• Generator cabinet. Each X-ray generator shall be supplied with a protective cabinet that limits leakage radiation measured at a distance of five centimeters form its surface such that is not capable of producing a dose in excess of 0.25 millirem (2.5 uSv) in one hour.

• Open beam configurations shall be provided with a readily discernable indication of X-ray tube “on-off” status located near the radiation source housing, if the primary beam is controlled in this manner; and or Shutter “open-closed” status located near each port on the radiation source housing, if the primary beam is controlled in this manner.
  
  **Note:** Open beam techniques should only be used after all attempts at an enclosed system have proven this to be impracticable.

• Each X-ray tube housing shall be equipped with an interlock that shuts off the tube it is removed from the radiation source housing or if the housing is disassembled.

• The analytical X-ray equipment user should be in immediate attendance at all times when the equipment is in operation unless otherwise approved by the Radiation Protection Program.

• When not in operation, the equipment will be secured in such a way as to be accessible to or operable by analytical x-ray equipment users only (i.e. keylocks).

• Particular attention should be given to viewing devices to ensure that lenses and other transparent components attenuate the radiation beam to minimal levels when alignment involves working near the open primary beam. The beam current should be reduced when a fluorescent alignment tool is used; dimming the room light will permit a significant reduction in beam current. The fluorescent alignment tool should be long enough to permit the analytical x-ray equipment user’s hand to be kept a safe distance from the beam.
Section 7: Radiation Surveys

The following surveys are required for use of analytical X-ray equipment:

1. Radiation survey will be performed prior to each new use of the x-ray equipment.
2. Weekly surveys should be performed to ensure radiation levels have not changed.
3. Periodic surveys should be performed on the local components to check for leakage radiation when the set-up is changed.

The RADIATION PROTECTION PROGRAM will make surveys according to the following guidelines:

1. Upon the installation of the x-ray equipment and at least once a year thereafter.
2. Upon any change in the initial arrangement, number, or type of local components in the system.
3. Upon any maintenance requiring the disassembly or removal of a local component in the system.

Notifications

- Notify EHS if the unit or tube is moved or modified before resuming use
- Notify EHS of new X-ray installations before use
- Notify EHS if X-ray equipment is removed from the University, becomes inoperable for a long period of time, or is placed into long-term storage
- In case of a medical emergency, call 911 immediately
- Report any real or suspected radiation exposure to supervisor and EHS.

You must read and sign that you have read and understood the above training and Applicable State Regulations attached.
Radiation Safety Inspection of Analytical X-ray Equipment

SURVEY DATE ___________________________ State Registration # ________________

LOCATION (Building/ Rm#/ Dept.) ____________________________

Equipment Type
☐ X-ray diffractometer
☐ R/F unit for non-human use
☐ Other

INSTALLATION DATE ____________________________

OTHER USERS ____________________________

MACHINE USED FOR ____________________________

Number of ports ____________________________

Normal operating parameters, such as maximum tube voltage and current, type of sample, amount of time the X-ray tube is on, and the extremes of goniometer travel

Description and diagram of apparatus used with diffraction unit, such as cameras, diffractometers, x-ray tube type, special slits, collimators, couplings, etc.

Special operating conditions for alignment, use with interlocks disabled, use without beam stop, etc.
Pre-operations check of diffraction unit

☐ Check of operating controls, safety devices, and experimental apparatus should be made before X-ray tube is turned on
☐ Note location and function of switches and indicator lights for electrical power, X-ray tube and shutters
☐ Unused X-ray tube ports have adequate shields which are fastened in place securely
☐ Shutter checked for smooth, positive and reliable action
☐ Apparatus, no missing parts, especially beam stops, shields, couplings, etc, parts should fit properly
☐ Viewing glasses in place and should be of lead glass
☐ Required shielding secured in place and not simply wrapped around parts or held with tape
☐ Any modification of the apparatus which could affect any of the safety devices or radiation level should be noted
☐ Check for radiation damage or corrosion of parts exposed to the direct beam, especially plastic parts or closefitting metal parts in locations where air is not free to circulate and corrosive gases can accumulate
☐ Electrical system power supply cabinet interlocks and high voltage cables checked for defects
☐ After pre-op checks X-ray tube should be turned on and set for maximum normal values of voltage and current. With all tube ports closed, tube housing, shutters and power supply should be checked for leakage.
☐ Shutter of port to be used should be opened and all parts of camera or diffractometer checked for excessive radiation levels.
☐ System should be operated with either normal sample or a dummy sample in position and should be checked throughout range of sample and detector orientations. (Negative angles can result in very high rad levels.) Completely closed systems should be insensitive to changes such as sample type and orientation.
☐ Mechanical parts such as sliding shutters and couplings which have number of possible positions should be surveyed throughout range of travel
☐ Check rad levels with ports open individually and in combination.

<table>
<thead>
<tr>
<th>Location</th>
<th>kV/mA</th>
<th>kV/mA</th>
<th>kV/mA</th>
<th>kV/mA</th>
<th>kV/mA</th>
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Survey Instrument Make & Model _______________________________ Serial No. _____________________
Cal Date _______________ Geometry _______________________________

☐ Posting. Each area or room containing analytical X-ray equipment shall be conspicuously posted with a sign or signs bearing the radiation symbol and the words “CAUTION - X-RAY EQUIPMENT” or words having a similar intent.
☐ Certificate of Registration Posted
☐ Notice to Employees (RH-F-12) Posted
☐ Copy of State Regulations pertaining to Use Posted or available
☐ Operating Procedures Posted
☐ List of Operators Posted
☐ Safety Device – prevents entry of any portion of an individual's body into the primary X-ray beam path or which causes the beam to be shut off upon entry into its path shall be provided on all open-beam configurations
☐ Warning Device – open beam configurations shall be provided with a readily discernable indication of X-ray tube status (ON-OFF) located near the radiation source housing, if the primary beam is controlled in this manner and/or Shutter status (OPEN-CLOSED) located near each port on the radiation source housing if the primary beam is controlled in this manner
□ Warning devices labeled so that their purpose is easily identified (on equipment installed after Jan 1, 1980, warning devices shall have fail-safe characteristics)

□ Unused ports on radiation source housings are secured in closed position in a manner which will prevent casual opening

□ Analytical X-ray equipment is labeled with a readily discernible sign or signs bearing the radiation symbol and the works:

1. “CAUTION - HIGH INTENSITY X-RAY BEAM,” or words having a similar intent, on the X-ray source housing; and

2. “CAUTION RADIATION - THIS EQUIPMENT PRODUCES RADIATION WHEN ENERGIZED,” or words having a similar intent, near any switch that energizes an X-ray tube if the radiation source is an X-ray tube; or

3. “CAUTION - RADIOACTIVE MATERIAL,” or words having a similar intent, on the source housing if the radiation source is a radionuclide.

□ Shutters. On open-beam configurations installed after January 1, 1980, each port on the radiation source housing shall be equipped with a shutter that cannot be opened unless a collimator or a coupling has been connected to the port.

□ Warning Lights.

1. An easily visible warning light labeled with the words "X RAY ON," or words having a similar intent, shall be located:
   a. near any switch that energizes an X-ray tube and shall be illuminated only when the tube is energized; or
   b. in the case of a radioactive source, near any switch that opens a housing shutter, and shall be illuminated only when the shutter is open.

2. On equipment installed after January 1, 1980, warning lights shall have full-safe characteristics.

□ Radiation Source Housing - Each radiation source housing shall be subject to the following requirements:

1. Each X-ray tube housing shall be equipped with an interlock that shuts off the tube if it is removed from the radiation source housing or if the housing is disassembled.

2. Each radioactive source housing or port cover or each X-ray tube housing shall be so constructed that, with all shutters closed, the radiation measured at a distance of 5 centimeters from its surface is not capable of producing a dose in excess of 2.5 millirem (0.025 mSv) in one hour. For systems utilizing X-ray tubes, this limit shall be met at any specified tube rating.

□ Generator Cabinet. Each X-ray generator shall be supplied with a protective cabinet which limits leakage radiation measured at a distance of 5 cm from its surface such that it is not capable of producing a dose in excess of 0.25 mrem (2.5 micro Sv) in one hour.

□ Procedures. Normal operating procedures shall be written and available to all analytical X-ray equipment workers. No person shall be permitted to operate analytical X-ray equipment in any manner other than that specified in the procedures unless such person has obtained written approval of the radiation safety officer.

□ Bypassing. No person shall bypass a safety device unless such person has obtained the approval of the radiation safety officer. Such approval shall be for a specified period of time. When a safety device has been bypassed, a readily discernible sign bearing the words "SAFETY DEVICE NOT WORKING," or words having a similar intent, shall be placed on the radiation source housing.

□ Repair of Modification of X-ray Tube Systems. Except as specified in 12VAC5-480-8620 B, no operation involving removal of covers, shielding materials or tube housings or modifications to shutters, collimators, or beam stops shall be performed without ascertaining that the tube is off and will remain off until safe conditions have been restored. The main switch, rather than interlocks, shall be used for routine shutdown in preparation for repairs.

□ Radioactive Source Replacement, Testing, or Repair. Radioactive source housings shall be opened for source replacement, leak testing, or other maintenance or repair procedures only by individuals authorized to specifically conduct such procedures under a license issued by the U. S. Nuclear Regulatory Commission, an Agreement State, or a Licensing State.
Electron Microscope Radiation Safety Survey

SURVEY DATE __________________________

LOCATION (Building/ Rm#/ Dept.) __________________________

Equipment Type________________________ Microscope MANUFACTURER & MODEL __________________________

☐ TEM (transmission electron microscope) ☐ SEM (scanning electron microscope) ☐ TEM and SEM
☐ Other __________________________

Serial Number __________________________

Installation DATE __________________________

OWNER/ AUTHORIZED USER/ Dept. __________________________

Contact Information (e.g. phone, email) __________________________

OTHER USERS __________________________

Normal operating parameters, such as maximum voltage and current, type of sample __________________________

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<thead>
<tr>
<th>Location</th>
<th>kVp / mA</th>
<th>kVp / mA</th>
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<td>Vicinity of Electron Gun (A)</td>
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<td>Camera/ Viewing Chamber (B)</td>
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<td>Specimen Chamber/ Changer and Column (C)</td>
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<td>High Voltage Tank &amp; Cable (D)</td>
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<td>Under Console (E)</td>
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<td>Operator Seating Area (F)</td>
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<td>Junctions btw. Column Sections (G)</td>
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<td>Power supply cabinet (H)</td>
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<td>Attachment Joints (I)</td>
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For measurements:
Use NaI probe to find leaks. If leaks found, measure with ion chamber.
All measurements must be < 0.5 mR/hr at 5 cm from the surface of the unit

☐ At start up of the microscope from cold
☐ Operate in “worst X-ray leak” condition, i.e. highest acceleration voltage, highest beam current
☐ The beam at cross-over on the specimen, low magnification (500 x)
☐ All apertures removed
☐ Monitor opposite each aperture as it is inserted into the beam off center
☐ Effect of a close up blocking of the beam with copper mesh grid bars near specimen chamber
☐ If metal foils studied, effect of inserting highest atomic number specimen
☐ Manufacture date before 1956?
☐ “Caution X-rays” labels posted
☐ State “Notice to Employees” Posted
☐ All users have read Safe Use Guide

Surveyor(s) ____________________________________________
eg – electron gun
C – condenser
S – specimen port
O – objective lens (& specimen holder)
Vp – vacuum pump
It – intermediate tube (& bellows)
P – projector lens
Vc – viewing chamber
Pc – photographic chamber
Radiation Survey of Electron Microscopes

A radiation protection survey is performed to see that the X-ray exposure rate is less than 0.5 mR/hr at 5 cm from all external points of the microscope, including column, high voltage cable, high voltage tank, console (top, sides, and beneath), and power supply cabinet. With experience in cross-correlating the radiation detector and the radiation calibrator, it should become possible to rely on only the estimates of leaks given by the detector. Particular attention is paid to the areas immediately opposite the operator (the viewing chamber and the potchamber).

The instrument is operated in its “worst X-ray leak” condition, i.e. highest acceleration voltage, highest beam current, the beam at cross-over on the specimen, low magnification (5000x), all apertures removed, and at start-up of the microscope from cold. The test should include monitoring opposite each aperture as it is inserted into the beam off center. Also the effect of a close-up blocking the beam with copper mesh grid bars should be measured near the specimen chamber. If metal foils are being studied, the effect of inserting the highest atomic number of specimen should be tried.

Recommended to use a high Z target and the normal kV for such a target to reduce possibility of damaging the EM.